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INVESTIGATION OF PROBLEMS ASSOCIATED WITH THE USE OF ALLOYED MOLYBDENUM SHEET IN STRUCTURES AT ELEVATED TEMPERATURES

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SUMMARY

The results of an experimental study to explore the capabilities and limitations of thin Mo-0.5Ti molybdenum-alloy sheet for structural applications at high temperatures are presented. Evaluation tests at temperatures ranging from room temperature to 3,000° F were made on resistance-welded corrugated-core sandwiches that were coated with a commercially available oxidation resistant coating known as W-2 and on coated oxidation and tensile specimens. The performance of the corrugated-core sandwiches in compressive strength and static oxidation tests, tensile properties of the coated molybdenum sheet, and the life of the coated specimens in static oxidation tests are given. A description of the equipment and procedures utilized in performing the evaluation tests is included.

INTRODUCTION

Molybdenum has often been considered to be a potentially useful material for structural applications at high temperatures. Numerous studies have been made to obtain basic knowledge on the characteristics and behavior of the metal, to develop usable and improved alloys, and to obtain coatings that permit its use at high temperatures. (See, for example, refs. 1 to 8.) The expected usefulness of molybdenum as a structural material is based on its high melting point of approximately 4,750° F and the retention of useful strength and stiffness above 2,000° F. The use of molybdenum has been limited, however, because of some undesirable characteristics that include brittleness, poor oxidation resistance, lack of uniformity and reliability, and numerous fabrication and joining problems.

In order to explore some of the capabilities and limitations of molybdenum for structural applications, a study was undertaken (1) to investigate fabrication methods including resistance welding of thin sheet, (2) to evaluate an available coating for protection of thin sheet from oxidation at high temperatures, (3) to develop reprocessing procedures that might improve the quality of commercially available sheet, and (4) to fabricate and perform evaluation tests on small structural components at high temperatures. The study was a coordinated effort between the National Aeronautics and Space Administration and several outside organizations. The design, testing, and evaluation of the specimens was performed by the Langley Structures Research Division. Preparation of the specimens including fabrication, coating, reprocessing of commercially available sheet, and other related efforts were performed by organizations outside the NASA.

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The results of various evaluation tests that include axial compression tests and oxidation tests on the sandwiches, tensile stress-strain tests on bare and coated sheet, and oxidation tests on small coated strips are reported herein. Details of the equipment and procedures used to perform the evaluation tests are also given.

SPECIMENS

The specimens in this study were fabricated from 0.010-inch-thick molybdenum-alloy sheet (molybdenum alloyed with 0.5-percent titanium). This molybdenum-alloy sheet is hereinafter referred to as Mo-0.5Ti sheet. The specimens consisted of the resistance-welded corrugated-core sandwiches shown in figure 1, the tensile specimens shown in figure 2, and small oxidation strips of 0.010 by 1/2 by 1 inch. All specimens were fabricated by the Aeronautical Division of the T. R. Finn & Company, Inc., Hawthorne, N. J. (now a part of Thermionic Products Company, Plainfield, N. J.).

Commercially available Mo-0.5Ti sheet in the stress-relieved condition was used in some of the tensile specimens for this study. The molybdenum-alloy sheet in all the other specimens was specially reprocessed.

Exploratory studies conducted by the fabricator revealed various fabrication difficulties which were believed to be caused by the nonuniformity and marginal quality of the commercially available molybdenum-alloy sheet. Accordingly, the commercially available sheet was reprocessed in an attempt to improve the quality of the sheet and to alleviate some of the fabrication difficulties. The reprocessing procedures, developed by the Thermionic Products Company, consist of a series of cold-rolling and heat-treating operations. The specific details of the reprocessing procedures are proprietary to the Thermionic Products

Company; however, some of the general details of these procedures are available and are presented in appendix A.

The reprocessing procedures improved the quality of the Mo-0.5Ti sheet and enabled the fabricator to produce uniform and consistent resistance welds. For these reasons, all of the resistance-welded corrugated-core sandwich specimens were fabricated from the reprocessed molybdenum sheet.

Hereinafter commercially available Mo-0.5Ti sheet in the stress-relieved condition will be referred to as stress-relieved sheet, and the Mo-0.5Ti sheet that was obtained in a stress-relieved condition and was subsequently subjected to the reprocessing procedures will be referred to as reprocessed sheet.

The oxidation-resistant coating used in this study is commercially available and is known as W-2. This proprietary coating was applied on the specimens by the Chromalloy Corporation, White Plains, N. Y. With the exception of a few bare specimens tested at room temperature, an 0.001-inch-thick coating was applied on the corrugated-core sandwiches and the tensile specimens. The W-2 coating on the oxidation specimens was applied by a modified coating process. The coating thickness for these specimens was 0.002 inch.

RESULTS AND DISCUSSION

Mechanical-Properties Tests

Tensile stress-strain tests were performed to determine mechanical properties for bare and coated sheet in the stress-relieved and the reprocessed condition. Details of the test procedures and the testing equipment are given in appendix B.

Some of the tensile properties obtained from room-temperature tests on bare and coated Mo-0.5Ti sheet in the stress-relieved and in the reprocessed conditions are noted in tables I and II, respectively. In these tests and in all other tests involving loading on the coated sheet, the cross-sectional areas of the specimens prior to application of the coating were used to reduce the applied loads to stresses. A graphical comparison of the properties listed in tables I and II is given in figure 3. The yield stress of the bare sheet in the reprocessed condition exceeds the yield stress of the stress-relieved sheet by approximately 20 percent. After application of the W-2 coating which involves exposure of the sheet to temperatures in the vicinity of 2,000° F for a few hours, the yield stresses of both sheets were approximately the same. In contrast, considerable difference exists between the elongation values. The elongation values of the bare and the coated reprocessed sheet were 1/3 and 1/2, respectively, of the elongation values of the bare and coated stress-relieved sheet. Examples of the room-temperature tensile stress-strain curves representative of the bare and coated sheet in the stress-relieved and in the reprocessed conditions are shown in figure 4. Figure 5 indicates the types of fractures that were obtained with the tensile specimens. Fractures characteristic of a brittle material were obtained in all specimens.

Elevated-temperature tensile stress-strain curves are shown in figure 6 for the coated reprocessed molybdenum-alloy sheet, and significant material-properties data obtained from these stress-strain tests are listed in table III. Plots of the variation of these material properties with temperatures from room temperature to 3,000° F are presented in figures 7 to 9. Examples of the fractures that were obtained in the tests at several temperatures are shown in figure 10. The fractures are representative of a brittle material.

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The grain structures in the uncoated stress-relieved sheet and in the uncoated reprocessed sheet are given in figures 11 and 12, respectively. The fibrous-grain characteristics of rolled molybdenum sheet appear more uniform in the reprocessed sheet in figure 12 than in the stress-relieved sheet shown in figure 11. The polishing procedures and the etchant used in the preparation of specimens shown in figures 11 and 12 and in all other photomicrographs in the present report parallel the procedures described in reference 9.

Oxidation Tests of Coated Molybdenum Sheet

In order to determine the oxidation protection afforded the molybdenum sheet at elevated temperatures by the W-2 coating, small strips of the 0.010-inch-thick reprocessed sheet were coated and exposed at elevated temperatures in air in a tube furnace. These oxidation specimens were approximately 1/2 inch wide and 1 inch long. A description of the equipment and test techniques is given in appendix C.

The W-2 coating is based on molybdenum disilicide as a major component (ref. 8) and is applied at a relatively low temperature which does not recrystallize the molybdenum. The method of application parallels the application of chromized coatings that are applied by pack deposition techniques (ref. 3).

A few exploratory oxidation tests were made at 2,500° F to calibrate the test equipment and to develop test techniques. The specimens tested in these preliminary studies were 0.010 inch thick with an

0.001-inch-thick W-2 coating. This thickness of coating in some cases provided oxidation protection for the sheet for several hours at 2,500° F; however, considerable variation in failure time was obtained and failure of the coating usually occurred at the edges of the specimens. In order to overcome this tendency for edge failures, the supplier of the coating modified the method for application of the W-2 coating. In the modified method, approximately 90 percent of the coating was applied in the initial coating operation. The specimens were then liquid honed to remove irregularities in the coating particularly at the specimen edges. The specimens were again subjected to the coating operation and the remaining 10 percent of the coating was added in this second operation. The final thickness of the coating applied by this modified method was approximately 0.002 inch.

The life of the oxidation specimens coated by this modified method is given in figure 13 and in table IV. In figure 13 the life of the specimen is defined as the exposure time at test temperature during which no visible evidence of molybdenum trioxide was obtained. When molybdenum trioxide was noted, rapid oxidation of the specimen generally occurred. Complete destruction of the small oxidation specimen usually occurred within 30 minutes after the first evidence of molybdenum trioxide was obtained. Note that the minimum life was obtained in the vicinity of 2,600° F. Above this temperature, the coating contains a liquid phase that promotes self-healing of flaws in the coating and extends specimen life. No tests were made above 2,800° F because of the limited quantity of test specimens. On the basis of test results given in reference 3, it appears that if the curve shown in figure 13 were extended above 2,800° F the curve would approach zero specimen life in the vicinity of 3,300° F to 3,500° F.

Cross-sectional views of the coated Mo-0.5Ti sheet in the reprocessed condition are given in figures 14 to 16. In figure 14 a nominal 0.001-inch-thick coating on the 0.010-inch-thick reprocessed sheet is indicated. This thickness of coating produces a 4-percent weight increase in the molybdenum sheet. This coating is representative of that applied on both the tensile specimens and the corrugated-core sandwiches. In figure 15 is shown the W-2 coating with a nominal coating thickness of 0.002 inch. This coating thickness produced a 7-percent weight increase in the bare sheet. This view was obtained from one of the oxidation specimens that were coated by the modified coating method previously outlined.

Views of the edges of the W-2 coated sheet are given in figure 16. Figure 16(a) indicates the edge of a sheet with a nominal 0.001-inch W-2 coating. Note that an irregular buildup of the coating occurred at the edge that would be prone to fracture. A view of an edge of a sheet that was coated to a nominal thickness of 0.002 inch by the modified coating method is shown in figure 16(b). Some improvement in the uniformity of the coating on the edge of the sheet was obtained with this modified coating method. Further improvement in the uniformity of the coating on the edge was obtained by rounding and beveling the edge of the sheet as indicated in figure 16(c). It appears that the coating shown in figure 16(c) would be least prone to fracture and that the coating shown in figure 16(a) would be damaged most easily. The edges of the oxidation specimens for which results are given in figure 13 generally resembled the edge view shown in figure 16(b).

Compressive Strength Tests of Corrugated-Core Sandwiches

The resistance-welded corrugated-core sandwich specimens were subjected to axial compressive loading tests at temperatures ranging from room temperature to 3,000° F. These tests were made to establish maximum compressive strength and to determine the performance of the resistance welds and the coating under compressive loading at elevated temperatures. A description of the equipment and procedures used in these compressive strength tests is given in appendix D.

The maximum compressive strengths determined from these tests are given in figure 17 for the temperature range from room temperature to 3,000° F, and examples of curves of average stress plotted against unit shortening obtained from tests at temperatures from room temperature to 2,100° F are given in figure 18. The symbols shown in figure 17 identify the maximum compressive strengths which are given in table V and the trend of these data is indicated by the solid curve. The tensile yield stress obtained from figure 7 is shown by the dashed curve. Note that the compressive strength of the sandwiches exceeds the tensile yield stress for the material at the higher test temperatures up to approximately 2,700° F. The higher compressive strengths of the sandwiches compared to the tensile yield stress may be the result of several factors. It was assumed that the load-carrying cross-sectional area of the coated sandwich was equal to the cross-sectional area of the bare sandwich. The 0.001-inch-thick coating increased the overall sheet thickness by 20 percent. This increase in thickness provided additional load-carrying material and additional stiffening for the sheet. Furthermore, the grain direction of the sheet in the sandwiches was perpendicular to the direction of loading. By contrast, the grain direction in the tensile specimens was parallel to the direction of loading. In view of the cold work to which the reprocessed sheet was subjected, differences in the mechanical properties between the with-grain and cross-grain directions were anticipated. These differences in mechanical properties would contribute to differences between the compressive strength of the sandwich and the tensile yield stress of the material.

The proportions of the molybdenum sandwiches were selected so that local buckling of the individual plate elements at elevated temperatures would occur in the inelastic or plastic stress range of the Mo-0.5Ti sheet. In the axial compressive strength tests on the sandwiches, wrinkles rather than local buckles were obtained. The wrinkles extended across the face sheets at right angles to the direction of loading shown in the view of the tested specimen in figure 19. The formation of the wrinkles probably occurred because of large spot-weld spacing. The nominal spacing for the spot welds was 1/3 inch. The maximum strength of the sandwiches was probably affected by the formation of these wrinkles; however, an accurate estimate of the magnitude by which the maximum load was reduced cannot be made without compression stress-strain data which were not obtained in this study. The distance between welds was dictated by practical considerations associated with the resistance welding of thin molybdenum sheet. Attempts to produce resistance welds that were spaced more closely resulted in shunting of the electrical power through the adjacent weld. Further efforts will be required to develop resistance welding techniques that will permit close spacing of the spot welds. On the basis of the strength test results the performance of the resistance welds was judged to be satisfactory at all temperatures. It is recognized that axial loading tests may not provide a critical test for the quality of the welds. Various additional tests would be required to establish the performance and limitations of the resistance welds.

A representative example of the resistance welds in the sandwich is shown in figure 20. The face sheet and corrugation of an uncoated sandwich in the vicinity of a weld is shown in figure 20(a) to indicate the grain structure of the weld. An enlarged view of the well nugget is shown in figure 20(b). The weld indicated in figure 20 was obtained from a sandwich specimen that had been subjected to an axial compressive strength test. No evidence of cracks or other signs of failure were found in the weld area.

Oxidation Tests on Corrugated-Core Sandwiches

Two W-2 coated sandwich specimens were tested at 2,400° F and 2,700° F, respectively, to evaluate the performance of the welded and coated specimens under prolonged exposure at high temperatures. The nominal thickness of the W-2 coating on these specimens was 0.001 inch. The specimens were inserted into a heated furnace and maintained at test temperature until portions of the sandwiches were destroyed by oxidation. A description of the test equipment and test procedures is given in appendix E.

One of the sandwich specimens was exposed at 2,400° F for 2.2 hours. Evidence of oxidation characterized by the formation of visible molybdenum trioxide was obtained after approximately $1\frac{3}{4}$ hours of exposure. The other sandwich specimen was exposed at 2,700° F for 3.2 hours. In this test molybdenum trioxide was noted after approximately 2 hours of exposure. An example of the type of failure that occurred in the oxidation test is shown in figure 21.

The coated sandwich specimen prior to testing is shown in figure 21(a), and the specimen after exposure at 2,700° F for 3.2 hours is shown in figure 21(b). Oxidation damage is visible; however, the full extent of this damage is not evident because the coating covers some of the oxidized areas. The full extent of the oxidation damage is indicated in part (c) of figure 21 which shows the same specimen viewed L
from approximately the same direction. A probe was used to remove the 1
coating to reveal the holes that had been oxidized in the molybdenum 0
sheet. Examination of the oxidized specimen indicates that most of the 0
oxidation damage occurred at the spot welds. It appears that the welds 4
were not coated adequately although the areas along the weld lines between the welds were protected satisfactorily by the coating. This result suggests that the welds were not cleaned sufficiently prior to application of the coating. It is believed that the surface cleanliness in the weld area rather than difficulty of access for the coating is primarily responsible for the type of oxidation failure that was obtained.

A cross-sectional view of the weld area in the coated sandwich is shown in figure 22. This specimen was obtained from a coated sandwich that had been subjected to axial compressive loading at room temperature to determine maximum strength. The upper sheet shown in the figure indicates the 0.010-inch-thick face sheet and the lower curved sheet indicates the corrugation. Note that a relatively uniform layer of W-2 coating is visible on the upper surface of the face sheet and on the lower surface of the corrugation. By comparison, relatively little coating is visible on the lower surface of the face sheet or on the upper surface of the corrugation particularly in the area adjacent to the weld. An enlarged view of a portion of this weld area is shown in part (b) of figure 22. This enlarged view shows the right-half portion of part (a) of figure 22 and is included to illustrate more clearly the lack of coating on the inner surfaces adjacent to the weld area. Practically no evidence of the coating can be seen on the surfaces at the junction between the face sheet and the corrugation.

Attention is called to the fact that the coating on the sandwiches was applied in one operation and produced an 0.001-inch-thick protective layer. The life of these sandwiches consequently was not expected to be in close agreement with the results given in figure 13 for the oxidation strips. The coating on these strips as noted previously was applied by using a modified coating procedure which produced a coating 0.002 inch thick. In view of the thicker coating and greater simplicity of the oxidation specimen, the longer life at high temperatures obtained with the oxidation strips was expected.

SUMMARY OF RESULTS

This study was made to investigate the capabilities and limitations of coated Mo-0.5Ti sheet for structural applications at high temperatures. The following comments and conclusions are made on the basis of this experimental study:

1. The W-2 coating produced some decrease in the room-temperature strength of the molybdenum sheet and practically no change in Young's modulus or elongation. This result indicates that if a thin sheet of alloyed molybdenum is acceptable for structural application in the uncoated condition, the application of an oxidation resistant coating will not necessarily embrittle the sheet or otherwise impair its usefulness for structural applications.

2. A 0.001-inch-thick W-2 coating did not give adequate oxidation protection for thin molybdenum sheet at high temperatures. With this thickness of coating, oxidation strips displayed a tendency for edge failure which produced erratic life at high temperatures. The corrugated core sandwiches with the same coating thickness failed by oxidation of the resistance-weld areas. Considerable improvement in elevated-temperature performance was obtained with a 0.002-inch thickness of coating on oxidation specimens. The marginal performance in the coated sandwiches in the oxidation tests is attributed in part to an inadequate deposition of the W-2 coating in the weld areas. The use of the thicker coating of 0.002 inch is expected to help overcome this deficiency. It also appears that further study is needed to improve the methods of application of the coating on welded sheetmetal assemblies.

3. Further efforts to improve resistance-welding techniques for thin molybdenum sheet appear necessary. The resistance welds in the corrugated-core sandwiches performed satisfactorily; however, the spacing along the weld lines was greater than desired. The minimum spacing was dictated by practical welding considerations. When the spacing between successive welds was too small the electrical power shunted through the adjacent welds.

4. It is expected that many of the fabrication and coating problems encountered in this study will be alleviated when improved quality molybdenum sheet becomes commercially available. The difficulties experienced by the fabricator in resistance welding of the corrugated-core sandwiches were alleviated considerably when reprocessed molybdenum sheet was used. This improvement is attributed in part to superior surface finish, lack of surface oxides and other contaminants, and the elimination of delaminations and local imperfections within the sheet. In view of this

experience, it appears that continuing efforts are needed to improve the uniformity and overall quality of molybdenum-alloy sheet.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Field, Va., May 26, 1960.

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APPENDIX A

REPROCESSING PROCEDURES

Reprocessing procedures were initiated in this study for the purpose of improvement in the overall quality and reliability of the alloyed molybdenum sheet. In the development of these procedures, commercially available stress-relieved sheet was subjected to various operations including cold rolling and heat treating. Some of the procedures that were investigated were patterned after methods that had been developed by Thermionic Products Company in drawing and annealing of refractory metal wires. On the basis of limited exploration the following reprocessing procedure was selected for the sheet material used in this study. First, the commercially available stress-relieved sheet was subjected to cold rolling to reduce the thickness approximately 0.002 inch and to detect sheets of poor quality which delaminated or showed evidence of edge cracking. Following this cold-rolling operation, the sheet was subjected to heat treatment in special atmospheres and further cold rolled to reduce the thickness to within 0.003 inch of the desired thickness. The sheet was again heat treated in special atmospheres and cold rolled to the final thickness of 0.010 inch.

