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ANALYSIS OF PROPELLANT STORAGE TANKS AFTER FOUR YEARS HYDRAZINE STORAGE

FINAL REPORT - MAY 1976 - APRIL 1977

BELL AEROSPACE TEXTRON
DIVISION OF TEXTRON, INC.
P. O. BOX ONE
BUFFALO, NEW YORK 14240

AUTHORS: E. J. KING
H. G. KAMMERER

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Prepared for:

AIR FORCE ROCKET PROPULSION LABORATORY
DIRECTOR OF SCIENCE AND TECHNOLOGY
AIR FORCE SYSTEMS COMMAND
EDWARDS AFB, CALIFORNIA 93523

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FOREWORD

This report was submitted by Bell Aerospace Textron, Division of Textron, Inc., P. O. Box One, Buffalo, New York 14240, under Contract No. F04700-76-90043, Job Order No. 305911RM with The Air Force Rocket Propulsion Laboratory, Edwards, CA 93523.

The Project Manager was E. J. King; the Project Metallurgical Engineer was H. G. Kammerer. In-depth analyses were conducted by H. G. Kammerer and D. G. Roberts. X-ray diffraction studies were conducted by D. Roberts and electron microprobe studies by E. Tomes.

This report has been reviewed by the Information Office/DOZ and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public, including foreign nations.

OREE DYES
Project Manager

WARREN R. HAYDEN, Lt Col, USAF
Chief, Propulsion Applications Branch

EDWARD E. STEIN, Deputy Chief
Liquid Rocket Division

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This report covers the examination and metallurgical evaluation of four tanks after they had been subjected to over four years storage with hydrazine propellant. The tank materials included 17-7 PH stainless steel and AM350, also a precipitation

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20. ABSTRACT -

hardened stainless steel. Only one instance of serious corrosion was found in the tanks and that was found to be most likely caused by an external source in the over one year time elapsed between removal of the tanks from hydrazine storage, and the start of evaluation.

The hydrazine storage occurred in two periods, the first of which was one year during which the temperature control was a problem (nominal 83.3°F). During this time some hydrazine decomposition did occur and venting was required to relieve pressure buildup. After modification and improvement of the insulation and temperature control equipment in the building, a temperature of 120°F was held steadily for the 3-1/3 years of remaining storage time, with no further pressure rise or hydrazine decomposition.

Some minor difficulties were present in these tanks, including some shallow corrosion of the stainless steel inlet/outlet tubes, and the likelihood that the flange cover closure bolts had loosened slightly over the extended storage period. In general, however, these precipitation hardened stainless steel tanks showed good compatibility with hydrazine and storability for times in excess of four years.

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SECTION I

INTRODUCTION

The advent of long term Air Force Weapon System Missions has made it necessary to evaluate long term storability of liquid rocket propellant systems. This contract was concerned with the Metallurgical evaluation of simulated aerospace tankage after storage of hydrazine for over four years, in unique environmental exposure areas at the Air Force Rocket Propulsion Laboratory (AFRPL). The storability program was designed to demonstrate environmental compatibility of tankage alloys with rocket engine propellants, thereby providing fundamental information on tankage materials, to be used over long storage periods.

The program conducted by BAT consisted of:

1. Documentation of as-received exposure vessels and components.

2. Definition of anomalies and defects that altered the functional capability of the components and exposure vessels.

3. In-depth metallurgical analysis of four component/tanks.

The identities and test histories of the four tanks evaluated in this program are summarized in Table I. This report covers the third in a series of programs conducted by Bell Aerospace Textron for the Air Force Rocket Propulsion Laboratory, all with the same objective, of metallurgically evaluating tanks and systems after propellant storage at Edwards Air Force Base. The first program, reported in Reference 1, covered over 70 tanks and systems, ranging from small one pint cylinders to complete prepackaged systems, with various oxidizer and fuel propellants. The second program, reported in Reference 2, covered the detail analysis of a Bullpup Missile with integral fuel and oxidizer tanks, after it had been defueled for UDMH recovery following over ten years of propellant storage.

Metallurgical examination of the exposure vessels/components identified the nature and extent of corrosion and other anomalies that had occurred over more than four years of storage. An effort was made to build a comprehensive matrix of positive and negative observations resulting from the analysis. Anomalies and failure modes were related to exposure conditions and when applicable to the mechanical characteristics that were deteriorated by the types of corrosion taking place. Processing and environmental effects were analyzed to determine their role in the abnormality or defect observed.

Mechanical properties were obtained from specimens machined from the test hardware, and include base metal properties as well as weld properties. These mechanical tests were necessary to verify heat treatments and to establish the extent, if any, of degradation from exposure, as well as verifying the integrity of weld joints.

SECTION II

PROGRAM STRUCTURE

This contract continued the storability program initiated by the Air Force Rocket Propulsion Laboratory (AFRPL) in its effort to bridge the gap between laboratory coupon tests and austere evaluation of tankage materials that have endured long term storability of earth-storable fuels and oxidizers. Tankage materials investigated were those common to the aerospace industry, where strength to density requirements are a vital economic factor, in the design of advanced space systems. They included aluminum alloys- cryogenic formed austenitic stainless steel, and an age hardenable stainless steel. This program dealt with the evaluation and demonstration of long term storage (up to 6 years), of tankage, components and integrated feed systems. The internal environment of these components was the oxidizers nitrogen tetroxide (N_2O_4) and chlorine pentafluoride (ClF_5), hydrazine (N_2H_4) and the mixed hydrazine fuel, MHF-5, (26% N_2H_4, 55% MMH, 19% hydrazine nitrate).

Reports by AFRPL illustrating the details and some of the early results of this storability program may be found in References 3 and 4.

The items included in this overall AFRPL program may be divided into three basic groups (1) small containers, (2) representative type tankage and (3) tankage systems with associated expulsion devices and/or feed system components.

The tanks being evaluated after storage in this portion of the program are all from Group II, Representative Tankage. The tanks in this group are of the 10 to 15 gallon size, which makes them typical of the types that would be used in reaction control systems. The tankage in this group was fabricated by current or advanced state-of-the-art methods. Therefore the range of fabrication and quality control problems encountered in manufacturing these vessels simulate those likely to be encountered during the manufacture of an operational liquid rocket system.

SECTION III

TEST FACILITIES AND PROCEDURES

A. LONG TERM STORAGE FACILITY - AFRPL

Storage of all of the hydrazine filled tanks examined in this study was conducted in a Quonset hut storage test building at the Air Force Rocket Propulsion Laboratory, Edwards Air Force Base, California. This is part of the extensive facility for storage testing of fuel and oxidizer tankage and systems that has been developed at the Rocket Propulsion Laboratory. The fuel storage building, separated from the oxidizer storage, can be controlled at any temperature between +65° and +165°F. Temperature conditioning is maintained by a heating and refrigeration system. There is no control of relative humidity. The building is insulated with a fire retardant, spray-in-place insulation. The facility is also equipped with a trace gas analyzer to detect any unnoticed propellant spillages, but this vapor detector is not tied into the building air conditioner system for automatic shutdown as is the case in the oxidizer storage building. The tanks being evaluated in this program had been in storage for approximately one year (1969/70) when the fuel storage facility was shutdown and tanks drained for extensive modification and upgrading of the facility. This included better temperature control equipment and the installation of the fire retardant insulation mentioned above. Thus, the first year of storage was under less stable temperature control than the final 3-1/3 years. All fuel loaded tanks were equipped with pressure gages to monitor pressure rise during storage.

B. POST-STORAGE TANKAGE ANALYSIS - BAT

The destructive examination of these tanks was conducted in the Bell Aerospace Textron Metallurgical Laboratories. The facilities required to conduct the metallurgical evaluation of these tanks were available and utilized within these laboratories. The evaluation procedures used are outlined below and suggest the types of equipment required. The equipment used in this work is described below.

After visual examination and photographic documentation of the as-received and as-sectioned vessels, they were examined in detail for corrosion, anomalies or defects using both binocular microscopes at low magnification and higher magnification research microscopes, such as the one shown in Figure 1. Photomacrographs of local corrosion and other anomalies were taken on view cameras, as seen in Figure 2. Cross sections of leaks, corroded areas, welds, etc. were prepared using the automatic rotary and vibratory metallographic polishing equipment shown in Figure 3. Photomicrographs of these metallographic sections, in the as-polished condition and after etching to reveal the microstructure, were taken on the research microscope shown in Figure 1. It was occasionally necessary to use radiographic inspection equipment, shown in Figure 4, especially to examine the suspected leak areas in the girth weld of tank S/N 3. Microprobe and X-ray diffraction analysis were carried out on corrosion products from S/N 5.

Mechanical properties were determined on all tanks evaluated in this effort and the majority of tanks in past efforts to establish the heat treatment condition or presence of degradation due to corrosion or other long term storage effects. A wide range of universal testing machines and electrohydraulic closed loop testing systems were available and used to determine these mechanical properties, depending on the load range and any special loading conditions required. A typical tensile test specimen taken across a weld from one of these tanks is shown in Figure 6, with examples of fractured and unfractured mechanical test specimens. A tensile test in progress is shown in Figure 5, utilizing one of the universal testing machines with a load range of 3,000 to 300,000 pounds.

Other facilities and equipment were used in an auxiliary or routine manner during various portions of this evaluation program. These included hardness testing equipment such as conventional Rockwell or Vickers, Leitz microhardness and a Sonodur for automatic microhardness traverses. Tank sectioning was performed on abrasive cutoff saws, lathes and bandsaws.

C. PROCEDURES

The procurement of test hardware and the environmental testing of this hardware with earth-storable propellants has remained essentially unchanged, since initiation of this long term compatibility program, Reference 1. Although these procedures have been previously documented they are also presented here to maintain completeness of the presentation and to provide a convenient reference for the post-test evaluations of exposed hardware being reported on.

Test articles evaluated in this program were procured from aerospace contractors, where primary responsibility for quality control and quality assurance of the test articles was vested. This hardware was fabricated according to specific procedural specifications encompassing detailed inspection and cleaning procedures, as dictated by the alloy being manufactured.

Helium leak testing of all individual tankage in the as-received condition was performed to ensure against the development of leaks and the introduction of contamination during shipment of the test articles from the manufacturer. Upon completion of the leak test, the tanks were loaded with propellant and placed in the appropriate storage facility for storability testing. The fuel tanks were monitored for both leakage and excessive pressure rise.

In the event of excessive pressure rise in a fuel tank, the tank is vented and propellant and ullage gas samples are taken. Tanks which exhibit continued pressure rise are removed from testing and analyzed to determine whether the pressure increase was due to an isolated instance or is indicative of a lack of storability of the material/propellant combination.

Following the above exposure test procedures, in this program and past programs, tanks were selected for destructive examination to ascertain the cause of failure or other observed anomalies. The metallurgical procedures used in the assessment of corrosive damage consisted of an examination of external and internal surfaces of the storage vessels with an in-depth analysis following the procedure outlined below. This procedure was submitted for approval of the project officer prior to initiation of these analyses.

1. APPEARANCE DOCUMENTATION

a. Those anomalies which are in large components will have the anomaly and surrounding material segment cut down for ease of handling.

b. Take photomacrographs of anomaly surfaces; remove for analysis any corrosion products or deposits, and take additional photomacrographs if surface changes or new features are involved.

c. If not already visible, section away from defect to reveal inside surface of anomaly area and take photographs of this inside surface.

2. EXAMINATION OF LEAK SURFACES

(Those components analyzed for surface pitting, etc. where no leak or deep corrosion is involved, were examined per 3 below.)

a. If a leak was suspected but not pinpointed, radiographs to verify location and extent were utilized.

b. The leak area was removed carefully from the surrounding metal.

c. The leak area was broken open by hand bending or tensile fracturing, to expose corrosion surfaces.

d. After microscopic examination at 10X to 60X, photomacrographs of exposed corrosion surfaces were taken.

e. A high magnification microscope examination was performed of one-half of exposed corrosion surface, to determine topography and significant features of corroded surface.

3. EXAMINATION OF PITTED SURFACES

(For those analyses where no leak is involved.)

a. Section through pitted region in a careful manner (usually with jeweler's saw) so that at least two segments of essentially equal pitting were available for study.

b. Perform microscopic examination of one-half of pitted surface to determine topography and significant features of pitted surface.

4. MICROSTRUCTURE AND RELATION TO CORROSION, LEAK OR ANOMALY

a. Mount a cross section through critical area of anomaly.

b. Polish using conventional metallographic techniques.

c. Examine in unetched condition for corrosion penetration of grain boundaries or similar effects and take photomicrographs.

d. Etch with appropriate reagents to bring out microstructure of weld and/or parent metal.

e. Examine and take photomicrographs of microstructure, both as it relates to corrosion effects and also to determine matrix microstructure and material effects.

5. CHEMICAL ANALYSIS OF CORROSION PRODUCTS AND CORRODED MATERIAL

a. If corrosion products were removed in Step a, Item 2, they were analyzed by X-ray diffraction or other analysis techniques.

b. If there is any suspicion that tank materials or weld filler metal was not of the alloy expected (based on microstructure or other observations), spectrographic analysis of component material was performed.

SECTION IV

FABRICATION HISTORY OF TANKS EXAMINED

In the analysis of corrosion behavior of any component it is instructive, and often necessary, to know the methods of fabrication and the processing details involved, in order to arrive at meaningful conclusions to the cause and significance of observed corrosion effects. Thus, in this program of analysis of representative tanks, after hydrazine propellant exposure, it has been necessary to collect as much fabrication history as possible to aid in the evaluation. This history is summarized in this section, and is then referred to in detail in the confirmatory analyses discussed in Section V on tank failure analysis. The reports and references from which this fabrication history were obtained are tabulated in the References (Section VII), with the specific reports from the manufacturer listed where applicable in Table II which accompanies this section. None of the tanks evaluated were fabricated at Bell Aerospace Textron, therefore, all of this section represents information obtained from reports or observations on the tanks themselves by investigators experienced in many phases of aerospace hardware fabrication.

The four tanks in this study were all part of "Storability Test Articles" of the Group II, Representative Tankage, portion of the AFRPL storage program. The tanks had all been manufactured by the Denver Division of Martin Marietta Corp. under U.S. Air

Force Contract F04611-68-C-0080. The summary of fabrication characteristics and specific reference to Martin Marietta reports documenting their work, are included in Table II. These tanks are of 10 gallon capacity and were fabricated and assembled using techniques and processes typical for those of production, flight type hardware and systems.

SECTION V

DISCUSSION OF RESULTS

A. VISUAL AND MACROSCOPIC EXAMINATION OF EXTERNAL AND PROPELLANT EXPOSED TANK SURFACES

The first stage in any examination of hardware for corrosion effects is a thorough examination and documentation of the surface appearance. This examination must be done by trained and experienced observers who will pay careful attention to preferential attack of welds, crevices and other susceptible regions. The initial examination of these tanks, after exposure to various propellants and storage room environments, was done in this manner, with complete photographic documentation of the external and, after preliminary sectioning, internal surfaces. The primary purpose of this initial examination (Phase I of metallurgical effort) was to identify those failures, anomalies or unusual conditions which would warrant a more detailed examination and analysis in the Phase II portion of the metallurgical effort. Accordingly, this section of the report documents the surface condition of the tanks as received from their various test exposures, and identifies those anomalies, failures or other corrosion and service effects, which will be considered in Section V, under C; Metallurgical Examination of Failures and Anomalies.

The four tanks received for post-storage test evaluation had all been drained and purged of their hydrazine (N_2H_4) storage fluid at least one year before the current examination.

One of the tanks had been partially sectioned at RPL before delivery to BAT, however, this tank has been evaluated in depth with the other tanks as part of the program.

Photographs of the as-received condition of each of the tanks are shown in Figures 7-10. In each case the flange cover had been removed from the flange end of the tank. As can be seen in the four views, the external surfaces of these precipitation hardenable stainless steel tanks were quite clean and unattacked. The cylinder and dome sections had the typical lightly frosted appearance of etched and passivated stainless steel, while the machined flange end member is of a shiny machined and passivated appearance.

One tank, S/N 5, which had been partially sectioned by RPL, shows evidence of corrosive attack on the small outlet fitting (sometimes called "Beanie" in Martin Marietta fabrication report, Reference 5) and adjacent fitting to tube weld. This etched or attacked region extends down onto the membrane section of the fitting with a localized corroded spot which may have penetrated through the wall. This region is shown in Figure 11 in greater detail. Also shown in Figure 11 is a comparative view illustrating that a section of the tube and tube to tube fitting weld was not in the original delivery of hardware for this evaluation program. The tube segment was eventually shipped and found to have only minor corrosive attack.

A detail view of this tube is also shown in Figure 11. The tube surface itself was lightly etched in the region immediately adjacent to the etched zone on the outlet fitting. There was no attack nor any evidence of defects or leakage on the tube to tube fitting weld or the tube fitting itself. This leakage area is one of the anomalies on which a Detail Metallurgical Analysis was performed. The results of that analysis are documented in Section V.C.

Tank S/N 3, shown in Figure 8, was received with four spots marked with circles on the girth weld nearest the flange. The exterior or crown of the weld in each of these spots showed a minor protrusion or spot, apparently remnants of incomplete fusion in rewelding or stop/start effects. Although these outer surface spots on the weld were obvious, they did not appear to represent weld defects. Review of the test log for these tanks showed that these spots had been marked before the initial loading of the tanks with propellant, based on slight indications during a leak test. Therefore, this anomaly was selected as one of those to be studied in detail. A complete discussion of results is found in Section V.C.

The interior, hydrazine exposed surfaces of the tanks were examined by sectioning the tanks lengthwise. This allowed the tank surfaces to be photographed in their entirety and be examined for overall effects such as "waterlines", which would indicate storage in a partially full condition. Photographs of the tank interiors are shown in Figures 10 and 12 through 15. There are "waterline" markings indicative of a partially full condition in a horizontal position in tanks 1 and 3. Tanks 4 and 5 show indications of only a small, residual liquid in a line that would represent the tank lying on its side also. Since all of these tanks had been taken out of storage test at least one year before being shipped to BAT for analysis, it is not surprising that these markings occurred. All of the reported test storage (the four year, four month test time in two periods) was in the "normal" orientation with the long tank axis vertical and the flange end up. However, the out of test time was not controlled and the tanks are known to have been in a holding area for some time awaiting disposition and eventual shipment to BAT.

The interiors of all four tanks were clean and shiny with no anomalies, other than the faint "watermarks" already discussed. There was no evidence of corrosion, or decomposition products or other foreign material on the tank surfaces. Even the spot on the interior of the outlet fitting of tank S/N 5, later found to be penetrated with corrosion, was clean and shiny in appearance. The inside surfaces of all flange covers were likewise clean. The exposed surfaces of the soft metal (aluminum) sealing rings in the flange to flange cover were somewhat frosted with the light powdery corrosion product usually obtained from atmospheric corrosion of aluminum. Aside from this very minor effect, the flange and sealing area were quite clean and unaffected by the propellant exposure.

B. METALLOGRAPHIC AND MECHANICAL PROPERTIES OF TANK SHELLS AND WELDS

In order to verify the general quality of these tanks and insure that no corrosion degradation or other unusual long term storage effect has taken place, all critical welds and representative base metal samples have been examined metallographically. In addition, tensile properties were determined from the cylinder section and across one of the girth welds of each tank as a further study of the condition of the tank wall material.

The combination of mechanical tests and metallographic analysis was performed on the four tanks sectioned: S/N 1 of AM350 stainless steel, and S/N's 3, 4 and 5 of 17-7 PH stainless steel. All four of these tanks show properties which can be favorably compared with typical manufacturers literature or handbook values for the appropriate material conditions. In addition, none of these tanks show any unusual or anomalous microstructure conditions in either weld or base metal. Nor do any of these tanks show any evidence of corrosion attack, except for the corroded outlet on tank S/N 5 which has been described in previous Section V.A, and is covered in detail in Section V.C.

The results of the tensile tests, three tests each of the parent metal in the cylinder section and across the cylinder to dome girth weld, from each of the four tanks, are shown in Tables III through VI. The AM350 tank, S/N 1, in Table III shows properties indicative of the cold rolled and tempered (CRT) condition of that alloy. It is not clear from the various Martin reports on the fabrication of this type of tank, exactly what temper and processing history was used. For either the AM350 CRT or AM350 SCT 1000 conditions, the results are reasonable and indicate no degradation due to either corrosion or the long term storage. The transverse weld tests show substantially lower strengths, particularly yield strength. However, these yield strengths (94,000 - 97,000 psi) are still well above the design yield strength of 75,000 psi used for this material (Reference 5). The sharp drop in properties is typical of what will be expected for welds used in the as-welded condition and again indicates the lack of any apparent corrosion effects.

The tensile properties obtained from the three 17-7 PH stainless steel tanks, S/N's 3, 4 and 5, are shown in Tables IV, V and VI. These tests again show typical and desirable properties, indicative of the TH1050 sheet metal condition and as-welded weldment condition which are expected from the fabrication history of the tanks, Reference 5. The very high sheet metal strengths 200,000 - 208,000 psi yield strength and

215,000 - 220,000 psi ultimate strengths are somewhat higher than the usual typical values (185,000 psi yield and 200,000 psi ultimate strengths) but are within the expected spread of data. No evidence of material property degradation was seen. In the same way, the transverse weld tests of as-welded material from tanks S/N 4 and 5 show typical values which meet the 80,000 psi yield strength design criteria of Reference 1, and indicate no degradation.

The properties across the girth weld of Tank S/N 3, as shown in Table IV, were somewhat lower than the properties across the weld in Tanks 4 and 5. This is believed to be due to the fact that Tank S/N 3 was rewelded, which would cause additional heat to further "anneal" the parent metal on either side of the weld. This heat affected, edge of weld region is where the tencile fractures occurred. Thus, the properties obtained represent this region, and the extra heat affects have caused the 10,000 to 15,000 psi drop in yield and ultimate strengths. This weld is the one which contained the "suspect" areas as marked by RPL. These areas are discussed in detail in Section V.C. under Detail Metallurgical Analyses of Anomalies. It is evident that some of the effects and appearances which caused these areas to be suspect, are probably due to the rewelding of this girth weld.

The microstructure of the AM350 sheet metal in the cylinder and dome portion of tank S/N 1 is shown in Figure 16. No unusual conditions are seen, with the most pronounced feature at lower magnification being the islands or stringers of delta ferrite which are typical of this alloy. The higher magnification views in Figure 17 show only the extremely fine and unresolvable transformed or strained matrix of tempered martensite. Sample edges are shown in Figure 16 to illustrate that there was no observable surface attack of either the internal, hydrazine exposed or outer, atmosphere exposed surfaces of the tank. In addition, it can be seen that there was no difference in apparent microstructure between the cylinder section which is simply rolled from sheet, and the dome which is explosively formed to the desired ellipsoidal shape.

The three 17-7 PH stainless steel tank sheet metal component microstructures are shown in Figures 18 through 23. As with tank S/N 1, views are shown in Figures 18, 20 and 22 which include both interior and exterior surfaces of both components to illustrate that no corrosion effects either from hydrazine or atmosphere could be seen. The higher magnification views in Figures 19, 21 and 23 show the typical and desired structure, a matrix of transformed and precipitation hardened martensite with delta ferrite stringers present. As with the AM350, the structure of the transformed martensite with precipitated intermetallic phases is too fine to be resolved with optical microscopy. It is evident, however, that the typical and desired microstructure has been achieved. Visible in the photomicrographs of Figures 18 through 23 are also a number of massive carbides and other inclusions. These particles are typical and normal for the alloy and do not represent any unusual condition.

In the case of tanks S/N's 3 and 5, there was some difference evident between the cylinder, formed from rolled sheet metal, and the dome, explosively formed. The explosively formed components view (b) in Figures 18, 19, 22 and 23, showed a consistently darker etching characteristic. This would generally be the result of cold work or strain in the matrix, which is logical to expect in the explosively formed dome.

Equally important with the sheet metal components are the quality and structure of the welds joining the components to form the tank. Metallographic sections of representative welds are shown in Figures 24 through 31. The girth and dome to outlet or flange fitting welds shown in these views exhibited a variety of configurations from smooth welds with reasonable crown and drop-through (view (b) of Figure 30) to badly mismatched and crowned welds such as shown in view (c) of Figure 24. All of the configurations, even those with serious mismatch, showed sound welds with good fusion of the weld bead to the adjacent parent metals.

The flange and outlet components in each tank were machined from thick forgings and contained a very coarse structure, indicative of the deformation that such processing conditions achieve. This coarse structure is shown in Figures 27, 29 and 31. Even where the welds met this coarse, nonoptimum structure, no defects or unusual conditions were noted.

All welds were closely examined for evidence of corrosion on either the internal, hydrazine-wetted surface or the external atmosphere exposed surface. No evidence of corrosive attack was found. Views are shown in Figures 25, 27, 29 and 31 from the edge of weld location for welds from each of the four tanks sectioned. This location is considered to be most susceptible to corrosive attack, because of the sharp gradient in structure and thermal exposure in this region. It was evident that there was no unusual attack of any of the tank welds or adjacent heat affected zones in these tanks.

In addition to the welds in the shell portion of the tanks, the welds connecting the various portions of the inlet and outlet tubes and associated fittings were sectioned and evaluated. All tanks contained the same tube configuration; a short length of AISI 321 stainless steel tube welded to the flange cover or outlet fitting, and then that tube welded to one side of a MS27856 connector. These fittings, developed by AFRPL, use a chevron-shaped ring, sealing element between the connector fittings, and were used in this program to obtain long term storage and leak tightness information on this connector, which has potential for improved sealing in manifold portions of liquid rocket systems. On the other side of the MS27856 fitting another short length of stainless steel tube is welded. This segment of tube terminates in a standard tube fitting and plug. Since this was standard hardware, and showed no anomalies or corrosion effects, none of this hardware was evaluated.

The tube-to-tube or tube-to-tube fitting welds were produced by the "Astro Arc" process. This is an automatic tungsten inert gas (TIG) welding process in which an electoode is automatically circled around the joint periphery. The compact size of the unit and the fact that the electrode rather than the tube turns, makes this process particularly useful for connecting tubing in complex manifolds. The joint is made without having to turn the tubing or otherwise disturb it.

The welds produced by the Astro Arc process in stainless steel tubing are usually quite sound and of high integrity. Examples from Tanks 1 and 5 are shown in Figures 32 and 35, which demonstrate the sound, fully fused and penetrated nature of the typical tube welds. There is no corrosion or other unusual effects in these welds.

