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METHODS TO DETERMINE THE COMPATIBILITY AND DEGRADATION OF ORGANIC MATERIALS

Robert B. Bonk
Linda J. Morlan
Kathleen A. Chabot

July 1991

U.S. ARMY ARMAMENT RESEARCH, DEVELOPMENT AND ENGINEERING CENTER

Armament Engineering Directorate

Picatinny Arsenal, New Jersey

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Robert B. Bonk, Linda J. Morlan, and Kathleen A. Chabot

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13. ABSTRACT *(Maximum 200 words)*

Some of the various methods used to evaluate the compatibility and degradation of organics when exposed directly or indirectly to energetic and nonenergetic materials is described in this report.

The compatibility and degradation of materials has been and will continue to be a repeated problem with both developmental and field commodities. The deterioration process is due to numerous factors, i.e., time, temperature, humidity, heat, types of exposure (storage - direct or indirect), materials involved, etc. If this type of information is not available prior to fielding an item, catastrophic failures could result. Therefore, it is imperative that information on the compatibility of systems be known or generated prior to fielding.

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FIGURES

INTRODUCTION

Long-term compatibility of organic materials with energetics has been a repeated problem with both developmental and fielded commodities. The degradation of the material usually occurs over a long exposure time, the deterioration process is dependent on the materials involved, storage temperature, and the type of contact. Also, the solvents and oxidizers contained in such energetic materials as single-, double-, and triple-base propellants can act as plasticizers, and these plasticizers can destroy the engineering properties of some adhesives, polymeric and composite/organic materials over extended periods of time.

The compatibility of materials with energetics is determined by the elevated-temperature testing of an intimate mixture of known quantities of the contact materials and, concurrently, the identical quantities of the control samples. The behavior of the mixture as compared to the total behavior of the controls is indicative of the degree of reaction. If the reaction is negligible or slight, the materials are considered compatible for limited contact under normal storage (real life situations); if there is an excessive reaction, the engineer generally tries alternate materials.

The results of the elevated-temperature testing are measured as gas evolved, mass temperature differential, time to bleaching of indicator paper, or resistance to explosion over a period of time. In all of these, the measurement is the tendency of the energetic to show degradation due to contact with the inert material.

The single exception to the general methods of compatibility testing is the storage type. In this, specimens of the material are stored at elevated temperatures in direct and indirect contact with the energetic and are withdrawn periodically for physical testing and comparison with controls stored concurrently. Here, the measurement is the resistance of the inert material in contact with the energetic; did it harden, soften, or exhibit some other type of physical change. However, in earlier work at Picatinny Arsenal, the contact energetic was also tested periodically, for comparison with the values obtained from the same energetic stored concurrently for use as a control.[1]

Reactivity testing conducted at the Army Research, Development and Engineering Center provides information on the effects of the polymer material on the energetic material. The reaction between the two materials may either sensitize or desensitize the energetic. Neither increased sensitization nor desensitization is desirable. On the other hand, prolonged storage will reveal the deleterious effects, if any, on the mechanical integrity of the polymer.

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*Additional information on this work can be found in Picatinny Arsenal report 2595 (AD310 262), March 1959.

Compatibility of armament items is not solely restricted to exposure to energetics alone, but also to direct and indirect contact and exposure to adhesives, sealants, paints, lubricants, chemicals, etc. These agents have been known to be detrimental to the performance of the end item causing malfunctions to occur in the field. One example of this is the aeroballistic housing used on the GATOR mine. In this case, if the adhesives used to adhere to mine to the body are not cured properly, the constituents in the adhesive will react with the acrylonitrile-butadiene-styrene (ABS) plastic housing causing it to stress crack.

BACKGROUND

Energetic compatibility testing at Picatinny Arsenal started early in World War II. The concept was brought about by a casual concurrence of (1) a need to know what had happened to certain armament hardware where energetics contacted other materials and (2) the development of a test which under closely controlled conditions, could reveal if degradation was occurring. As an illustration, something happened to a particular lot of grenades that were manufactured during this time frame. If was found that the grenades had rusted internally prior to loading with a western cartridge (WC) ball powder (a single base propellant grain with nitroglycerine coating). Through a series of reactivity tests of the iron rust in contact with all of the main ingredients of the WC ball powder, it was discovered that nitroglycerine and iron rust (suprisingly) are incompatible when in contact with each other.

From this investigation, it was a short and logical step to require previous knowledge of the compatibility of energetics with the inert materials that they contacted. Many materials are compatible; only a few are extremely incompatible. However, unless there is supporting information in considerable quantity and variety, it is unsafe ever to assume satisfactory compatibility behavior for any combinations involving energetic and nonenergetic materials. To illustrate, two fairly well-behaved materials are amatol and hydrocarbon wax. Put them together and they will detonate in 20 minutes at 100°C.

The bottom line which is of greatest concern to the design engineer is the decision on how much reaction between energetic and inert material can be tolerated in ammunition designed for 10 to 20 years of shelf life or storage.

METHODS EMPLOYED IN TESTING FOR COMPATIBILITY/DEGRADATION

Reactivity Test (Vacuum Stability Test)

Concept

The contact of an energetic with a contiguous material may, in time, cause the chemical deterioration of the energetic. This may lead to the development of non-standard or hazardous conditions, or to spontaneous ignition of the energetic material. The reactivity test, by subjecting intimate mixtures of the energetic and materials it might contact to prolonged high temperatures, simulates the possible effect of such contact under normal conditions.

The procedures and apparatus needed to perform the testing are found in MIL-STD-286B, Method 403.1.2 (ref 1). A typical test apparatus can be found in figure 1. The preparation of specimens for testing may require glass plates (for film drying) or tools for cutting, rasping, or grinding, as suggested below.