The initial thicknesses of the commercially available stress-relieved molybdenum sheets obtained for this study ranged from 0.020 inch to 0.038 inch prior to reprocessing. Because of the exploratory nature of this reprocessing study only 2-inch-wide strips were reprocessed and these strips were the widest sheet available for the specimens in the present study.

APPENDIX B

EQUIPMENT AND PROCEDURES FOR TENSILE STRESS-STRAIN TESTS

The tensile stress-strain tests were performed on the tensile specimens indicated in figure 2 in a 120,000-pound universal hydraulic testing machine.

All tests were performed at a nominal strain rate of 0.005 per minute up to the yield stress and at an increased rate of approximately 0.05 per minute above the yield stress. In the elevated-temperature tests, the specimens were heated to the desired test temperature and exposed at temperature for 0.1 hour prior to loading.

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In the room-temperature tests the strain in the specimen was determined over a 1-inch gage length with the aid of two linear variable differential transformer gages mounted back-to-back on the specimen. The tungsten extensometer assembly shown in figure 23 was used to determine the strains in the specimen in the elevated-temperature tests. This extensometer consisted of an assembly of tungsten rods that were clamped 1 inch apart on the test specimen by means of the tungsten disks. The specimen strain was transferred by tungsten strain transfer rods to a lever system that actuated linear variable differential transformer gages. The tungsten extensometer performed satisfactorily in the high-temperature tests. No evidence of slipping of the tungsten rods was observed. Slipping did not appear likely because the coated specimens provided a coarse surface on which the rods were clamped. Furthermore, the assembly was self-tightening with increasing temperature because the coefficient of thermal expansion of the test specimen exceeded that of the tungsten rod and disk assembly. Oxidation of the uncoated tungsten extensometer occurred at the high test temperatures. The magnitude of the oxidation was slight and this oxidation did not impair the usefulness of the assembly.

Heating of the tensile specimens was accomplished with an induction heater and graphite susceptor shown in figure 24. An overall view of the equipment utilized in the tensile stress-strain tests is shown in figure 25. Further details on the induction heating apparatus and the susceptor are given in appendix D. The temperature of the specimens was determined from measurement of the air temperature in the vicinity of the 1-inch gage length with the aid of a platinum-platinum, 13 percent rhodium thermocouple. The thermocouple was not attached directly on the tensile specimen in order to avoid possible damage to the W-2 coating. In addition, the use of uncoated thermocouples in the presence of the W-2 coating did not appear feasible. Limited experience with the W-2 coating and platinum-platinum, rhodium thermocouples indicated that a high-temperature reaction occurred between the wire and coating. This reaction caused melting of the thermocouple wires in contact with the coating and led to destruction of the thermocouple junction.

Calibration tests were made to determine whether significant temperature differences existed between the heated specimen and the air temperature near the heated specimen. These tests were made with the use of a nickel-alloy tensile specimen to which thermocouples were spot welded. Temperature differences between the nickel-alloy specimen and the air near the heated specimen as well as the magnitude of the temperature variations along the heated length of the specimen were determined in these calibration tests. The difference between specimen temperature and air temperature was obtained in tests up to 2,000° F. Temperature variations along the 1-inch gage length as well as differences between the specimen and air temperature were found to be on the order of ±0.5 percent.

APPENDIX C

EQUIPMENT AND PROCEDURES FOR OXIDATION TESTS

Oxidation tests that were conducted to evaluate the protection afforded the molybdenum sheet by the W-2 coating were made in the tube furnace shown in figure 26. The heated chamber of this furnace consisted of two alumina tubes of $1\frac{1}{2}$-inch inside diameter mounted horizontally. These tubes were heated by 2 pairs of silicon carbide heating elements supported horizontally in the furnace at right angles to the alumina tubes. One pair of the heating elements was supported below the alumina tubes and the other pair above the alumina tubes. The electrical power supplied to the upper and lower pairs of heating elements was independently controlled. A uniformly heated zone approximately 6 inches long was available at the midlength of each alumina tube. Calibration tests were made to determine the magnitude of the temperature variations along the heated length and the magnitude of the temperature fluctuations with time in the heated zone. These calibration tests indicated that the temperature variations along the length and the temperature fluctuations with time did not exceed $\pm\frac{1}{2}$ percent of the test temperature in the range between 2,100° F and 2,800° F.

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The coated molybdenum strips were placed on a small zirconia block and inserted into the uniformly heated zone of the furnace. The specimens were maintained at the test temperature until oxidation failure occurred. Failure was assumed to have occurred when the first visible evidence of molybdenum trioxide was obtained.

The oxidation specimens were expected to survive many hours of exposure at high temperatures and continuous monitoring of the oxidation test by an operator was not practical. For this reason a detection device was used to indicate the presence of molybdenum trioxide. This detection equipment is shown beside the furnace in figure 26. The detection equipment was connected to the alumina tube containing the oxidation specimen by means of the flexible tube as shown in figure 26. The detector operated at a slightly reduced air pressure which caused a small flow of air through the heated alumina tube into the flexible tube and to the detector. When oxidation of the specimen resulted in the formation of molybdenum trioxide, the trioxide was drawn into the flexible tube and into the detection apparatus. The apparatus utilized a sensitive photoelectric cell to detect the presence of the molybdenum trioxide and a timer was energized when evidence of the trioxide was obtained. The sensitivity of the detection apparatus was adjustable over a wide range so that detection of the presence of varying amounts of molybdenum trioxide was possible.

APPENDIX D

EQUIPMENT AND PROCEDURES FOR COMPRESSIVE STRENGTH TESTS OF CORRUGATED-CORE SANDWICHES

Axial compressive loading tests were performed on the corrugated-core sandwiches in a 120,000-pound universal hydraulic testing machine. Tests through 2,100° F were made with the aid of the equipment shown in figure 27. In this figure are shown the upper and lower loading rams, one quartz-tube-lamp radiator assembly, and an extensometer for measurement of axial shortening of the test specimen. Two quartz-tube-lamp radiator assemblies were used to heat the specimen to test temperature; however, in this view one of the radiator assemblies has been removed to expose the molybdenum sandwich specimen mounted in the lower ram. The sandwich specimens were supported in a stainless-steel adjustable-wedge slot in the lower loading ram and fitted into a similar slot in the upper loading ram. Both upper and lower rams were heated by electric heating elements within the rams. Ceramic fiber insulation was wrapped around the loading rams to minimize heat losses.

The sandwich specimens were heated to test temperature by the indicated radiator assembly and were exposed to the test temperature for 0.1 hour prior to loading. Electrical power supplied to the radiators was automatically controlled by a temperature controller utilizing a thermocouple supported in air within the test specimen. With this temperature control system it was possible to maintain the specimen temperature over a 1-inch gage length at the specimen midheight within ±2 percent of the desired temperature. Temperature variations with time in the gage length were less than ±1/2 percent of the test temperature.

Axial shortening of the specimens was measured during the tests with the aid of the extensometer assembly shown in figure 28. In this figure the extensometer assembly and the lower loading rams have been removed from the hydraulic testing machine. Two pairs of tungsten rods are shown extending from the sandwich specimen to form a portion of the lever assembly used to transfer specimen motion to the two linear variable differential transformer gages. The ends of the tungsten rods that contacted the test specimen were tapered to conical points. The conical points were seated in small holes of approximately 0.02-inch diameter that were drilled in the corrugated core of the sandwich. The holes were drilled 1 inch on centers to obtain a 1-inch gage length at the midheight of the specimen. All the extensometer assembly with the exception of approximately 1/2 inch of the tapered ends of the tungsten rods was shielded from the direct radiation of the quartz-tube-radiator assembly by means of asbestos-cement wall boards.

Compressive strength tests on the molybdenum sandwiches were made at $2,400^{\circ}$ F, $2,700^{\circ}$ F and $3,000^{\circ}$ F with the aid of an induction heating apparatus utilizing a graphite susceptor. An overall view of this apparatus is shown in figure 29. The induction heating unit consisted of a 20-kilowatt electronic tube generator with a frequency of approximately 450 kilocycles per second. The output of this generator was supplied to a 16-to-2 air-core radio-frequency output transformer to step down the voltage on the work coil and thus minimize arcing. The radio-frequency power was transmitted from the transformer through copper bus bars to the copper work coil. The induction heating coil shown in figure 30 was water cooled and consisted of approximately 11 turns of 1/4-inch-diameter copper tubing. The temperature of the specimen was maintained by manual adjustment of the power controls of the induction heating apparatus. Specimen temperatures were recorded during the tests by using the oscillograph temperature recorder shown in figure 29.

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The loading rams and the induction heating coil are shown in greater detail in figure 30. The copper induction coil was protected by glass-fiber sleeving and was insulated from the graphite susceptor by ceramic fiber insulation. The graphite susceptor was supported on the lower loading ram. Both the upper and lower loading rams were capped with 1-inch-thick boron nitride plates. Boron nitride was selected because of its low thermal conductivity which retarded heat transfer from the specimen into the loading ram, and because of good electrical insulating properties at elevated temperatures. Furthermore, the boron nitride was easily machined into the desired shape and also possessed adequate compressive strength for these elevated temperature tests.

Details of the graphite susceptor and associated equipment are shown in figure 31. The graphite susceptor consisted of a cylindrical tube 5 inches long with an outside diameter of $3\frac{1}{4}$ inches and wall thickness of 3/8 inch. This susceptor was heated to high temperatures with the induction heating apparatus and in turn heated the specimen by radiation. The graphite susceptor was used because of greater simplicity of the induction heating coil associated with a cylindrical specimen as well as the anticipation of more uniform heating of the sandwich specimen by radiation from the heated graphite.

The sandwich specimen was supported in a slot in the graphite loading ram shown in figure 31. The base of this slot was lined with a thin molybdenum sheet which distributed the compressive load from the specimen into the ram. The rams were flame sprayed with alumina to minimize oxidation of the graphite. Platinum-platinum, rhodium thermocouples were installed through holes in the rams and were supported with alumina thermocouple tubing. Three thermocouples were installed in each graphite ram so that measurement of temperatures at six representative stations within the specimen could be made. No attempt was made to determine shortening of the specimen during application of the compressive load in the tests utilizing the induction heating apparatus.

APPENDIX E

EQUIPMENT AND PROCEDURES FOR OXIDATION TESTS OF CORRUGATED-CORE SANDWICHES

Oxidation tests on the W-2 coated molybdenum sandwiches were performed in a heat-treating furnace. This furnace was heated by silicon
carbide heating elements and temperature was maintained automatically L
by an on-off temperature controller. The dimensions of the heated cham- 1
ber were 12 inches by 12 inches by 24 inches. On the basis of calibra- 0
tion tests, the maximum variations in temperature in one location in the 0
heated zone of the furnace were found to be not greater than ±2 percent 4
of the desired test temperature.

In the oxidation tests the furnace was heated to test temperature before insertion of the test specimen. The specimen was supported on an assembly of alumina rods protruding from a ceramic base. This assembly was used to support the specimen with the face sheets parallel to the furnace floor and permitted circulation of the hot air on all sides of the specimen. Visual examination of the specimens was made every 15 minutes to determine their physical condition. The specimens were not removed from the furnace for this periodic examination.

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TABLE I

ROOM-TEMPERATURE TENSILE PROPERTIES OF BARE AND COATED

Mo-0.5Ti SHEET IN STRESS-RELIEVED CONDITION[a]

Specimen	0.2-percent-offset tensile yield stress, ksi	Ultimate tensile stress, ksi	Young's modulus, ksi	Elongation in 2-inch gage length, percent
Bare sheet				
1	101.8	121.0	42.6×10^3	8
2	104.0	124.0	43.7	10
3	100.6	123.2	42.7	10
Average	102.1	122.7	43.0	9
W-2 coated sheet				
4	84.8	98.8	42.5×10^3	2
5	91.0	96.3	44.4	8
6	84.8	100.7	40.7	10
7	83.0	96.9	41.8	9
8	83.5	96.8	41.4	10
9	85.3	103.2	41.7	10
10	84.8	102.1	43.3	10
11	90.5	108.0	42.2	8
Average	86.0	100.4	42.2	8

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[a]Thickness of bare sheet, 0.013 in.; thickness of W-2 coating, 0.001 in.; strain rate in tensile test, 0.005 per minute up to yield and 0.05 per minute thereafter; cross-sectional areas of coated specimen prior to application of coating were used to convert applied loads to stresses.

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TABLE II

COMPARISON OF THE ROOM-TEMPERATURE MECHANICAL PROPERTIES OF

Mo-0.5Ti STRESS-RELIEVED SHEET AND REPROCESSED SHEET

Condition of material	Number of specimens tested	0.2-percent offset tensile yield stress, ksi	Ultimate tensile stress, ksi	Young's modulus, ksi	Elongation in 2-inch gage length, percent
Bare sheet					
Stress-relieved	3	102.1	122.7	43.0×10^3	9
Reprocessed	1	121.1	136.2	42.2	3
W-2 coated sheet					
Stress-relieved	8	86.0	100.4	42.2×10^3	8
Reprocessed	2	87.4	101.5	42.6	4

TABLE III

TENSILE PROPERTIES OF REPROCESSED Mo-0.5Ti SHEET

WITH W-2 COATING[a]

Specimen	Test temperature, °F	0.2-percent-offset tensile yield stress, ksi	Ultimate tensile stress, ksi	Young's modulus, ksi	Elongation in 2-inch gage length, percent
1	80	87.0	102.5	42.0×10^3	5
2	80	87.8	100.5	43.2	3
3	1,200	67.7	70.6	38.7	2
4	1,500	62.4	65.9	35.4	2
5	1,800	54.3	60.3	31.0	2
6	2,100	34.5	35.6	26.1	(b)
7	2,100	(b)	38.7	(b)	4
8	2,400	(b)	16.8	(b)	6
9	2,400	15.7	18.8	(b)	3
10	2,400	17.6	21.5	19.3	6
11	2,700	(b)	15.7	(b)	11
12	2,700	8.2	16.1	11.6	10
13	2,700	7.2	12.6	14.5	29
14	3,000	6.9	12.2	(b)	19
15	3,000	6.9	13.5	9.6	18

[a]Thickness of bare sheet, 0.010 in.; thickness of W-2 coating, 0.001 in.; strain rate, 0.005 per minute up to yield and 0.05 per minute thereafter; 0.1-hr exposure at test temperature prior to loading.

[b]Data not obtained.

L
1
0
0
4

TABLE IV

OXIDATION TEST RESULTS ON W-2 COATED Mo-0.5Ti SHEET[a]

Specimen	Test temperature, °F	Time to failure, hr
1	2,100	141.0
2	2,100	141.0
3	2,200	83.0
4	2,200	77.5
5	2,300	69.0
6	2,300	52.0
7	2,400	27.9
8	2,400	19.1
9	2,400	24.0
10	2,500	18.4
11	2,500	18.7
12	2,600	14.0
13	2,600	19.0
14	2,600	16.7
15	2,700	27.0
16	2,700	20.6
17	2,700	20.5
18	2,800	36.8
19	2,800	26.2

[a]Specimen size, 1 in. by $\frac{1}{2}$ in. by 0.010 in.; thickness of W-2 coating, 0.002 in.

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TABLE V

COMPRESSIVE STRENGTH TEST RESULTS FOR RESISTANCE-WELDED CORRUGATED-CORE Mo-0.5Ti SANDWICHES[a]

Specimen	Nominal test temperature, °F	Average stress at maximum load, ksi	Remarks
1	80	72.3	Bare; all other specimens coated
2	80	82.2	
3	80	89.2	
4	1,200	79.5	Heated to test temperatures with quartz-tube-lamp radiator assembly shown in figure 27 (specimens 4 to 7)
5	1,500	75.9	
6	1,800	67.6	
7	2,100	65.5	
8	2,400	23.7	Heated to test temperatures with induction heating equipment shown in figure 29 (specimens 8 to 10)
9	2,700	9.0	
10	3,000	7.9	

[a]Thickness of W-2 coating, 0.001 in.; 0.1-hr. exposure at test temperature prior to loading; all specimens fabricated from reprocessed sheet.

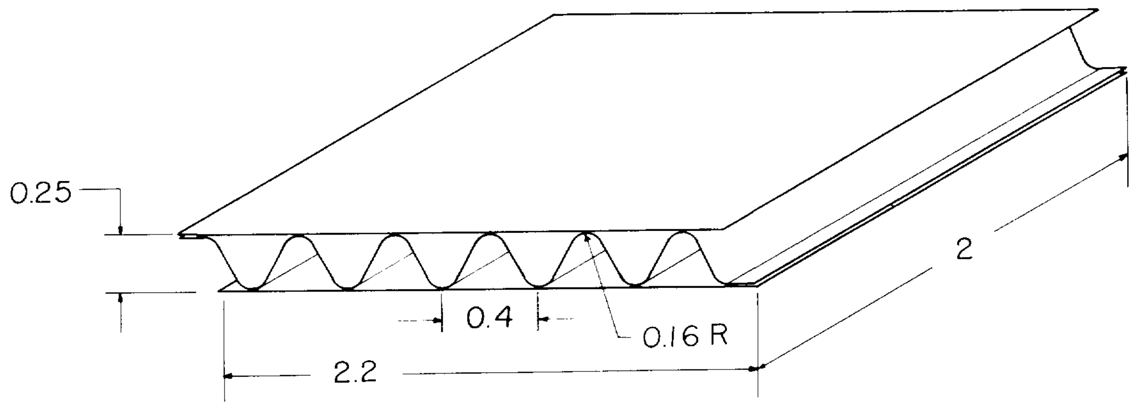


Figure 1.- Dimensions of resistance-welded corrugated-core Mo-0.5Ti sandwich specimens. All dimensions are in inches.

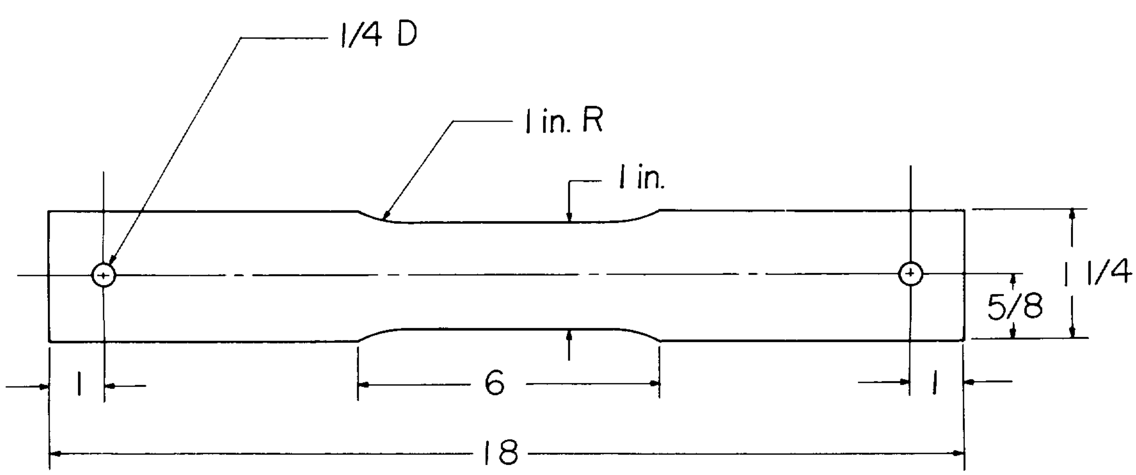


Figure 2.- Dimensions of 0.010-inch-thick Mo-0.5Ti tensile stress-strain specimens. All dimensions are in inches.

