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**RELIABILITY FACTORS FOR ELECTRONIC
COMPONENTS IN A STORAGE ENVIRONMENT**

By

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FINAL TECHNICAL REPORT
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20. these environmental stresses are discussed in terms of mechanical, thermal-mechanical, chemical and thermal effects. Further specific discussions are included on hermeticity, polymers, oxides and statistics. The conclusions and recommendations include several items which suggest changes in component reliability policies and recommendations are made for future research efforts in storage reliability. An extensive bibliography is included.

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Redstone Arsenal, Alabama 35809

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SUMMARY

The long-term reliability of U.S. Army tactical missile systems in storage should differ significantly from that of other non-missile systems, which are normally operating systems. While much information has been accumulated on the failure rates and reliability physics of operating electronic components, relatively little knowledge exists on the behavior of similar components in storage. The overall MIRADCOM storage reliability program has as one of its objectives the development of methods for predicting the reliability of missile systems that may be in storage for periods greater than five years. One approach would be to use statistical methods to establish predictions based upon failure rate data accumulated from field experience on older missile systems in storage or the storage failure rates of discrete components kept "on-the-shelf" for a number of years. The other approach, that we have basically pursued in our program, is to focus on the failure mechanisms and materials degradation processes which will possibly cause the failure of electronic components in missile systems under long-term storage conditions. The limitations of the statistical method are: 1) there are insufficient failure rate data to make reliable predictions; 2) the different electronic technologies associated with the wide range of Army missile systems introduce an unknown factor when predictions based upon failure rate data from one technology, e.g. discrete transistors and diodes, are applied to components using a different technology, e.g. integrated circuits or MOS devices; and 3) the precise environmental conditions are most often not known or not directly related to actual missile storage environments. On the other hand, the materials degradation approach that we have used is necessarily subjective in nature and therefore will not provide numerical estimates of failure rates for devices in storage. It is suggested, however, that, from a careful assessment of the potential materials degradation processes for electronic devices in storage, alternate circuit designs can be chosen which will ensure the improved reliability of future missile systems.

The need for an advance assessment of potential materials degradation processes in storage makes it essential that a knowledge of the missile

storage environment and the associated environmental stress parameters be developed. The storage environment, as defined in this report, includes all the environmental conditions a missile will experience from the time of its manufacture until it is fired. The variety of conditions include magazine storage, air, land or sea transportation, dump storage and temporary deployment to combat units. Some missiles are expected to spend much of their storage lives in earth-covered concrete magazines, or igloos, where daily temperature variations are small. Other missiles may spend considerable time stored under field conditions involving widely varying ambient environments corresponding to their location, with extreme conditions in the arctic, desert and tropical areas. The transportation and field environments may involve wide short-term temperature and humidity variations as well as some mechanical shock. In addition, the field environment can include rain, ice, salt spray and other atmospheric pollutants. Most Army missiles are electrically dormant throughout their storage life. Even those missiles which are periodically checked remain dormant most of the time. For these systems, however, the reliability of the ground test equipment may be a contributing factor to failures in the missile's electronics systems. This particularly would be the case for electronic circuits based on MOS technology where excessive voltages or transients from the ground equipment could cause breakdown of the gate oxides. According to our findings, very little data exist which directly relate storage degradation of electronic parts to relevant missile environmental conditions or which enable correlation of device failures with the fractional storage life in each environmental condition.

The storage degradation processes are determined principally by the most critical storage environmental stresses. These environmental stresses are chemical and physical quantities which induce changes in the structure of a microcircuit and therefore include such quantities as temperature, electrical potential, chemical potential and mechanical stresses. The two primary sources of mechanical stresses in a storage environment are inertial forces and thermal-mechanical interactions. Inertial forces occur from both the cyclical accelerations associated with vibrations and the transient accelerations due to shock. Except for some of the large hybrids, most

microcircuits have very small masses so that their parts are not particularly susceptible to damage through inertial forces. Thermal-mechanical stresses are introduced due to the differential thermal expansion between materials in a microcircuit and between subassemblies and interconnections. Temperature gradients can also lead to differential expansion between different regions of the same material. An important distinction should therefore be made between slow "thermal cycling" which is normally employed in microcircuit screen tests and "thermal shock" where the temperature changes are rapid. Polymers present the potential for very severe thermal-mechanical problems and thermal shock is most important for materials having high thermal impedance, such as ceramics. Thermal shock as a screen test can cause failures of package seals. Chemical stresses may develop in microcircuit structures from a variety of potential chemical interactions. Some sources of chemical stresses are concentration gradients, residual process chemicals, evolved gases, trace contaminants, moisture, environmental gases introduced through faulty hermetic seals, stress-accelerated chemical reactions and galvanic cells. Moisture has been identified as the most critical of the environmental stresses because of the corrosion processes it induces directly and because, in combination with trace contaminants, it causes the occurrence of reactions or physical processes. The establishment of allowable moisture levels in recent modifications to MIL-STD-883B of 6000 ppm for hybrid microcircuits and 500 ppm for integrated circuits represent conditions which may be adjusted downward as manufacturing methods and moisture measurement techniques are improved and when moisture-induced degradation processes and thresholds are better defined. Thermal stresses can affect the rate of chemical reactions and cause atomic diffusion. While storage temperatures are not generally high, this is not the case for high temperature screen or accelerated testing. The importance of some reactions may be greater at particular elevated temperatures than at the lower temperatures. Furthermore, certain chemical reactions may well be arrested above a temperature where the moisture is driven off surfaces. The dew point of the package ambient gas is thus a significant factor in determining whether moisture-induced chemical reactions will occur. The failures of stored devices from such mechanisms should be expected to be greater than those of operating devices where the temperatures are elevated above the storage level due to power dissipation in the device.

The physical mechanisms leading to failure in storage cannot be assumed to be the same as those normally found to cause operating devices to fail. The environment of a device in storage may be markedly different physically and chemically from that of an operating device. For example, the storage environment is normally free from the electrical stresses and the higher temperatures associated with operating devices. However, where there are similar chemical or physical driving forces the failure mechanisms for devices in storage may not vary significantly from those which occur in operating devices, but the relative importance of these failure mechanisms can be very different.

A wide range of mechanical failure processes have been identified in microcircuits. Those most likely to occur in response to the mechanical storage environmental stresses are described in detail in the report. The greatest area of concern for mechanical damage is at geometrical configurations where stress concentration occurs. Thus, the several investigations of fatigue damage due to temperature cycling include studies of aluminum interconnections crossing oxide steps. Other areas of stress concentration include metallization steps, scratches, bonds and various interfaces between ductile and brittle materials. Configurations most susceptible to thermal cycling induced mechanical fatigue damage include solder connections on printed circuit boards, polymer materials, die attach bonds, package seals, wire bonds and particular metallization regions such as at oxide steps. Complete mechanical failure is not necessary, however, for a mechanically-induced failure to occur in a microelectronic component. Mechanical defects such as voids, slip lines, partial bonds, fatigue damage, etc. can alter the electronic operation of a device and eventually result in either degradation or catastrophic failure. Predictions of reliability will require first a knowledge of how mechanical defects or damage affect the electronic performance.

Chemical failure processes which limit the long-term reliability of microelectronic components in storage result from the influence of chemical contaminants introduced either from the environment or during fabrication. Moisture is the single most important of these chemical contaminants and moisture-induced corrosion is the most critical chemical failure process.

Secondary effects of moisture include the formation of phosphoric acid in microcircuits employing phosphorous-doped passivation glass and the growth of gold dendrites in packages containing sufficient moisture and halogen ions. Other moisture-induced failure mechanisms include crack propagation in brittle materials such as ceramic seals, glass passivation layers, nitride coatings, laser trimmed resistors and the silicon chip itself. Efforts to protect parts by polymer coatings may be defeated, because moisture is transported through plastics and will displace the polymer bond.

Thermal degradation processes would ordinarily be expected to be less important for stored missile systems since storage temperatures of the electronic components are generally low compared to those of most operating devices. However, there is concern that low temperature atomic diffusion might occur, particularly at interfaces in thin multiple layer structures, and that diffusion rates will be enhanced by strain, mechanical defects, grain boundaries and contaminants in other thin or thick film composites.

While the three predominant storage stresses, mechanical, chemical and thermal are responsible individually for failure processes, the synergism of these stress factors is also expected in storage. For example, certain chemical reactions would probably not proceed under low temperature storage conditions were it not for the introduction of mechanical stresses due to temperature changes. Also, certain mechanical and chemical degradation processes are found to be accelerated by temperature cycling through the dew point of the package atmosphere.

Particulate contamination, especially that including conducting particles, represents a major potential storage failure mechanism since effective detection is often not accomplished during screen testing. Trapped conducting particles may be released during storage due to the severe vibration encountered in the transportation of a missile and they will cause problems when they short out critical interconnections or bonds. Conducting particles may be introduced into a package at many stages of the fabrication process. They include wire fragments, broken silicon chips, metallization flakes, solder balls, weld balls and extraneous metallic debris. Some success has been

achieved with glassivation and plastic conformal coatings to minimize the shorting problem. However, improved methods for detecting the particles and better fabrication procedures to reduce them to a tolerable level would be a preferable approach.

The principal conclusions and recommendations are:

- ° Army tactical missiles normally incorporate state-of-the-art electronic components as of the time of missile design. There will always be a need, therefore, for an evaluation of the potential storage failure mechanisms of the device technologies current at the design stage.
- ° The most important environmental stresses in storage are mechanical, chemical and low thermal. The synergism of these three primary storage stresses is critical.
- ° The failure mechanisms of greatest importance in storage are those related to various marginal manufacturing mistakes, corrosion processes and mechanical fracture. Moisture within a microelectronic package is probably the most important factor for both corrosion and mechanically induced failures.
- ° The hermeticity of microelectronic packages is a paramount concern for long-term storage conditions.
- ° When hybrid microcircuits employ polymeric materials for die attach, the compatibility of these materials with electronic materials or devices enclosed in the same package must be proved.
- ° Missiles placed in storage should never contain electronic parts employing polymers for package seals.
- ° Thermal shock should never be used as a screen test stress for hermetically-sealed devices intended for missiles that will be in storage for long times.

- ° Meaningful screen testing parameters need to be developed based upon the determination of stress-duration levels required for revealing well-defined device defects.
- ° Only general environmental data are currently available for the temperature, environmental gases, vibration, etc. expected in storage. There is need for specific information concerning the interior environment of a missile in storage in order to make judgments concerning future reliability factors. A carefully planned measurements program should be established.
- ° Future efforts in the storage reliability program should be directed towards determining the response of the materials used in microcircuit structures to the storage environmental stresses. This will require the application of advanced analytical techniques to the measurement of chemical, mechanical and thermal threshold levels for device degradation processes.
- ° Missile storage reliability is determined primarily by the stability, in the storage environment, of the materials used to fabricate individual components in the missile. These age-sensitive materials should be well characterized. There is a strong need, therefore, for compiling material degradation data, performing experiments on the commonly used materials and carrying out theoretical calculations.
- ° A strong electronic parts reliability program should be maintained at MIRADCOM as an essential aspect in the development of new missile systems.

FOREWORD

This program was sponsored by the U. S. Army Missile Research and Development Command under Contract No. DAAH01-75-C-0782 as a part of the overall program on "Storage Reliability of Missile Materiel" initiated by MIRADCOM in 1974. The major objective of the broad program is to establish an adequate base of data and technology that will allow cost-effective storage reliability requirements to be developed and assure that these requirements are met throughout the missile design, development, production and deployment cycle. The period of performance covered by this report is 21 April 1975 through 31 July 1977. A portion of the funding during the final phase was provided by the Manufacturing Methods and Technology Program.

We wish to acknowledge the contributions of our colleagues at Georgia Tech, Dr. John L. Lundberg, Callaway Professor, who prepared the material on polymers and accelerated testing, Chapters VII and IX respectively, and Dr. Harrison M. Wadsworth, Professor, who prepared the material on statistics in Chapter X. Mr. Richard L. Buckelew provided excellent guidance during the course of the program. Discussions with Maury Q. Bahan and Les Conger of MIRADCOM were beneficial in providing perspective on the overall storage reliability program. We also are sincerely grateful for the comments and suggestions made by the numerous individuals in industry and government whom we contacted.

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I. INTRODUCTION

The long-term reliability of U.S. Army tactical missile systems in storage should differ significantly from that of other non-missile systems, which are normally operating systems. Missile systems may be in storage for up to twenty years and for the most part they are dormant or inoperative. The basic requirement is that these systems maintain their operational readiness after long periods of dormancy under a variety of storage conditions. A large body of knowledge has been accumulated on the failure rates and reliability physics of electronic components subjected to various operating tests including continuous operation, temperature-bias tests, power on-off cycling and accelerated life tests. Relatively little knowledge exists however on the behavior of such electronic components in storage.

The broad range of missile types and the differences in the electronic technologies used limit the applicability of reliability predictions based upon statistical data on failures, yet this approach can give initial estimates of system reliability. A direct but subjective approach would consider the fundamental materials degradation processes which would potentially lead to failure of electronic components of missile systems in an extended period of storage.

The purpose of our program therefore was to evaluate the reliability factors that would be most significant for electronic components in a storage environment. Known facts on the performance and degradation of particular devices were collected and analyzed, the results of screening and accelerated life tests were examined, and the identification of failure mechanisms which would be most likely in a storage environment was made. Much of the information was assembled through numerous visits to government and industrial laboratories and discussions with key technical personnel. These visits and contacts are documented in Appendix A.

The types of missile systems and the detailed description of storage environments were essential to provide the proper perspective for our study. Moreover the different environmental stresses provide the driving forces for failure processes. These topics are considered in Chapters II and III respectively. A detailed discussion of failure processes for electronic components in storage is contained in Chapter IV. Subsequently special topics are treated in more detail in separate chapters, that is, hermeticity

in Chapter V, oxides in Chapter VI, and polymers in Chapter VII. The discussions on screening and accelerated life tests are included in Chapters VIII and IX, respectively, and a preliminary treatment of some statistical approaches to storage reliability is included in Chapter X. Conclusions and recommendations are listed in Chapter XI. The appendices include a listing of visits and technical contacts and a summary of the responses to questionnaires on microcircuit technologies sent to various industrial laboratories.

A Workshop on Failure Mechanisms of Electronic Devices in Storage sponsored jointly by Georgia Institute of Technology and the U.S. Army Missile Research and Development Command was held at Georgia Tech in Atlanta, Georgia on May 24-25, 1977. Working groups addressed four specific problem areas: thermal effects, package integrity, chemical effects and oxides. A summary report on the Workshop will be published separately.

II. MISSILE SYSTEMS AND TECHNOLOGIES

A. Introduction

The U.S. Army's programs include a wide spectrum of missile systems for tactical operations in land combat and for the defense against aircraft or ballistic missiles. These systems range from man-portable, shoulder-fired weapons to complex, surface-to-air guided missiles. Missile systems have been under development since World War II and particularly since 1950 when the Army established its rocket and guided missile program at Redstone Arsenal in Alabama. A parallel development in electronic component technology has also taken place and therefore, the electronic sub-systems of a particular missile will reflect the state-of-the-art of component development at the time when system design specifications have been completed and approved. For example, the PERSHING long range, ballistic missile originally (1958) incorporated electromechanical analog computers for its guidance and control functions. In 1970 a single digital solid state guidance and control computer replaced the older design. In PERSHING II further improvements have been made by replacing circuits which use discrete transistors with advanced circuitry employing monolithic IC's. A similar comparison can be made between the electronic components used in HAWK, developed prior to 1960, and IMPROVED HAWK, which was approved for production in 1971.

The storage reliability program has as one of its objectives the prediction of the reliability of missile systems that have been in storage for periods greater than five years. Predictions based on previous data on failures of electronic components in storage, either in missiles or as discrete parts "on-the-shelf," are likely to give only approximate statistical estimates. Furthermore, there are just not sufficient data to determine device failure rates for particular failure mechanisms or technologies. In our program we have focussed on failure mechanisms and materials degradation processes which will possibly cause the failure of electronic devices in missile systems under long term storage conditions. Based upon the basic understanding obtained, which should be supplemented by further studies of materials degradation processes, it ought to be feasible to arrive at confidence limits for the storage reliability of both current and future electronic device technologies.

The purpose of this chapter is to provide some perspective on the types of Army missile systems by giving illustrations of a few missiles and their electronic subsystems, examples of storage containers that are used, and a discussion of electronic component technologies, currently being used or expected to be incorporated in future systems. Following this overview subsequent chapters will focus on specific failure mechanisms and materials degradation processes at the component level.

B. Survey of Missile Systems

PERSHING/PERSHING II is a two-stage, solid propellant ballistic missile with a selective range capability. A truck and trailer combination carrying the missile, mounted on an erector-launcher (Figure 2-1), is capable of travelling over roads or cross-country. The missile is periodically checked out with ground test equipment and thus the reliability of the electronic components may be altered by repeated power on/off cycling or by transient voltages from the test equipment.

HAWK/IMPROVED HAWK (Figure 2-2) is a low/medium altitude anti-aircraft weapon which uses a radar homing system for guiding the missile by following the reflected electromagnetic waves from an illuminated target. A "certified round" concept is used with IMPROVED HAWK in the field; individual rounds are checked out with sophisticated test equipment and failures are subjected to complete failure analysis by the manufacturer (Raytheon). In addition, documentation is maintained on each round so that failures may eventually be correlated with the accumulated time in each of several storage conditions.

CHAPARRAL/IMPROVED CHAPARRAL (Figure 2-3) is a single-stage, solid propellant, infrared heat seeking missile used in forward areas for defense against aircraft. The basic CHAPARRAL missile is essentially the Navy developed SIDEWINDER modified for ground-to-air rather than air-to-air launch. The improved CHAPARRAL uses guidance, fuzing and warhead components designed for all-aspect encounters and is optimized for the ground-to-air role. The launcher on the tracked vehicle, as shown, contains a rotating turret that contains four missile launch rails and provides the gunner with the necessary means to aim and fire the missile at a target. The shipping and storage container for the CHAPARRAL (Figure 2-4) is

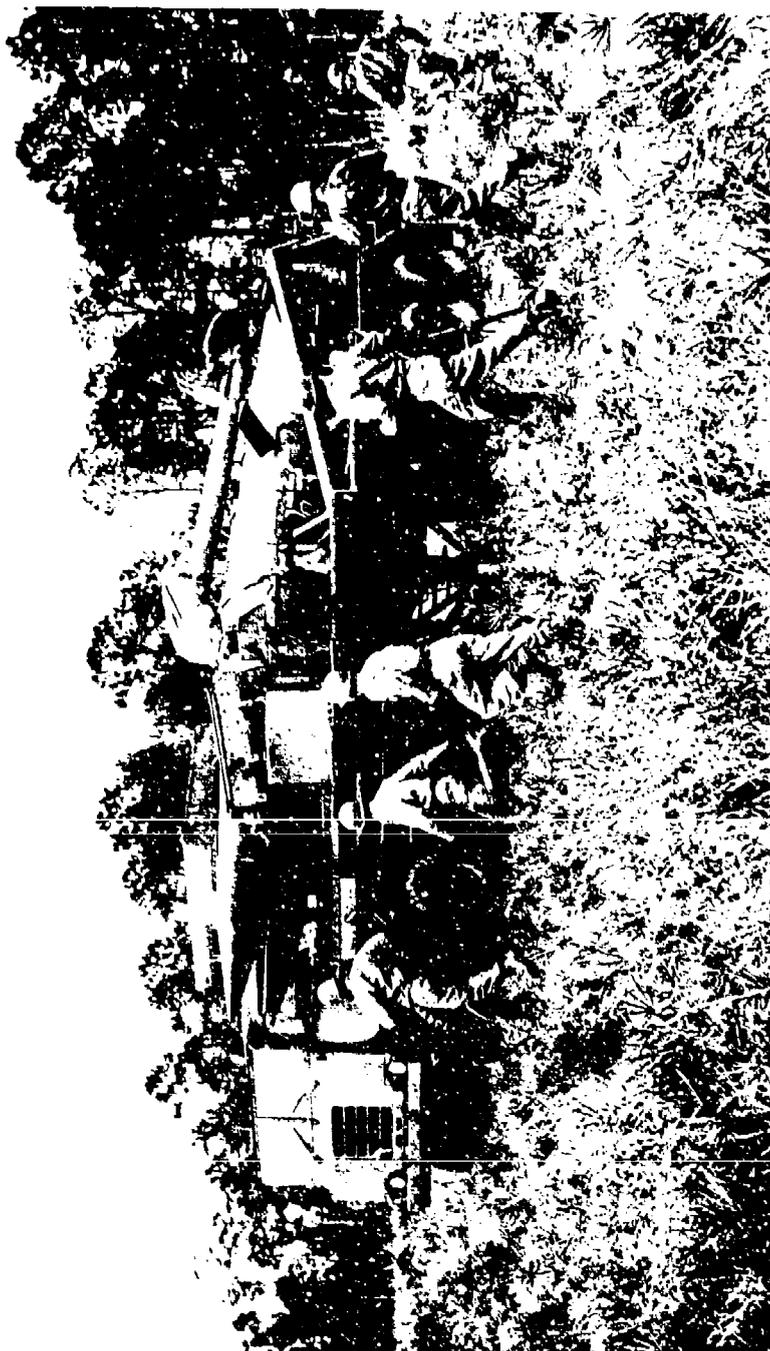


Figure 2-1. The PERSHING Missile Mounted on Erector-Launcher.

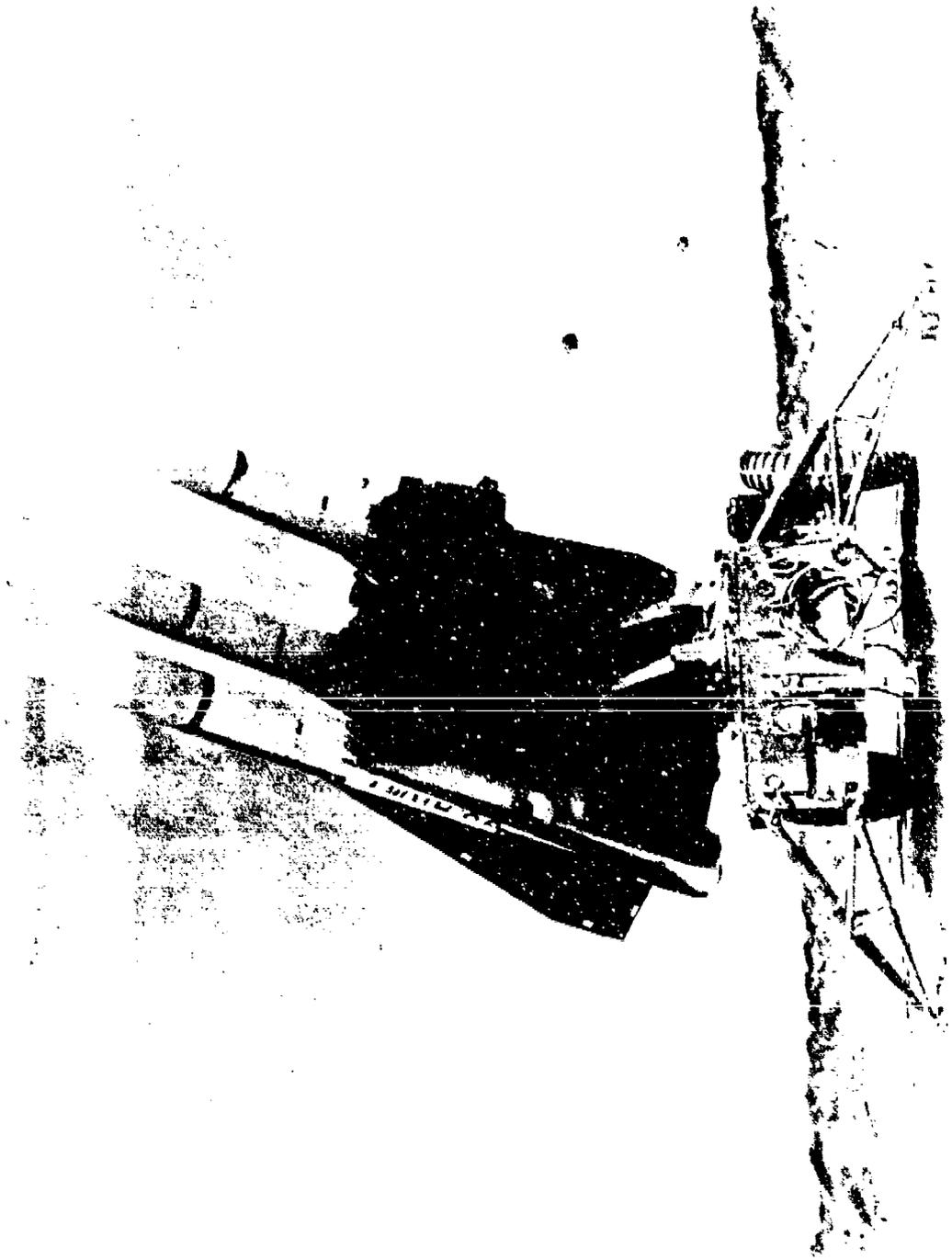


Figure 2-2. HAWK Missiles on Launcher.

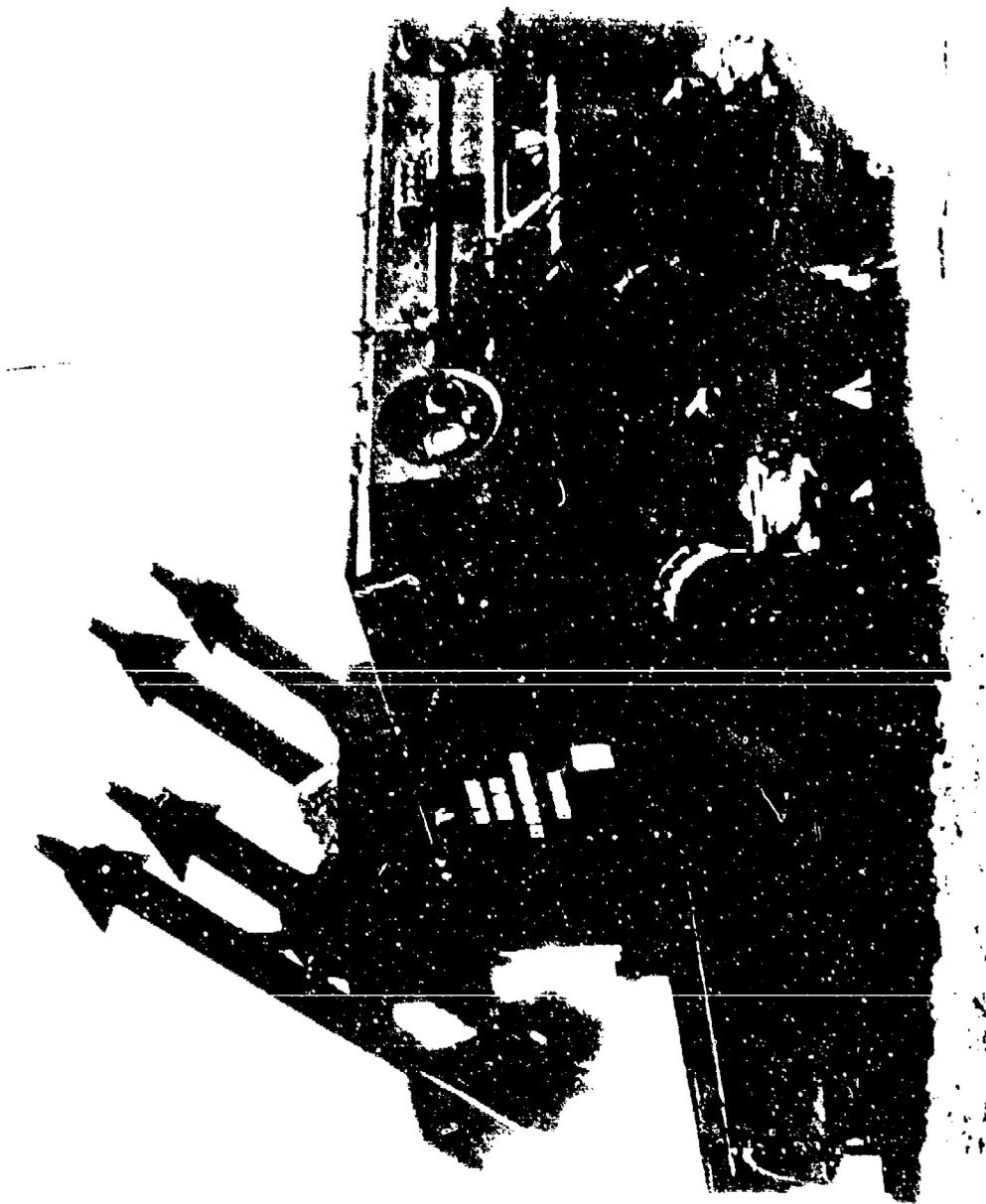


Figure 2-3. CHAPARRAL Missiles on Tracked Vehicle.



M570 SHIPPING AND STORAGE CONTAINER

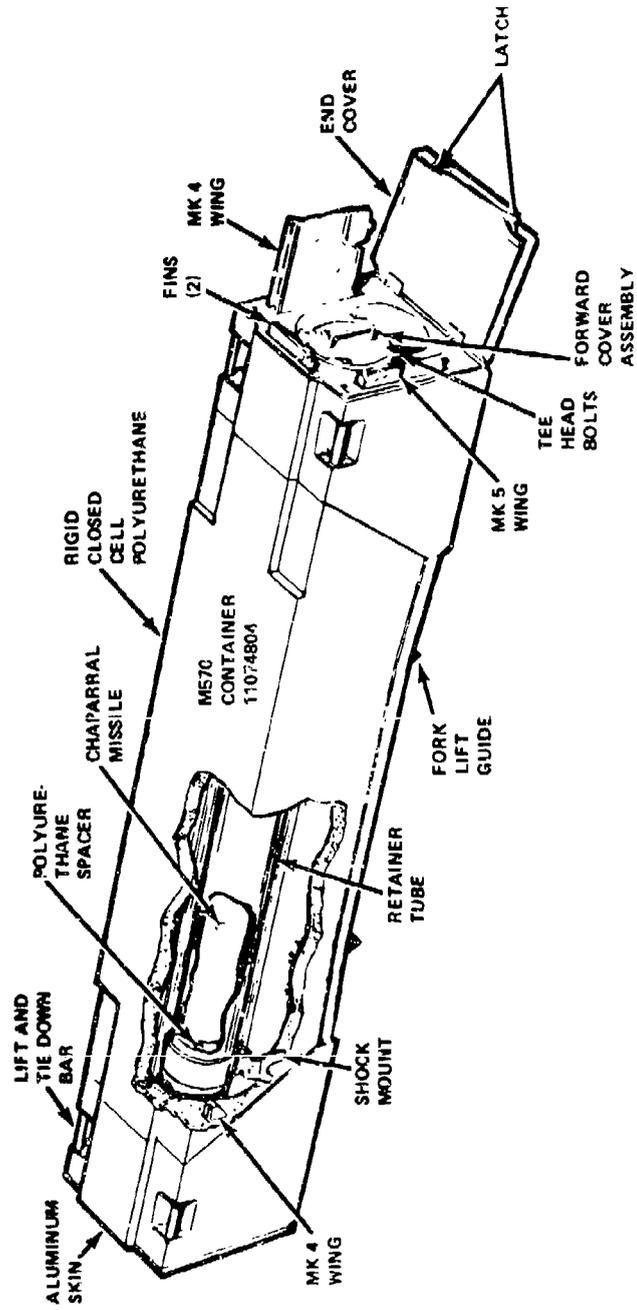


Figure 2-4. Shipping and Storage Container for CHAPARRAL.

insulated to provide a thermal lag for reducing large temperature excursions in storage.

The internal packing of the electronics in the missile is shown in Figure 2-5 and a typical hybrid microcircuit used is shown in Figure 2-6. Twenty-one hybrids are used in the guidance package of the CHAPARRAL.

TOW is a heavy anti-tank missile which is optically tracked and guided by signals sent along a wire attached to the missile. A shipping and launch container forms an extension of the launch tube when the missile is in position for firing.

STINGER is a ground-to-air missile, at present in engineering development, that uses infrared tracking and is fired from the shoulder (Figure 2-7). A view of STINGER in its shipping and storage container is shown in Figure 2-8. The arrangement of components in the missile is shown in Figure 2-9. The STINGER/Post concept (Figure 2-10) is the stacking of hybrids on a common base. A typical hybrid used on the STINGER/Post arrangement is shown in Figure 2-11. Note the rather unconventional package used.

COPPERHEAD (Figure 2-12) is a 155 mm cannon-launched guided projectile (CLGP), still in engineering development, that can be used against moving tanks or other hard point targets from miles behind the front line. This system is managed by the Project Manager, Cannon Artillery Weapons Systems, the Armament Research and Development Command. In operation, the target is illuminated with a laser beam by a forward observer on ground or in the air and the projectile homes-in on the target using the reflected laser signal.

COPPERHEAD is fired and handled just like any other round of ammunition and therefore requires no special treatment or preflight testing. Like other ammunition it will be stored either in the open or under cover, in all climates. The rounds must survive the shock, vibration and handling of self-propelled howitzers in addition to the environment typical of both tracked and wheeled ammunition supply vehicles. The "wooden round" concept is used with COPPERHEAD, as with other missile systems, since no testing is envisioned during storage or prior to firing.

The missile electronics contains approximately 700 electronic parts and a number of plastic-encapsulated integrated circuits. Accelerated

CHAPARRAL MISSILE ELECTRONICS

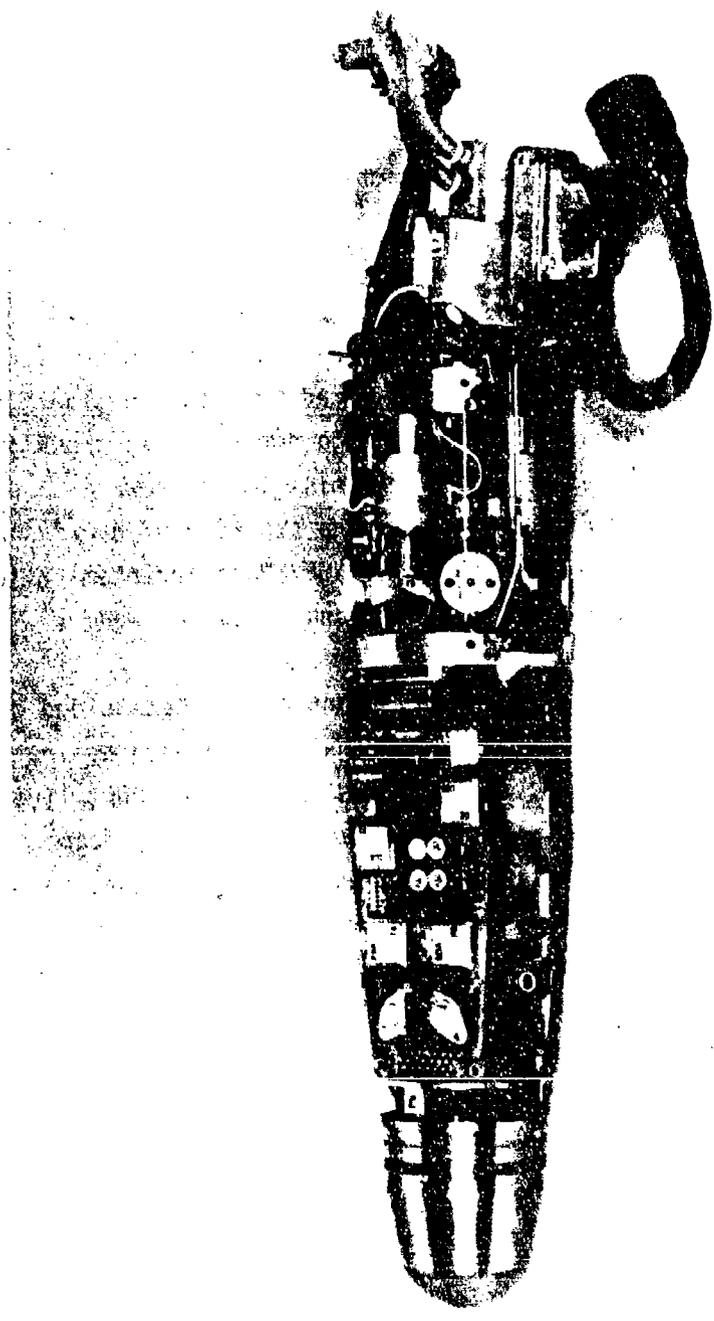


Figure 2-5. CHAPARRAL Missile Electronics.

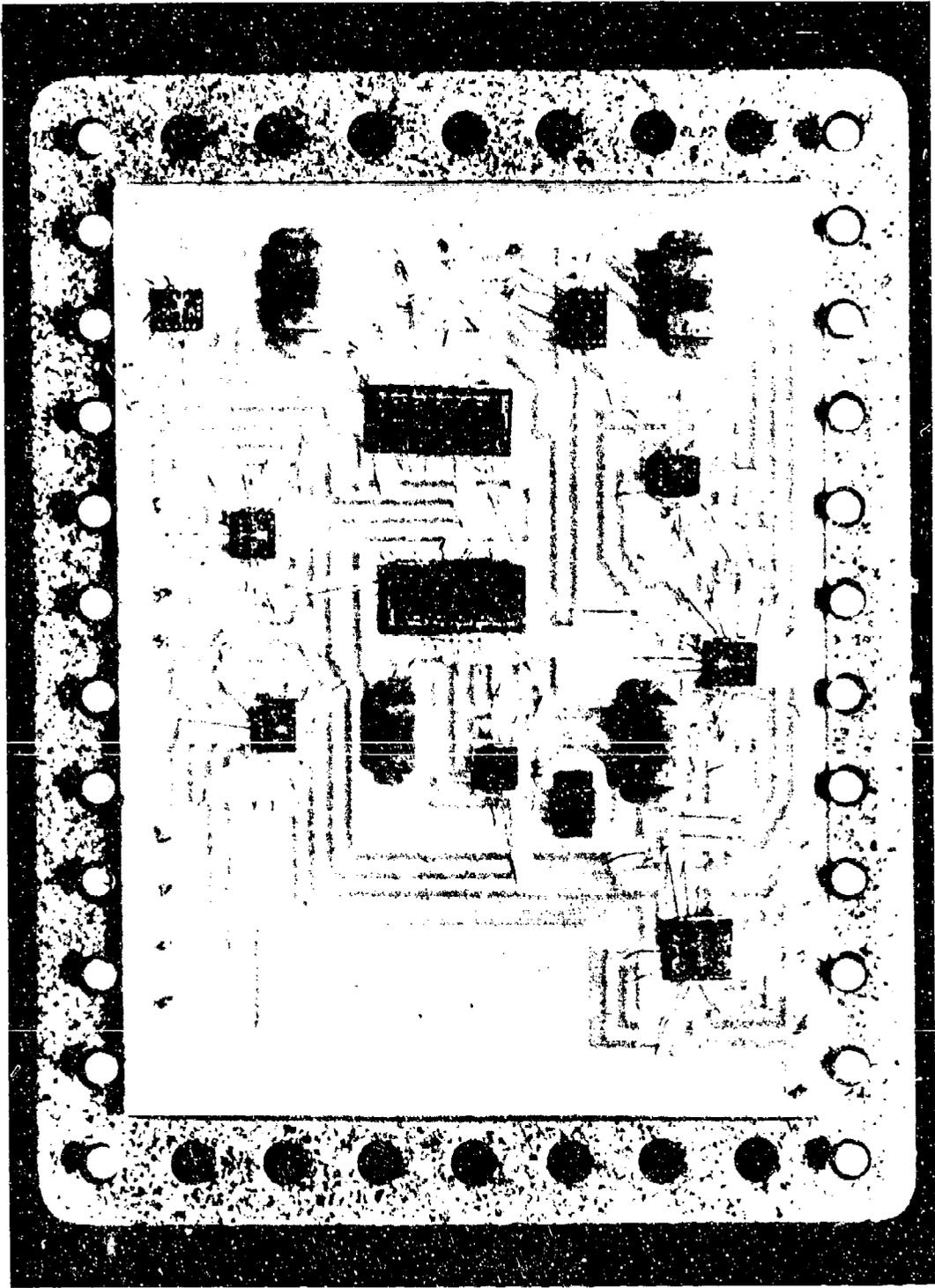


Figure 2-6. CHAPARRAL Hybrid Microcircuit.



Figure 2-7. STINGER.

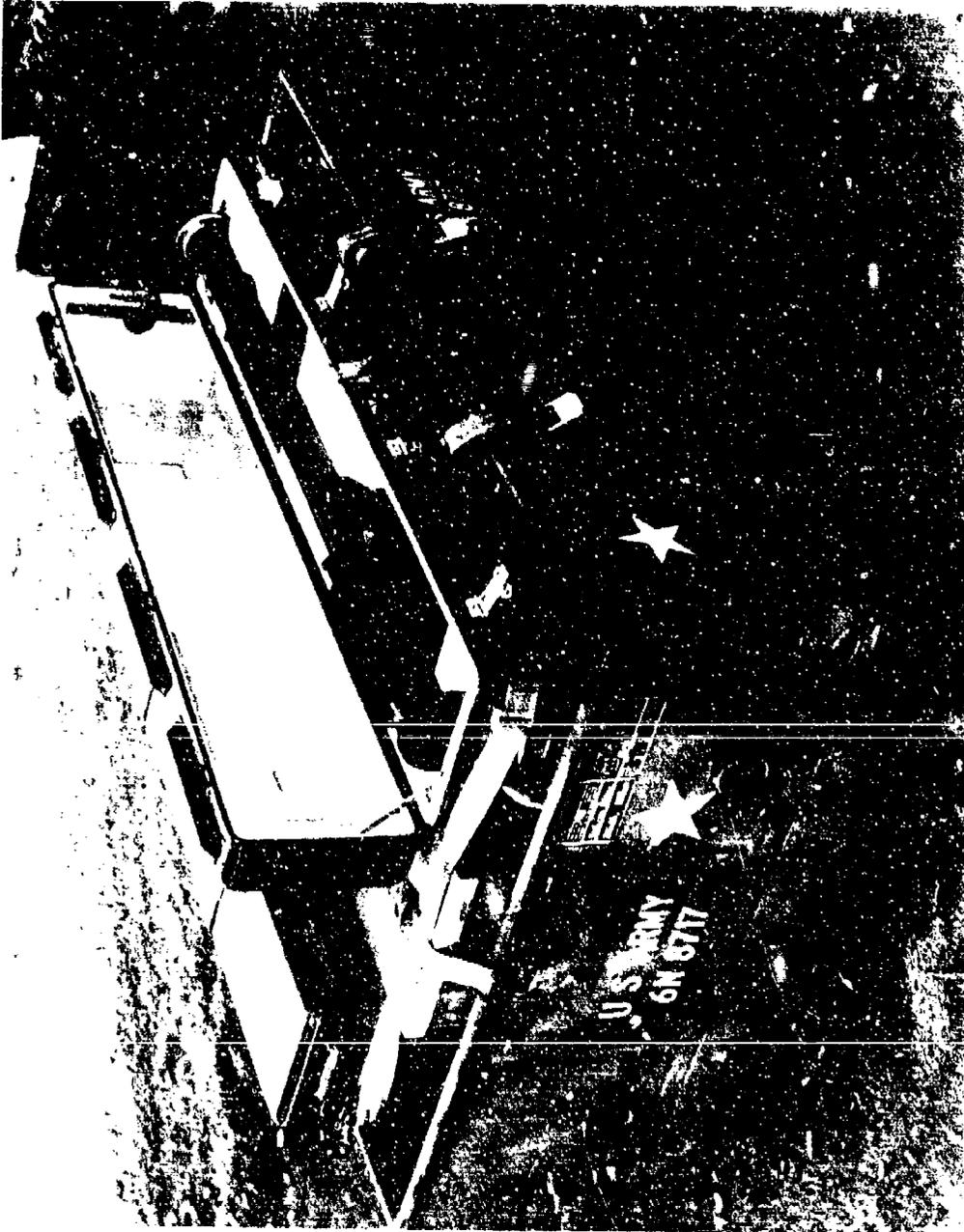


Figure 2-8. STINGER in Its Shipping and Storage Container.

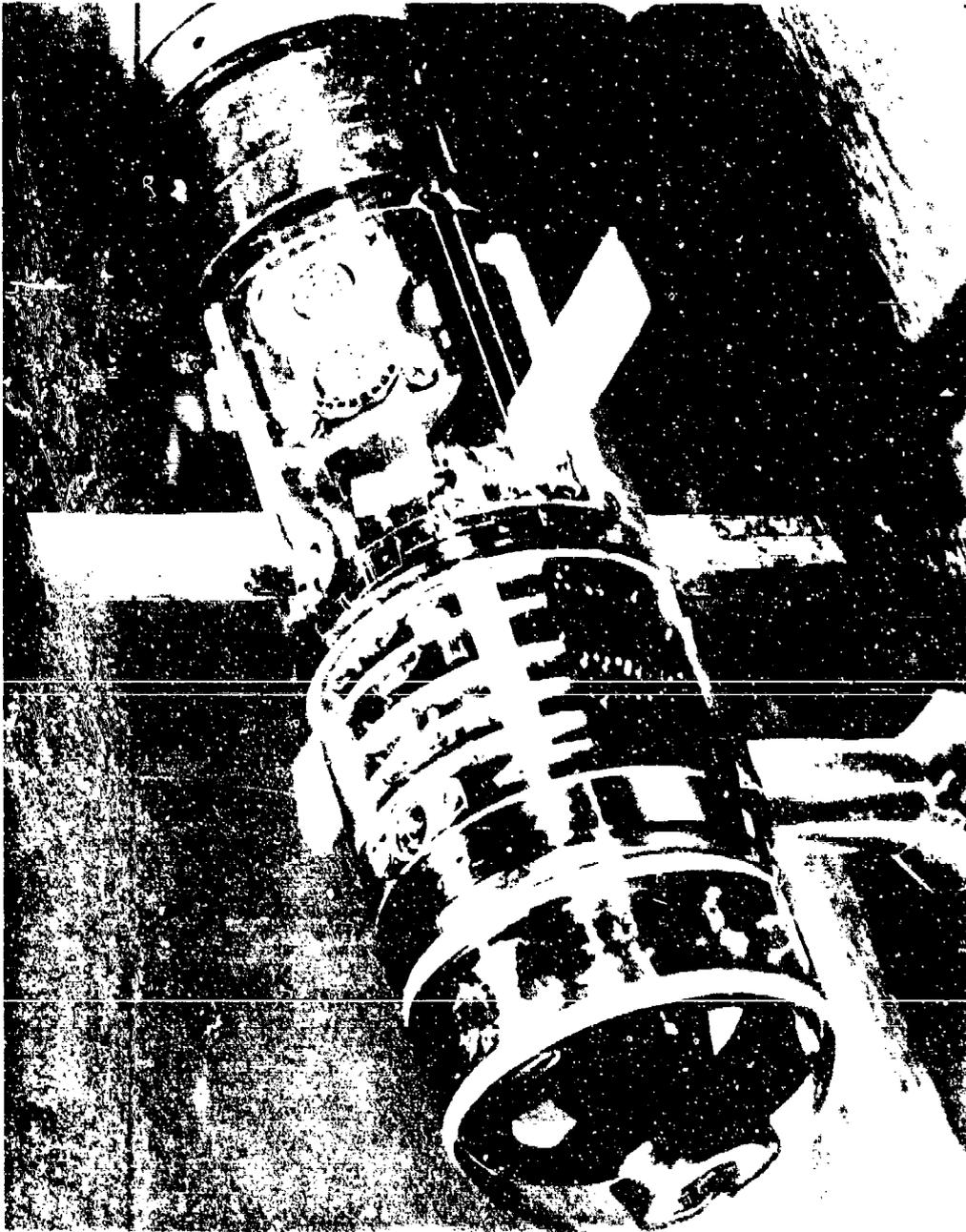


Figure 2-9. STINGER Missile Electronics.

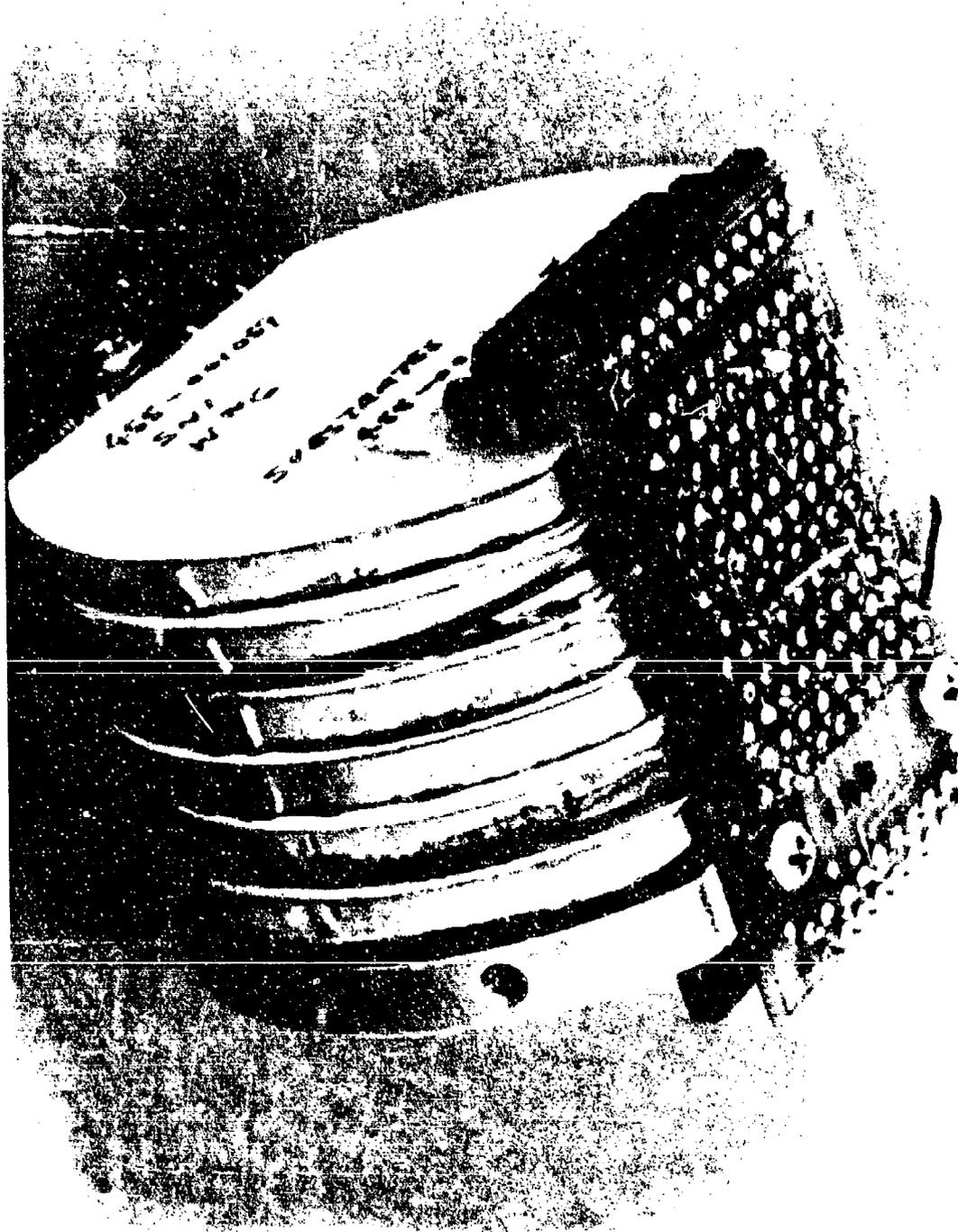


Figure 2-10. STINGER/Post Electronics.