One tube weld in the S/N 4 tank was found to be misaligned and hence only partially penetrated (Figure 34). Although the weld fusion extended through the entire 0.065" wall thickness, the center of the weld was at least 0.07 inch from the original joint line. Normally this type of defect is easily seen in radiographic inspection of the joint and would not be expected to reach deliverable hardware. The lack of penetration is approximately one-third of the joint thickness and on one side terminates as a sharp line. Even with a substandard quality weld, there is no evidence of corrosion or structural damage. The other tube welds of Tank S/N 4 were normal, high quality welds which also had no indication of corrosion or other damage.

The welds joining the tubing to the flange cover or outlet fittings in each tank involved the tube being inserted within a machined boss region and a fillet weld joining them. This causes an inevitable crevice between the tube and the inner diameter of the boss, and also raises the possibility of not achieving full fusion between the fillet weld and parent members. No such difficulties were seen in any of the tube to tank welds. A typical pair of welds from Tank S/N 1 are shown in Figure 33. They illustrate the deep crevice, exposed to the tank interior, of this tube to boss weld design. Even in this region no corrosion or deposits, indicative of hydrazine decomposition, were found. The weld penetrations into the tube or boss were shallow but adequate to insure a well fused, leak tight joint.

The coarse, somewhat banded structure of the flange cover and outlet fitting is evident in Figure 33. This was observed in all tanks and represents the consequence of using heavy forgings for these components. This coarse structure caused no difficulty in welding even though in some situations it would be expected to be deleterious. Except for the corrosion on the exterior of the outlet fitting of Tank S/N 5 (discussed earlier), no corrosion was found on the interior or exterior of any of these components.

The segment of the outlet tube from Tank S/N 5 which was missing from the original shipment was evaluated. The tube segment is immediately adjacent to the corroded outlet fitting and there was hope that evaluation of this component would aid in understanding the corrosion in this region.

When matched with the adjacent fitting, Figure 11b, the tube is seen to be only very lightly etched in the portion of the periphery adjacent to the corroded fitting surface. The bulk of the tube is completely unattacked. Even the streak which is etched is only very lightly attacked with no significant depth to the attack.

The tube to connector fitting weld just below this corroded and etched region was sectioned and examined. The weld was sound and fully fused, although it did exhibit a slight amount of root concavity, as shown in Figure 38. The amount, less than 0.010 inch would not be considered rejectable in normal production hardware. There were no cracks, defects or other anomalies that might indicate any leakage through this weld during or after storage exposure. Detail views of the outside and inside surface of the weld at an edge of weld location are shown in Figure 38. These views illustrate that there is no corrosion attack of either the inner, hydrazine exposed surface, or the outer, atmosphere exposed, surface.

In the examination of the tubing at the flange end of Tank S/N 5 (the tank with corrosion penetration in the outlet fitting and which was partially sectioned by RPL), considerable brown or reddish brown residue was deposited around the interior of the tubing. This is discussed in more detail in Section V.C as part of the Detail Metallurgical Analyses.

C. DETAIL METALLURGICAL ANALYSES OF ANOMALIES

The specific anomalies selected for detailed analysis were based on the initial visual examinations performed on all tanks and on discussions with the AFRPL Project Officer. Although only one anomaly represents an actual corrosion penetration of the tank wall, all four represent unusual conditions warranting further investigation. All four anomalies have significance in the handling and long term storage of Air Force liquid rocket propulsion systems. The specific anomalies are listed below and are then considered individually in the succeeding section:

1. Leak area in outlet fitting of tank S/N 5.
2. Deposits in flange tube fittings of tank S/N 5.
3. Suspect areas marked by RPL on girth weld of tank S/N 3.
4. Sealing surfaces and soft metal seal ring on flange and flange cover of tanks, particularly S/N 3.

1. LEAK AREA IN OUTLET FITTING OF TANK S/N 5

a. Test History

Tank S/N 5, as with the other three tanks in this study, had been stored with hydrazine for over four years. During this time, particularly the first year of storage, there were several instances of pressure buildup which required that the tank be vented to reduce the pressure. No pretest leakage was reported, nor was there any report of leakage (fuming, pressure loss or liquid puddles) during the early stages of storage test. During the final years of test, surveillance was less rigorous and there are no specific records of test behavior. The tank was removed from the storage test and drained more than one year before evaluation was performed. All tanks in this group were reported to have leaked, generally "around bolt hole". As is discussed in greater detail in analysis #4, there is some chance that this leakage report was based only on the presence of fuel on the floor in the general vicinity of the tanks. There was sufficient concern about leakage in this tank to start a metallurgical investigation at Edwards Air Force Base.

The tank was shipped to BAT from RPL where a preliminary metallurgical examination had been initiated. The area of concern to Edwards Air Force Base personnel was an area of attack on the small outlet fitting and adjacent fitting to tube weld. This area of attack was on the outside of the tank, on the fitting referred to as a "Beanie" in the Martin fabrication report, Reference 5, and in Section IV. This "Beanie" had been removed from the tank by Edwards personnel and sectioned near the area of attack in a region that was obviously quite thinned. A very small triangular segment was removed in the preliminary Edwards evaluation, however the study was interrupted, and the tank was shipped to Bell for further analysis.

b. Observations

1. The major area of concern in this investigation is shown in Figure 11, where view c shows a lightly etched area on the outlet extending down to a pitted region on the flat portion of the fitting. The outline and progression of this etched and pitted region seemed to indicate that a corrosive liquid dripping onto the fitting had collected in a puddle in the depression produced by the surrounding fitting to dome weld. As will be discussed in more detail in the Metallurgical Analysis which follows, this collection of liquid for eventual pitting could only occur when the tank was inverted from its normal, flange-up, test storage position. An indication of the serious nature of the pitting in this area is shown in views c and e of Figure 36.

2. In this detail metallurgical evaluation of the outlet fitting, particular attention was paid to the outlet tubing components shown in Figure 11, view a. In this view components of this outlet from tank S/N 5 are compared to tank S/N 1. One section of tubing from S/N 5, missing from the original shipment, was received later and is shown in view b of Figure 11. Etched areas indicative of a slight surface attack are marked with black arrows on this tube. This tube segment adjacent to the corroded outlet fitting was examined carefully for leaks and corrosive attack. The tube was found to be only very lightly etched in the portion of the periphery adjacent to the corroded fitting surface. The remaining length, the interior of the tube and the connector fitting were completely unattacked. The area where etching did occur on the outside surface was only very lightly corroded with no significant depth to the attack. This tube segment therefore indicates that the etching that did occur was the result of runoff of a corrosive media in this area.

3. Examination of the outlet fitting showed that it was quite thin, even in the areas not attacked by the corrosive media. These areas were measured at 0.008 to 0.012 inch thickness, compared to a nominal expected thickness of 0.030 inch, which is the thickness of the explosive formed dome that mates with this fitting. This thinned nature of the fitting is seen in Figure 36, view b. Only the area immediately

around the pit visible in view b was thinned by pitting corrosion, the smooth but thinned wall of this fitting was evidently due to mismachining of this fitting. Shown in view d of Figure 36 is the corresponding fitting area from tank S/N 4. This component was also thinned, but only to a thickness of 0.015 to 0.020 inch. The outlet fitting from tank S/N 3, the other 17-7 PH stainless steel tank in this group, was not thinned at all, having a minimum thickness of 0.025 to 0.030 inch.

4. The interior surface of the outlet fitting was clean and bright with no evidence of attack or pitting. The area immediately under the pitted exterior was closely examined, and a crack-like discontinuity was found under the deepest portion of the pit. This is shown in view a of Figure 36.

5. Metallographic examination of the outlet fitting in the pitted region showed the pitting to be quite deep, and to penetrate the wall in one spot. This is shown in the views of Figures 36 and 37. The external pitting is seen to be quite extensive around the deeply pitted area, view a of Figure 37. When the metallographic samples are examined in the etched condition, Figure 37, the coarse and oriented nature of the ferrite stringers are quite evident. This outlet fitting is machined from a forged block and therefore has much coarser microstructure than the sheet material used in the shell. The

ferrite stringers were in general not attacked in the pitting, as the corrosive medium attacked the transformed martensite matrix more readily. In some locations the initial attack was seen to concentrate on the ferrite to martensite phase boundary, as can be seen in view c of Figure 37. The corrosion resisting chromium alloying element will tend to be more concentrated in the ferrite than the austenite which transforms to martensite, so the slightly better corrosion resistance of the ferrite is understandable.

6. The tube-to-tube connector weld just beyond the pitted and etched areas was closely examined to determine whether any possible leak or anomaly in this region could have contributed to the corrosion observed. No leak, anomaly or even corrosion of this weld was found, on either the exterior or interior. The weld was sound and fully fused, as shown in view a of Figure 38. It did have a slight root concavity but this is often encountered with these welds and does not degrade the weld. Close examination of the inside and outside surface of the weld, view b of Figure 38, showed no evidence of any corrosion in or near this weld.

c. <u>Metallurgical Analysis</u>

The above observations have shown clearly that the corrosive attack on this outlet fitting was started from the outside and proceeded through pitting and intergranular attack to form a deep pit which completely penetrated the fitting wall. The fitting wall at this point had been mismachined to produce a wall thickness of only 0.008 to 0.012 inch, and therefore much less corrosion was required to produce the complete penetration than would normally have been required.

The orientation and configuration of the pit and surrounding etched areas indicate that this attack occurred when the tank was not in its normal storage, flange upright, condition. Instead the attack was most likely when the tank was inverted, which would allow the corrosive medium to collect in the depression formed in the area surrounded by the fitting to dome weld, which was mismachined and hence had a somewhat dished shape.

The corrodant which caused this pitting and attack has not been identified. Because it does not seem likely that the corrosion occurred during normal storage, it is particularly difficult to predict the exact corrodant. A rather strong acid is generally required to produce this type of attack in 17-7 PH stainless steel. The most likely possibility is hydrochloric acid, since it is most often associated with pitting corrosion in these stainless steels. This acid is quite prone to cause pitting in dilute solution, and could have been generated by the hydrolysis of vapors from residues of ClF_3 or ClF_5 propellants in other tanks or hardware placed in the same holding area as this tank after its storage test. Although details of this holding area are not known, there was a time period of over one year between removal from test and metallurgical analysis.

Lending further support to this hypothesis that some corrosion process occurred after completion of the storage test is the analysis which follows in Item 2. This analysis concerned corrosion and deposits which were found in the tubing of the flange end of this same tank S/N 5. This analysis of deposits in the tube also shows that the orientation of the deposits could only have occurred when the tank was no longer in its normal, vertical and flange-up orientation.

2. CORROSION DEPOSITS IN FLANGE TUBE AND FITTING OF TANK S/N 5

a. Test History

The storage of S/N 5 has been discussed in the previous section. For reasons unknown to BAT, this particular tank (S/N 5) was subjected at some period either during storage (4 years) or after storage to conditions which differ from the other two tanks fabricated from 17-7 PH (S/N's 3 and 4). The above statement is supported by the fact that for reasons unknown to BAT tank S/N 5 showed corrosive attack while S/N's 3 and 4 did not. Although S/N 3 was suspected of leaking, no leak areas were found and corrosion products existent in the inlet and outlet fittings of S/N 5 were nonexistent in tanks 3 and 4. Weld areas of S/N 3 previously marked by Edwards Air Force Base did not leak even after 30 days submerged exposure under pressure.

b. Observations

In the flange end tube of S/N 5 corrosion product deposits and some attack were found inside the tube. An over-all bronze color existed throughout the length of the fitting. A weld exists in the central portion of this tube where the 347 stainless steel RPL tube connector fitting is joined to 321 stainless steel tubing. Spectrographic analysis confirmed the materials to be those called for in the Martin fabrication report, Reference 5.

The corrosion deposits were most pronounced in two areas, (1) the central portion of the weld and (2) in an area closely adjacent to the weld on the inside surface of the 321 stainless steel, Figure 39. These deposits suggest puddles of liquid dried in these two areas. Corrosion products from this area were removed and placed on a beryllium plate and a microprobe analysis was made. The elements found as being a part of the corrosion products in this area were iron, chromium, nickel and oxygen with trace amounts of manganese. The iron, chromium and nickel were in the approximate proportions for stainless steel. It is evident that the deposits are the result of stainless steel corrosion and resultant oxidation. No evidence of chlorine could be found. Figure 39 shows views of the inside of this tube. View c of Figure 39 shows a cross section through the weld, particularly the concave root area which provided a collection point to hold liquid when the tank and its tubing were lying horizontally on the side.

View d of Figure 39 shows a high magnification view of the inner surface and subsurface region of the weld at the location where the corrosion deposit was found. Shallow, but significant, corrosion of the stainless steel weld metal and edge of weld can be seen. This corrosion, to a depth of 0.0008 inch or less is not sufficient to cause structural degradation to the tube and tank integrity, but is sufficient to produce the volumes of corrosion product noted, particularly when it is collected in puddles from a larger area before evaporation and deposition.

A compression seal used in the same fitting, tightened to the flange surface of this tube with a three-quarter hexagon nut, was also found to exhibit these deposits. Once again this condition was not found in any other tank in this program, neither at the inlet or outlet end of these tanks. The compression fitting is common to all tanks being part of the special RPL connectors used in the tubes of all of these Martin tanks. Sectioning of this compression fitting exposed similar products of corrosion as those found in the tube itself, Figure 40, views b and c. These deposits also are characteristic of the dried residue of small drops of liquid in the tube. Once again a microprobe analysis was run on the corrosion products. Elements found were iron, nickel, chromium, a trace of manganese and phosphorous. The corrosion products were reddish brown to dark brown with areas containing lighter brown streaks.

In a further effort to define the corrosion products, X-ray diffraction analysis was performed on a tube section with the corrosion products in place. The patterns obtained on the tube showed only definite patterns for austenite to be present. It was not possible to identify any characteristic peaks for the corrosion product. Likewise, an X-ray powder diffraction pattern of scrapings of the corrosion product only showed no definite peaks. It is assumed that the corrosion product was deposited as an amorphous film, rather than any easily identified crystalline product.

c. Metallurgical Analysis

The initial observation of these deposits in the flange end tubing of tank S/N 5 prompted the possibility that they represented the decomposition products of hydrazine, since they were in the upper, vapor phase portion of the tank. However, electron microprobe and X-ray diffraction techniques identified only those elements and phases associated with corrosion or oxidation of austenitic stainless steel, which is the composition of the tubing and associated tube fittings and connectors. No elements or phases that could be attributed to decomposition products of hydrazine were found.

These deposits observed are the result of corrosion of the adjacent tubing, weld and fittings, and perhaps minor corrosion in other portions of the same tubing lines, collected in one or two locations by "puddling" and eventual evaporation of the corroding medium. The orientation of the two major dried "puddles" in the tube and weld make it obvious that they corroded and dried in these locations when the tank was no longer in the vertical position. The puddles had to have been formed when the tank and tubing were lying on their side.

The source of the corroding medium is difficult to pin down. One possibility is the hydrolysis of vapors from hydrazine which can form hydrochloric acid from the minor amount of chloride impurity in hydrazine. The lack of any chlorine element being found in the deposits by the microprobe analysis makes this possibility remote. Another possibility is that water was either accidentally or deliberately introduced into the tank during draining and purging after hydrazine exposure. This water, collecting in puddles for the extended time between draining and the start of evaluation, could have caused the observed corrosion if it had picked up acid impurities from this or other tanks being drained after propellant storage test.

3. SUSPECT AREAS ON GIRTH WELD OF S/N 3

a. Test History

This particular tank was marked by Edwards Air Force Base as being suspected of leaking at/in the girth weld. If one refers to the log book on this tank, the following sequence of events took place after fabrication and before and during the exposure period. The sequence of events follow that reported in the log:

1. Pressure checked by Martin at 150 psi.

2. Clean and passivated by Martin on 29 January 1969.

3. Mass spectrometer checked at 70 psi helium on 4 March 1969. (This and subsequent operations at RPL.)

4. SF_6 checked in plastic bags 14 March 1969. (Leaked)

5. Four small leaks were found during the above examination which were marked, and then the tank was put into test and found to be satisfactory.

6. On 10 November 1969 the tank pressure was 100 psi at a temperature of 105°F, and the tank was vented to 50 psi.

7. On 17 November 1969 the tank pressure was 102 psi at a temperature of 110°F and the tank was vented to 50 psi.

8. On 18 November 1969 the tank pressure was 105 psi at a temperature of 112°F; the tank was vented to 50 psi.

9. On 26 November 1969 the tank pressure was 105 psi at a temperature of 112°F; the tank was vented to 50 psi, drained and removed on 26 May 1970.

10. On 4 November 1971 the tank was reloaded and put back into storage. This was true of all tanks and was required due to maintenance required on the building.

11. In February 1975, this tank (S/N 3), plus the other three tanks in this group, was drained of hydrazine and removed from service. The welds had been marked where leakage

was suspected in the prestorage testing, and one bolt hole on the flange inlet end of the tank was also marked as being a suspect leak area.

Macroscopic examination at BAT, radiographic inspection and dye penetrant inspection did not reveal any gross leak paths. After these investigations were conducted, BAT replaced the flange cover, sealing off the outlet end with a plug and immersed the tank in a water bath under 20 psi of helium. This leak test was performed for a total exposure of 36 days. After six days of immersion, the tank was reexamined and once again no leakage was observed. The test was then continued under helium pressure for thirty days and the result was negative, no leakage at the flange or in the welds was found. This test takes on greater meaning when one considers the fact that these flanges contain a sealing ring made from aluminum and are not considered reusable.

Since the flange cover had originally been removed at Edwards Air Force Base prior to shipment to BAT, an effort was made to determine if torque values on the flange bolts were measured prior to removal of the flange. These efforts, conducted jointly with Edwards Air Force Base, have revealed that torque values were not obtained. In addition, the original bolts holding the cover were no longer available.

b. Observations

The S/N 3 tank is shown in its as-received state in Figure 8. The girth weld areas marked at Edwards Air Force Base are plainly visible. These areas were examined in depth and BAT analysis on a macroscopic scale showed irregular protrusions of the weld crown and weld drop-through to be present.

Close-up views of the exterior or crown of the girth weld are shown in Figure 41. Views a and b of this figure show the appearance of the weld on a macroscopic scale where oxides or slag are present. At first it was thought that this scale and the manner it existed on the weld surface was indicative of a repass weld or second bead being made. The inside or underbead surface of the same weld areas are seen in Figure 42. The surface oxide had broken and solidified in an irregular manner which contributed to the discontinuous appearance of the weld.

Cross sections of this weld did not reveal a second pass. These views are shown in Figures 26 and 43. The overall shape of the weld is depressed, however, the weld is sound and shows that good fusion and dilution occurred. The higher magnification views in Figure 43 show no evidence of discontinuous freezing pattern which would indicate a second pass. As described in Section VB, tensile properties across this weld did indicate a reweld, from the somewhat lower strengths compared to tanks S/N's 4 and 5.

Further examination of this weld in which a view near the midplane of suspect area 1 was made by polishing down on the face of the weld is shown in view a, Figure 44. View b of the same figure is a vertical section along the centerline of the weld in the area marked 4. No leak paths or other anomalies were found in either of these sections which encompassed the entire area marked in each case. Although there were isolated instances of microporosity seen in the sequential grinding and polishing through these two suspect areas, no interconnection or leak path was found. Isolated microporosity is common in the detail examination of most welds.

A positive of a radiograph taken in these areas is shown in Figure 45. Actually this radiograph was made in the early stages of the investigation of this weld. The radiograph only substantiates the irregular geometry of the weld and when first examined gave the impression that drop-through and some weld bead undercutting could have been present. All further studies emphasized an irregular pattern, no undercutting and sound fusion. Microstructurally no area was found which would indicate the presence of a preexistent leak path.

c. Metallurgical Analysis

The efforts to discover a leak in S/N 3 are fully discussed in Sections a and b above. In all of the examinations made by BAT no leak path was discovered either in the girth weld or the flange. Although there is no proof other than the irregular shape of the weld and leftover slag deposits, it is surmised that somewhere in the history of this tank a reweld was made in the areas marked as suspect leak sites 1, 2, 3 and 4. BAT could not confirm this suspicion metallurgically.

4. SEALING SURFACES AND SOFT METAL SEALING RING ON FLANGES

a. Test History

The tanks being evaluated in this program were removed from storage test with the note "Leaked Around Bolt Hole" in three out of four cases. More than a year elapsed before these tanks were specifically committed for evaluation under the current BAT contract. Because of the extended time period, it was difficult to get specific verification of the nature of the leakage, and there was some possibility that not all of these tanks actually leaked. It may be that puddles of liquid under a tank were taken to indicate leakage of that tank, when the puddle may actually have come from a neighboring tank or from a manifold leak not strictly associated with any tank. However, the comments on the tank identity tags did represent a serious anomaly that required investigation.

The bolt holes referred to in the tags are the circle of fourteen bolts used to attach the flange cover to the flanged opening of the tanks. As described in the Martin fabrication reports (References 5 and 6), these openings were designed into these tanks (as they are in almost all flight hardware) to allow for internal fixturing of the girth welds, visual and radiographic inspection of welds, and in the case of some tanks, for inserting and servicing any positive expulsion devices. The end fitting containing the flange was machined from a forged block of 17-7 PH stainless steel and contained the typical heavy walls and built-up corner sections to resist bending loads and provide a stable surface for cover attachment and sealing. The cover plate was machined from 17-7 PH stainless steel plate. The soft metal sealing ring was of aluminum, 1/4 inch wide and 0.1 inch thick, which fit into a machined groove in each member.

After receipt at BAT, there was concern about the reported leakage and about the suspect areas in Tank 3, discussed in the preceeding section. Therefore, tank S/N 3 was reassembled with its flange cover and leak tested with helium pressure for first six, and then thirty days. In both cases, leakage was inspected for by both monitoring gage pressure and also completely immersing the assembled tank in a water bath. No gage pressure drop or formation of any tank surface bubbles was found in either of these tests that would indicate any leak path in the tank or associated cover plate or inlet/outlet tubes.

b. <u>Observations</u>

An overall view of the flange of tank S/N 3 with the aluminum sealing ring in place is shown in Figure 46. The corresponding view of the flange cover sealing side is shown in Figure 47. These views were taken after the 30 day water immersed leak test. Slight staining of the stainless steel and of the aluminum surfaces exposed to the water had occurred, but these are not readily apparent. Close inspection of the groove and sealing ridges in the flange cover showed the groove to be reasonably smooth with only a very slight ripple to the surface at some locations. The ridges were quite smooth and appeared to have an excellent machined surface that should have provided very good sealing with the soft aluminum ring, assuming sufficient clamping force was used. This is shown in Figure 48.

When the aluminum sealing ring was removed from the flange side of the joint, and the groove and sealing ridges on the flange closely examined, they were found to be considerably less smooth and poorly machined. Several areas with chattering and smeared or torn out metal were seen, such as is shown in view a of Figure 49. The areas of tearing on the shoulder and crest of the sealing ridges should be of greatest concern, since it is those ridges bearing into the soft metal sealing ring which produce the leak-tight joint characteristics.

The groove in the flange side contained some "sandy" residue, as seen in view b of Figure 49. This material had been wedged between the inner or outer diameter of the sealing ring and the side of the groove. There was no evidence of it having been pressed between the bottom face of the sealing ring and the bottom of the groove, since the particles would have impressed into the aluminum surface if they had been in that location. No clue could be gained as to whether this residue was primarily wedged between the outer diameter or inner diameter.

Identification of this "sandy" material was attempted through the use of X-ray diffraction powder pattern techniques. A definite pattern for silica, SiO_2 was found. Electron microprobe analysis of other portions of the "sandy" residue disclosed particles high in calcium and oxygen, with trace amounts of magnesium, aluminum and potassium. A likely source of this very fine "sand" would be wind blown particles in the desert environment of Edwards Air Force Base. The flange to cover gap would be a likely location for trapping wind blown particles. These particles are rounded and have occasional irregular, foreign particles mixed in. These are probably the calcium containing particles found by microprobe and may be gypsum or some variety of limestone, common in desert areas. These particles had no effect on the sealing capability of the flange and cover.

The soft aluminum sealing ring itself was also closely examined. Around the outer periphery of the ring, where it had been in contact with water for the extended leak test, some minor staining was present. This is seen in view a of Figure 50. The side of the sealing ring that was against the flange had received no handling or disturbance in all of the holding and evaluation time since hydrazine exposure testing. This surface of the sealing ring showed a definite staining of the inner portion of the ring, from the inner diameter to the first sealing ridge. This would be the region exposed to hydrazine during the over four years of storage testing. This staining is shown in view b of Figure 50. No significant corrosion depth was associated with either this hydrazine staining or the water induced staining discussed above.

The depressions formed in the aluminum ring by the sealing ridges of the flange and flange cover were examined for sealing capability. The locations where chattering and tearing of the ridges in the flange had been found showed corresponding disturbance of the aluminum. An example is seen in view c of Figure 50. This flow of the aluminum into and around torn and roughened regions of the sealing groove provides the capability for good sealing even when the flange and cover are in less than ideal machined condition. As long as sufficient sealing force is applied and the chattering or tearing does not leave long paths of very deep or hidden crevices, the soft aluminum sealing ring should be able to provide adequate sealing.

A metallographic section through the flange in the region of one of the torn areas disclosed no material anomalies to explain the poor machined surface at that point, Figure 51, view a. The microstructure of the flange is relatively coarse, being machined from a large forged block of 17-7 PH stainless steel, and is shown in Figure 51, view b. There was no evidence of any site for leak paths through the flange material. Although the ferrite stringers are oriented perpendicular to the sealing surface, the flange in this region is over one-half inch thick, and it is extremely doubtful that any leak path could be formed, even along the ferrite stringers that are present.

c. Metallurgical Analysis

As mentioned in the test history, the examination of the flange seal was deemed important, even though no leak was directly observed in the extended leak test of one of these tanks. The experimental observations showed occasional evidence of poor machining of the sealing grooves and ridges in the flange and flange cover. This had been observed by the manufacturer, Martin-Denver, and reported in the fabrication report, References 5 and 6, as the cause of a few instances of failure to meet the leak test requirements in the as-fabricated condition. According to the fabrication report, rework of the sealing groove and ridges allowed any tank which had failed leak test to become acceptable.

It is evident from evaluation of this tank that some instances of poor machining did not cause any leak test difficulty. This is undoubtedly due to the soft aluminum seal being able to flow and fill any torn regions. The ability of the aluminum to flow and seal small crevices and torn regions will be a function of the applied force from the flange bolt tightening and the microgeometry of the crevice. It seems likely that leaks would be found in the as-fabricated condition at this seal only when the bolt tightening was not sufficient (or nonuniform) or when some unusual crevice geometry is formed.