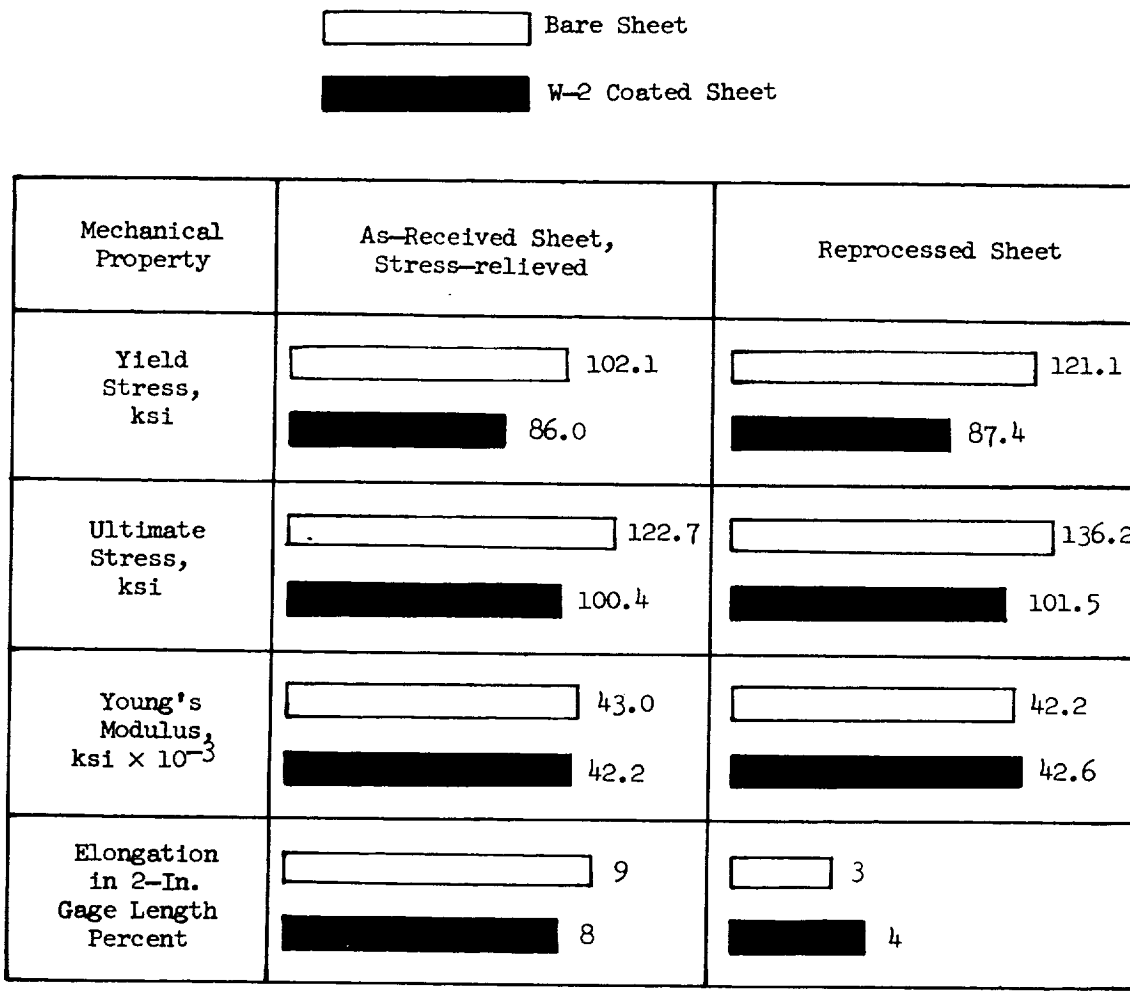


Figure 3.- Comparison of room-temperature tensile properties of Mo-0.5Ti stress-relieved sheet and reprocessed sheet.

L-1004

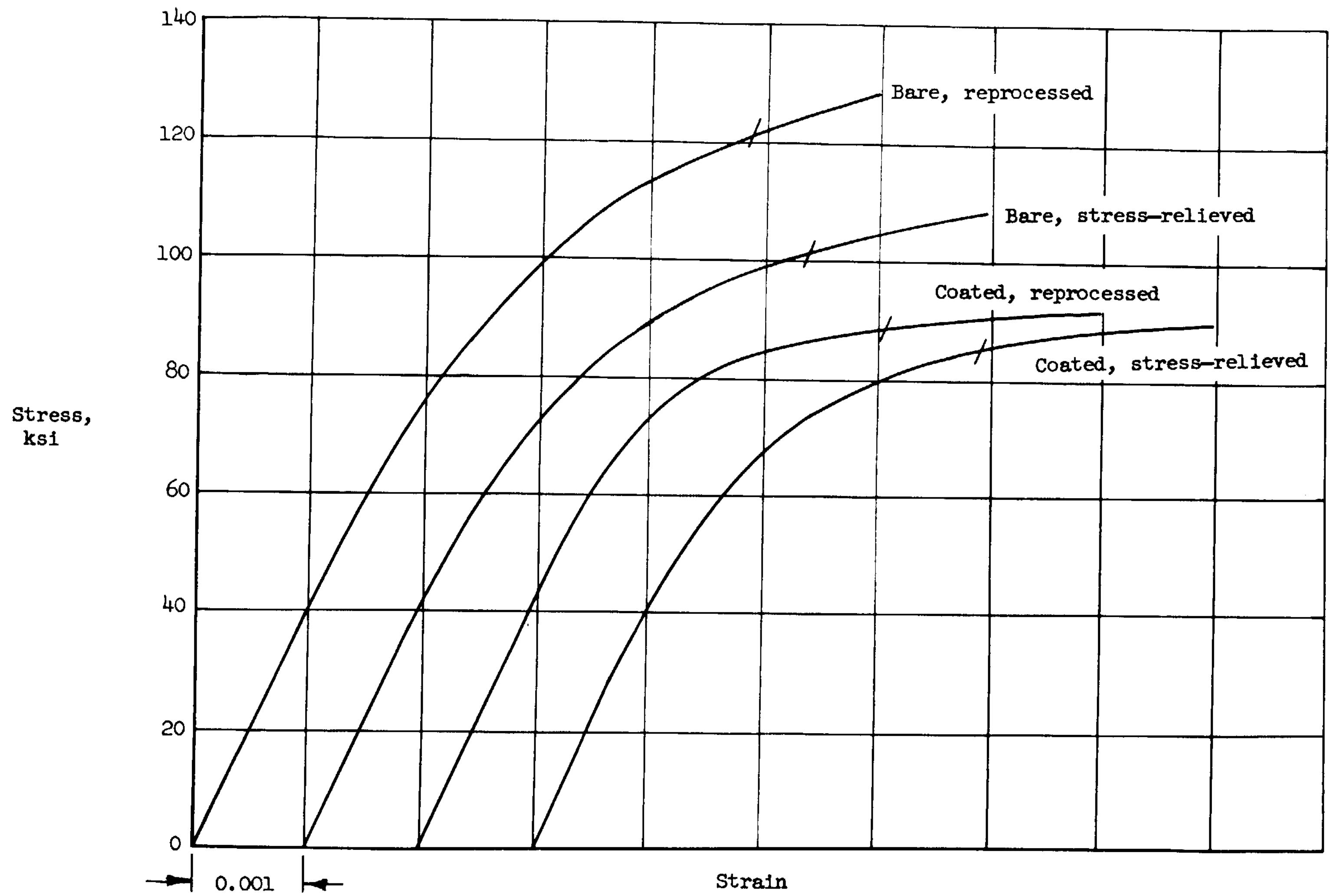
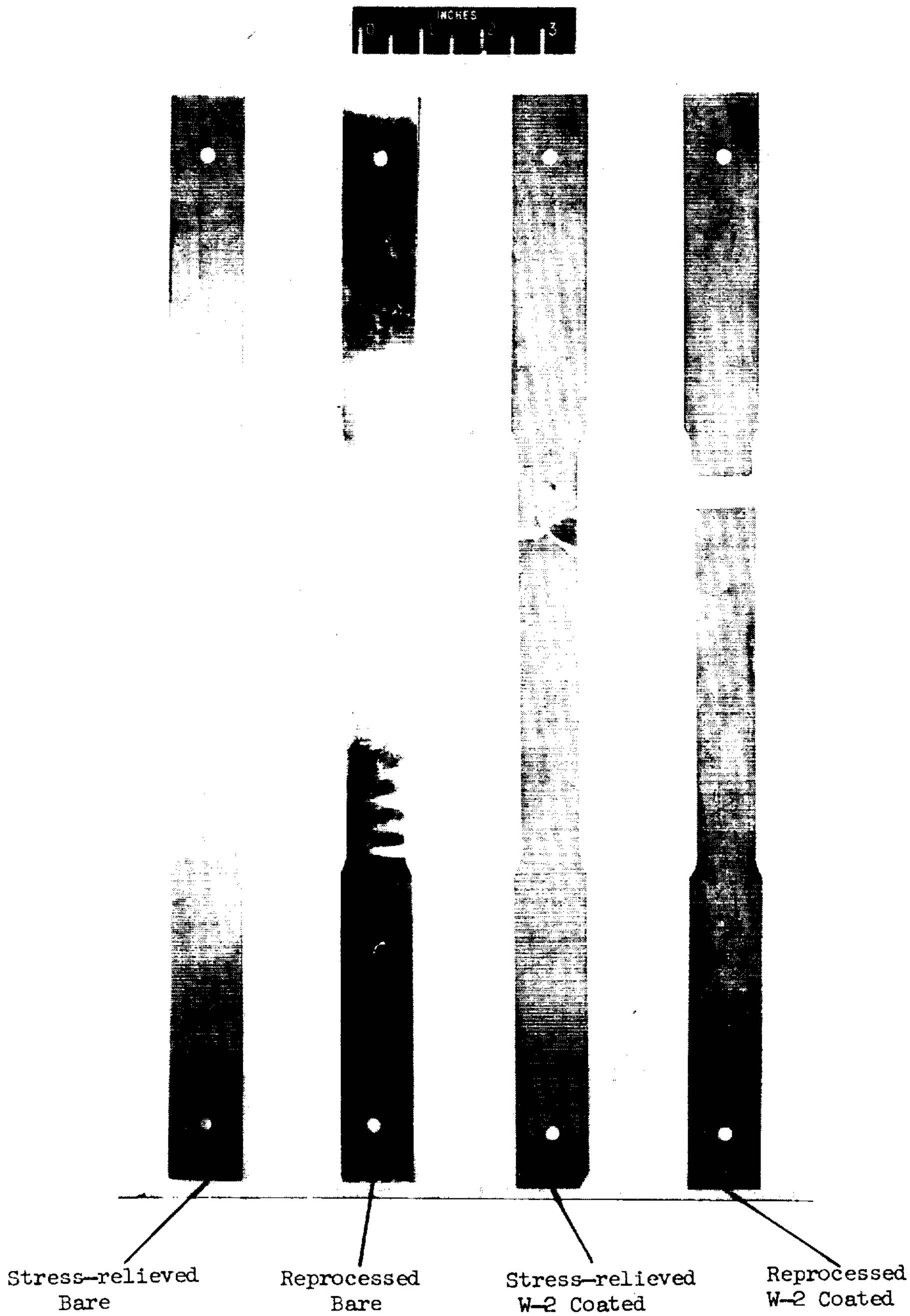


Figure 4.- Room-temperature tensile stress-strain curves for bare and coated Mo-0.5Ti sheet in the stress-relieved and in the reprocessed conditions. Nominal thickness of W-2 coating, 0.001 inch.



L-59-7933

Figure 5.- Examples of fractures obtained in room-temperature tensile stress-strain tests on bare and coated Mo-0.5Ti sheet. Thickness of stress-relieved sheet, 0.013 inch; thickness of reprocessed sheet, 0.010 inch.

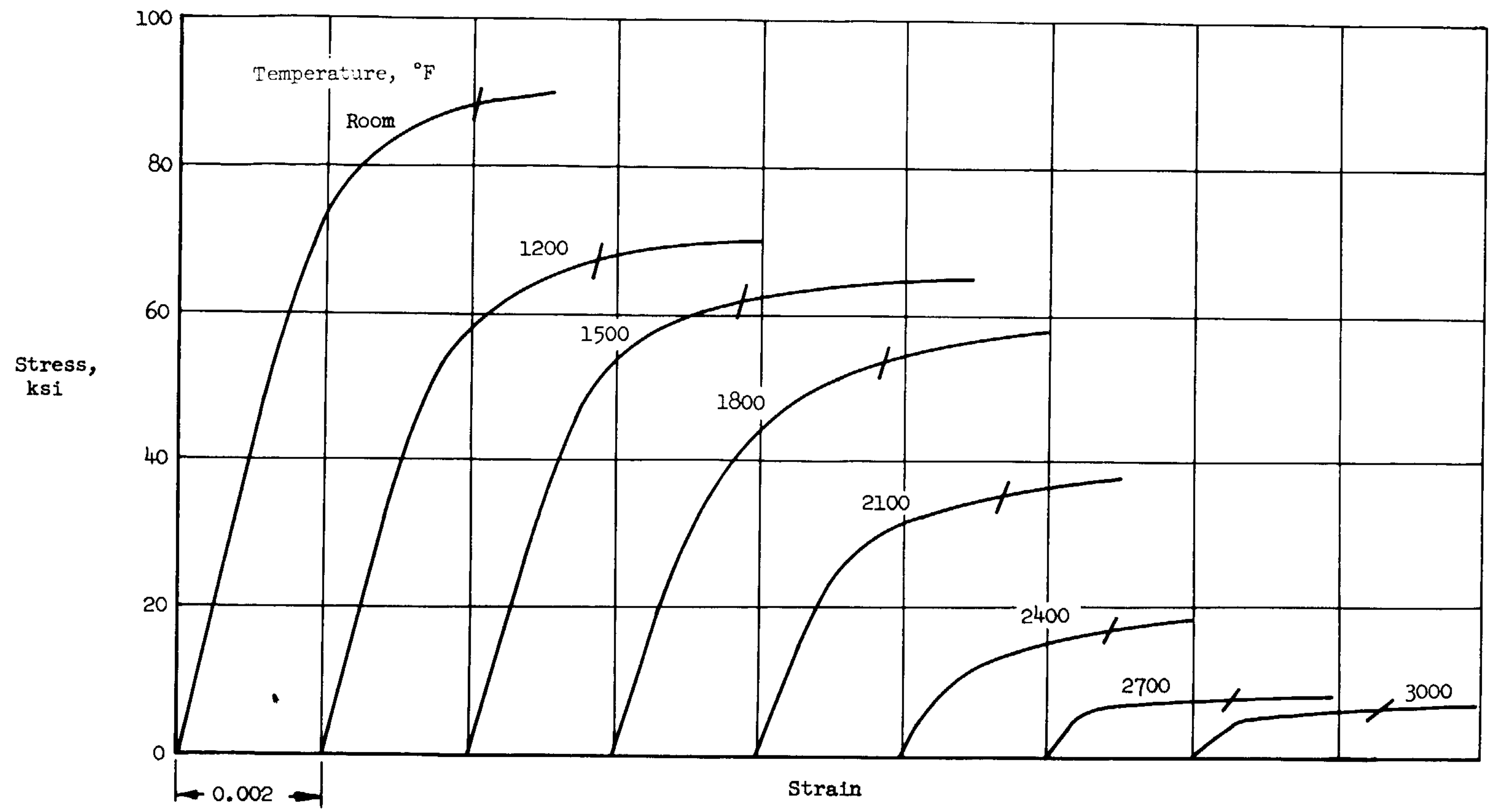


Figure 6.- Tensile stress-strain curves for W-2 coated Mo-0.5Ti sheet in the reprocessed condition. Thickness of bare sheet, 0.010 inch; thickness of W-2 coating, 0.001 inch; strain rate, 0.005 per minute up to yield and 0.05 per minute thereafter; 0.1-hour exposure at test temperature prior to loading.

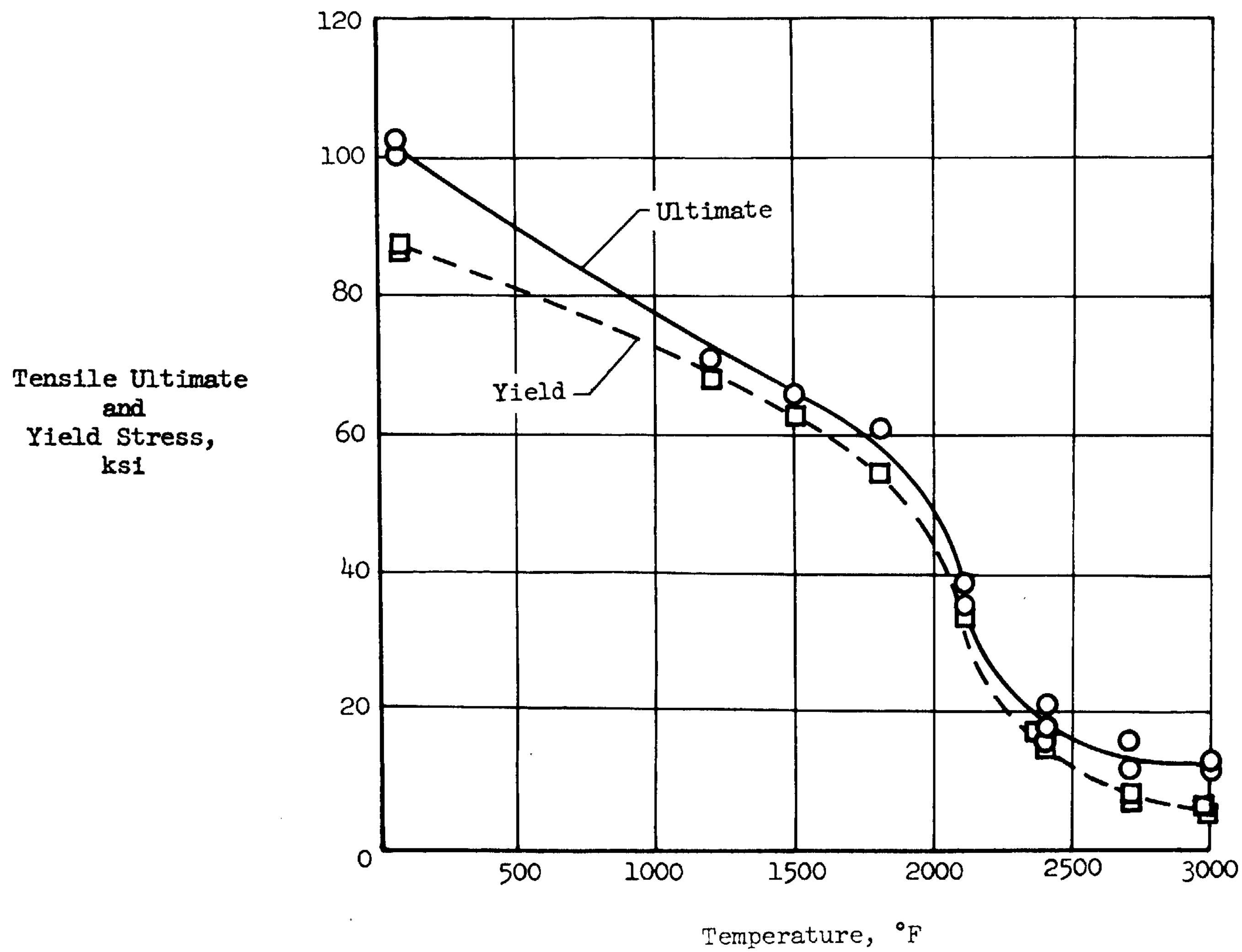


Figure 7.- Variation of ultimate tensile stress and 0.2-percent-offset tensile yield stress with temperature for W-2 coated Mo-0.5Ti reprocessed sheet.