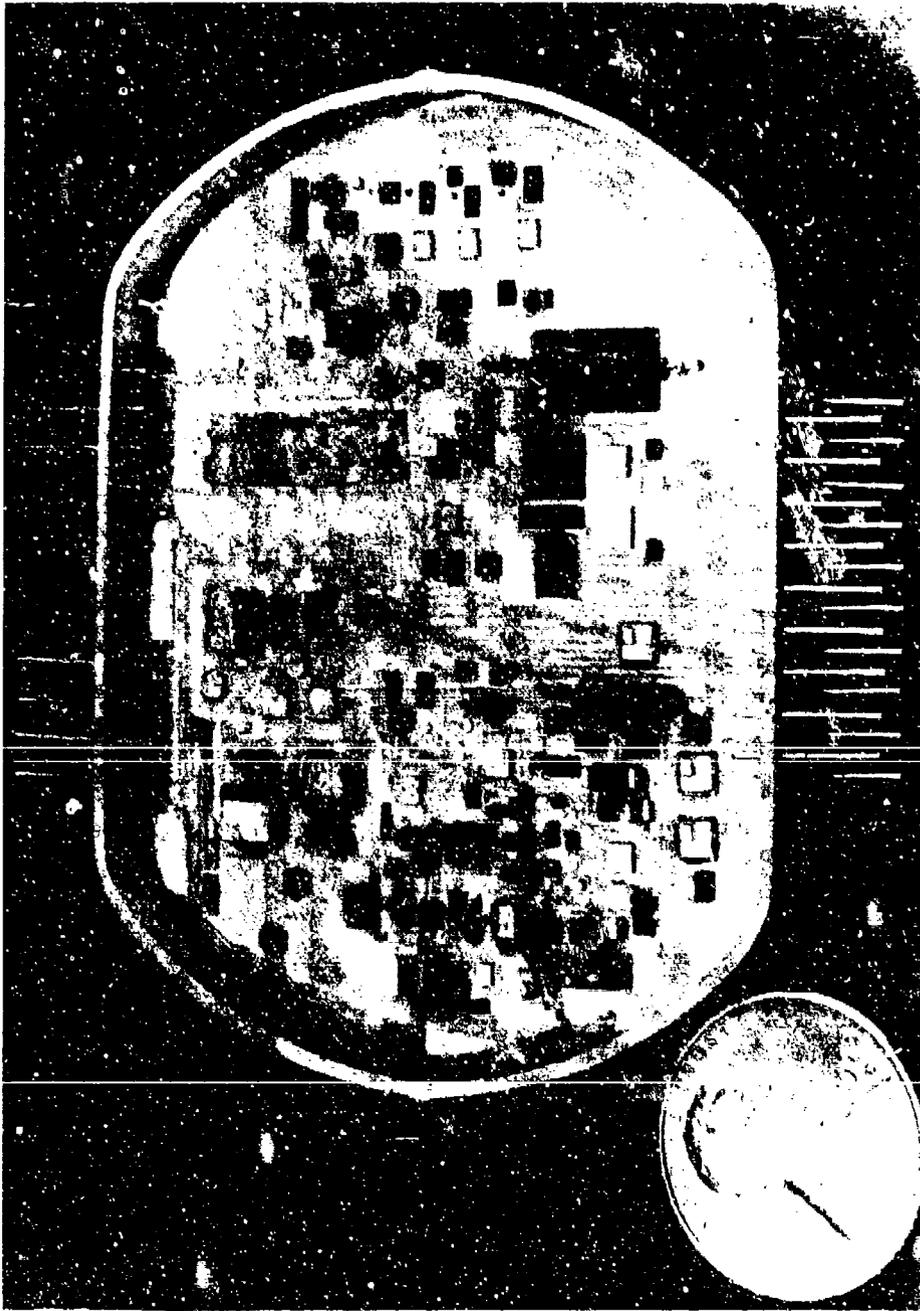


Figure 2-11. STINGER/Pest Hybrid Microcircuit.

COPPERHEAD (CLGP) PACKAGING CONFIGURATION

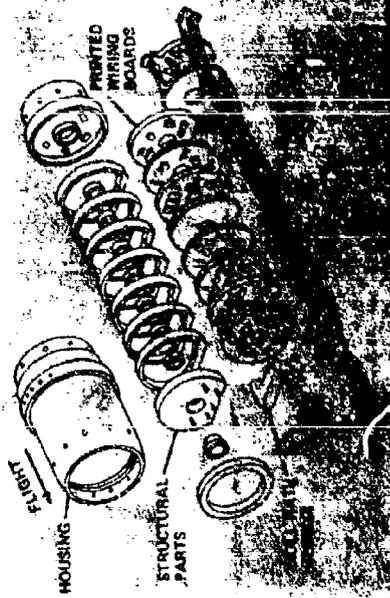


Figure 2-12. COPPERHEAD Missile Electronic Package Assembly.

tests of these parts at high temperature and humidity are planned as well as real-time aging tests in which the parts are to be sealed in plastic bags and stored at room temperature and ambient conditions with electrical measurements made at 6 month intervals.

PATRIOT (Figure 2-13) is a single-stage, solid propellant, surface-to-air missile, now in engineering development. The PATRIOT system includes a phased array radar that can simultaneously detect and track multiple targets and issue guidance commands to individual missiles in flight. The missile uses the "certified round" concept. The guidance system is predominantly digital and the electronic packaging technology (Figure 2-14) includes discrete components, IC's with dual-in-line packages (DIP's) and hybrids mounted on multilayer printed circuit boards. The ground equipment is also packaged in a similar configuration, that is, discrete components, IC's in both flatpacks and dual-in-line (DIP) and hybrid microcircuits mounted on multilayer printed circuit boards.

The PATRIOT canister (Figure 2-15) serves both as a shipping/storage container for the missile and as a launch tube. The canister, shown in the drawing of Figure 2-16, consists of flat panels stiffened by external frames. The missile is supported by a launch rail assembly with two support positions located at the missile support frames; it is restrained longitudinally, laterally and vertically. Thermal protection to provide a thermal lag and to maintain the missile temperature below +130°F is provided by internal polyurethane foam insulation. The forward end cover permits a missile flythrough capability during launch. The aft cover is designed to blow-off at ignition due to the rocket motor exhaust. An environmental control system for the canister includes controlled breathing to limit the variations of inside and outside pressures and a desiccant system to maintain the relative humidity below 60 percent.

C. Current and Future Technologies

In the early part of our program a survey was made to determine industry opinions on trends in electronic technologies that may be relevant in the storage reliability program (see Appendix B for questionnaire used and summary of results from the survey). For example, there appears to be a trend toward the use of more hybrids in missile system electronics. The hybrids will probably include more circuit elements in larger packages;



Figure 2-13. PATRIOT Launcher With Missile.

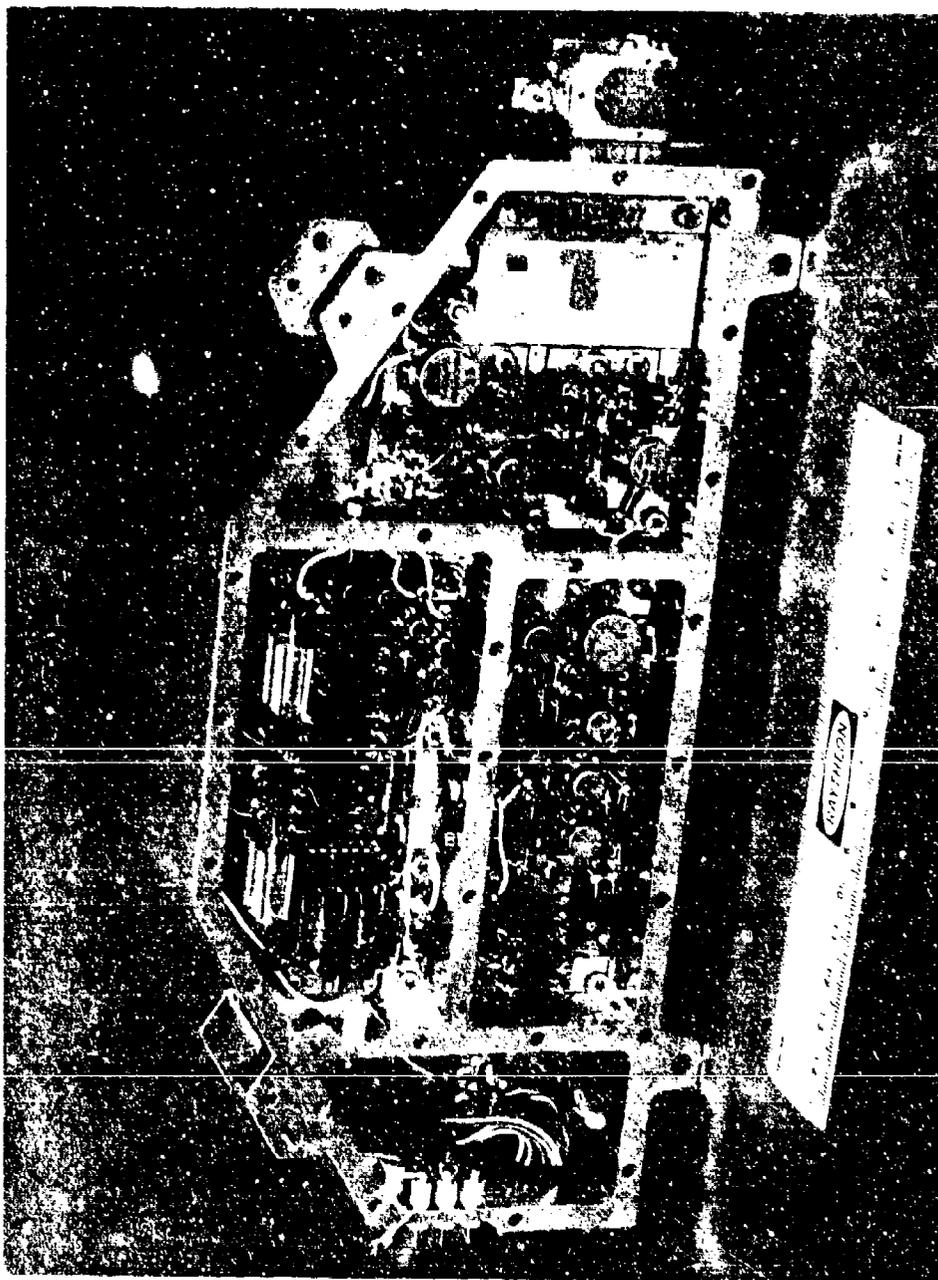


Figure 2-14. Portion of PATRIOT Guidance System Electronics.

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GUIDED MISSILE, INTERCEPT (XIMIM-104)



MISSILE ROUND



MISSILE



Figure 2-15. PATRIOT Missile and Canister.

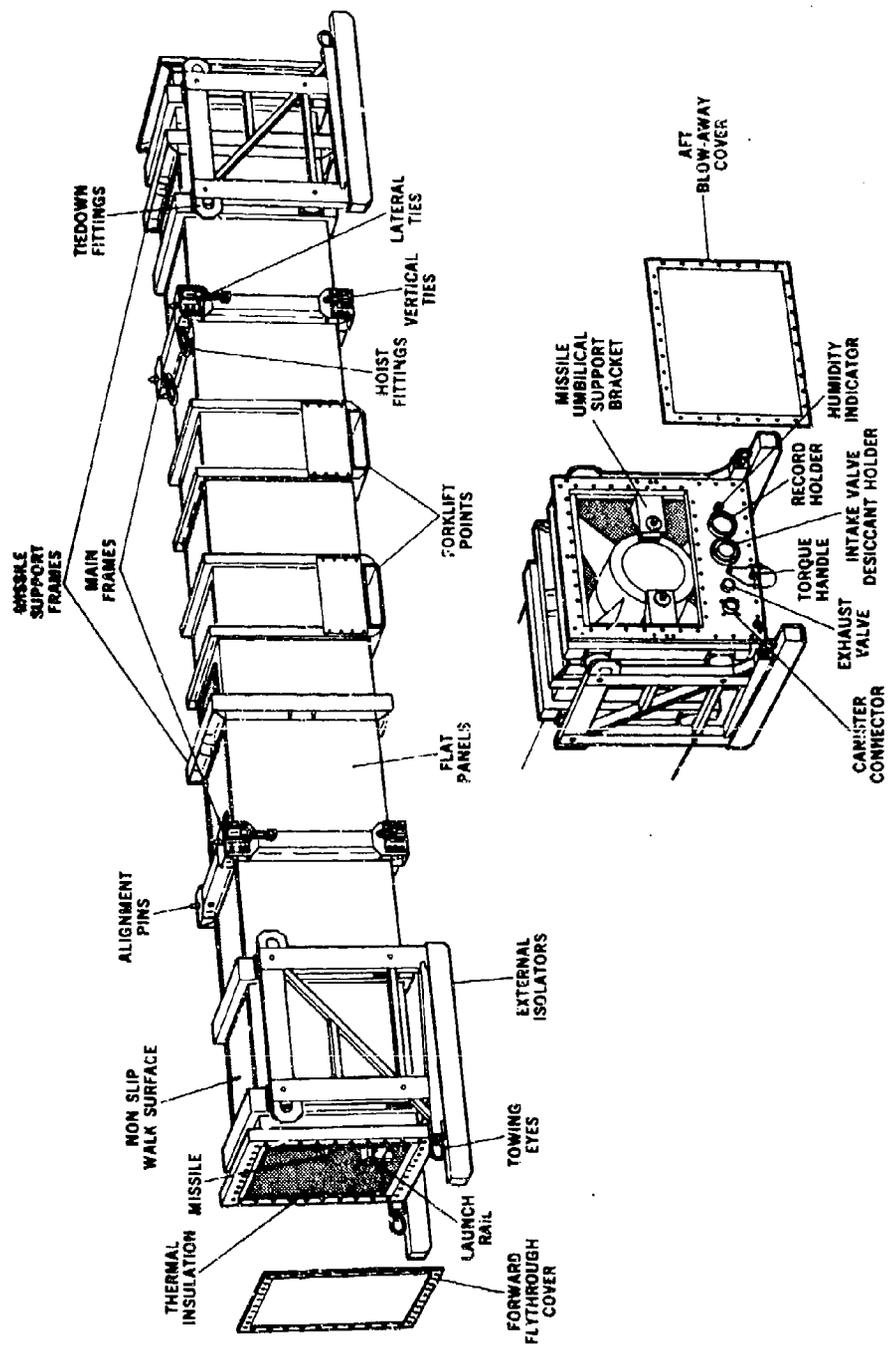


Figure 2-16. PATRIOT Canister.

however, the increased complexity and cost requires that die attachment and the bonding of chip capacitors and resistors be accomplished with epoxies to permit in-process repairs. Thick films, especially for interconnections, will predominate except in circuits where greater precision is required, such as resistive networks or microwave circuits. It is expected that the current industry use of dual-in-line packages and flat packs for hybrids will continue. For high reliability a welded seal as opposed to solder is preferable but the question of hermeticity remains as one of the most important considerations for storage reliability (see Chapter V).

An increased use of digital rather than analog circuitry is predicted. MOS/LSI components based on CMOS should see greater applications in the future; the use of microprocessors should continue to grow; and the dynamic random access memory which has progressed from 4K to 16K bits in a few years will likely be available with 65K bits in the future.

Charge-coupled device technology is being applied to reduce the size of RAM cells below the 1 mil^2 needed for 65K bit devices. Furthermore, CCD's for visible and infrared imaging and for the processing of analog signals should find applications in missile systems. For example, the Air Force MAVERICK, an air-to-surface missile, is one of several US weapons which use TV guidance systems. Replacing these systems with CCD-based systems is conceivable in the future.

III. TACTICAL MISSILE ENVIRONMENTS

A. Introduction

The environment seen by a tactical missile during storage is critical to long term reliability. The need for an advanced assessment of potential storage degradation makes it essential to develop a knowledge of potential environmental stress parameters. This topic has therefore been approached in numerous conversations with experienced missile personnel in both government and industry as well as a review of published reports. Unfortunately the environmental conditions expected during the life of an Army tactical missile system can never be clearly defined in advance. While it is generally intended that missiles will be placed in well protected magazines for storage, military needs often dictate their movement to practically any location on earth for short duration. Therefore the assessment of potential storage environments of Army missile systems must take into consideration worldwide ambient conditions.

The degradation of missile electronic parts under realistic long term storage environments apparently has never been subjected to thorough analysis. Individuals long associated with missiles state that both high costs and rapidly changing technology are responsible for this situation. It is expensive to pull missiles from storage, test a statistically significant number and conduct failure analysis on all defective or marginal parts. Funds have not been generally available for conducting the detailed failure analysis and record keeping needed to develop storage reliability information. Detailed records are maintained for certain strategic missile systems such as MINUTEMAN and TRIDENT but the controlled environments of a MINUTEMAN silo or a TRIDENT submarine are far less severe than the conditions seen by tactical missile systems. The available body of data from these systems must therefore be carefully evaluated to determine how or if it might be applied to assessing storage reliability under environments related to Army requirements. According to our findings, very little data exist which directly relate storage degradation of electronic parts to relevant missile environmental conditions. Practical methods for addressing this important need should be put into effect so that data can accumulate to guide the design of future missile systems.

B. Definition of Storage Environment

The term storage environment used here will include all the environmental conditions a missile experiences from the time of its manufacture until it is fired. The term therefore takes into account magazine storage, air, land or sea transportation, dump storage and temporary deployment to combat units. Some missiles are expected to spend much of their storage lives in earth covered concrete magazines, or igloos, where daily temperature variations are small. For example, the temperatures of missiles stored in this type of magazine at the Anniston Army Depot range between 10°C and 21°C over the year with daily temperature variations less than 2°C. However, the relative humidity in the magazines is normally above 70 percent. Certain missiles may spend considerable time stored under field conditions involving widely varying ambient environments corresponding to their location including such extremes as arctic, desert or tropical areas. Many storage locations are essentially under covered sheds with open sides. Examples of these conditions are shown in Figures 3-1, 3-2 and 3-3. All systems are subject to periodic transportation based on military requirements. While the conditions in a magazine are fairly constant, the transportation and field environments may involve wide short-term temperature and humidity variations as well as some mechanical shock. For example, thermocouples implanted in CHAPARRAL missiles revealed that components near the surface can reach maximum temperatures of 75°F while exposed to solar radiation in Arizona.¹ Daily temperature cycles greater than 50°C are therefore probable under such conditions. Alternately, the field environment can also include rain, ice, salt spray and other atmospheric pollutants.

C. Monitoring of Missile Storage Environments

Storage periods of fifteen years or more are mandatory for many Army systems. Unfortunately, a system for cataloging actual detailed storage environments is not employed for any of the Army's missile systems and therefore storage environment histories of active systems cannot be traced. While many systems can be stored primarily in the magazine environment mentioned above, others will by the nature of their mission have to be exposed to severe ambient conditions for extended periods. Extreme weather conditions therefore must be used to guide evaluations of the potential for

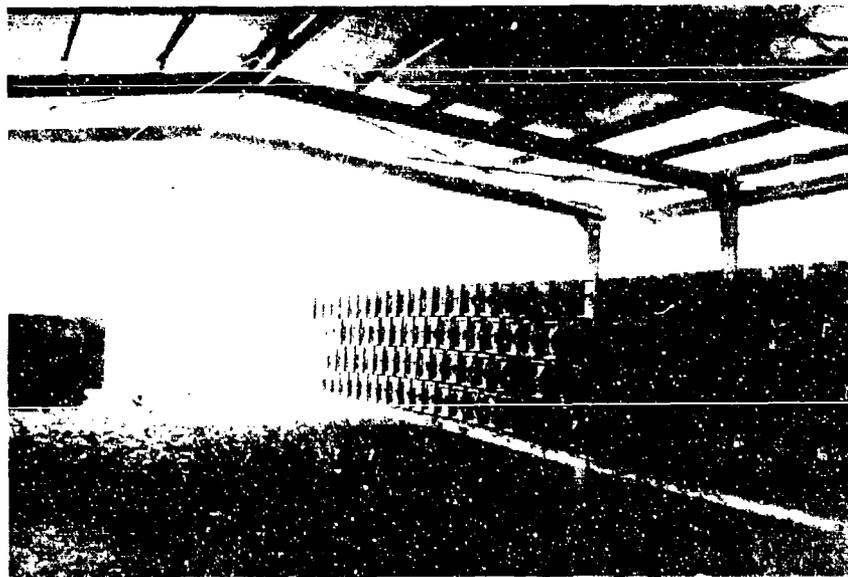


Figure 3-1. Tropical Missile Storage Under Simple Sheds. The Missiles Shown in the Upper Photograph are TOW and those in the Lower Photograph are SHILLELAGH.



Figure 3-2. Storage of TOW Missiles in Desert Sheds Within the United States.

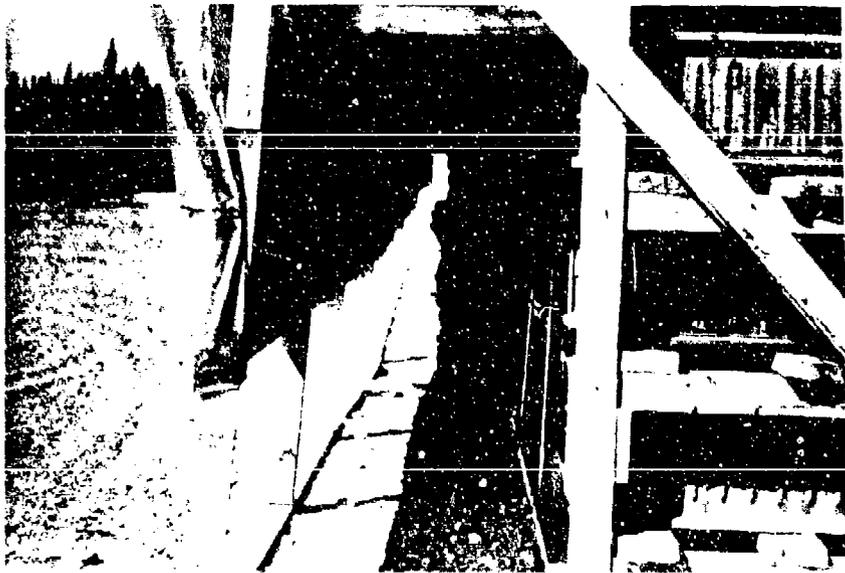


Figure 3-3. Arctic Storage Sheds for SHILLELAGH Missiles.

environmentally activated degradation processes. Use of recorded weather parameters must be done carefully since certain extreme values do not occur simultaneously and individual extremes may have a low probability for occurrence. Some of the anticipated extremes are summarized in Table A.

The data in Table A include extreme measured values for missile storage environments recorded in various published reports. It should be noted that certain of the extreme values may rarely be achieved. For example, the maximum temperature of 75°C was measured at the surface of a small missile exposed to solar radiation in the desert. The time at temperature was small. Schafer² states that the use of too wide a temperature range in design specifications "may be one of our most all-pervading errors." This is particularly true where unnecessary costly material substitutions are thereby required. A detailed analysis of the temperatures actually expected in relevant parts of the missile structure should be made prior to final design. For example, Schafer concludes from his data that the design temperature range for a missile motor grain should be more like -30°C to 55°C.³

With only two clear exceptions, the Army missiles are electrically dormant throughout their storage life. This is an essential factor in consideration of the anticipated long term reliability of these systems. The trend for Army tactical missiles has been towards CERTIFIED ROUNDS which are never tested during storage. Even those missiles which can be electrically checked remain dormant most of the time. The HAWK office estimates that their field reliability monitoring program results in a random sample of 15-20% of the HAWK missiles being subjected to operational test periods of about 30 minutes each year. A missile system's ground support equipment, however, may be operated more extensively.

A comprehensive study was made by Dantowitz and Hirschberger⁴ of field failures in an aircraft weapon system deployed in Southeast Asia and Coastal areas of the United States. Although this study concerned many systems other than missiles, it is useful here because it represents one of the few degradation surveys of modern tactical equipment. They found, for example, that fifty-two percent of approximately 46,000 field failures are identified as having been induced by environmental conditions. Temperature, vibration and moisture were the major environmental factors found to cause failures. Temperature-cycling increased the failure rate of some equipment by four to eight times that of equipment operating at constant temperatures.

Table A

Estimated Extreme Storage Environmental Parameters

<u>Environmental</u>	<u>Estimated</u>
a Maximum Temperature	+ 75°C *
b Minimum Temperature	-50°C *
c Temperature Cycling	$\Delta T \approx 70^\circ C$ *
d Moisture	Up to 100% Humidity
e Moisture	Direct Contact with Water on Exterior of Missile
f Atmospheric Pollutants	Sea Spray
g Atmospheric Pollutants	Industrial and Other Pollution Agents
h Thermal Shock	Small
i Mechanical Shock and Vibration due to Transportation and Handling	$\approx 10g$
j Bacteria, Fungus	Heavy Exposure
k Nuclear Radiation	Not Applicable
l Electromagnetic Fields	Not Applicable

* These values vary in different parts of a missile structure.

A summary of these data is given in Figure 3-4. A subsequent report by Hirschberger and Dantowitz⁵ analyzes additional data with regard to failure rates associated with operating equipment under various environmental stresses.

A significant body of information available in government documents provides a guide for specifying the environmental conditions seen by missiles in storage. Although a full review of their data is not practical within the context of this current report, a number of these documents are listed in the bibliography. The report by Durben and Smith⁶ "The Environmental Conditions Experienced by Rockets and Missiles in Storage, Transit, and Operation," contains a comprehensive collection of environmental data and references. Army Regulation AR 70-38,⁷ "Research, Development, Test and Evaluation of Materiel for Extreme Climatic Conditions," breaks worldwide climatic parameters down into the eight categories. Temperature, moisture and solar radiation extremes are tabulated for each of these categories in Table B. Comprehensive investigations of worldwide weather parameters are conducted by the Geographic Applications Division, USA ETL at Fort Belvoir, VA. Publications of this group providing useful background data relevant to missile storage environments are listed as references.⁸⁻¹³ Other worldwide temperature data are contained in a report by Stokes and Jorgensen.¹⁴ The military standard for climatic extremes are contained in MIL-STD-210B¹⁵ which lists extreme values of quantities such as temperature, absolute and relative humidity, solar radiation, rain, snow, wind and atmospheric pressure. The data contained in these weather summaries provide a basis for estimating the general environmental conditions of military equipment according to location.

The various elements which influence the temperature of a missile have been assigned the term "thermal forcing functions," by Ulrich and Schafer.¹⁶ Thermal forcing functions considered by these workers include:

- a. Direct radiation from the sun
- b. Reflected solar radiation from the atmosphere
- c. Reflected solar radiation from the ground
- d. Convective heat from or to the ambient air
- e. Heat transfer resulting from precipitation

These functions have both directional and time dependence. A missile mass exhibits a "thermal response" to the forcing function in terms of such measured thermal parameters as:¹⁶

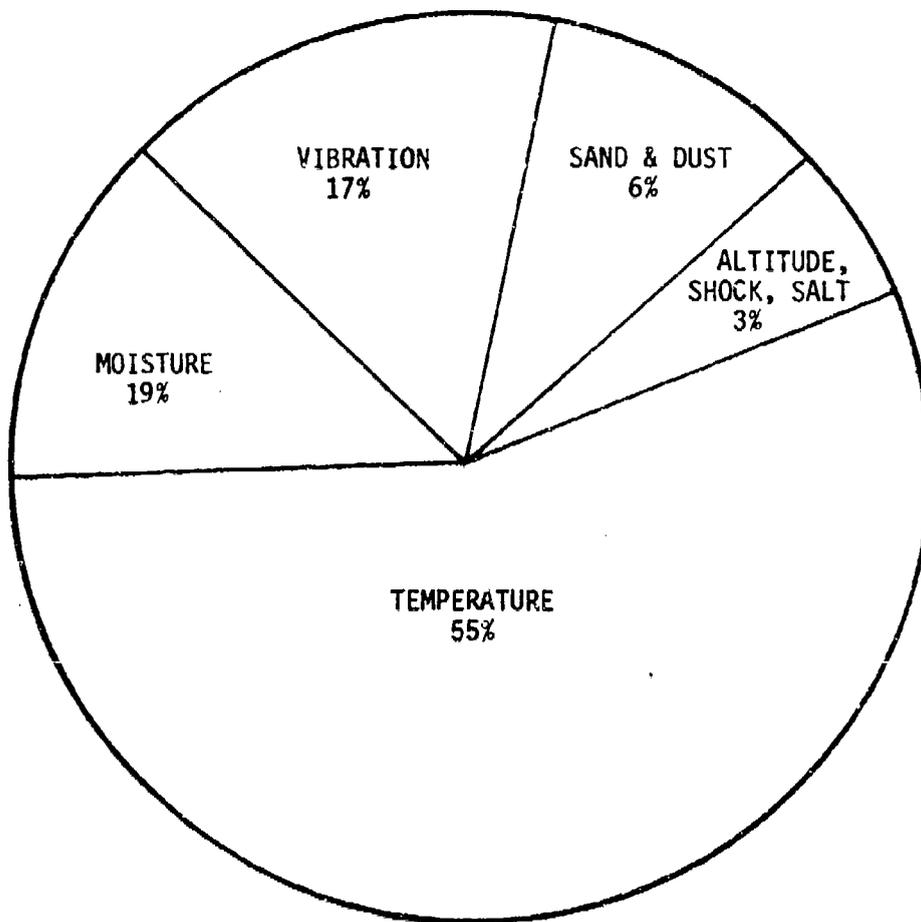


Figure 3-4. Distribution of Environmentally Related Costs
(from Dantowitz, et al., Ref. 2).

Table B. Summary of Temperature, Solar Radiation, and Relative Humidity Diurnal Extremes (AR 70-38)⁷

CLIMATIC CATEGORY	OPERATIONAL CONDITIONS			STORAGE AND TRANSIT CONDITIONS	
	AMBIENT AIR TEMPERATURE °F	SOLAR RADIATION Btu/ft ² /hr	AMBIENT RELATIVE HUMIDITY %	INDUCED AIR TEMPERATURE °F	INDUCED RELATIVE HUMIDITY %
1 WET-WARM	Nearly constant 75	Negligible	95 to 100	Nearly constant 80	95 to 100
2 WET-HOT	78 to 95	0 to 360	74 to 100	90 to 160	10 to 85
3 HUMID-HOT COASTAL DESERT	85 to 100	0 to 360	63 to 90	90 to 160	10 to 85
4 HOT-DRY	90 to 125	0 to 360	5 to 20	90 to 160	2 to 50
5 INTER- MEDIATE HOT-DRY	70 to 110	0 to 360	20 to 85	70 to 145	5 to 50
6 INTER- MEDIATE COLD	-5 to -25	Negligible	Tending toward saturation	-10 to -30	Tending toward saturation
7 COLD	-35 to -50	Negligible	Tending toward saturation	-35 to -50	Tending toward saturation
8 EXTREME COLD	-60 to -70	Negligible	Tending toward saturation	-60 to -70	Tending toward saturation

- a. Maximum surface temperature
- b. Temperature-time variation at a few discrete points on and within the body
- c. Maximum temperature gradients
- d. Average or bulk temperature
- e. Possible local or average heat flux

Part of the critical information needed for considering potential storage degradation processes in missile materials is the thermal response of the missile mass to the thermal forcing functions. Since missiles are quite complex structures which vary widely in detail from one missile type to another, Ulrich and Schafer have been developing a thermal standard¹⁶ which hopefully provides thermal response data which can be related to various missile systems. The standard mass is a simple sphere filled with a particular fluid. Sound relationships must be established between the thermal response of a given missile and the standard. Where applicable, the extensive data taken from thermal standards located around the world should guide the thermal analysis of general sections of a missile structure without having to compile comprehensive measurements on a specific missile system.

The information in the weather documents referenced above permit correlations to be made between various extremes. For example, very high temperatures and high humidity do not occur simultaneously. The highest temperature at which a 100 percent relative humidity has been recorded over the ground is 84°F and near the ocean surface only 93°F. The lowest recorded humidity of 2 percent occurred at 110°F.¹⁵ The chart^{17,20} shown in Figure 3-5 summarizes likely relative humidity values for a range of air temperatures in worldwide environments. MIL-STD-210B provides climatic extremes of military equipment in terms of the possibility that a particular extreme value will occur at a single earthwide location. The highest recorded air temperature is 136°F or 58°C. However, the 1, 5 and 10 percent extreme high temperatures are listed as 120°F (49°C), 115°F (46°C) and 113°F (45°C), respectively. The lowest recorded temperature is -90°F or -68°C whereas the 1, 5 and 10 percent extreme low temperatures are -78°F (-61°C) -70°F (-57°C) and -65°F (-54°C), respectively. These values would represent thermal forcing functions whereas the all important thermal response of a particular missile system will be determined by specific structural and materials details. The listed percentages correspond to the relative amount

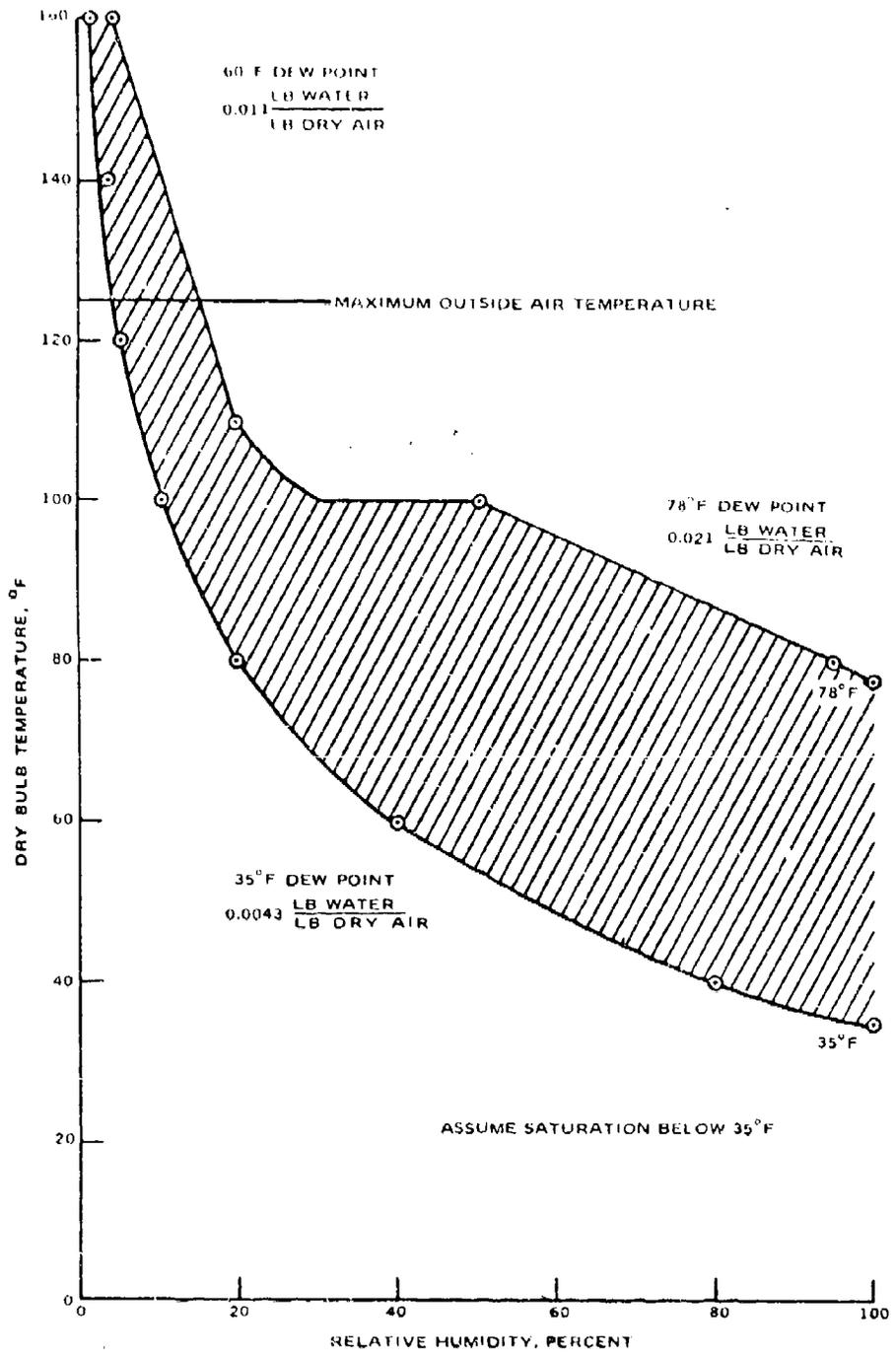


Figure 3-5. Worldwide Humidity Environment From Reference 17, Page 38.

of time the respective temperature extremes will be exceeded in an extreme environment. Equipment is not normally designed to survive extreme environmental values but rather a certain specified percentage criteria.

MIL-STD-1670(A),¹⁷ "Environment Criteria and Guidelines for Air-Launched Weapons," contains valuable design criteria for a wide range of possible environments seen by Navy missiles, much of which are of interest to the Army as well. This document lists the following situation-dependent environments:

1. Temperature
2. Humidity
3. Precipitation
4. Small Particulate Matter
5. Sand and Dirt
6. Wind
7. Pressure
8. Corrosion
9. Dissociated Gases
10. Fungus
11. Solar Radiation
12. Electrostatic

The transportation and storage situations of interest here include:

- A. Transportation and Handling
 1. Flatbed Truck (exposed)
 2. Van Truck
 3. Box Car
 4. Flatcar
 5. Handling Equipment
 6. Exposure on Deck of Cargo Ship
 7. Hold of Cargo Ship
 8. Hardstand
 9. Cargo Aircraft
- B. Storage and Handling
 1. Igloo Magazine
 2. Uninsulated Sheet Metal Building
 3. Roofed Structure with no Sidewalls
 4. Dump Storage (exposed)
 5. Dump Storage (revetment)
 6. Railroad Siding

The Naval Weapons Center at China Lake, California is an important source of actual ordnance thermal response measurements for equipment stored in a wide range of environments. H. C. Schafer and his associates have accumulated a vast amount of thermal field data during the past twenty years from stations established at representative locations around the world. Although the emphasis of the NWC effort is naturally with Navy weapons, much of their information corresponds to situations of concern to the Missile R&D Command, and was done with Army use in mind.³ They have placed instrumented Army, Air Force and Naval Ordnance under conditions approaching extreme environments and in most cases, continuously recorded internal and external temperature responses. Schafer indicated that most of their data is unpublished but they have produced a significant number of documents having information of value to Army missile personnel. Details from some of these reports¹⁸⁻²⁴ are extracted in following discussions.

D. Dump Storage

A severe storage situation occurs when missiles must be kept in an open storage dump. Dump storage is usually necessary when an Army must operate under mobile circumstances or in a new location outside the United States. In addition, the purpose of certain missiles such as HAWK requires some units to be in ready condition at all times. Solar radiation raises the temperature considerably above the ambient air temperature. The daily extremes over a one year period for the skin of a Navy SPARROW container at China Lake is shown in Figure 3-6. The skin temperature is seen to vary more than 100°F on some days. Even on cold days variations greater than 60°F are common. Daily temperatures of the skin of a SPARROW Rocket Motor are shown in Figure 3-7 for a year. A thermal inertia is associated with the heat capacity of a relatively massive rocket motor. One important consequence of a large thermal mass being located with a container is to introduce a phase difference between the temperatures of the container and the missile. The rocket itself never reaches container extremes. These effects are clearly illustrated in Figure 3-8 by Schafer's data showing hourly temperature measurements taken at the container skin, at just inside the motor and at the center of the grain.

Although these curves are not for electronic parts, the temperature, as expected, varies less as measurements are made closer to the center of

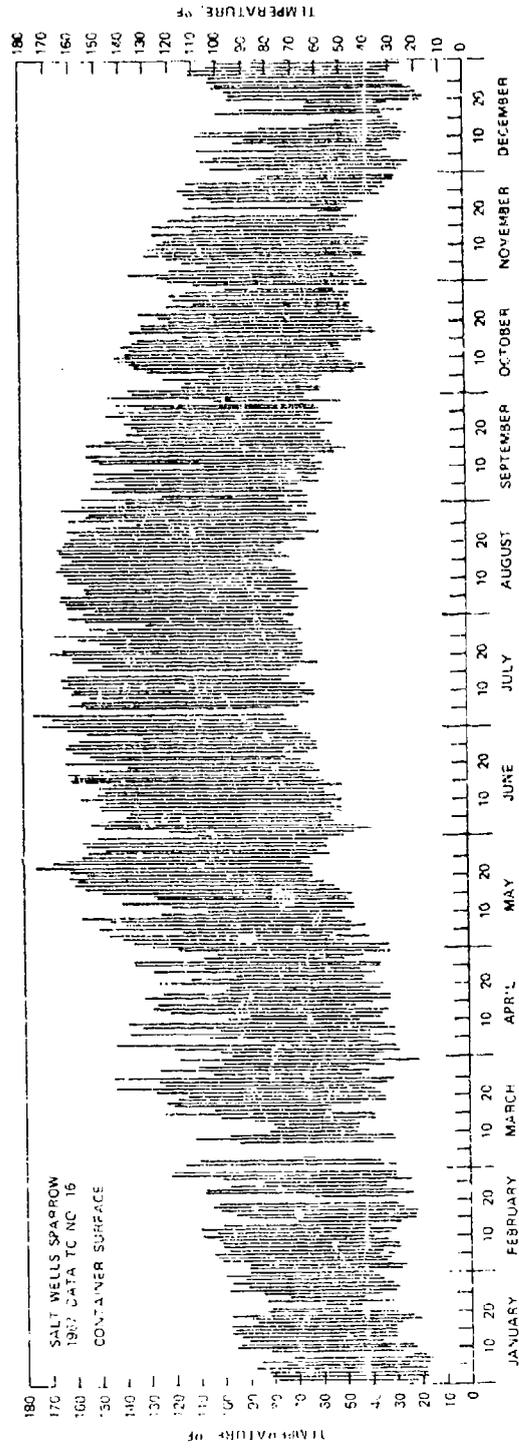


Figure 3-6. Temperature Profile of SPARROW Shipping Container Skin Dump-Stored at NWC - 1967. Reference 19, Page 44.

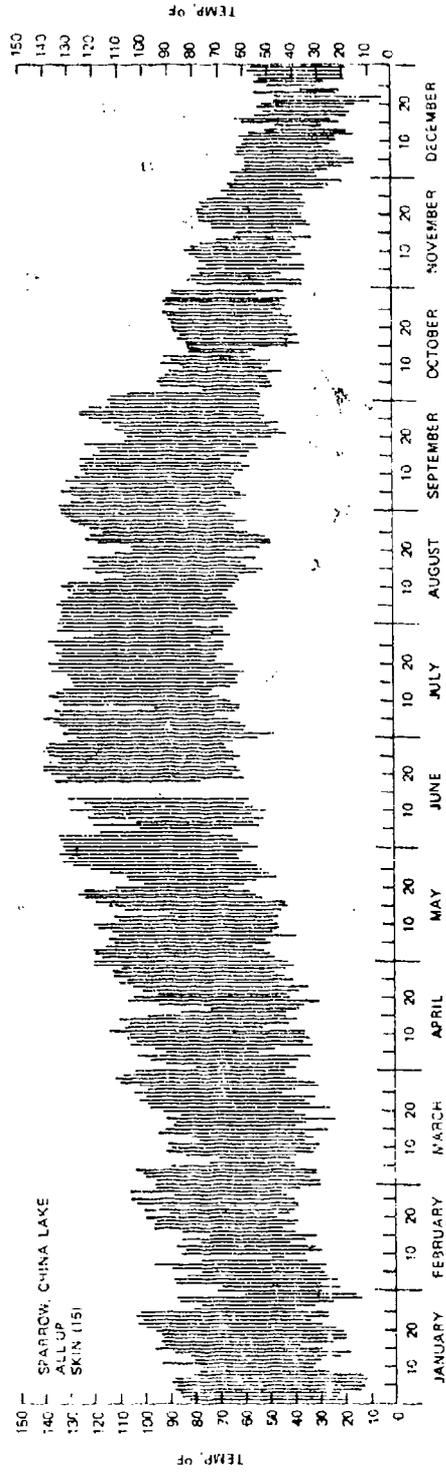


Figure 3-7. Temperature Profile of SPARROW Rocket Motor Skin, Dump-Stored at NWC as an All-Up Round - 1968, Reference 19, Part 1, Page 51.

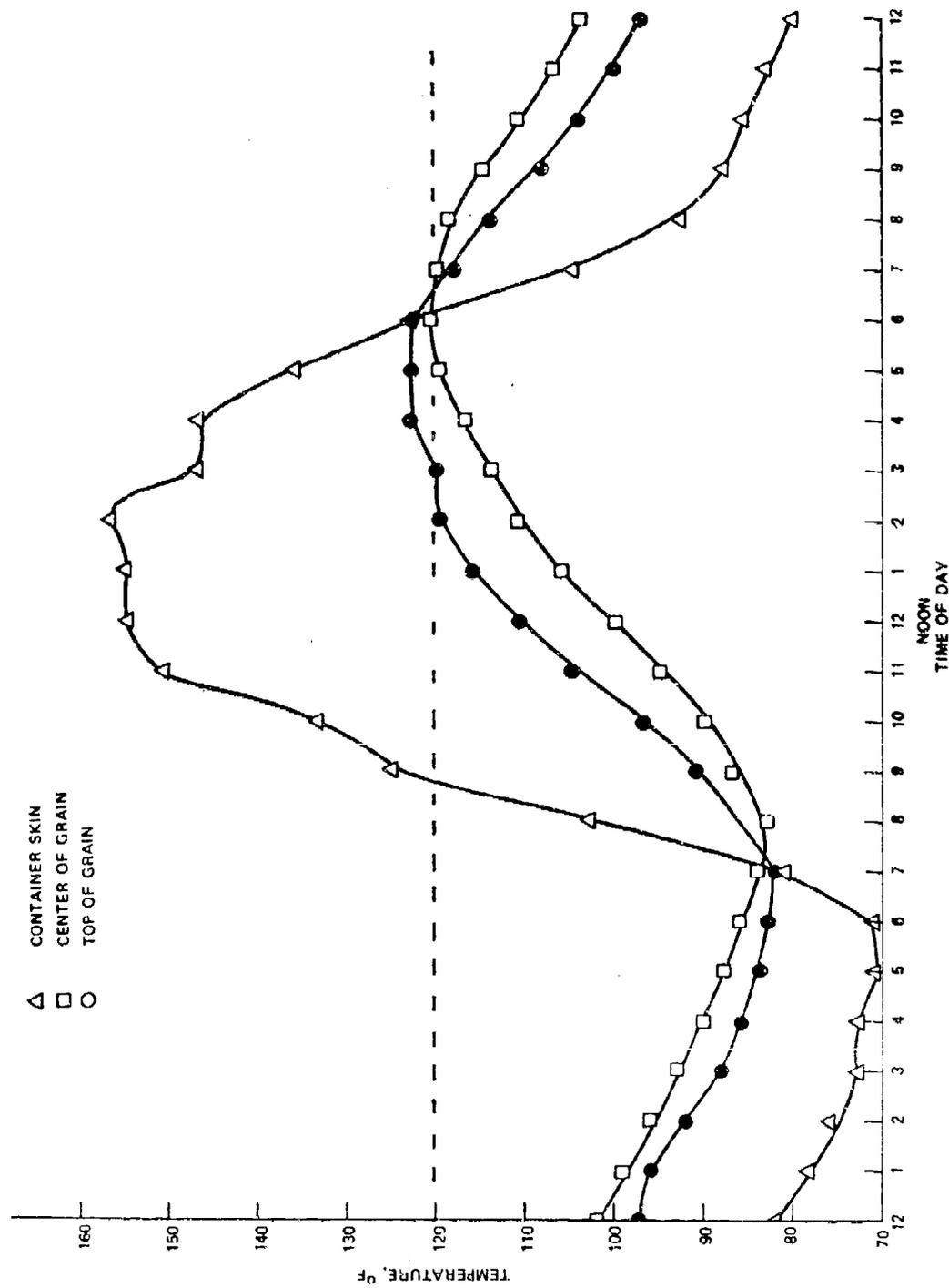


Figure 3-8. Temperature Profiles of SPARROW Motor in Shipping Container for 31 July 1969. Reference 19, Part 1, Page 46.

the mass and are representative of that expected of the guidance control package. Temperature profiles are out of phase so that the propellant achieves its maximum temperatures well after the hottest time of the day. K. K. Mitchell²⁵ of Redstone points out that the ideal situation is for the internal missile temperature to have a twelve hour phase difference with the container skin which, he noted, is achieved with the container designed for the PATRIOT missile. Schafer argues that by directing attention to the rate of heat transfer, radiation, convection, etc., into and out of the container, it should be possible to develop data and procedures for evaluating temperature profiles of ordnance based on surface characteristics and the thermal properties of the missile. The cumulative distributions over a year of temperatures of a larger (12 inch diameter) missile are shown in Figure 3-9 for a missile dump stored in the Philippines. The effect of the missiles' large thermal mass is clearly seen in a statistical manner by the relatively lower temperatures experienced within the missile mass.

Temperature profiles for CHAPARRAL missiles exposed to the environment at Yuma, Arizona have been comprehensively investigated by Mitchell.¹ Data from the skin of a CHAPARRAL mounted on launch rails are shown in Figure 3-10. A comparison of the outer skin and inner temperatures over a single day for an exposed CHAPARRAL near the ground is seen in Figure 3-11. The importance of thermal mass and the container is clearly seen by comparing Figure 3-11 with the SPARROW data of Figure 3-8. The cumulative temperature distribution for a launch rail mounted CHAPARRAL taken at various points within the electronics package is provided in Figure 3-12. A summary of Mitchell's CHAPARRAL measurements¹ showing maximum temperatures achieved on launch rails are provided in Table C.

Factors such as the paint color strongly influence weapon temperatures. Schafer compared maximum temperatures for fuel air explosive weapons painted olive drab and white. His data showed that white paint reduced the weapon temperature as much as 30-35⁰F. Similar conclusions are well represented in the China Lake data² shown in Figure 3-13.

E. Magazine Storage

Almost any type of cover will be a strong aid for protecting equipment from ambient temperature extremes. The insulation of trapped air and heat capacity of the air and stored equipment is effective for moderating

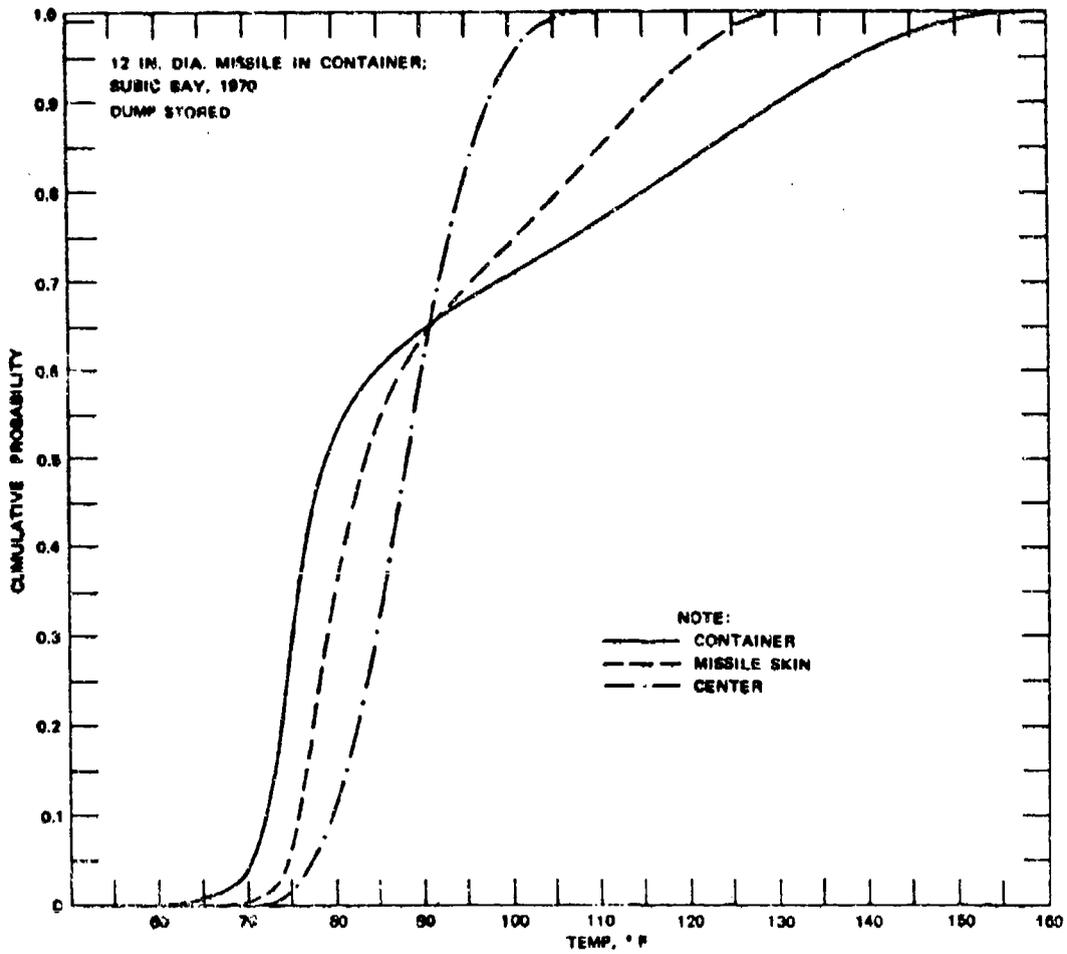


Figure 3-9. Cumulative Distribution Summary of Dump Stored ASROC Temperatures at Subic Bay for 1970. Reference 19, Part 1, Page 35.