Preparation

Since the efficiency of the test is commensurate with the degree of contact between the materials under study, insure an intimate contact by reducing all solids to a practical fineness by cutting or milling into chips, rasping into shreds or granules, or pulverizing, always observing established safety procedures. For some materials for which reactivity data may be required, special preparations will be necessary. Typical of such materials are films (paints, lacquers, adhesives, sealants, etc.) may be predried on glass plates, stripped, and chipped. Those which form nonremovable films (metal coatings such as parkerizing and anodizing) may be deposited on chips of the metal involved and then tested.

Procedure

Normally the test temperature is 100°C for explosives and 90°C for propellants; in special cases the temperature may be raised to 120°C or lowered to 75°C. The duration of the test is usually 40 hours.

For the basic unit (one energetic and one contact material) selected three sample tubes. Into the first tube place 2.5 ± 0.01 grams of the energetic material, into

the second tube place .25 ± 0.01 grams of the contact material, and into the third tube placed 2.5± 0.01 grams of the energetic material and 2.5 ± 0.02 grams of the contact material.[2]

Blend the materials which have been placed in the third tube by appropriate agitation, being careful not to lose any material or get it onto the ground-glass throat of the sample tube (this might make for an insecure junction between the sample tube and the manometer). Complete the three assemblies by joining the capillary tubes to the sample tubes and proceed with the vacuum stability test.

Evaluation

In determining the degree of reactivity of the materials under test, the materials processed separately are used as controls. The reactivity (or chemical deterioration) of the energetic is measured by comparing the volume of gas generated by the mixture of the energetic and the chosen contact material with the volume of gas generated by the controls.

The extent of reactivity is then calculated by the following equation

$$R = C - (A + B)$$

where

R = extent of reactivity of volume of gas generated by the mixture in excess of the controls

C = volume of gas generated by the mixture

A = volume of gas generated by the energetic

B = volume of gas generated by the contact material

[2]These weights are standard. Variations are sometimes imposed by (1) the wishes of the engineer, (2) limited supply of the materials to be tested, or (3) limitations on the amount of energetic that is safe to test.

Estimate the degree of reactivity by comparing the extent of reactivity (gas volume) with the following adjective-rating table:

Extent of reactivity excess gas, ml	Degree of reactivity
Negligible (a)	0.0 - 3.0
Moderate/normal (b)	3.0 - 5.0
Excessive (c)	5.0 and above

(a) Amount of gas evolved by mixture in comparison with that evolved by materials individually indicates a negligible reactivity.

(b) Amount of gas evolved by mixture in comparison with that evolved by materials individually indicates a moderate reactivity.

(c) Amount of gas evolved by mixture in comparison with the evolved by materials individually indicates an excess reactivity.

Differential Thermal Analysis (DTA)

The technique of DTA[3,4] measures the difference in temperature between an inert reference material and a sample as both are heated at the identical rate. By this technique, the temperature or temperature changes at which a sample undergoes endothermic or exothermic changes (i.e,, melting or decomposition) can be determined. This work gives as a basis for evaluation the following:

A	Compatible	0 - 2°C
B	Slightly sensitized	3 - 5°C
C	Sensitized	6 - 15°C
D	Hazardous	15°C and above

[3]Honeywell Inc., Ordinance Division Report "Compatibility of Explosives with Foreign Ingredients by Differential Thermal Analysis," August 1968, revised August 1969.

[4]A recommended source for information on DTA is "Differential Thermal Analysis," W. T. Smathers and Yas Chiang, Chemical Publishing Company.

A = Safe for use in any explosive design.

B = Safe for use in testing, when the device will be used in a very short period of time. Not to be used as a binder material, or when long-term storage is desired.

C = Not recommended for use with explosive items.

D = Hazardous. Do not use under any conditions.

LONG-TERM STORAGE AND MECHANICAL/PHYSICAL TESTING

Specimen Types and Storage Conditions

Specimens used for long-term evaluations are usually of the following types: (1) dumbbell or straight conforming to ASTM D412, Method A (ref 2); (2) tensile bars conforming to ASTM D638 and D638M (refs 3 and 4); (3) Naval Ordnance Laboratory (NOL) rings conforming to ASTM D2291 (ref 5); and (4) cubes (1 cubic inch) or discs (1.0 in. dia. x 0.5 in. thick). Occasionally, single lap shear type specimens conforming to ASTM D1002 (ref 6) are used to evaluate adhesive joint deterioration. Various types of specimens used in compatibility/degradation evaluation testing are illustrated in figure 2.

There are numerous types and variations of storage containers used for the long-term compatibility study. A typical storage container used for tensile bars, for dumbbell or strip type specimens, and in some instances, for slugs or discs is illustrated in figure 3. The size and shape of the container is not standard and can vary. For clarity and identification purposes, the illustration shows only three test specimens partially immersed in the energetic. Under normal long-term storage conditions, five to ten specimens would be exposed to the energetic material. Another type of container used for storing NOL ring type specimens is illustrated in figure 4.

Specimen contact may vary from test to test depending upon the type of exposure the end item will see in real life. Examples are as follows: (1) total, the entire specimen is immersed in the energetic; (2) partial, a portion of the specimen is immersed in the energetic; and (3) indirect, specimens are suspended above the energetic (in this situation the fumes/gases liberated from the energetic act upon the material). The result of stacking specimens one on top of the other and placing them in the energetic in that fashion is illustrated in figure 5. The clear (unshaded) area shows where the specimens were in direct contact with each other and not the energetic; the darker (shaded) area shows where the specimens were in intimate contact with the

energetic. If specimens are stored for extended periods of time in this stacked fashion, the uppermost specimen will leave an indentation in the specimen below it. Therefore, in order to obtain accurate and reliable results, specimen positioning is of utmost importance.