L-1004

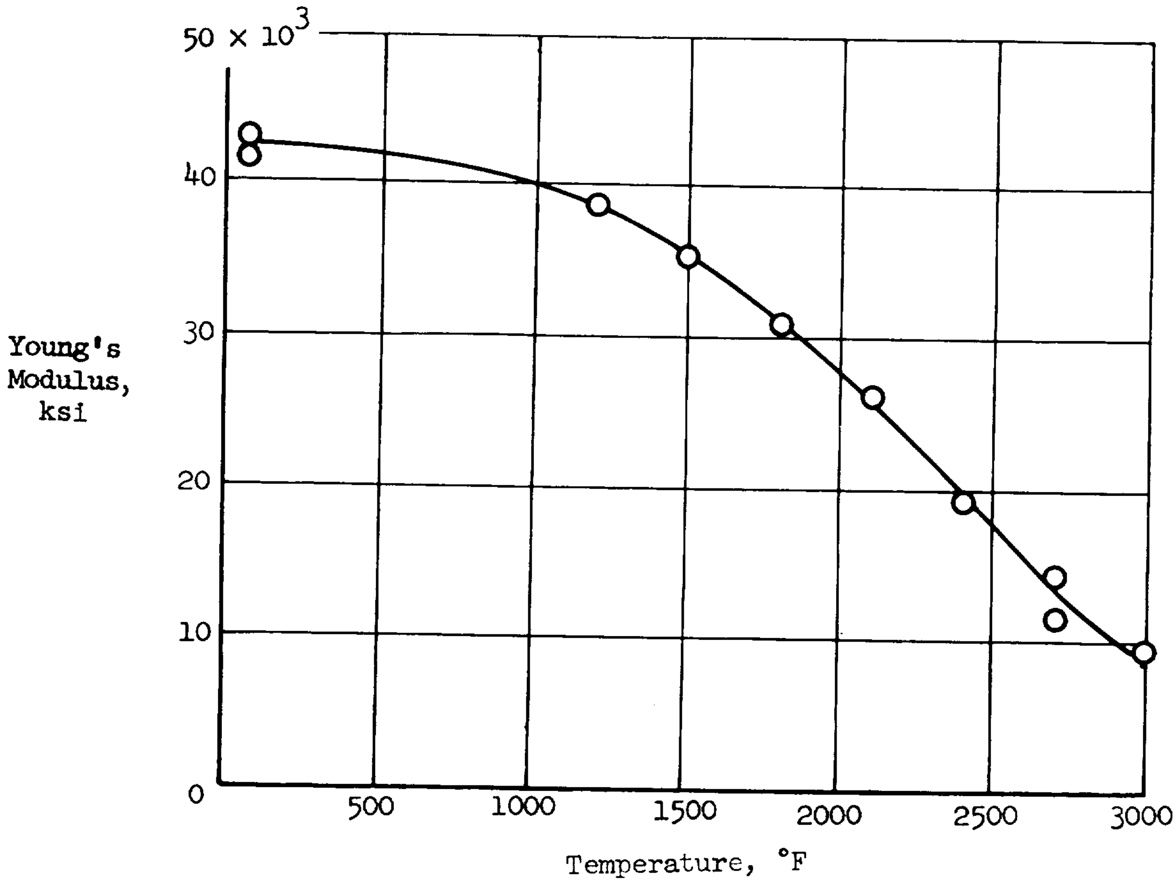


Figure 8.- Variation of Young's modulus with temperature for W-2 coated Mo-0.5Ti reprocessed sheet.

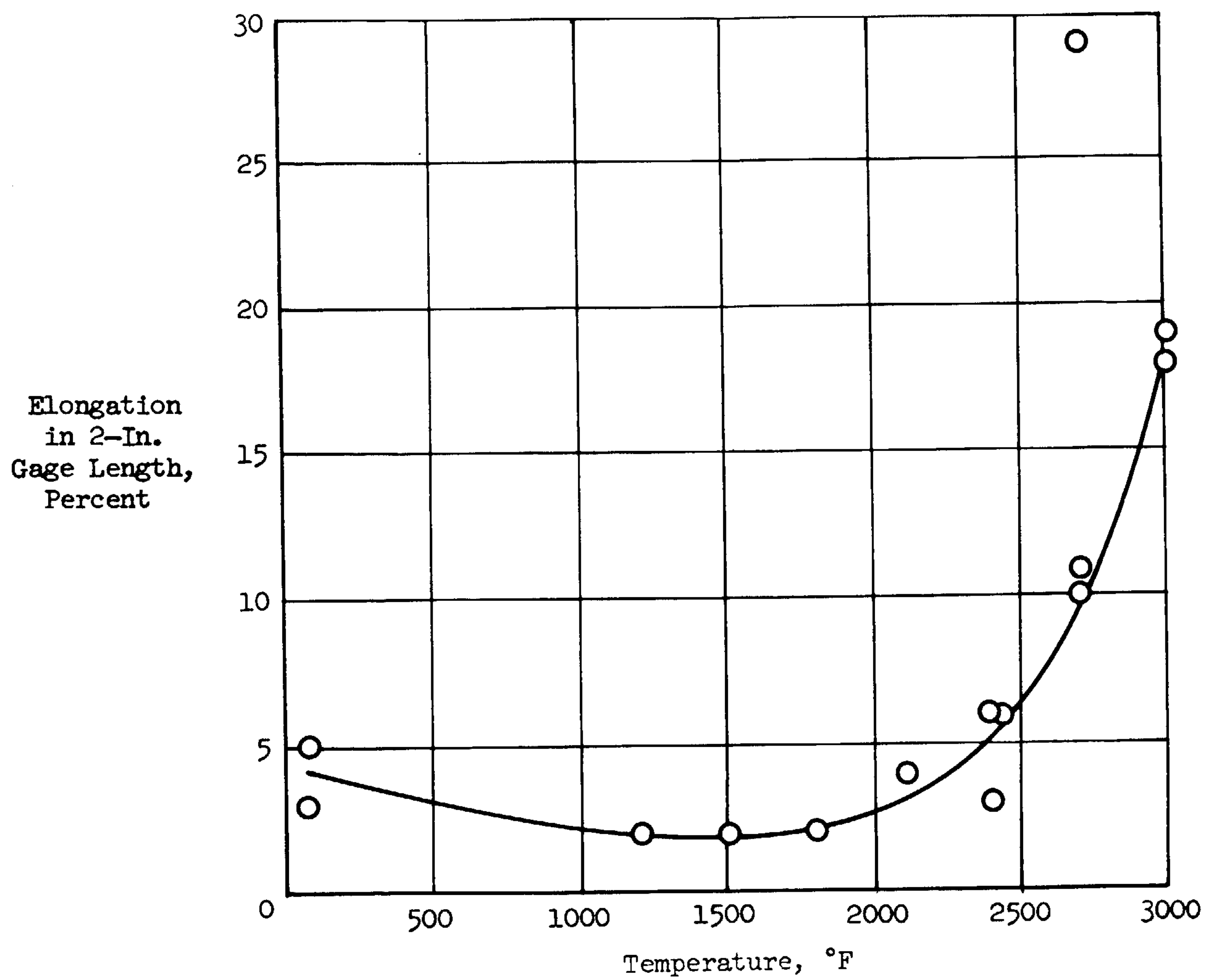


Figure 9.- Variation of percent elongation in 2-inch gage length with temperature for W-2 coated Mo-0.5Ti reprocessed sheet.

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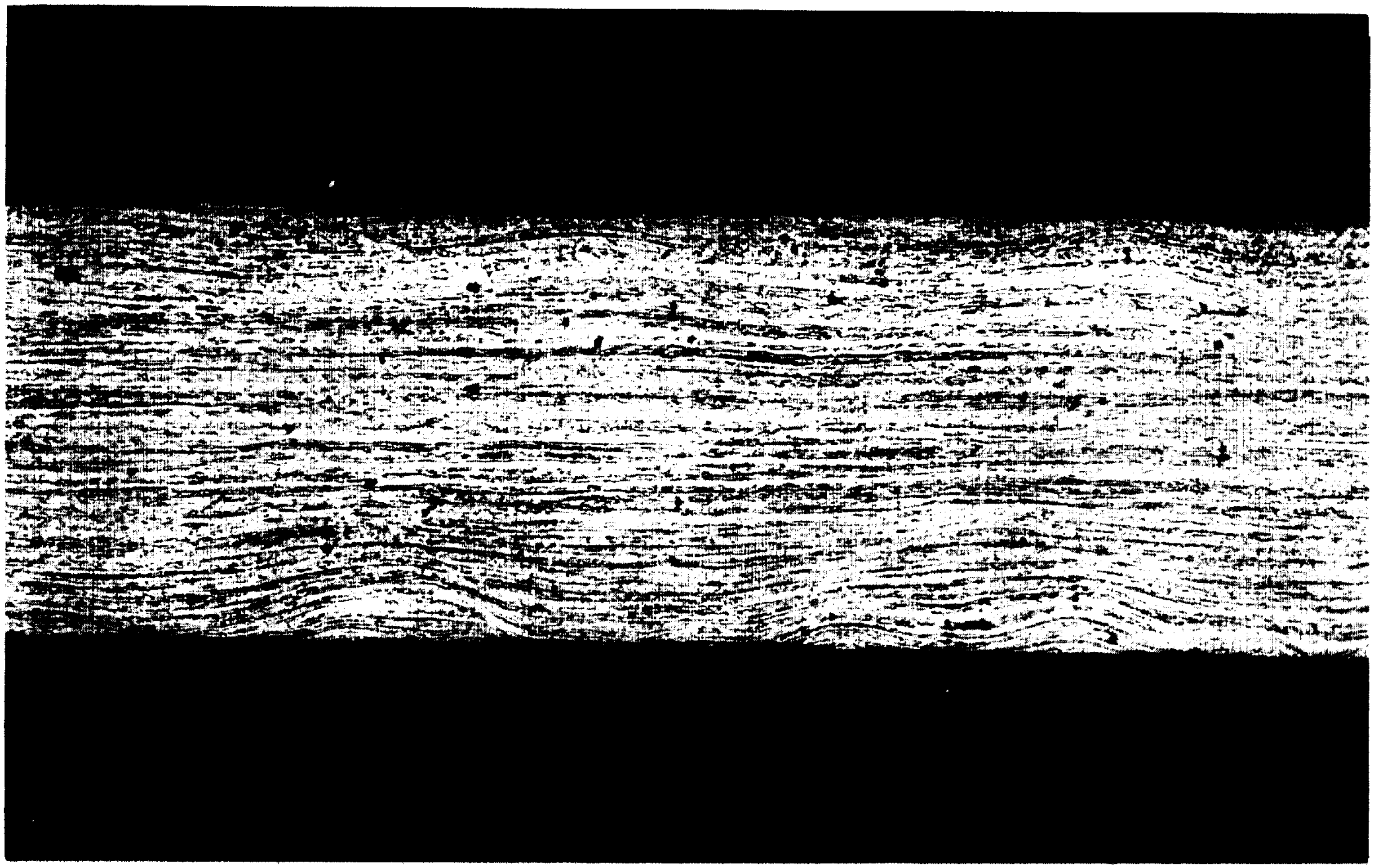
Room Temperature | 1200° F | 2100° F | 2700° F

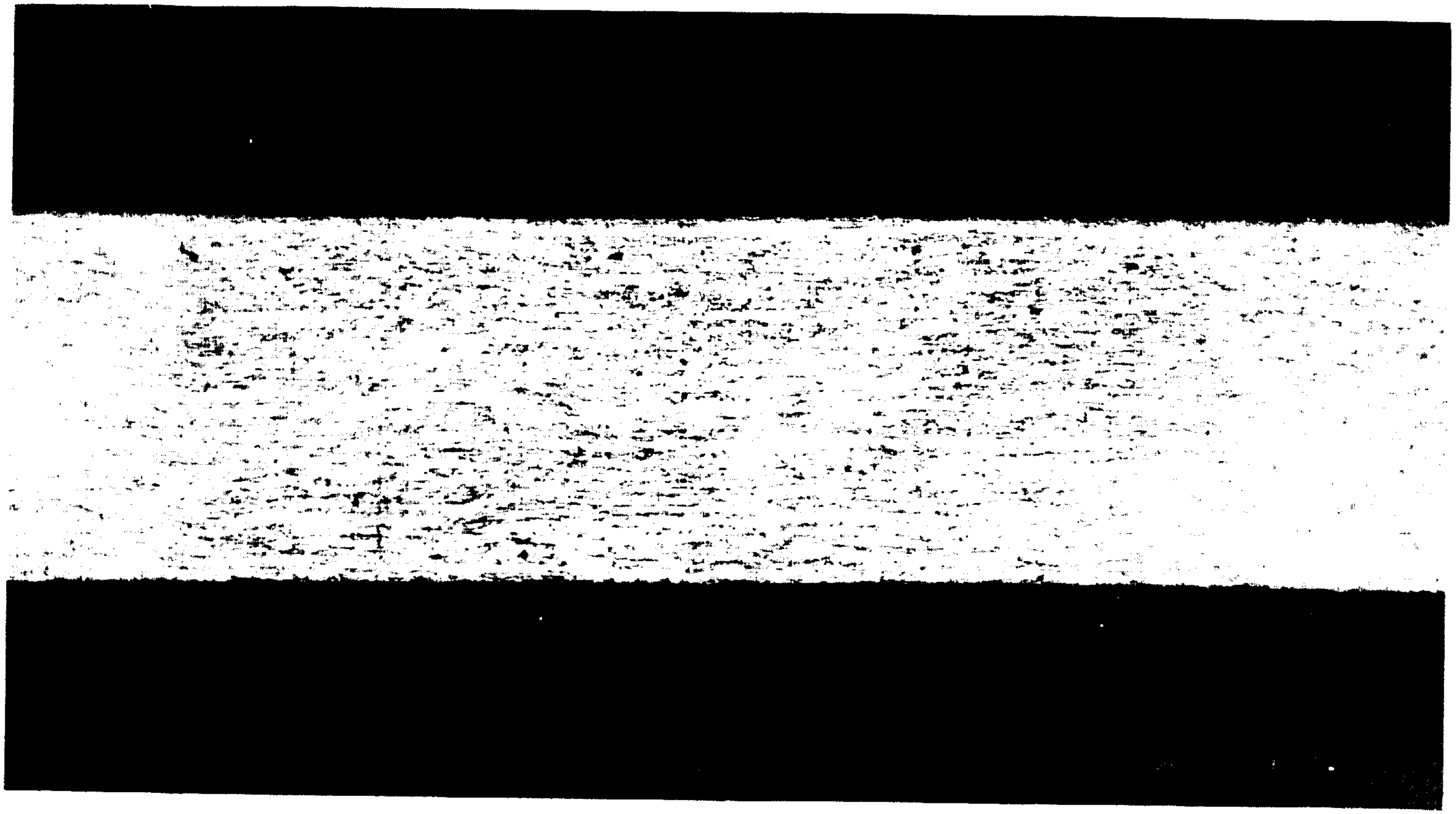
L-59-7935

Figure 10.- Examples of fractures obtained at several temperatures in tensile stress-strain tests of W-2 coated Mo-0.5Ti sheet in reprocessed condition. Sheet thickness, 0.010 inch; W-2 coating thickness, 0.001 inch.



L-60-2453

Figure 11.- Grain structure of 0.013-inch-thick stress-relieved Mo-0.5Ti sheet. x200.



L-60-2454

Figure 12.- Grain structure of 0.010-inch-thick reprocessed Mo-0.5Ti sheet. ×200.

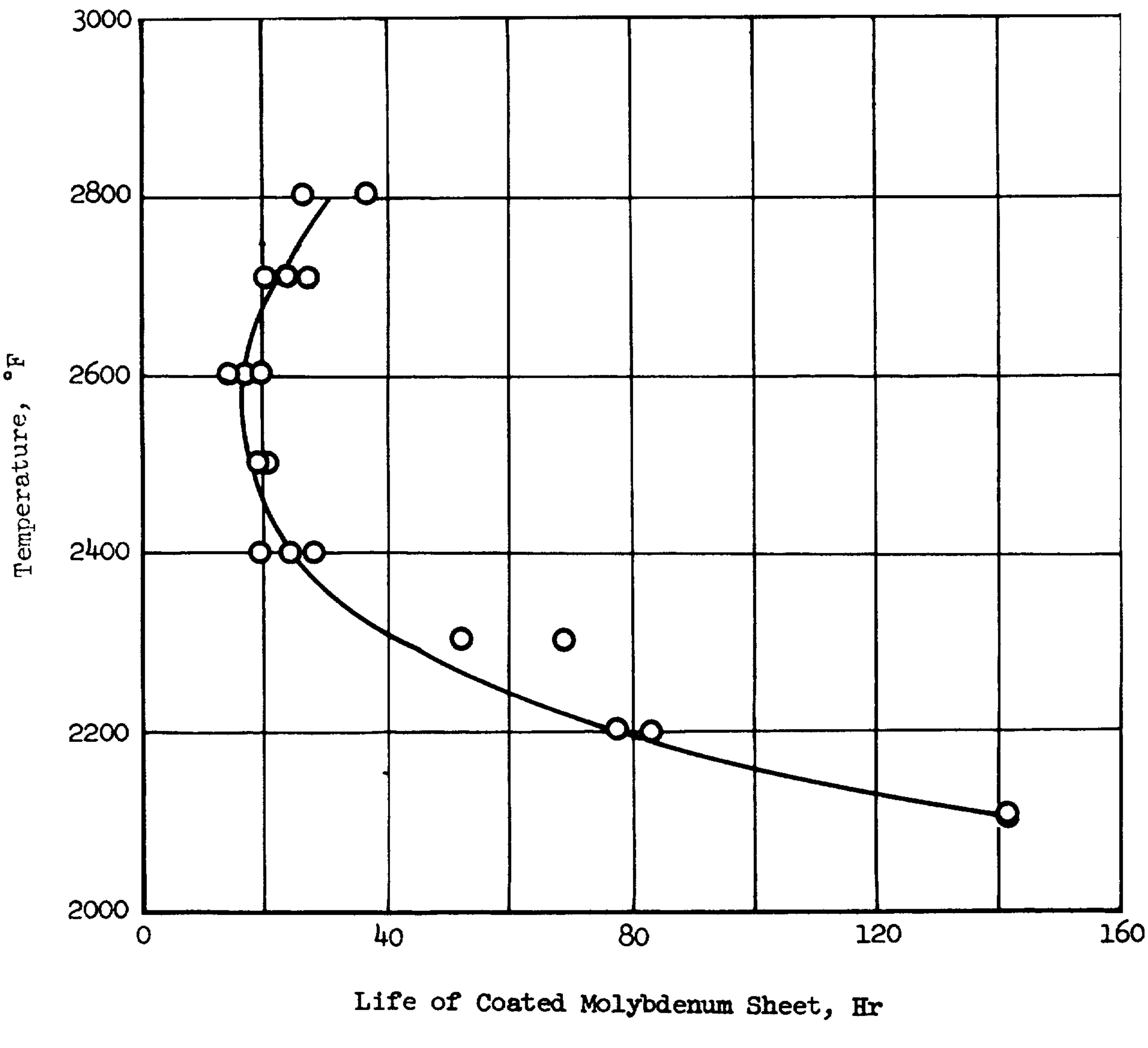


Figure 13.- Life of W-2 coated oxidation specimens tested in air in furnace. Thickness of W-2 coating, 0.002 inch.