The original sets of flange bolts from these tanks were not saved after draining and disassembly of the tanks, nor were the loosening torque values recorded. Therefore, it is impossible to evaluate whether the bolts loosened or stretched due to long term creep, the moderate temperature excursions known to have occurred or from vibration or corrosive effects. When fresh bolts were used to reseal tank S/N 3, no leakage was found. This occurred despite the fact that these metal sealed joints are not generally considered reusable.

It seems most likely to expect that the reported "leakage around the bolt hole", if there was in fact leakage, was probably due to loosening of the bolts due to some long term storage effect. Future evaluation work in support of this "Long Term Storability of Propellant Tankage" program should be planned to investigate the final tightness and long term stability of bolts used in the closure of tanks such as this. This item is also discussed in the Recommendations which follow.

SECTION VI

CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

The detailed analysis performed on these four tanks fabricated from two grades of precipitation hardenable stainless steels exposed to hydrazine for at least four years are as follows:

1. There was no evidence of a basic incompatibility existent between either the AM350 tank or the three 17-7 PH tanks with the stored propellant hydrazine.

2. Corrosion was observed in only one of the tanks, S/N 5 (one of the 17-7 PH tanks), and this corrosion was confined to the outlet fitting and inlet tubing and fitting. Since the fittings on the other tanks showed no corrosive effects, it is concluded that the corrosion observed occurred after the exposure period, and was the result of residue propellant vapors that hydrolyzed prior to the metallurgical examination. Hydrolysis of hydrazine could have produced a dilute hydrogen chloride environment which attacked the martensite structure of the stainless steel.

3. The corrosion of the outlet fitting resulted in the complete penetration of the wall by pitting from the exterior. The wall in this area had been thinned by mismachining to 0.008 to 0.012 inch, so less corrosion was necessary than would normally be expected. Corrosion inside the tubing at the opposite end

of the tank (flange end) produced only slight attack of the tube wall (less than 0.001 inch deep), but did have corrosion product deposits that would be sufficient to cause cleanliness difficulties if the tank and tubing were to be reused.

4. Microprobe and X-ray diffraction analysis of corrosion products within the tube did not reveal the source of the corrosion but did establish that corrosion of a heat treated martensite structure occurred. The corrosion was selective in that the delta ferrite present was not attacked, while the martensitic matrix was. This selective attack was in evidence in the metallographic studies.

5. Although chlorine was not found in the corrosion products, the surface attack and pitting that occurred are indicative of dilute acid attack.

6. The source of the propellant vapors could not be precisely identified. The following areas can be considered suspect:

a. Leakage at valves or fittings.

b. Residual propellant liquid or vapors left inside the tank.

7. Detailed analysis of the flange cover seals found no explanation of the leakage at these locations other than the possibility that fittings and flange bolts were not periodically checked to insure that torque values were maintained over the prolonged storage period. It is concluded that to some degree

creep relaxation could have occurred which would allow for vapor or liquid leakage to occur.

8. Although one tube weld was found with a misaligned weld which gave incomplete penetration, the tube welds from all tanks except S/N 5 were sound with no evidence of leakage, corrosion attack or other anomalies. The tube to flange or tank outlet welds, although containing a deep crevice, which could be a source of corrosion difficulty, showed no corrosion effects or other anomalies, except on the S/N 5 outlet previously described.

B. RECOMMENDATIONS

Based on the analysis made on the four tanks submitted, one fabricated from AM350 and three fabricated from 17-7 PH stainless steel, the following recommendations for future programs, both long term storage programs and Air Force production systems utilizing these types of tanks are made:

1. Thoroughly inspect all welds in the storage system to verify quality, pressurize system with helium gas and use leak detector to establish pressure tightness of system prior to propellant loading and storage.

2. Isolate each storage system to prevent externally induced corrosion effects produced by hydrolysis of leaking propellant vapors or liquids.

3. When tanks are removed from service and are to be stored for some period of time, it is imperative to drain and purge the tank and associated fittings of all propellants. In this program vapor or residual propellant left in fittings caused more corrosive damage than actual storage with hydrazine.

4. Monitor the storage building environment to detect presence of leaking propellant vapors; or install liquid level gages on each storage tank to detect propellant liquid loss.

5. Thorough draining and flushing operations should be performed on propellant tanks after long term storage if they are to be reused. Complete drying and sealing of the vessel in a dry, relatively airtight container should preclude post-storage corrosion effects. This recommendation cannot be overemphasized, since BAT has not found corrosion of internal tank surfaces regardless of the propellant stored. Corrosion found in the past two programs and in this program has been associated with fittings and has been of a nature which indicated that it occurred after the actual storage period or was outside surface attack due to the environment.

6. It is also recommended that all fittings and flanges be checked for the required torque values on a periodic basis to insure that leakage from this source is eliminated or positively identified.

7. When leakage is suspected at or near flange closures upon removal from test, the breakaway torque to remove closure bolts or fittings should be carefully measured. This is the only positive manner to determine whether loosening of nuts or creep of the bolt has occurred during storage service or test.

SECTION VII

REFERENCES

1. Technical Report AFRPL-TR-74-82 "Analysis of Liquid Rocket Tankage" Final Report, Bell Aerospace Textron, J. Salvaggi, H. G. Kammerer, E. J. King, April 1975.

2. Technical Report AFRPL-TR-75-73, "Analysis of Liquid Rocket Tankage from Model LR58-RM-4 Liquid Propellant Thrust Unit for the Bullpup Missile", Bell Aerospace Textron, E. J. King and J. Salvaggi, February 1976.

3. Technical Report AFRPL-TR-69-82, "Long Term Storability of Propellant Tankage and Components", J. E. Branigan, April 1969.

4. Technical Report AFRPL-TR-72-126, "Long Term Storage Testing of Propellant Tankage", H. M. White, Capt. USAF, December 1972.

5. Technical Report AFRPL-TR-69-112, "Design and Fabrication of Hydrazine Storability Test Tanks", Martin-Denver Co., A. W. O'Brien and C. L. Caudill, June 1969.

6. Technical Report AFRPL-TR-65-194, "Design, Fabrication and Test of Small-Scale Storable Propellant Vessels", Martin-Denver Co., L. D. Berman, January 1966.

TABLE I

COMPILATION OF TANKS RECEIVED AND
EFFECT OF LONG TERM STORABILITY ON PROPELLANT TANKAGE

TANK DESCRIPTION	TANK MATERIAL	PROPELLANT STORED	DATE PLACED IN STORAGE	DATE REMOVED FROM STORAGE	TEST LOG HISTORY	TANK S/N	VISUAL EXAMINATION - 6X MAG: EXTERNAL SURFACE	VISUAL EXAMINATION - 6X MAG: INTERNAL SURFACE
MARTIN 10 GALLON TANK	AM350 Stainless Steel	N_2H_4 (Hydrazine)	Period A: 5/22/69	5/04/70	Period A: Pressure rose above 100 psi at temps. of 110°-120°F four times. Vented back to 50 psi each time.	001	Lightly stained and frosted over much of tank. Brushed clean and bright near welds. Tubes, flange and fitting generally clean and bright.	Generally clean and unattacked. Lightly frosted or etched on sheet surfaces, clean and bright from wire brushing near welds. No deposits or foreign material evident. Faint evidence of "waterlines" indicating tank was partially full, lying on its side at some time.
			Period B: 11/04/71	2/28/75	Period B: No excessive pressure rise, temp. at 140°F.			
MARTIN 10 GALLONG TANK	17-7 PH Stainless Steel	N_2H_4 (Hydrazine)	Period A: 5/22/69	5/04/70	Same as S/N 001 preceeding	003	Same observations as S/N 001.	Same observations as S/N 001.
			Period B: 11/04/71	2/28/75				
MARTIN 10 GALLON TANK	17-7 PH Stainless Steel	N_2H_4 (Hydrazine)	Period A: 5/22/69	5/04/70	Same as S/N 001 preceeding	004	Same observations as S/N 001.	Same observations as S/N 001, except no "waterlines" visible, only a band along one side indicating some residue had evaporated while tank was on its side.
			Period B: 11/04/71	2/28/75				
MARTIN 10 GALLON TANK	17-7 PH Stainless Steel	N_2H_4 (Hydrazine)	Period A: 5/22/69	5/04/70	Same as S/N 001 preceeding	005	Same observations as S/N 001. Tank had been partially sectioned by RPL. Outlet fitting (opposite end of tank from flange) was found to be heavily corroded in a small spot with area around it somewhat etched.	Same observations as S/N 004.
			Period B: 11/04/71	2/28/75				

TABLE II

SUMMARY OF SIGNIFICANT FABRICATION CHARACTERISTICS FOR TANKS EVALUATED

TANK MATERIAL	SERIAL NUMBER	MANUFACTURER	GENERAL TANK DESCRIPTION	COMPONENTS	TYPE OF WELD & FILLER WIRE	HEAT TREATMENT AFTER WELDING	SOURCE OF MATERIAL	FORMING	HEAT TREATMENT	REFERENCE
17-7 PH Precipitation Hardening Stainless Steel	3, 4 & 5	Martin-Marietta Denver	10 gallon cylinder with domed ends; flanged at one end, outlet port at other end.	Barrel	Machine TIG DC current; Argon torch & backup 17-7 filler wire.	None	.040 inch sheet	Roll form single longit. weld.	Solution treat & age after forming to TH1050 condition.	RPL TR-69-112 "Design & Fabrication of Hydrazine Storability Test Tanks" (Ref. 5)
				Dome	" "	None	.040 inch sheet	Explosively formed.	" "	
				Outlet fitting plus flange	" "		Forging bar.	Machining	" "	Previous report (Ref. 2) included more detail on 17-7 PH tank fabrication.
				Outlet Tube.	Tube to flange or fitting manual weld. Tube to tube Astro Arc					
AM350 Precipitation Hardening Stainless Steel	1	Martin-Marietta Denver	10 gallon cylinder with domed ends; flanged at one end, outlet port at other end.	Barrel	Machine TIG DC-SP current Argon torch & backup gas; AM350 filler wire.	None	.044 inch sheet	Roll form single longit. weld.	Subzero cool & age after forming to SCT.	RPL TR-69-112 "Design & Fabrication of Hydrazine Storability Test Tanks"
				Dome	" "	None	.044 inch sheet	Explosively formed.	"	
				Outlet fitting & flange	" "		Forging bar.	Machining	"	
				Outlet tubes	Tube to flange plate or fitting manual weld. Tube to tube Astro Arc					

TABLE III

MECHANICAL PROPERTIES OF AM350
BASE METAL AND GIRTH WELDS

FROM TANK S/N 1 (CRT TEMPER) TEST SPECIMENS WERE REMOVED FROM LONGITUDINAL AXIS OF TANK (A)

SPECIMEN NUMBER	ULTIMATE TENSILE STRENGTH (PSI)	YIELD STRENGTH 0.2% OFFSET (PSI)	ELONGATION % IN 2 INCHES
		BASE METAL	
1.	201,200	168,400	7
2.	200,100	167,900	7
3.	200,300	167,400	8
		WELD METAL (B)	
1.	170,200	94,600	5 (C)
2.	177,000	97,400	6 (C)
3.	177,400	96,600	6 (C)

(A) Since the test bars were removed from the tank's central section along the longitudinal axis of the tank, the properties are believed to represent the transverse direction of the original rolled sheet if the assumed fabrication sequence is correct.

(B) Welds not ground flush.

(C) Broke at edge of weld.

TABLE IV

MECHANICAL PROPERTIES OF 17-7 PH (TH1050)

BASE METAL AND GIRTH WELD

TANK S/N 3

TEST SPECIMENS WERE REMOVED FROM LONGITUDINAL AXIS OF TANK (A)

SPECIMEN NUMBER	ULTIMATE TENSILE STRENGTH (PSI)	YIELD STRENGTH 0.2% OFFSET (PSI)	ELONGATION % IN 2 INCHES
	BASE METAL		
3C - 1.	212,700	197,600	6
3C - 2.	211,900	198,100	6
3C - 3.	216,200	203,200	6
	WELD METAL (B)		
3GW - 1.	136,500	75,900	4 (C)
3GW - 2.	128,700	70,700	3 (C)
3GW - 3.	130,900	67,100	3 (C)

(A) Since the test bars were removed from the tanks central section along the longitudinal axis of the tank, the properties are believed to represent the transverse direction of the original rolled sheet if the assumed fabrication sequence is correct.

(B) Welds not ground flush.

(C) Edge of weld failure.

TABLE V

MECHANICAL PROPERTIES OF 17-7 PH (TH1050)

BASE METAL AND GIRTH WELDS

TANK S/N 4

TEST SPECIMENS WERE REMOVED FROM LONGITUDINAL AXIS OF TANK (A)

SPECIMEN NUMBER	ULTIMATE TENSILE STRENGTH (PSI)	YIELD STRENGTH 0.2% OFFSET (PSI)	ELONGATION % IN 2 INCHES
	BASE METAL		
1.	215,600	201,600	7.0
2.	216,300	202,900	7.0
3.	216,000	197,000	7.0
	WELD METAL (B)		
1.	143,200	86,400	4.0 (C)
2.	140,000	85,800	4.0 (C)
3.	137,700	80,400	4.0 (C)

(A) Since the test bars were removed from the tanks central section along the longitudinal axis of the tank, the properties are believed to represent the transverse direction of the original rolled sheet if the assumed fabrication sequence is correct.

(B) Welds not ground flush.

(C) Edge of weld failure.

TABLE VI

MECHANICAL PROPERTIES OF 17-7 PH (TH1050)

BASE METAL AND GIRTH WELDS

TANK S/N 5

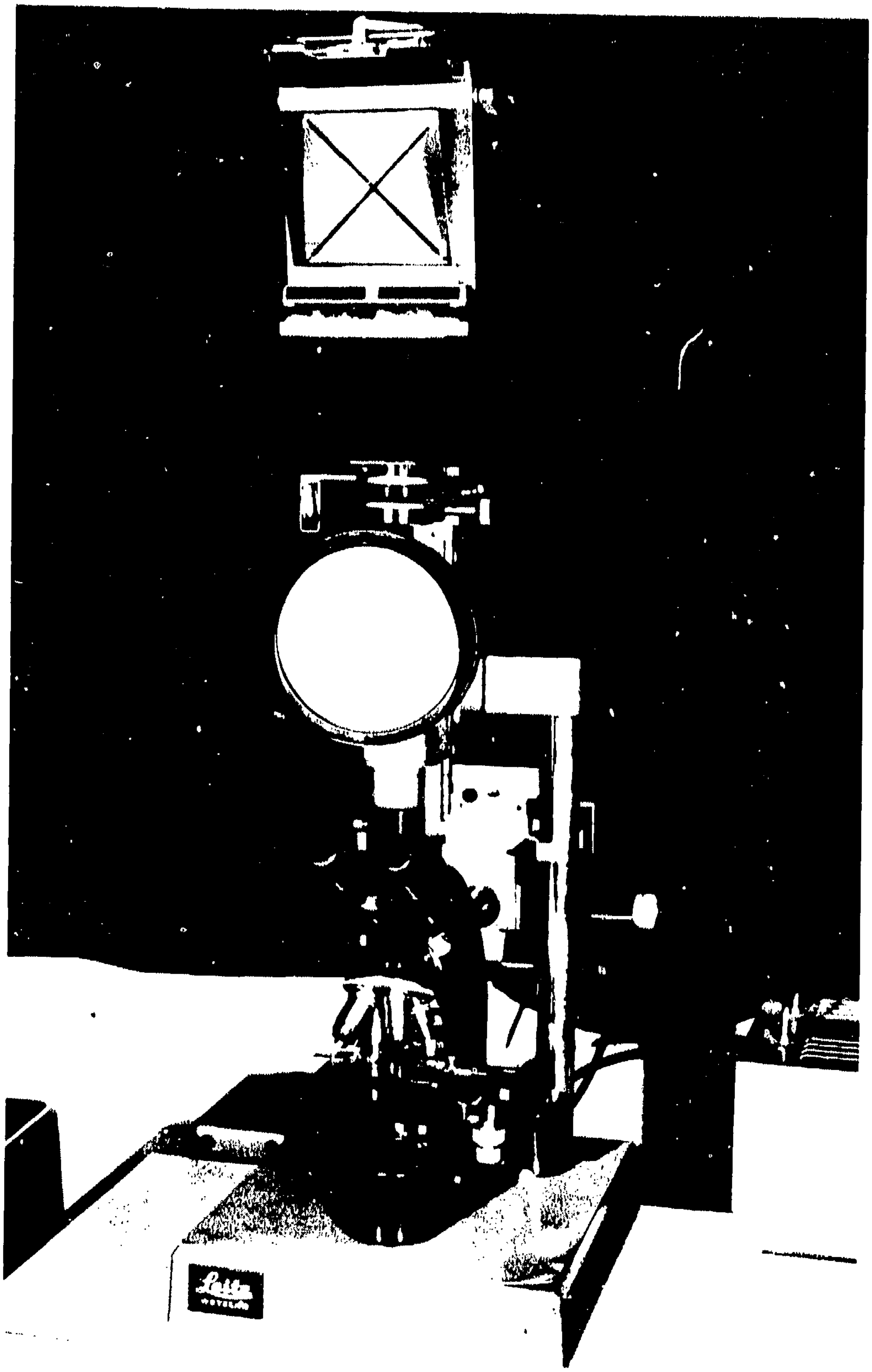
TEST SPECIMENS WERE REMOVED FROM LONGITUDINAL AXIS OF TANK (A)

SPECIMEN NUMBER	ULTIMATE TENSILE STRENGTH (PSI)	YIELD STRENGTH 0.2% OFFSET (PSI)	ELONGATION % IN 2 INCHES
	BASE METAL		
1.	220,000	207,000	8
2.	220,000	208,000	7
3.	220,000	207,000	7
	WELD METAL (B)		
1.	137,000	89,400	5 (C)
2.	139,000	85,500	4 (C)
3.	141,500	93,000	4 (C)

(A) Since the test bars were removed from the tanks central section along the longitudinal axis of the tank, the properties are believed to represent the transverse direction of the original rolled sheet if the assumed fabrication sequence is correct.

(B) Welds not ground flush.

(C) Edge of weld failures.



FIGU

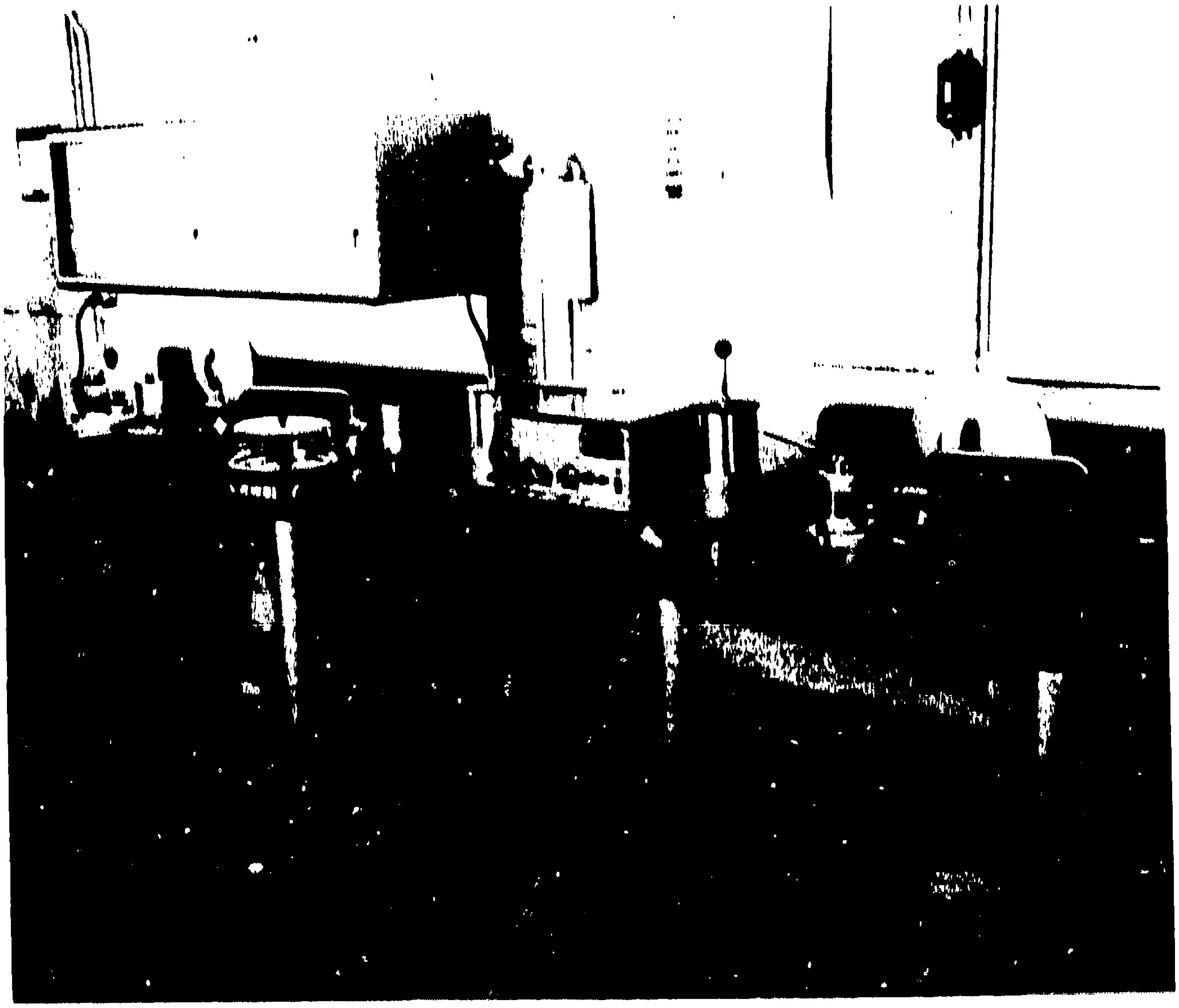


FIGURE 3. VIEW OF AUTOMATIC POLISHING EQUIPMENT IN METALLURGICAL LABORATORY

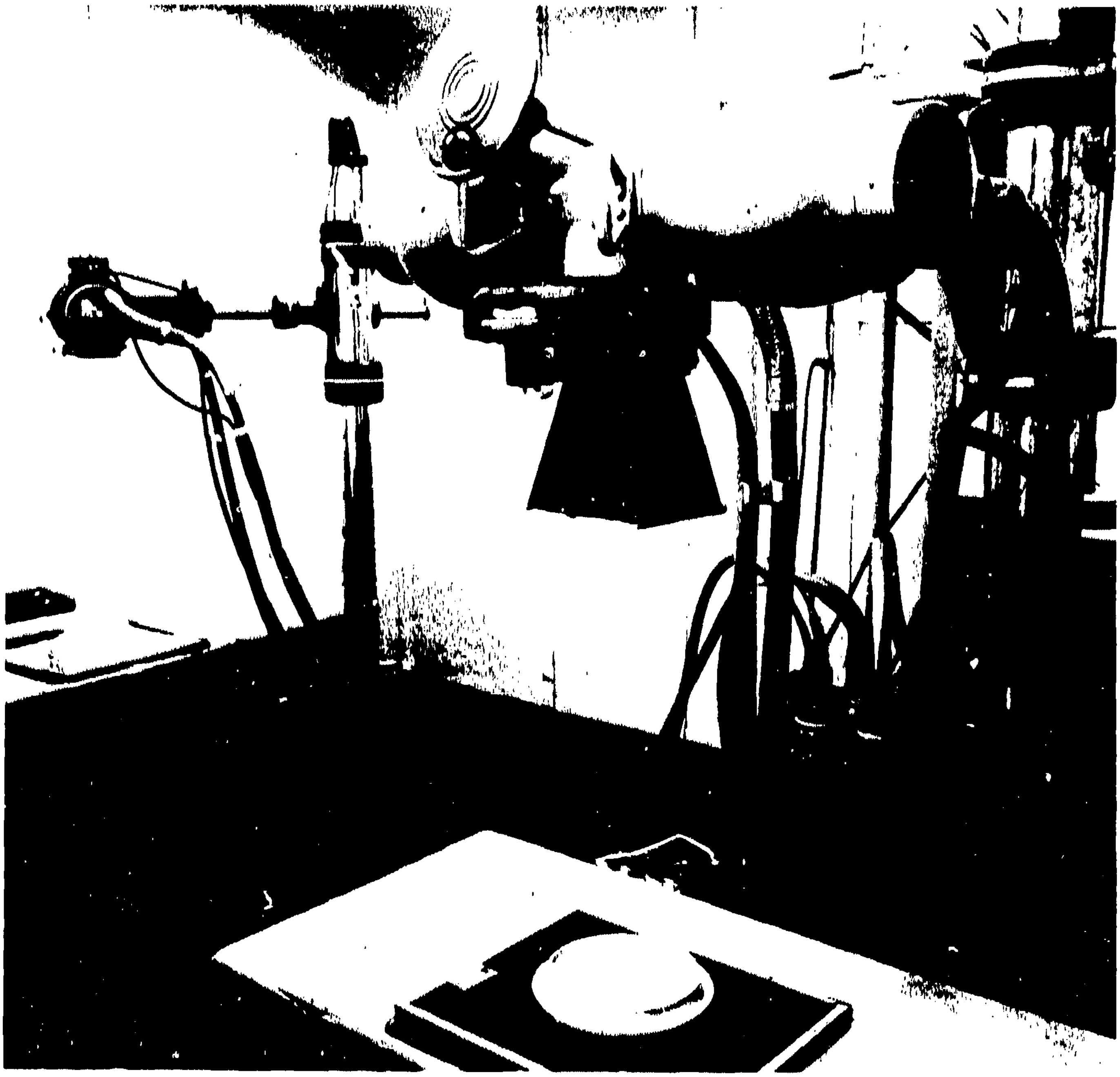
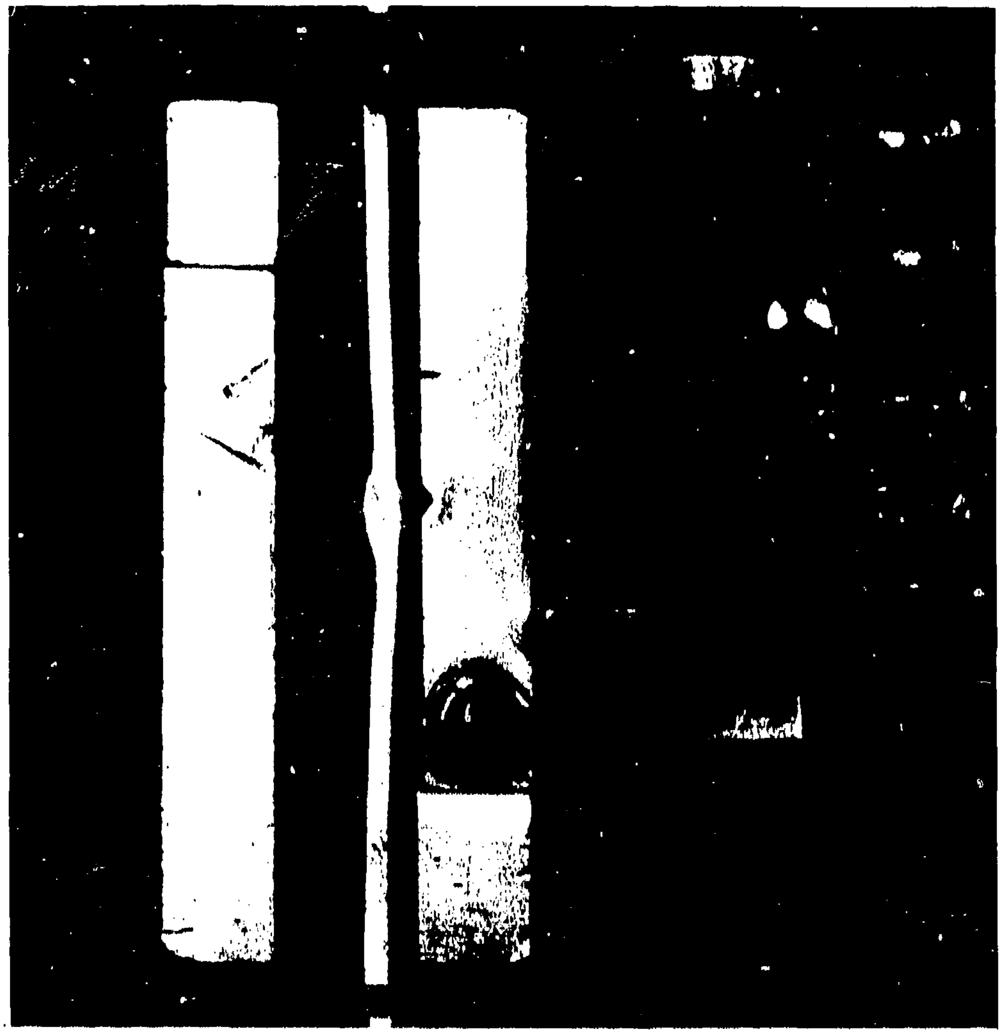
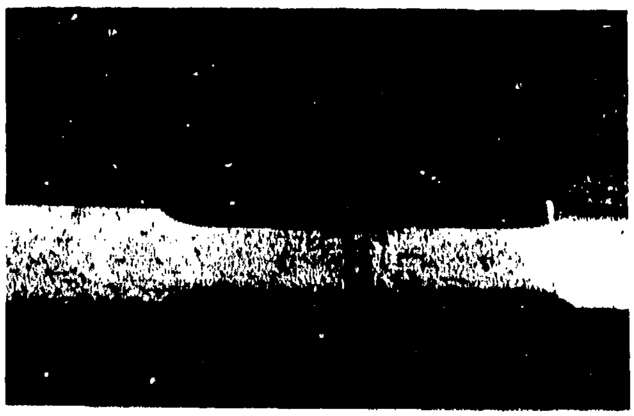