Specimens are stored in appropriate sealed containers at standard laboratory conditions, at elevated temperature (140°F), at 140°F with the energetic and in a magazine/bunker type environment with energetic. Five to ten specimens are withdrawn at each condition after 1, 3, 6, 12, 18, 24, and 36 months and tested for changes in mechanical and physical properties. In selected cases, the storage phase can be extended for up to 60 months. However, before extending the time limit the question of whether the energetic will be safe to handle or will it become desensitized must be addressed.

Prior to initiation of the storage phase, mechanical and physical tests are performed on the contact inert materials in order to establish room temperature control values which will be used later to determine if degradation has occurred. The physical and mechanical properties tested are the following: visual inspection; weight and dimensions; tensile strength, modulus and elongation (refs 2 and 3); lap shear strength (ref 6); hardness (refs 7 and 8); compressive shear strength (ref 9); composite and fiber stress and extension (ref 10); and specific gravity (ref 11).

Additional testing that can be performed to evaluate the compatibility/degradation of materials are as follows:

1. Modified vacuum stability (reactivity). This test consists of increasing the time of the standard test from 40 hours to 120 hours.

2. Accelerated long-term storage. This test consists of storing the inert material in contact with the energetic for 90 to 120 days at 160°F to 165°F.

3. Bending stress test. This test evaluates the compatibility/degradation of adhesives/solvents or any chemical reagent with organic materials. Bending stress fixtures designed by General Electric are used. Specimens 0.125 in. thick by 0.500 in. wide and 3 in. long are affixed to bent strip fixtures at various stress levels. Bending stresses are determined by the curvature of the fixture and range from 500 to 2500 psi. A sketch of the fixture and specimen is shown in figure 6. After fixturing, the specimens are coated with a thin layer of the adhesive/solvent to be evaluated. Control specimens are run concurrently with the coated samples. Controls consist of a stressed specimen without an adhesive/solvent, and an unstressed strip. Specimens are maintained at standard laboratory conditions for 10 days (this can be increased) and are visually

inspected periodically for indication of any stress cracking. Investigations can be accelerated by placing stressed specimens with and without adhesive/solvent and unstressed specimens in a high temperature/humidity environment.[5]

4. Stressed durability test. This test assesses the adhesive lap shear joint durability while under a sustained load in contact with air, air in equilibrium with certain solutions, water, aqueous solutions, or other environments at various temperatures. The procedures and apparatus necessary to perform this test are located in reference 5. The test specimens are fabricated according to reference 6. Each assembled test apparatus (fig. 7) is loaded to a percentage of the lap shear strength and then placed in an environmental chamber. The durability of various adhesive joints is reported as time to bond failure. This test may be used (1) as an accelerated screening test for assessing durability of various adhesive joints, (2) to measure durabilities of adhesive joints when exposed to various environmental conditions as experienced in service, and (3) to determine the effects of various adherends and surface preparations on the adhesive durability. A possible replacement for this test is ASTM D2294 (ref 12).

CONCLUSIONS

In evaluating compatibility/degradation data, it is necessary to recognize that two distinct effects may be present. On the one hand, there is the effect on the energetic materials caused by the inert material. This effect is generally chemical in nature and is detected by the short-term tests such as vacuum stability, differential thermal analysis (DTA), differential scanning calorimetery (DSC), thermogravimetric analysis (TGA), and various short-term heat tests. On the other hand, detection of the effects of the energetic material on the inert material usually requires a test of much longer duration with continued storage of materials in contact with each other.

The short-term effects on the energetic material usually involve chemical processing resulting in gas release or energy changes; the long-term effects on the inert material may be more subtle. Slow diffusion of a plasticizing component from the energetic may result in softening and/or mechanical property changes in the inert substance without any apparent chemical reaction. Chemical changes also may occur in this case but usually only after a considerable time lag. In these long-term tests (as previously stated) the materials are generally stored together and samples are withdrawn at various intervals. Any changes in properties such as appearance, sample weight, and mechanical properties are monitored.

[5]This test is similar to ASTM D3929-80, "Standard Practice for Evaluating the Stress Cracking of Plastics by Adhesives Using the Bent-Beam Method."

For either short- or long-term testing, an elevated temperature is generally used in order to accelerate any changes that may occur. While convenient and necessary from a practical point of view, this acceleration does require care in interpreting the results since ambient use conditions are generally milder. Nothing is really clear-cut and well defined. Numerous studies have shown that the visual examination of results indicate large scatter among replicates and a disturbing difference in averages, even among controls (refs 13 and 14). This, therefore, has suggested the use of methods to obtain an estimate of whether these differences can be explained by the point scatter about the average procedure.

Where distribution of data is not normal, or where sample sizes are small, it is useful to use nonparametric or distribution-free statistical methods which are considered valid regardless of the shape of the population distribution (ref 15). Although not generally as powerful as parametric statistics, it is very useful for small sample sizes in mechanical/physical testing experiments where experience had indicated that the normal distribution does not apply.

In initial testing and evaluation of data, the Wilcoxon sum of ranks test (ref 16) is used for comparison of data sets. This test has the advantage of simplicity and ease of use. The Wilcoxan sum of ranks test is nonparametric and therefore is a less powerful test than an equivalent student's t test for the normal distribution. However, numerous works conducted at Picatinny Arsenal have concluded that populations that are identical within 95% confidence level indicate no significant difference between samples.

When data scatter is found to be significantly large enough to be troublesome, a second procedure or method is suggested as a means to obtain some insight into the nature and extent of the differences. For this purpose, Weibull distribution statistics are used because they can accurately predict mechanical/physical behavior of materials (ref 17). Weibull distribution statistics work under the assumption that failures occur at the weakest link of a system (ref 18), that material flaws are independent of each other, and that the weak links remain unchanged during the stressing process (ref 19).