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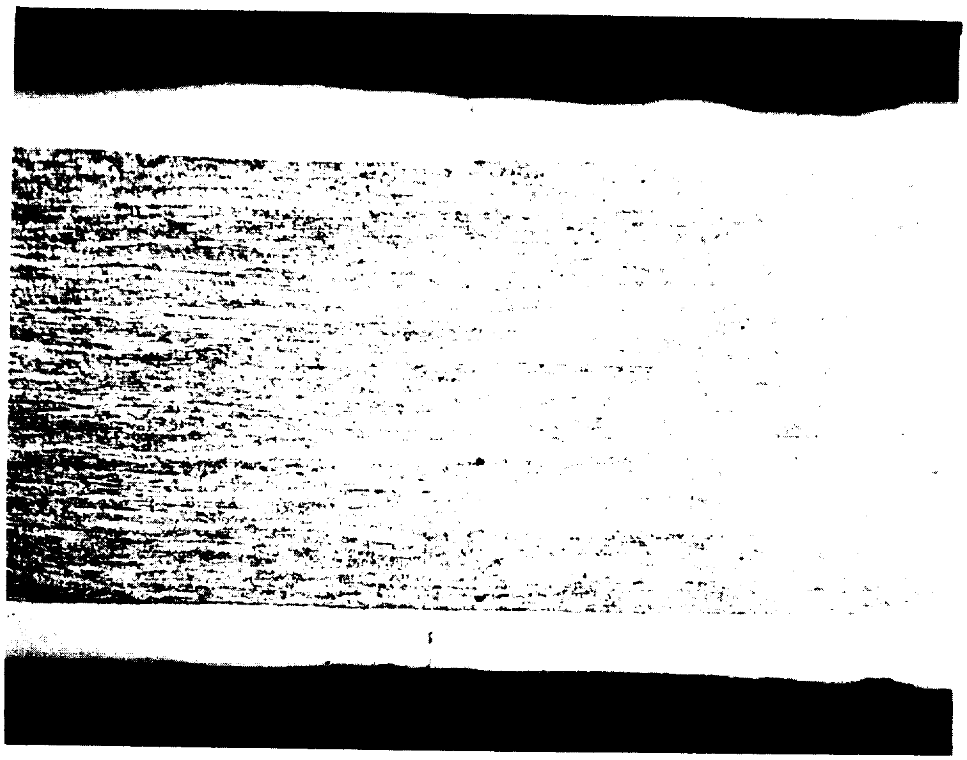
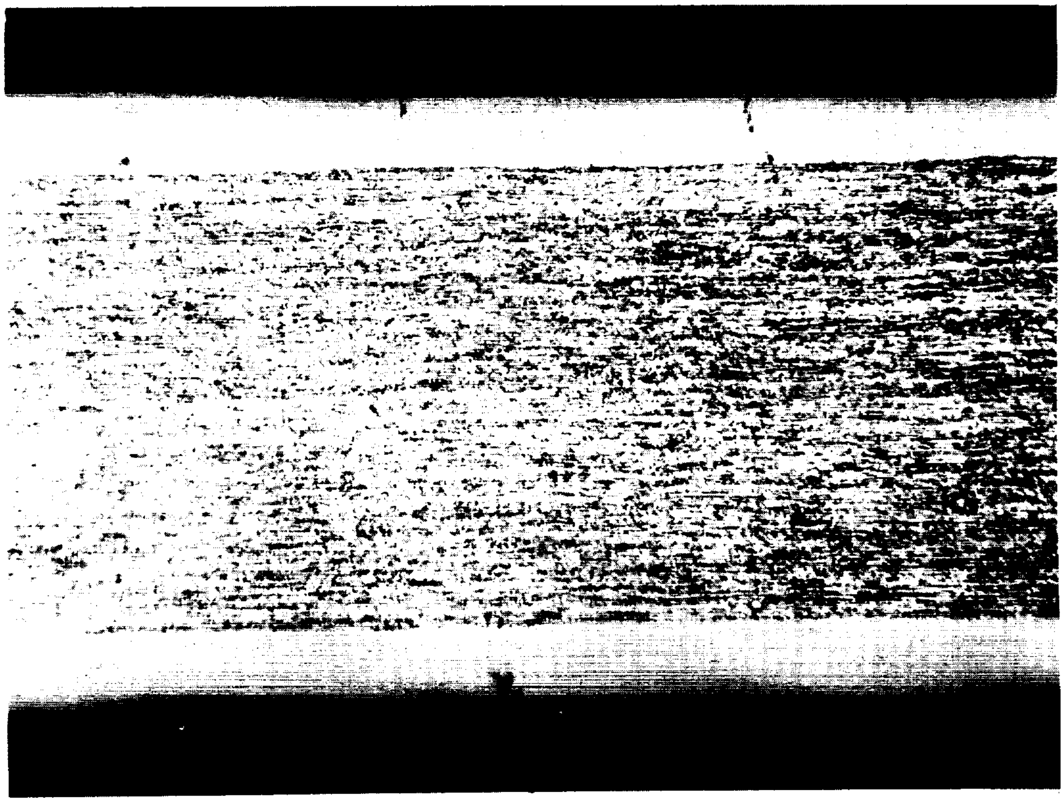


L-60-2455

Figure 14.- The W-2 coated Mo-0.5Ti sheet in reprocessed condition. Sheet thickness, 0.010 inch; coating thickness, 0.001 inch (this is representative of the coating used on tensile stress-strain specimens and corrugated-core sandwiches); ×250.



L-60-2456

Figure 15.- The W-2 coated Mo-0.5Ti sheet in reprocessed condition. Sheet thickness, 0.010 inch; coating thickness, 0.002 inch (this is representative of the coating used on oxidation specimens and was applied by modified coating process); ×250.

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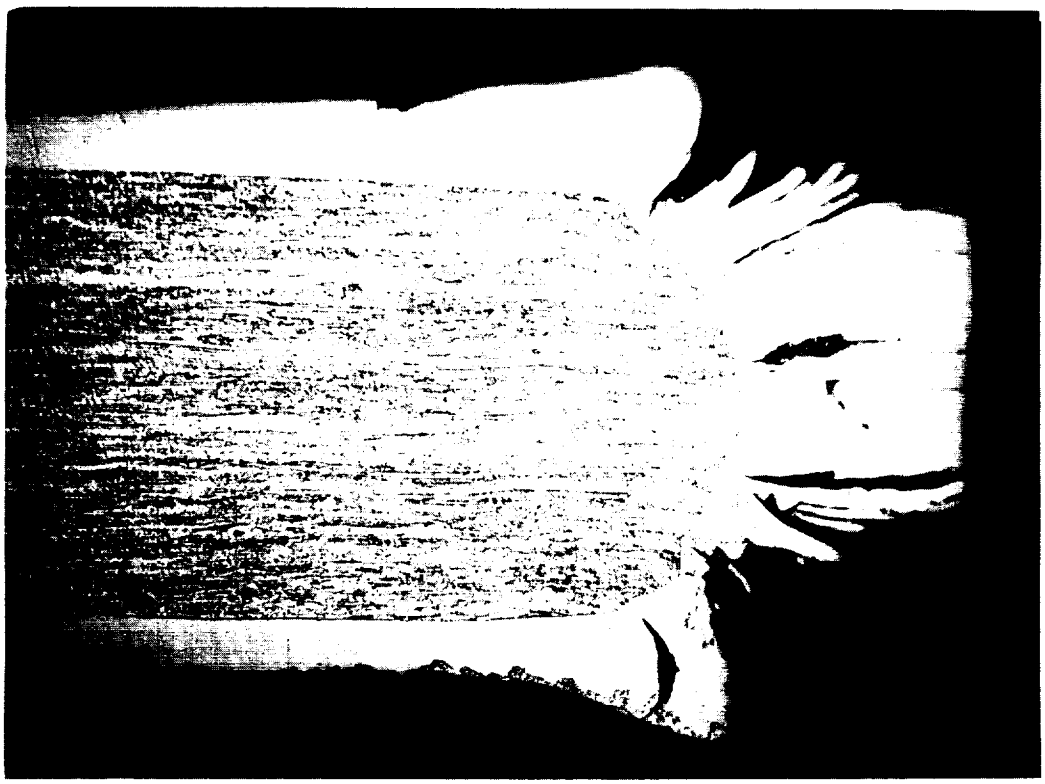
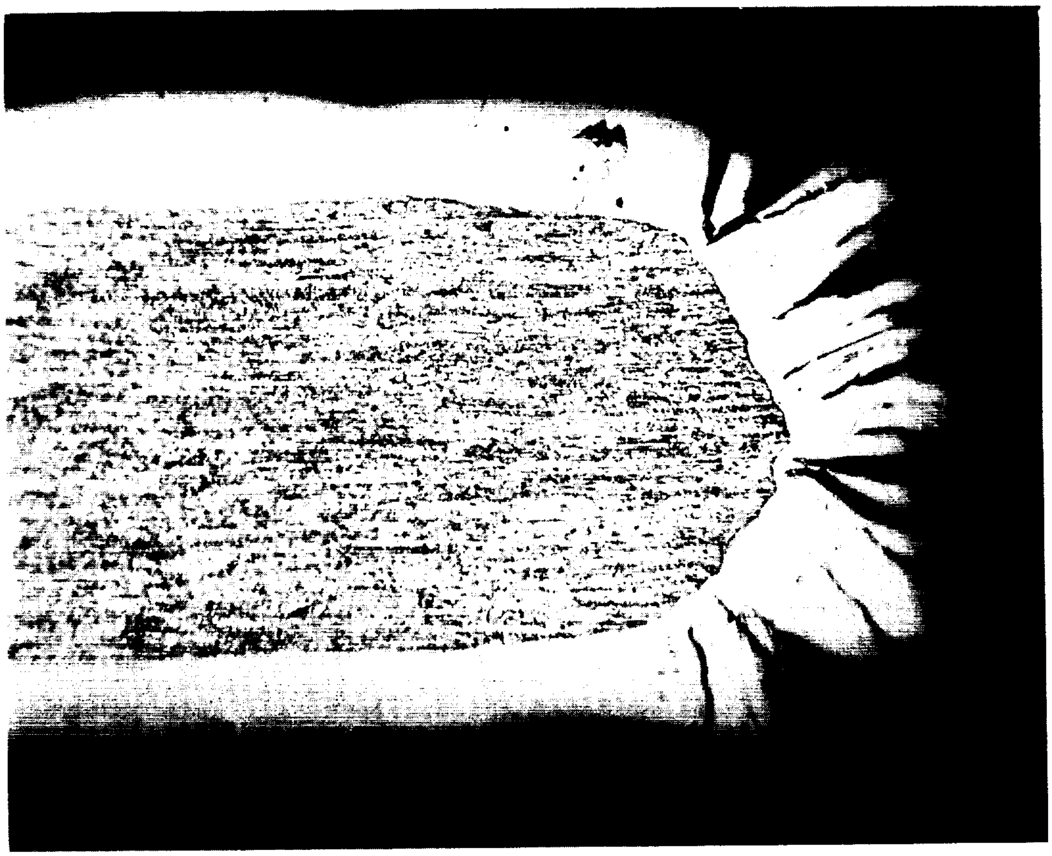


(a) Coating thickness, 0.001 inch; ×250. L-60-2457

Figure 16.- Edge view of W-2 coated oxidation specimen. Mo-0.5Ti sheet in reprocessed condition. Sheet thickness, 0.010 inch.

L-1004

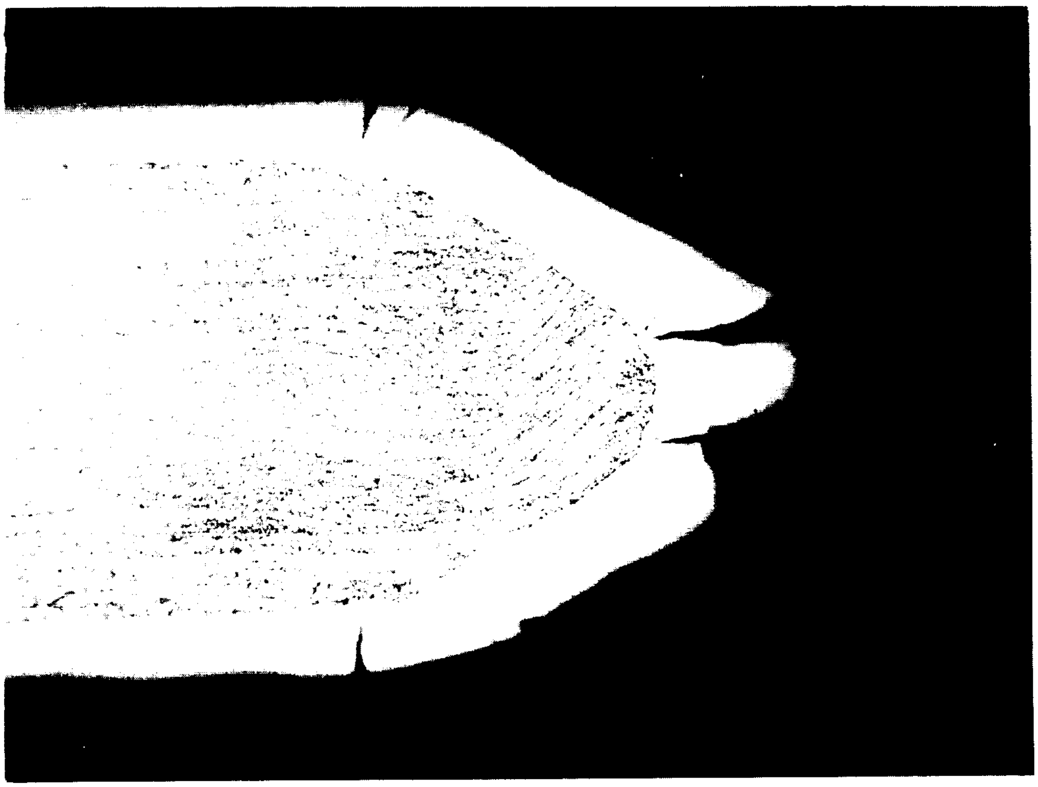


L-60-2458

(b) Coating thickness, 0.002 inch applied by modified coating process; ×250.

Figure 16.- Continued.

L-1004



L-60-2459

(c) Coating thickness, 0.002 inch applied by modified coating process; edge of sheet beveled to improve uniformity of coating; ×250.

Figure 16.- Concluded.

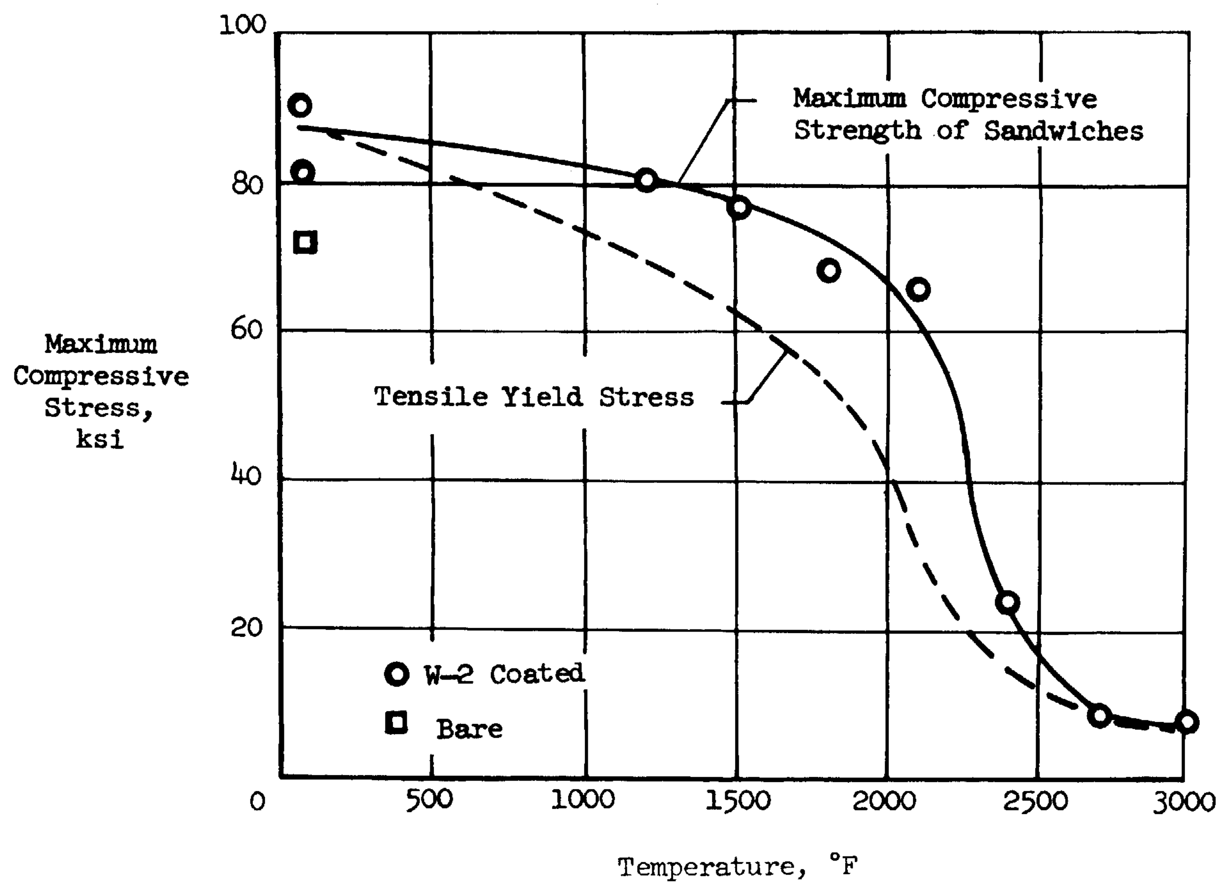


Figure 17.- Compressive strength of resistance-welded corrugated-core Mo-0.5Ti sandwiches. Thickness of W-2 coating, 0.001 inch; 0.1-hour exposure at test temperature prior to loading; all sandwich specimens were fabricated from reprocessed Mo-0.5Ti sheet.

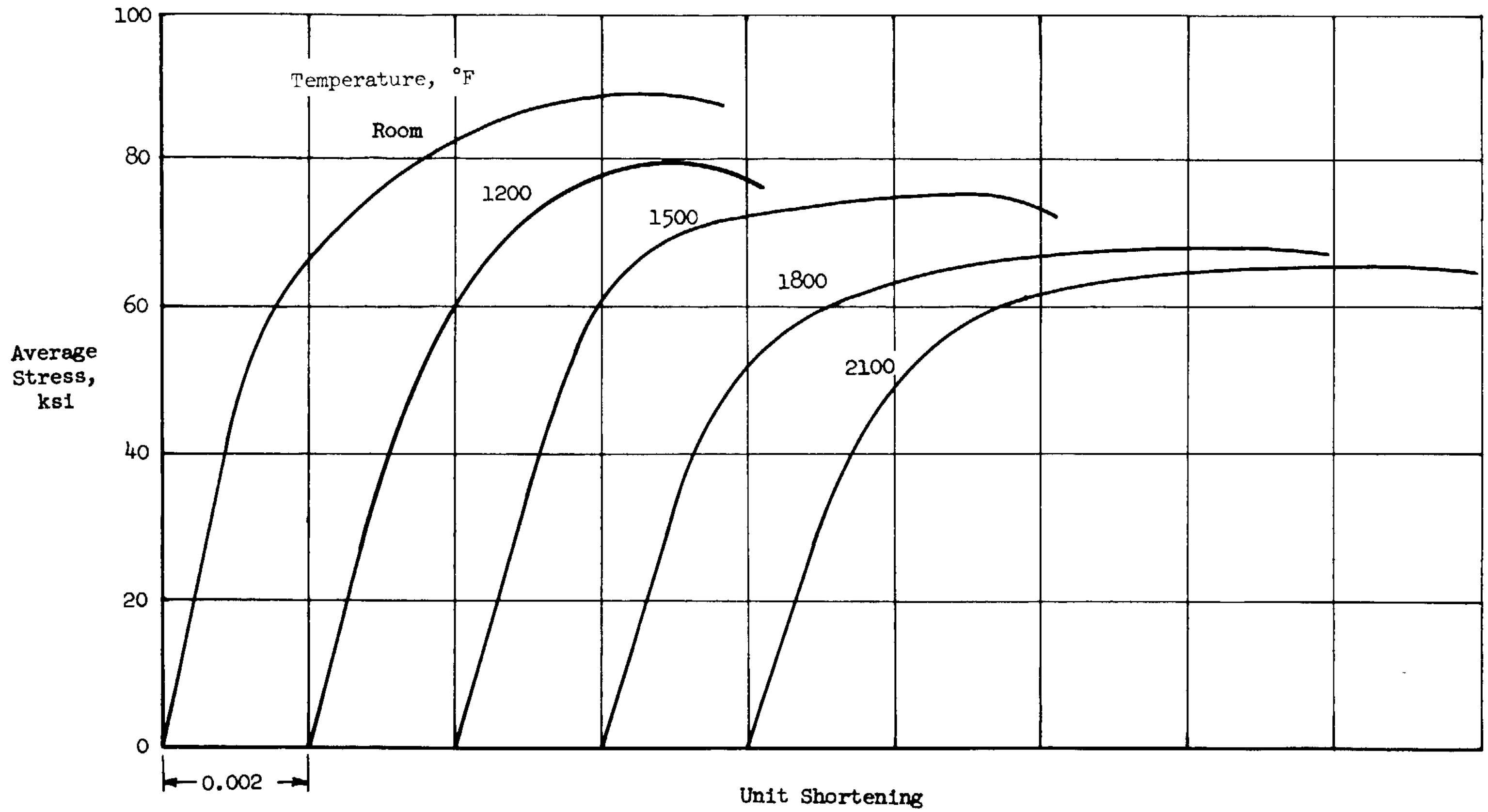


Figure 18.- Curves of average stress against unit shortening for resistance-welded corrugated-core Mo-0.5Ti sandwiches.

