NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

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SOME PROPERTIES OF HIGH-PURITY SINTERED WROUGHT MOLYBDENUM METAL AT TEMPERATURES UP TO 2400° F

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SUMMARY

The tensile-strength properties of sintered wrought molybdenum were investigated at temperatures from 1800° to 2400° F, as well as the effects of swaging, recrystallization, and "test-section" area on these tensile-strength properties. Additional studies were made on stress-rupture properties, types of fracture, directional tensile properties of rolled plate, and metallography of swaged and recrystallized molybdenum.

At temperatures in excess of 1800° F, commercially pure sintered wrought molybdenum metal had excellent tensile strength, ductility, and stress-rupture life comparable with or superior to other proposed high-temperature materials. At a temperature of 1800° F, the short-time tensile strength varied between 25,310 and 33,670 pounds per square inch. At 2400° F, the tensile strength varied between 13,070 and 26,100 pounds per square inch, depending upon manufacturing swaging variables and whether the evaluation temperature was above or below the recrystallization temperature of the specimen evaluated.

Recrystallization lowered the tensile strengths of molybdenum at all temperatures. Recrystallized metal had ductility at elevated temperatures but was brittle at room temperature. The amount of swaging on the particular bar sizes investigated had little effect upon increasing the tensile strength, but increasing amounts of swaging progressively lowered the recrystallization temperature. Increasing the test-section cross-sectional area of a specimen had a negligible effect upon the evaluation of the tensile strength.

Wrought molybdenum had a 100-hour stress-rupture life at approximately 19,300 ±300 pounds per square inch at 1800° F. Recrystallized metal had a 100-hour life at approximately 10,000 pounds per square inch.

"As-swaged" molybdenum exhibited a transgranular failure under tension at all temperatures until effective recrystallization occurred.

Recrystallized molybdenum exhibited intergranular failure at room temperature; from 1500° to 2000° F, the fracture was predominately transgranular; from 2000° to 2400° F, the fracture reverted to the intergranular type.

Rolled molybdenum plate possessed definite directional properties, having the greater tensile strength in the transverse direction to rolling and the greater ductility in the longitudinal direction of rolling.

Different manufacturers' products, having a similar swaging reduction, had considerably different strength and recrystallization properties.

INTRODUCTION

The present need for engineering materials having satisfactory mechanical properties at temperatures in excess of 1800° F has necessitated thorough investigation of all promising materials. Although the element molybdenum is a tough, dense, refractory metal with a melting point of 4750 ±50° F, it has never been considered suitable for use at elevated temperatures, in other than a vacuum or controlled atmosphere, because of its poor oxidation resistance. Recently, however, coatings have been developed that protect molybdenum from oxidation at elevated temperatures.

A literature survey disclosed almost no available data on the elevated-temperature mechanical properties of molybdenum (except in the form of wire a few tenths of a millimeter in diameter) manufactured by powder-metallurgy methods. In the temperature range of room temperature to 1600° F, 0.625-millimeter molybdenum wire has a tensile strength of 105 to 56 kilograms per square millimeter (149,500 to 79,700 lb/sq in.) (reference 1). Some data have been published on the elevated-temperature properties of arc-cast molybdenum (reference 2).

A limited amount of unpublished data was obtained from one of the manufacturers of wrought molybdenum on 0.400- to 0.410-inch-diameter swaged molybdenum rod. The values obtained for the swaged bars varied considerably:

Testing temperature (°F)	Tensile strength (lb/sq in.)	Elongation in 2 inches (percent)
Room	76,400 ±2400	18.5 ±13.5
400	56,000 ±2000	18.0 ±4.0
800	46,800 ±3800	12.0 ±4.0
1200	44,500 ±4500	10.5 ±.5
1500	33,000 ±6000	10.5 ±2.5
1900	27,500 ±6500	9.0 ±2.0

As few data are available on the properties of swaged molybdenum rod, an investigation was started at the NACA Lewis laboratory to determine some of the mechanical properties of swaged molybdenum rods and rolled plate in the temperature range of 1800° to 2400° F.