Specimens tested under a given set of test conditions can be represented by the following Weibull cumulative distribution function (ref 18):

$$\text{Log Log}\frac{\left[\quad 1 \quad\right]}{\left|1 - F(x)\right|} = \text{Log}\alpha + \beta \text{ Log } x$$

Where F(x) is the distribution function, i.e., the fraction of samples showing a mechanical/physical property value of x or below, α is the scale parameter (y-intercept), and β is the slope. A plot of the left-hand side of the equation versus log x should give a straight line of the slope β and intercept. The plot can be interpreted as the strength versus the probability of failure at that strength (ref 17). The scale parameter, α, indicates the location of the line and is a measure of how long the sample lasted (ref 19).

The slope β is called the Weibull modulus in material analysis and is a quantitive indication of the test variability (ref 17). If there are more than 20 observations, F(x) in the equation is obtained as a fraction of the total number of items that are less than or equal to each value of the variable divided by the total number of items in the test. If there are 20 or fewer observations, tables of plotting positions that make an adjustment for the smallness of the sample size are used (ref 17).

RECOMMENDATIONS

1. In compatibility/degradation testing of materials, never assume anything.

2. End-users often establish the anticipated service life for their particular application. While manufacturers can furnish information on the chemical and physical properties of their products, it is not usually possible to predict results from these data for the different parameters of organic types (adhesives, sealants, polymers, elastomers, etc.), surface preparations, service conditions, etc., all of which can affect the compatibility/durability of the end item. The most accurate determination of service life would involve measurement of actual changes that occur in the service environment. Although duplication of all conditions can seldom be expected, certain exposures to accelerated laboratory conditions or natural environmental conditions should provide pertinent information.

3. Prior to recommending or adapting any system consult the ARDEC Plastics Technical Evaluation Center (PLASTEC) Data Bases - COMPAT, HAZARD, and Corrosion.

4. If searching through the data bases yields negative results or proves to be inconclusive, contact the Adhesives Section, Organic Materials Branch, AED. They are the experts in the area of compatibility/durability/degradation of organic materials (adhesives, coatings, polymers, etc.) and lubricants. Also, they have the latest data available and will know if changes in formulations have occurred and if the data obtained on a particular system is still valid or if it should be requalified.

5. Evaluate the system using established procedures and experienced personnel.

6. As test data are generated, they will be incorporated into the various data bases at PLASTEC, disseminated to the cognizant material developers (i.e., mortars, mines, demolitions, small and large caliber ammunition, tank, etc.) and DoD contractors, and the findings will be published and briefed to the defense industry through ADPA, JANNAF, SAMPE, Journal of Hazardous Materials, etc.

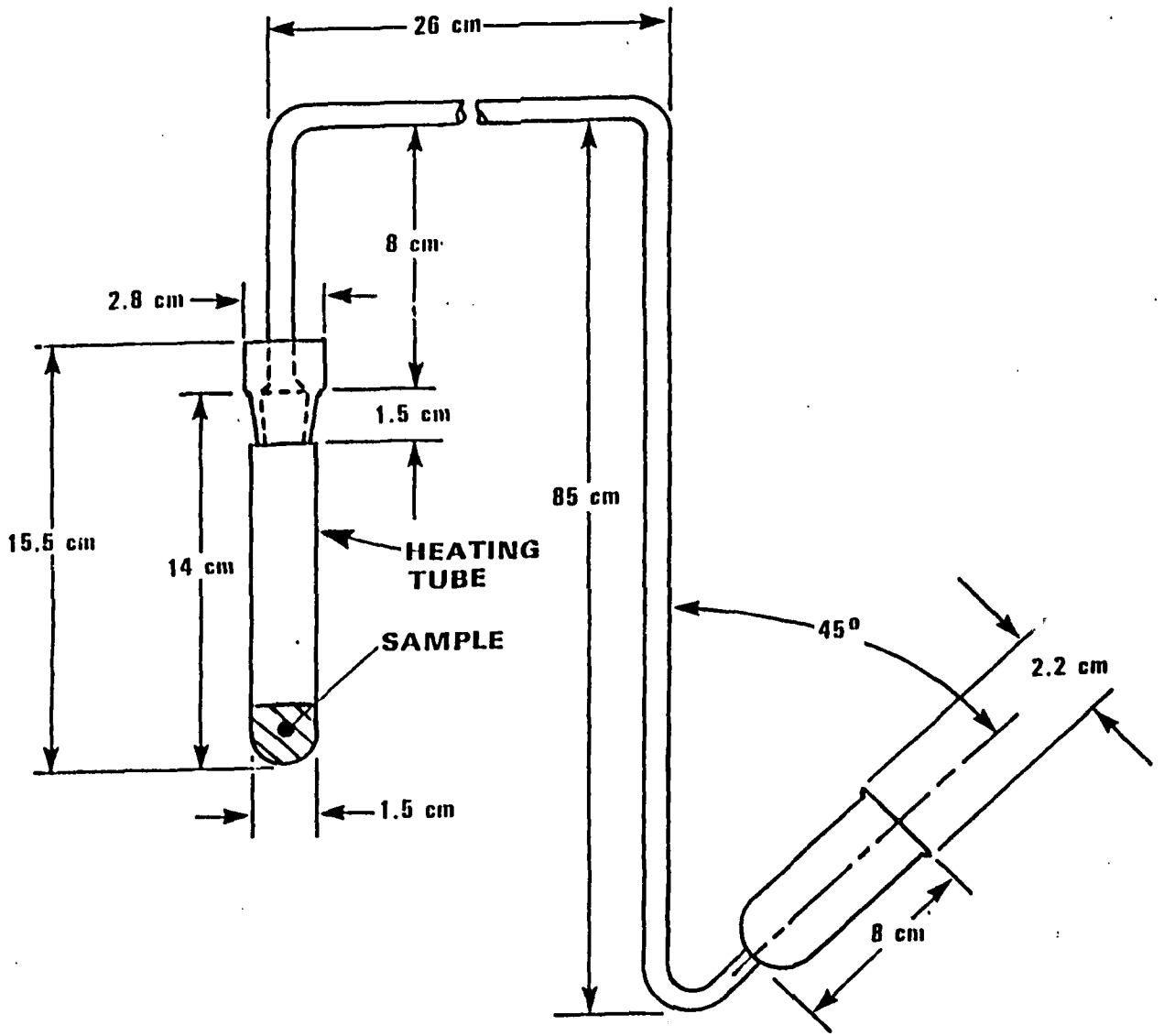
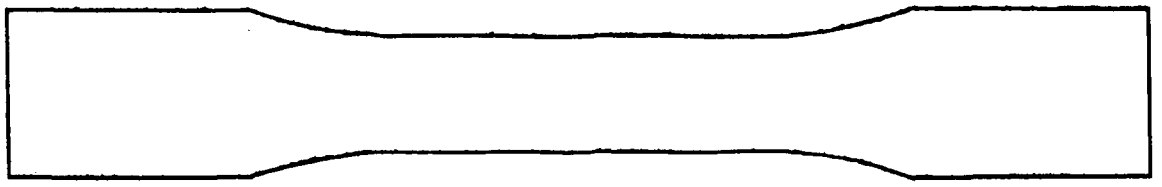
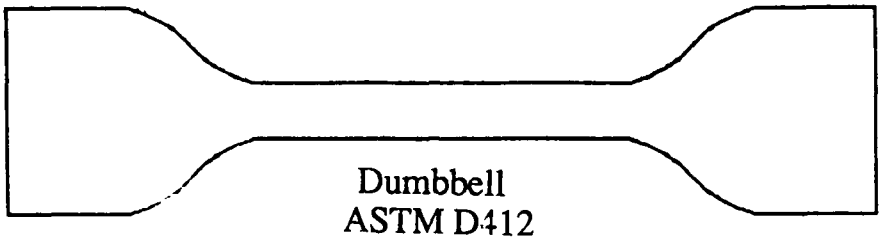