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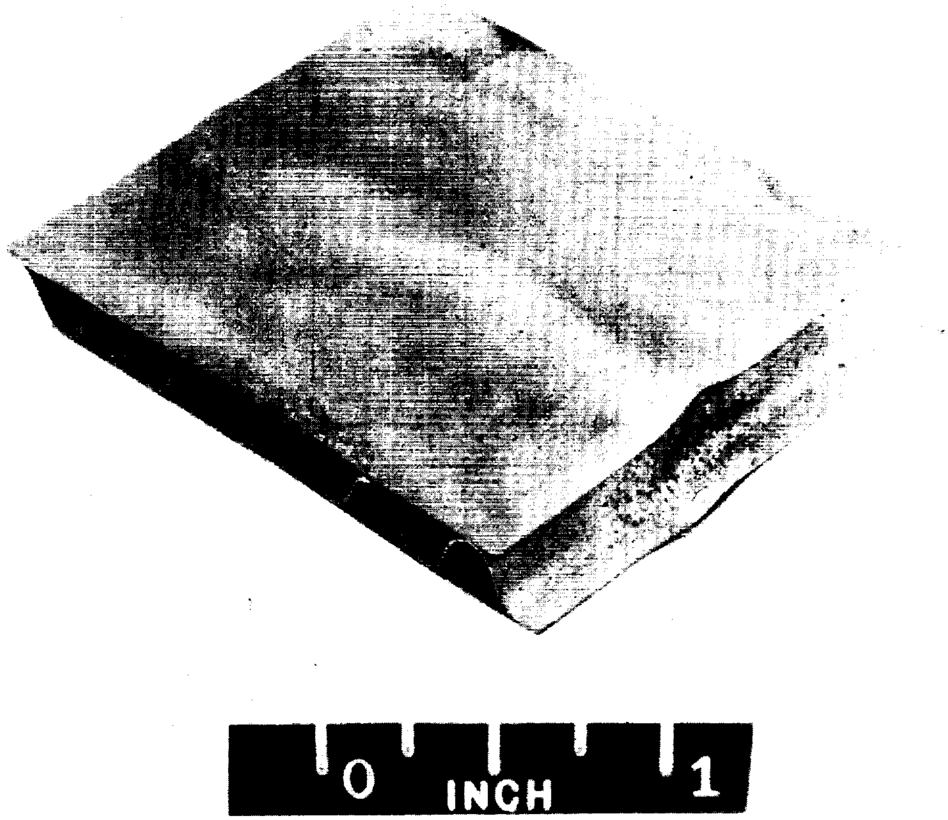
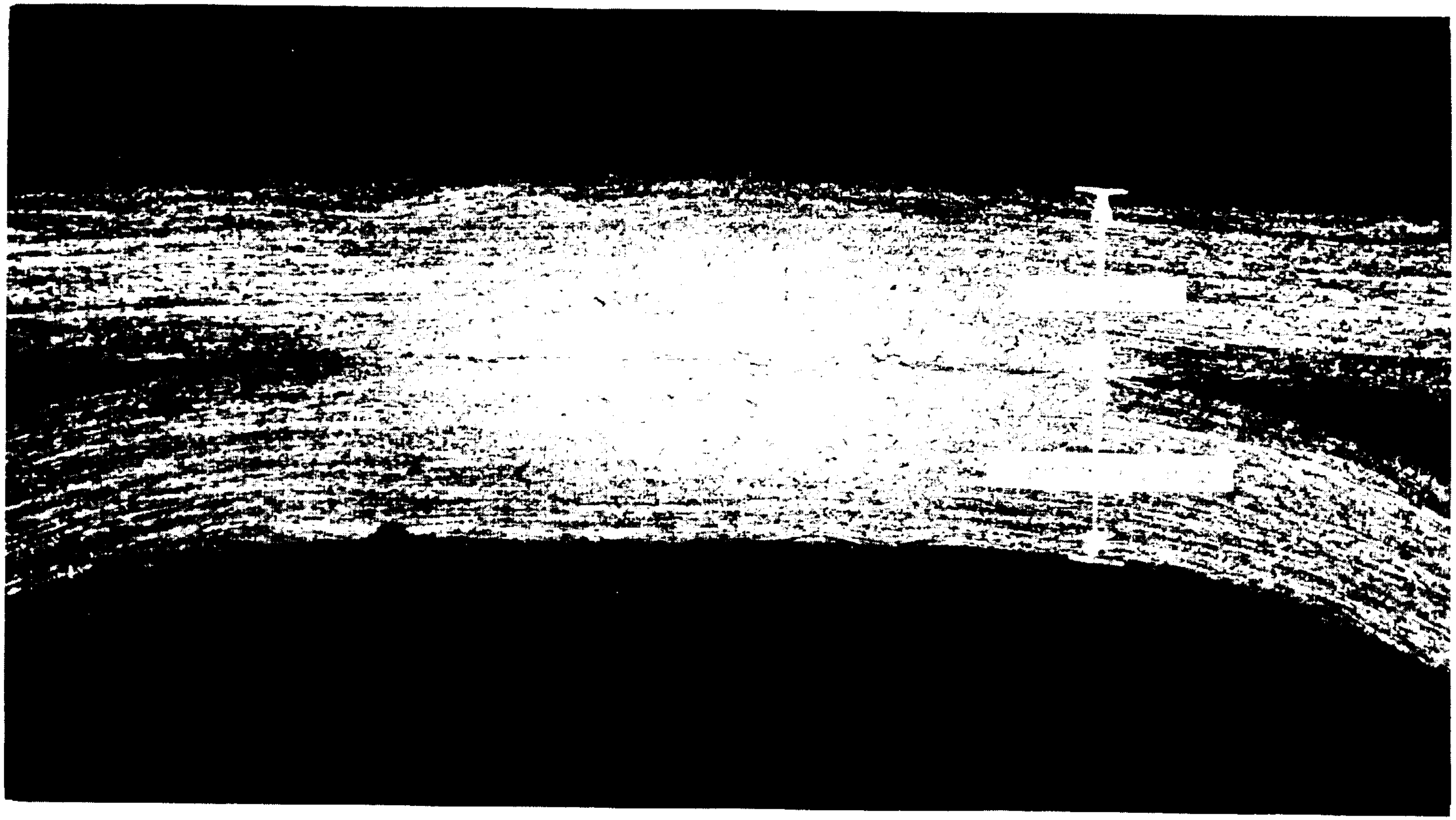


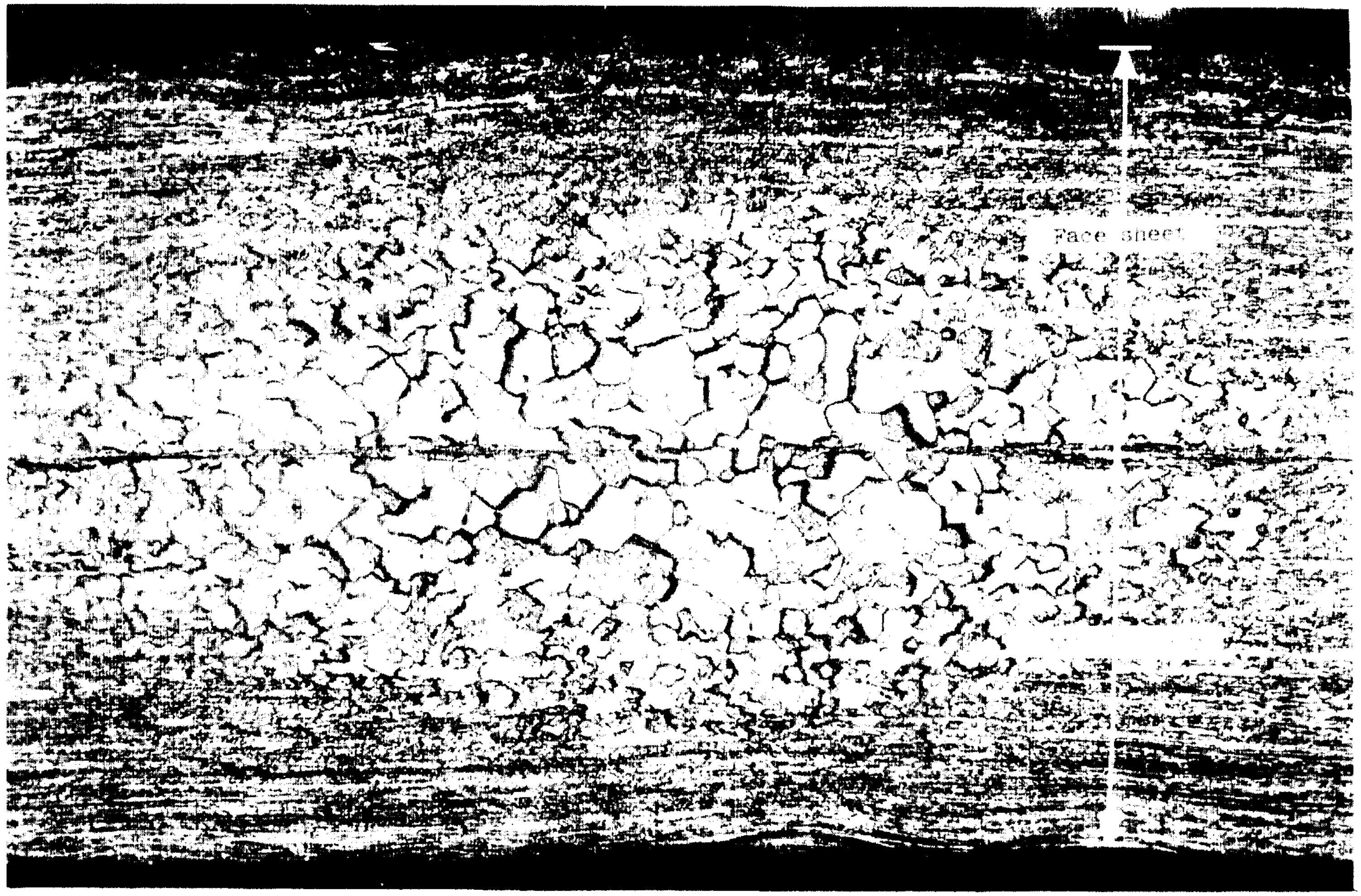
L-59-7009

Figure 19.- Molybdenum sandwich after axial compressive strength test. Test temperature, 2,400° F; compressive load was applied in the direction of the corrugations.



(a) ×100. L-60-2460

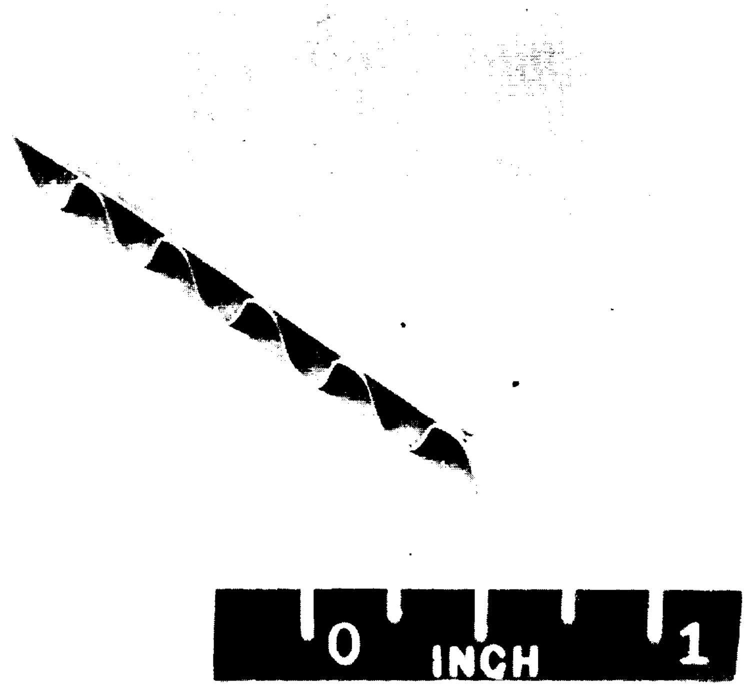
Figure 20.- Example of resistance weld in corrugated-core Mo-0.5Ti sandwich.



(b) x250. L-60-2461

Figure 20.- Concluded.

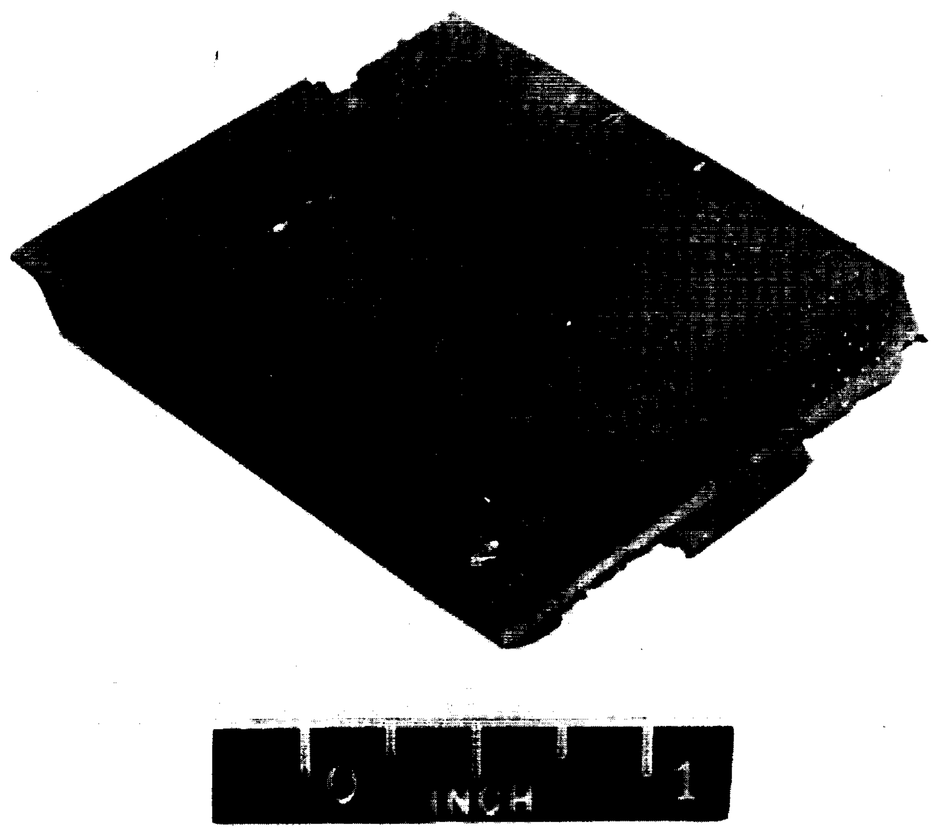
L-1004



(a) W-2 coated sandwich prior to oxidation test. L-59-7006

Figure 21.- Example of W-2 coated Mo-0.5Ti sandwich before and after oxidation test in furnace at 2,700° F. Time at test temperature, 3.2 hours.

L-1004

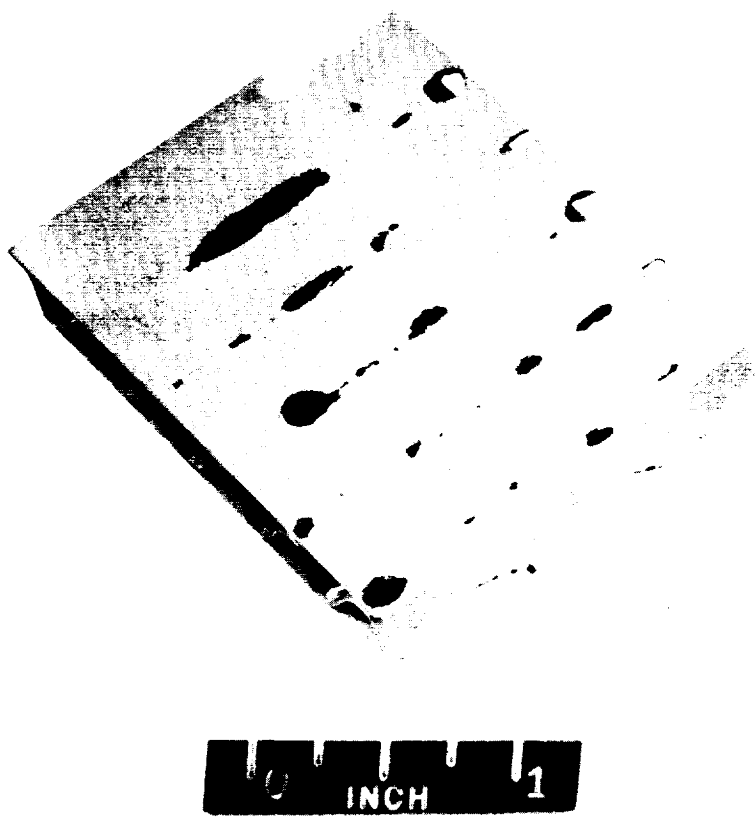


L-59-7004

(b) Appearance of sandwich specimen after removal from furnace at 2,700° F.

Figure 21.- Continued.