FIGURE [illegible]. RADIOGRAPHIC INSPECTION ARE[illegible]
METALLURGICAL LAB[illegible]

METALLURGICAL LABORATORY [illegible]



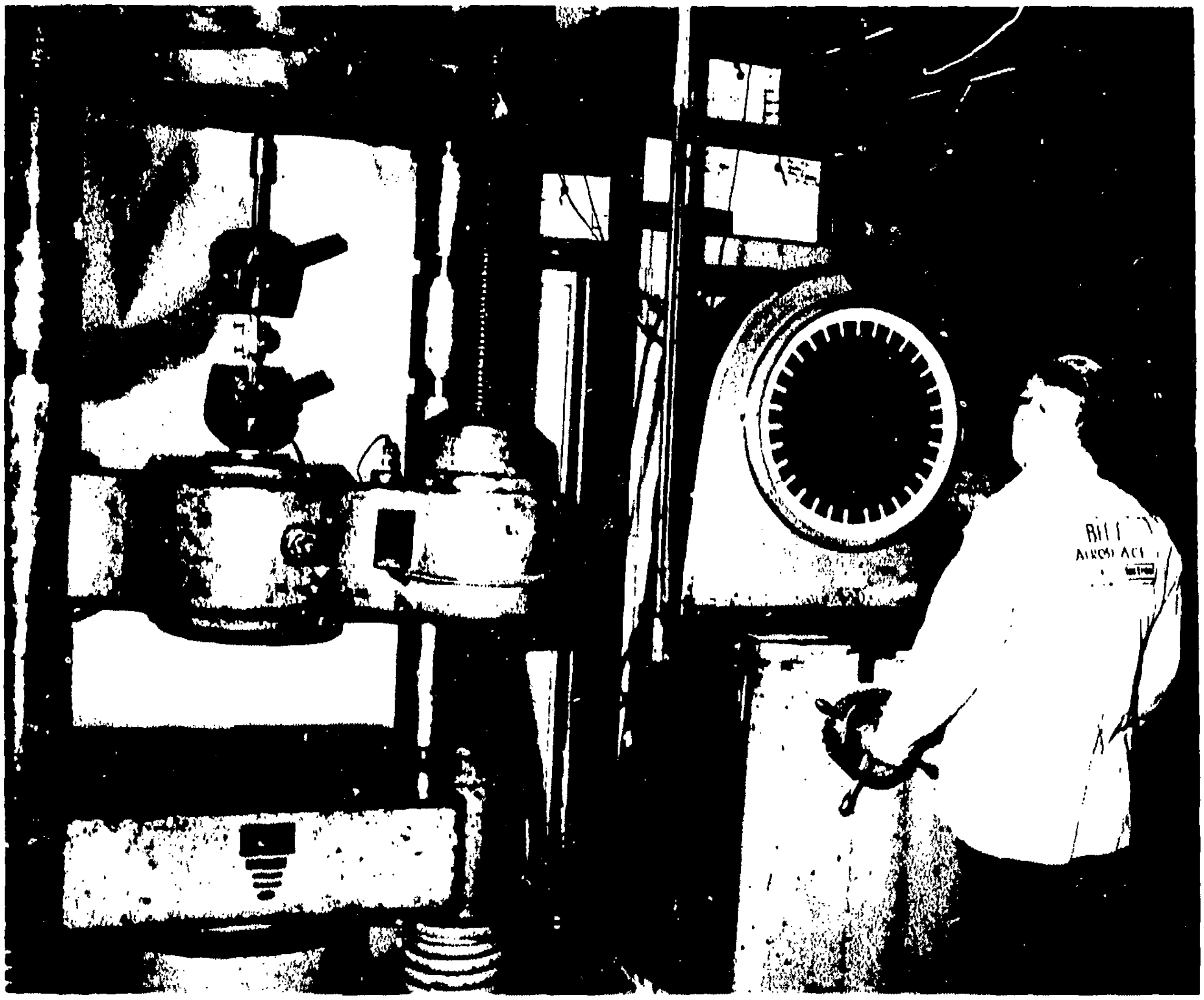
Closeup view of welded specimen ready for testing with extensometer in place



Plan view of tensile specimen, both before and after testing

FIGURE 6. EXAMPLES OF TENSILE SPECIMENS [illegible] FROM PROPELLANT STORAGE TANKS.

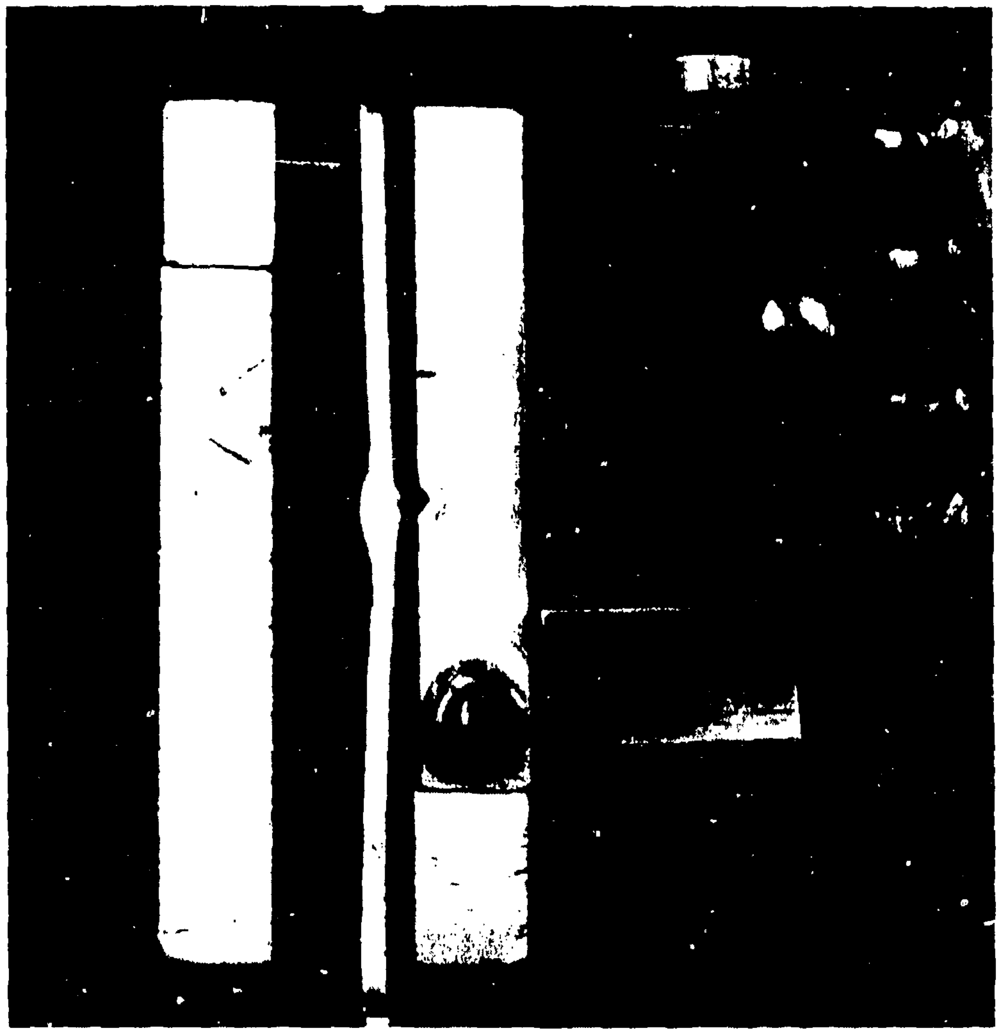
METALLURGICAL LABORATORY MET NO. 70-[illegible]



Autographic recorder for [illegible] curve for yield strength [illegible] of [illegible] bar. [illegible]

FIGURE [illegible]

[illegible]



[illegible] view of [illegible] specimen ready for [illegible], with [illegible] in place

[illegible] view of tensile specimen, [illegible] after [illegible]

FIGURE [illegible]

[illegible] LABORATORY [illegible]

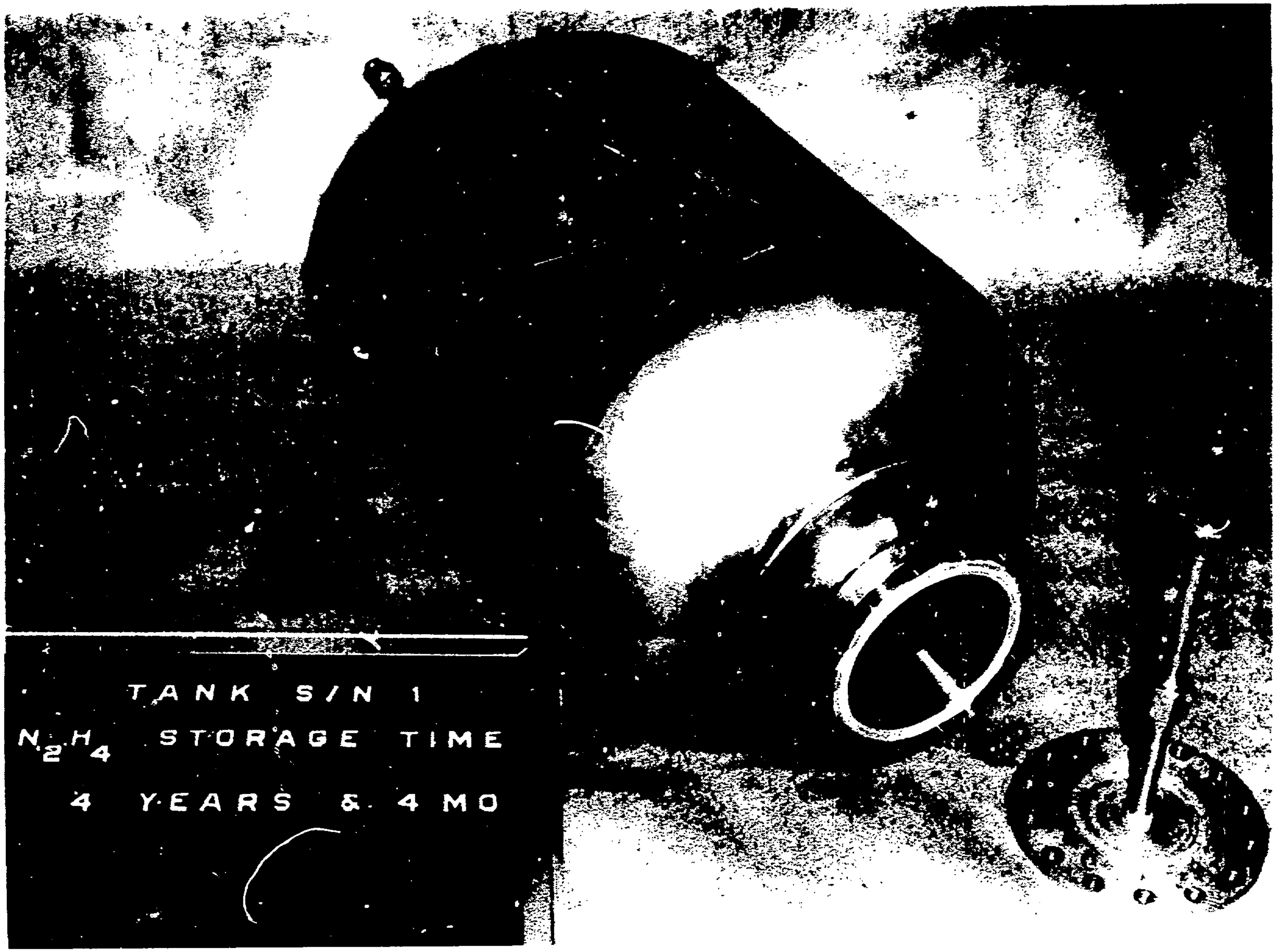


FIGURE [illegible]. TANK S/N [illegible] IN THE AS-RECEIVED [illegible] AFTER EXPOSURE TESTING [illegible]

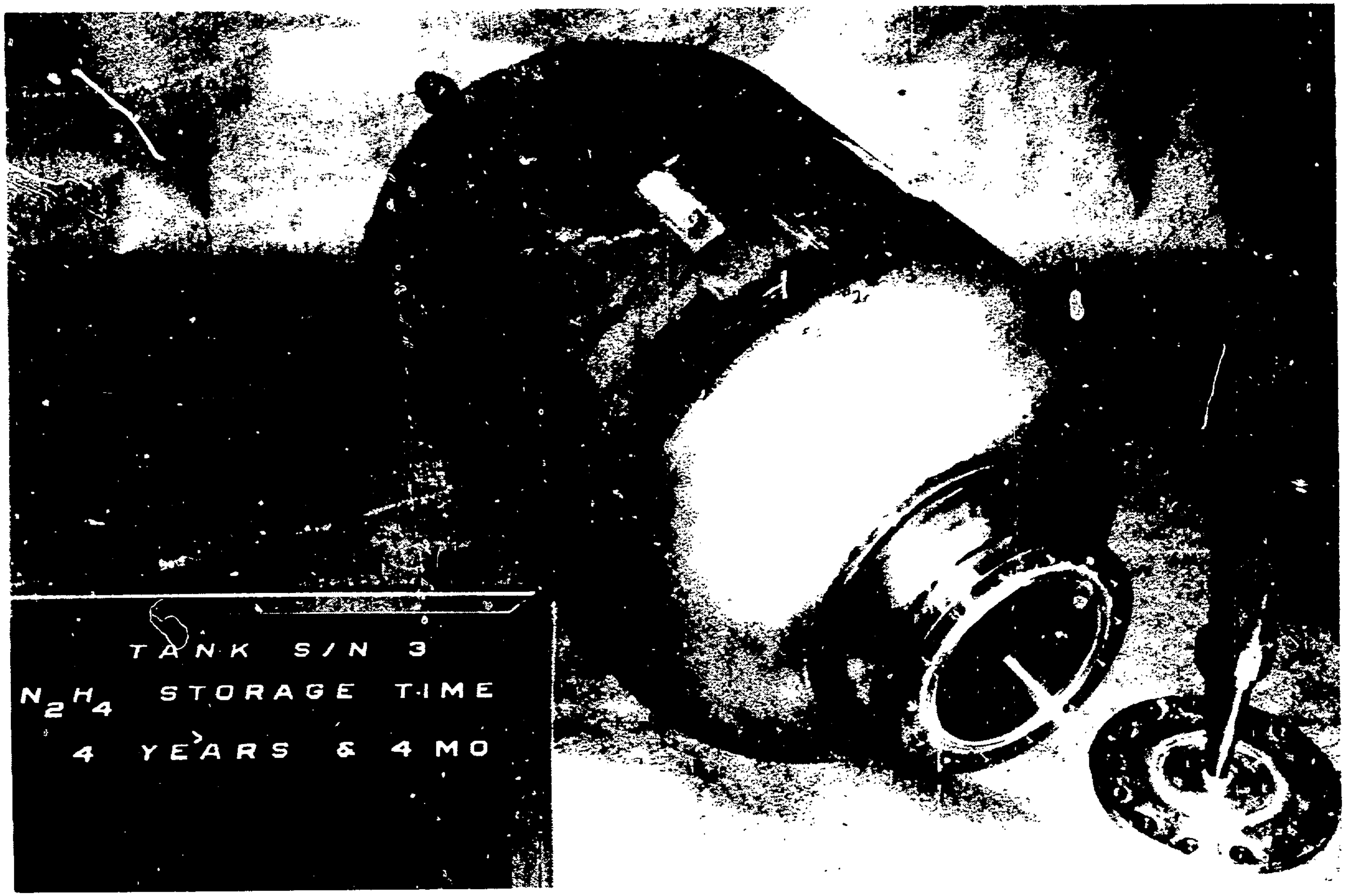


FIGURE [illegible]. TANK S/N 3 IN THE [illegible] [illegible]
AREAS OF THE WELD HAD BEEN [illegible] IN THE PAST. [illegible]
NOT DEFECTS OR LEAKS, [illegible]
[illegible] AND DROP-THRU WERE PRESENT.

METALLURGICAL LABORATORY MET NO. [illegible]

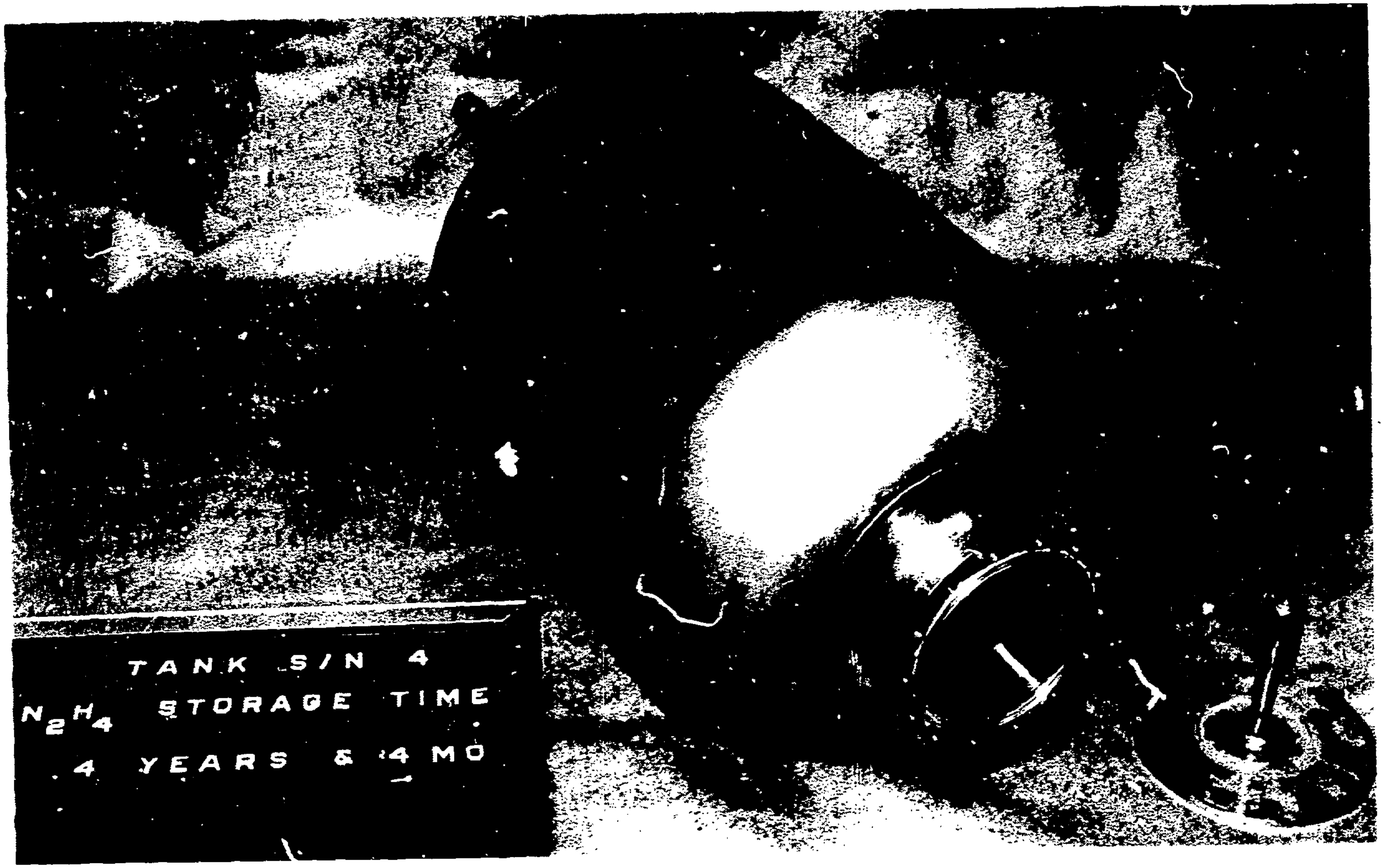
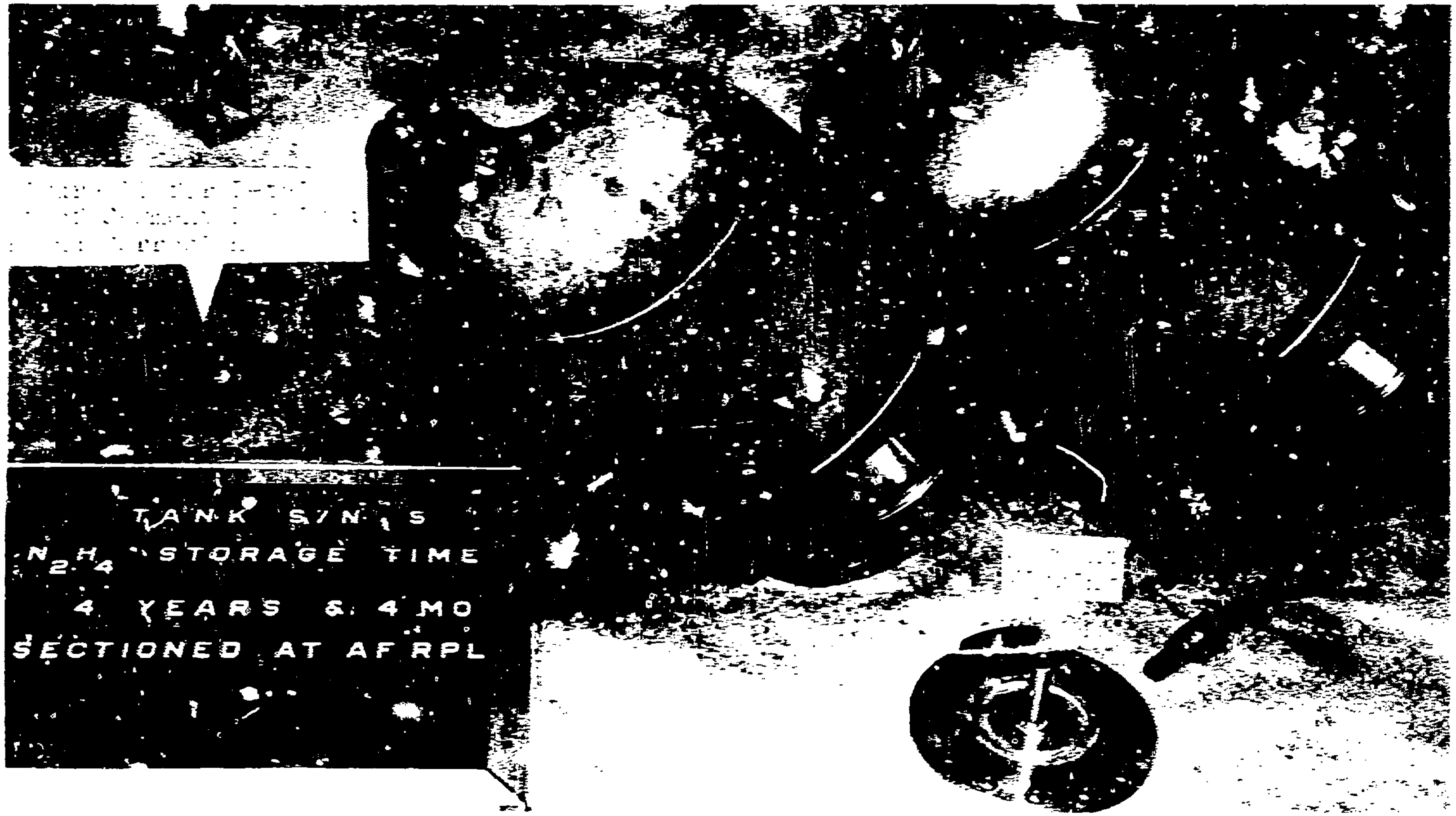


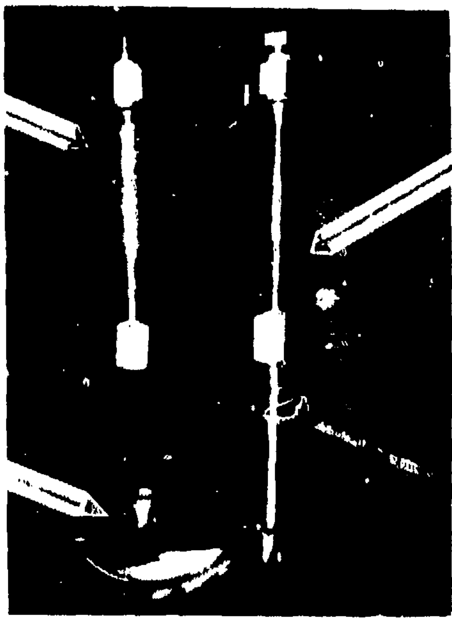
FIGURE 9. TANK S/N 4 IN THE AS-RECEIVED CONDITION AFTER STORAGE TESTING. THIS TANK, LIKE S/N 1, HAD LEAKED AROUND THE SOFT METAL GASKET SEAL AT THE FLANGED FILL.