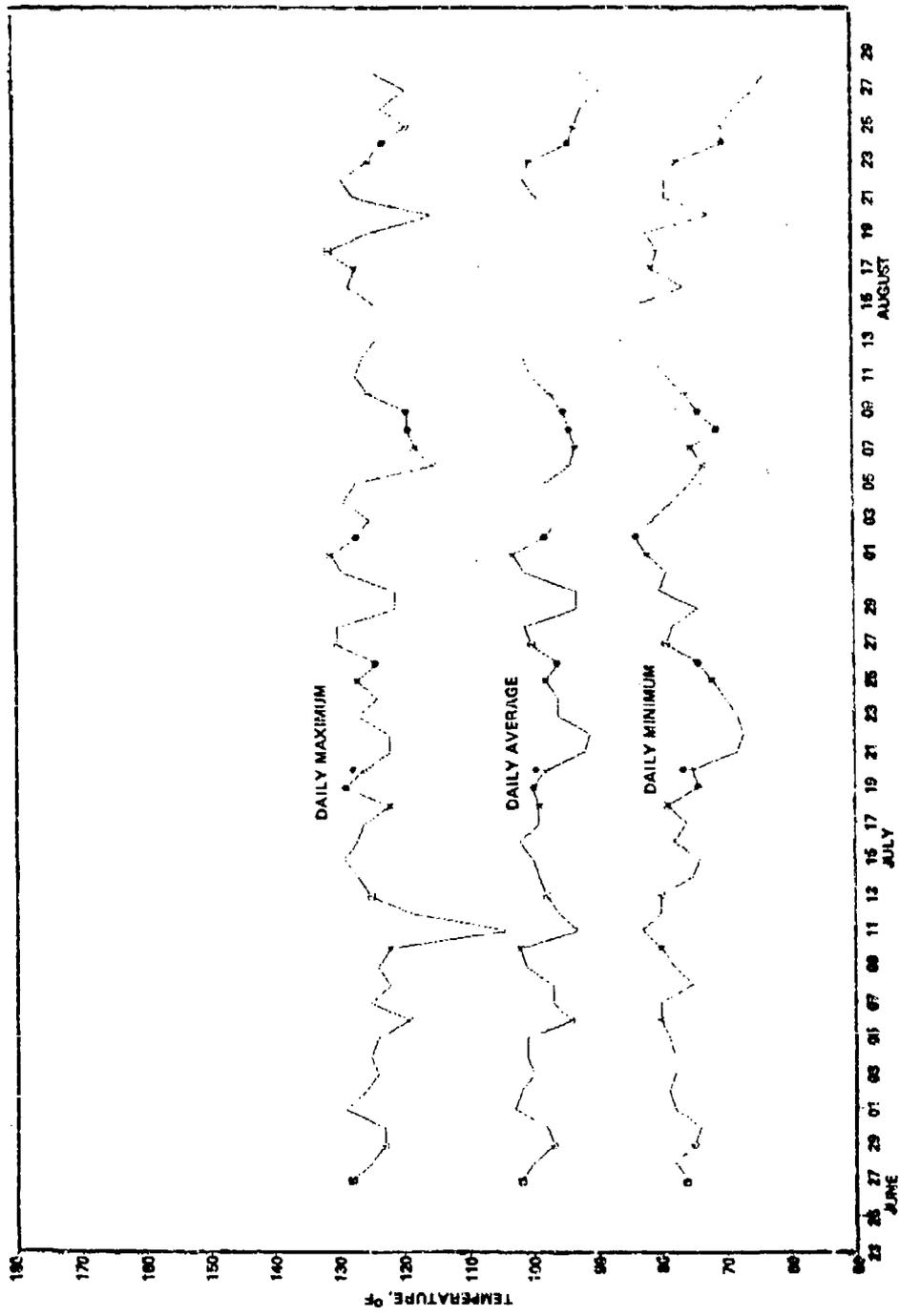


Figure 3-10. Temperature Profile of a CHAPARRAL Missile Skin, 8 Inches From Aft Flange. August 1973. From Reference 1, Page 91.

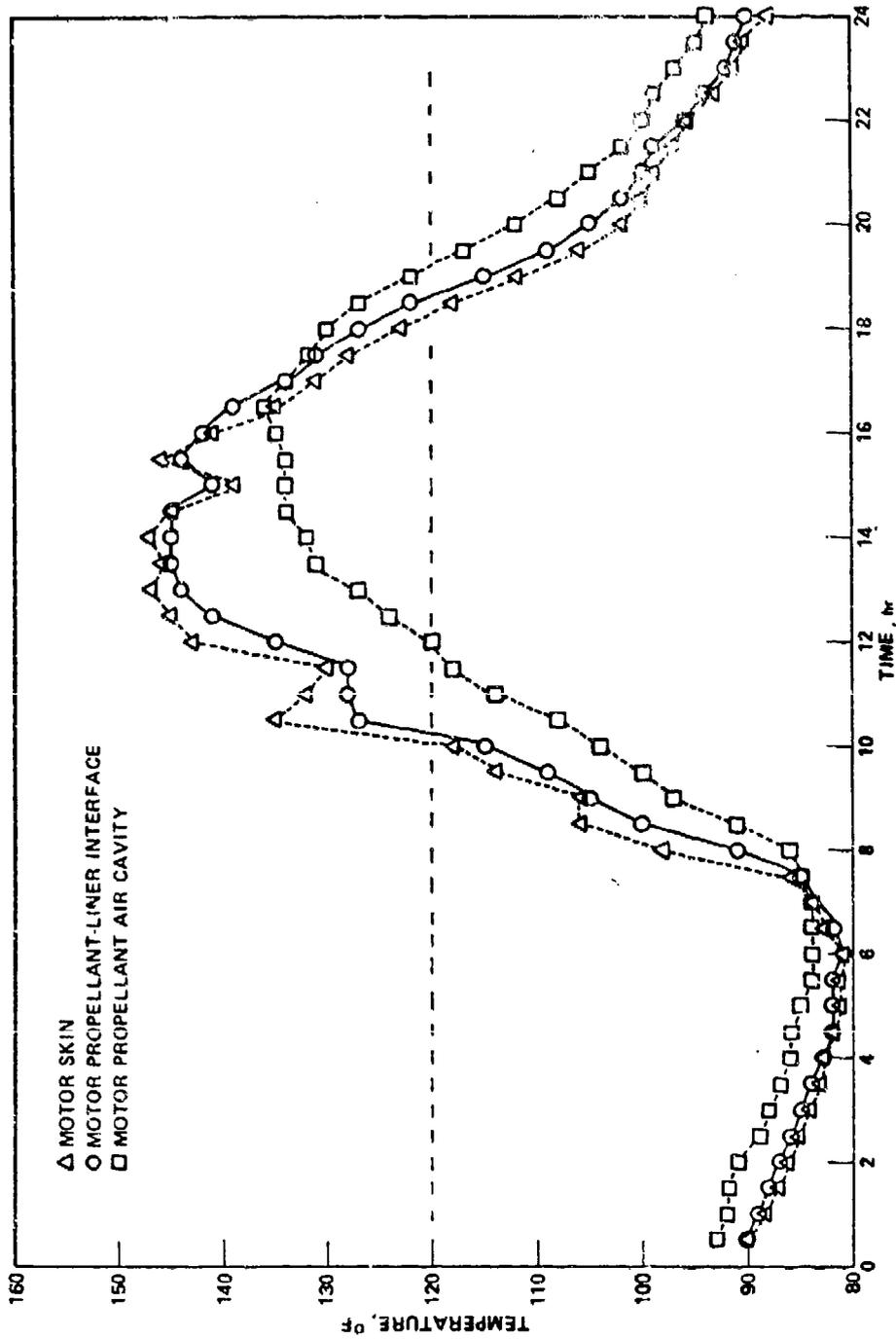


Figure 3-11. Diurnal Temperature Cycles of a CHAPARRAL Missile, 1 August 1973.
 Temperature Readings at Three Indicated Locations.
 From Reference 1, Page 143.

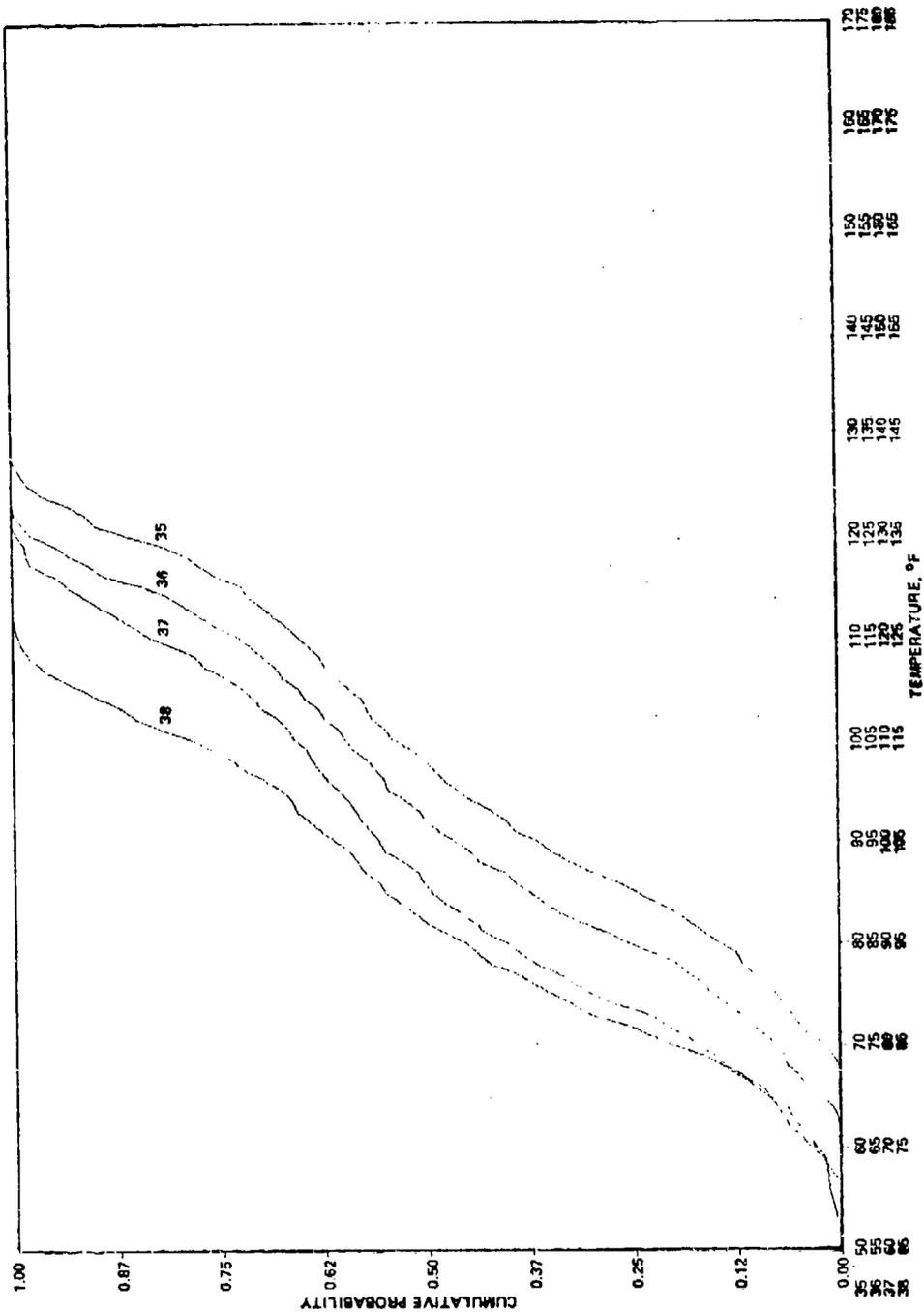


Figure 3-12. Cumulative Temperature Distribution for CHAPARRAL Missile at Four Regions: Seeker Section (T.C. 35), Precision Amp Transistor (T.C. 36), Electronic Section, Skin (interior) (T.C. 37), and Electronics Section, Skin (interior) (T.C. 38). From Reference 1, Page 164.

Table C. Maximum Temperatures Recorded for CHAPARRAL Missiles on Launch Rails, Solar Radiation at Yuma

Location	T.C. No.	Temperature (°F)	Missile No.	Launcher Position	One Date (1973)
<u>Motor</u>					
Skin	43	140	1	1	Aug 22
Propellant-liner interface	47	136	2	1	Aug 22
Propellant air cavity	51	126	2	1	Aug 22
<u>Warhead</u>					
Skin	89	132	4	1	Aug 1
Explosive-liner interface	68	123	2	2	Aug 1
Explosive center	84	122	3	2	Jul 2
<u>TDD</u>					
Skin	76	132	2	2	Aug 1
<u>GCG, Not Operating</u>					
Skin	77	131	2	2	Aug 1
Seeker sect (flask holder)	35	128	1	4	Aug 11
Precision amp transistor	36	128	1	4	Aug 16
Elect sect skin (interior)	37	129	1	2	Aug 5
Elect sect skin (interior)	38	127	1	4	Aug 11
Elect sect ambient air	39	128	1	2	Aug 27
Elect sect ambient air	40	122	1	1	Aug 18
Servo sect ambient air	41	122	1	4	Aug 11
Cylinder b c.k	42	122	1	4	Aug 11
Elect sect ambient air	16	121	1	4	Aug 11
<u>GCG, Operating</u>					
Seeker sect (flask holder)	35	175	1	3	Aug 7
Precision amp transistor	36	155	1	3	Aug 7
Elect sect skin (interior)	37	138	1	4	Jul 25
Elect sect skin (interior)	38	134	1	4	Jul 25
Elect sect ambient air	39	157	1	2	Aug 1
Elect sect ambient air	40	178	1	3	Aug 7
Servo sect ambient air	41	124	1	4	Jul 25
Cylinder block	42	124	1	2	Aug 1
Elect sect ambient air	16	149	1	3	Aug 7

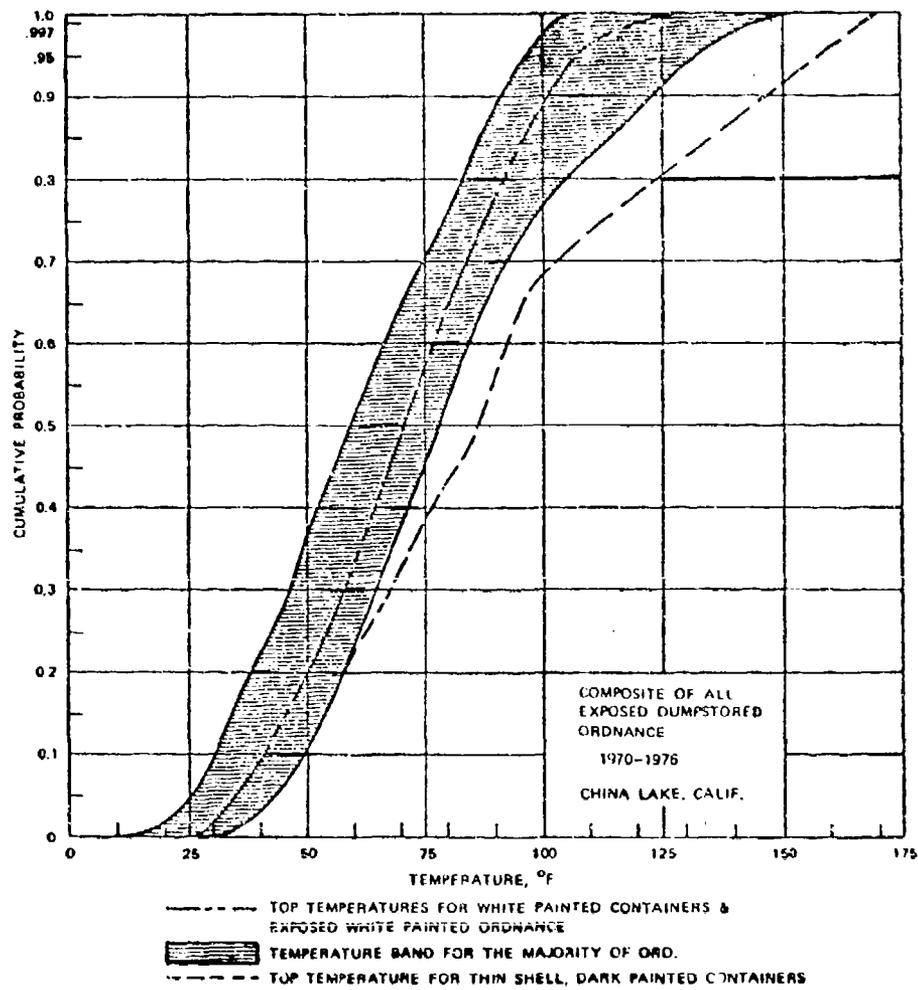


Figure 3-13. Composite of All Dump-Stored Ordnance at China Lake NWC, 1970-1976. Reference 2, Page 13.

diurnal temperature cycling. Earth covered concrete magazines are particularly effective due to the additional insulation and large heat capacity of the earth. Earth covered magazines have been shown to reduce daily temperature amplitudes within the magazine as much as 1/20 the external ambient amplitude. However, the actual internal temperatures follow the mean external values.

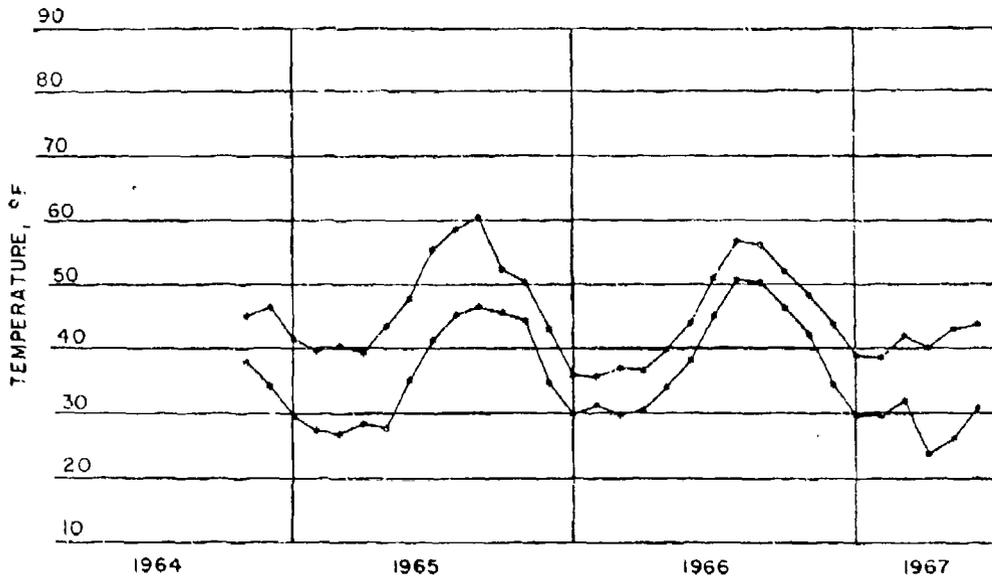
Schafer and his associates at China Lake have collected a vast amount of data from various military magazine storage facilities at representative locations around the world. Some of these data are available in a series of comprehensive reports²² according to magazine location in very cold, desert, tropical, and other extremes. The reader is referred to the report of Reference 23 in which the data from representative regions of the world are summarized in graphical form. It is clear that earth covered magazines are most favorable for protecting missile materials from both large daily temperature cycles and extreme temperature values.

Kurotori and Schafer²² compared temperatures in concrete magazines both with and without an earth covering in a number of environments. Figure 3-14 presents data taken over a several year period from magazines in Iceland. The average monthly maximum and minimum temperatures are shown as separate curves. The benefit of the earth covering is shown by the almost three to one decrease in temperature variations for earth covered relative to uncovered magazines.

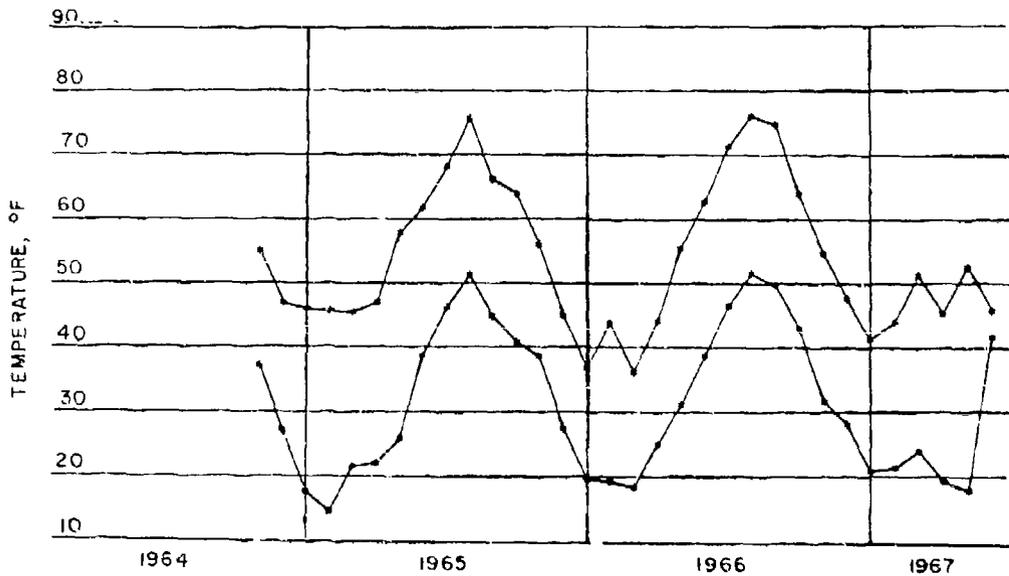
F. Transportation of Missiles

A last major area of concern is the environment seen by a missile during normal transportation. An Army tactical missile may have to be moved to many parts of the world during its storage life. There is no way to predict how often a missile will be moved or to what locations since such movements will be determined by international events.

Once again, Schafer and his associates at China Lake have documented data which provide the only known quantitative information available to guide our assessment of this problem. These missiles should always be expected to receive great care during handling. Therefore it is useful to examine some of the worst case situations noted by Schafer. For missiles transported within the hold of a ship,²⁶ temperatures cannot vary greatly because of the stabilizing effect of the ocean. However, the humidity will



The Average Maximum and the Average Minimum Temperatures of Earth-Covered Magazines at NS, Keflavik, Iceland.



The Average Maximum and the Average Minimum Temperatures of Non-Earth-Covered Magazines at NS, Keflavik, Iceland. Reference 22, Page 11.

Figure 3-14.

always be high. Therefore the concern for missiles transported by ship is directed towards the possible introduction of moisture with only moderate temperatures and temperature variation expected.

Studies of temperature profiles of air transported materials also show that temperature problems are not severe. Schafer and Dickus conducted a series of measurements involving several types of transport aircraft flying under very cold conditions. In a case where the heating system of the airplane was intentionally cut down to simulate partial failure, the temperature of the cargo remained at relatively high values. These data taken from a C-141 flight are shown in Figure 3-15. The outside temperature on this flight approached -70°F but the heat capacity of the cargo coupled with what heating remained maintained the cargo within the temperature envelope shown above about 40°F . Note that the cargo compartment floor did go down to about 5°F . Measurements on other flights were less severe as far as lowering the temperature of the cargo due to the flight itself is concerned. Based on these data it appears that the movement of missiles in normal air transports will not introduce low cargo temperatures anywhere near approaching the very cold values of the outside air at high flying altitudes as indicated in MIL-STD-210B. The recorded temperature of the cargo floor indicates severe extremes will probably never occur. Schafer noted that the lowest cargo temperature measured during his air transportation studies was only 19°F with a true outside air temperature of -82°F .

The decreased atmospheric pressure at high altitudes can, however, be a most significant factor. The decreased pressure will create conditions where leakage occurs through the seals of large containers. Upon returning to earth the large pressure differential has been seen to crush large missile containers.²⁵ Apparently it is easier for air to escape from containers than it is to leak back in. In fact pressure relief valves are often used which let air out when a 5 psi differential pressure occurs.³ For cases where the geometry of a container does permit air to leak back inside, a secondary problem must be considered, particularly if the landing occurs in a humid environment. The air replacing the original dry nitrogen may then contain a large amount of moisture. This effect corresponds to the problem of moisture accumulation inside a container with temperature cycling over sufficiently wide extremes to cause air exchange between the missile container and the outside air. In many cases the missile will be

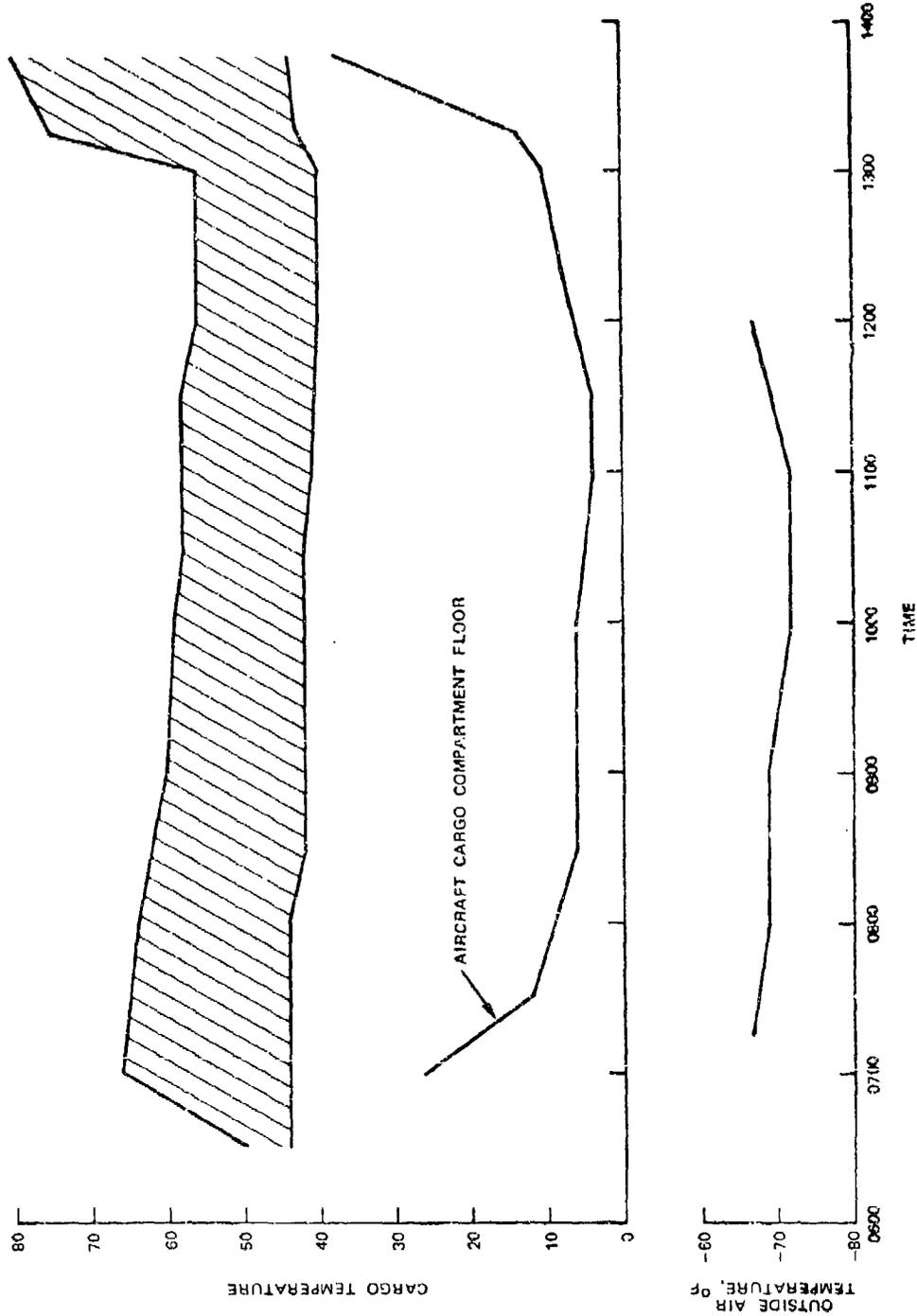


Figure 3-15. C-141 Flight From McGuire AFB to Rein-Main, Germany (1/18/69). The Shaded Area is the Material Temperature Envelope. From Reference 18, Page 10.

at a lower temperature when the air is subsequently forced out again. The net effect is that condensation of moisture will occur on the relatively massive missile motor and the air subsequently expelled may be correspondingly lower in moisture content. Schafer has noted large quantities of water collect in sealed containers cycled under humid conditions. This factor should be of major concern for missiles which remain in storage after transportation or storage conditions which cause this water pumping mechanism to develop.

Truck and rail transportation have also been examined by the Naval Weapons Center at China Lake. A study by Martin and Schafer¹⁷ measured the temperature profiles of truck cargos under both extremely cold and extremely hot weather conditions. They found that the truck enclosure offers protection from external extremes. For example, in a case where the outside temperature went to about -20°F , the cargo temperature reached -3°F . Corresponding extremes for hot weather tests showed that 128°F outside air temperature resulted in only 116°F cargo temperatures. These values are probably the most severe temperatures a missile would ever experience during transportation. Additional missile environment investigations are being conducted by personnel of the White Sands Missile Range.²⁷ They have been concerned with measurements on production hardware in the field.

G. Fungus

Fungus can grow and become destructive to missile materials under certain conditions. As living creatures these organisms must have food, moisture, oxygen and a favorable temperature range. Dr. Clossmyer²⁸ identifies the ideal temperatures as $20-40^{\circ}\text{C}$ and the optimum relative humidity as 85-100%. The food may be a contaminant or some ingredient of the missile system. The organisms are practically always present in the atmosphere with densities on the order of millions per cubic inch of air. During growth, the organisms collect minerals and become conductive for small currents. Missile materials are usually made resistant to fungus growth if the potential problem is always kept in mind by the designer and manufacturer. However, fungus problems are encountered with contaminants, improperly cured polymers, wrong paints and poor storage conditions. Growth can spread from a susceptible material to a normally non-susceptible material.²⁹ For example,

where missiles are stored within a tropical zone under a tree canopy, materials washed from plants onto the missile storage containers by rain-water can initiate fungus attack. The key to avoiding fungus problems in storage is to employ recommended materials for fabrication and keep the missile dry. The organisms certainly will not grow in a nitrogen atmosphere and if the relative humidity is less than 50% inside the missile container, the potential for growth is small.²⁸

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IV. FAILURE PROCESSES IN ELECTRONIC DEVICES IN STORAGE

A. Introduction

The physical mechanisms leading to failure in storage cannot be assumed to be the same as those normally found to cause operating devices to fail. The environment seen by a device in storage may be very different physically and chemically from that of an operating device. An operating device is subjected to physical stresses associated with applied electrical potentials, electrical currents, elevated temperatures and some mechanical stresses. The electrical potentials and currents may cause a variety of electrochemical processes to occur. A storage environment is normally free from electrical stresses and the higher temperatures associated with operating devices. The chemical and physical stresses associated with electronic devices in storage are therefore determined by ambient conditions and whatever materials may be within a package. Failure mechanisms which are common to devices in operation and in storage will be those having similar chemical or physical driving forces.

The evaluation of potential storage failure mechanisms must carefully consider the environment experienced by a device as well as details associated with construction, materials and fabrication processes employed. Environments seen by Army tactical missile systems are discussed in some detail in Chapter III of this report. It probably will never be possible to specify accurately in advance the environment a tactical missile will experience during its storage life since Army assignments will always be uncertain and tactical missiles must be readily available to activated Army units. Most types of tactical missiles remain electrically dormant for many years and are never activated electrically until just prior to actual firing. Therefore the failure mechanisms associated with electrical currents and applied bias so well documented for operating devices are not necessarily of importance to Army systems which operate only briefly after the very long storage period. Missile electronic systems which can be tested periodically, require separate consideration.

Many of the missiles currently being manufactured and stored employ electronic technologies which have primarily been developed within the last few years. Anticipated storage periods are as great as fifteen or twenty

years. Failure data are simply not available for these technologies based on relevant environments sufficient to establish statistical failure rates. This situation is not likely to change in the near future since rapid developments in electronic device technology are expected to continue for many years. Storage life data for specific technologies will therefore seldom be available to guide missile design. Therefore a more fundamental approach is needed to assess the long-term reliability of electronic devices placed in storage than has generally been necessary for normal operating applications.

The approach to long-term storage reliability must be built around a well founded understanding of the various degradation processes likely to cause failure. The ambient conditions in storage which become significant with long time periods include diurnal temperature variations, the presence of atmospheric components such as water and oxygen, and a variety of pollutants. In addition, a number of fabrication mistakes can be classified as storage reliability problems. Potentially critical manufacturing mistakes include residual process chemicals, a variety of chemical contaminants, particulate matter, incipient cracks in brittle materials including the package seal, conductors too thin and poor alloy thermal treatments. These same manufacturing mistakes are, of course, important for devices placed in regular operation as well as those in storage. However, the relative importance of particular defects in storage and in operation should be evaluated and judgments made about relevant screen test sequences. The fact that a perfectly fabricated device is not an easy accomplishment is reflected in the many compromises allowed in the MIL-STD-883 internal visual analysis criterion. An important concern for devices in storage is how stable the allowed defects remain over the long time periods characteristic of tactical missile storage.

B. Factors Influencing Microcircuit Failure Processes

Although the failure mechanisms introduced in storage may not be significantly different from those which occur in operating devices, the relative importance of these failure mechanisms can be very different. For example, many of the metal migration degradation processes associated with applied electrical fields in operating devices would not proceed in dormant situations. Electromigration^{1,2} wear-out processes associated with the combination of high current density and temperature are not likely to occur in

tactical missile systems. Unfortunately there is not sufficient long-term field failure information for device technologies of current and future missile system interests to provide a sound experience basis for the analysis of potential storage failures. The high reliability of modern solid state designs generally means that real life failure rate data will not be available soon enough to influence the design. Accelerated testing methods based on elevated temperature³⁻⁷ and electrical stresses⁸ have been applied for guidance in making operating life predictions. The theoretical foundation for accelerated testing based on thermal stress is discussed in greater detail in Chapter IX. Specific accelerated test methods have not yet been developed and proved applicable to storage environments. Knowledge of the detailed physical and chemical degradation processes which may occur in electronic systems exposed to Army missile storage environments is therefore critical to evaluating potential storage failure mechanisms.

Microcircuits are precision composite structures composed of materials which have been selected to provide desired electronic functions. Conditions which alter the intended geometrical arrangement of materials in minute detail may well be catastrophic to electronic operation. It is generally true that materials employed in most microcircuit structures will change very slowly when well protected. Properly fabricated integrated circuits might well remain stable for some decades if stored in a perfectly dry environment at a low and constant temperature. This conclusion is supported by experiences at the Naval Weapons Support Center in Crane, Indiana where very low degradation rates have been found for high reliability devices placed in dry nitrogen chambers located in a constant temperature room for a number of years. Unfortunately these conditions do not apply to tactical missile systems. As in most metallic structures, the materials combinations in a microcircuit are not in thermodynamic equilibrium. Material reactions occur in all device structures at rates which depend upon specific fabrication materials and various physical and chemical environmental factors. Realistic evaluations of storage failure rates expected of electronic devices therefore require that the basic processes leading to failure be well characterized. Only then can intelligent judgments be made concerning the effect departure from ideal conditions will have on device reliability. The purpose of this chapter is to discuss some of the more important stress factors and material reactions which ultimately determine the reliability of electronic devices in storage.

The investigations into various factors important to storage reliability were significantly aided by visits to missile storage depots, military laboratories, missile manufacturing facilities, industrial laboratories and to semiconductor manufacturers. Technical discussions were conducted with more than 200 individuals, each having knowledge and experience with some technical aspect identified as influencing the long-term storage reliability of electronic materials. Many of these individuals formulated constructive concepts based on their experiences even though few of the organizations had ever addressed nonoperating device reliability questions. Those groups which had done the most work towards details involving the long-term storage of electronic materials have been primarily concerned with environments much less severe than those Army tactical missiles will encounter and generally with older device technologies. The intuition concerning degradation processes of those workers closely involved with electronic materials has been most valuable to our assessments of potential storage failure mechanisms.

C. Storage Environmental Stresses

The term "environmental stress" used in this report includes various chemical and physical quantities which, as independent variables, induce changes in the structure of a microcircuit. Quantities such as temperature, electrical potential, chemical potential and mechanical stresses are therefore included in this very general definition. This section discusses how certain of these quantities take on much greater importance than others in storage. The storage degradation processes will always be determined by the most significant storage environmental stresses. The synergism of two or more of these generalized stresses acting at the same time can obviously have great consequences which might easily be overlooked.

1. Mechanical Stresses

Two primary sources of mechanical stresses in a storage environment are inertial forces and thermal-mechanical interactions. Inertial forces occur from both the cyclical accelerations associated with vibrations and with transient accelerations such as shock. Inertial forces often represent a major area of concern for electronic modules in military equipment. Both airborne and ground vehicle mounted systems must endure long-term vibration

and systems such as the cannon launched guided projectiles experience very high transient accelerations. The design of these systems therefore must protect electronic parts and connections from high operational mechanical stresses. The most critical concern with vibration is the excitation of mechanical resonance within sections of an electronic system where effective spring constants and masses approach critical values. The vibration amplitude and resulting material strain at resonance generally becomes greater than the materials can withstand. Solder joints crack, seals open and electrical leads fracture from fatigue damage due to these dynamical stresses.

A comprehensive analysis of the dynamic deformation modes in electronic structures has been compiled by Steinberg.⁹ He has shown the importance of sound construction practices for maintaining mechanical integrity in circuit structures. For example, his analysis of resonance modes shows that parts should be soldered at both sides of a hole when mounted to a circuit board. The solder joint fails by fatigue with a "shear tear-out" type of failure due to vibration induced bending of component lead wires.

a) Inertial Stresses. Except for some of the large hybrids, most microcircuits have very small masses so that their parts are not particularly susceptible to damage through inertial forces. For example, the linear density of 0.001 inch diameter bond wires is 0.01 mg/mm for gold and 0.0013 mg/mm for aluminum. Since most bond wires are in the range of 2-4 mm long, the mass is only a few micrograms for aluminum and a few tens of micrograms for gold wires. Similarly, a 50x50 mil silicon chip would have a mass of about 1 mg. Estimated storage accelerations of about 10 g therefore correspond to entirely negligible inertial forces for microcircuit materials. The maximum force on a 1 mil diameter gold bond wire would be less than 0.1 mg whereas normal pull strengths for good gold bonds are greater than 5 grams. However, at vibrating frequencies corresponding to a mechanical resonance at sufficient power, bond wire fatigue failure can occur at small g values. Electronic parts mistakenly placed in an ultrasonic cleaner are found to fail from bond wire fatigue in very short time periods.¹⁰ Ultrasonic excitations are not anticipated in any of the missile storage environments reviewed in these investigations. In any case, the only con-

cern for inertial stresses during the storage life of a missile should occur during transportation or handling within a storage area. The missile containers appear able to provide dampening for both vibration and shock. It is therefore concluded that a "clean" microcircuit would suffer mechanical damage due to inertial stresses only under extraordinary circumstances.

b) Thermal Mechanical Stresses. Differential expansion between materials within a circuit and between subassemblies and interconnections due to temperature changes introduce stresses in dormant as well as operating structures. Temperature changes occur in operating devices due to Joule heating of conducting elements as a circuit is turned on and off or as power levels are altered. Mechanically induced failures from temperature changes in operating devices are well documented.¹¹⁻¹⁶ However, it should be noted that such mechanical failures probably result from more than the mechanical stress alone as is sometimes implied. The failure mechanism may be due to the combined influence of thermal energy, electrical conduction and mechanical deformation processes. For example, electromigration² will alter the localized composition of conductor alloys which may induce conditions which initiate first stage fatigue cracks.^{17,18} Subsequent microfatigue damage will increase the localized resistance and Joule heating which accelerates thermal diffusion. Thermal diffusion processes^{19,20} in turn contribute to fatigue damage by several mechanisms such as the creation of Kirkendall voids, impurity concentration and the segregation of alloy dopants at grain boundaries.^{21,22}

Thermomechanical stresses occur in composite structures such as microcircuits due to two closely related phenomena. First, the materials in a microcircuit have different coefficients of linear expansion so that large mechanical stresses can result from even slow, uniform temperature variations. Second, temperature gradients also lead to differential displacements due to separate regions of a circuit being at different temperatures. Temperature gradients occur in microcircuits because of Joule heating at critical high resistive locations such as junctions, resistors and bond wires and also because of fast ambient temperature changes. It is important here that the difference in response of materials to slow and fast temperature changes be well understood. The term "thermal cycling" is normally employed in microcircuit screen testing, etc. for phenomena associated with

slow, uniform temperature changes and "thermal shock" has to do with very rapid temperature changes. Table A contains a list of thermal parameters and elastic modulus values for some of the materials used to fabricate microcircuits. Polymers present the potential for very severe thermal mechanical problems as discussed separately in Chapter VII. Other effects associated with thermal-mechanical phenomena include the residual mechanical stresses which usually exist throughout a microcircuit structure because oxides, nitrides and metal films are grown and bonds are made at elevated temperatures and then cooled down to the ambient.

An analysis of the stresses induced through temperature changes is based on the geometrical configuration of the circuit and the thermal and mechanical parameters of the construction materials. However, it should be understood that the thermoelastic problem presented by even simple microcircuit structures is likely to be too difficult for exact solutions. Approximation solutions must usually be obtained by constructing simplified models of a circuit structure. Methods for formulating models appropriate to microcircuits have been outlined by Howland and Zierdt.²³ They provide solutions for several idealized configurations. A comprehensive treatment of thermal stress and the mechanical response of materials in general is given by Manson.²⁴ Structures having constraints are most subject to thermal induced stress problems and microcircuits have many structural regions and interfaces which cannot be stress relieved. Microcircuits are dynamic structures since they are generally subject to localized and variable heating within particular circuit elements. The principal thermoelastic stresses of importance to missile storage are those associated with the ambient and with residual stresses developed during fabrication²⁵⁻²⁹ or screen testing procedures. It is clear from the values listed in Table A that the expansion coefficients of materials used in microcircuits vary widely. There is, for example, almost a ten to one differential for the silicon chip and aluminum conductor expansion coefficients. A similar differential exists for Si and SiO₂. Even more drastic differentials occur when polymers are used as discussed in some detail in Chapter VII.

Clearly thermal shock is most important for materials having high thermal impedance such as ceramics^{14,15} but can also be important for metals. Even homogeneous materials fracture or warp under mechanical stresses intro-

Table A

Thermal and Mechanical Parameters for
Materials Used in Microcircuits

Material	Temperature Coefficient of Expansion $\alpha \times 10^{-6} (\text{OC})^{-1}$			Thermal Conductivity watt/cm $^{\circ}\text{K}$	Young's Modulus $\text{Nt/m}^2 \times 10^{10}$
	200 $^{\circ}\text{K}$	300 $^{\circ}\text{K}$	500 $^{\circ}\text{K}$		
Aluminum	20.0	23.2	26.4	2.4	7
Copper	15.1	16.8	18.3	4.0	12
Gold	13.4	14.1	15.0	3.1	8.1
Molybdenum	4.6	5.0	5.3	1.4	29
Nickel	11.0	12.7	15.2	0.9	21.4
Palladium	10.8	11.6	12.6	0.7	11.8
Platinum	8.4	8.9	9.5	0.7	16.7
Silicon	1.3	2.5	3.5	1.5	
Tantalum	6.3	6.5	6.8	0.54	18.6
Titanium	7.2	8.5	9.8	0.2	11.6
Tungsten	4.1	4.5	4.6	1.7	36
Kovar		(5.0)			
Al_2O_3	(4.6-5.5)		(7.1-7.3)	0.4	31
SiO_2 (vitreous)	0.1	0.42	0.56	0.014	7.3
Glass (pyrex)		(7.8-9.7)		0.010	6.2
Lead-Tin (solder)		(25.1)			

These values are taken from the Handbook of Chemistry and Physics,
Chemical Rubber Publishing Co. and the American Institute of Physics
Handbook, McGraw-Hill.

duced by the thermal gradients from rapid cooling or heating. One part of a material expands or contracts differently from another part simply due to the temperature difference. However, this matter is made much more complex in the composite structure of microcircuits because of the many interfaces and bonds involved in a packaged device. The differences in expansion coefficients come into play here just as they do for slow variations. Certain phase transitions are also introduced by fast temperature changes. Thermal shock is particularly severe for the ceramic materials used as feedthrough insulators in packages. Some of the mechanical stresses which must be evaluated in storage are listed in Table B.

Army tactical missiles will be thermal cycled at various times during storage. Because the electronic systems are located within the thermal mass of a missile, device temperature changes should always be slow. The electronic parts in a dormant missile should therefore never experience thermal shock even for sudden, extreme changes in the ambient temperature. The number of temperature cycles an individual missile will experience during storage life is impossible to predict because these systems should be in earth covered magazines unless military needs determine otherwise. However, notable examples where missiles were stored in less protected environments for extended periods were found. The maximum temperature span during a given day due to diurnal temperature cycling will be less than 70°C so that the rate of change will be less than $6^{\circ}\text{C}/\text{hour}$. The actual values will be less than these ambient extremes by amounts depending upon the specific location of electronics packages and the thermal inertia of a missile and its container. If a missile system is turned on, either for firing or for operational checks, internally generated thermal shock may occur within some individual circuits. During the course of this program a particular high power LED was found to fracture due to the thermal shock which occurred when the device was cooled just prior to firing the missile. Although this thermal shock is not strictly a storage problem, it should be clear that devices may have to handle operational shock. In this case an altered design of the electrode support to accommodate expansion apparently solved the problem.

2. Chemical Stresses

Chemical stresses may develop in microcircuit structures from a large number of potential chemical interactions.³⁰ These interactions

Table B

Mechanical Stresses in Microcircuits

1. Residual Stresses in Thin Deposited Layers
 - a) Due to Differential Expansion During Growth Phase
 - b) High Defect Density: Dislocations, Point Defects
 2. Interface Microstresses
 - a) Lattice Parameter Misfit
 - b) Impurities
 - c) Incomplete Adhesion
 3. Static Stresses Imposed During Fabrication
 4. Inertial Stresses
 5. Differential Expansion
 - a) Due to Temperature Gradients
 - b) Due to Different Thermal Expansion Coefficients
 6. Cyclical Mechanical Stresses Due to Temperature Cycling
-

include solid-solid metallurgical processes³¹ as well as chemical reactions with outside contaminants.^{32,33} As stated above, the metallurgical configurations within microcircuit structures are not in thermodynamic equilibrium. The time rate of degradation will depend upon temperature and a number of metallurgical factors such as specific materials, microcracks, compositional variations, grain size, dislocation density and impurities. A large number of contaminants have been found to cause failures in microcircuits. These include halogen ions, alkali metal ions, residual process chemicals, hydrogen, oxygen, various atmospheric pollutants and water. Water is identified as a particularly critical contaminant and all possible steps should be taken to minimize moisture content within circuit packages. It is very difficult to detach water from solid surfaces even at ultra-high vacuum and elevated temperatures so there is always the strong possibility that some water will remain within a package which otherwise was fabricated under clean conditions.

Himmel³³ has identified a number of contamination problems for hybrid microcircuits. He has classified these according to their introduction during the various manufacturing processes. Some of the contaminants listed are:

1. Solvent contaminants
2. Metal impurities
3. Bath impurities
4. Resist impurities
5. Etchant impurities
6. Paste contaminants
7. Printer lubricants
8. Environmental gas impurities
9. Weld or solder splatter
10. Flux residues
11. Chip fragments
12. Adhesive migration
13. Wire contaminants
14. Wire fragments
15. Moisture
16. Oxides

17. Smog
18. Human contaminants (perspiration, skin, etc.)
19. Airborne particles
20. Grease

Some of the more important sources of chemical stresses in microcircuits in a storage environment are listed in Table C. In addition, packaging techniques which include polymers may introduce moisture problems either due to outgassing from trapped moisture within the polymer or by moisture conductance if a polymer seal is employed. Metal or ceramic hermetic seals may develop leaks with time. The important subject of hermeticity is covered as a separate topic in Chapter V and relationships with polymer seals in Chapter VII.

The thresholds for contaminant induced chemical reactions are generally unknown if indeed such thresholds exist. It is, however, clear that very small quantities of certain contaminants can be serious. State-of-the-art analytical techniques have been available in some laboratories to identify microcircuit failures generated by trace contaminants. Unfortunately many chemical failure analysis problems remain unresolved and defense organizations must grope with the imposition of impurity requirements based on scant knowledge. For example, a particular missile manufacturer was found to require a hybrid microcircuit supplier to insure that their packages contained less than about 40 ppm moisture. While this value may be most desirable, it was apparent that neither the hybrid manufacturing methods nor the package moisture monitoring apparatus used in this situation were appropriate for work even close to this moisture level. The recent modifications to MIL-STD-883B, Method 1018 and Method 5008 have addressed this problem by setting initial moisture standards of less than 6000 ppm for hybrid microcircuits and 500 ppm for integrated circuits. Thomas has said these values represent starting points and will be modified according to future experiences concerning manufacturing methods, moisture measurement techniques and moisture induced reliability degradation.

Because of the long time periods involved with missile storage, trace chemical contaminants can be catastrophic even where reaction rates are small. Moisture is critical because of the corrosion processes it induces directly and because the presence of moisture activates such contaminants as residual chloride ions. The applied biases in operating devices intro-

Table C

Sources of Chemical Stresses

1. Concentration Gradients Within the Microcircuit Structure
 2. Process Chemicals Remaining From Fabrication Steps
 3. Gases Evolved From Device Materials
 4. Contaminants Including Moisture
 5. Environmental Gases Introduced Through Deficiencies in Hermetic Seals
 6. Stress Accelerated Chemical Reactions
 7. Galvanic Cells
-

duce certain electrochemical processes not likely to present problems in storage. However, some caution is necessary here because initial testing and screening procedures may initiate degradation processes which could cause problems later. In addition, galvanic cells within the materials involved in a circuit can generate potentials sufficient to cause problems even in dormant storage.

Solid-solid interactions are important in a number of microcircuit locations. Diffusion across interfaces is necessary for such processes as making bonds and obtaining good adhesion between thin film layers. However, because of the very small dimensions involved, over-diffusion can detrimentally modify (by alloying) pure metal regions. A common concern here is the diffusion of Si into Al. Failures such as open conductors and shorted junctions are common results from over-diffusion. A number of metallurgical factors influence the rate and extent of solid-solid interactions. For example, atomic diffusion is greatly accelerated by defects such as microcracks and dislocations and by microstructural characteristics such as grain boundaries and surfaces. Particular dopant atoms employed for special purposes may diffuse rapidly and become detrimental to device operations. Details relative to the grain structure of metallization layers and bond wires are also important since individual grains extend through or across a considerable fraction of a circuit feature dimension.