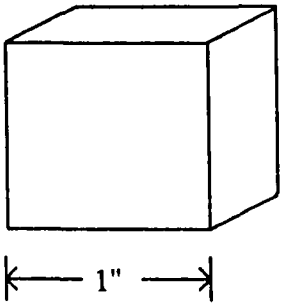
Figure 1. A typical vacuum stability test apparatus



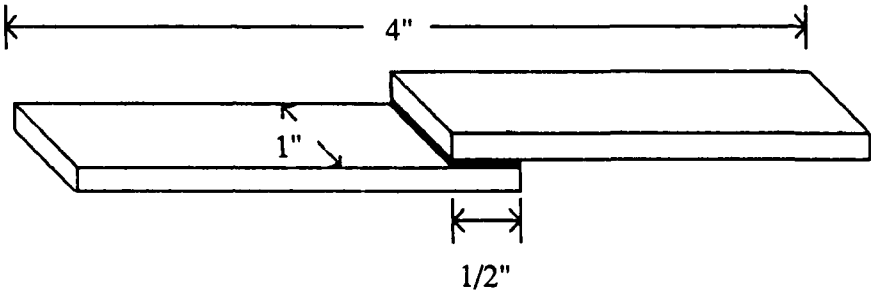
Tensile Bar
ASTM D638



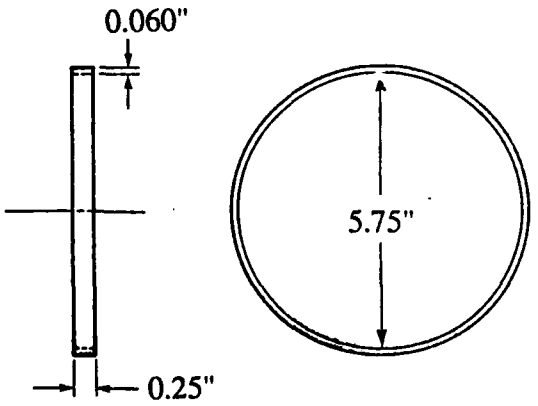
Dumbbell
ASTM D412