METALLURGICAL LABORATORY MET NO. 7[illegible]-79.1[illegible]

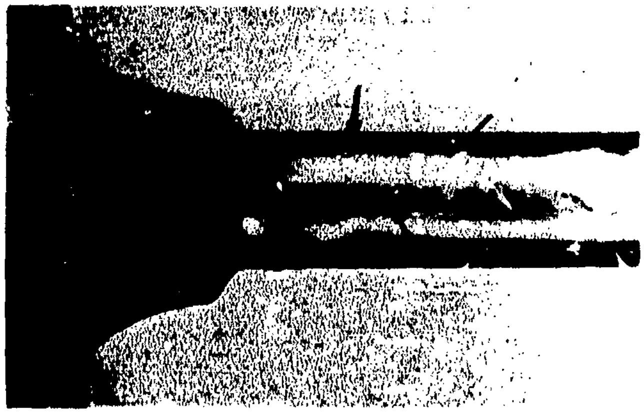

TANK S/N 1 S
N_2H_4 STORAGE TIME
4 YEARS & 4 MO
SECTIONED AT AFRPL

Segments supplied from Tank S/N 5

Typical components of Tank S/N 1 showing lengths and arrangement.



Area of corrosion detailed below.



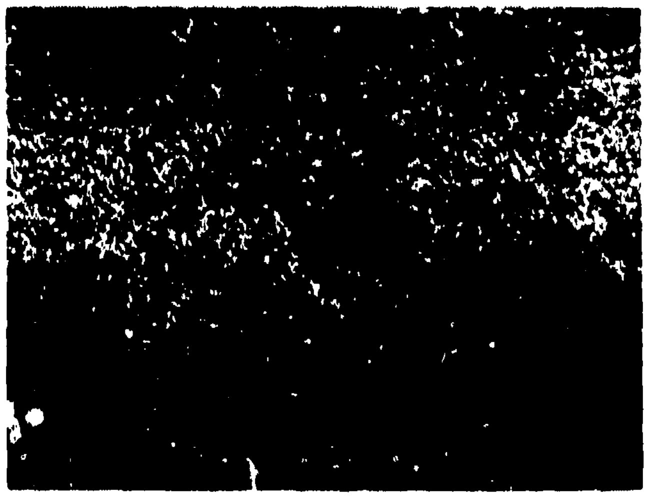
Mag: 1-1/2X

a) View of outlet tube components received from tank S/N 5 compared with unsectioned components of tank S/N 1 illustrating segment missing from original shipment

b) Tube extension from corroded outlet fitting showing slight surface etching but no significant corrosive attack. Etched regions are indicated by black arrows, unetched areas by white arrows.

Mag: 1-1/2X

c) Overall view of outlet fitting showing etched area and pit at bottom.



Mag: 20X

d) High magnification view showing localized corrosion.

FIGURE 11. DETAIL VIEW OF OUTLET PORT REGION OF TANK S/N 5 SHOWING EVIDENCE OF CORROSIVE ATTACK IN BOSS.

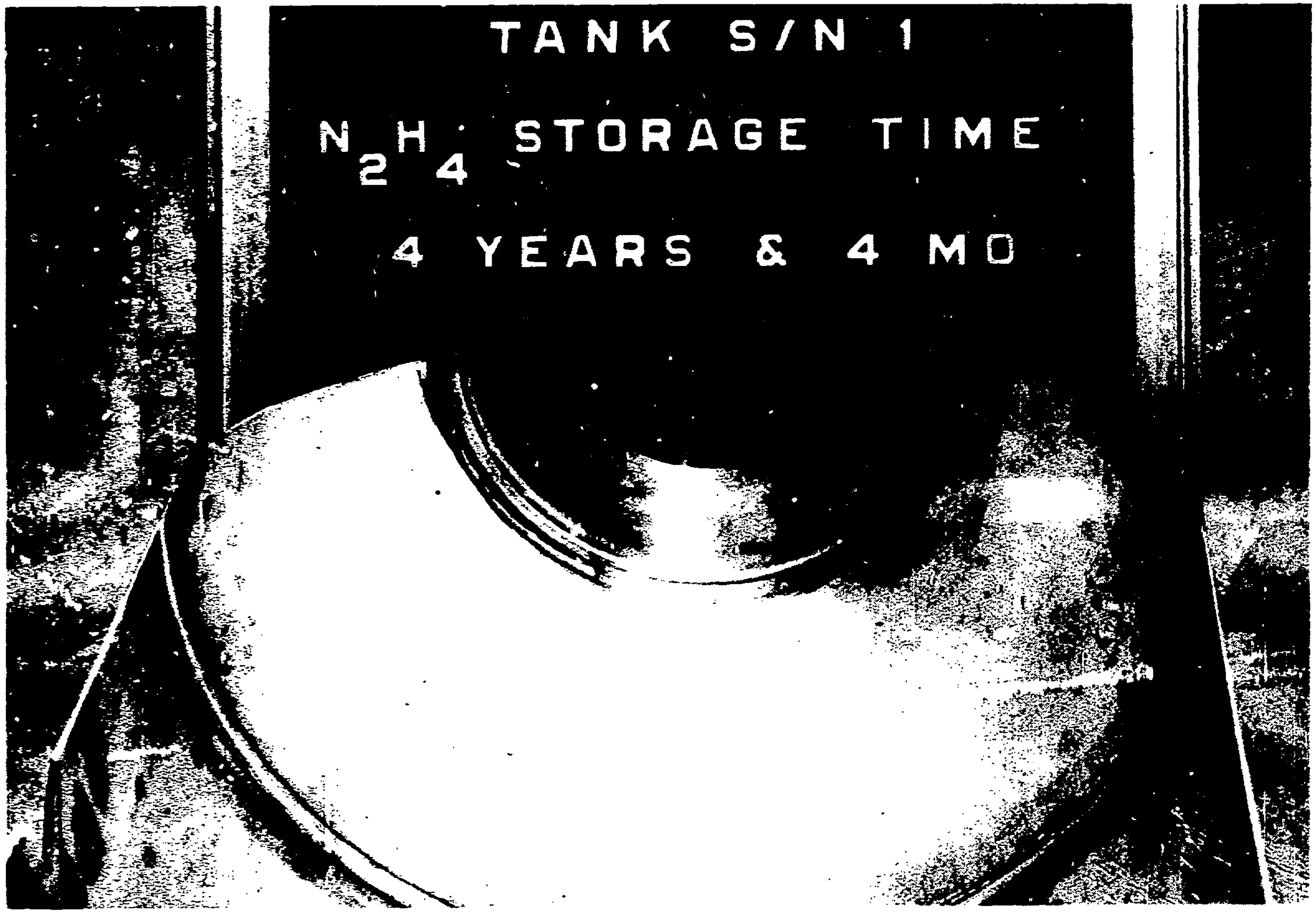


FIGURE 12. TANK S/N 1 IN THE AS-RECEIVED CONDITION AFTER SECTIONING SHOWING MARKINGS SIMILAR TO WATERLINES. THEIR PRESENCE IN THE FLANGE AREA MAY BE A RESULT OF POSITIONING AFTER REMOVAL FROM THE STORAGE RACKS.

METALLURGICAL LABORATORY MET NO. 76-79016

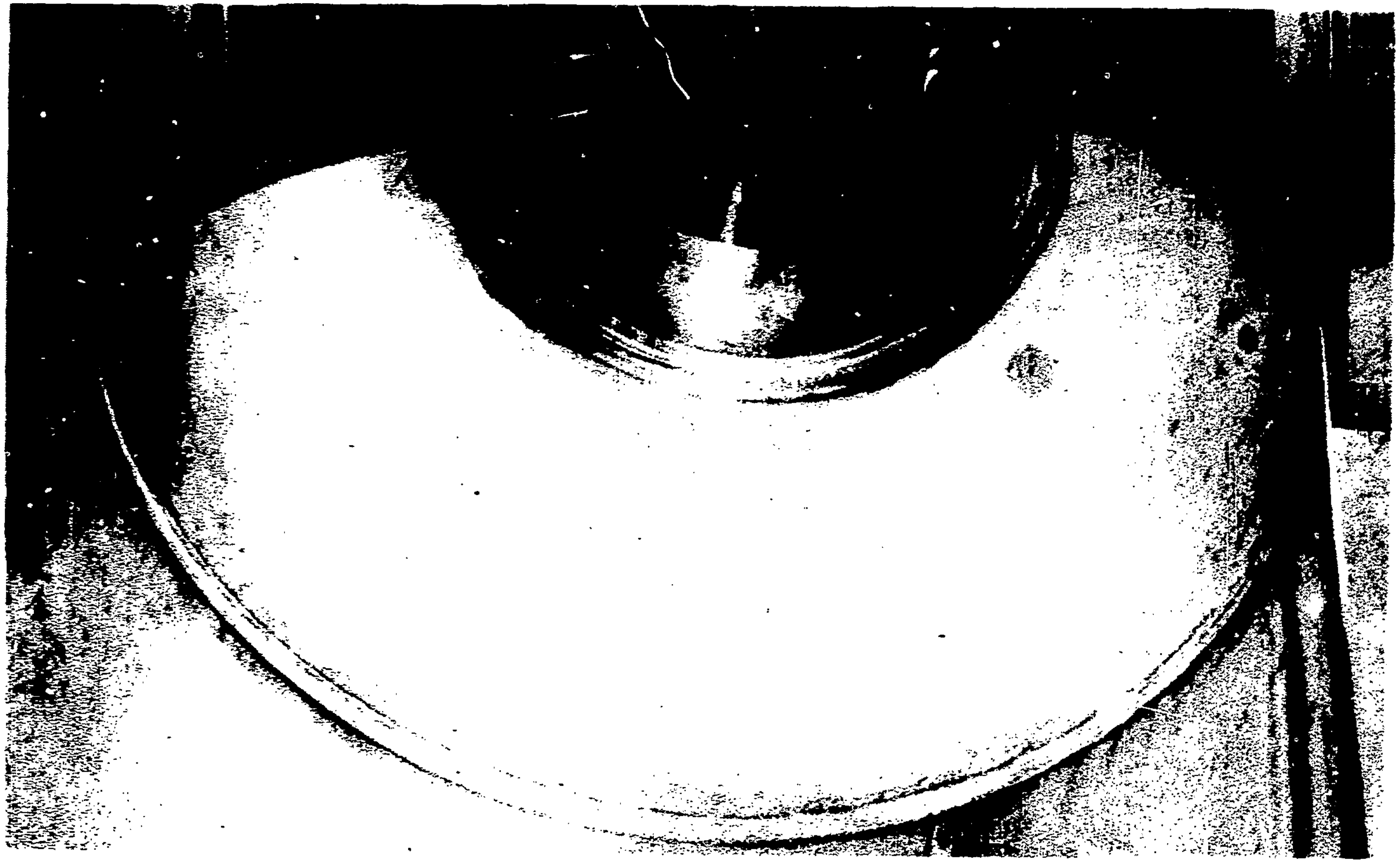
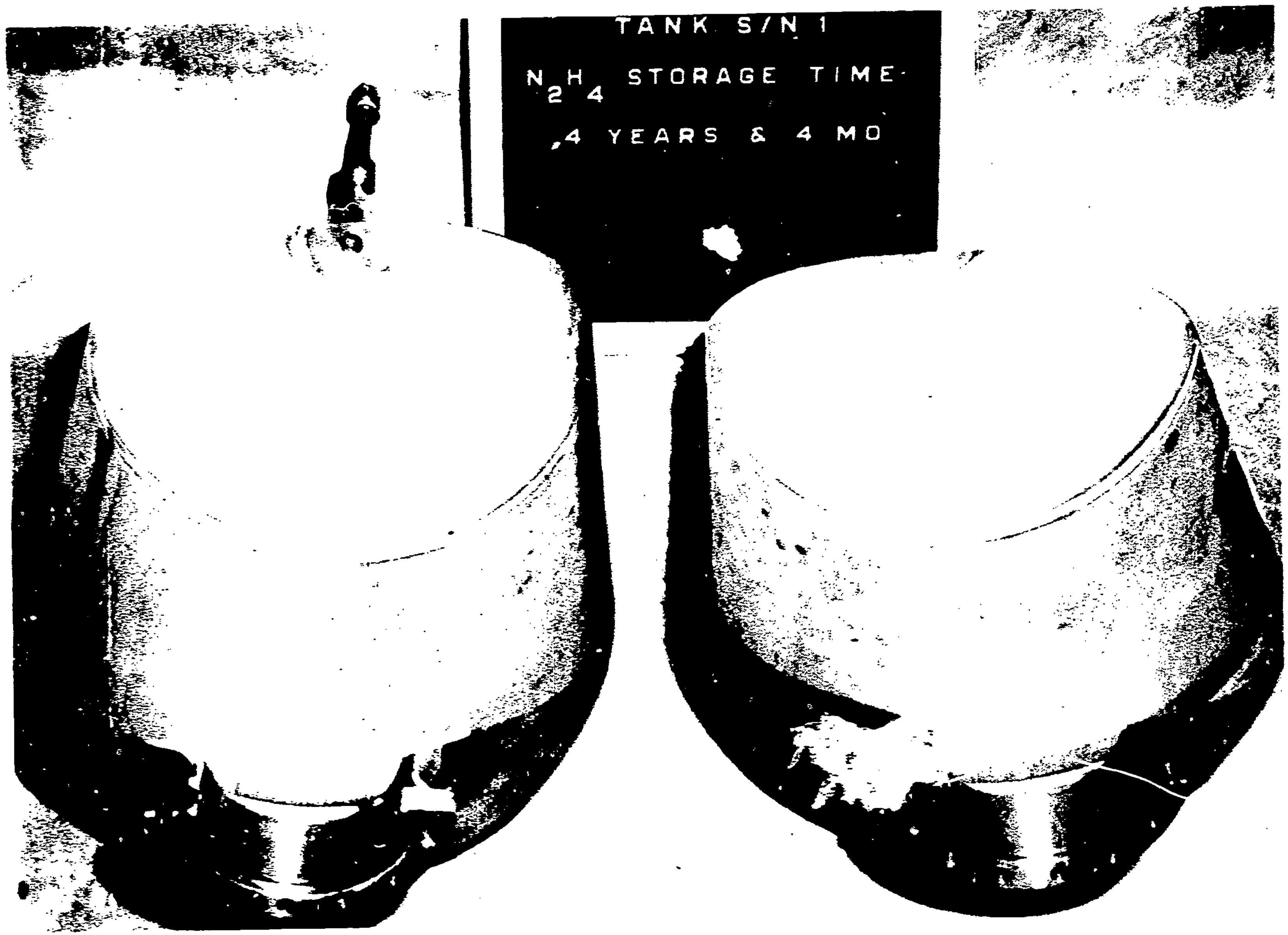


FIGURE 13. OPPOSITE HALF OF TANK S/N 1 IN THE AS-RECEIVED CONDITION SHOWING THE SAME TYPE OF MARKINGS AS SHOWN IN FIGURE 12. THESE PHOTOMACROGRAPHS SUBSTANTIATE THE OVERALL INTERIOR CONDITION OF THIS TANK. NO EVIDENCE OF CORROSIVE ATTACK WAS OBSERVED IN EITHER HALF OF THIS TANK ON A MACROSCOPIC BASIS.

METALLURGICAL LABORATORY MET NO. 76-79016


TANK S/N 1
N2H4 STORAGE TIME
4 YEARS & 4 MO

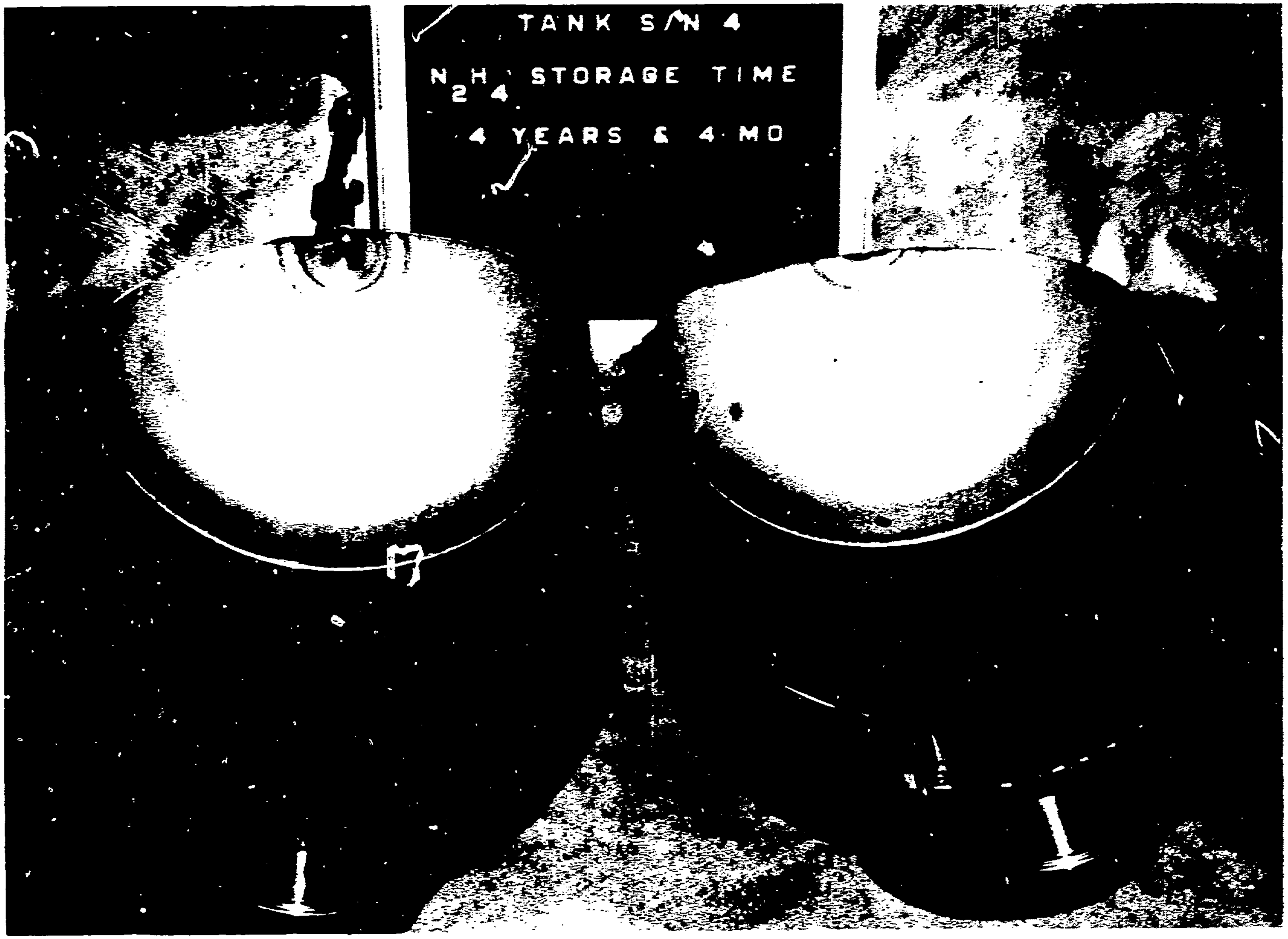
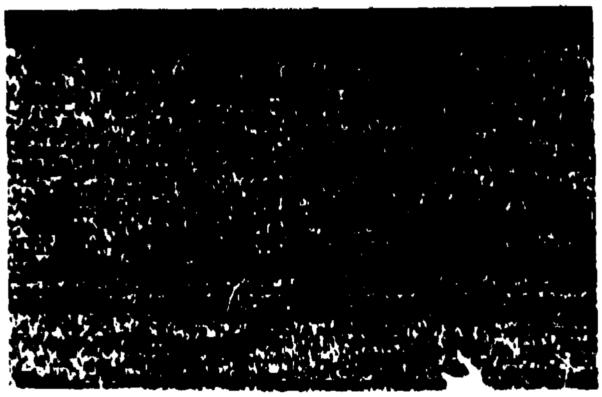


FIGURE 15. TANK S/N 4 SH[illegible] MARKINGS SIMILAR TO WATERLINES AT THE BOTTOM OF THE TANK. FROM THE MANNER IN WHICH THE TANKS WERE ST[illegible] THIS IS THE AREA WHERE MARKINGS OF THIS TYPE WOULD BE EXPECTED I[illegible] RESIDUAL N_2H_4 WERE PRESENT.

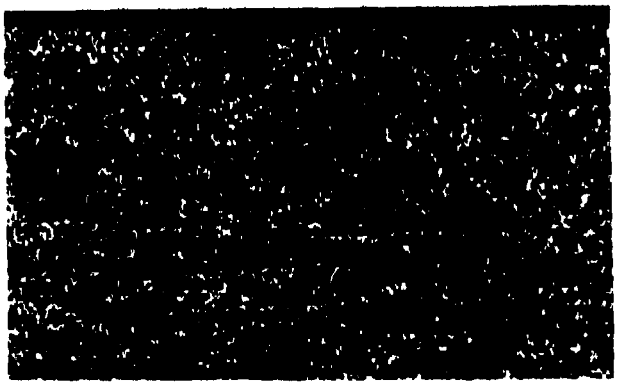
[illegible] RATORY NEG NO. [illegible]



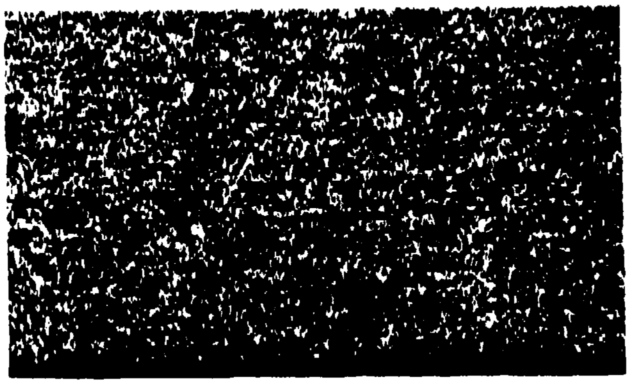
OD Surface in Circumferential Direction

ID Surface in Axial Direction

Cylinder Component



OD Surface in Circumferential Direction

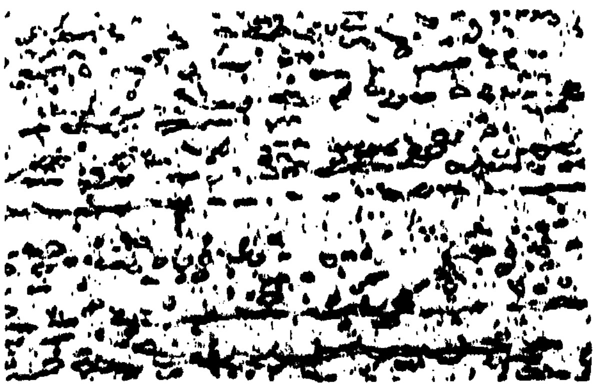


ID Surface in Axial Direction

Dome Component

All Views - Mag.: 100X   Etch: Ammonium Persulfate

FIGURE 16. MICROSTRUCTURE AND SURFACE CROSS SECTIONS OF AM350 STAINLESS STEEL MATERIAL USED IN TANK S/N 1.

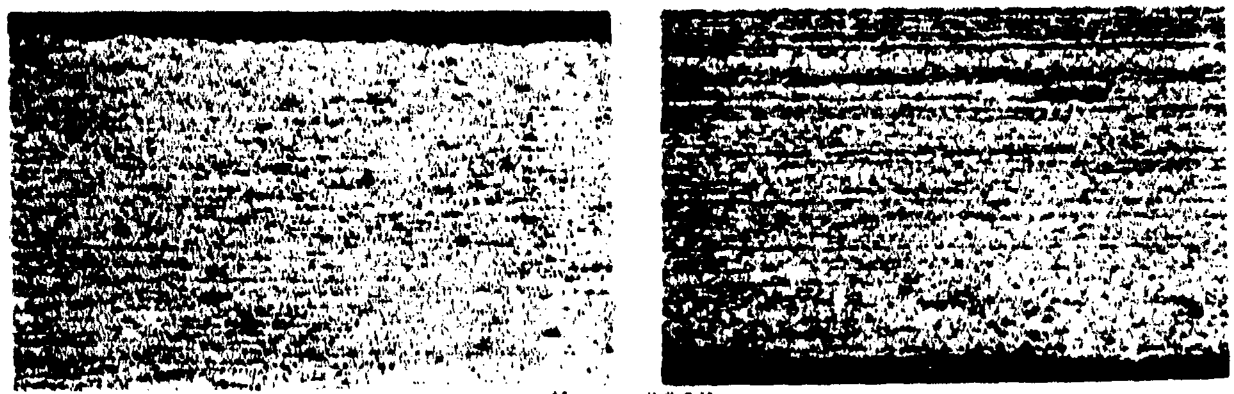


Mag.: 500X

a) Cylindrical Shell (Rolled Sheet)   b) Dome (Explosive Formed Sheet)

FIGURE 17. MICROSTRUCTURE OF AM350 STAINLESS STEEL SHELL MATERIALS USED IN FABRICATION OF TANK S/N 1.