3. Thermal Stress

Most chemical reactions proceed at rates generally described by an Arrhenius expression^{31,34,35}

$$K = \sum_{i=1}^N A_i e^{-Q_i/RT} \quad (4-1)$$

where K is the reaction rate, Q_i an activation energy characteristic of the i th reaction, R the gas constant, T the absolute temperature and A_i is a set of constants. Equation 4-1 implies that a number of reactions are anticipated and that the net effect corresponds to a superposition of many processes (see Chapter IX). Atomic diffusion processes in metals and alloys have diffusion constants, D, which are empirically found to have a similar

temperature relationship¹⁹

$$D = D_0 e^{-Q/RT} \quad (4-2)$$

where D_0 and Q may vary with composition but are independent of temperature. The quantity Q is related to the enthalpy, ΔH . The diffusion flux, J , for component i is then obtained from Fick's first law^{19,31}

$$J_i = -D_i \left(\frac{\partial C_i}{\partial x} \right)_t \quad (4-3)$$

where C_i is the concentration of component i . The many interfaces in the composite structure of a microcircuit introduce large concentration gradients $\frac{\partial C}{\partial x}$ which will therefore lead to atomic diffusion at rates determined by the diffusion coefficients, D_i , for each component. Since the important linear distances in a microcircuit are small, these structures are particularly sensitive to diffusion. Due to the exponential in both Equations 4-1 and 4-2 thermal activated processes must always be carefully examined to evaluate the material stability of a microcircuit structure. According to the data contained in Chapter III, the maximum temperature recorded at the skin of an exposed missile is about 75°C and this value existed only for a short period of time. The thermal mass of a missile will probably maintain electronic systems at still lower temperatures. Similarly, the thermal mass will also prevent the electronics package from reaching the recorded low temperature extremes in cold environments. These extremes are possible in any case only under unusually severe conditions. Therefore purely thermal degradation processes will generally be slow at storage temperatures and not of particular importance in stored missile systems. Some reactions are activated at particular elevated temperatures and do not occur at lower temperatures. Conversely, certain chemical reactions may well be arrested above a temperature where the moisture is driven from solid surfaces. The dew point of the package gas obviously has great significance in determining the particular chemical processes which will take place. Since operating device temperatures are normally well in excess of those in storage, the temperature determined reaction rate processes in stored electronic devices should be expected to differ from operating devices.

D. Failure Mechanisms

Careful consideration of environmental stresses and materials is required in order to evaluate which failure mechanisms have greatest significance for missiles in storage. The response of microcircuit materials to storage stresses in combination with manufacturing mistakes will determine the failure mechanisms in storage. Failure processes for electronic devices in general are described in a large number of publications within the open literature, government reports and various industrial publications. Corrosion processes particularly applicable to microcircuits have been reviewed recently by Kolesar.³⁰ Harman²⁵ has described several metallurgical factors affecting the reliability of wire bonds and Scnnable and Keen³⁶ surveyed failure processes having particular importance to LSI arrays.

Microcircuit structural features such as bonds, oxide steps and packages are of critical concern in the storage environment just as they are for operating devices. Bonds will always be critical because of the basic nature of bonding processes which join sometimes dissimilar metals at interfaces which often include oxides or other contaminants. The metallurgical processes involved in bonding are discussed in some detail below because of their importance to microcircuit reliability. Features such as oxide steps and other sharp material discontinuities are critical as points of mechanical stress concentration and as highly susceptible regions for possible fabrication mistakes. The package is critical because it must protect the circuit materials from physical and chemical environmental effects for many years. However, a hermetic package requires material discontinuities such as metal-ceramic interfaces and must itself be sealed by an extensive joining process.

Manufacturing mistakes present problems for both operating and stored devices. The most severe circuit defects will be detected during an appropriate screening sequence. However, certain marginal defects could present greater reliability problems in long-term storage than would be the case for normal operation. Marginal defects such as poor metallization adhesion, partial wire bonds, conducting particles, microcracks in glassivation films or the chip, and residual process chemicals or other sealed-in

contaminants are to be considered as important storage reliability factors. The range of potential storage failure mechanisms is determined by the variety of materials employed in the microcircuit composite structure, the processing chemicals and chance contaminants. These variables are so numerous when considering the different technologies that detailed consideration here can be given only to the more important factors but all factors must ultimately be evaluated to the greatest extent practical before using particular device technologies in systems to be stored.

1. Fault Tree Analysis

It has been useful in our analysis of failure mechanisms to construct fault trees^{38,39} leading to particular failure modes as illustrated in Figures 4-1, 4-2, and 4-3. The failure processes chosen for illustration here are primarily those which can be related to dormant conditions although a few branches include mechanisms which would be associated with operating devices.

SYMBOLS



Events A + B + C are all required to cause Event D



Event A or Event B or Event C can result in Event D independently

Electromigration problems of the type indicated in Figure 4-1 should not occur in dormant storage. However, combinations such as thermal cycling plus thin metallization could result in failure by mechanical fatigue following a finite number of cycles. Figure 4-2 illustrates some of the factors which can lead to bond failure. Figure 4-3 is concerned with particle induced problems. It should be mentioned that although Figure 4-3 does not so indicate, charged nonconductive particles can introduce charge inversion problems in MOS devices.

2. Factors Affecting Intermetallic Bonding

An intermetallic bond interface may bridge such property differences

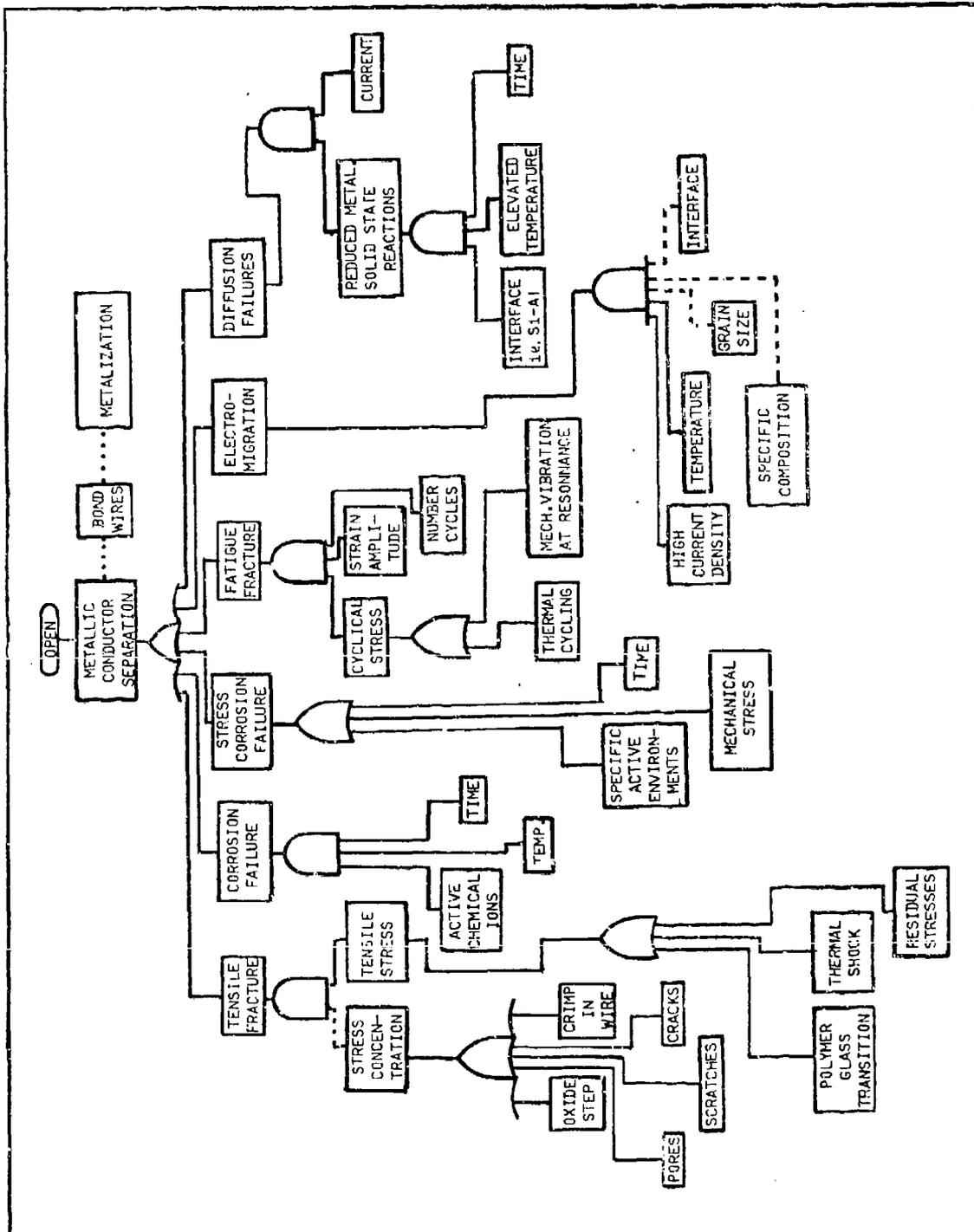


Figure 4-1. Fault Tree Showing Potential Sources of Circuit Opens.

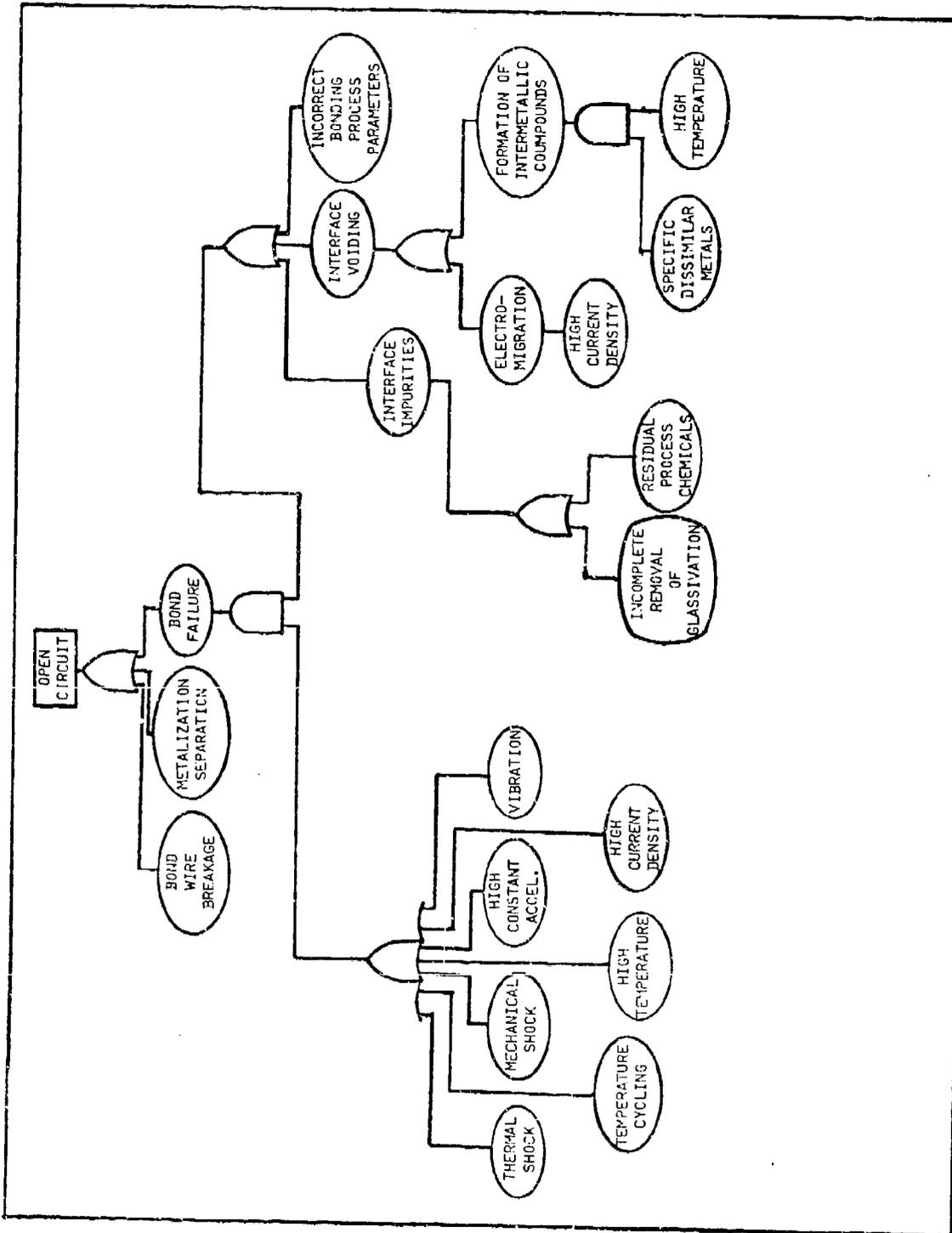


Figure 4.2 Fault Tree For Bond Problems.

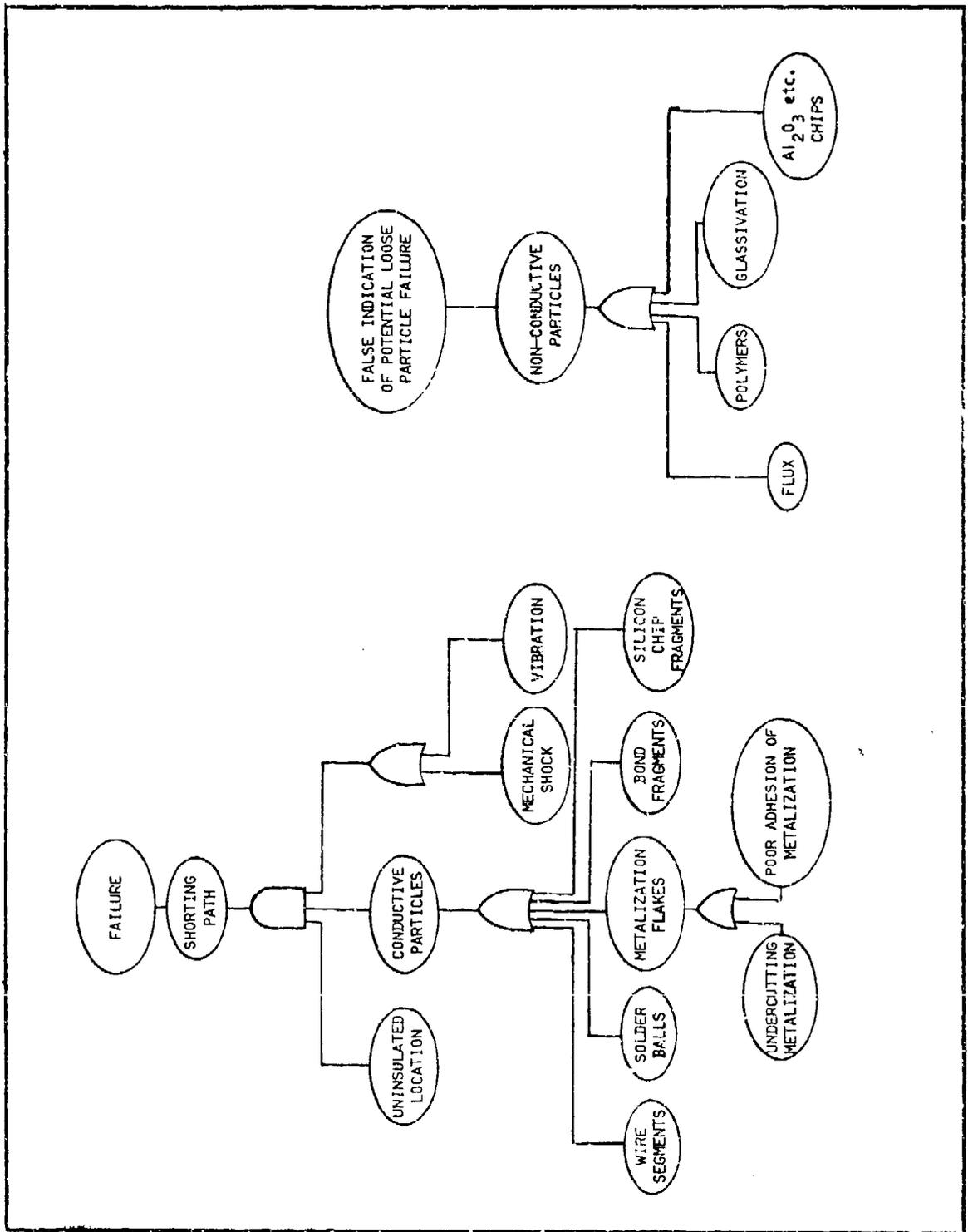


Figure 4-3. Fault Tree Illustrating Potential Particulate Matter Problems.

as crystal structures, lattice constants, elastic constants, chemical and metallurgical characteristics and thermal expansion coefficients. Interatomic diffusion has to occur between the members to develop a sound coupling interface but the extent of diffusion must be limited in many cases to avoid detrimental effects such as brittleness sometimes associated with the formation of intermetallic compounds.

The bonding of electrical conductors is accomplished by a number of methods.⁴⁰⁻⁴¹ Some of the more recent techniques include thermal compression bonding,⁴² ultrasonic bonding⁴³⁻⁴⁶ and diffusion bonding.⁴⁷⁻⁴⁹ Joining is generally described as taking place in two stages:

1. Mechanical deformation due to pressing the members together
2. Interatomic diffusion

Surfaces are never perfectly flat so that the net area in contact upon first touching is a very small fraction of the actual surface area. Application of sufficient mechanical force plastically deforms surface asperities so that the metals flow into depressions, etc., and thereby achieve contact. A somewhat elevated temperature then permits atomic diffusion to establish an intermetallic interface. If the mechanical deformation is only elastic, removal of the force may result in rupture of bonded regions leading to a poor bond. Recrystallization and grain growth in the interface is also found to occur in establishing bonds between some metals. Grain reorientation and other factors usually associated with sintering processes⁵⁰ are also involved here. The strength of certain bonds is increased at critical combinations of temperature and aging time.^{51,53} Surface oxides and coatings of other foreign materials add significantly to the problem of establishing metal to metal contact. The surfaces to be bonded must be carefully cleaned and steps taken to disperse oxides before the metallurgical bonding can occur. The tenacious oxide of aluminum is particularly difficult to handle and has led to many bond problems.

Structural metals have long been successfully fabricated by fusion welding. In recent years, however, solid state bonding methods have increasingly been applied to situations where the fusion of metals from a melt is not advantageous. The manufacture of electronic devices has presented a new range of materials and metal joining problems. Many bonds used in integrated circuits, for example, are microscopic in size and require not

only structural strength and corrosion resistance but must remain stable with relatively high electric current densities and with temperature excursions.

The interface of a metallurgical bond may extend from a few lattice spacings up to many microns. The important property considerations include structural factors, chemical and metallurgical interactions, thermodynamic factors and the mechanical properties and characteristics of the two members. Structural factors affecting the bond include the crystal structures of the components, grain sizes, crystallographic orientation and the lattice parameters. Studies reported by Sikorski⁵³ on intermetallic adhesion showed fcc metals stick together better than do bcc metals and that adhesion involving hcp metals is poor. Buckley and Johnson⁵⁴ also examined adhesion for different crystal structures as it related to friction and wear characteristics in vacuum and obtained similar conclusions. They reported, for example, that hexagonal cobalt had significantly lower friction and wear than did fcc cobalt. Buckley⁵⁵ subsequently showed friction to be lower along a slip direction and also that lower friction occurs for Cu-Au alloys which were ordered. Although hcp metals are successfully bonded, it is usually much easier to make bonds between two face centered cubic metals. This is partly due to the availability of multiple slip systems so that the plastic deformation may proceed to bring the metals into intimate contact. Two fcc metals also have a higher probability for matching similar crystallographic planes. Gold and aluminum are both fcc metals. The data in Table D show that the lattice mismatch is small and their elastic constants are also close. However, these two metals do not form solid solutions over a significant range as seen in the Au-Al phase diagram of Figure 4-4.

A lattice parameter mismatch may be accommodated by a network of interfacial dislocations formed more or less in the plane of the interface. Van der Merwe⁵⁶ and later Jesser and Kuhlmann-Wilsdorf⁵⁷ calculated misfit dislocation network spacings for abrupt interfaces based on interfacial energy and elastic strain energy considerations. These networks have been investigated experimentally for thin crystal film overgrowths using several fcc metal-metal combinations.⁵⁸⁻⁶¹ For very thin film overgrowths where the lattice misfit is small, the strain energy is distributed throughout the thickness of the film. For thicker coatings and the bulk material bonds of interest here, the strain must be accommodated by misfit disloca-

Table D
Selected Metal Parameters

Metal	Crystal Structure	Lattice Constants		Distance of Closest Approach	Elastic Shear Modulus	Melting Point
		$\overset{\circ}{\text{A}}$ a	$\overset{\circ}{\text{A}}$ c	$\overset{\circ}{\text{A}}$	10^{11} dyne/cm ²	$^{\circ}\text{C}$
Ag	fcc	4.0856		2.888	3.0	961
Al	fcc	4.0490		2.862	2.70	660
Au	fcc	4.0783		2.884	2.78	1063
Co < 400 $^{\circ}\text{C}$	hcp	2.507	4.069	2.506	8.1	1495
Co > 400 $^{\circ}\text{C}$	fcc	3.552		2.511	8.1	1495
Cr	bcc	2.8850		2.498	7.4	1875
Cu	fcc	3.6153		2.556	4.8	1083
Fe	bcc	2.8664		2.481	8.3	1534
Ir	fcc	3.8389		2.714	21.3	2450
Mo	bcc	3.1466		2.725	12.8	2620
Nb	bcc	3.3007		2.859	3.7	2468
Ni	fcc	3.5238		2.491	7.4	1453
Pd	fcc	3.8902		2.750	4.45	1552
Pt	fcc	3.9237		2.775	6.25	1769
Re	hcp	2.7609	4.4583	2.740	27	3180
Rh	fcc	3.8034		2.689	15.3	1966
Ru	hcp	2.7039	4.2816	2.649	17.2	2500
Ta	bcc	3.3026		2.860	6.85	3000
Ti	hcp	2.9504	4.6833	2.89	3.98	1668
V	bcc	3.039		2.632	4.66	1900
W	bcc	3.1648		2.739	16.0	3380

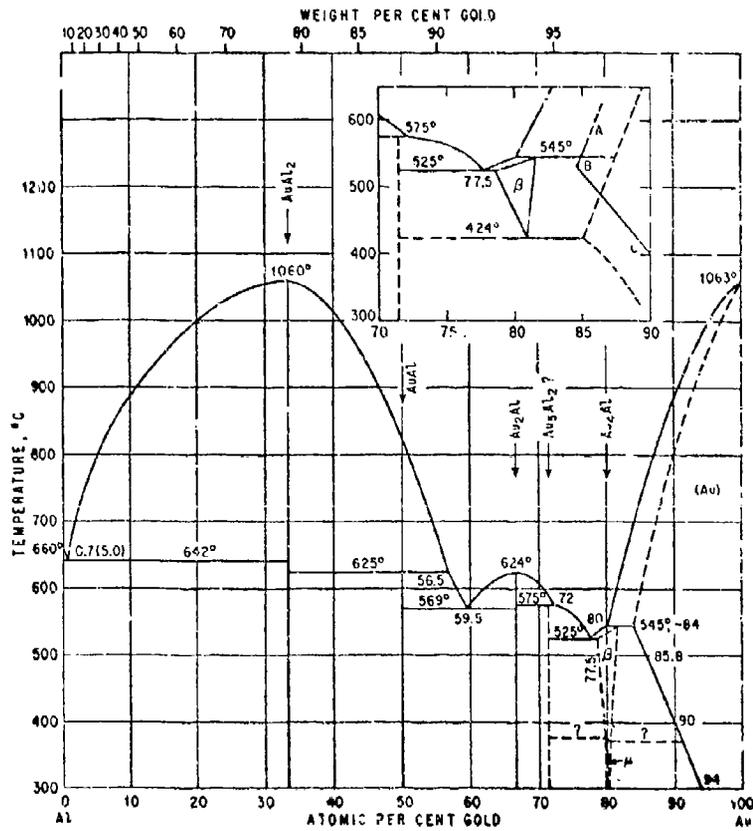


Figure 4-4. Phase Diagram For Aluminum-Gold (from M. Hansen, Constitution of Binary Alloys McGraw-Hill, 1958).

tions of properly oriented⁶² Burgers vectors being generated and gliding into the interface. The inability of a particular abrupt metal-metal couple to establish a misfit dislocation network will probably cause microcracks at the interface and may result in bond lifting. Matthews⁶³ has reported cracking prior to the introduction of misfit dislocations, in Pb-PbSe couples. Cracks often develop in chromium overgrowths on copper^{64,65} due to high strain because of the poor lattice registry between the two metals.

Accommodation of a lattice misfit may be aided by extending an interface through interatomic diffusion. A model for extended interfaces treated theoretically by Fleisher⁶⁶ was used to evaluate contributions to strengthening resulting from gradients in both lattice parameters and elastic modulus. The strengthening effects of diffusion interfaces were later investigated experimentally^{67,68} for thin films evaporated onto copper crystals. A direct observation of the effect of diffusion on misfit accommodation was recently reported by Marcinkowski, et al.⁶⁹ Cu₃Au-Au interfaces were annealed in an electron microscope while observing the misfit dislocation network. Misfit dislocations were found to climb out of the network and annihilate one another as diffusion proceeded.

The chemical characteristics of the atomic species may influence several factors both prior to and subsequent to the formation of a bond. Chemical considerations determine the metallurgical nature of the interface and also influence environmental effects due to chemical reactions with a particular atomic species. Recent investigations⁷⁰⁻⁷⁵ of interdiffusion have demonstrated that thermodynamic and structural factors can cause compositional distributions to develop that are quite different from a smoothly varying function. For example, Tu and Bergy⁷⁰ have shown that the diffusion of Cu-Au bimetal films result in layered structures of Cu-Cu₃Au-CuAu₃-Au which had sharp steps between the three phases. In addition, it has been shown⁷⁴ that surface contaminations can have a marked effect on diffusion mechanisms. Theoretical treatments considering the available diffusion paths in a solid have indicated that concentration peaks are to be expected for diffusion from a thin surface film. The important gold-aluminum interface has been carefully studied by Campisano, et al.,⁷⁵ using He⁺ backscattering and glancing angle x-ray diffraction methods. They were thus able to follow details of the kinetics for formation of the intermetallic compounds of the Au-Al alloy system. The rate of growth of intermediate phases followed

a $(\text{time})^{1/2}$ dependence characteristic of diffusion-limited growth kinetics. They observed only four of the five intermetallic compounds contained in the Al-Au phase diagram of Figure 4-4. These were AuAl_2 , Au_4Al , Au_2Al and Au_5Al_2 . The purple phase, AuAl_2 , was observed to grow as an end phase also with a $(\text{time})^{1/2}$ dependence and with an activation energy of 1.2 eV. Even at temperatures as low as 85°C , the Au_2Al phase had measurable growth in minutes but AuAl_2 began to grow from this phase at higher temperatures, on the order of 175°C . Compisano, et al.,⁷⁵ did not detect the phase AuAl in their thin film couples.

A dissimilar metal bond interface therefore cannot reliably be described by smoothly varying functions bridging the different properties of the materials bonded. The complete bond interface may actually consist of five or more dissimilar metal interfaces^{70,75} according to the number of intermetallic compounds possible as well as the bonding parameters of temperature, pressure and time. Philofsky^{76,77} studied the AuAl interface system using butt-welded diffusion couples. He pointed out that the purple phase in itself is not weak but in fact, has greater tensile strength than does either gold or aluminum. He therefore suggested that the critical stage is the deformation of voids at the interface between phases. Lines of Kirkendall voids appear at interfaces when the AlAu couple is maintained at 300°C for long time periods. He also noted⁷⁶ that thermal cycling can cause voids to develop at lower temperatures and that temperature cycles much accelerated the formation of the voids at higher temperatures. He interpreted this behavior as due to thermo-mechanical induced microcracks accelerating atomic diffusion processes. Horsting²¹ subsequently investigated the importance of impurities to the growth of voids in intermetallic compounds of ultrasonic bonds between aluminum wire and gold platings. He showed that exposure of a clean interface system to high temperature resulted in the formation of different phases but not a reduced bond strength. However, the deliberate addition of impurities in the gold plating both resulted in the formation of voids and poor mechanical bonds. Horsting²¹ showed that the impurities were collected and swept ahead of the advancing diffusion front.

The mechanical strength of a bond is determined by the amount of intermetallic contact and various characteristics of the material in the region of the bond interface. Diffusion induced Kirkendall voids usually occur in

planar arrays⁷⁶ and may lead to failure due either to the reduced area supporting the load or else serve as incipient fatigue cracks for ultimate fatigue failure. A poorly dispersed or not absorbed surface contamination⁷⁸ can also result in a decreased load supporting area. Brittle phases may fracture internally when stressed but are also likely to fail at an interface with a neighboring phase due to abrupt differences in mechanical characteristics.^{79,80} An abrupt bond interface may be strong if there is good atomic adhesion⁶⁵ and if the lattice misfit and other property differences are not too great. However, microcracks result if property differences are large and abrupt. Bond integrity is determined not only by properties of the particular materials bonded but also by parameters associated with the bonding process and by the subsequent mechanical, thermal, electrical and chemical environments experienced by the bond in use. Solid state bonding processes have in common⁴⁰ the two principal stages of plastic deformation and diffusion described earlier. Methods usually described as "diffusion bonding"^{40,47-49} involve applying mechanical pressure normal to the surface at an elevated temperature for some time period. Because of the time at high temperature, the materials must normally be protected by conducting the operation in either a vacuum or an inert gas. The minimum plastic deformation required to bring the surfaces into contact is usually 30%-40% and diffusion times and temperatures are determined by the materials. Thermal compression bonding is similar to diffusion bonding but can be used in air for the bonding of small wires that do not require long bonding times. The bond members are heated quickly and cooled quickly so that diffusion can be controlled to some extent.

Ultrasonic bonds are usually made at room temperature by pressing the bond members together using tools excited with ultrasonic energies at a few tens of kilocycles. The precise mechanism of bonding is not clear. Until recently it was widely thought that interatomic diffusion occurred with high surface temperatures assumed to be generated by surface friction. However, Joshi⁴³ recently reported investigations in which a laser technique showed that a discontinuity in vibration amplitude did not exist at the bond interface. Therefore the explanation of interatomic diffusion must be other than the generation of elevated temperatures by friction. Plastic deformation is assisted by ultrasonic energy in metals and alloys as shown by the work of Lagenecker.⁸¹ The ultrasonic energy also aids in fractur-

ing and dispersing tough oxides such as Al_2O_3 . However, details of the diffusion mechanism required for the formation of ultrasonic bonds are still to be determined. The ability to make bonds at room temperature using ultrasonic methods makes this method valuable for a number of applications where elevated temperatures are not desired.

3. Mechanical Failure Processes

A wide range of mechanical failure processes have been identified in microcircuits. Those most likely to occur in response to the mechanical storage environmental stresses of Section IV C.1 are discussed in this section. The mechanical stresses of concern in storage include both static and dynamic modes having synergism with chemical and thermal factors. Cyclical stresses lead to fatigue damage by microstructural processes that are described in various reviews and texts.^{17,18} It is often found possible to describe the fatigue behavior of metals with a graphical S-N representation similar to that shown in Figure 4-5. The curve is a plot of the fatigue stress amplitude as a function of the number of cycles to failure. The data for a set of specimens would not actually fall on the solid line but points from individual specimens generally lie within the bounds of the dotted curves. Large stresses lead to specimen failure in only a few cycles, depending upon the material. The number of cycles to failure increases dramatically with a decrease in applied stress amplitude as indicated by the logarithmic scale on the horizontal axis. Mechanical degradation processes associated with the low stress end of the S-N curve are called "High Cycle Fatigue Processes" and those occurring in the high stress region are "Low Cycle Fatigue Processes".^{82,84} The low cycle fatigue processes are generally characterized by some plastic deformation. The dashed line extension in Figure 4-5 indicates the tremendous modification of fatigue life that can occur when the structure is exposed to specific chemical environments. This phenomena is called "Corrosion Fatigue." The term "Stress Corrosion Cracking"⁸⁴ is sometimes classified as a subtopic of corrosion fatigue but with a constant rather than a cyclical mechanical stress. The constant mechanical stresses causing stress corrosion problems are derived from residual stresses as well as from applied loads.

Most of the vast amount of mechanical behavior information available today was obtained through studies on relatively massive structural materials as compared to even the largest elements of electronic circuits.

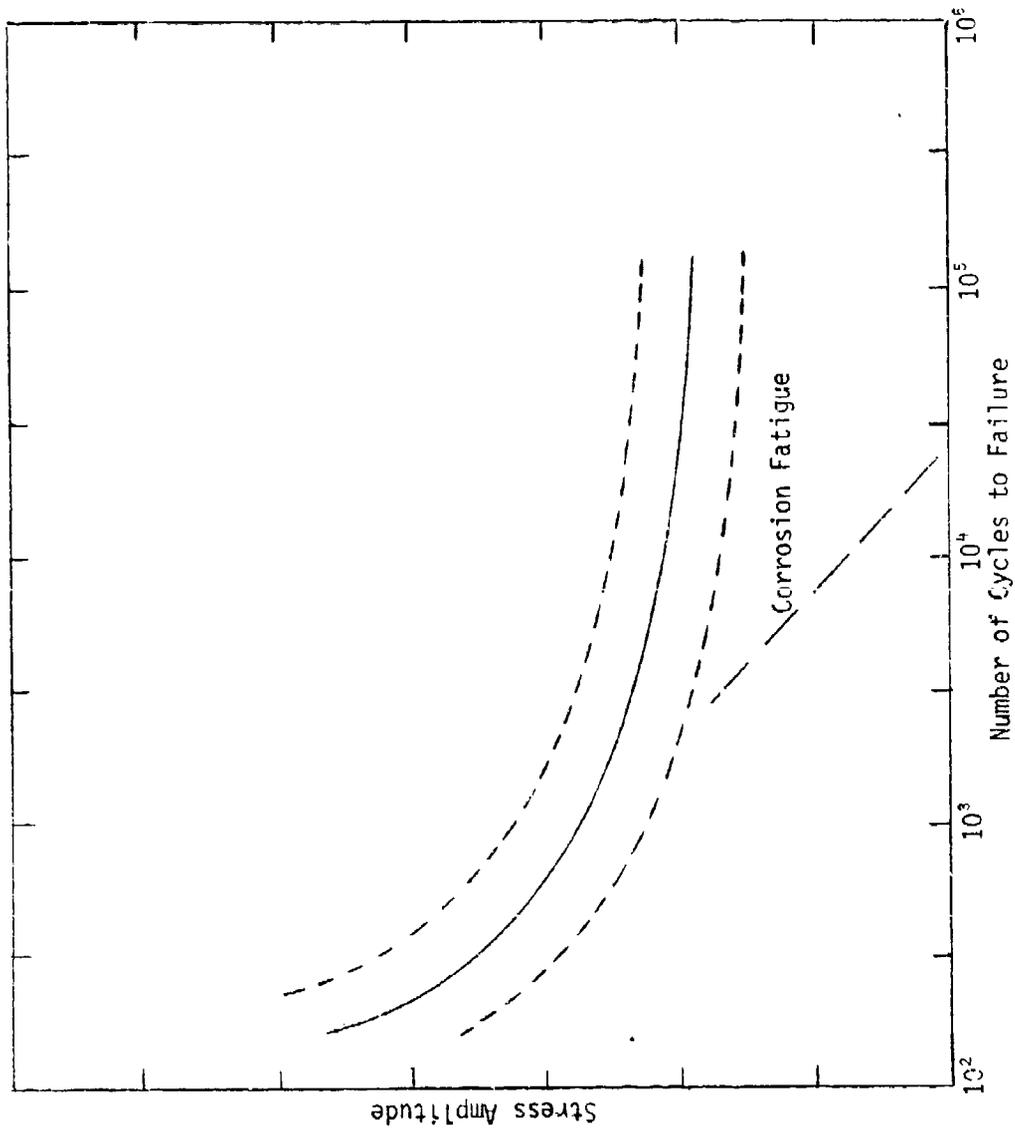


Figure 4-5. Fatigue S-N Curve Typical for Most Metals and Polymers. The Dashed line illustrates marked decrease in fatigue life due to certain environments.

However, methods have been developed⁸⁵⁻⁸⁸ for investigating thin film specimens having thicknesses corresponding to those employed in microcircuit structures. It is considered significant here that several failure analysis experts with extensive experience in analyzing both bulk failures and microcircuit problems stated they see many correlations between failure processes in normal structural materials and in microcircuits. Microstructural considerations in mechanical damage processes are most important to the degradation of electrical performance prior to catastrophic failure. Studies of fatigue damage due to temperature cycling of thin film interconnections have been described by several investigators.⁸⁹⁻⁹¹ Ghate and Blair⁸⁹ mechanically fatigued aluminum film interconnections crossing Si-SiO₂ steps by temperature cycling. After 1500 temperature cycles they noted substantial structural changes in the aluminum films in the form of hillock formation and striations. These features were interpreted in terms of dislocation dynamics and grain boundary sliding processes. It should be noted that slip bands in bulk materials are of comparable dimensions with many microcircuit components and of same order of size as the 8000^Å films studied by Ghate and Blair. Schematic representations of the surface slip bands formed under monotonic and cyclical mechanical stress conditions are shown in Figure 4-6.

The greatest area of concern for mechanical damage is at geometrical configurations where stress concentration occurs. Well documented areas include metallization steps, scratches, bonds and various interfaces between ductile and brittle materials. Bonding and connecting problems in microcircuits are also made more complex by the fact that, in many cases, a large fraction of the material in a member is modified by the bond. Both members are normally mechanically weakened.

Complete mechanical failure is not necessary for a mechanically induced failure to occur in an electronic subsystem. Mechanical defects such as voids, slip lines, partial bonds, fatigue damage, etc. can alter the operation of an electronic component and eventually result in either degradational or catastrophic failure. Predictions of the reliability of an electronic component require first a knowledge of how mechanical defects or damage affect the electronic performance. The interface between adjacent layers will influence the type and extent of mechanical deformation. The epitaxial growth of one layer on another introduces mechanical defects such as growth twins, misfit dislocation networks,²⁹ and interlayer diffusion which also influences the electronic performance of the components.

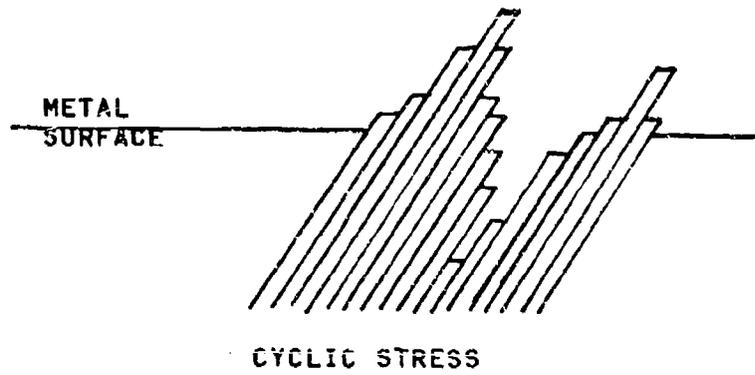
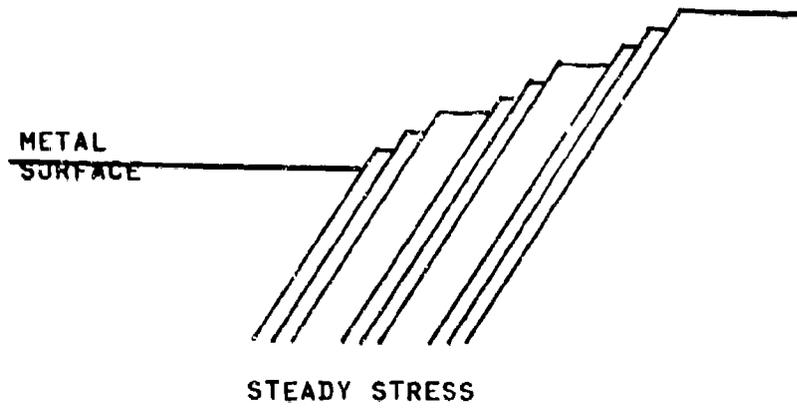


Figure 4-6. Comparison of Surface Damage Within Single Grains of Metals Subjected to Monotonic and Cyclical Stresses.

It is therefore important to determine the type of structural defects to be expected in the various subsystems and then determine their effect on electronic processes. The types of defects can, in some cases, be predicted from a careful evaluation of properties and geometry of the materials used in the construction. However, most of the particular material combinations employed have simply not been investigated to determine the mechanical behavior of the interface systems which appear in microcircuits. The mechanical stress magnitudes introduced by anticipated ambient temperature variations are below the level required for monotonic tensile fracture. However, the mechanical stresses are sufficient to introduce mechanical fatigue problems in critical materials configurations and these fatigue damage processes can be greatly accelerated under certain chemical environments. Configurations most susceptible to thermal cycling induced mechanical fatigue damage include solder connections on circuit boards, polymer materials, die attach bonds, package seals, wire bonds and particular metal-lization regions such as at oxide steps.

Solder alloys, as a general rule, have very poor fatigue strengths. Problems are compounded with less than ideal fabrication processes. Incomplete wetting or, at the other extreme, the exposure of terminals too long in liquid solder results in connections having poor fatigue properties. In the latter case, terminal metal atoms are dissolved into the solder and a brittle alloy results. In addition, geometrical configurations which aid automated fabrication procedures sometimes lead to mechanical stress levels in the solder materials which are much higher than intended.

The maintenance of a hermetic package over the long storage time periods is critical for minimizing integrated circuit corrosion processes. The bonding processes employed to establish package seals do not result in a homogeneous material. Instead, a non leaking seal is probably better described as one in which the voids and microcracks do not provide a continuous path for leakage. Seal leakage is usually best described as effusion. Temperature cycling coupled with environmental gases can cause microcracks to grow such that a previously "good" seal is no longer hermetic at some point in the storage life. The introduction of moisture or other contaminants may thereafter lead to rapid corrosion degradation processes. A mechanism leading to crack growth and subsequent seal failure is schematically illustrated in Figure 4-7.

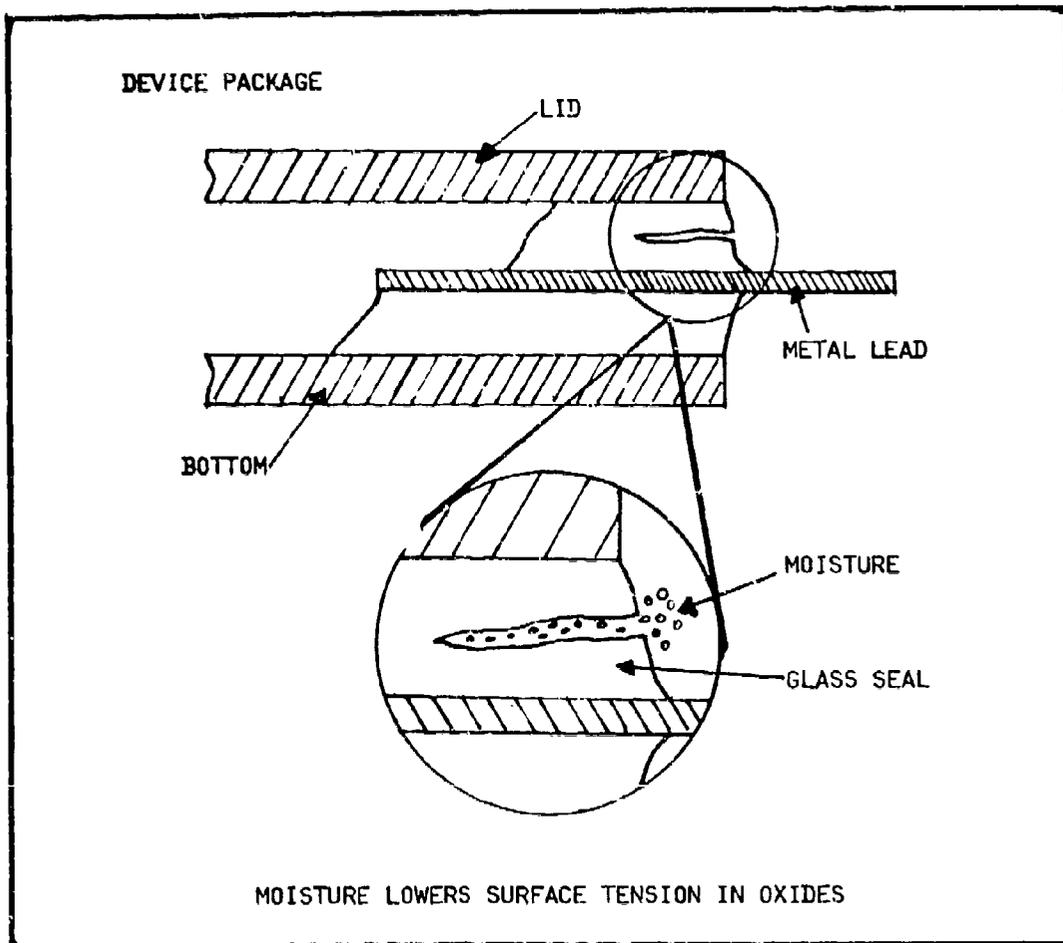


Figure 4-7. Moisture Diffusion Into Seal Crack.

The growth of a crack creates new surfaces. According to the Griffith criteria the average applied stress, S_n , at which a crack will spread is⁹²

$$S_n = \left(\frac{\sigma E}{2C} \right)^{1/2} \quad (4-4)$$

where σ is the specific surface energy, $2C$ is the crack length and E is Young's modulus. From expression 4-4 it is seen that once a crack starts, i.e., as $2C$ increases, the stress required to keep it growing decreases. Also, any quantity which decreases the surface energy, σ , might initiate crack growth. Moisture and other chemicals will reduce σ . The effect of moisture on the fracture strength of glass fibers is dramatic and well known. Subsequent exposure of brittle materials in microcircuits to chemical agents can therefore lead to crack growth at some point in the device storage period if a microcrack were formed either during manufacture or during screen testing.

Brittle materials having critical functions in microcircuits include the above mentioned ceramic seals, the silicon chip, glassivation coatings, oxides, nitrides⁹³ and thick films. Laser trimmed resistors often exhibit microcracks with the potential of changing resistance⁹⁴ if crack growth occurs during storage. The brittle nitride coating on beam lead devices represents the major reliability concern for this technology.⁹⁵ Mechanical stresses crack nitride coatings during wobble bonding operations. One hopefully can detect cracks through measurements of leakage currents as moisture penetrates. Swafford lists⁹⁶ the various failure modes found in beam lead devices and cracks in the nitride coating and in the silicon chip represent about 50% of their observed failures. He points out,⁹⁵ however, that they find significantly fewer defective beam lead devices than corresponding chip-and-wire type devices.

An excellent review of important long-term failure mechanisms which must be considered in the design of high reliability devices for long space missions is contained in a report by Stanley.⁹⁷ Although his concern was primarily with operating devices, much of the information compiled in this report is valuable background data for considering factors important to the tactical missile storage environment. This report deals with a number of mechanically induced problem areas and dwells on the importance of screen test procedures appropriate to the anticipated device usage.

A wide range of studies have been reported which relate to the mechanical integrity of wire bonds. It is outside the scope of this report to provide a detailed review of these studies but some of this work should be brought out here. Reynolds⁹⁸ has subjected integrated circuits and other types of parts to sustained temperature cycling. He reported that some devices exhibited significant numbers of wire bond failures with less than 200 temperature cycles. However, his data showed considerable scatter according to the manufacturer indicating that careful attention to maintaining proper bonding parameters will provide greatly improved bond reliability. Plastic devices were particularly susceptible to temperature cycling. Adams⁹⁹ conducted a type of temperature cycling study on dual-in-line plastic packages containing gold bond wires and noted failures due to fracture at wire mid-spans. He conducted a stress analysis based on the package geometry and estimated materials parameters to show that expected wire stresses were about 66 percent of the ultimate tensile strength. He interpreted the 45° fractures as due to grain growth and a particular slip process. His interpretation of the fracture occurring by extended slip along a single slip plane is interesting and should be verified using detailed mechanical testing procedures and microstructural analysis including x-ray diffraction. The development of single crystal regions in aluminum bond wires had previously been reported.⁹⁷ The present authors had noted that micrographs of some bond wire failures found at various industrial and government laboratories showed evidence of slip lines. Bond wires are subjected to conditions sometimes used for the growth of single crystals in aluminum by the stress-anneal technique. However, the gold wires are another matter. If it were verified that bond wires could sometimes be transformed into single crystals during, say, the high temperature bake screen test, then a potentially serious mechanical problem would be identified. Single crystals of fcc metals always deform at much lower stresses than do polycrystals.

A comprehensive series of bond technology investigations have been underway at the National Bureau of Standards.¹⁰⁰⁻¹⁰⁵ These investigations have examined details of the bonding processes and developed certain mechanical testing procedures for individual bond wires. At RCA, Hitch^{106,107} has been investigating details determining the mechanical characteristics of bonds made to thick film conductors. Hitch has evaluated the chemical

and physical properties of thick film materials in relation to the adhesion and solderability performance of these materials.

Fitch¹⁰⁸⁻¹¹⁰ has conducted a series of studies to evaluate the effects of temperature cycling on devices produced by various manufacturers. He established that mechanical fatigue wear-out processes are initiated with the first thermal cycle and damage progresses with subsequent cycling. He also found that the rate of change of temperature had little effect on wire bond strength but had a pronounced effect on package humidity and measured seal strength. The strength of devices in thermal cycling varied with vendors. In addition, a greater failure rate was noted for longer wires. Wear-out processes under thermal fatigue began to introduce most problems after 1000-4000 cycles.

Anderson¹¹¹ at General Dynamics, Pomona, established a special screen on purchased devices which involved more severe mechanical testing than normally employed. This test sequence includes 25 temperature cycles at a faster than normal rate, sometimes an air impact mechanical shock test and acoustic particle detection. They have thereby accumulated a large amount of mechanical failure data on devices which already had passed Class B screen tests. Principal problems include chip cracking under bonds and polymer induced bond failures for improperly cured materials. They noted that the temperature cycling sometimes aggravated intermetallic compound growth problems in bonds but generally find that the 25 cycles do not cause many bond failures on these previously screened devices.

The relationships between thermal cycling, T.C., and thermal shock, T.S., were brought up by many individuals. Unfortunately there exists a lot of confusion about the response of materials to these stress modes. Some individuals want to substitute T.S. for T.C. testing in screening procedures, primarily because of costs. This would be a serious mistake. The damage caused by T.S. to brittle materials such as seals and the chips is severe. Subsequent loss of hermeticity and drift in circuit gain is a risk here. The metallic components used in microcircuits are generally ductile and not likely to provide a different response for T.S. and T.C. As discussed earlier, T.S. is not one of the storage stress modes and therefore it should be carefully avoided because of other problems so introduced unless operational conditions dictate further consideration.

4. Chemical Failure Processes

The overwhelming area of concern as expressed by the micro-electronics personnel for long-term reliability is the influence of chemical contaminants introduced either from the environment or else during fabrication. As stated earlier, moisture may be the single most important factor to long-term storage reliability. The open literature and various government and private reports¹¹³ discuss a large number of corrosion induced failures in microcircuits. The amount of water required to degrade the materials within a circuit is known to be very small, perhaps even as little as one molecular layer. Some of the potential sources of contaminant ions were listed in Section C of this chapter and described in greater detail by Himmel.³³ Certain ions very rapidly degrade the microcircuit structure where moisture is available but might remain harmless indefinitely in a dry package. The amount of such ions necessary for corrosion is very small. For example, ions such as Cl^- transport metals in an electrolyte by forming metal complexes such as AlCl_3 . Once the Al is deposited, the Cl^- ion is available for repeated dissociation cycles.