Ductile molybdenum is being economically fabricated by powder-metallurgy and arc-casting methods. This report includes only metal fabricated by the following powder-metallurgy method: Pure molybdenum powder is cold-pressed into bar compacts and then sintered in a reducing atmosphere at temperatures near the melting point of the metal. The sintering is followed by hot swaging and drawing or rolling. Swaging and other reduction methods, performed hot, are supposedly done at a temperature below the recrystallization temperature of the material and therefore impart beneficial cold-working effects to the metal, which result in high strength and ductility at room temperature.

The investigation included the following parts:

1. The high-temperature tensile properties as influenced by swaging, recrystallization, and tensile "test-section" area of swaged bars

2. Stress-rupture strength for 100-hour life at 1800° F of a 1/2-inch-diameter swaged bar

3. The types of fracture in these tests

4. The directional properties of rolled plate

Studies on the metallography of molybdenum metal are presented herein and in the appendix.

MATERIALS

Various bar sizes of high-purity, cold-pressed, sintered and swaged molybdenum were procured from two separate manufacturers. Each bar size was fabricated from a different ingot and received a different amount of swaging. Each lot of metal therefore had possible different physical and mechanical properties. The following table gives the fabrication history:

Source	A				B	
Ingot dimensions (in.)	$1\frac{1}{8} \times 1\frac{1}{8} \times 24$				$\frac{9}{16} \times \frac{5}{8} \times 24$	
Weight of ingot (kg)	5.00				1.41	
Manufacturers' chemical analysis (percent)	0.01 iron; 0.015 nonvolatiles; balance, molybdenum				0.005 iron; 0.003 carbon; balance molybdenum	
Number of ingots swaged	1	3	2	2	1	1
Swaged-bar diameter (in.)	$\frac{1}{4}$	$\frac{1}{2}$	0.698	1	$\frac{1}{4}$	$\frac{1}{2}$
Reduction of ingot cross-sectional area (percent)	96	85	70	38	86	44
Specimen test-section diameter (in.)	0.125	0.250	0.125 .250 .375 .500	0.500	0.125	0.250

Directional properties of rolled plate were determined from a longitudinal rolled plate (1/4 by 1-5/8 by 80 in.) procured from source A. The plate was rolled to size from a 5-kilogram ingot.

Three lots of 1/2-inch-diameter stock from source A were used in the investigation. One lot (lot number 3, table II), which was used for stress-rupture evaluation, was heterogeneous and contained many flaws. The data from the sound bars of this lot have been included only as a

matter of interest. Of the two good lots, one was used to determine short-time tensile strengths and the other the 100-hour stress-rupture strength of molybdenum. Both lots of material were comparable with respect to tensile strength at room temperature and 1800° F, and varied only slightly as to grain size.

For determining the effect of tensile test-section cross-sectional area upon the evaluation of the mechanical properties of molybdenum, the 0.698-inch-diameter bar of source A was chosen for three reasons:

1. The recrystallization temperature of the material was above the highest evaluation temperature, thereby eliminating a troublesome variable.

2. The material was swaged a sufficient amount to allow the effects of swaging to penetrate to the center of the bar.

3. After receiving a medium amount of swaging, the bar remained large enough in diameter to permit the fabrication of tensile specimens with test-section diameters of 1/8 to 1/2 inch.

DESCRIPTION OF APPARATUS

Short-Time Tensile Evaluation of Molybdenum

Tensile-testing machine. - A commercial hydraulic-type tensile-testing machine with a low scale of 6000-pound-load capacity was used to fracture tensile specimens.

Heating furnace. - The tensile specimens and the gripping mechanism were heated in a platinum-wound, resistance-type, tube furnace (fig. 1), which was sealed at both ends with fire brick and glass wool.