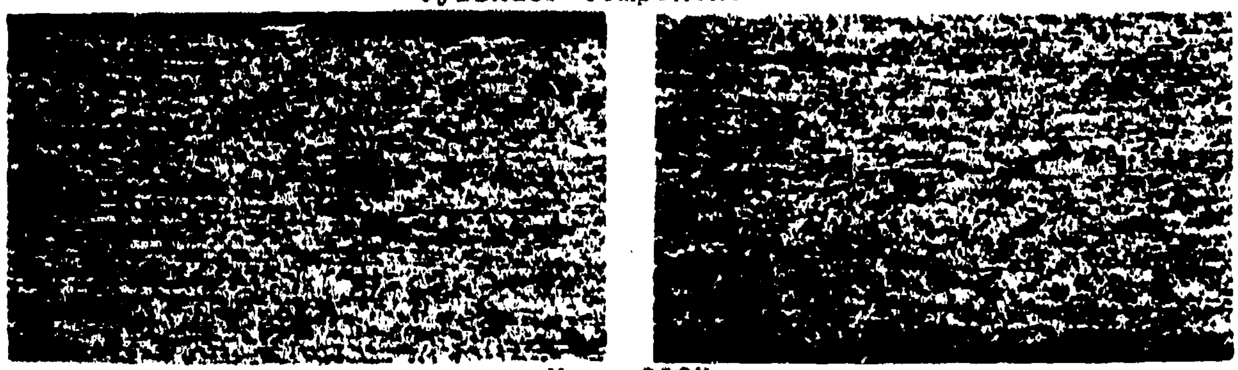
METALLURGICAL LABORATORY MET NO. 76-[illegible]



Mag: 200X

OD Surface in Axial Direction — ID Surface in Circumferential Direction

Cylinder Component

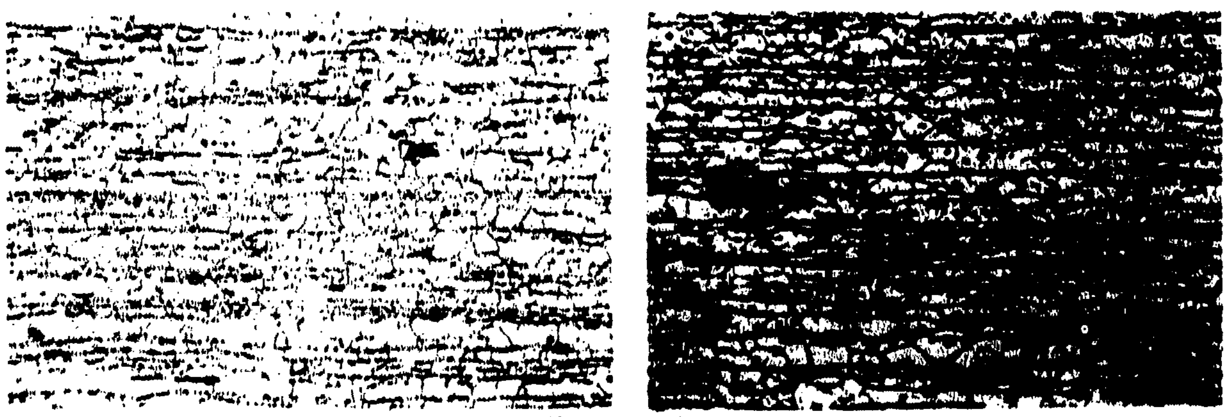


Mag: 200X

OD Surface in Axial Direction — ID Surface in Circumferential Direction

Dome Component

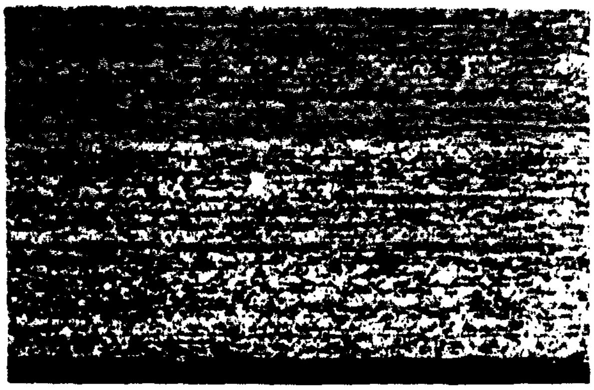
FIGURE 18. MICROSTRUCTURE AND SURFACE CROSS SECTIONS OF 17-7PH STAINLESS STEEL MATERIALS USED IN TANK S/N 3



Mag: 500X
Etch: Vilella's

a) Cylindrical Shell (Rolled Sheet) — b) Dome (Explosive Formed Sheet)

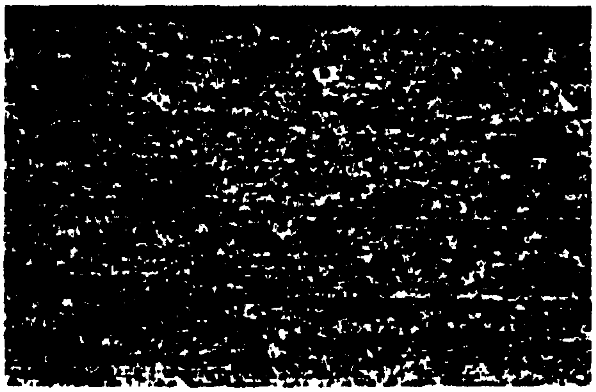
FIGURE 19. MICROSTRUCTURE OF THE 17-7PH STAINLESS STEEL SHELL MATERIALS USED IN FABRICATION OF TANK S/N 3

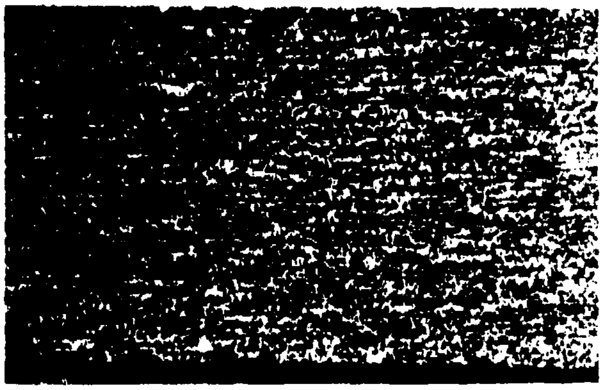


OD Surface in Axial Direction

ID Surface in Circumferential Direction

Cylinder Component





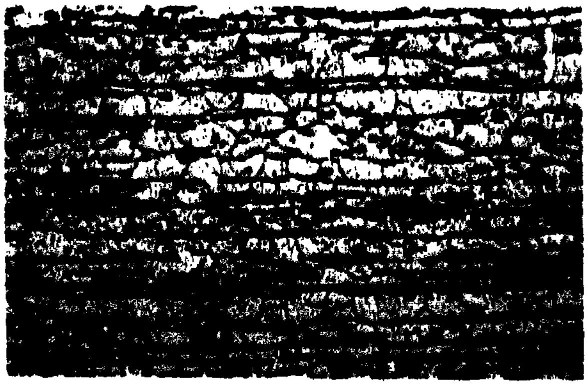
OD Surface in Axial Direction

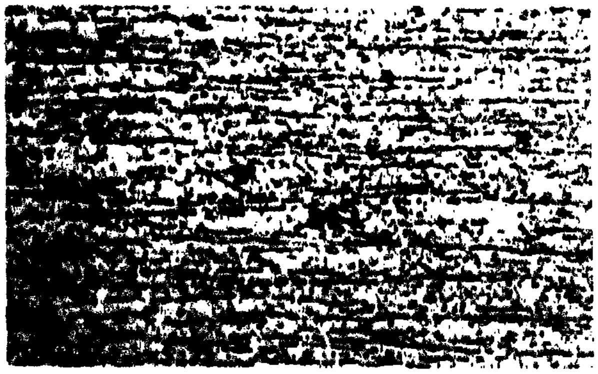
ID Surface in [illegible] Direction

Dome Component

All Views - Mag.: [illegible]X Etch: Vilella's

FIGURE 20. MICROSTRUCTURE AND SURFACE CROSS SECTIONS [illegible] STAINLESS STEEL MATERIALS USED IN TANK [illegible]



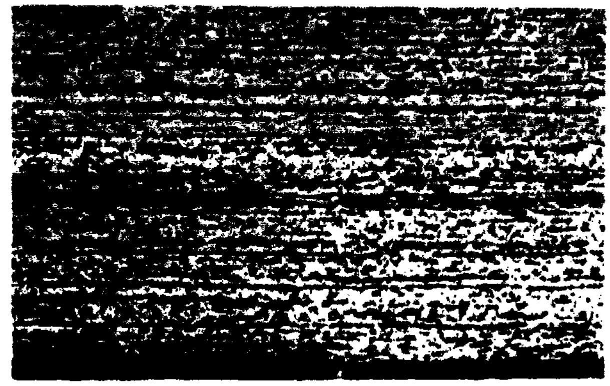


Mag.: 500X

a) Cylindrical Shell (Rolled Sheet) b) Dome (Explosive Formed Sheet)

FIGURE 21. MICROSTRUCTURE OF THE 17-7PH STAINLESS STEEL SHELL MATERIALS USED IN FABRICATION OF TANK S/N 4

[illegible] Surface in [illegible]xial Direction



ID Surface in Circumferential Direction

Cylinder Component

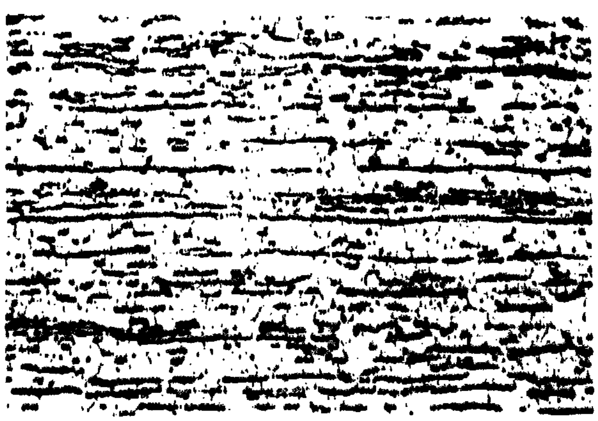
OD Surface in [illegible] Direction

ID Surface in Circumferential Direction

Dome Component

[illegible] Etch: Vilella's

[illegible]



Mag.: 500X

a) Cylindrical Shell (Rolled Sheet)

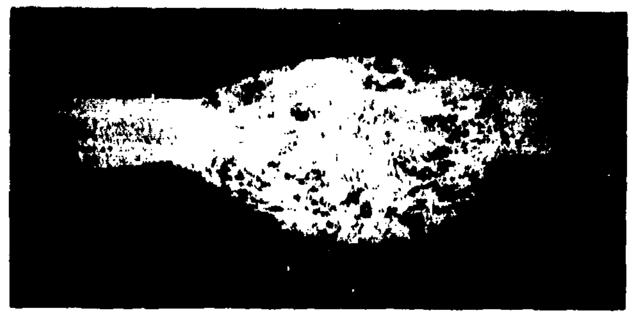
b) Dome (Explosive Formed Sheet)

FIGURE 23. MICROSTRUCTURE OF THE 17-7 PH STAINLESS STEEL SHELL MATERIALS USED IN FABRICATION OF TANK S/N 5.

METALLURGICAL LABORATORY MET NO. 76-79016

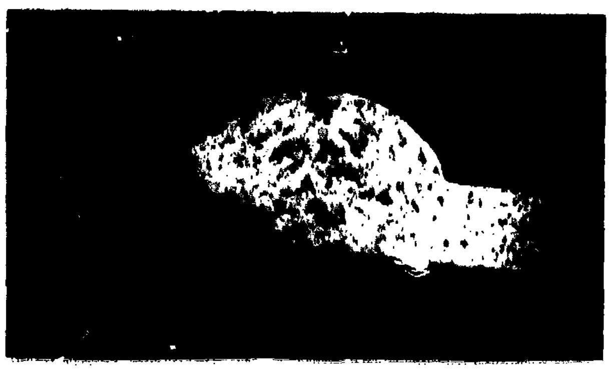
a) Flange to Dome Weld

Flange Dome to Cylinder



Outlet Dome to Cylinder

b) Girth Welds

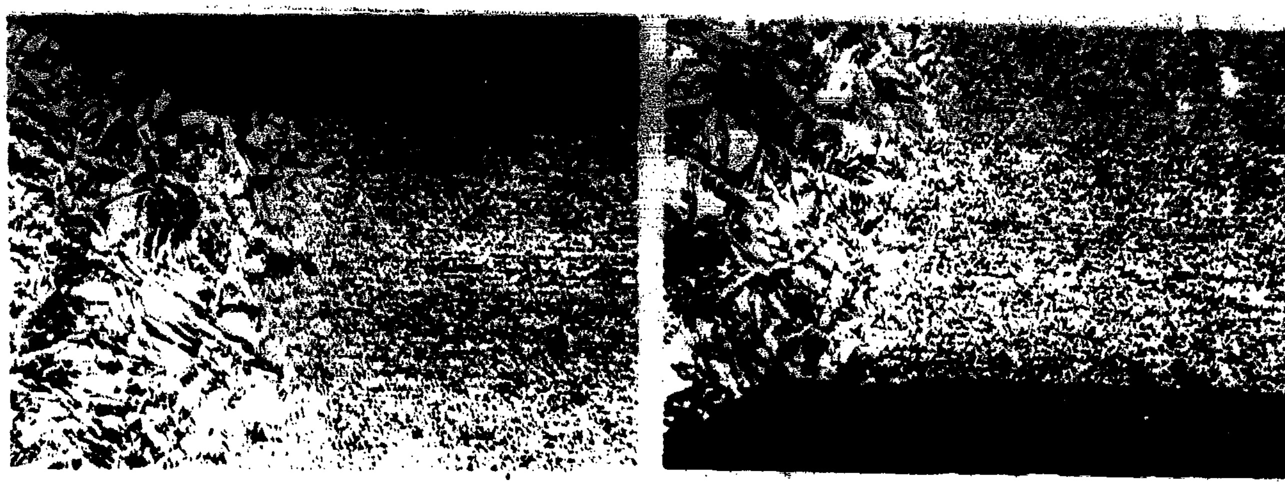


c) Outlet Fitting to Dome Weld

All Views - Mag.: 10X Mat'l: AM350 Etch: Ammonium Persulfate

FIGURE 24. CROSS SECTION OF TANK SHELL WELDS FROM TANK S/N 1 SHOWING WELD BEAD CONFIGURATION.

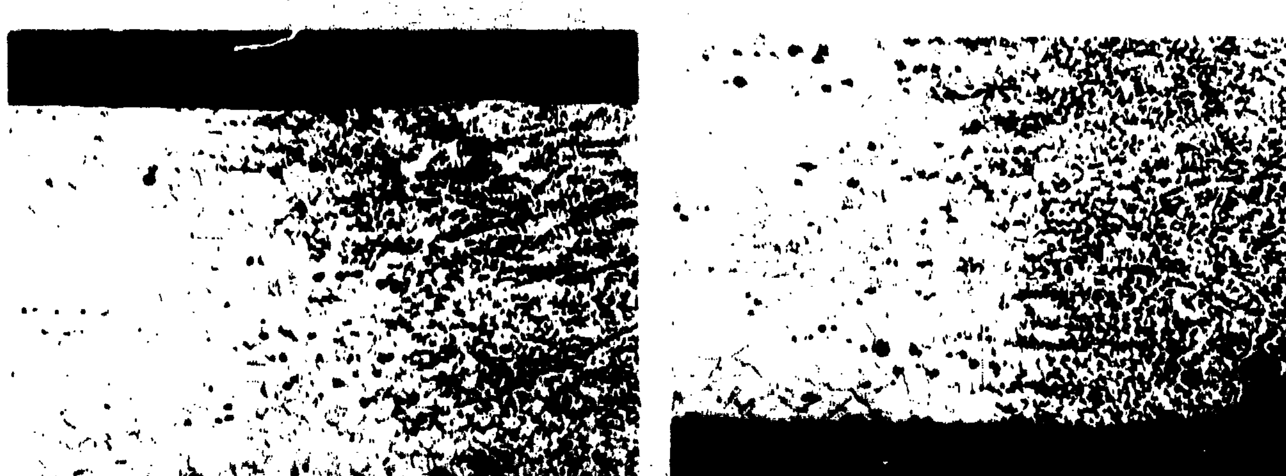
METALLURGICAL LABORATORY MET NO. 76-79016



OD Surface Mag.: 100X ID Surface

a) Girth Weld on Flange Dome Side

Mat'l: AM350



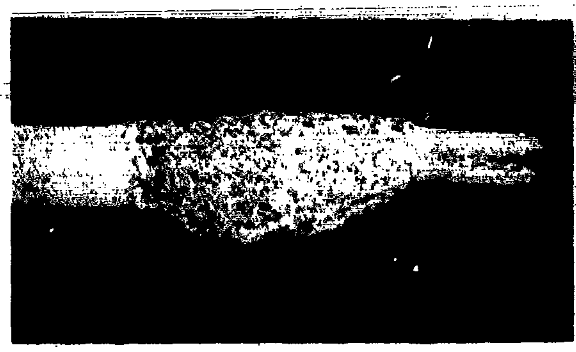
OD Surface Mag.: 100X ID Surface

b) Outlet Tube to Connector Fitting Weld

Mat'l: AISI 347 Stainless Steel

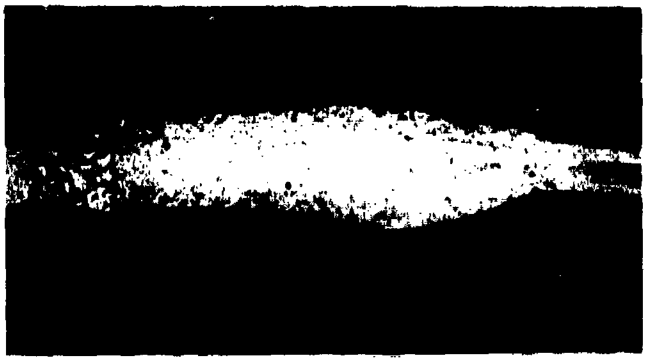
FIGURE 25. SURFACE CROSS SECTIONS SHOWING ABSENCE OF ANY SURFACE ATTACK IN TANK AND TUBE WELDS FROM S/N 1.

METALLURGICAL LABORATORY MET NO. 76-79016



a) Flange to Flange Dome Weld

b) Flange Dome to Cylinder
Girth Weld

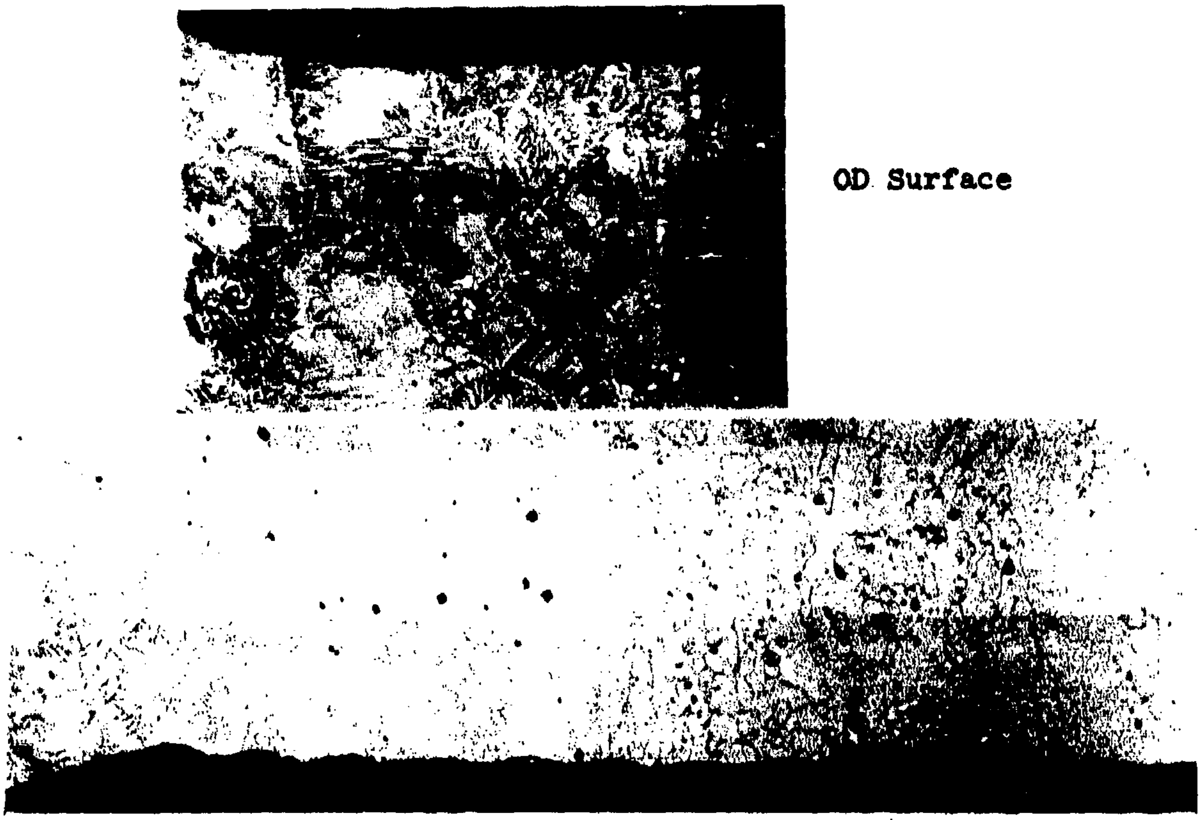


c) Outlet Fitting to Outlet
Dome Weld

All Views - Mag.: 10X Mat'l: [illegible] SS
Etch: Vilella's

FIGURE 26. CROSS SECTION OF TANK SHELL WELDS FROM TANK
S/N 3 SHOWING WELD BEAD PENETRATION

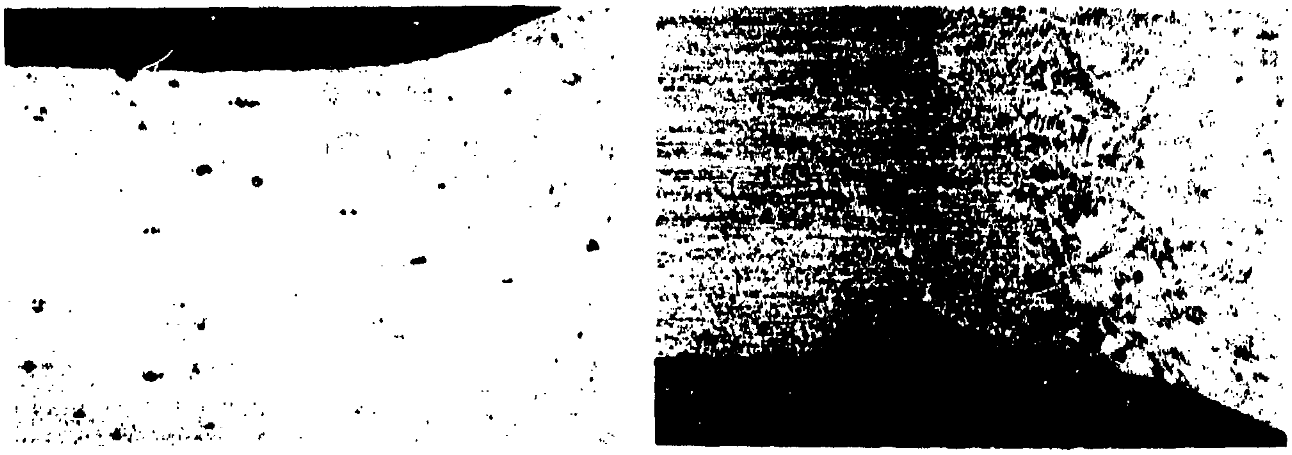
METALLURGICAL LABORATORY MET NO. [illegible]



OD Surface

Weld ID Surface Outlet Fitting

a) Outlet Fitting to Dome Weld showing coarse structure of Fitting but no corrosion or even alteration of as machined inside surface.



OD Surface ID Surface

b) Girth Weld on Cylinder Side

All Views - Mag.: 100X, Etch: Vilella's

FIGURE 27. HIGH MAGNIFICATION VIEWS OF TWO TANK S/N 3 WELDS SHOWING ABSENCE OF ANY SURFACE ATTACK.

METALLURGICAL LABORATORY MET NO. 76-79016

a) Flange to Dome Weld

Flange Dome to Cylinder — Outlet Dome to Cylinder

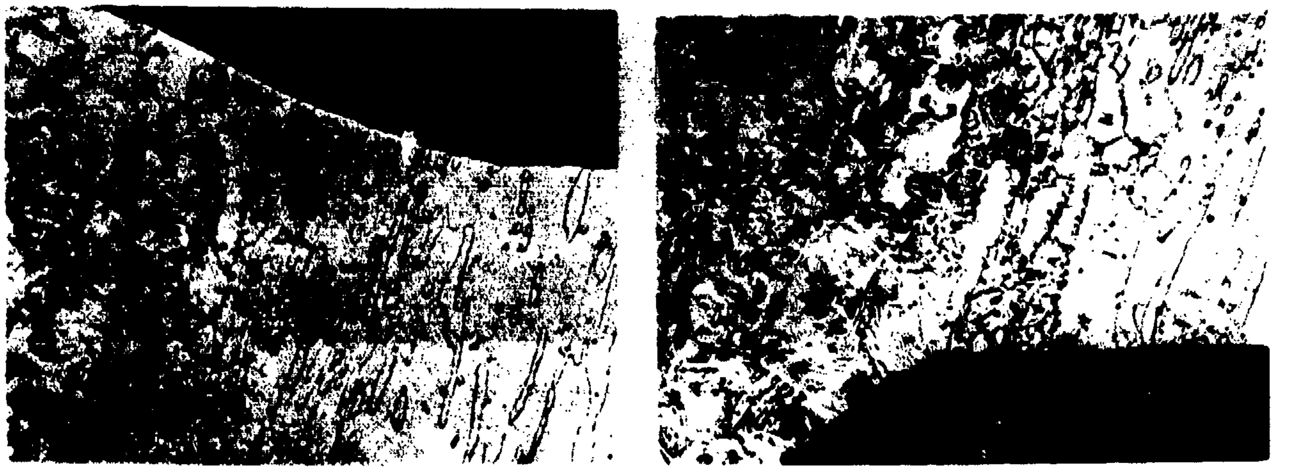
b) Girth Welds

c) Outlet Fitting to Dome Weld

All Views - Mag.: 10X Mat'l: 17-7PH SS Etch: Vilella's

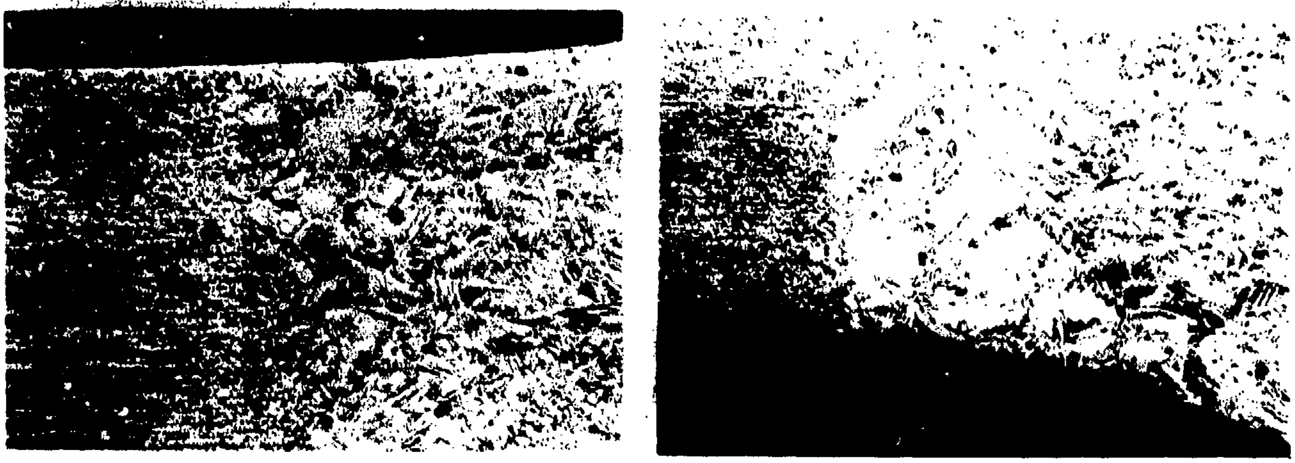
FIGURE 28. CROSS SECTION OF TANK SHELL WELDS FROM TANK S/N 4, SHOWING WELD BEAD CONFIGURATION.