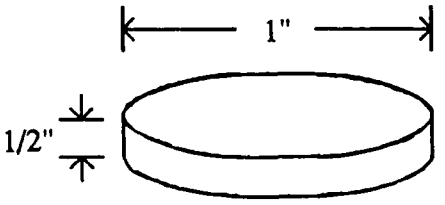
Cube



Single Lap Shear
ASTM D1002



NOL Ring
ASTM D2291



Disc

Figure 2. Typical compatibility specimens

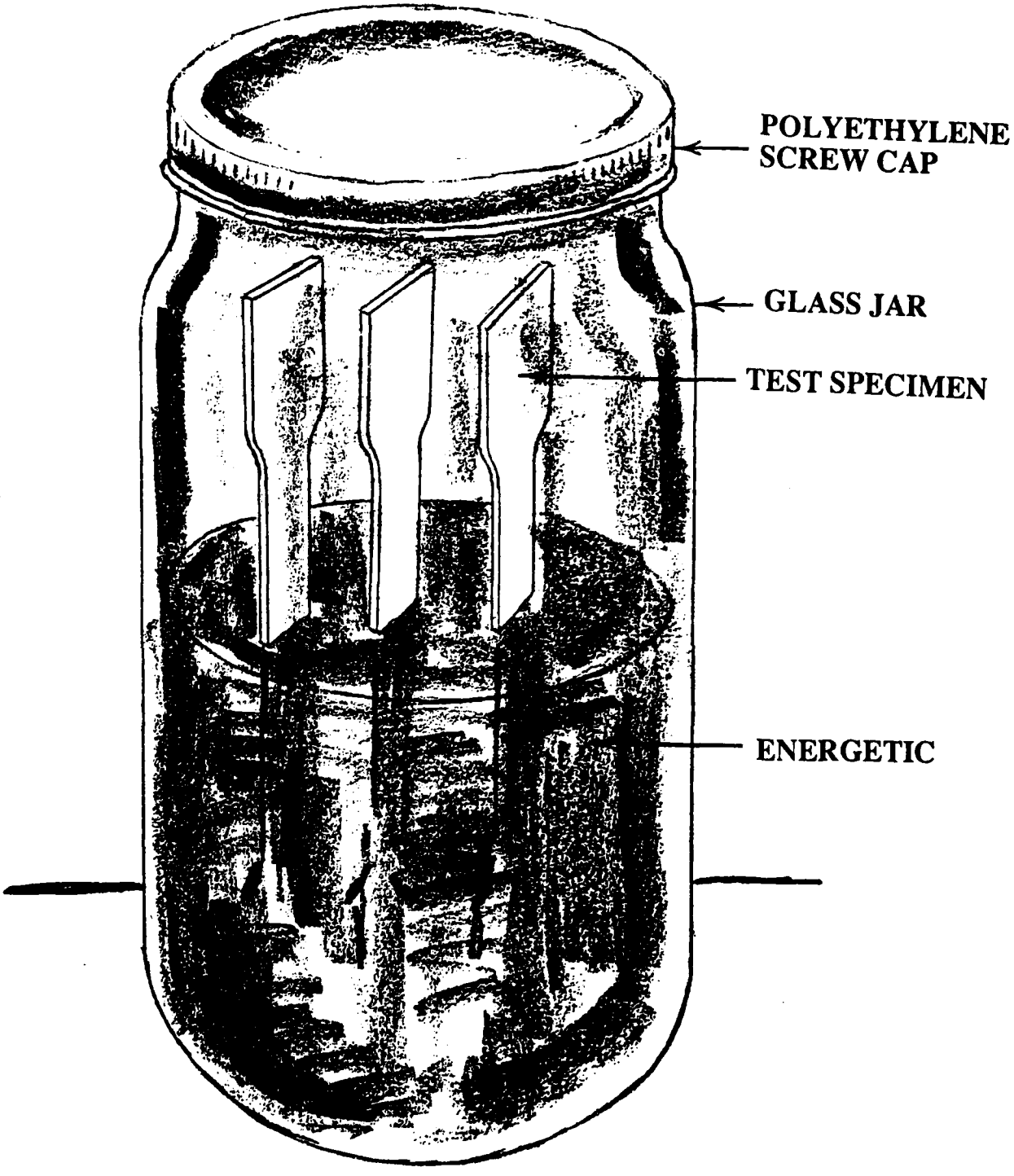


Figure 3. Storage test apparatus