Atmosphere protection. - Because molybdenum oxidizes rapidly in air at temperatures in excess of 1200° F, a protective atmosphere of helium gas was provided. A metal tube was placed around the entire length of the exposed parts of the tensile specimen, and a sufficient flow of helium gas was introduced through a smaller tube attached to the large one to exclude excessive oxygen. (See fig. 1.)

Temperature control. - The temperature of the specimen and the furnace was controlled by a thermocouple adjacent to the test section of the tensile specimen in conjunction with a potentiometer pyrometer. A parallel power circuit, with a variable resistor in the line, was used in conjunction with the standard on-off power controller to maintain a more constant temperature than could be obtained

with the on-off controller alone. This electrical arrangement provided current to the furnace at all times and thus made it possible to maintain the indicated temperature to ±3° F at the evaluation temperatures.

Tensile specimens. - The test sections of the tensile specimens were machined and finish-ground to 5 to 16 rms. The types used and the dimensions are detailed in figure 2.

Specimen-gripping mechanism. - Tensile specimens with either tapered or threaded ends were held in split collets and grips attached to extension arms with air-cooled pins. All metal parts used within the heating zone of the furnace were fabricated from Inconel X, which met all requirements of strength and oxidation resistance at the maximum test temperature of 2400° F.

Instrumentation. - Temperatures of the test section of the specimens were indicated on a potentiometer pyrometer with two 22-gage chromel-alumel thermocouples. The thermocouples were inserted through an opening in the atmosphere-protection tube and placed against the test section of the specimen. Preliminary calibration tests, using four thermocouples equally spaced along the entire length of the tensile specimen between the grip faces, showed a maximum variation of ±3° F in temperature at the evaluation temperatures.

Stress-Rupture Evaluation of Molybdenum

Stress-rupture machine and furnace. - A lever-arm-type rupture machine and a nichrome-resistance-type tube furnace (fig. 3) were used to determine stress-rupture values.

Atmosphere protection. - A protective atmosphere of argon gas with the use of cobalt-base-alloy grips proved to be the most satisfactory. An atmosphere-protection tube (fig. 3) was placed around the rupture specimen and recessed at both ends into the Haynes Stellite No. 25 alloy grip, which afforded constant atmosphere protection while the specimen was elongating.

Preliminary rupture testing with Inconel X grips and helium gas for a protective atmosphere proved unsatisfactory. Severe corrosion of the molybdenum occurred in contact with or closely adjacent to the Inconel X. This corrosion effect was accelerated with increasing volumes of helium.

Temperature control. - A temperature-control system, similar to that described under "Short-Time Tensile Evaluation of Molybdenum" was used and provided a control of ±5° F at an indicated temperature of 1800° F.

Stress-rupture specimens. - The test sections of the stress-rupture specimens were finish-ground to 5 to 16 rms (microin.). Dimensions are detailed in figure 2.

Instrumentation. - A chromel-alumel thermocouple was placed adjacent to the furnace wall in conjunction with a pyrometer indicating controller. The temperature of the rupture specimen was recorded on a potentiometer pyrometer with two 28-gage, chromel-alumel thermocouples, wired as illustrated in figure 3.

PROCEDURE

Short-Time Tensile Evaluation of Molybdenum

Heating. - Evaluation temperatures were attained as rapidly as possible and maintained for 1/2 hour to allow for temperature stabilization throughout the specimen before application of the load.

Testing temperature (°F)	Soaking period (min)	Time to reach temperature (hr)
1500	30	1.75 ±0.2
1800	30	2.25 ±.2
2000	30	2.50 ±.2
2200	30	3.00 ±.2
2400	10	3.50 ±.2

The specimens evaluated at 2400° F were soaked at 2390° F for only 10 minutes because of thermocouple difficulties at this high temperature. Before the load was applied, however, the temperature was raised to 2400° F.

Corrosion. - Adequate atmosphere protection was provided. A tensile specimen, heated to 2400° F and soaked for 10 minutes, had its test-section diameter reduced only 0.0002 inch.