METALLURGICAL LABORATORY MET NO. 76-79016



OD Surface | ID Surface

a) Flange to Dome Weld



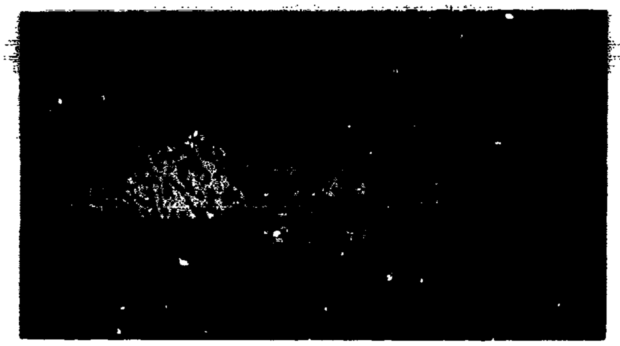
OD Surface | ID Surface

b) Girth Weld on Cylinder Side

All Views - Mag: 100X Mat'l: 17-7PH Stainless Steel
Etch: Vilella's

FIGURE 29. HIGH MAGNIFICATION VIEWS OF TWO TANK S/N 4 WELDS SHOWING ABSENCE OF ANY SURFACE ATTACK.

METALLURGICAL LABORATORY MET NO. 76-79016



a) Flange to Dome Weld Showing Considerable Variation in Joint Thickness

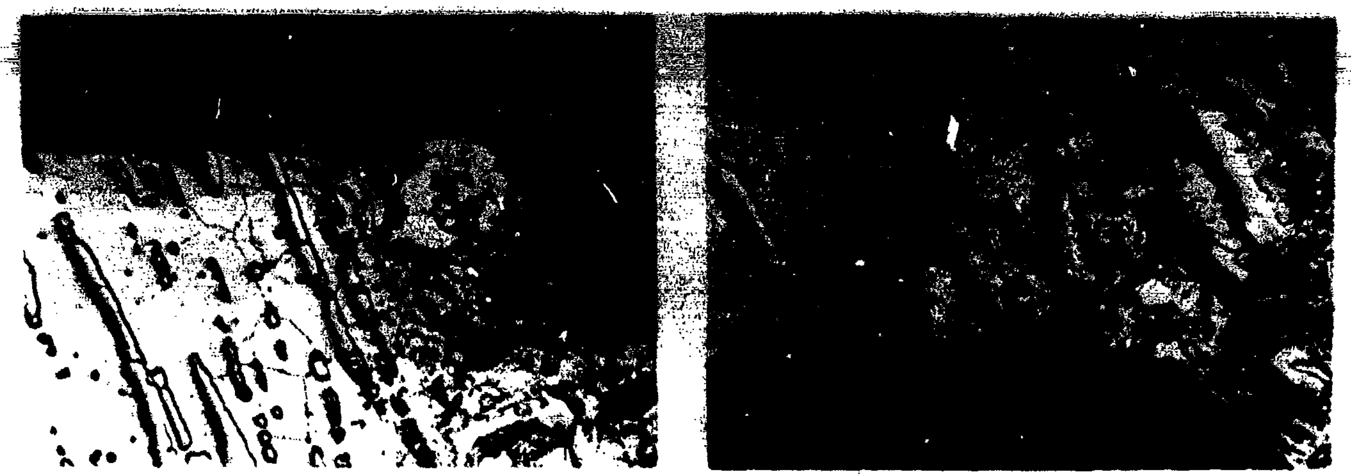
b) Flange Dome to Cylinder Girth Weld

c) Outlet Dome to Cylinder Girth Weld

All Views - Mag.: 10X Mat'l: 17-7PH SS Etch: Vilella's

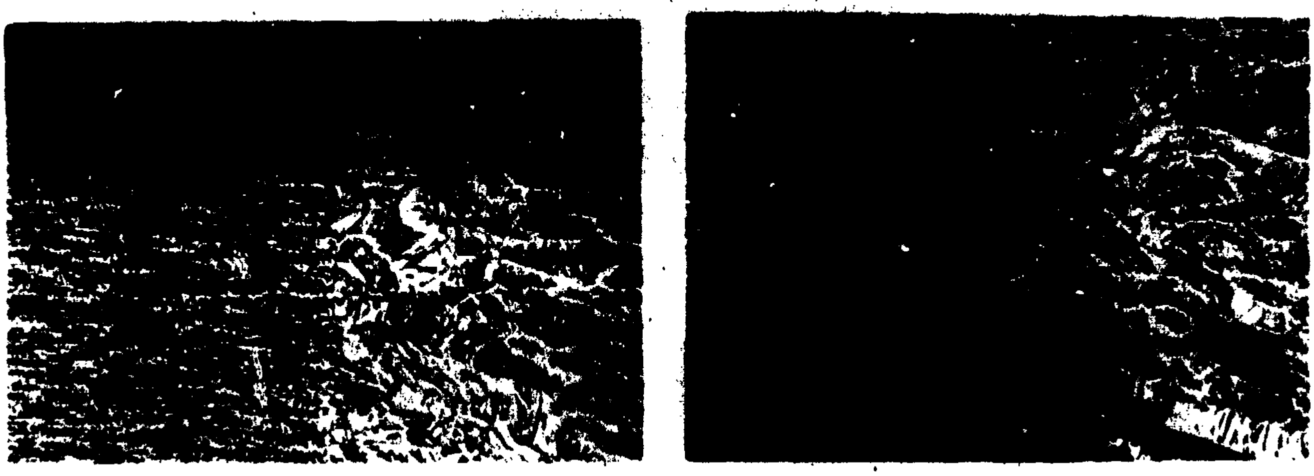
FIGURE 30. CROSS SECTION OF TANK SHELL WELDS FROM TANK S/N 5 SHOWING WELD BEAD CONFIGURATION

METALLURGICAL LABORATORY MET NO. 66 79016



OD Surface ID Surface

a) Flange to Dome Weld



OD Surface ID Surface

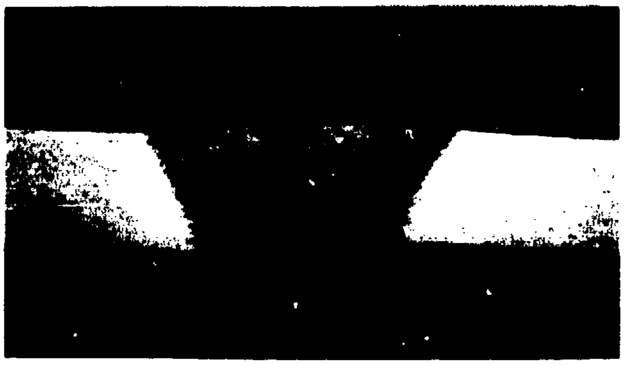
b) Girth Weld on Cylinder Side

All Views - Mag.: 100X Mat'l: 17-7PH Etch: Vilella's

FIGURE 31. HIGH MAGNIFICATION VIEWS OF TWO TANK S/N 5 WELDS SHOWING ABSENCE OF ANY SURFACE ATTACK.

METALLURGICAL LABORATORY MET NO. 76-79016

a) Flange tube to connector fitting weld



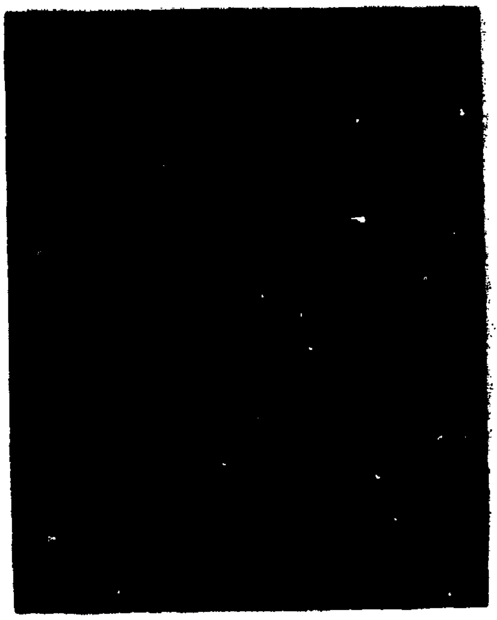
b) Connector fitting to extension tube weld

c) Outlet tube to connector fitting weld

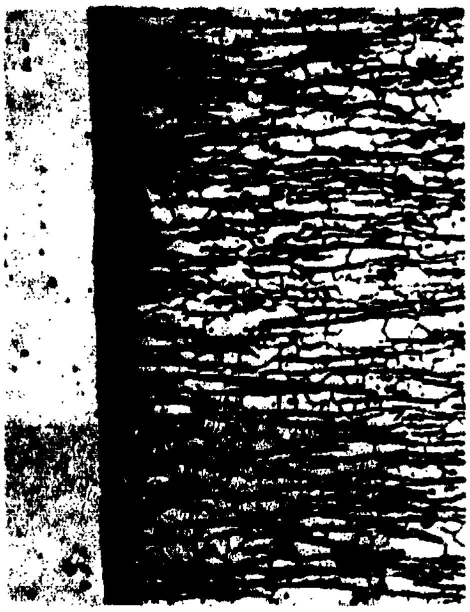
All views: Mag.: 10X Mat'l: AISI 347 SS Etch: Mixed Acids

FIGURE 32. CONFIGURATION OF TUBE WELDS FROM TANK S/N 1 PRODUCED BY "ASTRO ARC" PROCESS.

METALLURGICAL LABORATORY MET NO. 76-79016



Mag.: 10X



Mag.: 100X

a) Flange to tube weld showing no corrosion in extended crevice

Mag.: 10X

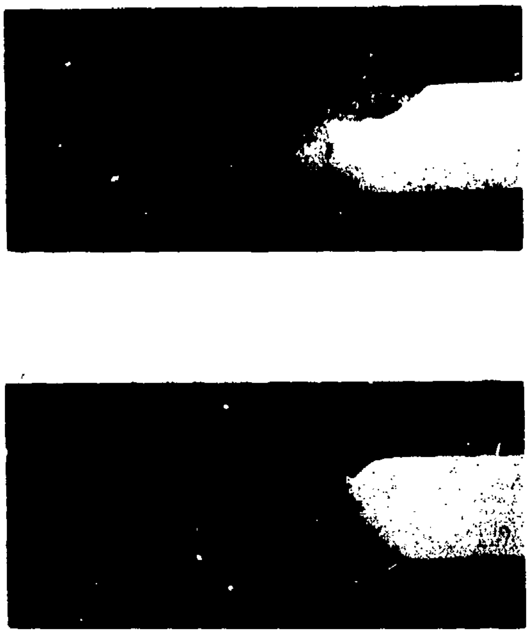
Mag.: 100X

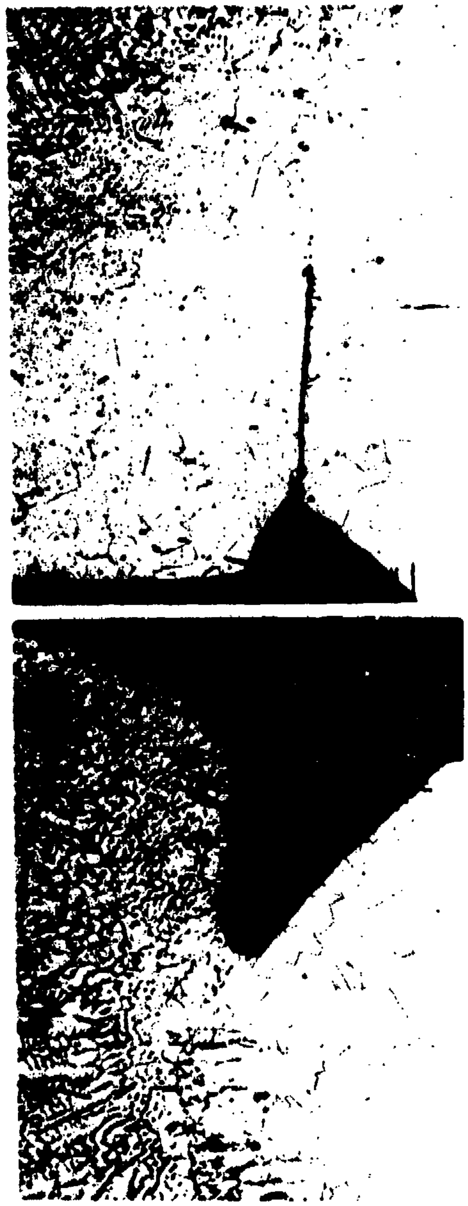
b) Outlet to tube weld with no apparent corrosion. This is region in Tank S/N 5 where corrosion was observed

All Views: Etch - Ammonium Persulfate Mat'l: AM350 Flange Welded to AISI 347 SS Tube

FIGURE 33. CONFIGURATION AND LACK OF CORROSION EFFECTS IN WELDS WHERE TUBING JOINS FLANGE OR OUTLET IN TANK S/N 1.

METALLURGICAL LABORATORY MET NO. 76-79016





Mag.: 10X

a) Overall view of weld joint showing considerable amount of original joint preparation still visible

Mag.: 100X

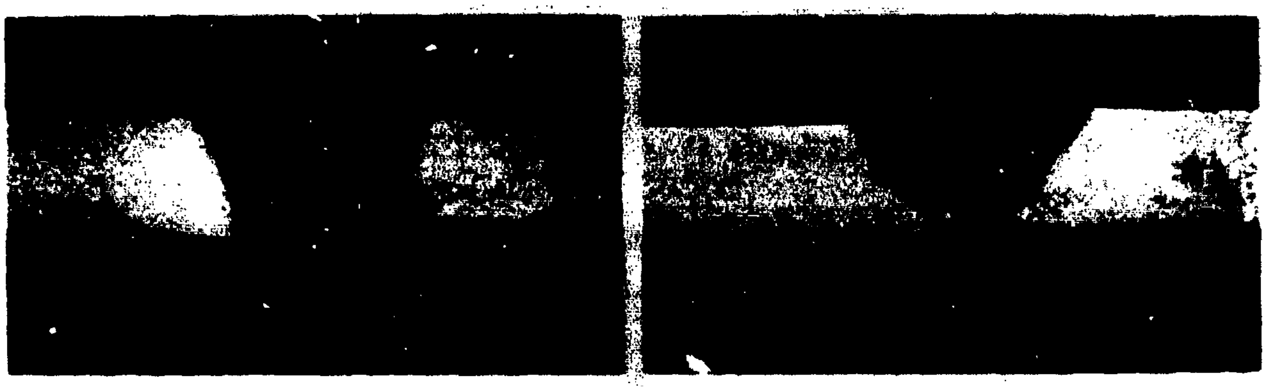
b) Detail views of unfused joint edges showing no corrosion or other effects

Mat'l: AISI 347 Stainless Steel

Etch: Mixed Acids

FIGURE 34. TUBE WELD FROM TANK S/N 4 (OUTLET CONNECTOR FITTING TO EXTENSION TUBE), SHOWING POOR LOCATING OF WELD TO ORIGINAL JOINT PREPARATION, GIVING CONSIDERABLE LACK OF FUSION BUT NO JOINT LEAKAGE.

METALLURGICAL LABORATORY MET NO. 76-79016

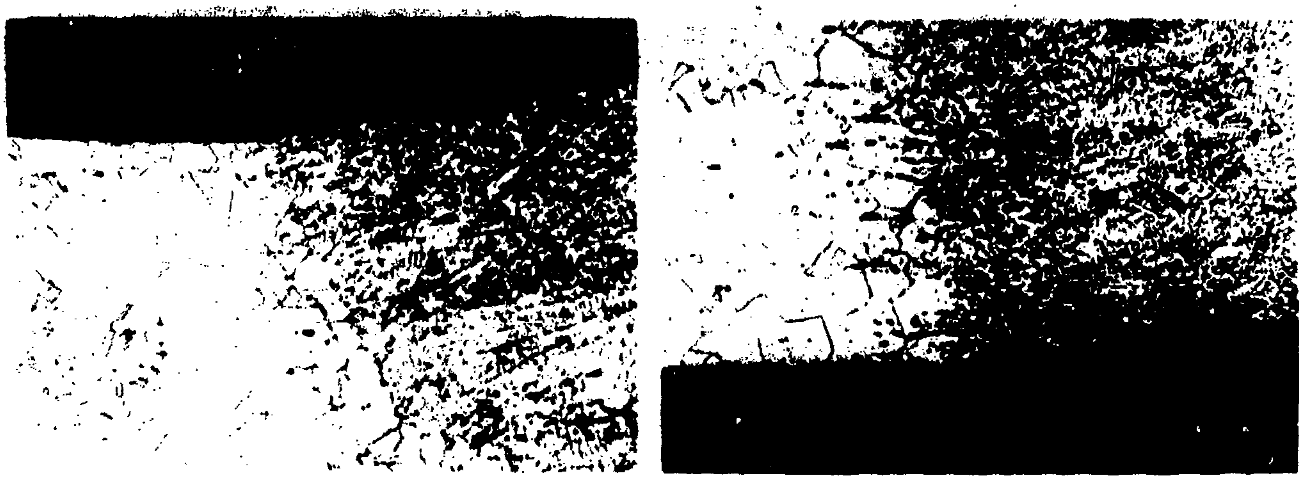


Mag.: 10X

Flange tube to connector fitting — Connector fitting to extension tube

a) Normal and desirable configuration of tube weld produced by Astro-Arc process

All Views: Mat'l: AISI 347 SS Etch: Mixed Acids



Mag.: 100X

OD Surface — ID Surface

b) Higher magnification view of connector to extension tube weld showing lack of corrosion effects

FIGURE 35. CROSS SECTION CONFIGURATION AND MICROSTRUCTURE OF TUBING WELDS AT FLANGE END OF TANK S/N 5.

METALLURGICAL LABORATORY MET NO. 76-79016

Mag.: 100X

a) Interior surface at location of corroded exterior of outlet fitting. Note crack-like discontinuity.

Mag.: 4X

b) Cross section of outlet fitting showing extreme thinning from machining plus corrosion

Mag.: 32X

c) View of corroded region showing extensive corrosion on exterior surface

Mag.: 4X

d) Cross section in outlet fitting of Tank S/N 4 showing thinning but not to extent of Tank S/N 5

Mag.: 200X

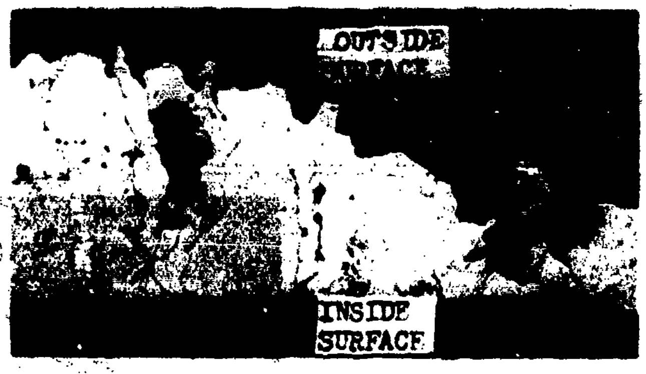
e) Close up view of corrosion attack and penetration to interior

FIGURE 35. INTERIOR AND CROSS SECTION VIEWS AT [illegible] OUTLET FITTING OF TANK S/N 5.

METALLURGICAL LABORATORY MET NO. 76-[illegible]

Mag: 50X

a) Overall view showing orientation of ferrite stringers across thinned down membrane of outlet fitting.

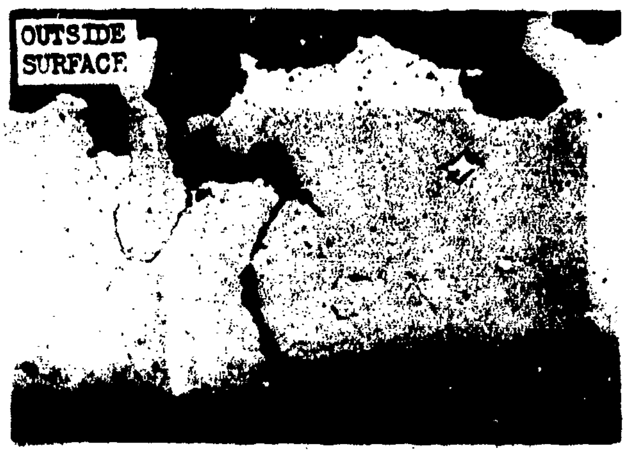


Mag: 200X

b) Area where matrix of transformed martensite has been preferentially attacked leaving ferrite stringers unattacked.

Mag: 500X

c) Detail view of area where attack is proceeding along ferrite to martensite phase boundary.



Mag: 500X

d) Detail view at leakage or complete penetration site.

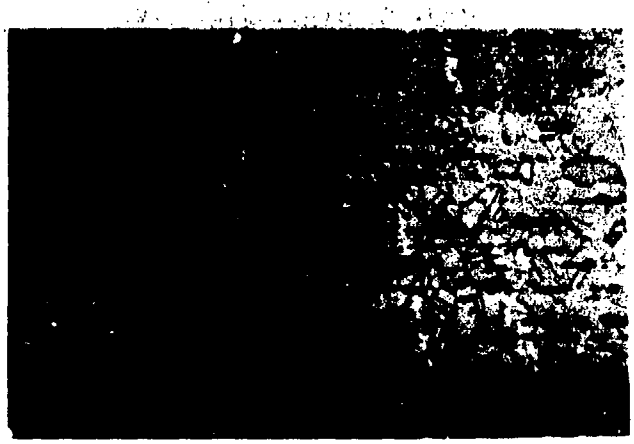
All views - Etch: Picric Acid/Hcl
Matl: 17-7 PH Stainless Steel

FIGURE 37. VIEWS OF LEAK AREA IN OUTLET OF TANK S/N 5 SHOWING RELATION OF ATTACK TO FERRITE AND TRANSFORMED MATRIX COMPONENTS OF MICROSTRUCTURE.

Mag: 10X

a) Overall view of tube to connector weld showing slight root concavity but a sound, fully fused joint.

Etch: Mixed Acids   Matl: AISI 347 SS

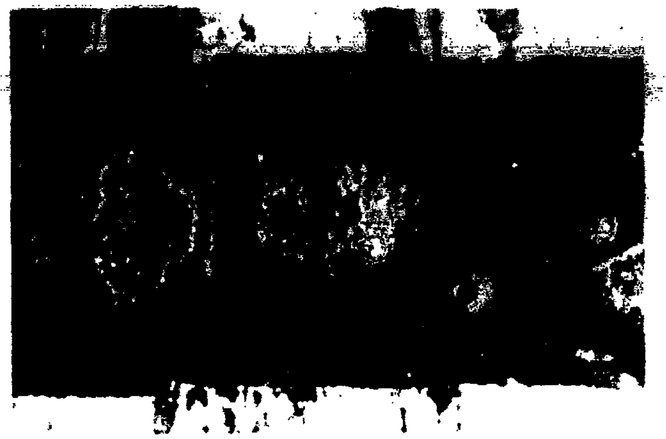


Outer Surface   Mag: 100X   Inner Surface   Mag: 100X

Edge of weld at exterior and interior surface showing no corrosive attack.

FIGURE 38. TUBE TO CONNECTOR WELD FROM OUTLET TUBE OF TANK S/N 5, ADJACENT TO AREA WHERE CORROSION WAS OBSERVED ON OUTLET FITTING.

METALLURGICAL LABORATORY
MET NO. 76-79016



Mag.: 5X

a) Overall view of sectioned tube showing deposits on weld and tube suggesting solids had dried in horizontal position

Mag.: 10X

b) Closeup view showing deposit crusted on surface of weld

Mag.: 10X

c) Cross section through weld illustrating concave underbead where deposit [illegible]

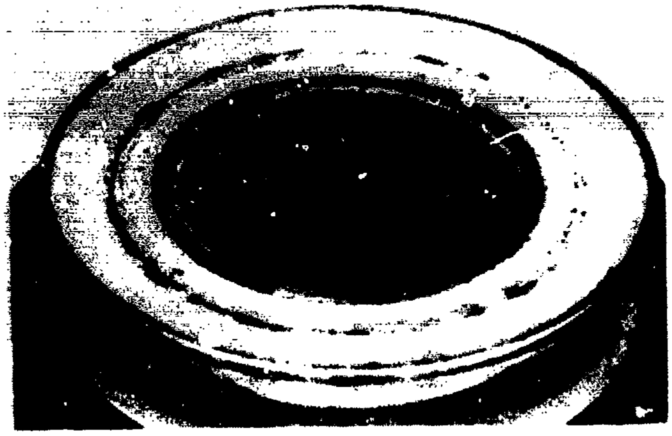
Mag.: 200X

d) Microstructure at weld underbead surface illustrating shallow corrosion observed

All views - Mat'l: AISI 321/347 Stainless Steel

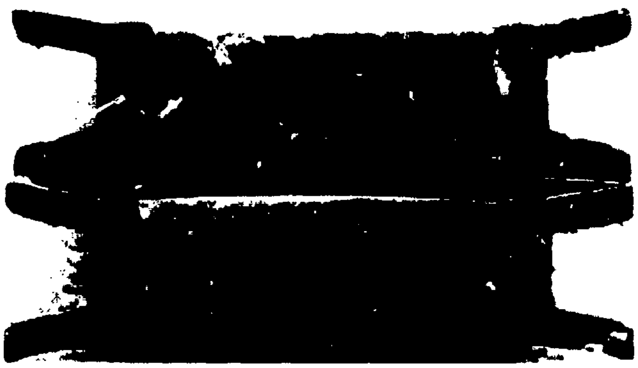
FIGURE 39. VIEWS OF DEPOSIT FOUND IN TUBE AT FLANGE END OF TANK S/N 5 AND CROSS SECTIONS OF UNDERLYING WELD.