Examples of important moisture induced corrosion processes include pitting of metallization layers, inversion in LSI devices, various types of metallic whisker growth, intermetallic bond degradation and various electromigration processes. Secondary effects of moisture include the formation of phosphoric acid in circuits involving phosphorous doped glass passivation. Where the phosphorous content is a little higher than the 4-6% desired for mechanical stability, water will combine with the phosphorous to make phosphoric acid. Aluminum films are quickly degraded by the acid. Gold dendritic growth (also called migrated-gold resistive shorts) is known to occur only in packages with moisture, halogen ions and electrical bias. Experimental investigations have indicated a threshold of 1.5% moisture ambient within the package¹¹⁴ is required for dendrite growth. However, additional study is needed to determine if 1.5% represents a fundamental threshold level or simply implies that the process didn't occur in the packages involved in the study below that value. Are there halogen ion concentration or electrical bias threshold levels? MGRS should not be a storage reliability problem unless the dendrite growth took place during testing but was not sufficient for early detection or else was able to proceed at a low rate during long dormant storage periods with galvanic

potentials providing the driving force. However, no experimental evidence for such galvanic induced dendrite growth processes were found. Other moisture induced mechanisms include crack propagation in brittle materials such as ceramic seals, glass passivation layers, nitride coatings, laser trimmed resistors and the silicon chip itself. Efforts to protect parts by polymer coating may be defeated because moisture is transported through plastics and will displace the polymer bond. Polymer coatings protect against particles, not moisture. Mechano-chemical processes also are important in microcircuits just as they are for normal structural materials. Stress corrosion and corrosion fatigue failures occur from residual stresses, vibration, thermal shock or thermal cycling where applicable. Microcircuit failure analysis has identified a large number of moisture induced degradation processes of tremendous importance to the reliability of solid state devices. However, very little work has been accomplished providing detailed data concerning the important fundamental parameters controlling the degradation reactions. Degradation threshold levels for moisture content and specific contaminating species need to be established where possible.

The problems associated with oxide impurities in MOS devices are discussed separately in Chapter VI and many polymer contamination problems are described in Chapter VII. There are general indications that plastic materials have been improved over the past several years. However, where plastics must be used for die attach, there remains the danger of a chemical agent being released from the material subsequent to sealing. The critical question then centers upon the specific chemicals which come out of the polymer used and how the particular circuit materials react to these chemicals. Extreme caution must be used whenever plastics are included inside a package.¹¹⁵ Most agree that the manufacturer must always be responsible for proving "chemical compatibility" for specific types of circuits and plastics. It is also noted that polymer suppliers will, at times, change the composition of their product without any notification.

Specific thick film materials also introduce potential chemical reliability problems as described by Hitch.⁹⁴ He has shown that gold frit is chemically stable although bonding is difficult because of glass at the surface. Silver is particularly bad about migration. Solders also take into solution metallic components of the thick films.¹¹⁵ The dissolution of various pure metal base wires by solder alloys is well known.¹¹⁷ The critical point is that the interface between the base metal and the solder

may consist of brittle intermetallic compounds subject to cracking and mechanical failure.

Corrosion processes and surface contaminants result in poor bonds. It should be noted that a reactive environment is not always detrimental. Rossiter¹¹⁸ found, for example, that oxygen apparently extended the time to failure of Au-Al bonds at 250°C. Jellison,¹¹⁸ however, demonstrated that impurities such as humidity and trace amounts of chlorine lead to corrosion induced bond degradation problems. Surface contaminations may occur from many sources. These include organic materials from photoresist residue, vapors from epoxies, long chain polymers not detected in deionized water and even adsorption from supposedly clean laboratory air.^{120,121} Other surface contaminants include fingerprint "grease," etching chemical residues, "specks of dust," residual glassivation and salt spray elements for plants near oceans. A bonding tool may be contaminated due to handling with bare hands and transfer impurities to the wire. In addition, the manufacturer does not always supply wire clean over its full length. All of these contaminants lead to poor bonds. One individual said a leaking vacuum pump exhaust near bonding machines led to a situation where only 1% of the bonds would stick at all.

Trace amounts of secondary materials within either the wire metallization or a plated surface lead to other types of problems. For example, we were told that one wire manufacturer is pushing gold wire with 0.5% Be to improve strength but this was said to introduce problems with grain boundary diffusion. In another case, trace amounts of cobalt were placed in the gold plating bath to increase hardness. However, this hardness made it difficult to achieve the desired amount of deformation during bonding so poor bonds resulted.

A well recognized problem concerns the silicon precipitates in 1% Si-Al bond wires. The phase diagram of the Al-Si alloy system is shown in Figure 4-8. The maximum solid solution for Si in Al is 1.65% Si at 577°C. It is clear that a wire production process slightly out of control could lead to the precipitation of silicon grains. The desired strengthening effect of the 1% silicon occurs from incipient precipitation. An over-aged 1% Si-Al wire would have silicon particles distributed along the length of the wire, making bonding dependent upon local variations in the distribution of these particles. One wire supplier sent micrographs showing that

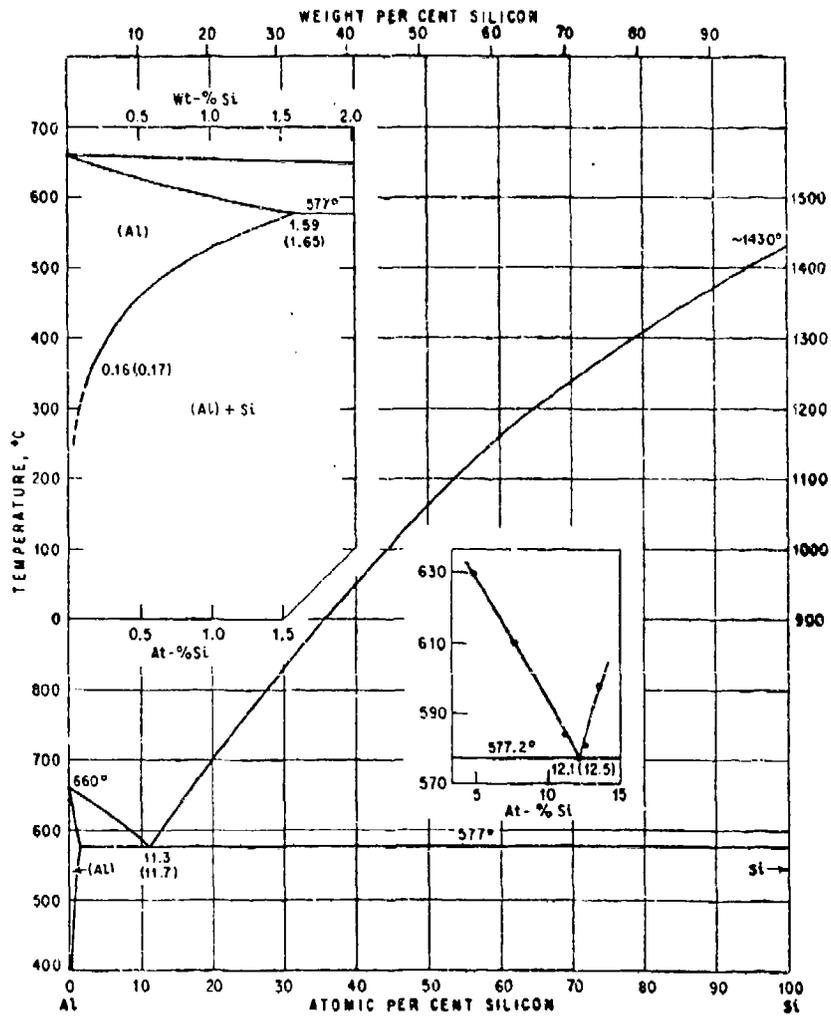


Figure 4-8. Aluminum-Silicon Phase Diagram. (From M. Hansen, Constitution of Binary Alloys, McGraw-Hill, 1958).

their wires have a homogeneous silicon distribution but the "competition" has silicon precipitates visible at 1500X. The "competition's" wire led to some important bond reliability problems for the Navy. Klein,¹²² formerly at the Crane Naval Weapons Center, said they periodically section and polish bond wires long-ways to track down this problem. It should be recognized that a previously homogeneous wire can have silicon precipitate out if it inadvertently experiences an elevated temperature for sufficient time.

Impurities within a coating may introduce problems through several mechanisms. Metallic impurities such as Ni, Cr, Co, Cu, Ti and Ag have been shown to move to the surface of a coating under the proper thermal-chemical conditions. These trace metals may be present as an actual impurity in an out of control plating bath or deposition system or else there may be an undercoating that diffuses into the gold layer. A large firm on the West Coast had bond sticking problems which ultimately were attributed to the presence of Cr_2O_3 at the surface of an Au film. The Cr was a sublayer film. Similarly, a large government laboratory identified TiO_2 at the surface of a multilayered Ti-Au film as the cause of a bond problem. The chemical potential favors the diffusion of such metals as Cr, Ti and Ni to the surface of Au in the presence of O_2 at an elevated temperature. It should be pointed out that these trace metal oxides are very thin and are usually detected only by Auger analysis.

Impurities may play a different part in the degradation of intermetallic bonds such as between aluminum and gold. The work of Horsting²¹ showed that impurities segregate at interfaces between aluminum and gold intermetallic compounds. The "impurities" include components of a thick film material. Horsting proposed that the impurity concentration reached some critical level as the diffusion front progressed through the gold and was arrested by precipitation and the occurrence of voids. This precipitation-void front developed adjacent to the AuAl_2 purple phase and was identified as the real cause of bond lifting in cases of the so-called "Purple Plague" discussed above. This work has been followed up by Newsome, et al.,¹²³ who showed that a thick film frit enhanced intermetallic formation and voiding at the gold-intermetallic interface. They found rapid growth of the intermetallics at low temperatures. Oswald, et al.¹²⁴ found that the resistance of such bonds increased significantly after being subjected to temperature

cycling. Apparently the thermal stresses cannot be relieved in the relatively hard impurity precipitation-void plane at the interface between AuAl₂ and gold.

Wagner¹²⁵ showed differences in contamination levels of incoming material and in certain stages of the cleaning process of microelectronic components. Comparison between AES results for a sputtered gold film and a typical gold thick film indicated that the sputtered gold surface was relatively free from contamination while the thick film gold surface displayed a large percentage and variety of contamination (including silicon from the glass frit). The gold surface with the least contamination gave superior bondability. In a gold fritless thick film the major impurity observed on the surface was copper.

In cleaning operations there exists the possibility of impurities being added rather than being removed in the process. An AES analysis of the surface of a gold bonding pad following cleaning revealed the presence of silver and iodine. The assumption was made that the cleaning solvents were contaminated by silver from conductive epoxies on the hybrid microcircuit and by iodine solutions used in gold etching operations.

A further example given by Wagner is a lift-off failure of aluminum ultrasonic wire bonds from an electroplated gold thin film following a time-temperature stress of 68 hours at 150°C. Prior to the stress, wire bonds had an average bond pull strength of 11.9 gm and the Auger analysis showed that the surface was relatively free of contamination. Subsequent to the stress impurities such as Ca, Ag and Cd appeared at the surface. It was concluded that the contamination resulted from the diffusion of electroplating bath impurities to the bond interface during the time and temperature stress.

Further evidence of the effect of time and temperature stresses on the diffusion of impurities to the surface of gold platings is presented by Unger and McKee, of NELC.¹²⁶⁻¹²⁸ As an example, a particular batch of material that showed acceptable bondability initially (bond pull strength of greater than 9 grams) degraded drastically after temperature stressing at 150°C for 68 hours. The AES analysis of the bond interfaces showed the presence of Ag, C, Ca, Cd, O and Zn impurities. As mentioned by Wagner,¹²⁵ the temperature stressing apparently caused the contaminants to migrate or diffuse to the bond interface, a resulting degradation of bond strength and a bond lift-off failure mode. Unger notes that, because of such results,

material bondability acceptance standards should include bondability after temperature stressing. One can readily see that the stabilization bake screen may have a similar effect on the bonds to contaminated gold platings and that during long periods of storage of Army missiles the diffusion processes will occur with resulting bond failures, possible in critical components of the missile electronics.

Several papers discussed the role of contaminants in the bondability of microcircuit materials at an ARPA-NBS workshop.¹²⁹ J. M. Morabito¹³⁰ reported on Auger results obtained on a gold plated ceramic after pre-bond cleaning. The pre-bond cleaning which included a heat treatment of 120°C for 1 hour increased the Cu to Au and Ag to Au ratios and there was a large increase in the oxygen peak suggesting that these impurities were present as oxides. It was believed that the Cu and Ag diffused to the surface during pre-bond cleaning. A correlation is shown by Morabito between the presence of Ag, Cu, O and Sn and lead failures. In the discussion of the paper it was stated by Thomas that the most frequently recurring bonding problem is that due to the oxides of copper, nickel or iron.

F. J. Gruthaner¹³¹ discusses the applications of x-ray photoelectron spectroscopy to the study of organic surface contamination, residues of etchants and copper in MOS structures, as well as the MGRS problem. McGuire¹³² gives an example showing Auger spectra for good and poor gold-plated surfaces. The presence of potassium, carbon, nitrogen and tin was associated with Au-plated surfaces having poor bonding characteristics.

Reich and Hakim¹³³ have been conducting extensive reliability tests on plastic devices in Panama. Hakim¹³⁴ notes that nearly all catastrophic failures of plastic encapsulated devices failed from aluminum metallization corrosion on top of the die. He identified the corrosive agents to be by-products of the encapsulation process, transmitted moisture and exterior reactive agents. They also stated that copper oxide combines with certain organic materials to produce water in sealed packages. Shumka lists¹³⁵ the principal corrosion problems noted in JPL work to be migrating gold resistive shorts, aluminum corrosion, dissolution of nichrome resistors and package corrosion.

A number of other chemical problems also have the potential for degrading devices in storage. It obviously is important to keep the devices clean. The unknown factor at this time is how clean a device must be for degradation

not to occur. Threshold levels have not been established for the important storage degradation processes.

5. Thermal Degradation Processes

The temperatures associated with stored missile systems are generally low compared to those of most operating devices. Atomic diffusion constants follow the temperature dependence of Equation 4-2 so that inter-metallic diffusion should be slow. However, there is concern¹³⁶ about low temperature diffusion problems such as Cr-Cu-Au interfaces at 100°C and Sn on 100μ Au at 125°C. Mechanical defects and contaminants have strong effects on these rates as discussed earlier. There are certain chemical reactions which have greatest importance near the dew point so that an electronic device is clearly not inert during storage. In fact, we were told that RCA now spends a lot of time on low temperature testing of C/MOS devices.¹³⁷ Higher temperatures drive contaminants off the chip surface and arrest the degradation process.

While the three storage stresses, mechanical, chemical and purely thermal, have been discussed as separate items, it is clear that no one of these stresses ever acts alone. The synergism of these three stress factors must be carefully evaluated for the specific condition expected within a package in storage. For example, certain chemical reactions would probably not proceed under low temperature storage conditions were it not for the introduction of mechanical stresses due to temperature changes. Conversely, chemical assisted mechanical failures must be evaluated where the strength of the composite structure would have been sufficient in a contaminant free package. The dew point has been pointed out as critical to reliability by many individuals. The mechanical and chemical degradation processes are found to be greatly aggravated by temperature cycling through the dew point of the package atmosphere. Obviously, a drier package is desired.

It is tremendously important to understand the aging of all materials going into missile systems. This point was of considerable concern to personnel at Aerospace¹³⁸ where they must worry about both the long-term storage and operation of military satellites. They said there appears to be no organized search for aging data. Age-sensitive design analysis of complex systems should be carried out by the contractor so that intelligent judgments can be made relative to combinations of long storage and long use times. What are the wear effects of excessive turn on-off cycles?

E. Particulate Contamination

Conducting particulates represent a major potential storage failure mechanism due to problems with their detection during screen testing. This particular matter was one of the most universally expressed areas of concern at the different facilities visited during this program. Many organizations have developed their own testing equipment¹³⁹ although complete systems are now commercially available. Conducting particulates are potentially introduced into a package at many stages of the fabrication process. They include wire fragments, broken silicon chips, metallization flakes, solder balls, weld balls, and extraneous metallic debris. Glass and plastic coatings have been developed to minimize the chances of their shorting-out most parts of a device.

The particles are a storage reliability problem because they are often trapped and not detected during screen testing but may somehow be released due to transportation vibrations while in storage. The approach to this problem is great care during every manufacturing step and 100% testing of devices using one of the vibration-acoustical noise monitoring test methods. Greater testing sensitivity and improved ability for operators to interpret test findings are current goals of many organizations. Considerable care is needed by the Army and their missile parts manufacturers to control particle problems.

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V. HERMETICITY

A. Introduction

Microelectronic components encapsulated in glass, ceramic, or metal packages are subjected to ambient atmospheres during their operating and storage life. Hermeticity implies a perfect package seal such that the electronic components are protected from the detrimental effects of atmospheric constituents, particularly water vapor. Screen tests for hermeticity in MIL-STD-883A are designed, however, to assure that leak rates are below a certain level, which is usually the sensitivity of the leak detection instrument used or below a specified standard. Therefore, it has not been possible to guarantee a hermetic seal, i.e., a zero leak rate. One should then expect that with a finite leak rate there will be a gaseous exchange process taking place during the time that missile systems are in storage. The primary concern for long term storage reliability should be the sensitivity of electronic components, bonds, metalizations, etc. to the presence of water vapor in the package. Sources of water vapor include the sealed-in atmosphere, epoxies used for die attachment, polymer conformal coatings, leakage through polymer seals, leakage through minimal leaks for devices that pass the screen tests and leakage due to seal failures occurring as a result of storage conditions, e.g., ambient temperature variations. Because of their importance the problems inherent to polymers and epoxies are addressed in a separate chapter of this report (Chapter VII). In this chapter we will provide summaries of Method 1014.1 of MIL-STD-883A on testing seals and Method 1013 on dew point measurements. The purpose of the dew point test is to detect the moisture present inside the microelectronic package in terms of device leakage currents. A more direct method for determining water vapor and other gaseous components within packages, involving quadrupole mass spectrometry, has been used by Dr. R. W. Thomas of RADC. This method and typical results will be discussed. Finally, the consequences of water vapor within the package on such effects as corrosion, ionic drift, phosphosilicate glass reactions and the formation of migrated-gold resistive shorts are presented.

B. MIL-STD-883A Test Methods

MIL-STD-883A establishes uniform methods and procedures for testing

microelectronic devices, including basic environmental tests and physical and electrical tests. Method 1014.1 has as its purpose the determination of the effectiveness (or the hermeticity) of the seal of microelectronic devices with designed internal cavities. This may be accomplished by a variety of fine and gross leak rate tests. Method 1010 has as its purpose the detection of moisture trapped inside a microelectronic package by the effect on device leakage current at the dew point, which for an acceptable device, should be lower than -65°C . Both of these test methods are included below in abridged form for convenient reference.

1. Test Method 1014.1

Test Condition A_2 of Method 1014.1 specifies that the maximum allowable leak rate into vacuum or more precisely the equivalent standard leak rate (L), calculated from the measured leak rate (R_1) and other parameters of the test, should not be greater than 5×10^{-7} atm cm^3/sec for packages with an internal cavity volume of less than 0.1 cm^3 and not greater than 5×10^{-6} atm cc/sec for packages with an internal cavity volume greater than 0.1 cm^3 .

The test procedures used, Test Condition A_2 or A_3 , involve first of all the introduction of helium as a tracer gas into the package either by bombing (A_2) or by prefilling with an ambient which contains a minimum of 0.2 atm of the helium (A_3). Upon completion of the bombing, or sealing the package in A_3 , there is a finite time during which the device package is at atmospheric pressure while it is being transferred to the vacuum chamber for testing with a mass spectrometer leak detector.

The equation that is used for bombed packages to relate the measured leak rate to the leak size is the Howl and Mann equation for molecular flow¹, given in Method 1014.1 of MIL-STD-883A as follows:

$$R_1 = \frac{LP_E}{P_0} \left(\frac{M_A}{M} \right)^{\frac{1}{2}} \left\{ 1 - \exp \left[\frac{-Lt_1}{VP_0} \left(\frac{M_A}{M} \right)^{\frac{1}{2}} \right] \right\} \exp \left[\frac{-Lt_2}{VP_0} \left(\frac{M_A}{M} \right)^{\frac{1}{2}} \right]$$

where

- R_1 = the measured leak rate of tracer gas (He) through the leak in atm $\text{cm}^3 \text{ sec}^{-1}$;
- L = the equivalent standard leak rate in atm $\text{cm}^3 \text{ sec}^{-1}$, also called *leak size*,

SEAL

1. PURPOSE. The purpose of this test is to determine the effectiveness (or the hermeticity) of the seal of microelectronic devices with designed internal cavities.

1.1 Definitions:

- (a) Standard leak rate. Standard leak rate is defined as that quantity of dry air at 25°C in atmosphere cubic centimeters flowing through a leak or multiple leak paths per second when the high-pressure side is at 1 atmosphere (760 mm Hg absolute) and low-pressure side is at a pressure of not greater than 1 mm Hg absolute. Standard leak rate shall be expressed in units of atmosphere cubic centimeters per second (atm cc/sec).
- (b) Measured leak rate. Measured leak rate (R_1) is defined as the leak rate of a given package as measured under specified conditions and employing a specified test medium. Measured leak rate shall be expressed in units of atmosphere cubic centimeters per second (atm cc/sec). For the purpose of comparison with leak rates determined by other methods of testing, the measured leak rates must be converted to equivalent standard leak rates.
- (c) Equivalent standard leak rate. The equivalent standard leak rate (L) of a given package, with a measured leak rate (R_1), is defined as that leak rate of the same package with the same leak geometry, that would exist under the standard conditions of 1.1 (a). The formula in paragraph 3.1.1.2 represents the L/R_1 ratio and gives the equivalent standard leak rate (L) of the package with a measured leak rate (R_1) where the package volume and leak test conditioning parameters influence in the measured value of (R_1). (See complete description for further details). The equivalent standard leak rate shall be expressed in units of atmosphere cubic centimeters per second (atm cc/sec).

2. APPARATUS. The apparatus required for the seal test shall be as follows for the applicable test condition.

2.1 Test conditions A_1 , A_2 and A_3 - Tracer gas helium (He) fine leak. Apparatus required for test conditions A_1 and A_2 shall consist of suitable pressure and vacuum chambers and a mass spectrometer type leak detector preset and properly calibrated for a He leak rate sensitivity sufficient to read measured helium leak rates of 10^{-9} atm cc/sec and greater. The volume of the chamber used for leak rate measurement should be held to the minimum practical, since this chamber volume has an adverse effect on sensitivity limits. The leak detector indicator shall be calibrated using a diffusion type certified standard leak at least once during every working shift. For test condition A_3 , all of the above apparatus except the pressure chamber is required.

2.2 Test condition B - Radioisotope fine leak.

(See complete description)

2.3 Test condition C₁ and C₂ - Fluorocarbon gross leak.

(See complete description)

2.4 Test condition D - Penetrant dye gross leak.

(See complete description)

2.5 Test condition E - Weight gross leak.

(See complete description)

3. PROCEDURE. Fine and gross leak tests shall be conducted in accordance with the requirements and procedures of the specified test condition. The fine leak test shall be performed prior to the gross leak test, unless otherwise specified by the applicable procurement document. Where bomb pressure specified exceeds the microcircuit package capability, alternate pressure and time (bomb and dwell) conditions may be used provided they satisfy the leak rate, pressure, time relationships which apply, and provided no less than 30 psig bomb pressure is applied in any case.

3.1 Test condition A₁ or A₂ - Tracer gas (He) fine leak. Test condition A₁ is a "fixed" method with specified conditions that will insure the test sensitivity necessary to detect the required measured leak rate (R₁). Test condition A₂ is a "flexible" method that allows the variance of test conditions in accordance with the formula of paragraph 3.1.1.2 or Table I to detect the specified equivalent standard leak rate (L) at a predetermined leak rate (R₁). (See complete description for further details)

3.1.1 Procedure applicable to "fixed" and "flexible" methods. The complete device(s), shall be placed in a sealed chamber which is then pressurized with a tracer gas of 100⁺⁰₋₅ percent helium for the required time and pressure.

The pressure shall then be relieved and each specimen transferred to another chamber or chambers which are connected to the evacuating system and a mass spectrometer type leak detector. When the chamber(s) is evacuated, any tracer gas which was previously forced into the specimen will thus be drawn out and indicated by the leak detector as a measured leak rate (R₁).

3.1.1.1 Test condition A₁ - Fixed method. Unless otherwise specified, the bomb pressure shall be 75 psig minimum, exposure time shall be one hour minimum, and the device shall be measured within 30 minutes after it has been removed from the pressure vessel. Unless otherwise specified, devices with an internal cavity volume of 0.1 cc or less shall be rejected, if the measured tracer gas leak rate (R₁) exceeds 5x10⁻⁸ atm cc/sec; and devices with an internal cavity volume greater than 0.1 cc shall be rejected, if the measured tracer gas leak rate (R₁) exceeds 5x10⁻⁷ atm cc/sec. This fixed method shall not be used if the maximum standard leak rate limit given in the procurement document is less than the limits specified herein for the flexible method.

3.1.1.2 Test condition A₂ - Flexible method. Values of bomb pressure, exposure time, and dwell time shall be chosen such that actual measured tracer gas leak rate (R₁) readings obtained for the devices under test (if defective) will be greater than the minimum detection sensitivity capability of the mass spectrometer. Unless otherwise specified, devices with an internal cavity volume of 0.1cc or less, shall be rejected if the equivalent standard

leak rate (L) exceeds 5×10^{-7} atm cc/sec; and, devices with an internal cavity volume greater than 0.1 cc, shall be rejected if the equivalent standard leak rate (L) exceeds 5×10^{-6} atm cc/sec. (See complete description for further details)

3.1.2 Test condition A₃ - Tracer gas (He) Fine leak. This test method may be used for packages that have been enclosed in such a fashion as to insure that the package ambient contains a minimum of 0.2 atmosphere absolute partial pressure of tracer gas (He) at standard temperature. Upon completion of the package seal, the device shall be transferred to a chamber connected to an evacuating system and a mass spectrometer type leak detector. Transfer time (total time between completion of seal and completion of test) shall be less than 30 minutes. Any tracer gas that leaks out will be indicated by the leak detector as a measured leak rate (R₁). The measured leak rate (R₁) is converted to the equivalent standard leak rate (L) by applying the following formula:

$$L \text{ (atm cc/sec)} = R_1 \text{ (atm cc/sec)} \times \frac{\text{Total internal pressure (atm absolute)}}{\text{He internal partial pressure (atm absolute)}}$$

Unless otherwise specified, devices with an internal cavity volume of 0.1 cc or less, shall be rejected if the equivalent standard leak rate (L) exceeds 5×10^{-7} atm cc/sec; and, devices with an internal cavity volume greater than 0.1 cc, shall be rejected if the equivalent standard leak rate (L) exceeds 5×10^{-6} atm cc/sec. A sampling inspection shall be conducted on each eight hour shift to verify that the specified amount of tracer gas (He) is actually being sealed within the package.

3.2 Test condition B - Radioisotope fine leak test.

(See complete description)

3.3 Test condition C₁ or C₂ - Fluorocarbon gross leak. C₁ is designed to detect package leaks $\geq 10^{-3}$ atm cc/sec. C₂ is designed to detect package leaks $\geq 10^{-5}$ atm cc/sec. Unless otherwise specified, test condition C₂ shall apply as a minimum. (See complete description)

3.4 Test condition D - Penetrant dye gross leak.

(See complete description)

3.5 Test condition E - Weight measurement gross leak.

(See complete description)

3.6 Retest. Devices which fail gross leak (test conditions C₁, C₂, D, and E) tests shall not be retested for acceptance. Devices which fail fine leak (test conditions A₁, A₂, A₃, or B) tests shall not be retested for acceptance unless specifically permitted by the applicable procurement document. Where fine leak retest is permitted, the entire leak test procedure for the specified test condition shall be repeated. That is, retest consisting of a second observation or leak detection without a re-exposure to the tracer fluid or gas under the specified test condition shall not be permissible under any circumstances. Preliminary measurement to detect residual tracer gas is advisable before any retest for test condition B.

4. SUMMARY. The following details shall be specified in the applicable procurement document:

- (a) Test condition letter when a specific test is to be applied (see 3).
- (b) When other than specified accept or reject leak rate applies for test conditions A₁, A₂, A₃, and B (see 3.1.1.1, 3.1.1.2, 3.1.2, and 3.2).
- (c) Where applicable, measurements after test (see 3).
- (d) Retest acceptability, test conditions A₁, A₂, and B (see 3.6).
- (e) Order of performance of fine and gross leak tests if other than fine followed by gross (see 3).

P_E = the pressure of exposure in atm;

P_0 = atmospheric pressure (1 atm);

M_A = the molecular weight of air (28.7);

M = the molecular weight of tracer gas (He)(4);

t_1 = the time of exposure to P_E , in sec;

t_2 = the dwell time between release of pressure and leak detection, in sec;

V = the internal volume of the device package cavity in cm^3 .

Davy² discusses simplifications of the Howl and Mann equation and problems related to helium leak testing of hermetic packages. Current problems are also covered in the NBS Special Publication 400-9 on Hermeticity Testing for Integrated Circuits.³ This publication is a report on the ARPA/NBS Workshop held at NBS on March 29, 1974.

Leak testing of microelectronic packages has routinely been performed in accordance with MIL-STD-883A screens but they often only reflect the capabilities of the package sealing techniques or the mass spectrometer leak detectors. Fundamental considerations like the buildup of water vapor and other gaseous contaminants in a package and the level of water vapor in a package that would not affect device performance and reliability have not until recently received much attention. To illustrate the situation dramatically, Davy calculates the rate, \dot{p}_w , at which the partial pressure of water vapor, p_w , accumulates inside a package of volume V for a use leak rate of R_U (the package exposed to ambient air at 1 atm). Assuming that an average value for the partial pressure of water vapor in air is 0.02 atm, the rate at which water vapor enters the package is about $0.025R_U$ and, multiplying by $10^6/V$, the rate of change of water vapor partial pressure is:

$$\dot{p}_w = 2 \times 10^4 R_U/V \text{ (ppm sec}^{-1}\text{)}$$

$$\text{or } \dot{p}_w = 6.3 \times 10^{11} R_U/V \text{ (ppm yr}^{-1}\text{)}$$

If, for example, the use leak rate for a 1 cm^3 package is $5 \times 10^{-6} \text{ atm cm}^3 \text{ sec}^{-1}$, the rate of water buildup would be

$$\dot{p}_w = 2 \times 10^4 (5 \times 10^{-6}) = 10^{-1} \text{ ppm sec}^{-1}$$

$$\text{or } 8.6 \times 10^3 \text{ ppm day}^{-1}$$

Such a package would be at equilibrium with the atmosphere in the order of days. For extended storage of missiles it is thus evident that either the leak test requirements should become more stringent or the storage conditions should be at low relative humidities at the least if current leak standards are maintained.

In a recent paper given at the 1977 Reliability Physics Symposium Strohle⁴ points out that a critical failure mechanism for long life applications is the electrolytic corrosion of aluminum interconnections. His investigations show that the maximum allowable leak rates specified in MIL-STD-883A are not sufficiently low to prevent the onset of corrosion and that a helium leak rate of 10^{-10} atm cm³ sec⁻¹ should be required, as well as a dry atmosphere during package sealing.

2. Test Method 1013

The presence of moisture in microelectronic packages is likely to have a disastrous effect on the electronic components in the package especially over the long periods of storage. Whether there will be an effect or not depends on the sensitivity of the internal devices or materials to moisture-related phenomena, such as, increased leakage currents and corrosion of aluminum metallization or bonds. Even in devices which are to be operated and not stored before operation it would be desirable to reduce the amount of moisture sealed into a device package. However, failure of a package seal at some later time in the operating or storage life could cause an almost immediate exchange of the residual gases with the external environment. Method 1013 is an attempt to design a test that will detect the moisture present inside microelectronic device packages in sufficient quantity to adversely affect device parameters. There are several comments that can be made on the usefulness of this test method.

The test description implies that device leakage current is the most sensitive indicator of moisture in the package yet the actual specification of the type device used and the appropriate test voltages and currents is left for the applicable procurement document. Some devices may not exhibit sufficient changes in leakage current and the addition of special devices mainly for monitoring moisture may not be economical or convenient.

DEW POINT

1. PURPOSE. The purpose of this test is to detect the presence of moisture trapped inside the microelectronic device package in sufficient quantity to adversely affect device parameters. The most sensitive indicator of moisture is device leakage current. This test specifies a lower temperature of -65°C for the normal dew point test. It may be desirable in some cases, where the presence of moisture in concentrations lower than that would be revealed at this lower temperature, to extend the lower temperature downward.
2. APPARATUS. The apparatus used in this test shall be capable of varying the temperature from the specified high temperature to -65°C while the parameter is being measured.
3. PROCEDURE. The voltage and current specified in the applicable procurement document shall be applied to the terminals and the device leakage current or other specified parameter(s) continuously monitored from the specified high temperature to -65°C and back to the high temperature. The dew point temperature is indicated by a sharp discontinuity in the parameter being measured with respect to temperature. If no discontinuity is observed, it shall be assumed that the dew point is at a temperature lower than -65°C , and the device being tested is acceptable. Devices which demonstrate instability of the measured parameter at any point during this test shall be rejected even though a true dew point is not identified. If a high temperature is not specified in the applicable procurement document, the device shall be taken to a temperature at least 10°C above ambient temperature to initiate this test and enable detection of dew point in devices which may already be at saturation. The rate of change of temperature for this test shall be no greater than 10°C per minute. The test voltage shall be at least equal to the rated breakdown voltage of the device since it is necessary to apply sufficient voltage to achieve ionization.
4. SUMMARY. The following details shall be specified on the applicable procurement document:
 - (a) Test temperature, high (see 3) and low if other than -65°C (see 1).
 - (b) Test voltage and current (see 3).
 - (c) Test parameter (see 1 and 3).

A dew point of -65°C or lower is considered for an acceptable device and instabilities of the measured parameter above that point would be cause for rejection of the device. Problems with these criteria are associated with the correct measurement of the dew point. The dew point is related to the amount of moisture in the residual atmosphere of the internal package volume. Upon cooling of the device it is possible that adsorption on the package walls will effectively lower the measured dew point. Furthermore, desorption from unbaked components or from the epoxies used in hybrid circuits will appreciably increase the moisture content, even for devices sealed in dry N_2 , and the measured dew point will invariably be above the -65°C value.

Another comment is that the dew point of -65°C corresponds to a moisture content of approximately 5 ppm by volume. Direct measurement of package ambients of this order by mass spectrometer techniques, described below, is difficult and, furthermore, recommendations have been made for acceptable moisture contents of higher values (up to 6000 ppm corresponding to a dew point of 0°C). Method 1018 of MIL-STD-883B describes measurement procedures and Method 5008 specifies a maximum water vapor content for hybrids of 6000 ppm at 100°C .

C. Analysis of Gases in Microcircuit Packages

A dynamic method for measuring the gas ambient within microcircuit packages has been developed by P. W. Thomas of the Rome Air Development Center.^{5,6} The method involves the measurement of the gases evolving from a microcircuit package after puncturing using a quadrupole mass spectrometer and a computer analysis system for processing the time-dependent data to give the relative percent by volume of each species of gas present.

The microcircuit packages to be analyzed are maintained under vacuum in a test chamber for 24 hours or longer at a temperature of 125°C to remove contamination on the walls of the chamber and the packages in addition to establishing a background level. After opening the package the gases leave the chamber at different rates (a typical result is shown in Figure 5-1) so that an integration over time is necessary to obtain the relative amount of each species present and a correction must be made for the sensitivity factor of each gas.

The moisture content of microcircuit packages is one of the most difficult measurements to make accurately in the gas analysis system. The

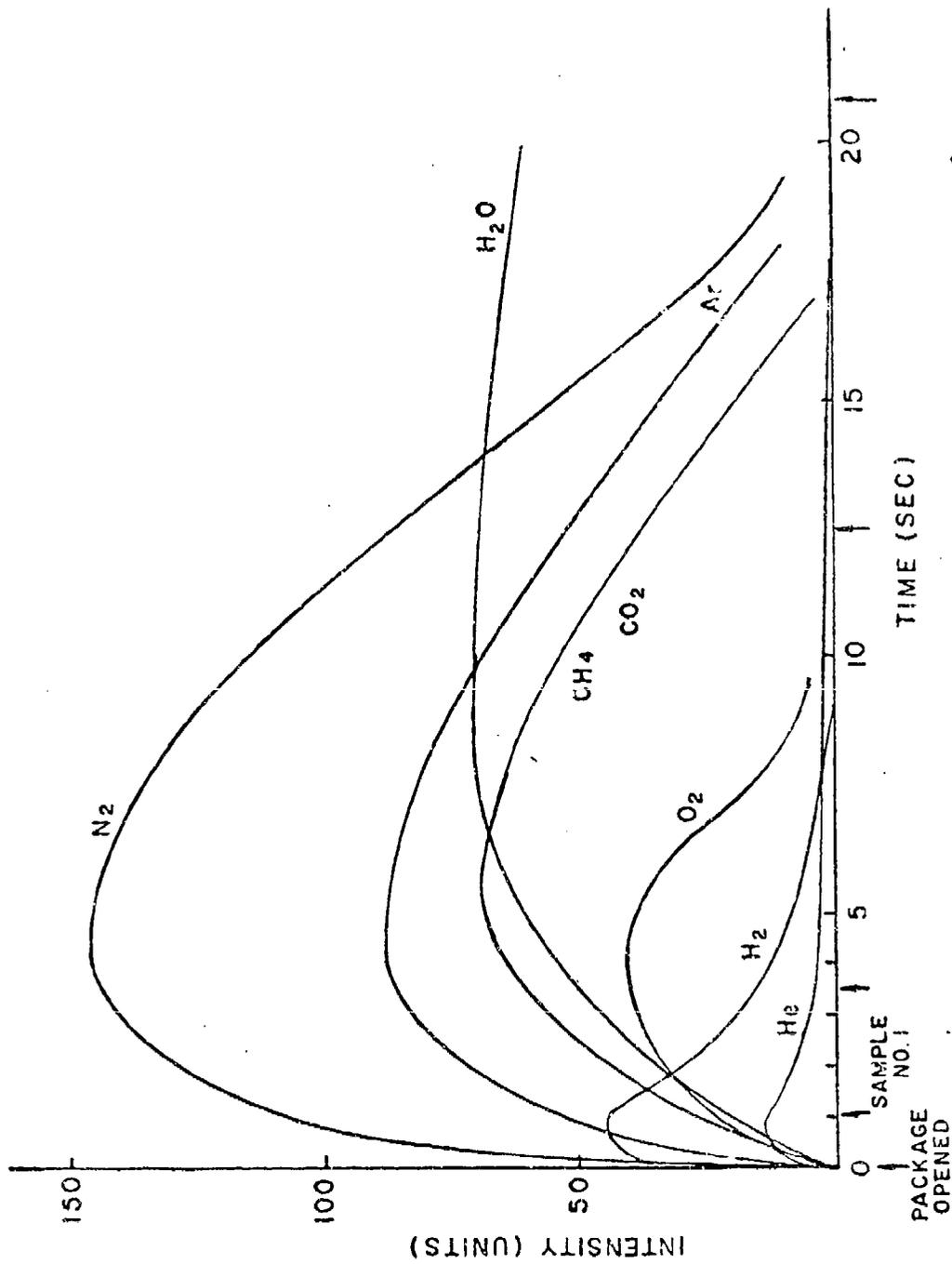


Figure 5-1. Evolution of Gases from Microcircuit Package (after Thomas⁶).

test chamber, the connecting tabulation to the quadrupole, and the quadrupole itself need to be maintained at an elevated temperature ($\approx 100^{\circ}\text{C}$) to remove moisture from the walls and to prevent adsorption during the measurement. Calibration of the system using gases of known moisture contents is an important part of the measuring technique. According to Thomas, moisture leaked into the system from the standard gas mixture at atmospheric pressure will reach a measured equilibrium value if a dynamic flow of the moisture mixture is maintained. However the accuracy of the specified moisture content of standard gas mixtures over large ranges (10 to 1000 ppm) is questionable. Therefore some secondary measurement of moisture content needs to be made or a way of generating gas mixtures "on-line" for calibration purposes needs to be developed.

The interpretation of the data obtained by mass spectrometric analysis of the gases evolved after puncturing a package is complex for at least two reasons. The composition of gases in microcircuit packages immediately after sealing can be much different when the gas analysis measurements are made. This is particularly true when unbaked parts are used. Thomas notes that for packages sealed in dry nitrogen (5 ppm moisture) with unbaked parts, the package ambient changes to 5000 ppm in a matter of seconds and to greater than 15,000 ppm in several days. The second reason is that at the elevated temperature during bakeout or measurement there are several possible reactions that may occur within the package. These include outgassing from the materials within the package (glass sealing frit, ceramics, gold platings, and die attach epoxies); reaction of oxygen with kovar, the sealing frit and ceramics; and the reactions of moisture with aluminum and other metals present in the package.

The possible presence of leaks in package seals below the minimum detectable leak, as discussed earlier, or the use of polymer seals will give gas analysis results which will depend primarily on the environmental exposure of the packages and not on the initial atmosphere sealed into the package. This is an important point to consider for long-term storage of missile systems. That is, the package ambient for non-hermetic sealed devices is bound to be directly related to the environments that the micro-electronic component will experience in the missile system in storage. The wide range of these storage environments is discussed in detail in Chapter III.

D. Consequences of Moisture in Microcircuit Packages

Some consequences of moisture in microcircuit packages are mentioned in this section. Whether the effects will be significant for semiconductor devices in stored missile systems depends upon a number of factors which need to be evaluated in each case. Certainly storage in a humid environment accompanied by failure of package seals due to repeated temperature cycling and exposed junctions will result in degradation, and possibly catastrophic failure of the devices. The moisture-related phenomena discussed below include: (1) corrosion of aluminum thin films; (2) reactions with phosphorous-doped passivation glasses; (3) ionic drift on the surface of oxidized silicon; and (4) the phenomenon of migrated-gold resistive shorts.

1. Corrosion

Corrosion is probably one of the most important wear-out mechanisms in semiconductor devices and it is expected to account for a larger percentage of storage failures in moist environments. Philofsky and Hall⁷ review the corrosion of aluminum and other limitations of aluminum thin films in semiconductor devices. In an environment of high relative humidity they point out that there are essentially three types of corrosion cells of interest: (1) galvanic, or dissimilar metal cells, (2) concentration cells, and (3) electrolytic cells. A review of corrosion in integrated circuits by Kolesar⁸ discusses these three cells in some detail. In galvanic corrosion, two dissimilar metals in an electrolyte have a potential difference between them because of the difference in electrochemical potentials. In a concentration cell, the electrodes are of the same material but there is a gradient in concentration. In the third, the electrolytic cell, the electrodes are similar and there is an applied potential between them.

Aluminum forms a passivating oxide which inhibits galvanic corrosion and corrosion in a concentration cell unless the native oxide is altered by the electrolyte or impurities within it. Aluminum does form an electrolytic cell in humid atmospheres where the electrolyte is the absorbed water on the surface connecting the two electrodes. The effect of the phosphorous content in passivation glass on the corrosion rate of aluminum under different temperatures, relative humidity and bias conditions is discussed in the following section.

2. Phosphosilicate Glasses

Paulson and Kirk⁹ studied the effects of phosphorous-doped CVD passivation glass on the electrolytic corrosion of aluminum. There are a number of variables that cause corrosion failures of microelectronic devices.^{7,8} Paulson and Kirk show that the corrosion rate of aluminum metallization under bias is increased by a factor of 25 when the phosphorous concentration of the passivation glass changes from 2 to 10 wt. percent and that increasing the relative humidity at 35°C from 85 to 100% caused an increase of corrosion rate by two orders of magnitude.

Passivation glass with no phosphorous-doping often exhibits cracking either during the deposition process or during later temperature cycling. Exposure of underlying aluminum films as a result of the cracking can also lead to corrosion under the right conditions of temperature, relative humidity and bias. Cracks in the passivation glass over nichrome resistors appear to act as sites for the condensation of water and where failures are initiated.⁵ It is interesting that unpassivated nichrome resistors are often less sensitive to moisture. The addition of phosphorous eliminates the tendency of the glass to crack but it introduces other problems. In addition to the electrolytic corrosion effects mentioned above it has been observed¹⁰ that, above certain critical concentration levels, the phosphorous can be dissolved out into water films. The formation of phosphoric acid could have severe detrimental effects on other parts of the microcircuit.

A review of various properties of the phosphosilicate glasses have been presented by Schlacter, et al.¹¹

3. Ionic Drift

The presence of adsorbed water and ionic contamination on the surface of semiconductor devices is known to influence their stability, performance and reliability. Attala, Bray and Lindner,¹² in 1959, reported that surface ions on thermally oxidized, diffused silicon junctions caused slow changes in the reverse current. The explanation was that the drift of ions on the surface of the oxide in the fringing field of the junctions caused the formation of channels, or inversion layers, on the lightly doped region of the junction. Shockley, et al.¹³ analyzed the surface ion motion with the assumption that the total density of surface ions is

independent of time and distance. Their result which was confirmed by experimental observations was that the surface-ion induced inversion layers formed in proportion to the square root of time. Subsequently Schlegel, et al. ¹⁴ considered the possibility that the total ion density was not constant but depended upon the electrostatic potential on the surface. In this case, the time-dependent behavior is related to the ionic mobility.

Factors which are important to the build-up of surface-ion induced inversion layers are the moisture levels in the package, the package temperature, contamination on the surface, the oxide thickness and the applied voltage across the junction. Evaporation and condensation of moisture during excursions of temperature may have an effect on the distribution of contaminants on the surface. Therefore, even under storage conditions, the surface ions may accumulate in critical regions near a junction and thus form an inversion layer. During the long storage period it is possible that ionic contaminants from other parts of the microcircuit will migrate to junction regions of active semiconductor devices and also cause a build-up of ion-induced inversion layers. The essential factor in all these instances, however, is the moisture level in the package.

4. Migrated-Gold Resistive Shorts

Shumka and Piety¹⁵ have described a moisture-induced gold migration failure mechanism that results in a resistive short between adjacent gold metallization stripes in microcircuits. Three elements are necessary for the migrated gold resistive short (MGRS) failure mechanism to occur; (1) the presence of reactive chemicals, such as the halogens, particularly iodine and chlorine; (2) a continuous layer of adsorbed water between neighboring gold stripes; and (3) an applied bias. Moisture levels in the microcircuit package of greater than 1.5% at 25°C are required but this value is expected to be temperature dependent. It would seem that since an applied bias is necessary this would not be a significant failure mechanism in storage. However, the effect often takes place quite rapidly and operation during the initial screening tests or during subsequent operational checkout of a missile system should be sufficient to initiate the gold migration mechanism, if the moisture levels are above the threshold value. Trace amounts of the halogens are usually present in both good and MGRS devices and are difficult to remove from the microcircuit by normal cleaning techniques.

E. Summary

1. MIL-STD-883A establishes uniform methods and procedures for testing microelectronic devices, including basic environmental tests and physical and electrical tests.
2. Method 1014.1 has as its purpose the determination of the effectiveness (or the hermeticity) of the seal of microelectronic devices with designed internal cavities. The fine leak rate test specifies a maximum allowable leak rate that may be too high when considering the exchange of gases between the package ambient and the external environment, especially in long term storage situations.
3. The probability of failure of package seals after the devices are incorporated in a missile system is expected to be high when thermal excursions result in significant mechanical stresses on the seal.
4. Method 1013 is a test designed to detect the moisture present inside microelectronic packages by measuring device leakage currents as the temperature is lowered below the dew point. The acceptable level specified of -65°C corresponds to a moisture content of approximately 5 ppm by volume. Most microcircuits probably have moisture contents much greater than this value but the test does not give a positive indication unless moisture-sensitive devices are incorporated in the microcircuit.
5. Mass spectrometer methods for measuring the gas ambient within microcircuit packages have been developed. The methods and procedures used have to be taken into consideration when comparisons are made between measuring systems. The use of unbaked parts and the outgassing of the various materials within the package lead to difficulties in interpreting the data obtained in terms of the atmosphere sealed into the package.
6. The possible presence of leaks in the package or the use of polymer seals will give gas analysis results which will depend primarily on the environmental exposure of the packages and not on the initial atmosphere sealed into the package.

7. It would be desirable to incorporate gas analysis as the first step in the failure analysis of microelectronic components returned from the field for missile systems which have parts surveillance programs.

8. The consequences of moisture in microcircuit packages are a number of failure mechanisms including corrosion, ionic drift, reactions with phosphorus-doped passivation glasses and the formation of migrated gold resistive shorts. Considering the long time, the temperature variations and other environmental conditions of missile systems in storage, all of these moisture-related failure mechanisms will be important to some degree.

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VI. OXIDES

Silicon dioxide, the oxide most extensively used in semiconductor device technology, is used for masking and surface passivation, as the oxide in MOS devices, as isolation layers in multi-level interconnections, and as a glassivation over the entire integrated circuit. Other dielectric films, e.g., Al_2O_3 and Si_3N_4 , are often combined with SiO_2 in multiple layer structures either to provide diffusion barriers for mobile alkali ions or to make unique memory devices, such as the MNOS devices where charge is stored at the nitride-oxide interface. Reliability problems with these oxides on bipolar or MOS devices relate to shorts in the oxide, surface migration of ions, mobile ions in the gate oxide of MOS devices, oxide breakdown, cracking of the glassivation or nitride layers and dissolution of the phosphosilicate glass. Many of these phenomena could cause failure or degradation of MOS devices in storage.

Metal diffusion through cracks, pinholes or discontinuities (as at the boundary of devitrified areas) in SiO_2 can cause shorts between metallization layers or between the metallization and silicon.

The migration of ions on oxides which is particularly enhanced by a water layer can result in excess leakage currents or induced inversion layers in the silicon. The formation of inversion layers depends upon the thickness of the oxide and the bulk doping of the silicon substrate. Some types of digital bipolar integrated circuits are based on dopant densities of 10^{16} atoms/cm³ or higher and are less surface sensitive than devices of lower dopant densities. The relationship between substrate dopant density and the effective oxide charge density necessary to invert an n-type silicon surface is shown in Figure 6-1 (from Schnable and Keen¹).

Mobile alkali ions in the gate oxide of MOS devices can migrate towards the silicon-silicon dioxide interface and cause changes in the threshold voltage. Temperature-bias stress tests are capable of determining the presence of these mobile ions, particularly at the wafer level with special test structures. In 1974 Bruce Deal² reviewed the understanding of charges in thermally oxidized silicon. Of the four types of charges—fixed surface state charges, mobile impurity ions, fast surface states, and traps ionized by radiation—the mobile ions are perhaps most important for long time effects. Comparison of drift rates for various alkali ions under

temperature-bias stresses is shown in Figure 6-2 (from Deal). Thus unless strict in-process controls are imposed, most gate oxides will include a concentration of mobile ions sufficient to cause significant threshold voltage shifts. Under temperature and bias the changes will occur rapidly, as indicated in the figure, but even without an applied bias one might expect some migration to occur when there is a nonuniform distribution of fixed and mobile ions in the oxide. Questions in regard to the use of the stabilization bake screen (125°C, 24 hours, no bias) for screening MOS devices for excessive mobile ions are being considered by Rockwell under a contract with MIRADCOM.³

The dielectric strength of amorphous SiO₂ is of the order of 10⁷V/cm. For a typical 1000 Å gate oxide an applied potential of 20 V will give a field of 2x10⁶V/cm, one-fifth of the dielectric strength. Since oxide integrity probably only approaches the ideal, due to variations in the thickness, localized defects, etc., breakdown voltages will generally show a range of values. MOS devices are therefore susceptible to damage due to static charging. In addition, voltage transients may exceed the breakdown voltage. The periodic testing of certain missile systems may introduce such transients from the ground test equipment. Most commercially available MOS devices include some form of input protection circuit but they are not always 100% effective.

The over-layer of chemically-vapor-deposited (CVD) SiO₂ glass doped with phosphorous provides mechanical protection of integrated circuits, improves electrical stability and minimizes the effects of loose particles. Insufficient doping, however, results in cracking due to residual stresses in the glass, cracking due to thermal cycling and cracking at contact windows. Increasing the phosphorous doping provides a composition that will getter mobile alkali ions. However, the phosphorous-doped glasses also react with water vapor to form phosphoric acid which can have a detrimental effect on exposed metallization layers. Paulson and Kirk of Motorola⁴ showed that the corrosion rate of aluminum metallization under bias increased by a factor of 25 when the phosphorous concentration changed from 2 to 10 wt percent. As discussed in the previous chapter, this is one of several situations which emphasize the importance of limiting the water vapor in microelectronic packages during storage.