Rate of loading. - An approximate rate of loading of 2500 pounds per minute was used for all sizes of specimens up to the load at which the specimen began to yield appreciably. Above this load, the rate of loading was increased as rapidly as possible to eliminate possible short-time stress-rupture factors and to prevent serious deformation of the gripping mechanism.

Elongation measurements. - Percentages of elongation were calculated by measuring the increase in the length of the specimens between two notches placed above and below the test section on a larger diameter than the test section (fig. 2). The elongation between these two notches was then calculated in terms of the actual test-section length.

At elevated temperatures, the rate of strain beyond the yield point may produce variations in the percentage elongation. Because the rate of strain beyond the yield point was not controlled, the elongation values are probably influenced by this factor. This influence may account for some of the variation, other than material irregularities, in the ductility values reported.

Stress-Rupture Evaluation

The rupture specimens were heated to the test temperature in approximately 4 hours and soaked for 1/2 hour before the full tension load was applied.

Recrystallization

Bars from both sources, intended for both tensile and stress-rupture testing, were recrystallized at various times and temperatures prior to testing in a helium protective atmosphere, except as otherwise noted in tables I and II.

RESULTS

High-temperature tensile properties as influenced by recrystallization, swaging, and "test-section" area. - Table I lists the short-time ultimate tensile strengths of commercial molybdenum at room temperature and over the elevated-temperature range of 1800° to 2400° F. The strength of swaged bars at 1800° F varied between 25,310 and 33,670 pounds per square inch and between 13,070 and 26,100 pounds per square inch at 2400° F.

Recrystallization has a detrimental effect upon the ultimate tensile strength of molybdenum both at room and elevated temperatures (table I and fig. 4). The ductility of recrystallized metal remained approximately the same as "as-swaged" material in the temperature range of 1500° to 2000° F (table I) but was negligible at room temperature and lower at temperatures in excess of 2000° F.

Swaging had a negligible effect on increasing the ultimate tensile strength (fig. 5), but had a pronounced effect on lowering the recrystallization temperature. Bars that had a swaging reduction of 85 percent or more had a rapid drop in strength between 2000° and 2200° F, as the recrystallization temperature was within or lower than this range. Those bars that received less than 70-percent reduction had recrystallization temperatures above 2400° F and therefore showed only a slight decrease in strength with increasing temperature.

The effect of additional swaged area, that is, increased test-section cross-sectional area, on the strength properties of a fairly homogeneous swaged rod is shown in figure 6. The strengths, with increasing test-section area and temperature were nearly the same, being within the general experimental scatter band, which indicates uniformity of properties across the areas investigated.

Stress-rupture strength for 100-hour life at 1800° F of 1/2-inch-diameter swaged bar. - The values of stress-rupture life for both as-swaged and recrystallized molybdenum are listed in table II. A logarithmic plot of these values (fig. 7) places the 100-hour life of as-swaged metal at 19,300 ±300 pounds per square inch and that of recrystallized metal at approximately 10,000 pounds per square inch.

Types of fracture. - As-swaged molybdenum exhibited a reasonably ductile, transgranular failure at all temperatures up to the recrystallization temperature. Recrystallized metal exhibited a brittle, intergranular failure at room temperature; from 1500° to 2000° F the fracture was ductile and predominately transgranular; from 2000° to 2400° F the fracture reverted to the intergranular type with a decrease in ductility.

Directional properties of rolled plate. - Rolled plate, 1/4 inch thick, was found to possess 10 to 15 percent greater tensile strength in the transverse direction than in the longitudinal (table III and fig. 8) at both room temperature and 1800° F. The ductility, however, was greater in the longitudinal direction (table III).

DISCUSSION OF RESULTS

High-Temperature Tensile Properties as Influenced by Swaging, Recrystallization, and Test-Section Area

Effect of swaging and recrystallization. - When swaged metal from source A was subjected to either a 70-, 85-, or a 96-percent swaging reduction, the ultimate tensile strengths of all bars at each

evaluation temperature from room temperature to 2000° F were essentially the same (fig. 5).