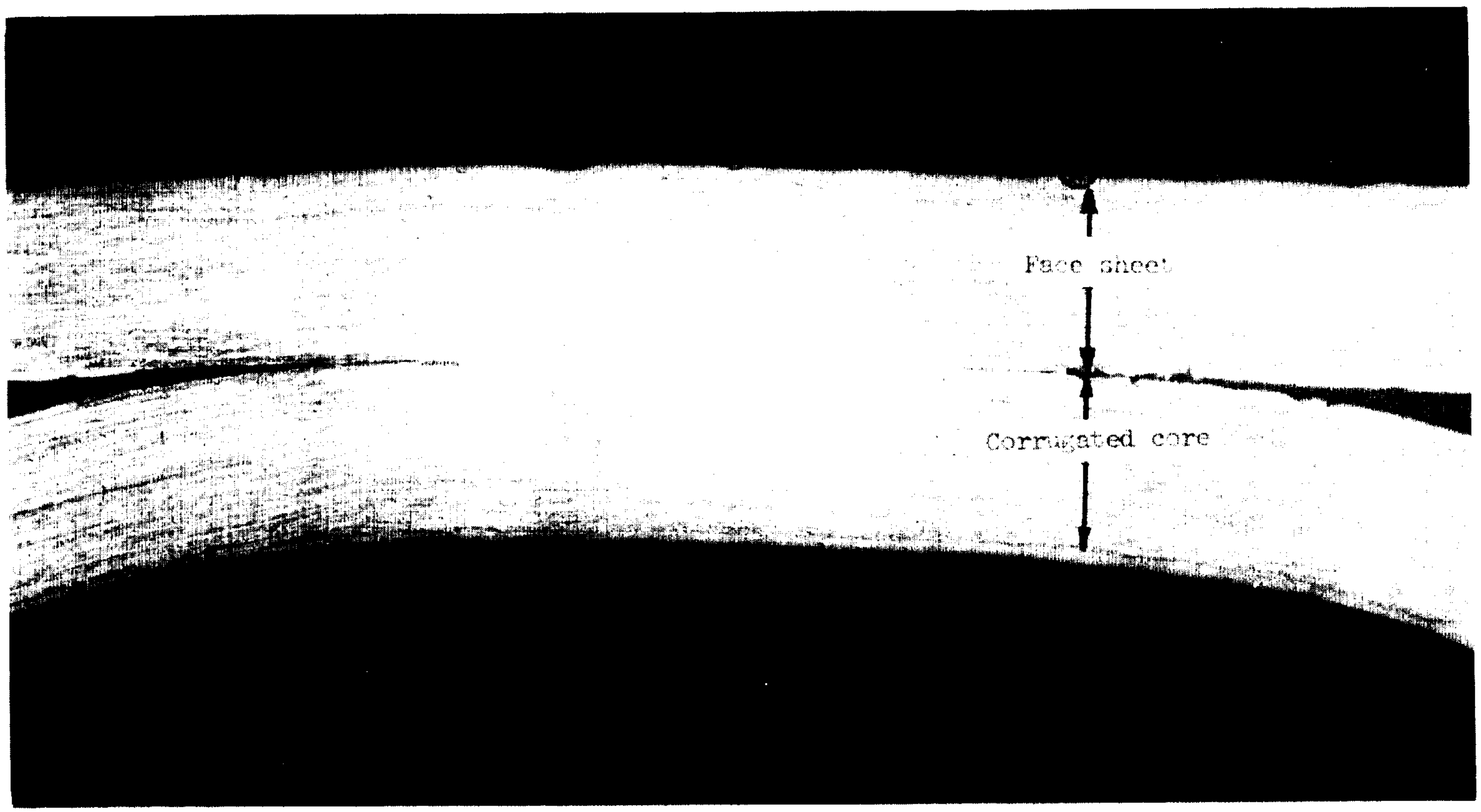
L-1004



L-59-8130

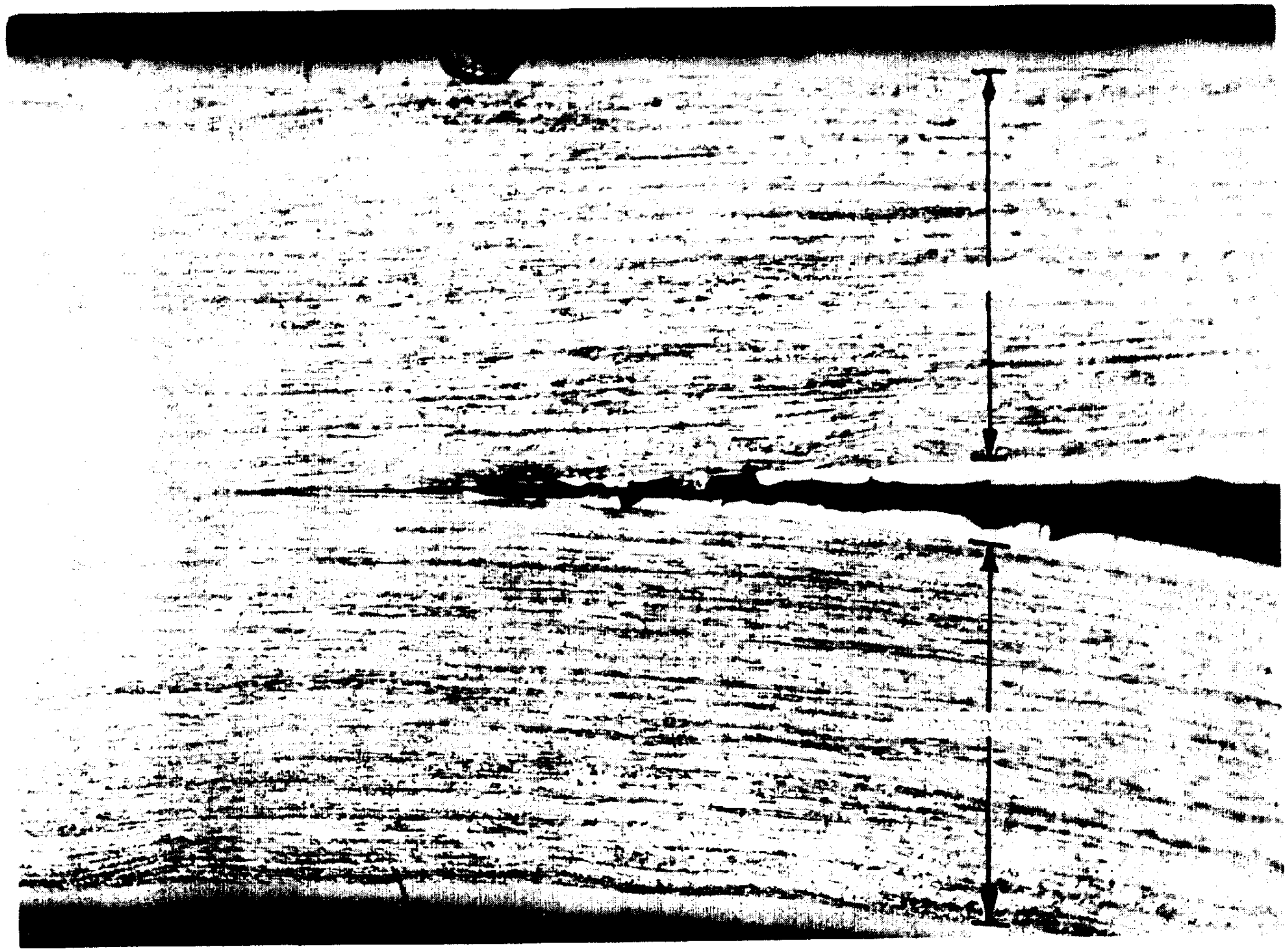
(c) Appearance of specimen after coating has been removed to expose oxidized areas in face sheet and core. The oxidized areas are located at the spot welds.

Figure 21.- Concluded.



(a) ×100. L-60-2462

Figure 22.- Example of resistance weld and coating in W-2 coated Mo-0.5Ti sandwich specimen.



L-60-2463

(b) ×300.

Figure 22.- Concluded.

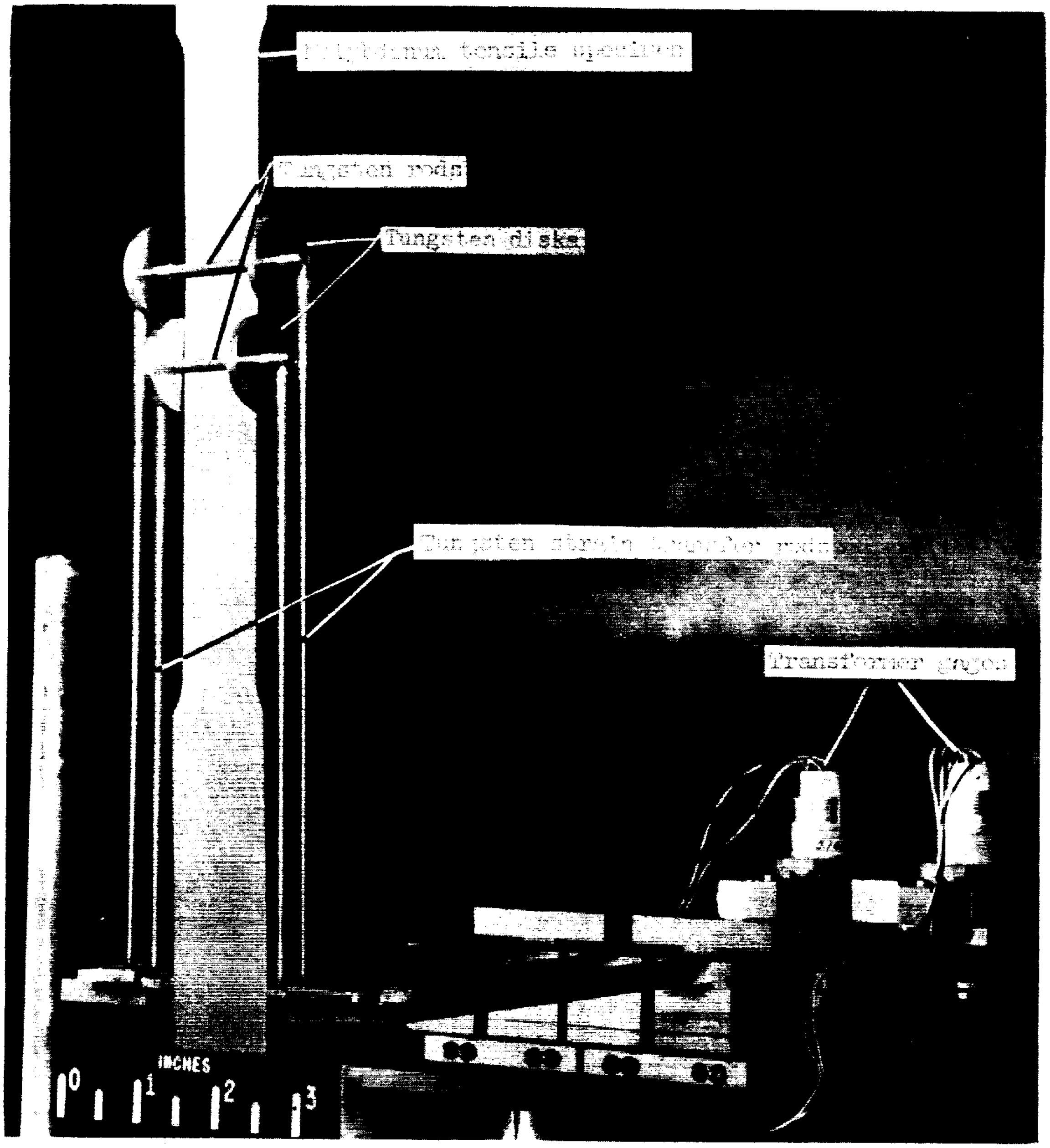
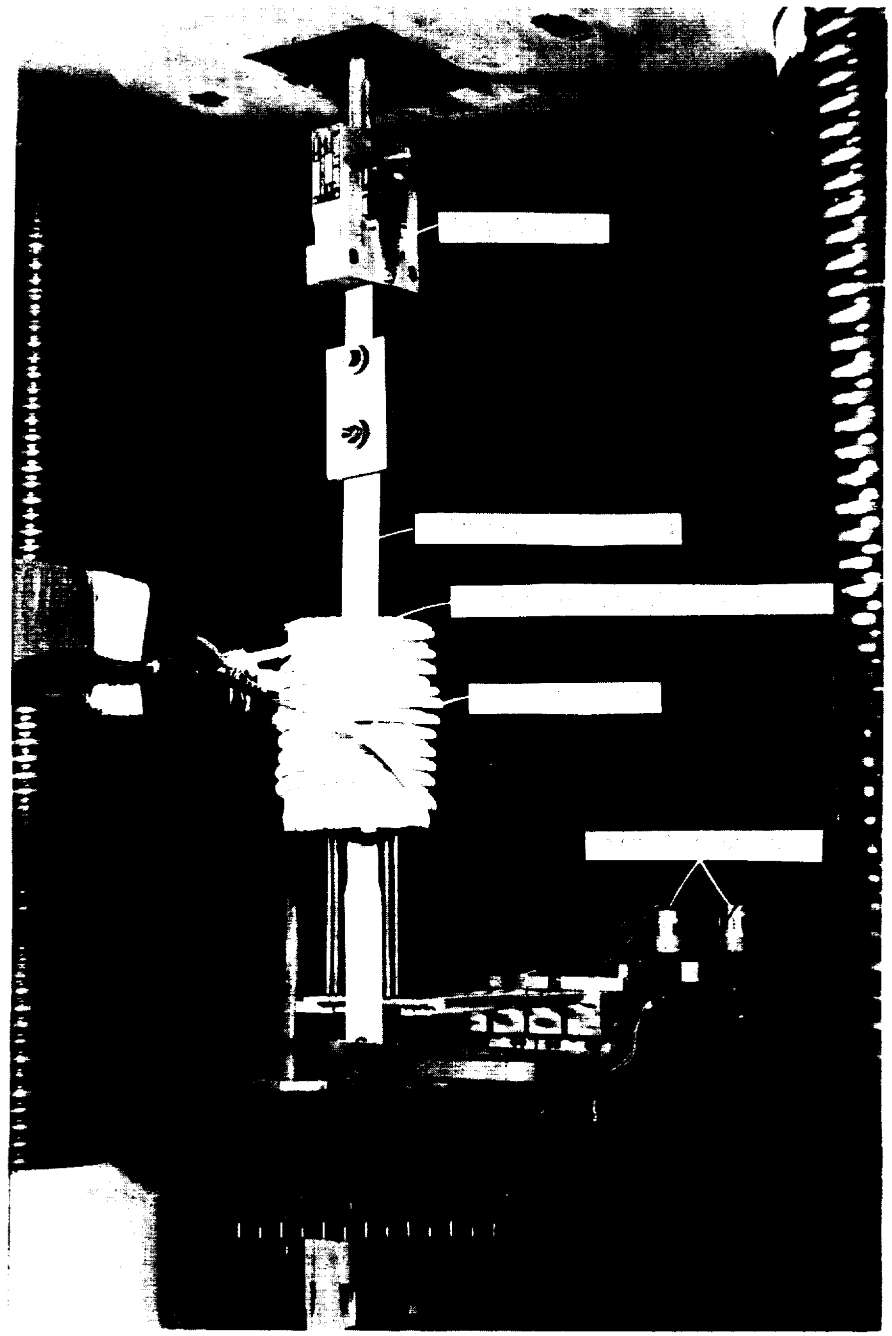


Figure 23.- Tungsten extensometer assembly. L-60-73.1

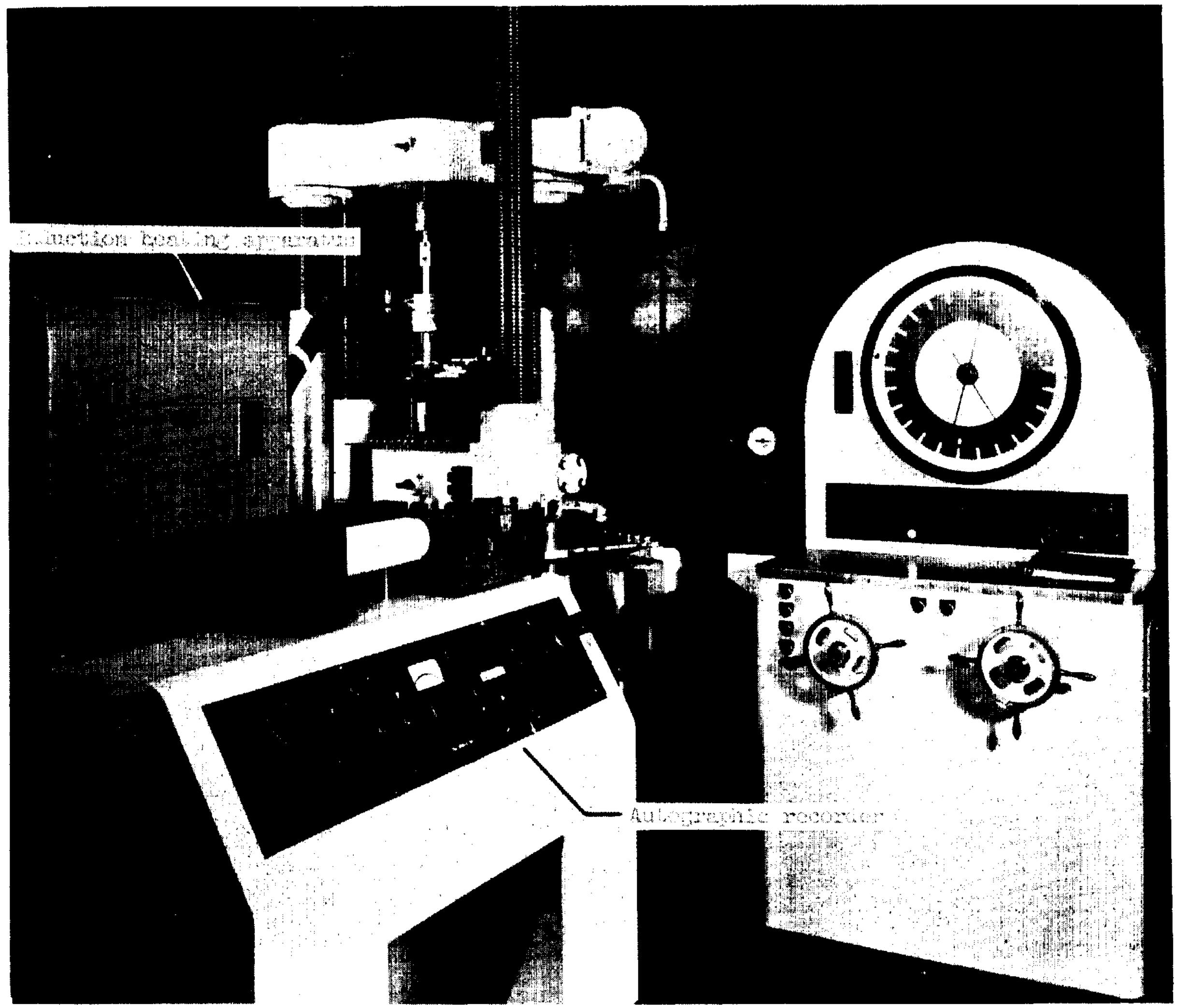
L-1004

L-1004



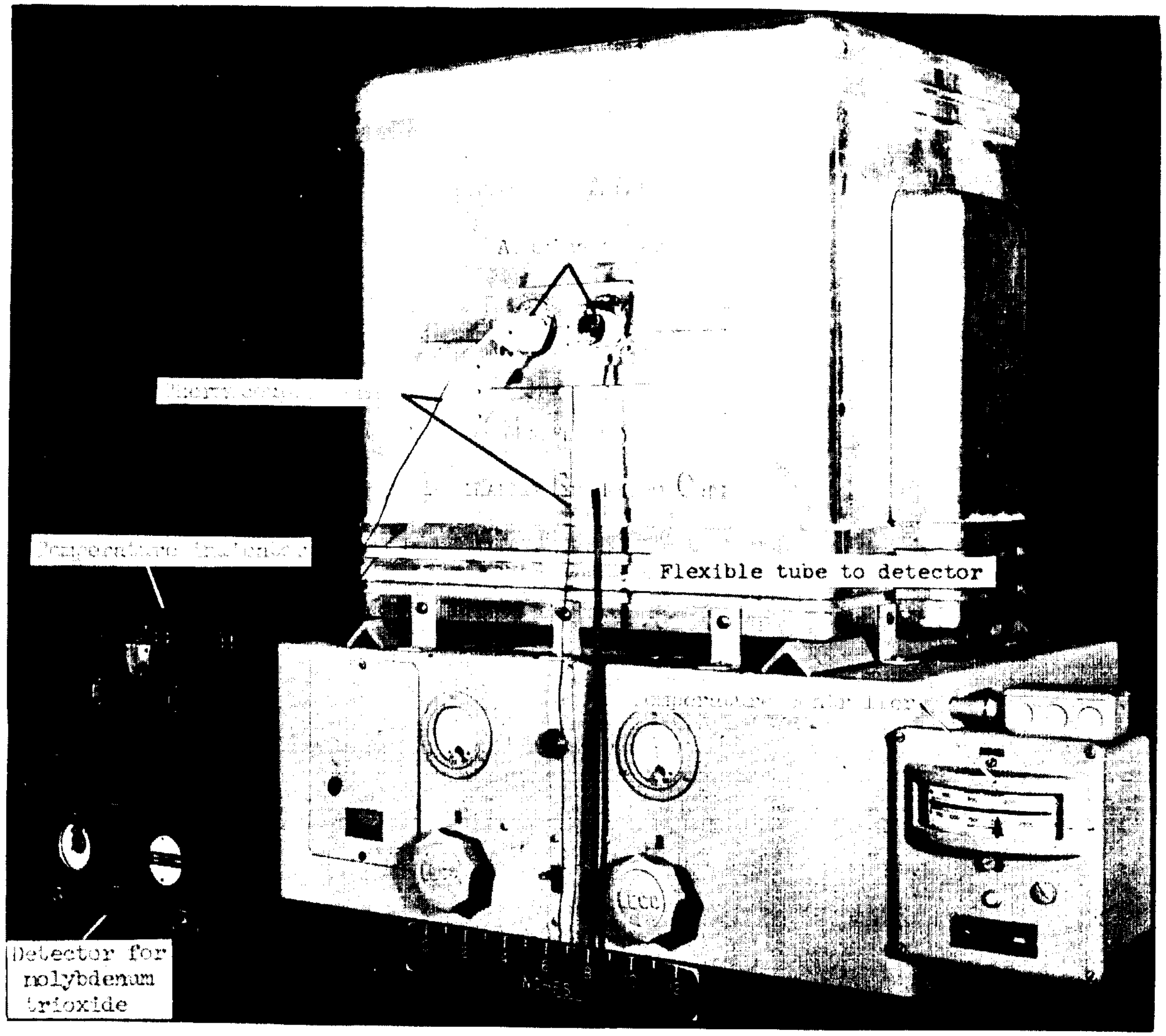
L-60-35.1

Figure 24.- Molybdenum tensile specimen mounted in universal hydraulic testing machine showing extensometer assembly, insulated graphite susceptor, and induction coil.