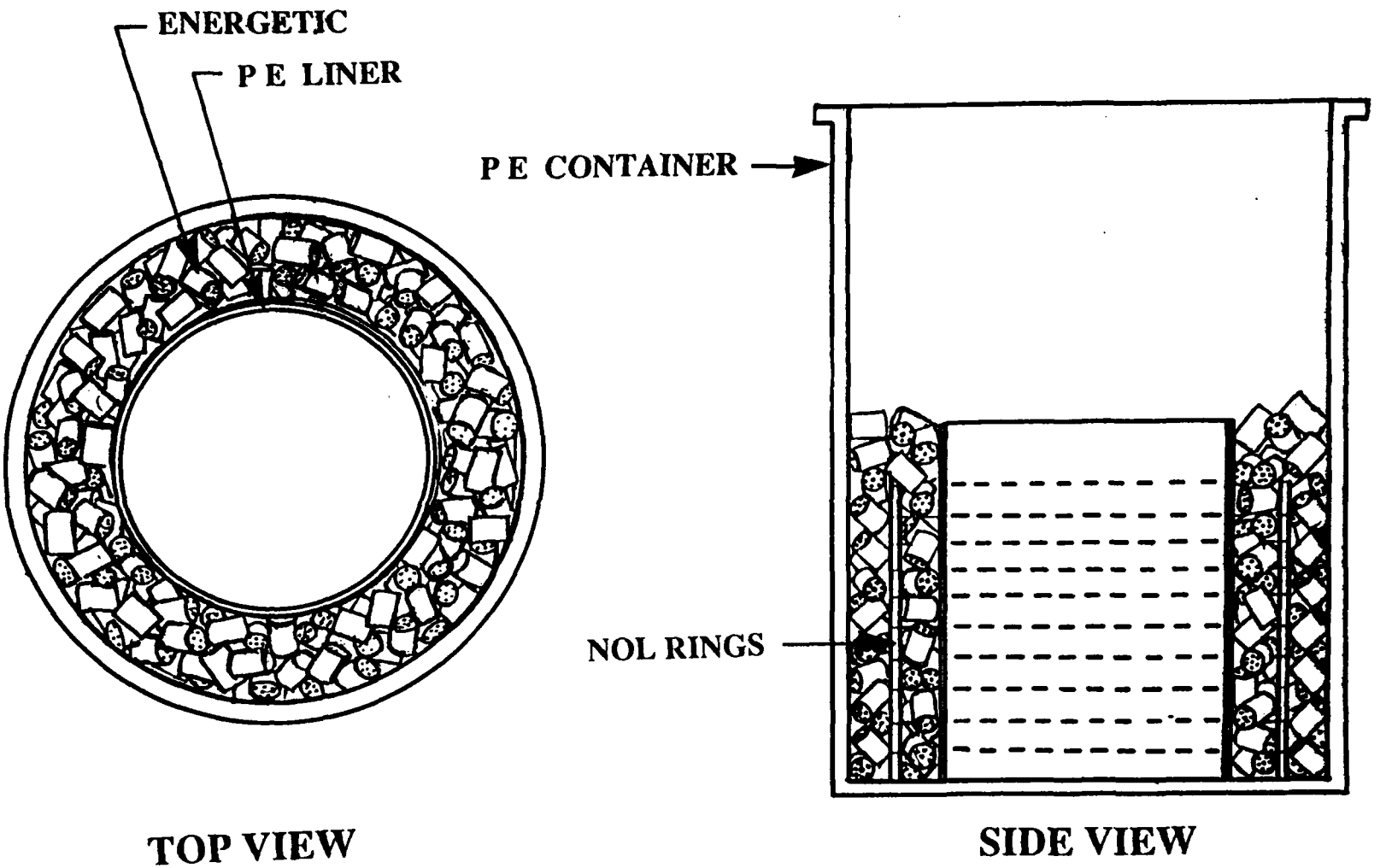


Figure 4. Storage of NOL rings

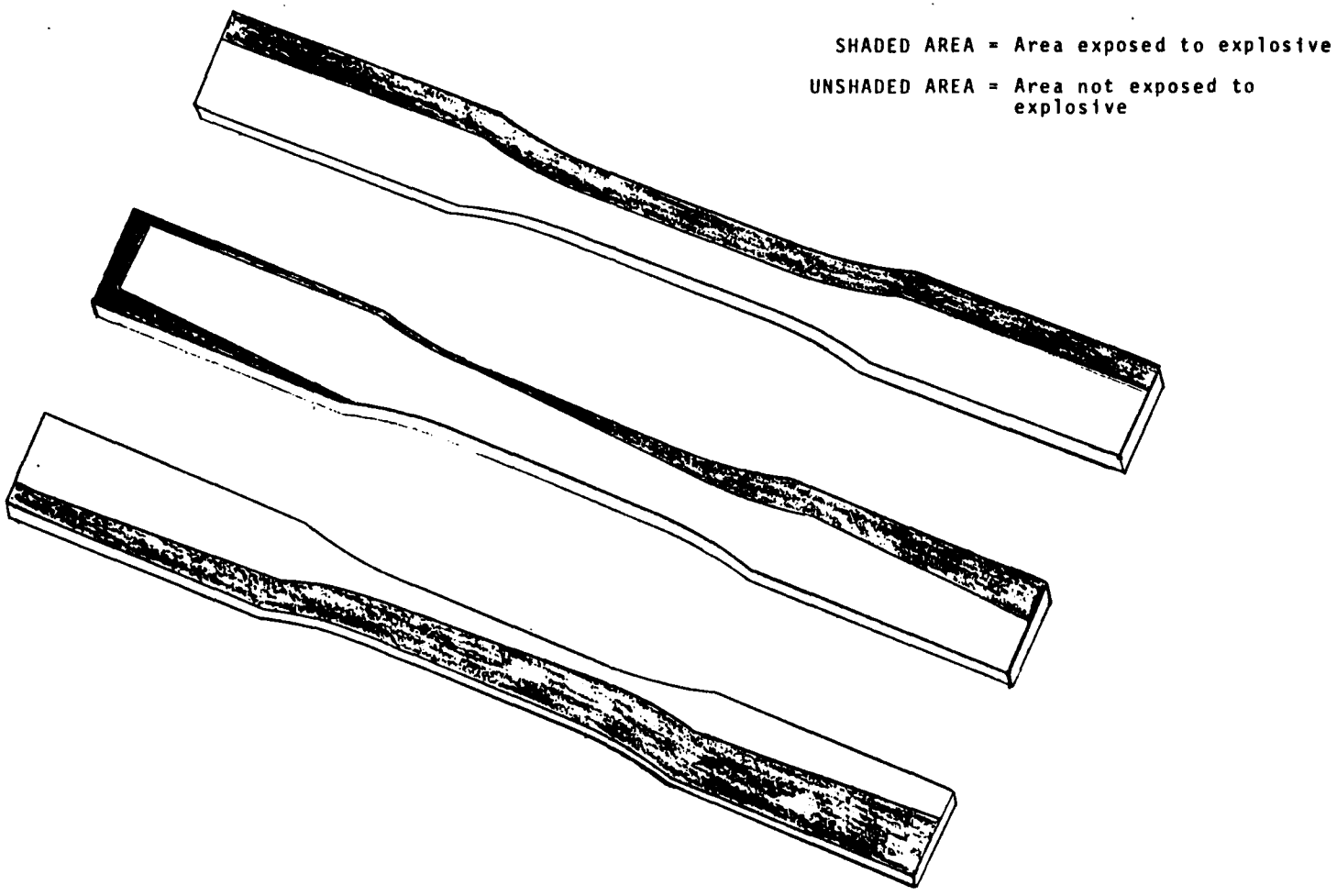


Figure 5. Illustration of specimens improperly positioned in storage test apparatus