The 1/4- and 1/2-inch-diameter (96- and 85-percent reduction) material showed a rapid drop in strength between temperatures of 2000° and 2200° F and a less rapid drop as 2400° F was approached. This rapid rate of change of strength was caused by the "effective" recrystallization of the metal (by "effective" is meant that recrystallization which affects the strength properties) during the period it was subjected to elevated temperatures. The photomicrographs in figure 9 show the grain directionalism imparted by the swaging operations and the structure of the metal after having been fractured in tension at a temperature of 1800° F (figs. 9(a) and 9(c)). Figures 9(b) and 9(d) are views of the same materials after fracture at 2200° F showing the recrystallized structures in both the 1/4- and 1/2-inch-diameter material.

The 0.698-inch-diameter material having a 70-percent reduction (fig. 5) showed a uniform decrease in strength up to 2400° F; such a decrease indicated that "effective" recrystallization had not occurred. Photomicrographs (figs. 9(e) and 9(f)) show no evidence of recrystallization. Two subsequent specimens heated for 48 hours, one at 1800° and one at 2200° F, showed no decrease in the 1800° F tensile strength. A third specimen tested at 2600° F, heated by induction, showed an appreciable drop in strength, which is evidence that the 1-hour recrystallization temperature would be approximately 2500° F.

The 1-inch-diameter swaged bar having a 38-percent reduction had a 15- to 25-percent-lower tensile strength than the other swaged bars (table I and fig. 5) at both room temperature and 1800° F. From 1800° to 2400° F, the uniform decrease in strength with no rapid drop indicated absence of effective recrystallization. Examination of figures 9(g) and 9(h) reveals no essential difference in the microstructure of the specimens tested at 1800° and 2200° F, respectively.

The 1-inch bar exhibited a microstructure different from the other bar materials. The structure shown in figures 9(g) and 9(h) represents a central core approximately 1/2-inch in diameter surrounded by an outer layer of grains similar to but larger and less fibrous than the grains of figure 9(a). The grains within the core material (75 to 100 percent of the area of the cross section of the specimen test section) were rather uniform, showing a mixture of fine strained and fragmented grains with only slight directionalism. Recrystallization studies of the core metal indicated a 1-hour recrystallization temperature of approximately 2825° F.

The evaluation of as-swaged molybdenum indicates that swaging had a negligible effect on increasing the tensile strength of the metal at all temperatures below the recrystallization temperature. The effect of swaging upon lowering the recrystallization temperature, however, is pronounced and limits the usefulness of molybdenum metal in the higher-temperature applications. Increased swaging reduction decreases room-temperature brittleness, but only if the swaging adds "effective" cold work to the metal. Brittleness is not evident in radially swaged material; it is evident in rolled plate in a direction normal to the plate surface.

In order to determine the over-all effect of recrystallization on the elevated-temperature tensile strengths of molybdenum, material from source B was used. The as-swaged 1/2-inch-diameter bar did not possess a very uniform crystalline structure. Representative photomicrographs from two tensile specimens (figs. 10(a) and 10(b)) showed elongated structures with definite directionalism, but a considerable difference in grain size.

Complete recrystallization (fig. 10(c)) was obtained by heating specimens for 48 hours at a temperature of 2400° F. This recrystallized structure possessed considerably less strength and less ductility than the original as-swaged material at room temperature. At 1800° F the tensile strength was about 80 percent of that of the original as-swaged material (table I). Above 2000° F, the as-swaged material was subjected to a temperature high enough and for a long enough time during testing to allow "effective" recrystallization to take place. The strength values obtained at 2200° and 2400° F for as-swaged material from source B and plotted in figure 4 therefore approach the values of the fully recrystallized material and are not true comparisons of strength between recrystallized and actual as-swaged molybdenum.