The following chart summarizes the failure modes and mechanisms which are likely to occur in C/MOS devices that are part of electronic systems of missiles in storage.

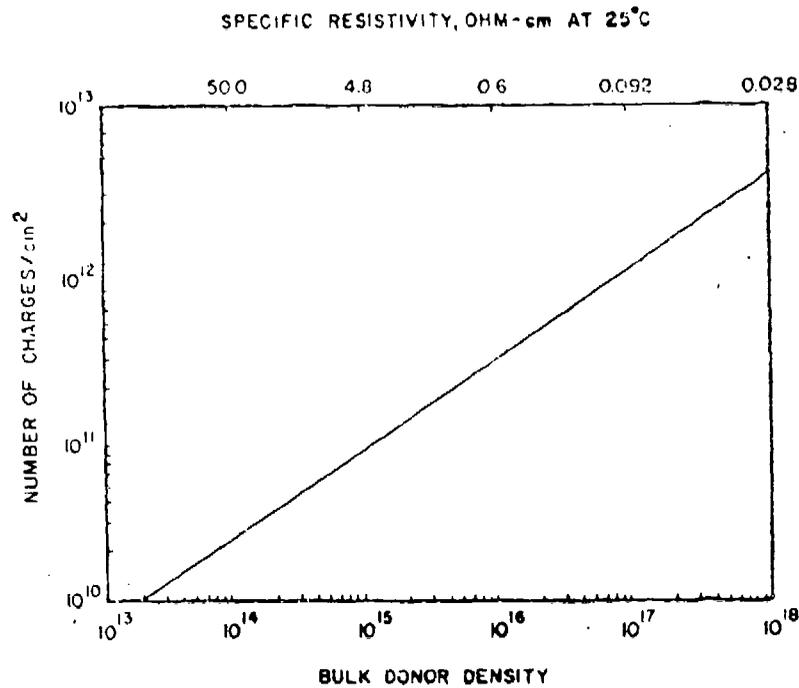


Figure 6-1. Plot of Resistivity Versus Number of Charges Needed to Invert n-type Silicon.

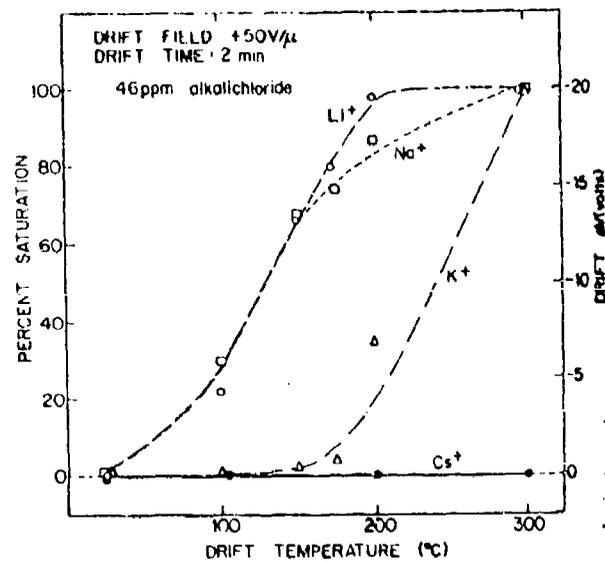


Figure 6-2. Comparison of Drift Rates for Various Alkali Ions Through 0.2 μ m Thermal Silicon Dioxide Over the Temperature Range 25-300°C.

FAILURE MODES AND MECHANISMS IN C/MOS

OPEN CIRCUITS DUE TO BOND BREAKAGE, POOR BONDS FROM CONTAMINATION LAYERS, OPEN INTERCONNECTS AS A RESULT OF SCRATCHES, THIN METALLIZATION, POOR COVERAGE AT OXIDE STEPS—BOTH WOULD LEAD TO HIGH CURRENT DENSITIES AND BURN-OUT, AND CORROSION IN THESE REGIONS CAUSING OPENS OVER A LONG TIME.

SHORT CIRCUITS DUE TO LOOSE METALLIC PARTICLES BRIDGING CONDUCTIVE AREAS, MISALIGNMENT OF METALLIZATION OR SMEARED METALLIZATION CAUSING REDUCED SPACINGS OR SHORTS—DIRECTLY OR IN COMBINATION WITH CONTAMINANT FILMS, AND SHORTS DUE TO PINHOLES IN THE OXIDE—USUALLY PRESENT IN THE THIN GATE OXIDE.

OXIDE BREAKDOWN DUE TO BUILDUP OF STATIC CHARGE AND THEN DISCHARGE THROUGH THE OXIDE WHEN ELECTRIC FIELD STRENGTH OF OXIDE IS EXCEEDED. MOST STATIC DISCHARGES OCCUR DURING HANDLING, TESTING OR REPAIR.

THRESHOLD SHIFT DUE TO DRIFT OF MOBILE IONS—USUALLY POSITIVE SODIUM IONS—IN THE OXIDE.

LEAKAGE CURRENTS, FOR EXAMPLE BETWEEN SOURCE AND DRAIN, DUE TO SURFACE CONTAMINATION WITH OR WITHOUT A CONDENSED WATER FILM.

FAILURES IN INPUT PROTECTIVE NETWORKS OR AS A RESULT OF TRANSIENT VOLTAGES THROUGH PATHS OTHER THAN INPUTS.

Reliability data on TI's 4096-bit MOS RAM⁵ obtained during a visit to Texas Instruments in Houston, Texas in 1975 reflected the experience with MOS failure mechanisms. While historically the major MOS failure causes included device instabilities caused by ionic contamination, gate-substrate shorts due to oxide defects, open metallization caused by poor oxide-step coverage, and various forms of leakage, the predominant failure cause of the 4K-RAM was associated with structural faults in the oxide of the capacitor or the transistor gate oxide due to particulate or other contamination occurring before, during or after growth of the thin oxide. Several changes in the process and screening technology were made subsequently to eliminate such failures. These included:

- 1) Instituting stricter specifications on the purity of the chemical materials used in the manufacturing process.
- 2) Minimizing slice contamination, both before and after growth of the gate oxide with improvements in rinse equipment and slice transportation.
- 3) Installing monitors of oxide integrity at critical process points.
- 4) Initiating voltage-stress screens.

One can conclude that for large scale integration of MOS devices (MOS/LSI) there is a potential for improving reliability by the implementation of in-process controls and monitors. The Rockwell program is considering such approaches in addition to evaluating the effectiveness of MIL-STD-883A screens for MOS devices.

In a paper⁶ presented at the Electron Devices meeting in December 1976 Barrett and Smith of Intel Corporation discussed the reliability of the 16K-bit, N-channel, MOS RAMs in terms of the primary failure modes. In contrast to the 4K, one-transistor, RAM, as used by TI for example, the 16K RAM uses a double layer polysilicon select gate which overlaps the polysilicon capacitor plate forming a CCD cell. The overall size of the memory cell is reduced but greater reliance on the oxide integrity is required.

The data in Table A, from Barrett and Smith, represent failure analyses of a random sample of 4K RAM field and laboratory failures. Package failures such as broken leads, lead corrosion, etc. were eliminated from this listing. Similar trends are observed for 1K and 16K RAMs. The significant

Table A

Die Related Failures of 4K RAMs

<u>Failure Mode</u>	<u>Number</u>	<u>% of Total</u>
Surface Defects	22	17.3
Oxide Defects	83	66.0
Metallization Defect	1	0.8
Masking Defects	7	5.5
Assembly Defects	10	8.0
Degraded Input	3	2.4

feature of these data is that the primary MOS RAM failure mode is oxide breakdown. In their discussion the authors conclude that while acceptable reliability can in theory be achieved by improving oxide quality alone, in practice it must be attained by screening out defective oxides. They then consider the best screen for oxide defects. In comparison with other failure modes which have activation energies in the range of 1 eV, and are therefore greatly accelerated by high temperature stresses, the oxide defects failure mode has an activation energy of only 0.3 eV so that a high temperature burn-in is not particularly useful. Rather, the most effective screen is one which employs an over-voltage stress.

In summary, oxides are expected to be an integral part of the electronic components used in missile systems and the increased use of such devices as MOS RAMs, C/MOS and CCDs will mean that the potential for failures in storage will correspondingly increase. A continuing evaluation of MOS technology and the examination of failures for identifying the predominant failure mechanisms should therefore be of vital concern to the Army.

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VII. POLYMERS

A. Barrier Problems

1. Hermeticity and Diffusion

The prime concern in use of plastics for encapsulation of electronic devices or as sealants is water resistance. Water is the most damaging of all outside agents which can cause failure in microcircuits. Clough and Collins¹ describe water attacks on plastic encapsulated circuits in four stages:

- 1) absorption of water by the polymer,
- 2) transport of ionic species through the polymer,
- 3) formation of conducting paths between electrodes, and
- 4) corrosion of the embedded materials.

Chapman and White² have pointed out that water can penetrate plastic by two mechanisms. Both mechanisms involve diffusion, one through the plastic and the other through the air trapped in the plastic. We consider a third mechanism for introducing water, namely by decomposition of encapsulated materials.

R. K. Traeger³ studied the hermeticity of polymeric lid sealants. Moisture permeability of polymeric packages was found to be around 3.7×10^{-5} cc(STP)/cm-sec-atm. With this permeability the interior of a package will reach 50% of exterior humidity in 6-10 hours. Figure 1 illustrates time scales for moisture to penetrate various sealant materials. The data listed in Table A illustrate differences between polymers. Epoxies are good moisture barriers compared to other organic materials, but the highly fluorinated polymers such as Teflon[®] could be an order of magnitude better. Unfortunately, Teflon[®] and low permeability epoxy are not usable as lid sealant polymers.

Jones, et al.⁴ have considered permeation of moisture through organic seals using the usual modification of Newton's and Fourier's equations for thermal conductivity used in permeability and drying studies. If the permeability P is constant, the time, t , for the partial pressure of water to increase from p_1 to p_2 is

$$t = \frac{1}{P} \left(\frac{LV}{A} \right) \left(\frac{273}{T \times 1 \text{ atm}} \right) \ln \frac{p_0 - p_1}{p_0 - p_2}$$

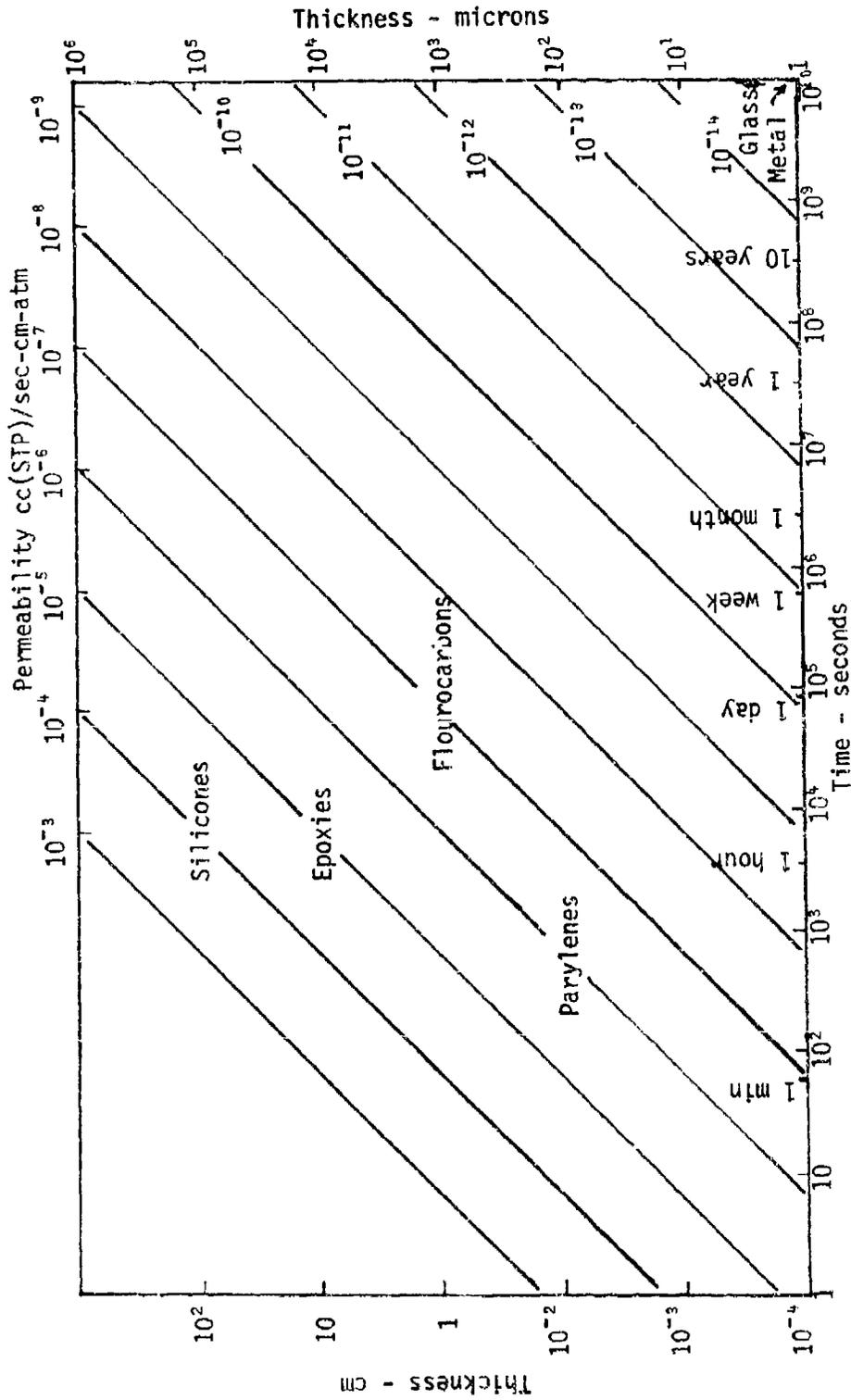


Figure 7-1. Effectiveness of Sealant Materials (after Trager³). Times to reach 50 percent of exterior pressure of penetrants are given for permeabilities from 10^{-3} to 10^{-14} cc(STP)/sec-cm-atm and for barrier thicknesses from 1 micron to 100 cm, from Equation 1.

Table A
Moisture Permeability of Various Polymers

<u>Material</u>	<u>Temperature</u>	<u>Permeability cc(STP)/sec-cm-atm·10⁶</u>	<u>Reference</u>
Silicone	38 ⁰ C	8.4	7
Silicone RTY	25 ⁰ C	240	3
		20-100	3
Silicone junction coating	25 ⁰ C	36-190	3
Polyurethane	25 ⁰ C	6-16	3
	25 ⁰ C	1.7-5.2	3
	25 ⁰ C	53	3
Epoxy, anhydride	25 ⁰ C	4.8	3
Epoxy, amine	25 ⁰ C	3.6	3
Epoxy, amine	25 ⁰ C	3.2-7.5	3
Epoxy, amine	25 ⁰ C	2.9-3.4	3
Epoxy, commercial	37 ⁰ C	0.64	7
	50 ⁰ C	0.96	7
	60 ⁰ C	1.2	7
Epoxy, commercial	38 ⁰ C	0.26	7
	60 ⁰ C	0.62	7
Epoxy, experimental	38 ⁰ C	0.16	7
	50 ⁰ C	0.26	7
	60 ⁰ C	0.38	7
Phenolic	60 ⁰ C	0.11	7
Poly (diallyl phthalate)	69 ⁰ C	0.88	7
Poly (ethylene terephthalate)	39 ⁰ C	1.7	3
Polycarbonate	25 ⁰ C	20	3
	25 ⁰ C	0.67	3
Polytetrafluoroethylene	40 ⁰ C	0.28	3
	28 ⁰ C	0.064	7
	50 ⁰ C	0.11	7
	60 ⁰ C	0.10-0.22	7
Polychlorotrifluoroethylene	38 ⁰ C	0.028	7
Polychlorotrifluoroethylene	30 ⁰ C	0.11	7
Polyethylene copolymer	45 ⁰ C	0.18	7
Polyvinylfluoride	25 ⁰ C	1.4	3
	38 ⁰ C	0.26	7
	50 ⁰ C	0.66	7
	60 ⁰ C	1.4	7

t - is time to reach p_2
V - is the free volume of the container
L - is the diffusion path length of seal
P - is the permeability of sealant
A - is the area of the seal exposed to the permeant

T - is the absolute temperature
 p_0 - is the external water vapor pressure
 p_1 - is the initial, internal water vapor pressure
 p_2 - is the final internal water vapor pressure

Traeger³ reports that moisture will permeate through silicones in minutes, epoxies in hours, and fluorinated polymers in days (Figure 7-2). Bubble testing (for gross leaks), helium leak tests, and device operation have been used to measure hermeticity of electronics packages. According to Traeger packages sealed with adhesives can pass both fine and gross leak tests. However, gases, including water vapor, permeate through organics and the organic seals do not provide long-term resistance to moisture. Perkins and Licari⁵ recently measured the moisture permeation as shown in Figure 7-3 for ceramic hybrid microcircuit packages sealed with epoxy resins. Their best seals have permeabilities in the 1.2×10^{-7} to 4.2×10^{-7} cc(STP)/cm-sec-atm range. This means that packages reach one half of ambient humidity in about 90 to 100 days.

Hamilton⁶ discussed water vapor permeability of polyethylene and other plastic materials and described apparatus for measurement of permeabilities. Low density polyethylenes have permeabilities of the order of 10^{-5} cc/cm-sec-atm at 25°C. High density polyethylenes have permeabilities from 1/3 to 1/6 that of low density polyethylene. The permeabilities of polypropylene are between those of high and low density polyethylenes. Figure 7-4 shows a comparison of nonolefinic polymers with a typical low density polyethylene.

Hadge and colleagues⁷ described the apparatus developed in the laboratories of the Allied Chemical Corporation for testing vapor permeability

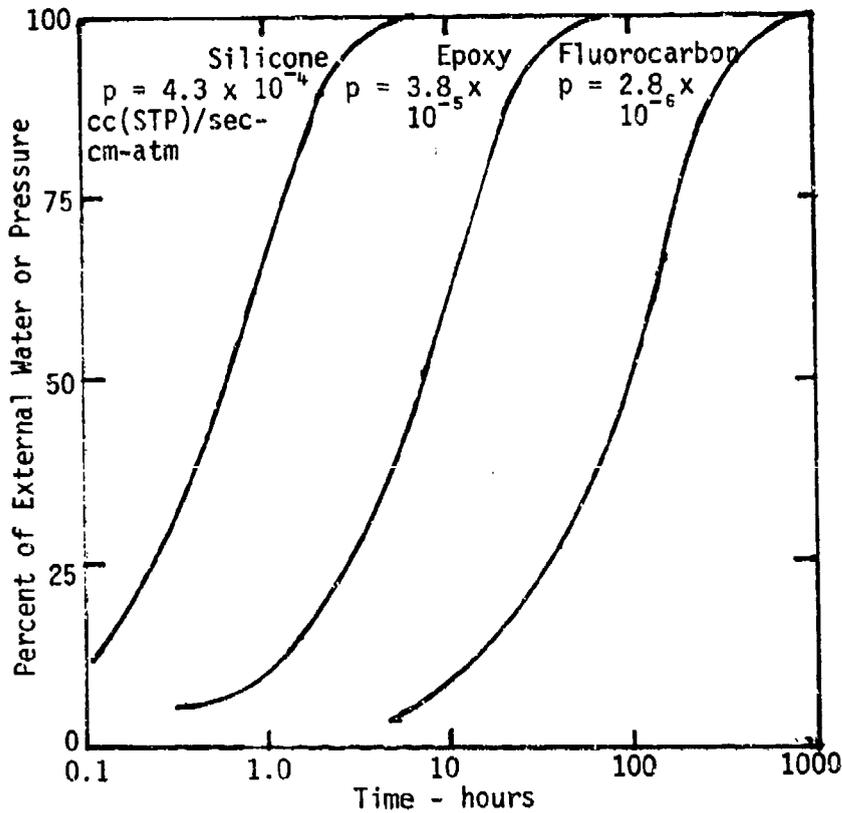


Figure 7-2. Rate of Moisture Permeation into a Hybrid Microcircuit Package (after Traeger³). Times to reach various fractions of external relative humidity are given for a package with seal aveg. 0.175cm^2 , seal length (thickness) 0.15cm , and internal volume 1.8cm^3 , from Equation 1.

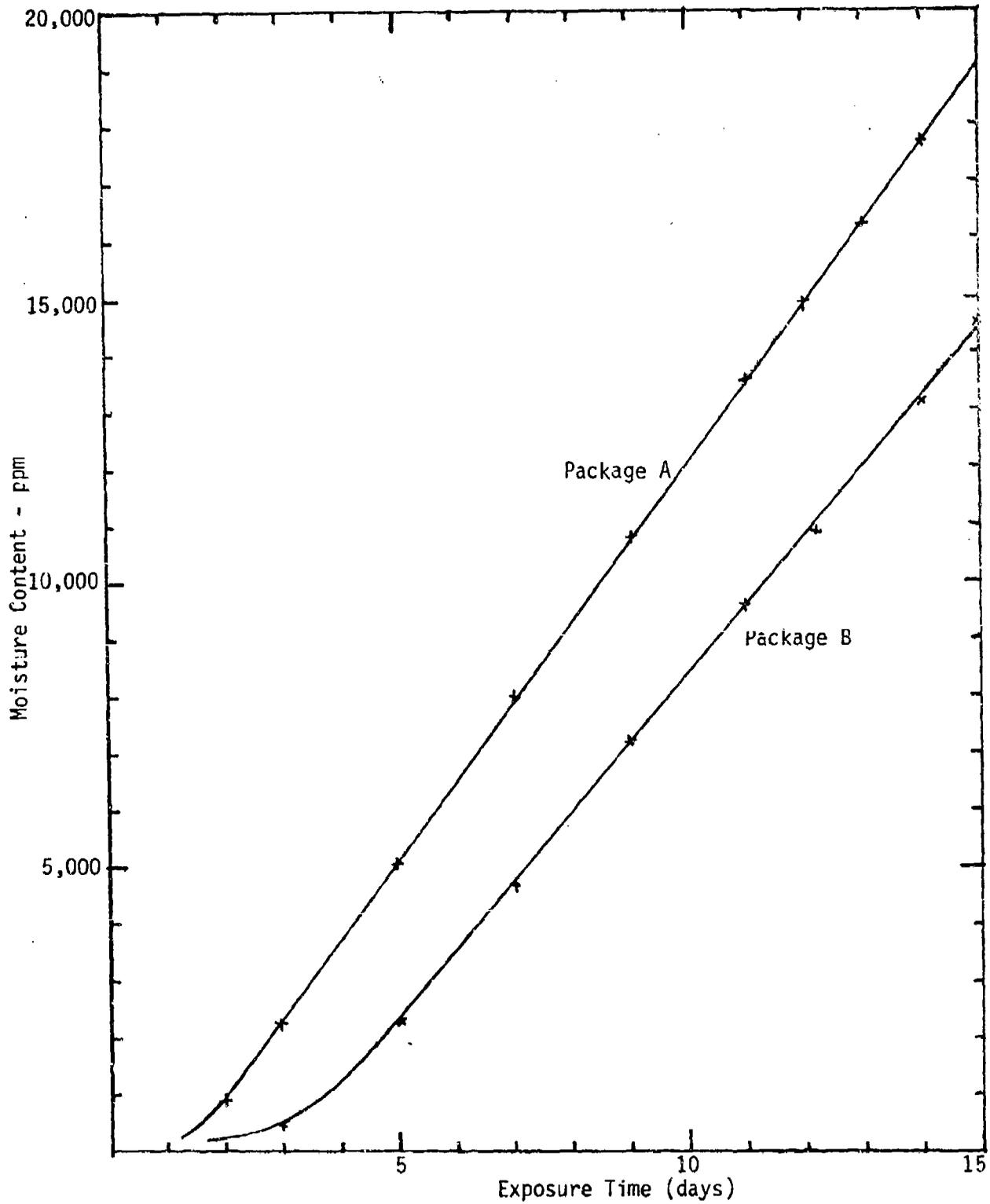


Figure 7-3. Moisture Permeation Into Adhesive-Sealed Ceramic Exposed to 60°C/98% RH. Each package contained moisture sensor. From Reference 5.

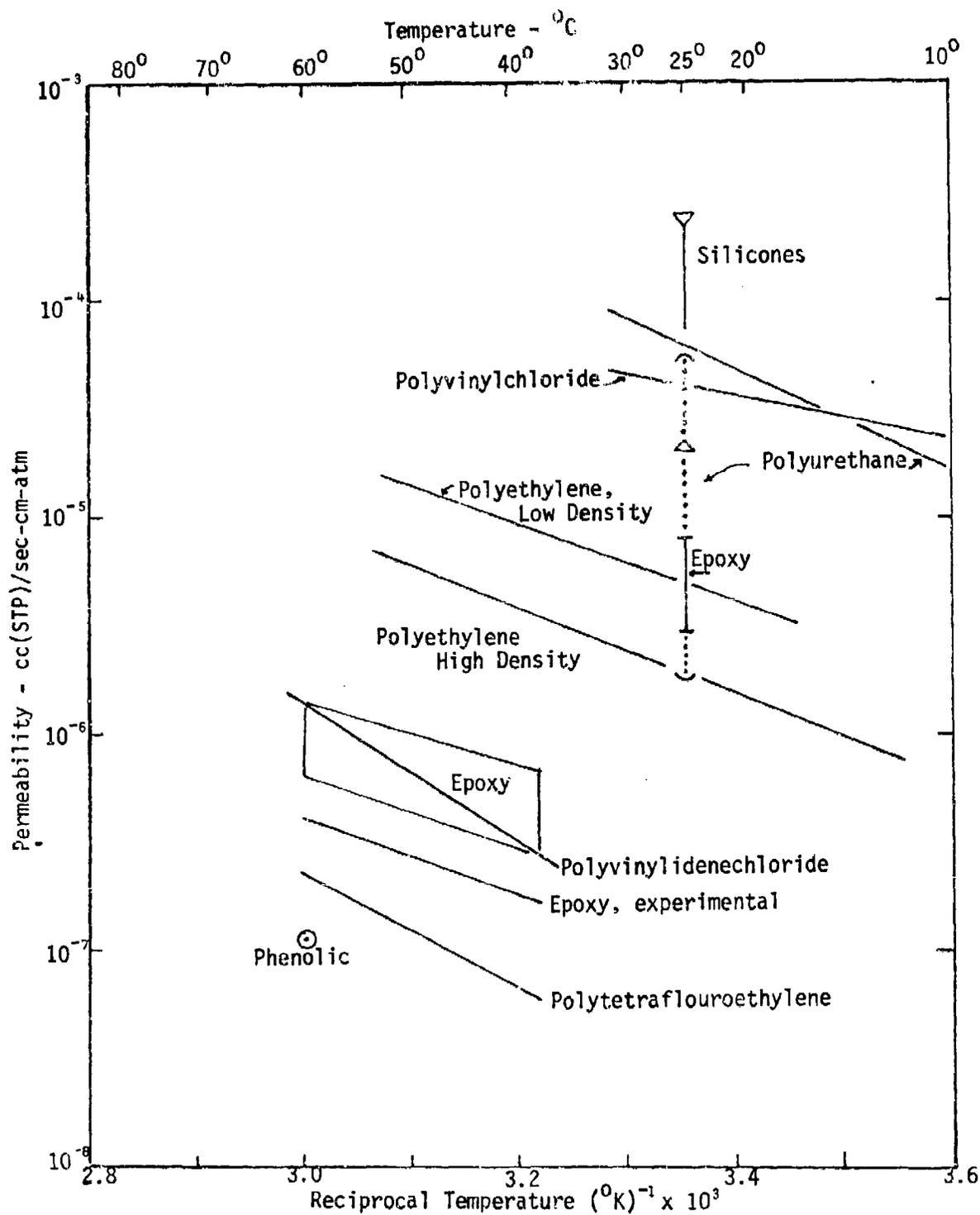


Figure 7-4. A Comparison of Permeabilities and Their Dependence on Temperature for Several Polymers (data from the literature^{3,7}).

for molded plastics in the thickness range of 0.16 to 0.32 cm. Table A includes their water vapor permeation results for various polymers.

In 1974, Thomas and Meyer⁸ noted that inside a semiconductor package water is a "killer." Water induces nichrome resistor failures, molybdenum and gold dendrite growth, aluminum hydroxide formation, and second order effects such as undesirable intermetallic growth and surface inversion. At present, package atmosphere analyses are made in a number of laboratories. Helium and Freon^R have been found inside "hermetically" sealed packages. There doesn't appear to be much if any correlation between fine helium or krypton leak rates and moisture levels.

Gas analyses of parts from the Apollo program which were sealed seven to nine years ago showed that although measured leak rates were in the 10^{-8} cc/sec-atm range, little or no oxygen, argon or water exchange was observed. This indicates that a true leak rate was lower than 10^{-8} (actually less than 10^{-9} cc/sec-atm) and that the parts could have been stored indefinitely without ambient degradation.

A leak rate of 10^{-8} cc(STP)/sec-atm is equivalent to a permeability of 1.6×10^{-7} cc(STP)/cm-sec-atm in a barrier 1.0 cm thick, if we take water as the penetrant at 37°C and 100% RH ($p_{\text{vap}} = 47.07$ mm Hg). In case the demand for storage life is 10 years (3.16×10^8 sec) this permeability would permit passage of water equivalent to ~ 82,000 molecular layers. Such accumulation of water is intolerable. We must conclude that a leak rate of 10^{-8} cc(STP)/sec-atm is much too high to use as a criterion for hermeticity.

We expect that one monolayer of labile water probably is necessary to permit surface migration of ions in electrochemical oxidation-reduction reactions leading to migration of metals and resultant failures in microcircuits and devices. Therefore, we propose as a realistic and prudent criterion for hermeticity a leak rate which corresponds to the admission of one monolayer of water vapor or gas in ten years. This leak rate is about 1.2×10^{-13} cc(STP)/sec-atm per square centimeter of adsorbing surface. We recognize that to dry any material, component, or microcircuit in the sense of removing the last traces (monolayer) of water is quite impossible and completely impractical were it possible. We emphasize that we must remove sorbed water which is mobile. Sorbed water can enter into reactions as water, and can facilitate ion migration. Further, we must prevent introduction of water at concentra-

tions at which water can be labile. This means that we must dry components, materials, microcircuits, etc. carefully; seal components under dry conditions, and seek leak rates less than about 1.2×10^{-13} cc(STP)/sec-atm in hermetic seals.

A barrier 10μ thick which would pass one monolayer of water (at 37°C and 100% RH) in ten years would have a permeability of 2×10^{-15} cc(STP)/cm-sec-atm. Water permeabilities in polymers are at least 10^8 times too high. See Figure 7-1.

Further studies of hermetically sealed electronic parts were reported by Thomas⁹ at the 26th Annual Electronic Components Conference in 1976. Moisture adsorbed on the surfaces of the package parts was the primary source of package contaminations. Thomas claims that moisture can be removed by extended bake-out procedures. Organic materials such as epoxy die attach compounds require special processing if used in hermetically sealed packages. He says that a minimum of 48 hours drying time is necessary for organic materials when baked in a vacuum at 150°C . We doubt that organic materials can be dried in vacuum in any reasonable time. One of us tried unsuccessfully to remove toluene from polystyrene by baking in vacuo at 100°C for two weeks¹⁰ and to remove water from carbon black by baking in vacuo at 500°C for ten days¹¹. The results of studies by Wiegand and Licari¹² on outgassing support this observation.

Plastic lead seals were found to be nonhermetic with respect to moisture and, therefore, unacceptable for use in high reliability applications. Experiments conducted on the plastic sealed samples⁹ showed that after less than 100 hours in 85% RH at 85°C packages contained more than 10% moisture by volume. These packages were "hermetic" with respect to the helium leak test. These studies showed the uselessness of testing plastic sealed packages using MIL-STD-883A helium fine leak test procedures. A new standard for determining the hermeticity based on moisture content is needed.

According to Thomas⁹, most microelectronic devices operate reliably with moisture contents of 1000 ppm (in the enclosed volume) or less. For high reliability parts he recommends 200 ppm as a maximum. Thomas suggests that this is a compromise between current measurement capabilities and the difficulty in achieving dry environment in a manufacturing facility.

In the past few years much effort was expended in development of new methods for measuring of moisture content inside packages. A new Al_2O_3 sensor was tested by Texas Instruments Inc., under contract from Rome Air

Development Center.¹³ The sensor detects moisture more quickly and cheaply than does mass spectrometry. The capacitance-resistance moisture sensor has a lossy dielectric of hydrated porous alumina formed by anodizing aluminum and covered with a moisture permeable gold film. Capacitance and conductance depend on partial pressure of water. Improved sensors using silica on silicon electrodes have been developed and are being tested at the National Bureau of Standards.¹⁴ This type of moisture detector can behave erratically with shifting calibration or become inoperative.

Water, free to participate in deleterious reactions, must be minimized in sealed devices and circuits. Water content in plastics has received attention out of proportion to its importance, perhaps, because water contents (solubilities) are easier to measure than the more important diffusivities or permeabilities. Modern Plastics Encyclopedia 75-76¹⁵ and other sources, for example Harper,¹⁶ give the values of water absorption of plastics as the only factor characterizing behaviour of water in plastics. No water-vapor permeabilities, diffusion coefficients, or leak rates are given. Measurements of water sorption were carried out according to the Standard ASTM D 570. Water absorption is specified as the percent weight gained by the specimen in 24 hours at 23°C.¹⁷ Figure 7.5 presents water absorption versus density.

Solubilities of water in hydrocarbon and fluorocarbon plastics should be and are low (<0.01%) except for polystyrene. Water contents of epoxies are greater, from 0.03 to 0.2%. Except for polar polymers like cellulose and proteins, all water must be considered to be labile water which can participate in deleterious reactions. Care should be exercised to maintain available water contents below levels which will provide conduction paths for electrochemical reactions on surfaces. Such levels must be determined.

2. Outgassing and Water Extraction From Organic Adhesives and Encapsulants

Products outgassed from cured organic adhesives and encapsulants in high reliability hybrid microcircuits can cause failures.

Perkins and Licari¹⁷ investigated methods for determining the outgassing of cured adhesives. Short-term tests of two hours using thermal gravimetry can identify the worst, high outgassing materials. Longer term, 1000 hour, tests are more instructive. The total amount of outgassing is

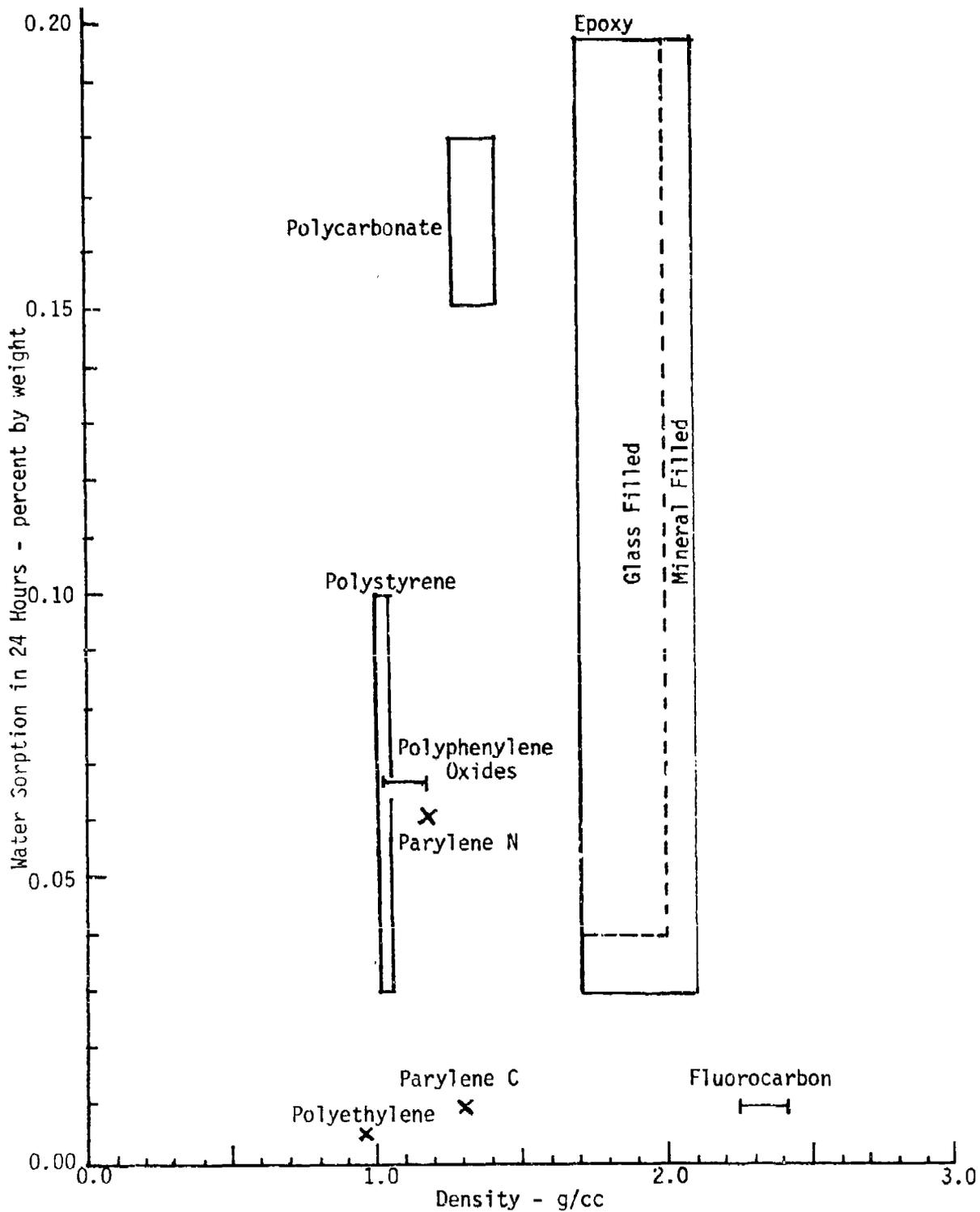


Figure 7-5. A Comparison of Water Sorption of Plastics. The percentage by weight sorbed in 24 hours is plotted versus density of polymer. No correlation of water sorption with density is expected.

important because high outgassing indicates that contamination and corrosion are likely to occur. The chemical nature of outgassed products is much more critical than is the total amount because some very active constituents can impair electronic devices when present only in trace amounts.

The results of gas chromatographic analyses by Perkins and Licari¹⁷ of several epoxy resin adhesives are shown in Table B. Only for the package containing Hysol 0151 was water reported; these results for water should be questioned. A constituent which probably is boron trifluoride was found in the packages containing three types of adhesives, Epo-Tek H61, Epo-Tek H31, and Epo-Tek M44. Water reacts with BF_3 to form boric acid and hydrofluoric acid, HF dissolved in water. Thus we may expect BF_3 content to correlate with degradation of device electrical parameters.

Wiegand and Licari¹² evaluated five epoxy resin adhesives intended for use in hybrid microcircuits. After extensive testing they concluded that the most reliable test to determine the amount of adhesive outgassing is 1000 hours at 150°C. DuPont 5504A and Ablebond 150-6 are high outgassers (4 and 7 percent by weight). Eccobond 104 and Ablebond 36-2 (0.1 and 0.5 percent, respectively) showed least outgassing (Figure 7-6)

The tests presented in Figure 7-6 were conducted in air. Wiegand and Licari recommended a nitrogen atmosphere for future 1000 hour outgassing tests, because nitrogen is the filling gas in sealed devices.

The outgassing tests described here are instructive, but less than definitive for two reasons. First, even the low outgassing materials give off huge amounts of volatile materials. Outgassing of 0.1 percent by weight corresponds to about 5×10^6 monolayers of adsorbed materials. Second, careful qualitative and quantitative analyses of gases and non-volatile decomposition products are necessary to an understanding of failure mechanisms in integrated circuit packages using organic adhesives, sealants, or encapsulants. These analyses should be sensitive to about two parts in 10^8 , corresponding to one monolayer of adsorbed molecules.

Chlorine and other halides such as BF_3 should be excluded from circuit packages of any kind. Results of water extraction analyses of six epoxy resin cements after digestion of 288 hours at 71°C showed chloride to be present from 2.7 to 380 parts per million¹⁷. That concentrations of chlorine as high as 500 parts per million might be tolerable¹⁸ is surprising if one

Table B
Results of Gas Analysis of FET Test Packages*

Adhesive	Amount (mg)	Constituent						
		H ₂ O (%)	O ₂ (%)	N ₂ (%)	BF ₃ ? (ppm)	Low Molecular Weight Hydrocarbons (ppm)		
Control	Zero	Zero	7.31	Balance		8		
Hysol 0151	22.7	0.10	1.31	↑ ↓		75		
Ablefilm 517	28.0	Zero	0.84		57			
Eccobond 104	30.0	↑ ↓	0.02		37			
Epo-Tek H61	47.7		4.72		347	3839		
Epo-Tek H74	47.6		Zero			63		
Ablefilm 529	41.5		Zero			21		
Ablebond 36-2	47.2		Zero			251		
Epo-Tek H31	42.1		3.43		147	4042		
DuPont 5504	48.2		0.08			2441		
Ablebond 58-1	100.3		4.52			559		
Epo-Tek H44	12.3		Zero		Zero	Balance	114	79

* After Perkins and Licari¹⁷

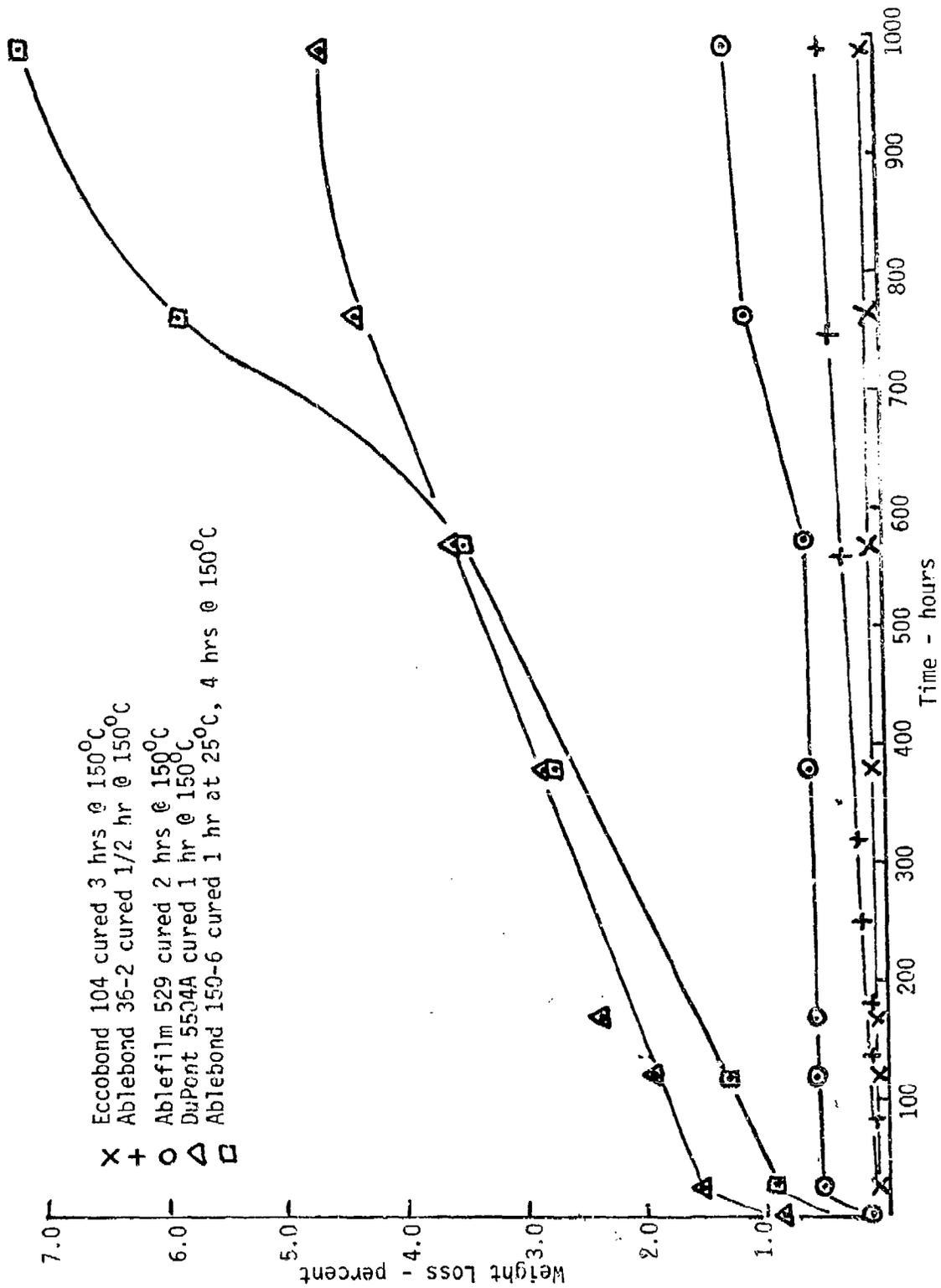


Figure 7-6. Weight Losses of Epoxy Adhesives Exposed to Air at 150°C for 1000 hours (after Perkins and Licari15).

considers that chloride ion need be present in small amount only in the locale of charged conductors to cause migration and failure.

Adhesion and viscoelastic behavior of organic adhesives, encapsulants, and sealants is not part of this study. Enough probably is known about making adhesive systems that desired adhesion and viscoelastic properties as well as adhesive wetting characteristics in the presence of sorbed water can be attained.¹⁹

B. Expansion Problems

1. Thermal Expansivities and Stresses in Encapsulated Systems

Almost all organic resins have much higher coefficients of thermal expansion than do metals, ceramics, and inorganic glasses. Therefore, electronic devices, integrated circuits, and components embedded in organic resins can be subjected to severe stresses in embedding processes or in storage and use. Differences in thermal expansivities, coupled with changes in viscoelastic responses of polymers, with changing temperature can produce sufficient stress that mechanical failures (particularly in leads and bonds to devices and circuits), increases in permeabilities or formation of leaks, and chemical reactions can occur.

Coefficients of thermal expansion (CTE) of inorganic materials and metals commonly found in hybrid circuits range from 3.2×10^{-6} to 24×10^{-6} per degree; the CTE's of unfilled resins are from 80×10^{-6} to $400 \times 10^{-6} \text{ deg}^{-1}$, and the CTE's of filled resins are from 15×10^{-6} to $110 \times 10^{-6} \text{ deg}^{-1}$,^{20,21} (see Table C). These differences are more than sufficient to produce stresses which can rupture leads and bonds with quite modest changes in temperature. For polymers below their glass transition temperatures typical moduli of elasticity are about 4×10^5 psi.²² This coupled with a difference in CTE's of $50 \times 10^{-6} \text{ deg}^{-1}$ and a ratio of cross sectional areas of ten to one could produce tensile stresses in leads as great as 2000 psi with a 10°C change in temperature.

Thermally induced stresses may be reduced by using filled polymers whose CTE's can be as low as $15 \times 10^{-6} \text{ deg}^{-1}$.¹ Of course, the filler cannot be a chemical contaminant or affect adversely the properties of either the circuit assembly or the resin.

Table C
Coefficients of Thermal Expansion - Linear

<u>Material</u>	<u>CTE $10^6 \text{ } ^\circ\text{C}^{-1}$</u>	<u>Ref.</u>
Silicon	3.2	1
Germanium	6.0	21
Alumina	6.5	1
Glasses	8-10	20
Steel	10-12	20
Gold	14	20
Copper	17	20
Aluminum	24	20
Filled Epoxy Resins	15-17	1
Filled Silicone Resins (no solvent)	30	1
Filled Phenolic Resins	35-110	1
Epoxy Resins (unfilled)	60-70	1
Phenolic Resins (unfilled)	80-110	1
Silicone Resin (unfilled, no solvent)	125	1
Polyurethane Resins Flexible (unfilled)	150	1
Silicone Rubbers	170-250	1
Silicone Gels	300-400	1

Instead of using rigid encapsulants, an obvious method of reducing, but not eliminating, thermally induced stresses is to use polymers at temperatures above their glass transition regions. Harper²³ has reviewed a method for making epoxy resins flexible. Polymeric resins can have large coefficients of thermal expansion above their glass transitions. Data of Baker²⁴ on an epoxy-glass composite structure are an example (Figure 7-7). Thermally induced stresses can be quite large in systems encapsulated in viscoelastic resins above the glass transition temperature range if temperatures are changed quickly. For example, if the temperature changes 10°C per second in an encapsulant with CTE $100 \times 10^{-6} \text{ deg}^{-1}$ and viscosity 10^{11} poises, the stress in a polymer may be over 1400 psi. This stress can be transformed to leads, bonds, etc., depending upon geometries. (However, rapid temperature changes can produce stresses in systems above the glass transition temperatures of encapsulants.)

Glass transition temperatures are strongly dependent on degree of polymerization, cure, presence of plasticizing material, etc. Glass transition temperatures for comparable epoxy resins ranged from 100°C to 200°C in one investigation.²⁵ This variations should be considered typical for proprietary resins.

C. Plastics Decomposition Products

Decomposition products from sealants, adhesives, and encapsulants can provide the deleterious materials which react electrochemically with components and integrated circuits and cause failure. Polymers such as epoxy resins can yield halides from epichlorohydrin and from curing agents such as boron trifluoride. Polyvinylchloride can give off hydrogen chloride; fluorinated polymers can yield hydrogen fluoride. Ammonia and amines from curing epoxy resins from polyamides can react with ions to form complexes and with acids or water to form cations. Acids from curing agents for epoxy resins, polyesters, polyamides, or the oxidation of any organic compound can form salts with metal ions aiding in the electrochemical oxidation and probably transport these metals. Water may be a decomposition or oxidation product of many materials.

In summation, the polymers used inside hermetically sealed systems can yield materials as destructive to integrated circuits and components as are those in the external environment.

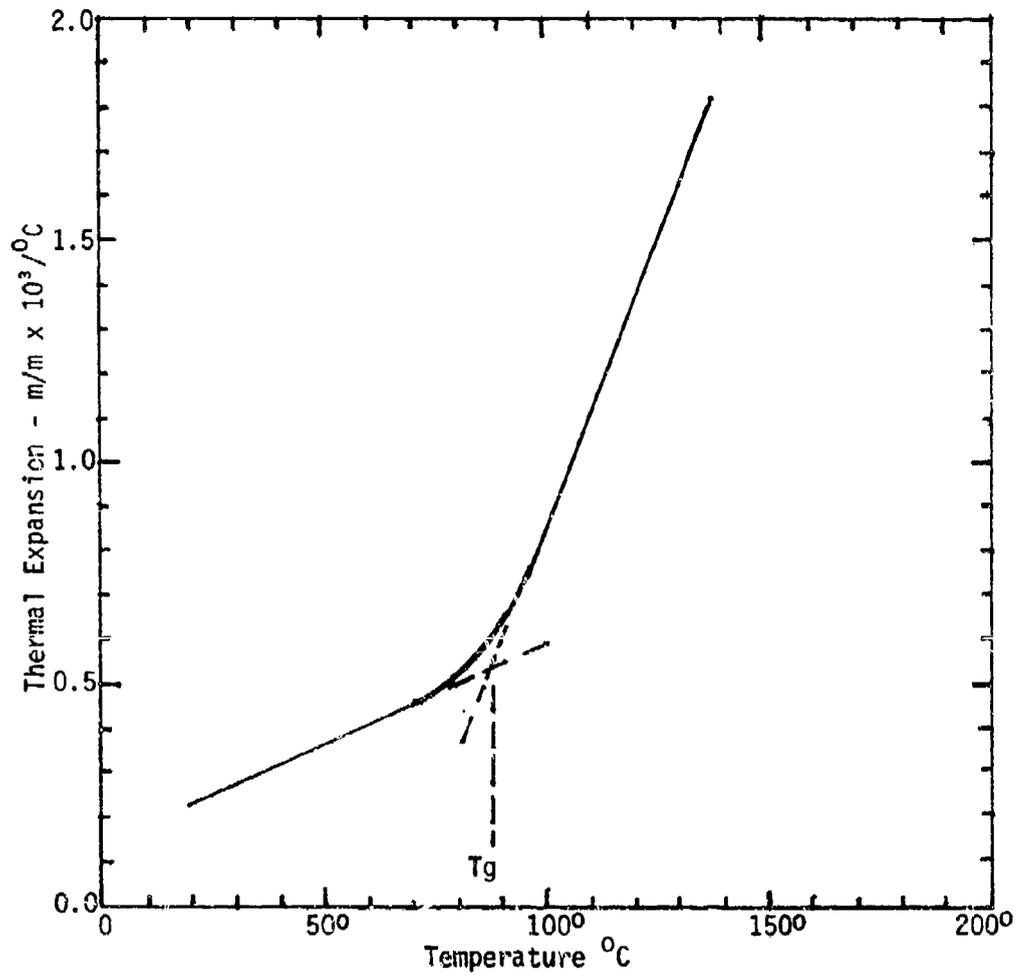


Figure 7-7. Thermal Expansion of an Epoxy-Glass Composite Measured Normal to the Surface (data from Baker²⁴).