METALLURGICAL LABORATORY MET NO. 76-79016



Mag: 5X

a) Overall view of RPL fitting sealing member showing deposits on inner surface in vapor phase.



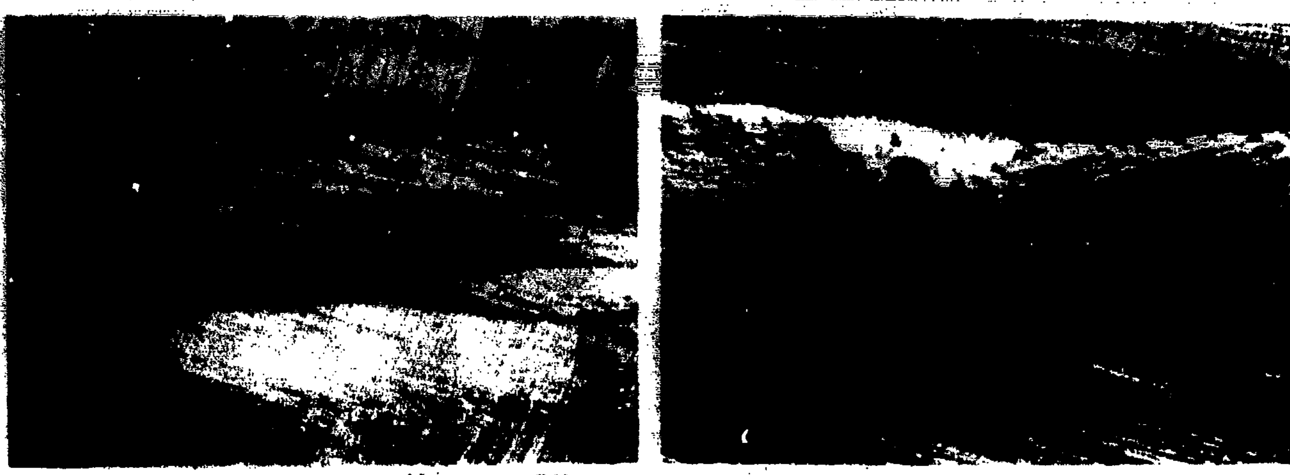
Mag: 5X

b) Deposits on surface of sealing member after sectioning.

Mag: 15X

c) Close-up view of deposit color is reddish brown to dark brown with occasional lighter brown streaks.

FIGURE 40. DEPOSITS ON INNER SURFACE OF SEALING RING FROM RPL CONNECTOR IN FLANGE TUBE OF TANK S/N 5.



Mag.: 3X Mag.: 10X

a) Area marked #1 showing spot of oxide or "slag" adhering to side of weld and giving the suggestion of remaining intact when a second pass (more rippled appearance) was made. Marked Area #1 is shown in Figure 8.

Mag.: 10X

b) Area marked #3 where a condition similar to (a) above has been partly obliterated by post-welding sanding or brushing. Marked Area #3 is shown in Figure 8.

FIGURE 41. SURFACE CONDITIONS VISIBLE ON WELD CROWN (EXTERIOR) IN LOCATIONS MARKED AS SUSPECT ON GIRTH WELD OF TANK S/N 3.

METALLURGICAL LABORATORY MET NO. 76-79016

Mag.: 3X

Mag.: 10X

Example of interior appearance (Area #1) where the surface oxide, probably from a previous weld has been cracked and broken during rewelding so that a discontinuous surface is formed. Marked Area #1 shown in Figure 8.

FIGURE 42. SURFACE CONDITIONS VISIBLE ON WELD UNDERBEAD (TANK INTERIOR) IN LOCATIONS MARKED AS SUSPECT ON GIRTH WELD OF TANK S/N 5.

METALLURGICAL LABORATORY MET NO. 76-79016

a) Section near (but not in) suspect areas showing no evidence of rewelding. At white arrow is the crest of a condition very similar to that shown in Figure 41.

Both views -

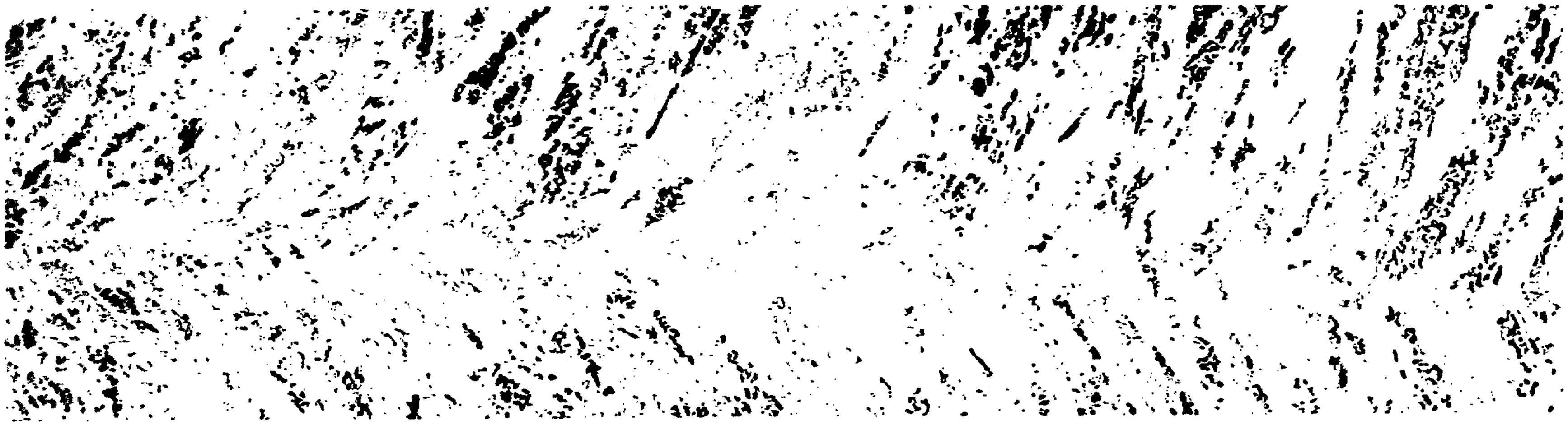
Mag: 32X

Etch: Picric Acid plus HCl

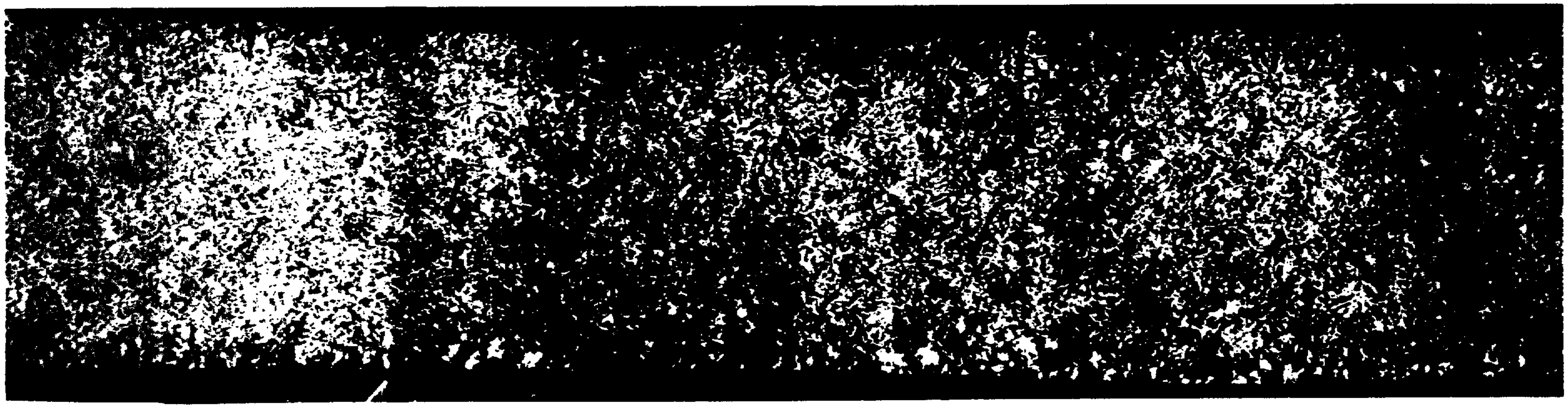
Matl: 17-7 PH Stainless Steel

b) Section at area approximately 180° from the marked suspect areas.

FIGURE 43. CROSS SECTIONS THROUGH PORTIONS OF THE GIRTH WELD OF TANK S/N 3.



a) Horizontal section near midplane of weld in area 1.



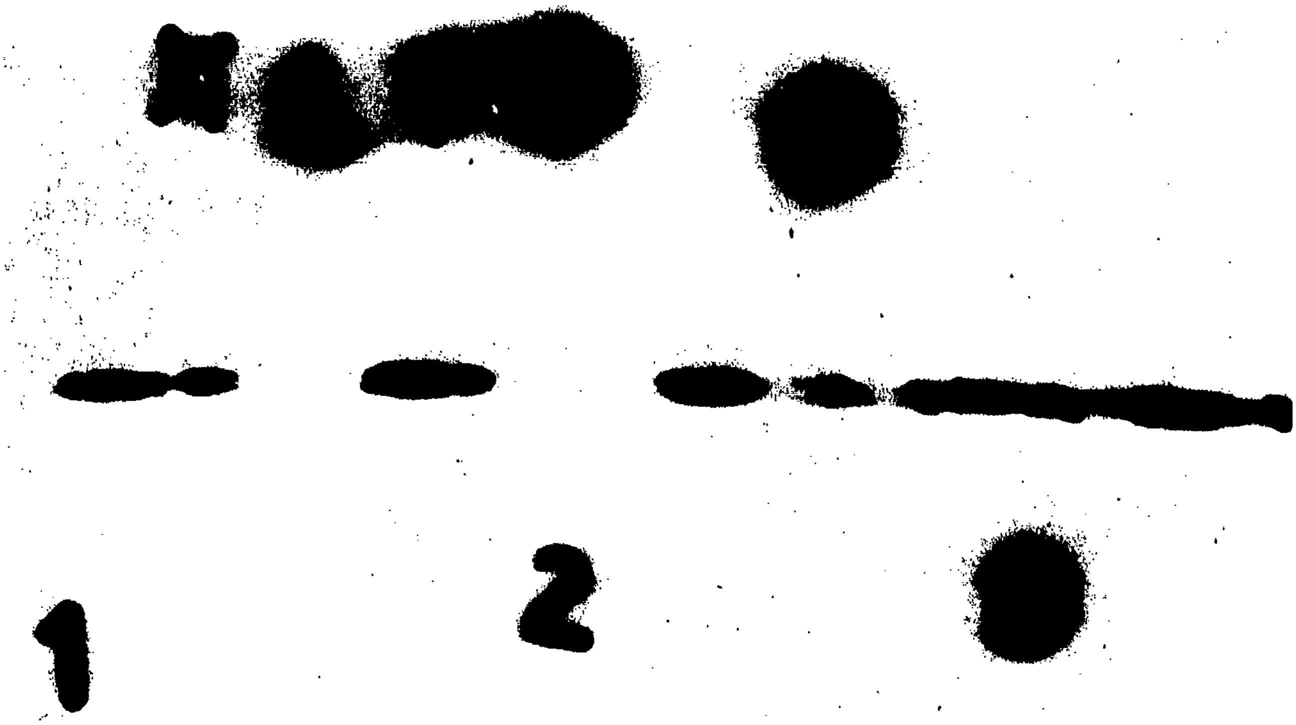
b) Vertical section along centerline of weld in area 4.

Mag: 32X

Etch - Picric Acid/Hcl   Matl: 17-7 PH Weld

FIGURE 44. EXTENDED VIEWS OF SECTIONS FROM REGIONS OF TANK S/N 3 GIRTH WELD WHERE SUSPECT AREAS HAD BEEN MARKED.

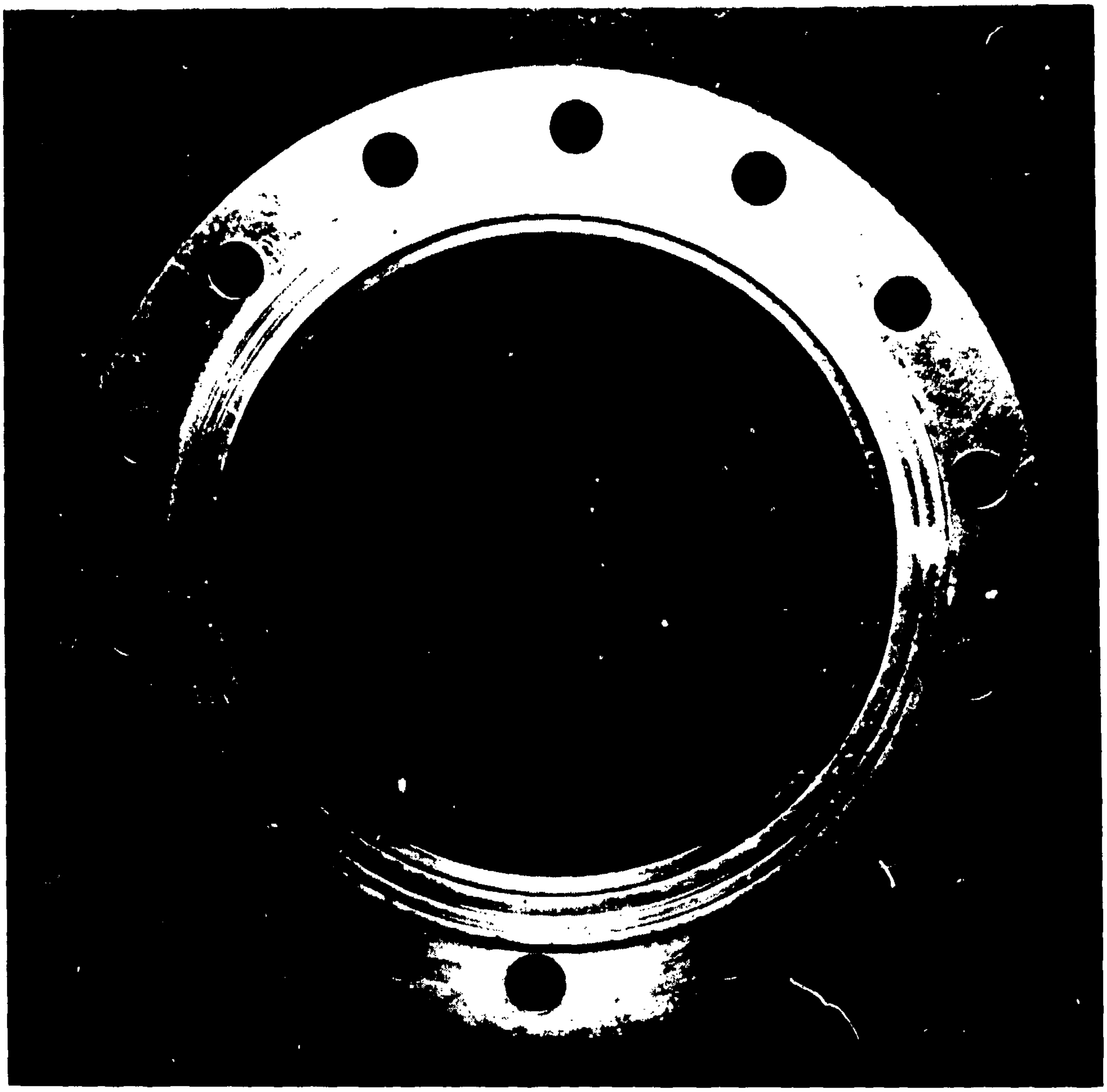
METALLURGICAL LABORATORY
MET NO. 76-79016



Print is magnified two times original size

Note: Reproduction of X-ray reverses the normal shading. Light areas on X-ray (meaning thicker section thickness) become dark areas in above print. Overall view of area studied is shown in Figure 8.

FIGURE 45. REPRODUCTION OF RADIOGRAPH FROM SUSPECT REGION OF GIRTH WELD OF TANK S/N 3.



[illegible] VIEW OF FLANGE [illegible]
[illegible] IN PLACE.

METALLURGICAL [illegible]
MET NO. [illegible]

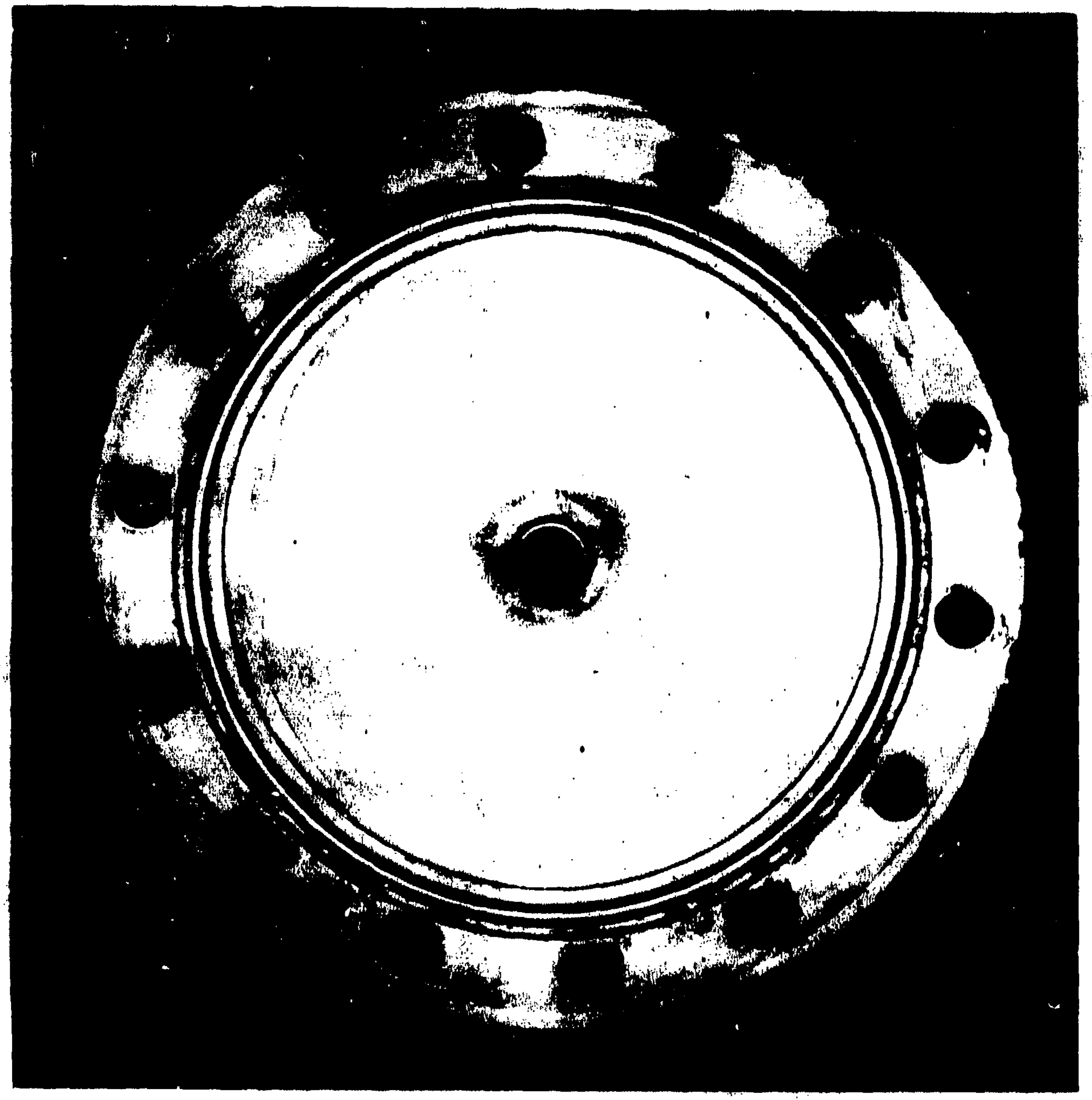
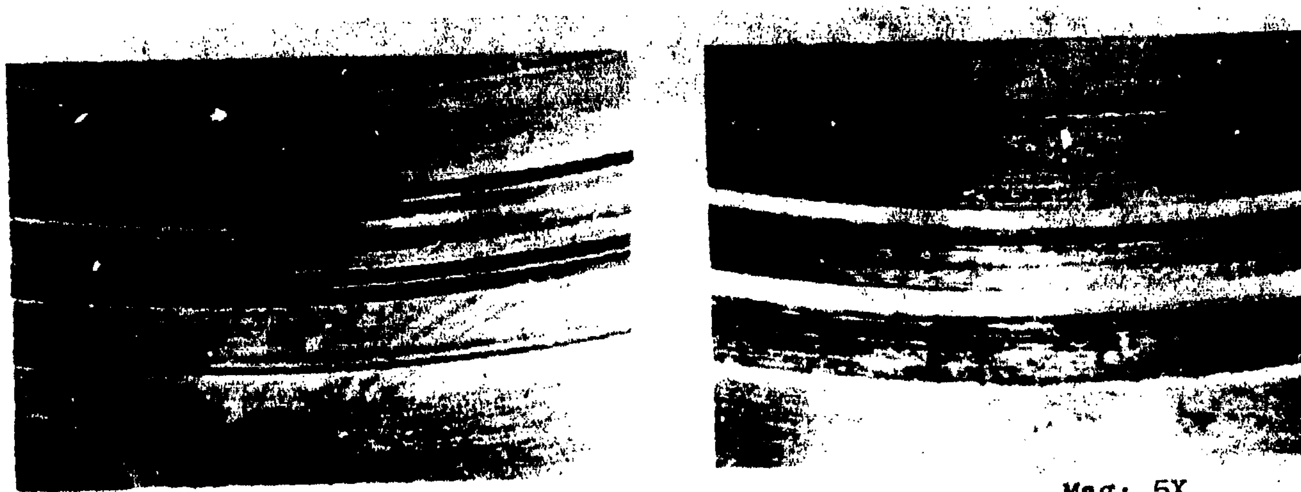


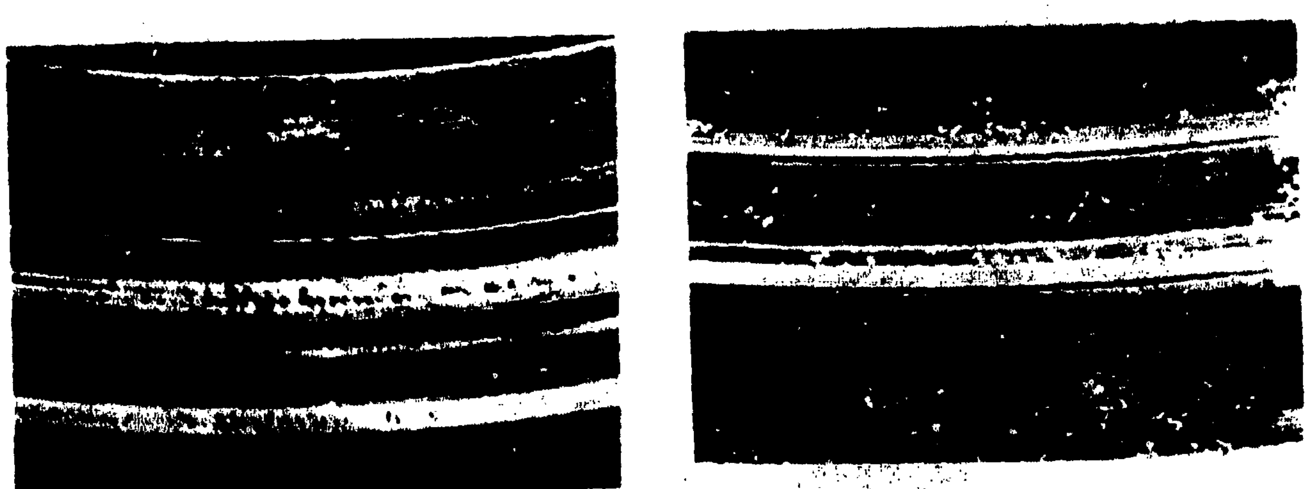
FIGURE 47. OVERALL VIEW OF FLANGE COVER FROM TANK S/N 3.

METALLURGICAL LABORATORY
MET NO. 76-79016



Mag: 5X　　　　Mag: 5X

FIGURE 48. DETAIL VIEWS OF SEALING GROOVE AND RIDGES IN FLANGE COVER. SLIGHT ROUGHNESS OR RIPPLING SHOWN AT RIGHT WOULD NOT AFFECT SEALING.



Mag: 5X　　　　Mag: 8X

a) Chattered and torn region.

b) Deposits and residue in form of "sand" found between seal ring and groove.

FIGURE 49. DETAIL VIEWS OF SEALING GROOVE AND RIDGES IN FLANGE. SMEARED AND TORN SURFACE ON RIDGE IS MOST CRITICAL TO SEALING CAPABILITY.

METALLURGICAL LABORATORY
MET NO. 76-79016

a) Sealing ring while still in place on flange. Minor staining of aluminum is result of one month water immersion for leak test.

b) Side of ring in contact with flange after removal. Note staining on hydrazine side up to first sealing ridge.

c) Sealing ring in region of chattered surface of flange. Note that aluminum formed around torn out ridge.

Mag: 5X

FIGURE 50. DETAIL VIEWS OF ALUMINUM SEALING RING FROM TANK S/N 3.

METALLURGICAL LABORATORY
MET NO. 7C-79016

Mag.: 10X

a) Cross section through sealing ring groove where some chattering occurred. Ridge at arrow was slightly torn. No material anomaly is visible to explain chattering and tearing.

Etch: Mixed Acids Mag.: 100X

b) Microstructure showing very elongated ferrite stringers in a matrix of transformed martensite.

FIGURE 51. MICROSTRUCTURE AND GRAIN ORIENTATION IN FLANGE UNDER SEAL RING GROOVE OF TANK S/N 3.

METALLURGICAL LABORATORY MET NO. 76-79016