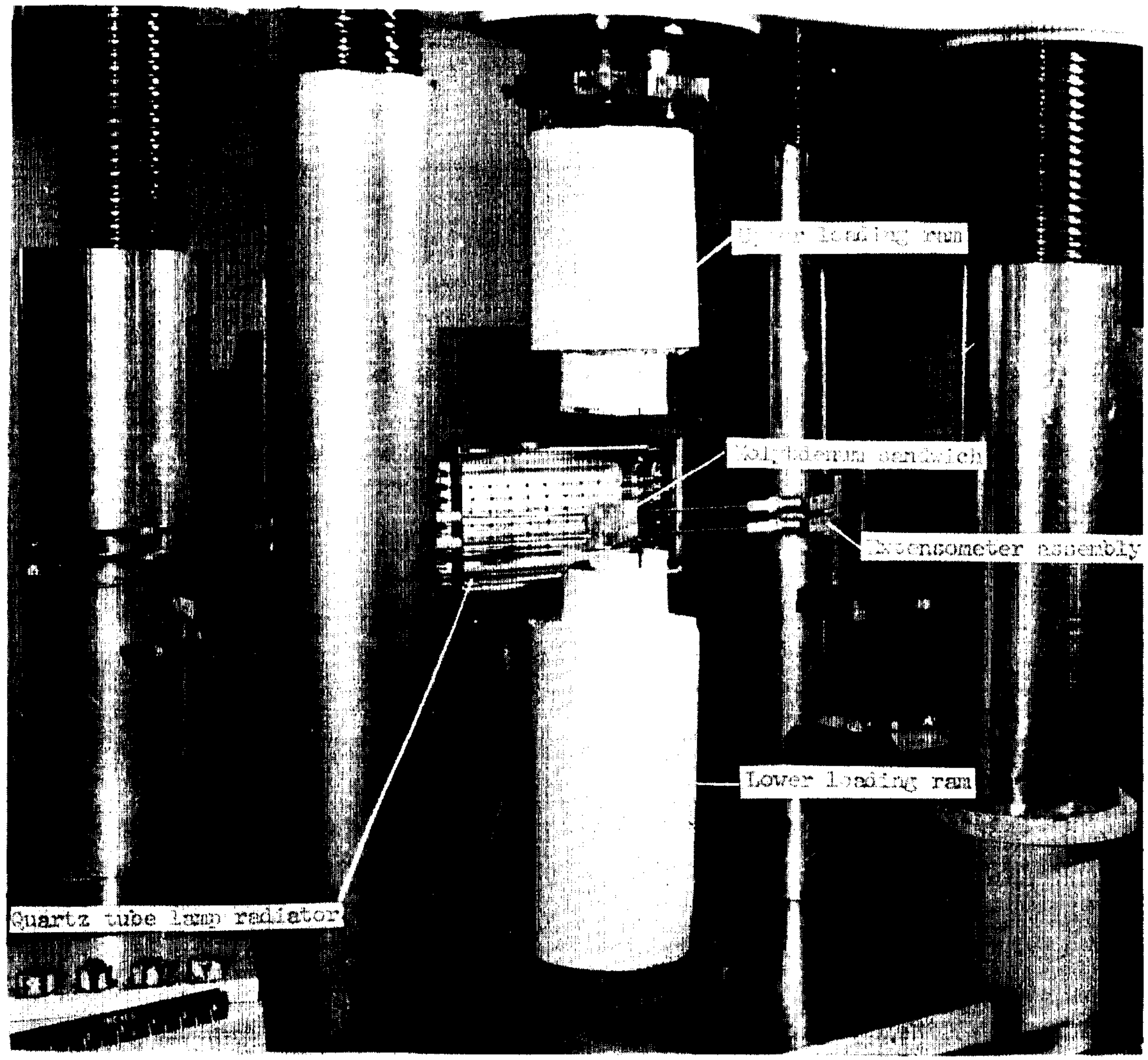
L-60-33.1

Figure 25.- Overall view of test setup for tensile stress-strain tests.



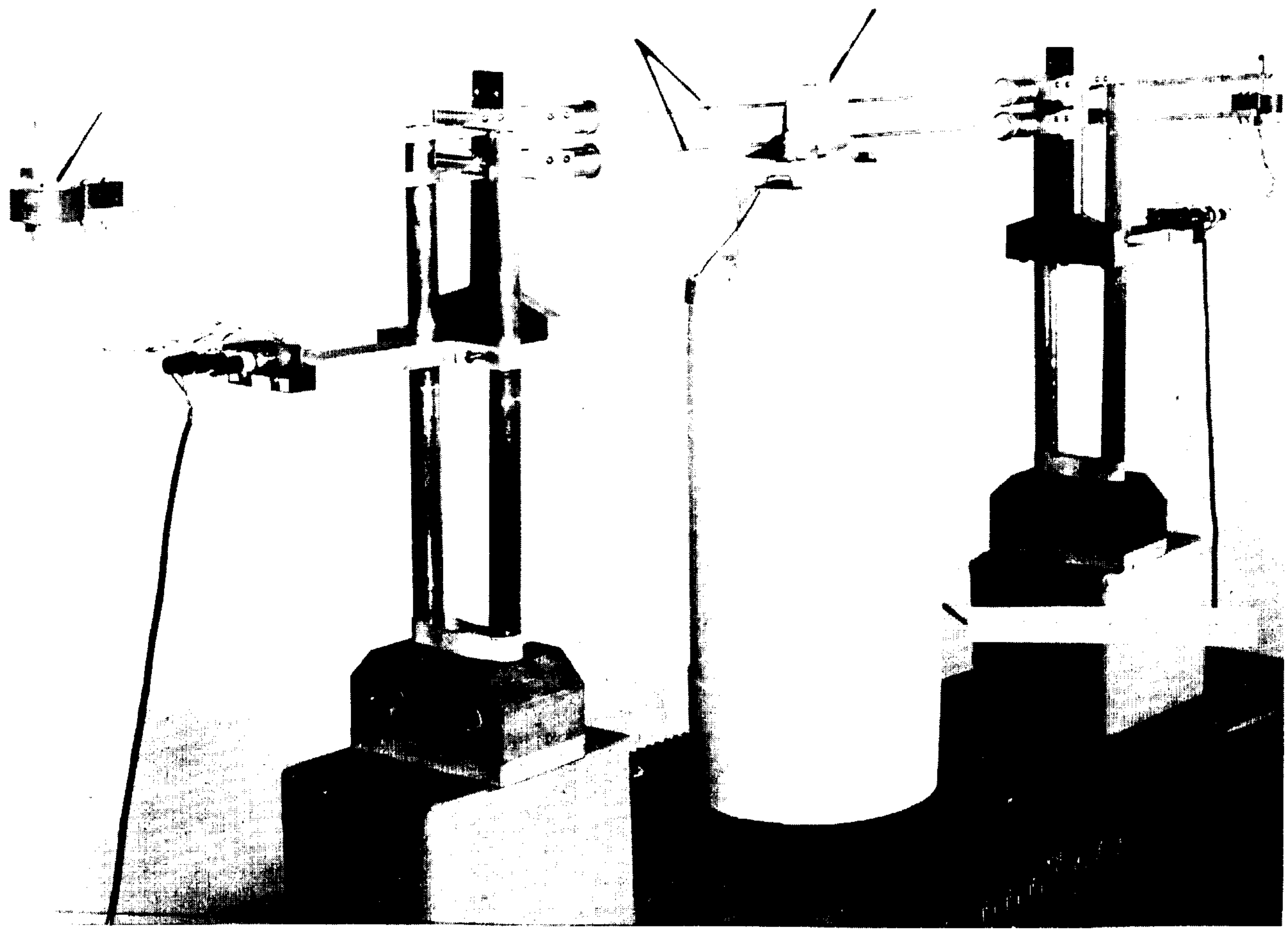
L-59-7998.1

Figure 26.- Tube furnace utilized for oxidation tests on W-2 coating.



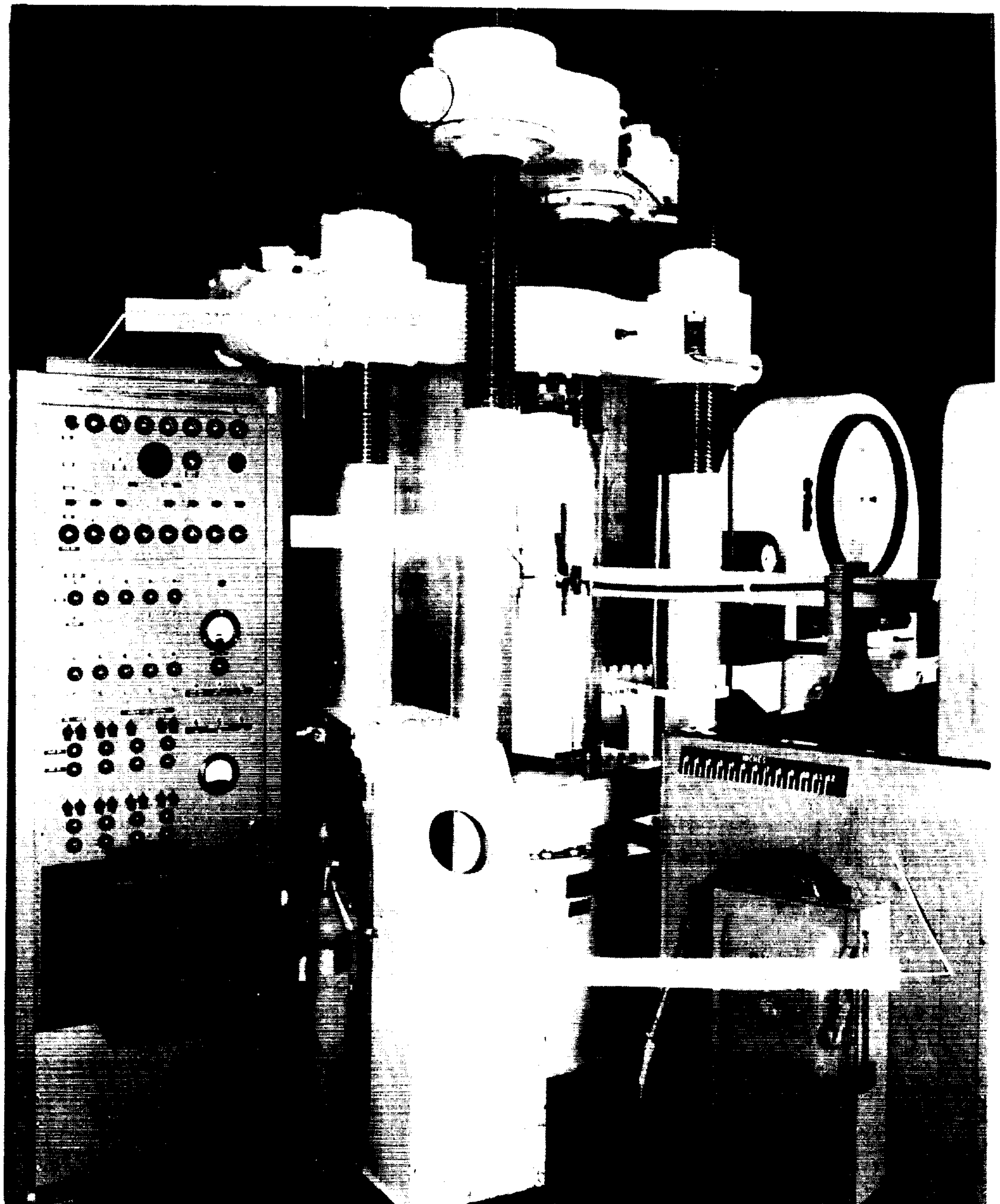
L-59-6369.1

Figure 27.- View of equipment utilized in performing axial compressive loading tests on corrugated-core sandwiches to 2,100° F.



L-59-6373.1

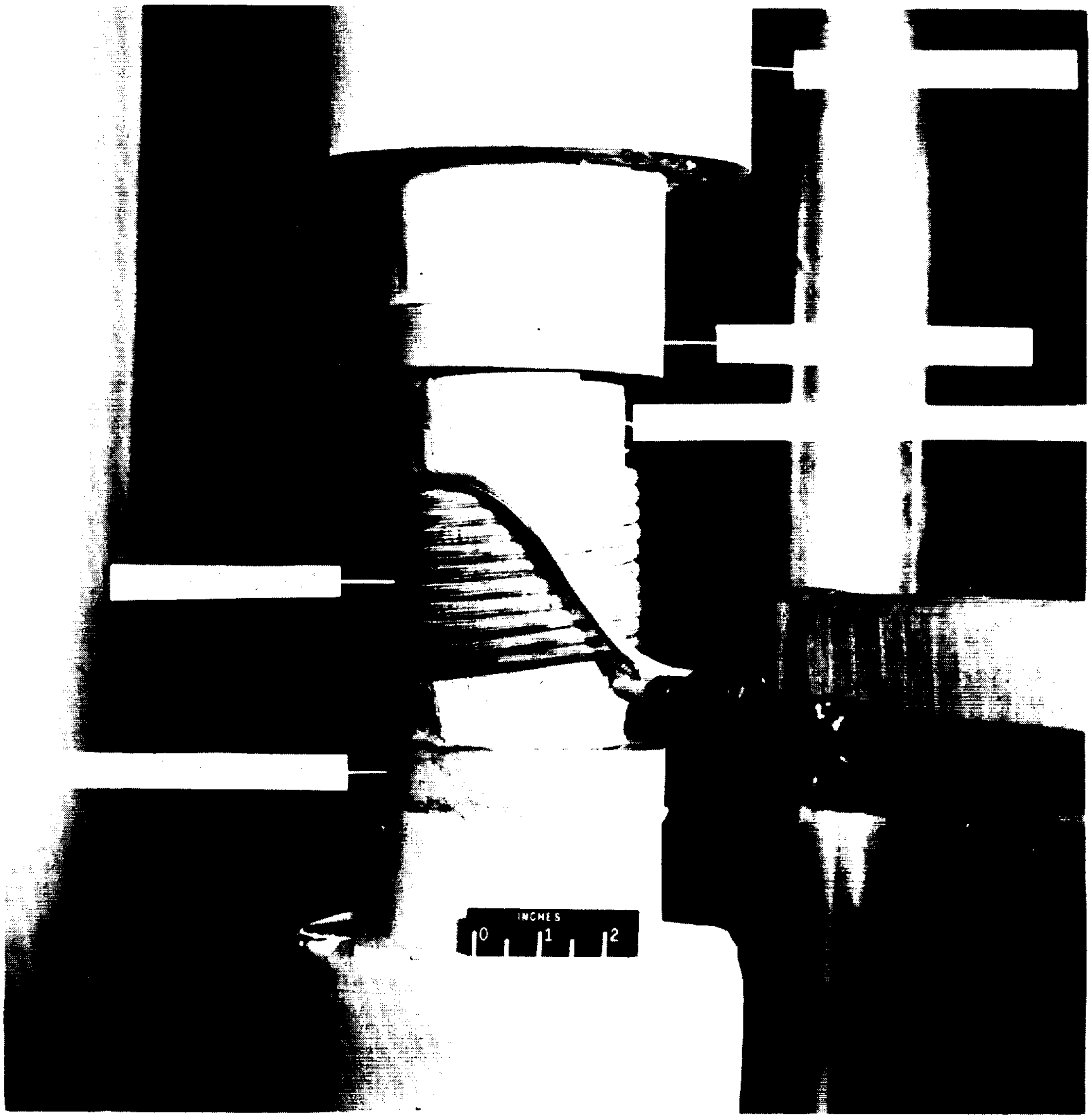
Figure 28.- Extensometer assembly utilized for measurement of axial shortening of corrugated-core sandwiches up to 2,100° F.



L-59-6378.1

Figure 29.- View of test setup utilizing induction heating apparatus for axial compressive strength tests on corrugated-core sandwiches from 2,400° F to 3,000° F.

L-1004



L-59-6379.1

Figure 30.- View of induction coil, insulated graphite susceptor and loading rams utilized in axial compressive strength tests on corrugated-core sandwiches.

L-59-6723.1

Figure 31.- View of induction coil, graphite susceptor, graphite loading rams, and thermocouple assembly.

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NASA TN D-447
National Aeronautics and Space Administration.
INVESTIGATION OF PROBLEMS ASSOCIATED WITH THE USE OF ALLOYED MOLYBDENUM SHEET IN STRUCTURES AT ELEVATED TEMPERATURES.
Eldon E. Mathauser, Bland A. Stein, and Donald R. Rummler. October 1960. 60p. OTS price, $1.50.
(NASA TECHNICAL NOTE D-447)

The results of an experimental study to explore the capabilities and limitations of thin Mo-0.5Ti molybdenum-alloy sheet for structural applications at high temperatures are presented. Evaluation tests at temperatures ranging from room temperature to 3,000° F were made on resistance-welded corrugated-core sandwiches with a W-2 coating and on coated oxidation and tensile specimens. The performance of the corrugated-core sandwiches in compressive strength and static oxidation tests, tensile properties of coated molybdenum sheet, and life of the coated specimens in static oxidation tests are

Copies obtainable from NASA, Washington (over)

I. Mathauser, Eldon E.
II. Stein, Bland A.
III. Rummler, Donald R.
IV. NASA TN D-447

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NASA TN D-447
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