Use of considerable care in selecting only stable epoxy resins without excess curing agents is necessary. Boron trifluoride should be avoided unless the absence of fluorides can be demonstrated. Use of hydrocarbon polymers, polyethyleneterephthalate, and well outgassed phenol-formaldehyde polymers is suggested. More reactive polymers, such as polyvinylchloride, epoxy resins, polymers, etc. probably should be avoided.

D. Conclusions

1. A quantitative definition of hermeticity is needed. We propose that a system be considered hermetically sealed if not more than one monolayer of deleterious material, e.g., labile water, be absorbed on critical working surfaces in the useful life (including storage time) of the device or circuit assembly. For example, a leak rate of about 1.2×10^{-13} cc(STP)/atm-sec would admit one monolayer of adsorbed gas (such as water vapor) on one square centimeter in ten years if the external pressure of the gas is one atmosphere.
2. Polymers cannot be used to seal circuits where even short-term storage reliability is required. Polymers are permeable to gases and water vapor and to liquids including water. Diffusion coefficients are high, in the $D \sim 10^{-9}$ to 10^{-5} cm²/sec range. Solubilities are sufficient that permeabilities range from $P \sim 10^{-10}$ to 10^{-4} cc(STP)/sec-cm-atm. These permeabilities are from 50 to 50 million times greater than the permeability of $P \sim 2 \times 10^{-12}$ cc(STP)/cm-sec-atm which would admit one monolayer at water through one centimeter thick material at 100% RH ($p_{\text{vap}} = 42.1$ mm Hg) at 37°C in ten years. If moisture barriers were to be ten microns thick, the permeability could be not greater than $\sim 2 \times 10^{-15}$ cc(STP)/cm-sec-atm. Permeabilities of polymers are too great by from seven to eleven powers of ten to serve as sealants for circuit assemblies.
3. Hermetic seals should be retained in circuitry components in U.S. Army missiles.
4. No chlorine or other halogen containing materials should be sealed in any circuitry components. Polymers used should be simple hydrocarbons or compounds of carbon, hydrogen and oxygen. Nitrogen containing polymers should be considered with skepticism.
5. Thermally induced stresses can be excessive in hybrid circuit systems encapsulated in polymers especially in rigid materials below glass transition temperatures.

7. Data on thermal expansion, glass transitions, and viscoelastic responses of polymer encapsulants and adhesives are too meager for design of circuit systems.

E. Recommended Studies

1. Leak rates of hermetic seals should be studied to define hermeticity, to develop leak tests, and to establish relationships among leak rates of water, helium, and other gases and vapors.
2. Measurements of permeabilities, diffusion coefficients, and solubilities of water in representative polymers should be made so that good data are available and effects of temperature, pressure, mechanical strain, previous sorption, and synergism of two or more penetrants be understood.
3. Measurements of allowable chlorine, chloride ion and water concentrations in microcircuits should be made. Water alone, chloride ion alone, or chlorine other than chloride probably can be tolerated at relatively high concentrations, but the chloride ion and water concentrations together must be low. These concentrations and concentration ratios probably control device and circuit life.
4. The measurement of water concentrations inside sealed devices and circuit assemblies should be continued and expanded. The "gettering" action of materials used in assemblies or added as "getters" should be studied.
5. The technique of water exclusion by displacement using polymeric encapsulants should be studied. For example, the efficacy of silicon rubber "sealants" for integrated circuits should be studied at various humidities including submersion. Time-temperature-humidity effects on performance with and without chloride ion present should be investigated.
6. Humidity-temperature-time tests should be studied. The immersion in 121°C (250°F) boiling water for 48 hours probably is too drastic in screening. Tests of 100°C immersion in fresh water and sea water and exposure to various humidities and immersion at lower temperatures should be made.
7. Careful attention should be paid to life testing in the telephone system. Performance for 10 to 40 years under extreme conditions such as in submerged or buried cables and repeaters is similar in rigors of conditions and time to those sought for storage and use of U.S. Army missiles.
8. The decomposition of and products formed from polymeric adhesives, encapsulants, sealants and substrates should be studied with care and in detail. This will require accurate and sensitive quantitative analysis in

the part in a billion range. Mass spectrometry, ion emission and absorption spectroscopy, careful gas analysis, x-ray fluorescence activation analysis, and chromatographic separation techniques will be necessary.

9. The tolerable levels of chloride ion water should be determined. In the presence of water, very little chloride ion should be present. Tolerable levels of cations such as Na^+ , Br^{++} , Mg^{++} , etc. should be determined.

10. The thermal and photolytic decomposition products of polymers used should be determined. This requires careful analyses of the one part in 10^6 level.

11. The effects of water and oxygen on the decomposition of polymers in closed systems should be determined.

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VIII. SCREENING

A. Introduction

Some aspects of screen testing are discussed in this chapter with special attention to the missile storage environment. A comprehensive screening procedure able to remove all types of weak or defective devices from the population has clearly not been realized. The potential failure mechanisms are numerous and the device construction details too diverse to realistically search out all weaknesses with a single set of stress tests. There actually are only a few ways to observe and exercise the delicate composite structure of a microcircuit without undue damage to good devices. Some steps in screen tests will rapidly extend a device population into the wear-out phase of the "Bath-Tub" curve if the stress parameters selected for the test are too severe. It is also known that latent defects can be created during screen testing. Obviously, this situation must be carefully avoided. Some of the significant structural or chemical mistakes made in the fabrication of devices are not revealed in reasonable times using current test methods. For example, it is necessary to destructively puncture a package to determine if the moisture level is high because direct measurements are not yet satisfactory. Furthermore, moisture induced failure modes often require longer time periods to appear than would be practical using current screening stresses. The screen must also be appropriate to the intended use of a device. Only after the important failure mechanisms are identified can the screen test procedure having the best chance of removing devices with appropriate defects be selected. We found that the organizations with the most effective parts quality programs used individuals having a strong understanding of device materials to make judgments concerning the emphasis of their screen testing. Based on long personal experience, they looked most closely for particular failure mechanisms in certain devices.

B. Screen Test Steps

High reliability screen testing normally includes steps from the following lists:

1. Visual Examination
 - a) inside package
 - b) exterior of package

2. Elevated Temperature at no Bias
3. Temperature Cycling
4. Thermal Shock
5. Mechanical Shock
6. Constant Acceleration
7. Nondestructive Bond Pull for Hybrids
8. Package Leak Tests
9. Burn-In Tests at Various Ambient Temperatures
 - a) forward bias
 - b) reverse bias
10. Electrical Parameter Measurements to Detect Changes
11. Radiography
12. Particle Impact Noise Tests

The visual examinations reveal obvious mistakes during fabrication at no risk of degrading the device. The details requiring attention are addressed comprehensively in Methods 2010 and 2017 of MIL-STD-883 for monolithic and hybrid microcircuits, respectively. Any indication that a chemical contaminant is present should reject the part. In addition, the criteria in MIL-STD-883 for cracks, scratches, voids and other geometrical features potentially producing mechanical stress concentration factors should be made more severe for stored than for operating devices. Mechanical stresses arising from sources discussed in Chapter IV will be most effective in causing device failure at the leading edges of sharp fissures. In addition, moisture tends to accumulate at such sites, thereby stimulating localized corrosion and chemo-mechanical processes. The thresholds for crack propagation in the thin elements of a microcircuit have not been either theoretically or experimentally evaluated for the conditions associated with storage stresses. Therefore, fundamental measurements will be necessary before visual crevice criteria appropriate for devices in storage can be specified.

The supposed benefits of maintaining unbiased devices at an elevated temperature are found to be a point of some controversy. High temperature bakes are generally considered inexpensive and nondegrading to properly constructed devices. It is clear from the investigations referenced in Chapters IV and

VII that material modifications will occur at the class B requirements of 24 hours at 150°C. Diffusion of the metal alloy and impurity atoms within critical features such as bond interfaces and also certain types of chemical reactions will proceed under these conditions. Although no one counts on the bake to reveal defects independently, it is often believed that subsequent screen stresses will fail devices having certain weaknesses due to the interactions occurring during the bake. For example, the accumulation of impurity atoms at a bond interface during the bake may cause the bond to lift later during temperature cycling or burn-in. Conversely, the high temperature bake can be beneficial as a metallurgical anneal for properly fabricated microcircuit structures. Vacuum and sputter deposited metal films are well known to have high dislocation densities equivalent to a highly work-hardened bulk material. In addition, mechanical stresses associated with the lattice constant misfit at interfaces between composite layers can be relaxed to some extent by the anneal. The metals in the neighborhood of a bond are also highly work-hardened so that the bake anneals residual stresses here as well as permitting additional interdiffusion at clean bond interfaces to make a better junction.

Two experimental investigations illustrate the factors discussed above. Sulouff¹ and Robertson² have both examined bonds subsequent to at least the bake and temperature cycling screen steps. Sulouff employed a bond pull force method and Robertson a bond resistance criteria to evaluate the effect of screening stresses. While many details differed in these two studies, both demonstrated thermal induced changes in the materials at some stage during the screen test steps. Sulouff and Robertson each found that the performance of poor bonds was made worse and that of good bonds better subsequent to the imposition of these stresses.

Temperature cycling is intended to provide cyclical mechanical stress of the microcircuit structure based on differences in the thermal expansion coefficients of device materials (See Table A of Chapter IV). However, there is widespread controversy concerning the optimum number of cycles for this step. During our many visits, we found practically no support for the usual MIL-STD-883A requirement of 10 cycles at limits of -65°C and 150°C.

One would not expect easily detected accumulated fatigue damage at these stress levels from only 10 cycles. Various organizations have adopted their own temperature cycling screen test procedures with the number of cycles ranging from 25 to about 300 cycles. Although these workers usually had some empirical confidence about the number of cycles adopted for their tests, no one appeared able to relate degradation of faulted devices with the stresses imposed by thermal cycling. The needed relationships between cyclical rate, types of faults, device construction, temperature extremes and number of cycles required to reveal particular faults have not been generated. As discussed in Chapter IV, there have been investigations evaluating the wear-out processes due to cycling of some wire bond and package systems. These studies indicate that wear-out begins at somewhere between 1000 and 4000 temperature cycles. With one exception, the several wear-out investigations did not examine specified faults or environments. These data, therefore, indicate that a temperature cycle screen should involve accumulated cycles considerably less than 1000 cycles.

Thermal shock is often substituted for thermal cycling by a number of organizations and is allowed by MIL-STD-883A Method 5004.3 for class B devices. Individuals expressed the opinion that an 80% correlation exists between defects revealed by thermal shock and temperature cycling stresses. Given the same achieved temperature extremes, the stresses involved in thermal shock should include those introduced by temperature cycling. It is usually less expensive and faster to conduct the thermal shock test. However, as discussed in section C of Chapter IV, additional factors come into play with thermal shock. The potential is greatly increased for unnecessary damage to brittle materials such as the chip, glassivation layers and ceramic seals during thermal cycling. For example, T.I. noted thermal shock damaged seals such that subsequent MGRS failures occurred. The biggest concern is that the damage done here might not be detected during any phase of screen testing. The storage environment is particularly apt to cause growth of the type of defects potentially introduced by thermal shock and lead to failure even years later. Therefore, the thermal shock option should be strongly discouraged for screening devices placed in Army tactical missiles.

Constant acceleration is used to detect poor die attachments and very weak gold wire bonds. With a linear density of about 0.01 mg/mm for 1 mil diameter gold wires the inertial force at 30,000 g's is 0.9 grams for a 3mm wire length. This force is shared by both bonds, so clearly the force on a single bond is not large. A similar length of 1 mil aluminum wire with a linear density of 0.0014 mg/mm experiences a total force of only 0.13 grams. A 1 mg chip feels 30 grams at 30,000 g. Obviously, one should never consider checking aluminum wire bonds by inertial stresses and lower accelerations will have quickly decreasing value, even for gold. The vulnerability of some large hybrid packages has limited constant acceleration testing to much smaller values of g so that wire bonds are really not tested at all. Mechanical shock based on instruments which are essentially air guns have been used for very small electronic devices, but are not considered appropriate for larger I.C.'s and certainly not hybrids. Factors associated with hermetic seal testing are discussed fully in Chapter V.

Particulate matter within a sealed package is detected using either radiographic techniques or the more recently developed acoustic methods. The low absorption of X-rays by low atomic weight materials severely limits the usefulness of radiographic methods. However, significant advances are currently being made in the art and science of loose particle detection by monitoring particle noise in a vibrated device package. One supplier now claims his system will detect particles as small as 1×10^{-7} grams. This corresponds to a sphere of aluminum with a diameter a little greater than 1 mil. A large number of organizations had adopted their own techniques for conducting particle testing as noted in reference 139 of chapter IV. However, no one has been able to differentiate between the particle material. Normally, one is only concerned about conductive particles, but many devices must be rejected because of harmless, non-conductive particulate matter. A concern for storage reliability is that many particles tend to be caught in crevices or are otherwise attached to some part of the package and then are not detected by the acoustic screen test. We found workers using various "standard" shock methods to free trapped particles. Electronic instrumentation for graded mechanical shock is now becoming available. The effectiveness of particle detection screen testing appears to be closely related to the operator at this time.

C. Deliberate Defect Investigations

The basic purpose of a screen test is to separate faulted or otherwise weak devices from the part population without doing significant damage to the remaining devices. This implies a knowledge of the type and amplitude of stress required to cause particular defects to be revealed. In reality, people have accumulated screen effectiveness information by subjecting device populations to typical screen stresses and then observing the failures. This procedure obviously can be neither precise nor efficient for establishing optimum screening parameters, since both the type and number of initial defects is generally unknown. The philosophy needed for developing meaningful screen testing parameters is to concentrate on determining the specific stress-duration levels required to reveal well defined device faults. This means that the capability is needed for fabricating devices with deliberate defect types, severity and number. This implies either quantitative measurements in advance of defects randomly introduced during fabrication or a deliberate modification in the manufacturing process to form these defects. Then the failures due to specific defects which occur during screen testing can be used to determine the effectiveness of particular screens. For example, graduated impurity levels can be introduced at bond interfaces. Experiments should then be performed to determine the effect of these impurities on bond durability in expected device environments. This information would provide the basis for an appropriate screen test to fail bonds having unsatisfactory durability related to interface impurity levels.

A current program undertaken by Rockwell International³ for MIRADCOM is designed to evaluate the effect of several screen steps on selected deliberately introduced defects. One of these is marginal bonds based on adjustments to an ultrasonic bonder. Weak bonds of two kinds were produced on a consistent basis following careful experiments using the bond machine parameters. Under bonded wires were fabricated with a mean pull strength of about 2 grams and the other defect was an over-bonded condition which would not have passed an alert visual examination. The materials included Al-Al, Au-Al and Au-Au couples. Although their results are not final, it is clear that 10 temperature cycles had no effect. Extended cycling, up to 500 cycles for Au and 300 cycles for Al, did not result in electrical opens for this

type of bond defect. However, the pull strengths of bonds with these defects were altered in an interesting manner subsequent to the extended temperature cycling. The strength of marginal bonds fabricated in the underbonded condition was increased whereas those made in the overbonded condition were significantly weaker. Subsequent investigations were conducted on this type of defect using a screen stress sequence of stabilization bake, temperature cycling and constant acceleration. Although this sequence did reveal some of the defective bonds as electrical opens, most of the defects remained undetected until destructive pull testing was conducted.

The Rockwell screening investigations have therefore demonstrated that temperature cycling is not effective for removing bonds which were defective because of a badly adjusted ultrasonic bonder. Although micrometallurgical examinations have not been done on these bonds, it is reasonable to assume that the under-bonded couples were made stronger by the additional interface diffusion at the high temperature regions of the temperature cycling tests. The reduced strength of over-bonded couples might have resulted from the thermal-mechanical stresses at the interface of the highly work-hardened metals within the bond region. The effectiveness of annealing here is undetermined. The scope of this program did not permit the examination of bonds having other types of normally encountered defects. For example, it would be particularly useful to know how effective this screen sequence is for detecting defective bonds based on selected interface, substrate or wire impurities. The Rockwell program represents a step in the development of effective screen test procedures based on actual knowledge of how a particular defect responds to specific screen stresses. However, the temperature cycling screen test study at Rockwell has not resolved the fundamental controversy relative to an optimum number of cycles. The limited number of variables examined did not include many additional factors relative to strip line defects, impurities, rate of cycling and chip defects known to be significant. Other aspects of the Rockwell International screen testing investigations concern the analysis of gases in hybrid packages and MOS/LSI process parameters. The package gas studies in progress are concerned with the precision of current state-of-the-art measurement methods and some effects of moisture on microcircuit materials. The MOS/LSI investigations are discussed below.

D. MOS Devices

The problems associated with the screening of MOS devices and, in particular, MOS/LSI devices have been discussed with a number of technical personnel in our visits to industrial and government installations. The predominance of alkali ion (mainly sodium) contamination in gate oxides and the presence of oxide defects (e.g., pinholes) are possible causes of long term drift in MOS devices in storage. There is not a general agreement on the use of high temperature reverse bias screens versus high temperature operating bias screens. One would expect that the screen which exercises the maximum number of gates would be preferable, however, the actual bias-time product would also be important in terms of the drift of mobile alkali ions in the field across the oxide. Internal visual inspection prior to capping would ordinarily pick up problems with mask alignment, coverage of oxide steps, etc., but would not identify oxide defects such as pinholes under the metallization. Destructive tests are usually necessary in that case and, therefore, would not constitute a screen test. The stabilization bake or high temperature storage screen (125°C, 24 hrs., no bias) has questionable value for MOS devices unless it functions to stabilize the amount of surface charge. However, if sufficient moisture is present a gradual change in surface charge can take place with time. Whether stabilization bake can also be considered a possible screen for oxide defects or mobile ions in the oxide needs to be examined. In order to resolve some of these uncertainties, the program with Rockwell International was initiated by MIRADCOM. The purpose of the program, with respect to MOS/LSI devices, was to introduce known defects at the wafer level and then to determine if the various screens could detect the presence of these defects by comparison of electrical parameters for devices with and without the known defects.

The Rockwell effort has made effective use of a process evaluation circuit (PEC) which was one of their standard (PMOS) circuits containing many different devices. Measurable parameters of the devices enable correlation of the electrical behavior with various process steps. For example, threshold voltage shifts are investigated by measuring V_T and V_{GST} of a small unit FET and an 8 x 8 mil unit FET; charge at the Si-SiO₂ interface is checked by C-V analysis on a gate oxide capacitor; leakage current is measured

for a serial parallel multiplier, an 8 mil wide gate FET and the 8 x 8 mil unit FET; and circuit speed is measured directly for the dynamic serial parallel multiplier. In addition to the PEC, a production MOS device which is a CPU for one of Rockwell's microprocessors was also fabricated. Since the PEC used $\text{SiO}_2/\text{Si}_3\text{N}_4$ gate dielectrics, C-V tests on the SiO_2 for sodium ion contamination were also done on test wafers prior to the deposition of Si_3N_4 and bubble tests were made on the combined dielectric. The results of the study show transistor data for three sets of parts subjected to stabilization bake at 200°C for 24 hours and 100 hours: (1) parts fabricated as close to process guidelines and tolerances as possible; (2) parts fabricated exactly the same as (1), but metallized using a contaminated sputtering target in a chamber known to be contaminated with sodium; and (3) parts fabricated exactly the same as (1), but with deliberate misalignment of the metal mask to expose gate oxide and bare silicon in contact areas. While it is not possible to state conclusively at this time on the sensitivity of the stabilization bake screen for oxide defects, it does appear certain that judicious use of a process evaluation circuit, along with production runs of MOS/LSI devices, is an advantageous approach for attaining higher reliability.

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IX. ACCELERATED TESTING

A. Introduction

Predictions are sought for the reliability of electronic devices and systems. We can do this only by experience, but we can't wait over the useful life of each device or system to make our observations. Therefore, we use "accelerated aging" tests and extrapolate in time. Experience is our only justification; this procedure has no scientific basis. We delude ourselves with mental gymnastics in kinetics to assuage our fears and mask our uncertainties. To approach the prediction of reliability with objectivity, we must understand the bases of kinetics and its application to predicting physico-chemical behavior in time to come.

Our electronic devices are complex systems undergoing mechanochemical, electrochemical, and mechanoelectrical changes. These changes must be considered together. At any time in an electronic device, several hundreds or thousands of interdependent chemical, mechanical, and electrical reactions and processes may be occurring simultaneously. How these processes add determines the behavior and fate of the device.

1. Rate Equations for Various Processes

The basis for all treatments of kinetics may be stated simply as, "the rate at which a system approaches equilibrium is proportional to the departure from equilibrium." Based upon this postulate we may write the rate equation

$$\text{Throughput} = \frac{\text{Push}}{\text{Drag}} = \text{Rate Constant} \times \text{Potential} \quad (1)$$

This equation describes the motion of a pendulum,¹² the conduction of heat,^{2,10} the flow of fluids,^{1b,3,4,5,6,7,8,9} diffusion of matter,¹⁰ conduction of electricity,¹¹ rates of chemical reactions,^{12,13,14,15} visco-elastic deformation of materials,^{3,16,17} rates of radiation of heat,^{18,19} and the generalized rate equations of so-called nonequilibrium thermodynamics.^{20,21}

Because rate equations for all or almost all physical and chemical phenomena are of the form of the linear rate equation given above, a simple linear rate equation can approximate the complex electrical, mechanical,

and chemical aging behavior of any system including electronic circuits. Although there is not a good example from electronics, the combustion of fuel oil illustrates the additivity of linear rate equations. The partial combustion of simple hydrocarbons involves several hundred chemical reactions;²³ the practical combustion of fuel oil probably involves several thousand or tens of thousands of reactions. In addition, flow of oil, separate into droplets, mixing with air, diffusion, convection, radiation, thermal conduction, charge transfer, and other phenomena are involved. Rates of these processes can be described by linear rate equations. These add for the multitude of parallel, sequential, competing, and branching processes so as to give an overall linear rate equation with only fuel and oxidant concentrations as the potentials.²³ We must realize that obtaining a fit of rate data by a near-equilibrium, linear rate equation tells us nothing about the processes involved in anything but the simplest, homogeneous systems.

B. Temperature Dependence of Rate Equations

van't Hoff showed that the temperature dependence of the equilibrium constant (expressed in terms of concentrations, that is, at constant volume) is given by the equation^{15a}

$$\frac{d \ln K_e}{dT} = \frac{\Delta E}{RT^2} \quad (2)$$

where K_e is the equilibrium constant, T the temperature, ΔE is the change in energy in going from reactants to products, and R is the gas constant. He also had observed that the equilibrium constant is given by the quotient of the forward and reverse reactions, this based upon Guldberg and Waage's ideas of mass action.^{15b} Combining these observations, Svante Arrhenius in 1889 stated that a reasonable equation for the temperature dependence of rate constants is²⁴

$$\frac{d \ln K_r}{dT} = \frac{\Delta E_{act}}{RT^2} \quad \text{or} \quad K_r = Ae^{-\frac{\Delta E_{act}}{RT}} \quad (3)$$

where K_r is a rate constant, A a constant, and ΔE_{act} is an energy of activation. Arrhenius believed that only molecules which had acquired energy

equal to or greater than ΔE_{act} could react. This interpretation persists today.

Arrhenius knew and appreciated the intimate connection between equilibrium and kinetic phenomena. Therefore, he expected that rate constants, and equilibrium constants should have temperature dependences of the same form. The postulate of near equilibrium kinetics leads to the same result. The argument is as follows: near equilibrium states can be described by thermodynamic functions similar to those of equilibrium states. Therefore, if the equilibrium constant at constant pressure K_p is given by ^{21b}

$$-RT \ln K_p = \Delta G^0 = \Delta H^0 - T\Delta S^0 = \Delta E^0 + \Delta pV^0 - T\Delta S^0, \quad (4a)$$

where ΔG^0 , ΔH^0 , ΔS^0 , ΔE^0 , and ΔV^0 are the changes in Gibbs free energy, enthalpy, entropy, energy, and volume respectively, in going from reactants to products at standard conditions (at some pressure p), then the rate constant K_p at constant pressure at some near equilibrium state may be written as

$$-RT \ln K_p = \Delta G^\ddagger = \Delta H^\ddagger - T\Delta S^\ddagger = \Delta E^\ddagger - p\Delta V^\ddagger - T\Delta S^\ddagger \quad (4b)$$

where ΔG^\ddagger , ΔH^\ddagger , ΔS^\ddagger , ΔE^\ddagger , and ΔV^\ddagger are the activation free energy, enthalpy, entropy, energy, and volume respectively in going from reactants to some near equilibrium, activated state capable of reacting. Eyrings' notation ²⁵ is used intentionally. We see that the Arrhenius and Eyring models are identical.

Use of time-temperature-potential (or driving force) superposition required that the rate equation be known exactly for each process and that the temperature dependence of each rate process be known precisely. The activation energy, E_{act} of Equation 3 or ΔE^\ddagger of Equation 4b, is usually a function of temperature. For transport and mechanical processes in polymers, the activation energies can be very strong functions of temperature. ²⁶

C. Limitations on Kinetics and Time-Temperature-Potential Superposition and Extrapolation in Time.

Treatments of kinetic phenomena are based upon the postulate of linear kinetics: "the rate at which a system approaches equilibrium is proportional to the departure from equilibrium". Therefore, our equations are limited to near-equilibrium systems. This limitation need not concern us in most cases. All

of our experience shows that linear kinetics is applicable to most systems and fails only in cases of extreme departure from equilibrium.

Three critical conditions must be met if time-temperature-potential superposition is to be valid. These conditions are:

1. All possible processes and combinations of these processes must be considered.
2. The processes and combinations thereof must occur over the whole range of temperature considered.
3. Temperature coefficients of the rates of all processes must be known. This means that "activation energies" of all processes must be invariant with temperature or their dependence on temperature be known precisely.

In almost all cases where we use time-temperature-potential-superposition and extrapolation in time from accelerated aging studies to predict useful service life of systems, none of the above conditions are met. Nevertheless, we use accelerated aging studies, as we must, because we have nothing else from which to predict useful life of systems. We must be cautious not to delude ourselves as to the quality and reliability of our projections from accelerated studies.

Quite obviously, time-temperature-potential-superposition is limited to small changes in systems. Drastic or permanent changes associated with phase changes, radiation damage, mechanical deformation, structural change, electrically and mechanically induced chemical reactions, chain reactions, and parallel reactions make time-temperature-potential superposition next to impossible. Processes of this sort occur in storage and use of electronic devices. For example, there is no way to separate mechanical and chemical processes in polymers. Electrical processes are usually ignored due to a lack of knowledge and at considerable risk. In electronic devices, components, and systems made up of conductors, semiconductors, and insulators, and including organic and inorganic polymers, one must consider electrical, electrochemical, and electromechanical processes as well as chemical and mechanochemical phenomena. In spite of the complexities of systems, there are attempts to make time-temperature-potential superpositions and extrapolations in time as though a single, small change was occurring in each system. On the basis of meager information and such interpretation, predictions are made that devices and circuits will not be harmed by particular materials, environmental conditions, stresses (potentials), etc. over long

periods of time. Such extrapolation is dangerous at best and more likely foolhardy if changing conditions and probable mechano-chemical-electrical processes are neglected.

The only basis for use of time-temperature-potential superposition and extrapolation in time is real experience in how specific accelerated tests correlate with actual performance.²⁷ What is now a relatively simple example of accelerated aging in thermal oxidation of polyethylene demonstrates this. For many years, measurement of rates of oxygen uptake at 140°C or 150°C has been used as a screening test for antioxidants for polyethylenes.²⁸ Quite obviously, oxidations of polyethylenes at 140°C are not representative of oxidations at lower temperatures if for no other reason than polyethylenes "melt" in the 110° to 115°C or the 120° to 135°C ranges depending on chain branching^{29a} and thermal history^{29b} and that only less ordered, noncrystalline polyethylene can be oxidized at temperatures below the melting ranges.^{28b,30} Sufficient experience has been accumulated to show that in spite of this and other limitations, the 140°C oxygen uptake rate is a good screening test for antioxidant systems for polyethylenes.^{28c} However, the only basis for use of this 140°C accelerated test is more than thirty years of experience³¹ from careful experiments.

Our problem is to apply our experience in correlating accelerated aging studies with actual performance. Electronic devices and systems are much more complex than the oxidation of polyethylene examples cited because we must consider electrochemical and electromechanical processes as well as chemical and mechanochemical phenomena. Considerable experience has been gained in the past forty years from aging studies of resistors, capacitors, tubes, insulation, transistors and some integrated circuits. This experience must be used carefully to avoid unwarranted conclusions being drawn. We must admit that almost all of our experience is qualitative and cannot be reduced to quantitative extrapolations in time.

D. Some Comments on Justifications of Accelerated Testing Models

1. Overinterpretation of Rate Theory

One example of the misuse of kinetics equations to gain unreliable or baseless conclusions is a purported derivation³² of the "power rule" for capacitors³³ from Eyring's formulation of rate equations.²⁶ The "power rule"

for capacitors expressed in differential form

$$\frac{d(\text{damage})}{dt} = \text{constant} \times (\text{voltage})^n \quad (5)$$

is exactly the near equilibrium rate equation applied to damage of capacitors. This equation is an overall rate equation which sums and averages over equations of the same form for chemical reactions, current flow, diffusion, thermal conduction, etc. Eyring's model is exactly the same near equilibrium rate equation with the rate constant expressed in terms of near equilibrium, state functions of thermodynamics, namely, the free energy, enthalpy, and entropy. Identification of the rate constant with thermodynamic state functions in no way constitutes a derivation of or gives a theoretical basis to the "power rule" for capacitors.

2. Acceleration Factors in Accelerated Aging

The assignment of numerical acceleration factors in time-temperature-potential superposition and extrapolation in time probably is not justified because of the primitive theory undergirding kinetics and the incompleteness of experiments describing aging processes. Because we can write overall rate equations summing over any number of processes and because many of these overall rate constants follow Arrhenius' temperature dependence, time-temperature-stress superposition and time extrapolation seem to work for accelerated aging of some devices, components, and circuits. We must be careful not to place too much reliance upon this apparent agreement between experiment and "theory." The summation of rates over many processes averages out information so that any details of processes are lost. This averaging appears to be as destructive to information as averaging over small ranges of size, shape, refractive index, etc. in scattering of electromagnetic radiation.³⁴

The use of and reporting of "acceleration factors" to two, three or four significant figures and extrapolations in time to two significant figures³⁵ cannot be justified. Such over reliance on extrapolation by authorities in the field invites disastrous error by those less skilled in the art.

3. Additivity Assumptions in Varying Potentials During Tests

The effects of stepwise and continuous increase in time-temperature-potential superposition are taken to be additive in accelerated aging studies.

In studies of electrical devices, components, or circuits the potential varied usually is voltage.³² However, potential in this context means "driving force" and can be mechanical stress, concentrations of chemical reactants, voltage, etc. For example, the effects of stepwise and continuous increase of voltage on the failure rate of capacitors has been analyzed assuming additivity of damage.³² This is exactly the additivity of the Boltzmann superposition principle³⁶ as applied in linear viscoelasticity.^{37,38} Strains produced by various components of stress in a loading program varying with time are added separately or stresses are added separately for strains in a strain program, and viscoelastic response to a stress program can be calculated from stress response to a strain program and vice-versa. That damage effects in capacitors are simple additive functions of potential is no more likely than stress-strain effects are additive in real materials at finite strains. Superposition simply does not work for viscoelastic response of real polymers at finite strains.^{38b,39,40}

Our considerable experience with departure from additivity in viscoelasticity can guide us in time-stress superposition in accelerated aging. Departure from additivity should be expected for strain (flow) processes which involve cooperative movement of structural moieties in anisotropic, heterogeneous, composite structures such as polymers. Electromechanochemical systems such as electrical devices, components, and circuits are at least as complex structures as polymers and are involved in at least as complex, cooperative processes as are polymers. Therefore, we should expect time-temperature-potential superposition and time extrapolation to be nonlinear in stress and temperature. (Consider the nonlinear temperature dependence of strain behavior of polymers.)²⁶ If we expect nonlinear superposition and extrapolation in accelerated aging, we must treat our linear superpositions and extrapolations with skepticism and care.

4. Comments on Distribution Functions Describing Rates of Failure

In life testing, if populations of devices, components, or circuits functioning at various times can be described by some convenient distribution function, manipulation and extrapolation of these rate data are simplified. No justifications from kinetics theories are mentioned in most works on life testing.

The most logical distribution in time might be that from simple first order kinetics which leads to an exponential decrease in number with time.

This is the case if the number of units which fail in unit time is proportional to the number of units operating. Compared to experience this distribution is not nearly broad enough; decay is too rapid with time.

Simple zero order kinetics in which the number failing is independent of the number operating gives a constant rate of failure for a given starting population. This gives a broad distribution in which the number operating varies inversely as time. Experience is that rate of failure decreases somewhat with increasing times at long times.

Necessary to represent the experiment well is a distribution that is broad and for which the rate of failure rises from zero at time zero through a maximum at some relatively short times and then decreases slowly over long periods of time. The Weibull distribution⁴¹ has these properties. Thus, for the fraction of units, $P(t)$, which have failed at time t

$$P(t) = 1 - e^{-t^\beta/\theta} \quad (6)$$

where β and θ are constants, the fractional rate of failure is

$$\frac{dP(t)}{dt} = \frac{\beta}{\theta} t^{\beta-1} e^{-t^\beta/\theta}. \quad (7)$$

The lognormal distribution⁴² behaves similarly. For the fraction failed $P(t)$ at time t

$$P(t) = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^z e^{-u^2/2} du \quad (8)$$

where u is a variable of integration and the probit⁴³ is

$$z = \frac{\ln t - \ln \bar{t}}{\sigma} \quad (9)$$

where \bar{t} is the geometric mean life of the population and σ is the standard

deviation of the logarithms of the life times. The fractional rate of failure for the lognormal distribution is:

$$\frac{dP(t)}{dt} = \frac{t^{-1}}{\sigma \sqrt{2\pi}} e^{-\frac{1}{2} \left(\frac{\ln t - \ln \bar{t}}{\sigma} \right)^2} \quad (10)$$

These distributions, the Weibull and lognormal, are quite similar. Neither has an obvious advantage in representing experiments. However, Peck and Zierdt prefer the lognormal distribution based upon their experience.⁴⁴ The lognormal distribution has been used enough that we tend to forget that its application in these aging studies is empirical. Smith and Vaccaro observed that a lognormal distribution of lifetimes should be expected for a multiplicative effect of randomly distributed variables.⁴⁵ That the mechano-electrical-chemical processes that lead to failure are randomly distributed and multiplicative is unlikely. More probable is that these processes are many in number and are sequential, parallel, competitive, and chain reaction processes whose effects are additive. If we sum over a relatively small number of such rate processes, we should get a broad distribution in time like the Weibull or lognormal distribution for unreacted species. We see this if we consider even two consecutive reactions or simple chain reactions⁴⁶ which lead to concentration profiles in time like the Weibull and lognormal distributions. Thermal degradations and oxidations of organic materials usually involve chain reactions. Mechanisms of chemical reactions at surface of solids are little understood; many of these combine consecutive and chain reactions. Mechanisms of electrochemical and mechano-chemical reactions and mass transport including ion migration are unknown. Rates of all processes add stoichiometrically to give the overall rates of failure and the distributions of species with time.

E. Comments on Treating the Combined Effects of Temperature and Humidity on the Lives of Devices, Components and Circuits

A few, different empirical mathematical models have been used to treat the combined effects of temperature and humidity on the service lives of devices, components and circuits.⁴⁷⁻⁵² These include using humidity as a potential (accelerating stress)⁴⁷ partial pressure of water as a potential,⁵⁰

and humidity squared as a potential.⁵¹ Reich and Hakim⁵² quite arbitrarily have used the expression for failure rate λ

$$\lambda = e^{A + B (T + RH)} \quad (11)$$

where A & B are constants, T is temperature, and RH is relative humidity in percent. Reich has compared these models using the doubtful method of calculating numerical acceleration factors; he concludes that more data are necessary to test the models.⁵³ The fact that different empirical models can be used to represent effects of humidity and temperature and that one of these, an arbitrary summing of temperature and relative humidity in an Arrhenius type equation,⁵² seems to work but gives widely variant "acceleration factors" probably is a result of writing overall rate equations to describe the rates of cumulative effects of hundreds or even thousands of processes. We probably should expect that narrowing choices to even a few models to describe these complex sums of many processes is unlikely.

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X. STATISTICAL APPROACHES TO STORAGE RELIABILITY

A. Introduction

The present Army program on storage reliability gives important recognition to the system reliability of missile systems in storage. The statistical analysis of field and laboratory data on electronic component failures requires an adequate grouping of failures in terms of failure mechanisms and technologies. However, as pointed out previously, there are limited data of this type currently available. Thus, the main thrust of our study was to identify potential failure mechanisms in terms of fundamental materials processes and the expected exposure to various environmental stresses. In this way we have been able to focus on the most critical situations which would limit the storage reliability of missile systems. Nevertheless, statistical approaches to analyzing failure rate data and for obtaining reliability predictions are of value in initial estimates of system reliability. This chapter includes discussions on three aspects of statistical methods: 1) a review of relevant literature on system reliability; 2) a procedure for modeling storage reliability when more than one failure mode exists; and 3) a consideration of the sampling requirements of MIL-STD-883A, a topic which would be relevant to the selection of sample sizes for small qualification lots of complex and expensive hybrid microcircuits.

B. Storage Reliability of Missile Systems

The need to predict the reliability of missile systems in storage or dormant states has been studied and numerous reports and papers have been published in an effort to develop a reasonable model that can be widely used. This section contains a review of some of that literature.

The first problem is the attempt to analyze the data. In order to do this one must first analyze all the different failure modes. Jordan¹ discusses one approach to this analysis that even reaches to the designing stage of the system. The method is called Failure Modes Effect and Criticality Analysis. The first step in a Failure Modes Effect Analysis has six parts.

- a) Define the system requirements.
- b) Establish ground rules to which the FMEA is performed.

- c) Describe the system hardware and functional blocks.
- d) Identify failure modes and their effects.
- e) Compile the Critical Item List.
- f) Document the analysis.

The second step, Criticality Analysis, is where the criticality numbers (c_p) are computed. The paper performs this analysis on a simple hardware system. There are several other papers which deal with the analysis of data. They are concerned with the analysis of accelerated life test data. Nelson² presents a graphical method based on the Arrhenius model. The methods require complete data. The paper is concerned with estimating the parameters, α , β , σ , where $\mu(x) = \alpha + \beta x$ ($\mu(x)$ is the logarithmic mean and σ is the logarithmic standard deviation). Nelson and Kielpinski³ present a method somewhat similar to the one presented by Nelson, but this one can be used on censored data and is based on the log normal and normal life distributions. This paper discusses common and optimal plans and shows how to arrive at these types of models. Plithides⁴ uses a different type of distribution than either of the previous ones, the Weibull distribution. This paper deals with a study conducted on the reliability of washing machines.

There are several general papers on some aspects of reliability. One written by Lucinda Mattera,⁵ though not very technical, discusses failures and the increasing importance of reliable parts since every system is made up of many smaller parts that, if they failed, would cause system failure. Calvin⁶ discusses, in a much more technical article, the modeling of the classic bathtub curve using a mixed Weibull distribution. The data he uses to illustrate his model are simulated.

Cherkasky⁷ employs several of the common approaches to analysis of system reliability after some extensive literature studies. He discusses some problems such as the lack of usable data and the use of the exponential distribution as a model

$$R(t) = e^{-\lambda_{nop} t_{nop} k_{nop}}$$

where nop stands for non-operating mode. The predicted reliability of the system is found by merely summing the operational readiness and mission success terms, and substituting this sum into the classical exponential formula with

$$(\lambda_t) = \{k_1(1-a)\lambda_1 t_2\} + (k_1 \lambda_1 t_3) + (k_2 \lambda_2 k_4)$$

There are diagrams in the paper which make this formula a little clearer. There is a paper written by Burns and Kapfer⁸ on the evaluation of C/MOS devices which discusses several different tests that these devices are subjected to. The tests include, for example, temperature cycling, moisture resistance, bias power and temperature step stress, and DC parameter tests. Colledge and Kimball⁹ discuss the COPPERHEAD Storage Reliability Verification. The paper presents the requirements from the Army for Material Reliability Availability Maintenance and Durability (RAM-D). It also shows why storage testing is necessary.

There is a paper written by Salt¹⁰ on the different approaches to the stress failure mechanism related to wires. The work is theoretical and tells the effects of defects such as notches on failures. There are no data from specific tests presented in this paper. Cottrell¹¹ makes us aware that there is some need for reliability studies on the nonelectronic parts of the system. Here we have no specific quality levels or the data which is available. The paper presents "no test" and "periodic tests" cases for ground and submarine situations. There are models given in each case which are based on the exponential distribution.

There are also several papers which have come from government sponsored research. One Boeing Company paper¹² presents results from 10,027 Motorola RTL electronic parts which were stored for 8 years. The measurements taken in 1975 were compared to those taken in 1967 in order to arrive at some sort of parameter drift model. Another of these papers¹³ is a handbook on electrical and electronic devices presented by the Raytheon Co. The handbook attempts to present models which describe the life cycle of a missile system. The missile material storage or non operating modes and failure rates were developed by the U.S. Army Missile Command. The operational data were extracted from existing sources such as MIL-HDBK-217B.

There is another paper written by Kobylarz and Graf¹⁴ on two specific devices, the 2N3029 silicon controlled rectifier and the (SRC)CD40074D integrated circuit. A minicomputer was used to collect the data. Several restrictions were placed on the data so only the data where the temperature of the parts was between 23^o and 25^oC were used. The object was to see if

more frequent testing leads to faster deterioration. Masterson and Miller¹⁵ present the input required and the output of a program concerned with failure distributions (the distribution used is Poisson). An example of the program used is given. Again the question is raised, how many of the failures are induced by testing?

Lastly, there are some papers on a particular type of accelerated testing, cycling. Gagnier¹⁶ prefers power cycling to temperature cycling, especially for certain types of failures, such as poor welds and bonds. He gives a very general model for this type of cycling. Wagner and Mischke¹⁷ present data for a specific wire type. They are concerned here more with stress than cycling. They use the Weibull rather than the normal or log normal distributions in prediction. Martin Marietta has a paper¹⁸ which is basically the same as Gagnier's.

The problems here are many. What type of accelerated testing gives the best results? How many times should a part be tested while it is in storage? How many failures will this testing cause? How can we make data more uniform so that it can all be used? Much further research is needed on all these questions, but perhaps we can find the beginnings of these answers in the research presented in these papers.

C. A Procedure for Modeling Storage Reliability

I. Determine Failure Mechanisms by Means of:

- a) Search of the Literature
- b) Interview With
 1. design engineers
 2. manufacturing and quality control engineers
 3. field personnel
- c) Testing Where Feasible

II. Determine Events Which Can Result in the Occurrence of the Above Failure Modes. These Will be Obtained by the Same Procedure. Along With the Identification of the Events, the Conditional Probability of Occurrence of Each Failure Mode When the Event Occurs must be determined.

This information may be obtained by detailed study of the types of failure mechanisms. Where insufficient data are available for such a study, the probability may be obtained by designed experiments run in the laboratory

making use of such things as accelerated test methods, shock and vibration tests, high temperature tests, etc. These experiments would be designed to simulate the occurrence of all possible combinations of the events leading to the various failure modes.

Suppose, for example, a missile has two failure modes and three events may happen causing the occurrence of these failure modes. The first event will cause the first failure mode to occur with probability k_1 . The second will cause the second failure mode to occur with probability k_2 . The third will cause both to occur with probability k_{12} . (The third event might be the occurrence of both events one and two or an entirely separate event.) If event three is not both of the other events, we can further say that it will cause failure mode 1 but not 2 with probability k_{10} , failure mode 2 but not 1 with probability k_{02} and neither to occur with probability k_{00} . Of course, here $k_{12} + k_{10} + k_{02} + k_{00} = 1$. If event one is the only event that can occur, the device failure rate would be $k_1\lambda_1$ and the reliability of the device could be expressed as

$$R(t) = e^{-k_1\lambda_1 t}$$

Similarly if event 2 is the only event that can occur the device failure rate is $k_2\lambda_2$ and the device reliability is

$$R(t) = e^{-k_2\lambda_2 t}$$

Thus, the failure rate would be either $k_1\lambda_1$ or $k_2\lambda_2$ in these two instances.

Now if event 3 occurs the failure rate would be $k_{12}\lambda_{12}$ for both modes, $k_{10}\lambda_{12}$ for mode 1 only, etc. Thus, the overall failure rate for mode 1 would be $k_1\lambda_1 + k_{10}\lambda_{12}$, for failure mode 2 would be $k_2\lambda_2 + k_{02}\lambda_{12}$ and for both modes $k_{12}\lambda_{12}$.

The reliability of the device would then be written as

$$R(t_1, t_2) = e^{-t_1[k_1\lambda_1 + (1-k_{00}-k_{02})\lambda_{12}] - t_2[k_2\lambda_2 + k_{01}\lambda_{12}]}, \text{ for } 0 \leq t_2 \leq t_1$$

and

$$R(t_1, t_2) = e^{-t_1 [k_1 \lambda_1 + k_{01} \lambda_{12}] - t_2 [k_2 \lambda_2 + (-k_{00} - k_{10}) \lambda_{12}]}, \text{ for } 0 \leq t_1 \leq t_2$$

Combining these two reliabilities we get the bivariate exponential distribution^{19,20}

$$R(t_1, t_2) = e^{-\lambda_1^* t_1 - \lambda_2^* t_2 - \lambda_{12}^* \max(t_1, t_2)}$$

$$\text{where } \lambda_1^* = k_1 \lambda_1 + k_{01} \lambda_{12}$$

$$\lambda_2^* = k_2 \lambda_2 + k_{02} \lambda_{12}$$

$$\lambda_{12}^* = k_{12} \lambda_{12}$$

$$t_i = \text{failure time for failure mode } i$$

If there are three or more failure mechanisms, the above formulation can be extended to the multivariate exponential distribution. For three modes this is

$$R(t_1, t_2, t_3) = \exp [-\lambda_1^* t_1 - \lambda_2^* t_2 - \lambda_3^* t_3 - \lambda_{12}^* \max(t_1, t_2) - \lambda_{23}^* \max(t_2, t_3) - \lambda_{13}^* \max(t_1, t_3)]$$

Here there are six events possible. Events 1, 2, and 3 cause failure modes 1, 2, and 3 respectively. Event 4 causes failure mode one with probability k_{40} , failure mode 2 with probability k_{04} , both modes 1 and 2 with probability k_{44} and neither with probability k_{00} . Here $k_{40} + k_{04} + k_{44} + k_{00} = 1$. Event 5 causes failure mode 1 with probability k_{500} , mode 3 with probability k_{005} , both one and 3 with probability k_{505} and neither with probability k_{000} . Again $k_{500} + k_{005} + k_{505} + k_{000} = 1$.

Event six causes failure mode 2 with probability k_{050} , mode 3 with probability k_{006} , both 2 and 3 with probability k_{066} and neither 2 nor 3 with probability k_{011} . Here $k_{050} + k_{006} + k_{066} + k_{011} = 1$.

$$\text{Then } \lambda_1^* = k_1 \lambda_1 + k_{40} \lambda_{12} + k_{500} \lambda_{13}$$

$$\lambda_2^* = k_2 \lambda_2 + k_{04} \lambda_{12} + k_{060} \lambda_{23}$$

$$\lambda_3^* = k_3 \lambda_3 + k_{005} \lambda_{13} + k_{006} \lambda_{23}$$

$$\lambda_{12}^* = k_{44} \lambda_{12}$$

$$\lambda_{13}^* = k_{505} \lambda_{13}$$

$$\lambda_{23}^* = k_{066} \lambda_{23}$$

The above expression assumes there is no event such that all three modes would occur simultaneously.

This may be generalized to n failure mechanisms, again assuming no more than two can occur simultaneously, as follows:

$$R(t_1, t_2, \dots, t_n) = \exp \left[-\sum_{i=1}^n \lambda_i^* t_i - \sum_{i < j} \lambda_{ij}^* \max(t_i, t_j) \right]$$

$$\text{where } \lambda_i^* = k_i \lambda_i$$

$$\lambda_{ij}^* = k_{ij} \lambda_{ij}$$

$$\text{and } \lambda = \sum_{i=1}^n \lambda_i^* + \sum_{i < j} \lambda_{ij}^*$$

and k_i, k_{ij} are generalizations of the k's discussed above for n=2 and 3. λ is the overall system failure rate.

D. Statistical Sampling Requirements of MIL-STD-883A

Test method 5005.2 of MIL-STD-883A specifies LTPD values for use with the sampling tables of MIL-M-38510A. These LTPD values vary from 3% to 20%

and include the following values: 3%, 5%, 7%, 10%, 15% and 20%. Specific plans corresponding to these LTPD values may be found in MIL-M-38510A.

1. Use of LTPD Plans in MIL-M-38510A

For all tests except Group C, Subgroups 4 and 5, the tests are attribute sampling plans and the values of LTPD refer to percent defective. For these percent defective plans, the LTPD is that quality, expressed as lot percent defective, which has probability of acceptance of 0.10. The tests in Group C, Subgroups 4 and 5, are life tests and, for these cases, LTPD means the device failure rate, expressed as percent of failures per 1000 hours, for which the probability of acceptance is equal to 0.10, assuming an exponential failure distribution. For these tests the values of n , the sample size, in MIL-M-38510 must be multiplied by 1000 hours to give the total test time to be used. For example, a test time of 45,000 hours may be accomplished by 45 devices tested for 1000 hours each, or 30 devices each tested for 1500 hours, or some other convenient combinations making up 45,000 test hours. In general, however, the standard indicates initial tests shall be for 1000 hours. Later, life tests may vary between 340 hours and 2000 hours. If tests are for less than 1000 hours and the stipulated number of failures occurs, additional test hours may be used, up to 1000 hours and a corresponding new acceptance number determined from Table B-1. For example, suppose the plan $n = 45$, $c = 0$ (LTPD = 5%) were used and 90 devices were put on life test for 500 hours. Note $90 \times 500 = 45,000$ hours. Now if one failure occurred during this 500 hour test, the test time could be increased up to 1000 hours, making a total test time of $90 \times 1000 = 90,000$ hours. The new acceptance number would then be found by reviewing Table B-1 to find that the test could be stopped, if no more failures occurred, at 70,000 hours. This would correspond to $70,000/90$ or 778 hours. Thus, to summarize, if 90 devices are put on test and no failures are encountered in 500 hours the lot could be accepted. If one failure occurred in 778 hours, the lot could be accepted. If two or more failures occur the lot should be rejected since the total test time required for two failures is 105,000 (which is greater than the maximum allowed test time for 90 devices of 90,000 hours.)