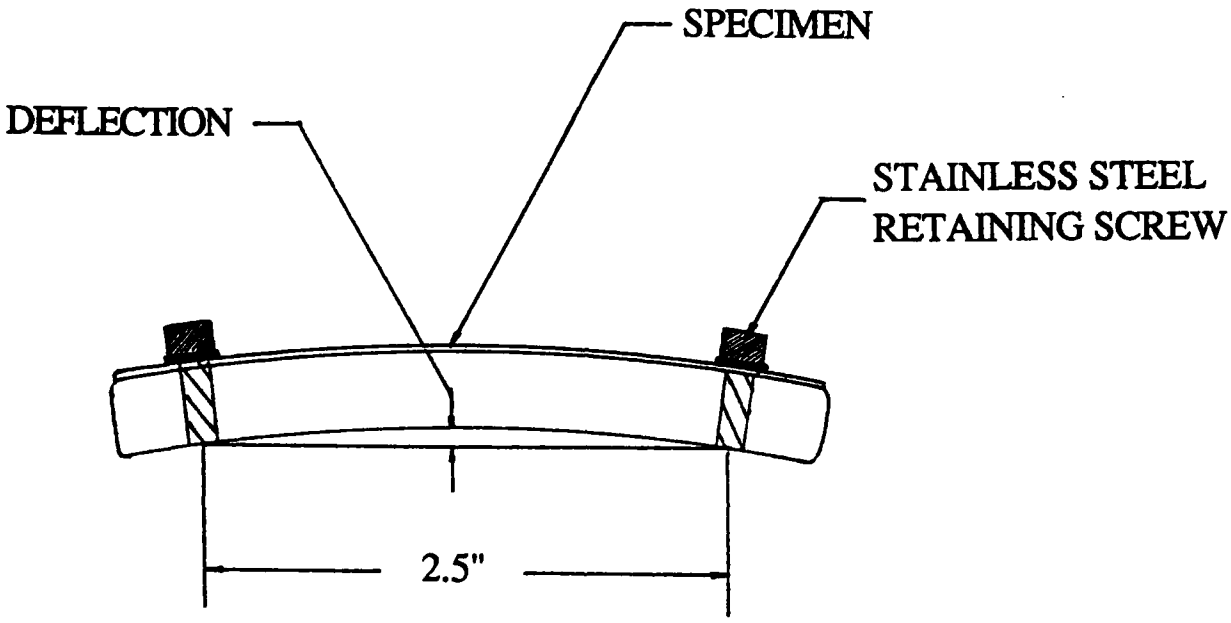


Figure 6. Bending stress fixture.

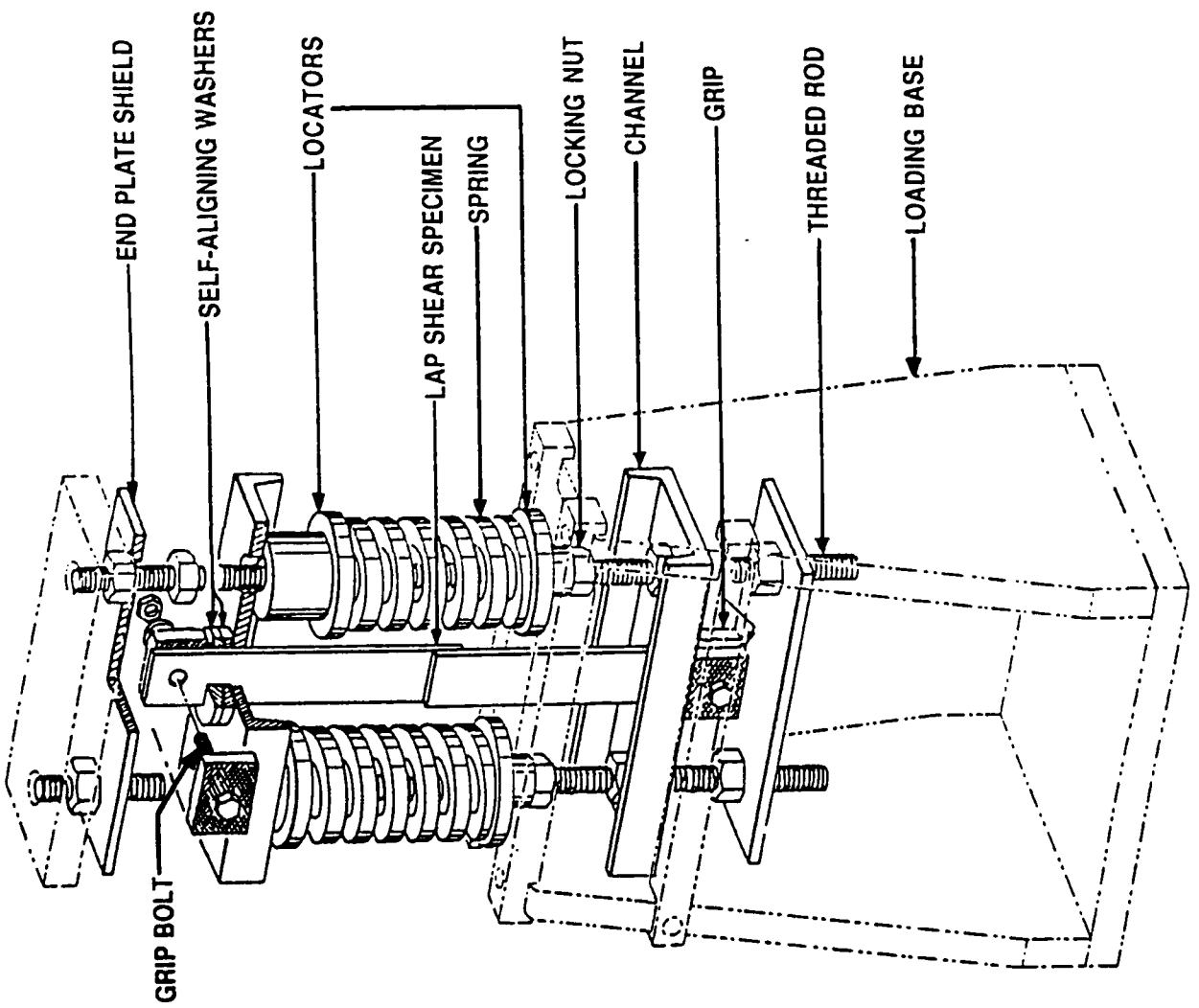


Figure 7. Assembled stressed durability apparatus

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