If a test time of more than 1000 hours is selected, with a correspondingly fewer number of devices tested, and more than the allowed number of failures occur, additional test samples may be used once to bring

the total test time up to that for which the observed number of failures are allowed. If no further failures occur the lot may be accepted, otherwise it is rejected. Thus, for example, for the 5% LTPD plan with $c = 0$ we might put 25 devices on test for 1800 hours. If one failure occurs during this 1800 hours we might test an additional 18 devices for the 1800 hours. If no more failures occur we may accept the lot, otherwise it would be rejected.

The above discussion centered around the use of Table B-I of MIL-M-38510A. This table is based on the Poisson Distribution which is a convenient approximation to the actual probability computations when the size of the lot is large relative to the sample size. For small lots the hypergeometric plans in Table B-II should be used. These plans are indexed by lot size, which runs from 10 to 200, and acceptance number which runs from 0 to 2. For each lot size, appropriate sample sizes are listed along with their AQL and LTPD values. The following table indicates the effect of lot size for the previous example, i.e., LTPD = 5% and $c = 0$. As the table indicates, the percent of the lot which is to be tested decreases as the lot size increases when the LTPD is maintained at 5%. Similar results are obtained for other LTPD values and acceptance number.

2. Derivation of LTPD Plans in Table B-I of MIL-M-38510A (based on Poisson Distribution)

LTPD is defined in this standard as that quality for which the probability of acceptance is 0.10. Thus, for each acceptance number, c , we merely go down the column of a Poisson table, such as that in the following table, until a value of 0.10 is obtained. The corresponding value of np is then read from the left margin. Thus, for example, for $c = 0$, the probability of zero defectives corresponds to a value of np of 2.30. The sample size is then obtained by dividing this value by the stipulated LTPD value. Thus, for LTPD = 5% and $c = 0$, the sample size is

$$n = \frac{2.30}{.05} = 45.$$

Some values of $np_{.10}$ for each acceptance number are listed below.

Table A

Effect of Lot Size on Sample Size for LTPD = 5% and $c = 0$

<u>N</u> <u>(Lot Size)</u>	<u>n</u> <u>(Sample Size)</u>	<u>n/N (%)</u>
10	--	100
20	--	100
30	25	83
40	32	80
50	32	64
60	32	53
80	40	50
100	40	40
120	40	33
150	40	27
160	40	25
200	40	20
Large (B-I)	45	--

Table B

Summation of Terms of Poisson's Exponential Binomial Limit
 1,000 x Probability of c or Less Occurrences of Event That
 Has Average Number of Occurrences Equal to c' or np'.

$c' \text{ or } np'$ \ c	0	1	2	3	4	5	6	7	8	9
0.02	980	1,000								
0.04	961	999	1,000							
0.06	942	998	1,000							
0.08	923	997	1,000							
0.10	905	995	1,000							
0.15	861	990	999	1,000						
0.20	819	982	999	1,000						
0.25	779	974	998	1,000						
0.30	741	963	996	1,000						
0.35	705	951	994	1,000						
0.40	670	938	992	999	1,000					
0.45	638	925	989	999	1,000					
0.50	607	910	986	998	1,000					
0.55	577	894	982	998	1,000					
0.60	549	878	977	997	1,000					
0.65	522	861	972	996	999	1,000				
0.70	497	844	966	994	999	1,000				
0.75	472	827	959	993	999	1,000				
0.80	449	809	953	991	999	1,000				
0.85	427	791	945	989	998	1,000				
0.90	407	772	937	987	998	1,000				
0.95	387	754	929	984	997	1,000				
1.00	368	736	920	981	996	999	1,000			
1.1	333	699	900	974	995	999	1,000			
1.2	301	663	879	966	992	998	1,000			
1.3	273	627	857	957	989	998	1,000			
1.4	247	592	833	946	986	997	999	1,000		
1.5	223	558	809	934	981	996	999	1,000		
1.6	202	525	783	921	976	994	999	1,000		
1.7	183	493	757	907	970	992	998	1,000		
1.8	165	463	731	891	964	990	997	999	1,000	
1.9	150	434	704	875	956	987	997	999	1,000	
2.0	135	406	677	857	947	983	995	999	1,000	

Table B

Summation of Terms of Poisson's Exponential Binomial Limit. (continued)

c c' or n/p'	0	1	2	3	4	5	6	7	8	9
2.2	111	355	623	819	928	975	993	998	1,000	
2.4	091	308	570	779	904	964	988	997	999	1,000
2.6	074	267	518	736	877	951	983	995	999	1,000
2.8	061	231	469	692	848	935	976	992	998	999
3.0	050	199	423	647	815	916	966	988	996	999
3.2	041	171	380	603	781	895	955	983	994	998
3.4	033	147	310	558	744	871	942	977	992	997
3.6	027	126	303	515	706	844	927	969	988	996
3.8	022	107	269	473	658	816	909	960	984	994
4.0	018	092	238	433	629	785	889	949	979	992
4.2	015	078	210	395	590	753	867	936	972	989
4.4	012	066	185	359	551	720	844	921	964	985
4.6	010	056	163	326	513	686	818	905	955	980
4.8	008	048	143	294	476	651	791	887	944	975
5.0	007	040	125	265	440	616	762	867	932	968
5.2	006	034	109	238	406	581	732	845	918	960
5.4	005	029	095	213	373	546	702	822	903	951
5.6	004	024	082	191	342	512	670	797	886	941
5.8	003	021	072	170	313	478	638	771	867	929
6.0	002	017	062	151	285	446	606	744	847	916
	10	11	12	13	14	15	16			
2.8	1,000									
3.0	1,000									
3.2	1,000									
3.4	999	1,000								
3.6	999	1,000								
3.8	998	999	1,000							
4.0	997	999	1,000							
4.2	996	999	1,000							
4.4	994	998	999	1,000						
4.6	992	997	999	1,000						
4.8	990	996	999	1,000						
5.0	986	995	998	999	1,000					
5.2	982	993	997	999	1,000					
5.4	977	990	996	999	1,000					
5.6	972	988	995	998	999	1,000				
5.8	965	984	993	997	999	1,000				
6.0	957	980	991	996	999	999	1,000			

Table B

Summation of Terms of Poisson's Exponential Binomial Limit. (continued)

c c' or np'	0	1	2	3	4	5	6	7	8	9
6.2	002	015	054	134	259	414	574	716	826	902
6.4	002	012	046	119	235	384	542	657	803	886
6.6	001	010	040	105	213	355	511	658	780	869
6.8	001	009	034	093	192	327	480	628	755	850
7.0	001	007	030	082	173	301	450	599	729	830
7.2	001	006	025	072	156	276	420	569	703	819
7.4	001	005	022	063	140	253	392	539	676	788
7.6	001	004	019	055	125	231	365	510	648	765
7.8	000	004	016	048	112	210	338	481	620	741
8.0	000	003	014	042	100	191	313	453	593	717
8.5	000	002	009	030	074	150	256	386	523	653
9.0	000	001	006	021	055	116	207	324	456	587
9.5	000	001	004	015	040	089	165	269	392	522
10.0	000	000	003	010	029	067	130	220	333	458
	10	11	12	13	14	15	16	17	18	19
6.2	949	975	989	995	998	999	1,000			
6.4	939	969	986	994	997	999	1,000			
6.6	927	963	982	992	997	999	1,000			
6.8	915	955	978	990	996	998	999	1,000		
7.0	901	947	973	987	994	998	999	1,000		
7.2	887	937	967	984	993	997	999	999	1,000	
7.4	871	926	951	980	991	996	998	999	1,000	
7.6	854	915	954	976	989	995	998	999	1,000	
7.8	835	902	945	971	986	993	997	999	1,000	
8.0	816	888	936	966	983	992	996	998	999	1,000
8.5	763	849	909	949	973	986	993	997	999	999
9.0	706	803	876	926	959	978	989	995	998	999
9.5	645	752	836	898	940	967	982	991	996	998
10.0	583	697	792	864	917	951	973	986	993	997
	20	21	22							
8.5	1,000									
9.0	1,000									
9.5	999	1,000								
10.0	998	999	1,000							

Table B

Summation of Terms of Poisson's Exponential Binomial Limit. (continued)

c or np'	0	1	2	3	4	5	6	7	8	9
10.5	000	000	002	007	021	050	102	179	279	397
11.0	000	000	001	005	015	038	079	143	232	341
11.5	000	000	001	003	011	028	060	114	191	289
12.0	000	000	001	002	008	020	046	090	155	242
12.5	000	000	000	002	005	015	035	070	125	201
13.0	000	000	000	001	004	011	025	054	100	166
13.5	000	000	000	001	003	008	019	041	079	135
14.0	000	000	000	000	002	006	014	032	062	109
14.5	000	000	000	000	001	004	010	023	048	088
15.0	000	000	000	000	001	003	008	018	037	070
	10	11	12	13	14	15	16	17	18	19
10.5	521	639	742	825	888	932	960	978	988	994
11.0	460	579	689	781	854	907	944	968	982	991
11.5	402	520	633	733	815	878	924	954	974	986
12.0	347	462	576	682	772	844	899	937	963	979
12.5	297	406	519	628	725	806	869	916	948	969
13.0	252	353	463	573	675	764	835	890	930	957
13.5	211	304	409	516	623	718	795	861	908	942
14.0	176	266	358	464	570	669	756	827	883	923
14.5	145	229	311	413	518	619	711	790	853	901
15.0	118	185	268	363	466	568	664	749	819	875
	20	21	22	23	24	25	26	27	28	29
10.5	997	999	999	1,000						
11.0	995	998	999	1,000						
11.5	992	996	998	999	1,000					
12.0	988	994	997	999	999	1,000				
12.5	983	991	995	998	999	999	1,000			
13.0	975	986	992	996	998	999	1,000			
13.5	965	980	989	994	997	998	999	1,000		
14.0	952	971	983	991	995	997	999	999	1,000	
14.5	936	960	976	986	992	996	998	999	999	1,000
15.0	917	947	967	981	989	994	997	998	999	1,000

Table B

Summation of Terms of Poisson's Exponential Binomial Limit. (continued)

c c' or np'	4	5	6	7	8	9	10	11	12	13
16	000	001	001	010	022	043	077	127	193	275
17	000	001	002	003	013	026	049	085	135	201
18	000	000	001	003	007	015	030	055	092	143
19	000	000	001	002	004	009	018	035	061	098
20	000	000	000	001	002	005	011	021	039	066
21	000	000	000	000	001	003	006	013	025	043
22	000	000	000	000	001	002	004	008	015	028
23	000	000	000	000	000	001	002	004	009	017
24	000	000	000	000	000	000	001	003	005	011
25	000	000	000	000	000	000	001	001	003	006
	14	15	16	17	18	19	20	21	22	23
16	368	467	566	659	742	812	868	911	942	963
17	281	371	468	564	655	736	805	861	905	937
18	208	287	375	469	562	651	731	799	855	899
19	150	215	292	378	469	561	647	725	793	849
20	105	157	221	297	381	470	559	644	721	787
21	072	111	163	227	302	384	471	558	640	716
22	048	077	117	169	232	306	387	472	556	637
23	031	052	082	123	175	238	310	389	472	555
24	020	034	056	097	128	180	243	314	392	473
25	012	022	038	060	092	134	185	247	318	394
	24	25	26	27	28	29	30	31	32	33
16	976	987	993	996	998	999	999	1,000		
17	959	975	985	991	995	997	999	999	1,000	
18	932	955	972	983	990	994	997	998	999	1,000
19	893	927	951	969	980	988	993	996	998	999
20	843	888	922	948	966	978	987	992	995	997
21	782	838	883	917	944	963	976	985	991	994
22	712	777	832	877	913	940	959	973	983	989
23	635	708	772	827	873	908	936	956	971	981
24	554	632	704	768	823	868	904	932	953	969
25	472	553	629	700	763	818	863	900	929	950
	34	35	36	37	38	39	40	41	42	43
19	999	1,000								
20	999	999	1,000							
21	997	998	999	999	1,000					
22	994	996	998	999	999	1,000				
23	988	993	996	997	999	999	1,000			
24	979	987	992	995	997	998	999	999	1,000	
25	966	978	985	991	994	997	998	999	999	1,000

Table C

Values of np for LTPD Plans

<u>c</u>	<u>np_{.10}</u>	<u>c</u>	<u>np_{.10}</u>	<u>c</u>	<u>np_{.10}</u>
0	2.30	5	9.28	10	15.41
1	3.89	6	10.53	11	16.60
2	5.32	7	11.77	12	17.78
3	6.68	8	12.99	13	18.96
4	7.99	9	14.21	14	20.13
				15	21.29

The operating characteristic of a sampling plan is the probability of lot acceptance when the quality (in terms of percent defective) is equal to any stipulated value, p . The operating characteristic of a sampling plan is often expressed as a curve on which the probability of lot acceptance is plotted against the percent defective. Such curves are commonly referred to as OC-Curves. Some examples of OC-Curves for some of the plans in MIL-M-38570 are included in this report.

3. Procedure for Computation of Points on the Operating Characteristic Curve for a Sampling Plan Using the Poisson Distribution (Table B-1 of MIL-M-38510A)

1. Select value of p (percent defective) desired.
2. Multiply each value of p (in decimal form) by the sample size.
3. Enter a Poisson Table to determine the probability of c or fewer defectives in the sample. This is the probability of acceptance of a lot which has p -percent defective using the sampling plan defined by the values n and c , where n is the sample size and c is the maximum number of defectives permitting lot acceptance.

The OC-Curves for 5, 10 and 15% LTPD plans using acceptance numbers $c = 0, 1, 2, 3$ are shown below. For these plans, it will be noted that all plans with the same LTPD go through the ($P_a = 0.10$, LTPD) point on that graph. The probability of lot acceptance for other values of p varies, with most variation occurring when the quality is significantly better than the specified LTPD. These plans show the effect of increasing the acceptance number on the probability of acceptance of a lot.

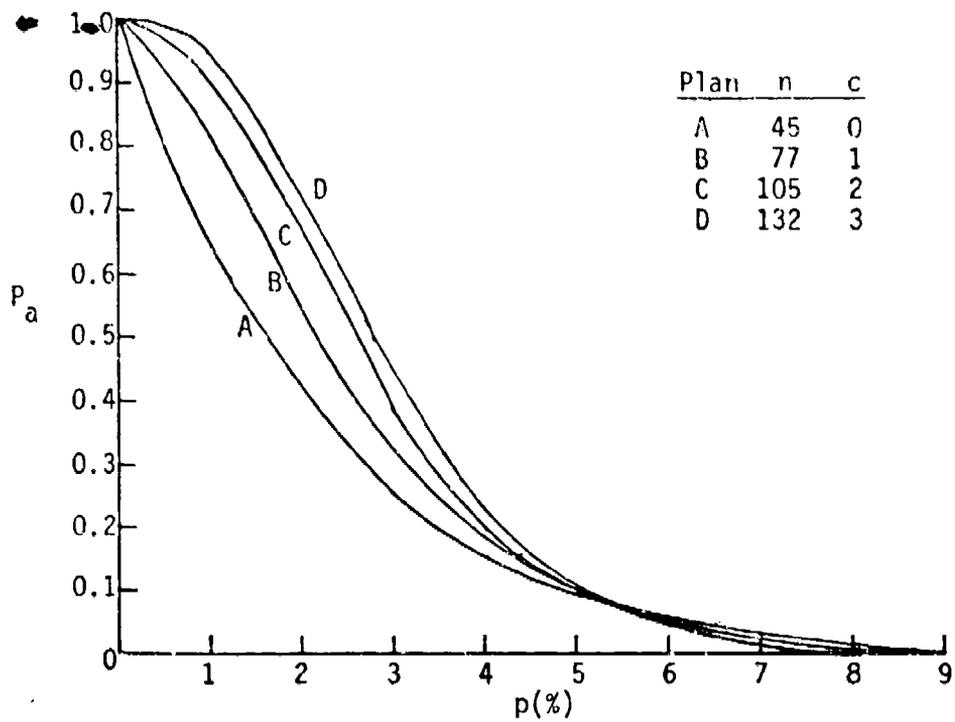


Figure 10-1. Operating Characteristic Curves for 5% LTPD Plans.

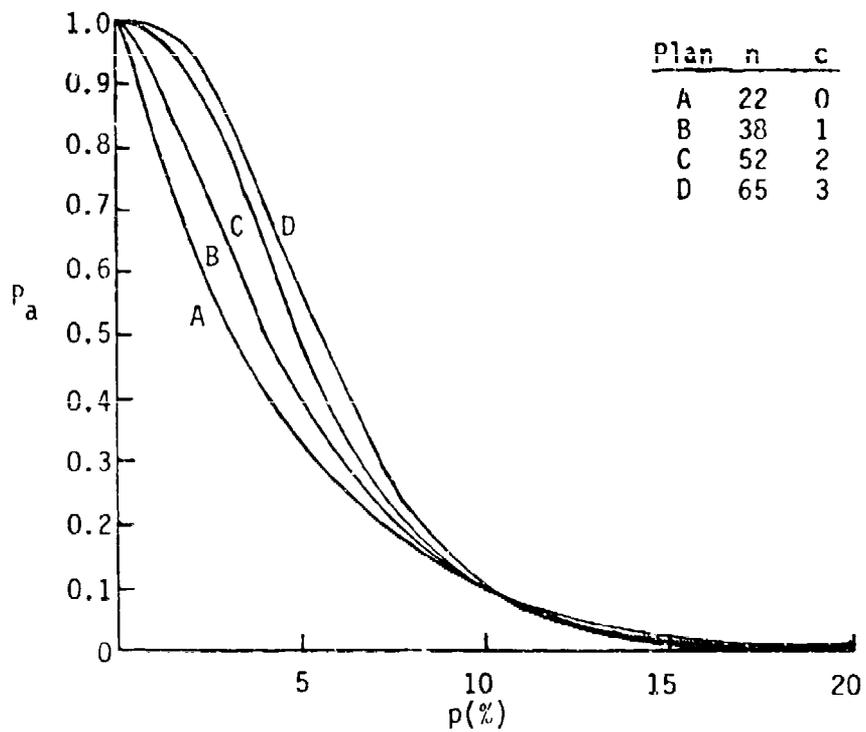


Figure 10-2. Operating Characteristic Curves for 10% LTPD Plans.

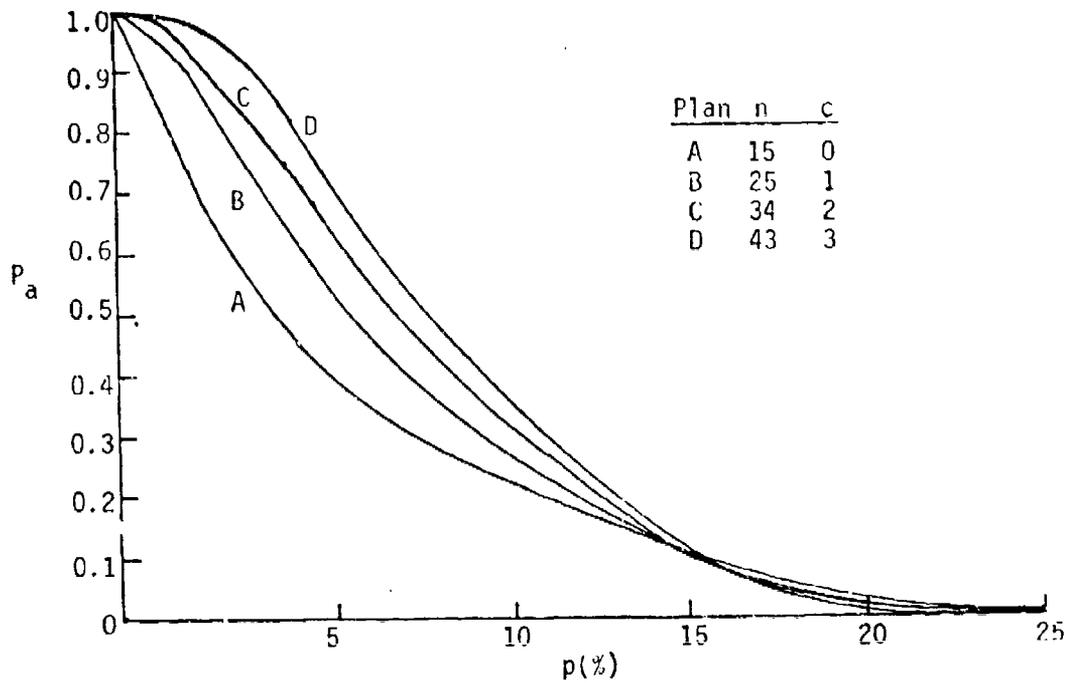


Figure 10-3. Operating Characteristic Curves for 15% LTPD Plans.

The following OC-Curves show, for $c = 0$ and $c = 2$, the effect of changing the LTPD on the probability of acceptance. It should be noted that this change results in corresponding changes throughout the quality range.

4. Hypergeometric Sampling Plans

These plans, found in Table B-II of MIL-M-38510A are, as previously discussed, more accurate for small lots. The plans based on the Poisson distribution (Table B-I) assume a large lot relative to the sample size. If this assumption is not met, the probability of acceptance obtained using the Poisson distribution may be somewhat in error.

The table gives values of AQL and LTPD. AQL is defined as that lot percent defective for which the probability of c or less defectives in a sample of size n is equal to or greater than 0.95. The LTPD is defined as before. This gives the user two points on the operating characteristic curve.

The hypergeometric distribution is as follows:

$$P(x) = \frac{\frac{r!}{x!(n-x)!} \frac{(N-r)!}{(n-x)!(N-r-n+x)!}}{\frac{N!}{n!(N-n)!}}$$

- where N = lot size
 n = sample size
 r = number of defectives in the lot
 x = number of defectives in the sample
 $P(x)$ = probability of exactly x defectives in the sample
 $p = \frac{r}{N}$ = fraction defective in the lot

Thus, for $N = 20$, $n = 4$, $c = 1$, the AQL and LTPD values are obtained, respectively, by solving the following for r and then p .

$$0.95 = \sum_{x=0}^1 \frac{\frac{r!}{x!(4-x)!} \frac{(20-r)!}{(4-x)!(16-r+x)!}}{\frac{20!}{4! 16!}}$$

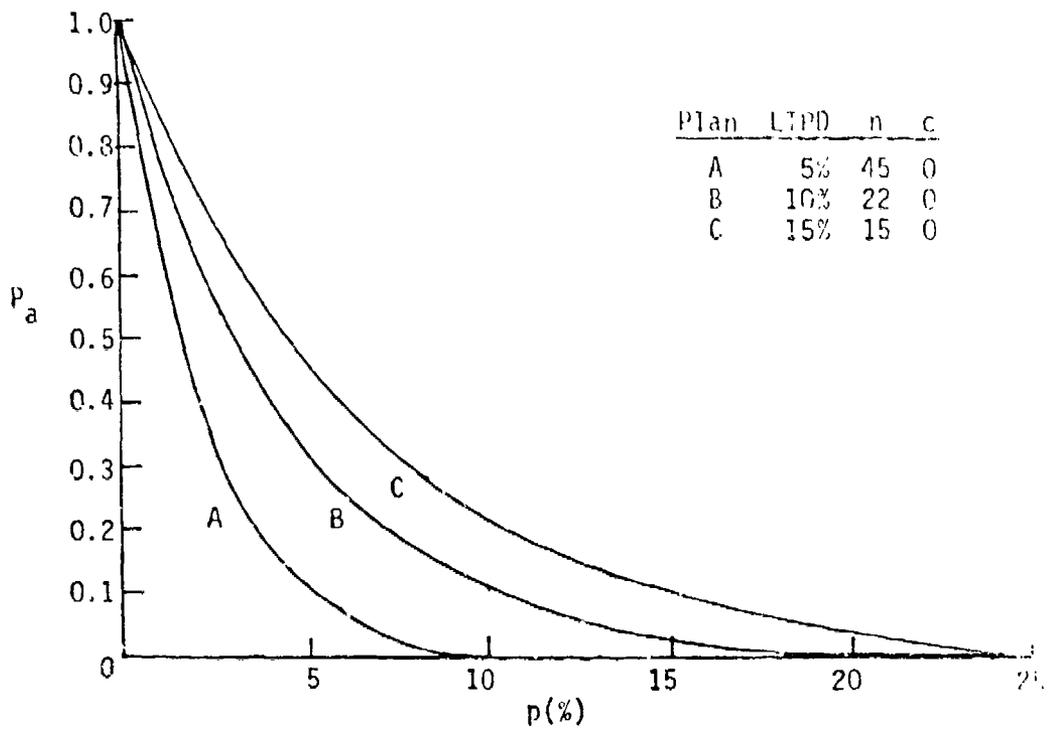


Figure 10-4. Operating Characteristic Curves for $c = 0$ Plans.

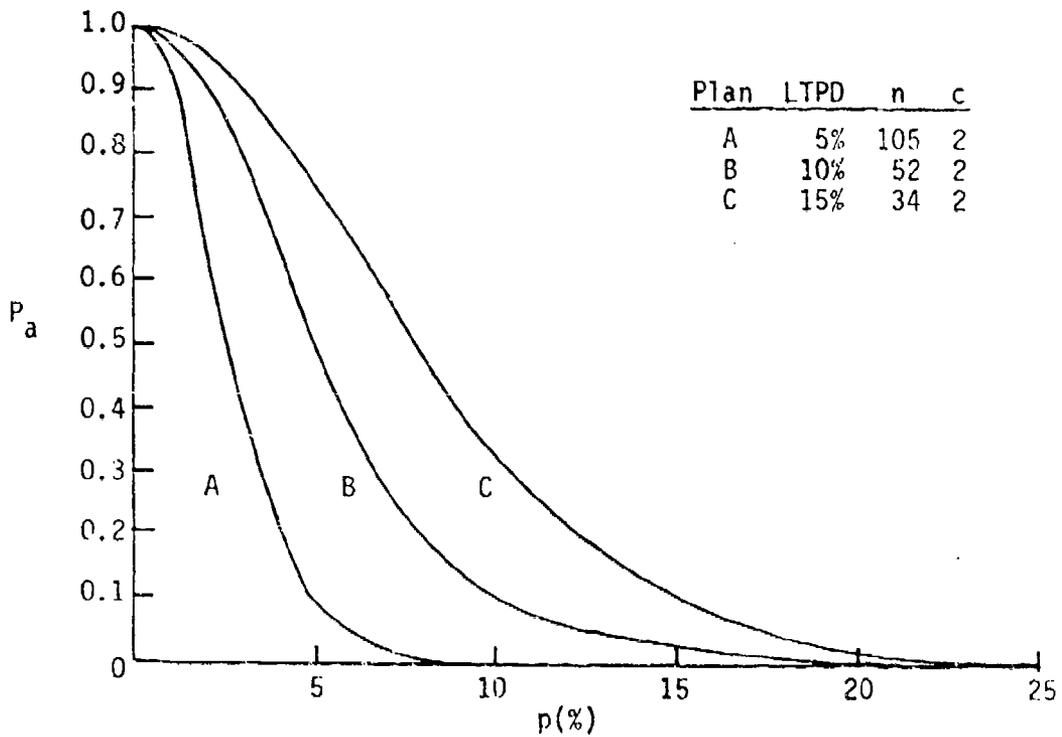


Figure 10-5. Operating Characteristic Curves for $c = 2$ Plans.

and

$$0.10 = \sum_{x=0}^1 \frac{r! (20-r)!}{x!(4-x)! (4-x)!(16-r+x)!} \frac{20!}{4! 16!}$$

The first equation gives an r of 2.40, which gives a value of p of $\frac{2.40}{20} = 0.12$ or 12%. The second equation gives an r of 13.2, which gives a value of p of $\frac{13.2}{20} = 0.66$ or 66%. Thus, AQL = 12% and LTPD = 66% for this plan as shown in Table B-II of MIL-M-38510A.

The operating characteristic curves for the sampling plans given in Table B-II may be plotted by using the two points tabulated in the table. If another point is desired, the hypergeometric expression may be equated to some other convenient value of P_a . For example, we might use $P_a = 0.50$. For the plan used in the above example ($N = 20$, $n = 4$, $c = 1$) this leads to

$$0.50 = \sum_{x=0}^1 \frac{r! (20-r)!}{x!(4-x)! (4-x)!(16-r+x)!} \frac{20!}{4! 16!}$$

Solution of this equation is $r = 5.53$, which yields a value of p of $\frac{5.53}{20} = 0.28$ or 28%. Thus, a 28% defective lot has a 50% chance of being accepted using this sampling plan. The OC-Curve for this plan is therefore shown in the following illustration.

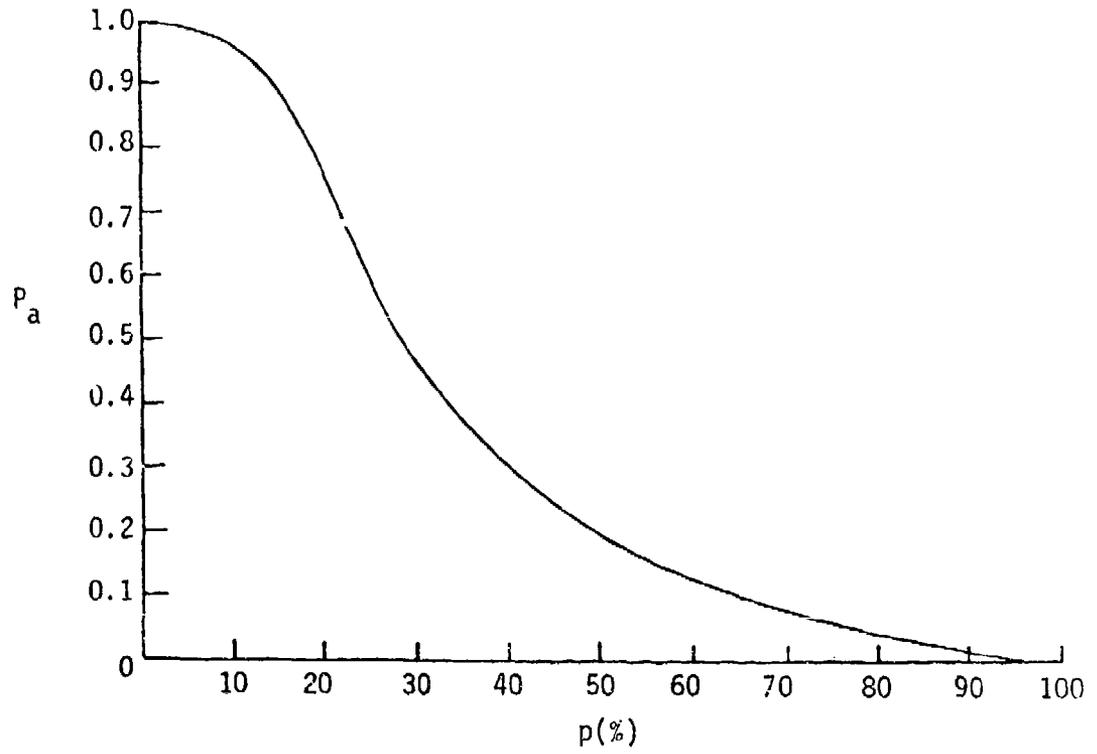


Figure 10-6. Operating Characteristic Curve for $N = 20$, $n = 4$, $c = 1$ Hypergeometric Plan.

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XI. CONCLUSIONS AND RECOMMENDATIONS

1. The tactical missiles used by the Army will normally employ state-of-the-art electronic components as of the time the missile design is frozen. Currently deployed missile systems therefore involve a range of electronic device technologies. It is impossible to compare failure rates across the full inventory of Army missiles due to the wide range of device technologies employed. There will probably never be a significant statistical basis for determining missile reliability using storage failure rate data obtained from old systems since the new technologies will generally involve very different structures. Consequently there is a need for the continued evaluation of potential storage failure mechanisms based on advancing current technologies.
2. The most important environmental forcing functions, or stresses, in storage are mechanical, chemical and low thermal. Mechanical stresses occur due to thermal-mechanical interactions and residual stresses. Chemical stresses result from contaminants such as residual process chemicals and environmental gases which are introduced through improper or failed seals. Although purely thermal stresses have much less importance in storage than operating environments, certain low temperature reaction rates and diffusion processes are temperature dependent.
3. The synergism of the three primary storage stresses is critical. Any one of the three acting alone may not be particularly damaging but the combined effect of two or three forcing functions acting together is likely to cause device failures.
4. Environmental extremes for Army missiles in storage have involved temperatures of -50°C to $+75^{\circ}\text{C}$, diurnal cycling of 70°C , 100 percent relative humidity, direct sea spray, industrial pollutants, some mechanical shock and fungus.
5. The failure mechanisms of greatest importance in storage have been identified as those related to various marginal manufacturing mistakes, corrosion processes and mechanical fracture. Electrical or potential current induced degradation processes should not be important in the storage environment. Moisture within a package is probably the most important

factor for both corrosion and mechanically induced failures in storage. Chemicals including moisture trapped within a package due to improper cleaning or because of evolution from materials such as polymers are a critical concern for long-term reliability. The package seal is also critical for keeping out atmospheric contaminants. Thermal-mechanical stresses aided by chemical agents will cause crack propagation in seals, passivation layers, bonds, metallization layers and the silicon chip.

6. New manufacturing methods such as the Tape Automated Bonding technology should be continually evaluated to determine if there are potential storage failure mechanisms. For example, are there detrimental effects in a storage environment from probable impurities introduced during bump plating and bonding operations?

7. The presence of defects such as impurities, dislocations, microcracks, interfacial faults and grain boundaries in the materials of a microcircuit structure can result in failure due to low temperature atomic diffusion processes.

8. The design of circuit configurations along with the choice of materials for electronic systems placed in storage should be based on a sound understanding of potential degradation processes in expected storage environments.

9. Particulate matter is one of the dominant concerns as a storage failure mechanism.

10. The hermeticity of microelectronic packages is an important concern for long-term storage conditions. The screen test for determining the effectiveness or hermeticity of the package seals includes a fine leak rate test. The maximum allowable leak rate specified for this test should be lowered to 10^{-10} atm cm³ sec⁻¹ for devices that are expected to be stored because of the exchange of gases between the initial package ambient and the external storage environment for packages with a finite size leak.

11. All microcircuit packages should be vacuum baked at 150°C for at least 4 hours and sealed in dry nitrogen without ever being exposed to moisture containing gases such as air. The moisture content of the nitrogen sealing chamber should be less than 100 ppm.

12. Significant improvements are needed in the measurement technology for moisture and other gases in microcircuit packages. Current methods are too

expensive and complicated while providing insufficient sensitivity and wide variations in numerical values for supposedly identical gas contents.

13. The fields across a thin gate oxide in MOS devices can often approach the dielectric strength of the oxide. However, because of various factors that are not easily controlled the breakdown voltages have a range of values. Consequently, any application of potentials to the gate electrode can be a possible cause of oxide breakdown, particularly when static charging is not avoided or if there are voltage transients present in ground test equipment.

14. The use of plastics introduces high risks of differential expansion problems which result in mechanical damage such as pulling apart leads.

15. Whenever polymeric materials are employed for die attach within hybrid microcircuit packages, they must be proved compatible with all enclosed electronic materials. No chlorine or other halogen containing materials should be sealed in any circuitry components. Polymers used should be simple hydrocarbons or compounds of carbon, hydrogen and oxygen. Nitrogen containing polymers should be considered with skepticism. The responsibility for proof of compatibility should be with the manufacturer for specific epoxies and circuit element combinations.

16. Missiles placed in storage should never contain electronic parts employing polymers for package seals. Polymers will transmit moisture and other gases.

17. Screening and accelerated testing procedures of Army missiles must have steps determined by potential storage failure processes. There is doubt that the screening sequence contained in MIL-STD-883A is fully appropriate to the storage environment.

18. There is widespread controversy about the optimum number of cycles in a temperature cycling screen test. Opinions vary from 25-300 cycles for effective screening but the use of only 10 cycles is not considered to be of any value. Results of the Rockwell International screen test program have not resolved this question.

19. Thermal shock should never be used as a screen test stress for hermetic devices placed in stored missile systems.

20. The metallurgical consequences of an upper limit of 150° vs. 125°C for temperature cycling and stabilization bakes with regard to solders should be investigated.

21. High temperature burn-in is a relatively effective screen for failure modes having high activation energies. For oxide defects the failure mode has a much lower activation energy. The high temperature burn-in is then not particularly useful. An over-voltage stress should be investigated for screening MOS devices for oxide defects.

22. Complex MOS/LSI microcircuits require a different approach to reliability than mere application of MIL-STD-883 screens. Attention to good quality control at the process level and the development of more appropriate screens are essential to improved reliability. In addition, the use of a specially designed process evaluation circuit providing device material parameters at the wafer level should be required for high reliability devices. This circuit should also be useful for developing and evaluating the effectiveness of screens.

23. The philosophy necessary for developing meaningful screen testing parameters is to concentrate on determining the stress-duration levels required to reveal well defined device faults. The capability is therefore needed for fabricating devices with deliberate defects of desired type, severity and number.

24. There is no scientific basis for the widely practiced time-temperature superposition and extrapolation methods used in accelerated aging studies. The only justification for particular test procedures is experience and good judgment. This judgment requires an understanding of the general processes involved and their interrelations. High temperature aging tests of operating microcircuits should not be expected to provide meaningful lifetime predictions for devices placed in storage environments.

25. Only general environmental data are currently available for the temperature, environmental gases, vibration, etc. expected in storage. There is need for specific information concerning the interior of a missile in storage in order to make judgments concerning future reliability factors. The chemical factors associated with moisture, evolved gases and fungus need to be developed at four levels:

1. Within the storage structure (iqloo, shed, etc.)
2. Within the missile container
3. Within the missile electronic system compartment
4. Within individual component packages.

A measurement program should be established so that actual data will be available concerning these factors.

26. The effectiveness of desiccant materials used within Army missiles should be evaluated. This topic was not pursued during this program but questions were raised by several organizations.

27. The various types of missile storage containers should be evaluated to determine how well they protect missiles from storage environments most critical to the electronic systems.

28. Procedures should be in effect to close the loop concerning the detailed analysis of parts failing in service and manufacturing parameters. Failures in field environments are generally more severe than indicated by initial predictions. Feed-back from service failures should be available to guide design decisions of future systems.

29. Future efforts in storage reliability should be directed towards determining the response of materials in microcircuit structures to the storage environmental forcing functions. This will require the application, and in some cases, the development of advanced measurement techniques in order to determine chemical, mechanical and thermal threshold levels for device degradation processes. Particular emphasis should be placed on quantitative evaluations of moisture induced failure processes so that contaminant requirements can be established. The basic threshold levels for degradation have to be established before effective screens and accelerated test methods can be designed.

30. Measurements of permeabilities, diffusion coefficients, and solubilities of water in representative polymers should be made so that good data are available and effects of temperature, pressure, mechanical strain, previous sorption, and synergism of two or more penetrants be understood. Data on thermal expansion, glass transitions, and viscoelastic responses of polymer encapsulants and adhesives are too meager for design of circuit systems. Measurements are needed here.

31. Age sensitive materials used in missile systems must be well characterized. Missile storage reliability is determined by the stability of the materials used to fabricate individual parts within the system while exposed to the storage environment of a tactical missile. There is a strong need for compiling material degradation data from the technical literature, directed experiments and theoretical calculations.

32. The purchase of Army missile parts should be controlled by the engineering staff. Government facilities having the strongest reliability performance usually have their engineering staffs closely involved and responsible for part purchasing, monitoring and testing decisions. A strong parts reliability program should be maintained at MIRADCOM as an essential aspect in the development of new missile systems.

APPENDICES

APPENDIX A

LIST OF TECHNICAL CONTACTS

A large number of individuals were contacted during the course of these investigations. Most of these involved personal visits, but some discussions were conducted over the telephone. There were additional individuals we would like to have visited because of their special expertise, but time and logistical considerations made those visits impossible. We are indebted to the individuals listed below for sharing with us their experiences and judgments.

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APPENDIX B
INDUSTRY SURVEY

An industry survey on missile system electronics related to storage reliability was conducted by using a questionnaire prepared by personnel at MIRADCOM. A copy of the questionnaire used is included in this appendix and an analysis of the responses follows below, with the items numbered according to the numbers on the questionnaire. The future was taken as 1980.

1. The predominant circuit type which is currently digital in most of the industries surveyed should see an increase in percent usage with a corresponding decrease in analog circuitry. RF circuits are a small percentage of the total except for those industries that have a significant need for microwave circuits. Overall there is practically no change in the percentage for RF circuits and a slight decrease for those industries which produce or use microwave circuits.
2. There was practically universal agreement that the predominant module design, present and future, is planar PC board soldered.
3. (a) Either TO Cans (single chip) or Dual In Line Packages (single chip) are used by most industries, one or the other. No significant change is expected in the future.
(b) The use of thick film multi-chip hybrids predominates over the thin film hybrids by a factor of 3 indicating the large use of thick film interconnections. Very little change is expected in the future except for isolated cases where the precision required for microwave circuits forces the choice toward thin-film circuits.
(c) Current use of SSI monolithic devices is expected to decrease slightly in the future in favor of MSI devices (>15 equivalent gates) and LSI devices (75-1000 gates).

4. Dual In Line hybrid packages and Flat Packs appear to be the industry trend with a major usage of vertical sidewall packages. Flat packs are used almost exclusively by MINUTEMAN.
5. Hermetic sealing is the predominant environment protection method used for microelectronic circuits and semiconductors with the majority of industries testing for minimum leaks of the order of 10^{-7} atm cc/sec.
6. Although the responses in this section reflect the nature of the industry and no semiconductor manufacturers were included, one can conclude that the predominant monolithic technology is now bipolar-T²L with the future seeing more use of MOS and C/MOS technology. It is interesting that the majority of the responses indicated a future use of CCD technology (5-10%).
7. Chip attachment to the substrate including chip resistors and chip capacitors is over 90% by epoxy bonding techniques. Eutectic bonding is used for high power devices and a few companies use soldering techniques for chip capacitors. The trend appears to be toward more epoxy bonding presumably because of the need to repair hybrids. Future use of beam lead chips is indicated, if they become more generally available.
8. Gold-to-gold accounts for approximately 6% for the present and is expected to increase to an average of 8% in the future. This increase depends to some extent on the use of gold metallization on silicon chips. The use of gold wires to aluminum metallization presently at the 40% level is expected to decrease to about 30% in the future. A surprisingly large number of industries use aluminum-to-aluminum connections with percentages being over 50% for the present and the future. The tradeoff here is apparently the problems with the gold-aluminum intermetallics versus the risks of aluminum corrosion. One company mentioned the need for developing other metallization contact schemes, e.g., copper-to-copper.

9. Oxide passivation is used for most chips with an increased use of silicon nitride in combination with the thermal oxide expected. Glassivation is used over the entire chip instead of polymer coatings.
10. Almost universal use of MIL-STD-883, Class B screens was noted with a few exceptions on the number of temperature cycles (25-50 cycles), the substitution of 100% mechanical shock for constant acceleration, the upper temperature limit for stabilization bake and temperature cycling (125°C instead of 150°C) and the use of 100% non-destructive bond pull tests.
11. A sampling of the remarks on recommended changes to screening methods includes the following statements:
 - "Add loose particle detection."
 - "Increase temperature cycling to 25 cycles."
 - "Special applications require special screening techniques."
12. The answers to the question on the adequacy and appropriateness of the MIL-STD-883 screening methods for long-term storage varied from a qualified "yes" when there was little knowledge about long-term storage to a "don't know" and "no way." In one case a suggestion was made that each materials system requires careful evaluation for long-term storage.
13. When used by the particular industry the screening methods for semiconductor devices generally followed MIL-STD-750.
14. There were few recommendations on changes to semiconductor device screening methods.
15. Answers to this question were similar to those of item 12.
16. One could not prove that the storage requirements included in development contracts were met but, in most cases, compliance was based upon the component screening methods used.
17. Plastics (epoxies) are used for die attachment, etc. Some information might be available on their use in long-term storage.



DEPARTMENT OF THE ARMY
HEADQUARTERS UNITED STATES ARMY MISSILE COMMAND
REDSTONE ARSENAL, ALABAMA 35809

QUESTIONNAIRE - Missile System Electronics
An Industry Survey for Storage Reliability

Please Indicate Future Time Frame (_____)

1. Predominant Type Circuit

- | | |
|--------------------|-----------------|
| a. Analog | Present _____ % |
| | Future _____ % |
| b. Digital | Present _____ % |
| | Future _____ % |
| c. RF | Present _____ % |
| | Future _____ % |
| d. <u>REMARKS:</u> | |

2. Predominant Module Design

- | | |
|-----------------------------|-----------------|
| a. Planar PC Board Soldered | Present _____ % |
| | Future _____ % |
| b. Planar PC Board Welded | Present _____ % |
| | Future _____ % |
| c. Cordwood Soldered | Present _____ % |
| | Future _____ % |
| d. Cordwood Welded | Present _____ % |
| | Future _____ % |
| e. <u>REMARKS:</u> | |

3. Discrete Components Used

- a. Semiconductor Devices

(1) Flatpack (Single Chip)	Present _____ %
	Future _____ %
(2) TO Can (Single Chip)	Present _____ %
	Future _____ %
(3) Dual In Line Package (Single Chip)	Present _____ %
	Future _____ %

b. Hybrids Devices

(1) Thick Film Hybrid (Multichip)	Present _____ %
	Future _____ %
(2) Thin Film Hybrid (Multichip)	Present _____ %
	Future _____ %

c. Monolithic Devices

(1) SSI Devices (Single Chip)	Present _____ %
	Future _____ %
(2) MSI Devices (Single Chip)	Present _____ %
	Future _____ %
(3) LSI Devices (Single Chip)	Present _____ %
	Future _____ %

d. REMARKS:

4. Predominant Hybrid Module Size

a. TO Cans	Present _____ %
	Future _____ %
b. Dual In Line Packages (Ceramic)	Present _____ %
	Future _____ %
" " " " (Vertical Sidewall)	Present _____ %
	Future _____ %
" " " " (Platform)	Present _____ %
	Future _____ %
c. Flat Packs	Present _____ %
	Future _____ %

d. REMARKS:

5. Predominant Environment Protection for Microelectronic Circuits and Semiconductors

- | | |
|---|-----------------|
| a. Hermetic Sealing (1 x 10 ⁻⁶) | Present _____ % |
| | Future _____ % |
| " " (1 x 10 ⁻⁸) | Present _____ % |
| | Future _____ % |
| b. Conformal Coating | Present _____ % |
| | Future _____ % |
| c. Semi-Rigid Encapsulation | Present _____ % |
| | Future _____ % |
| d. Rigid Encapsulation | Present _____ % |
| | Future _____ % |
| e. Foam Encapsulation | Present _____ % |
| | Future _____ % |
| f. Other | Present _____ % |
| | Future _____ % |
| g. <u>REMARKS:</u> | |

6. Monolithic Technology

- | | |
|---------------------|-----------------|
| a. Bipolar | Present _____ % |
| | Future _____ % |
| b. MOS | Present _____ % |
| | Future _____ % |
| c. C-MOS | Present _____ % |
| | Future _____ % |
| d. T ² L | Present _____ % |
| | Future _____ % |
| e. I ² L | Present _____ % |
| | Future _____ % |
| f. CCD | Present _____ % |
| | Future _____ % |
| g. <u>REMARKS:</u> | |

7. Chip - Attachment Techniques to Substrate

a. Chip and Wire

(1) Epoxy Bonding

Present _____ %
Future _____ %

(2) Eutectic Bonding

Present _____ %
Future _____ %

b. Beam Lead Chips

Present _____ %
Future _____ %

c. Flip Chips (Solder Bump)

Present _____ %
Future _____ %

d. LID's

Present _____ %
Future _____ %

e. Chip Resistors (Tantalum)

(1) Eutectic

Present _____ %
Future _____ %

(2) Epoxy

Present _____ %
Future _____ %

f. Chip Capacitors

(1) Soldered

Present _____ %
Future _____ %

(2) Epoxy

Present _____ %
Future _____ %

g. Other

Present _____ %
Future _____ %

h. REMARKS:

8. Chip Metallization Interconnection Contact Techniques

a. Gold to Gold

Present _____ %
Future _____ %

- b. Aluminum to Gold Present _____ %
Future _____ %
- c. Aluminum to Aluminum Present _____ %
Future _____ %
- d. Other Present _____ %
Future _____ %
- e. REMARKS:

9. Chip Surface Protection Techniques

- a. Oxide Passivation Present _____ %
Future _____ %
- b. Silicon Nitride Passivation Present _____ %
Future _____ %
- c. Glassivation Present _____ %
Future _____ %
- d. Dielectrics Present _____ %
Future _____ %
- e. Polymer Films Present _____ %
Future _____ %
- f. Uncoated Devices Present _____ %
Future _____ %
- g. REMARKS:

10. Screen Methods for Microelectronic and Hybrids Microelectronics (MIL-STD-883)
(If screening methods differs from MIL-STD-883, Class B, please discuss under remarks)

- a. Internal PreCap Visual (Microelectronic Devices, Per Method 2010, Test Condition B, Hybrid Microelectronic Devices, Per Method 2017) YES NO

REMARKS:

b. Stabilization Bake (Per Method 1008, 24 hrs Minimum, Test Condition C) (150°C)	YES	NO
	—	—

REMARKS:

c. Temperature Cycles (Per Method 1010 Test Condition C, -65°C to +150°C, 10 Cycles)	YES	NO
	—	—

REMARKS:

d. Constant Acceleration (Per Method 2001 Test Condition E min, Y Plane) Modify for Hybrids	YES	NO
	—	—

REMARKS:

e. Interim Electricals (Before Burn-In)	YES	NO
	—	—

REMARKS:

f. Burn-In Test (Per Method 1015, 168 hrs at 125°C Minimum)	YES	NO
	—	—

REMARKS:

g. Final Electrical Test	YES	NO
--------------------------	-----	----

(1) Static Tests 25°C	—	—
-----------------------	---	---

(2) Maximum and Minimum Rated Operating Temperature	—	—
---	---	---

g. Final Electrical Test (Continued)	YES	NO
(3) Dynamic/Switching Tests (at 25°C)	—	—
(4) Functional Test (at 25°C)	—	—

REMARKS:

h. Seal (Per Method 1014)	YES	NO
(1) Fine	—	—
(2) Gross	—	—

REMARKS:

i. Bond Pull Test (100% Non-Destructive for Hybrids Only)	YES	NO
	—	—

REMARKS:

11. Recommended Changes to Screening Methods

12. Are the Above Screening Methods Adequate and Appropriate for Electronic Components Which Will be Placed In Long Term Storage?

13. Screening Method for Semiconductor Devices MIL-STD-750

a. High Temperature Storage (48 hrs Minimum at T _a = 175°C)	YES	NO
	—	—

REMARKS:

b. Thermal Shock (Temperature Cycle, Per Test Method 1051, 10 Cycles)

YES NO

c. Acceleration (Per Method 2006, Y₁ orientation at 20,000 G Minimum)

YES NO

REMARKS:

d. Hermetic Seal (Per Method 1071)

YES NO

Test Condition G or H (Fine Leak)
Test Condition C or E (Gross Leak)

REMARKS:

e. Pre-Burn Electrical Test

YES NO

REMARKS:

f. Burn-In Test (Per Method 1026 Test)

YES NO

REMARKS:

g. Post Burn-In Electrical Test

YES NO

REMARKS:

14. Recommended Changes to Screening Methods

15. Are the Above Screening Methods Adequate and Appropriate for Electronic Components Which Will Be Placed in Long Term Storage?

16. Storage Reliability (AR 70-38)

a. What is your approach towards meeting the storage requirements (time oriented) placed upon you in development contracts?

b. Accelerated test methods to evaluate storageability of electronic components.

c. What type of failures have you observed from storage that differ from operating?

d. Have you observed mechanical failure mechanism on microelectronic circuits in storage which have been Class B screened?

17. Use of Plastic in Long Term Storage Which You Have Available Data Check

	YES	NO	UNDER INVESTIGATION
a. Die Attachment	_____	_____	_____
b. Polymer Seals for Hybrid Packages	_____	_____	_____
c. Substrate Attachment in Package	_____	_____	_____
d. Direct Encapsulated Semiconductors, Microcircuits, Hybrid Microcircuits	_____	_____	_____

IF YOU WISH A COPY OF THE FINAL REPORT, PLEASE FURNISH YOUR NAME AND ADDRESS BELOW:

NAME: _____

COMPANY: _____

ADDRESS: _____