The recrystallization of this material had considerable effect upon the ductility. At room temperature the metal was brittle and possessed much less ductility than the as-swaged material. From 1500° to 2000° F, the metal exhibited 20- to 30-percent elongation, which dropped rapidly to about 4 percent at 2400° F.

The determination of the 75-hour recrystallization temperature of 1/2-inch-diameter bar stock from source A shows an excellent example of the initial changes in mechanical properties caused by recrystallization. Figure 11(a) shows the elongated grain structure of the as-swaged material, which had a transgranular fracture at room temperature. Figure 11(b) shows that the material remained essentially in the as-swaged condition but with a few recrystallized grains

after having been heated for 75 hours at a temperature of 1800° F. This limited amount of recrystallization had no effect upon the strength, the ductility, or the type of fracture at room temperature.

After heating for 75 hours at 1900° F (fig. 11(c)), almost complete recrystallization was attained. A few unrecrystallized grains remain, the material not having had sufficient time at temperature to recrystallize completely. These specimens when tested at room temperature, failed with an intergranular-type fracture and at a strength that was 77 percent of the as-swaged strength. Also, the percentage elongation dropped from approximately 1.0 percent to zero.

The tensile strength at 2000° F was about 86 percent of the as-swaged strength. The ductility, though, remained essentially the same and there was a greater tendency toward an intergranular fracture at 2000° F.

The tensile strength of the recrystallized 1-inch-diameter molybdenum decreased to 43 percent of the as-swaged strength at room temperature and the percentage elongation decreased from 1.2 percent to zero (table I). At a temperature of 1800° F, the strength was 88 percent of that of the as-swaged material and the ductility was good.

Summary of recrystallization studies. - The data illustrate the importance of the recrystallization temperature when molybdenum is to be considered for elevated-temperature applications. If this recrystallization time-temperature limit is exceeded during application, detrimental changes occur, which would limit the usefulness of the material at both room and elevated temperatures.

A table summarizing all the recrystallization temperatures and times is included in the appendix.

Effect of tensile-specimen test-section area. - In the process of swaging, the metal adjacent to the circumference of a bar receives more mechanical working and reduction in area than the metal in the center of the bar. If this difference in the amount of working imparted considerably different mechanical properties to the material in the center and to that material removed from the center, the strength data obtained from a tensile specimen with a small test-section diameter would not be representative of the entire cross section of the bar.

The 0.698-inch bar had a typical over-all swaged structure consisting of elongated, severely strained grains, which were slightly

more fibrous near the circumference of the bar. The grains were fairly uniform in size and shape across the diameter of the bar stock but varied somewhat along the length of the bar. The X25 photomicrographs in figure 9(e) and 9(f) are typical examples of this variation.

The strengths obtained for all specimens regardless of test-section area varied only a maximum of about 3500 pounds per square inch at both room and elevated temperatures (fig. 6), which is within material variations. Other materials subjected to more severe swaging, and therefore having a more uniform structure, also had a strength fluctuation of several thousand pounds per square inch at the same temperature. If a difference of mechanical properties does exist from the center to the outside, it is too small to detect. It was found in the study of the effect of swaging that a considerable difference in the amount of swaging was necessary to change the strength enough to be detected and this large difference in metal working probably does not exist from the circumference to the center of this bar.

No difference in hardness was perceptible from the center to the circumference of the swaged bar. Rockwell A hardness readings varied from 53.5 to 56.0, with no definite pattern. A considerable difference in the amount of mechanical working has been found to be necessary before a definite trend in hardness could be detected (reference 3).

The effect of test-section area upon ductility could not be determined from the elongation values obtained because the ratio of test-section length to diameter of the different sizes of specimens varied from $2\frac{1}{2}$ to 6. The fractures of all the specimens evaluated at elevated temperatures were similar. Because fracture type, tensile strength, and hardness did not vary appreciably with test-section area, it is believed that ductility would also remain essentially constant.

Stress-Rupture Strength for 100-Hour Life at 1800° F of 1/2-Inch-Diameter Swaged Bar

The value of 19,300 pounds per square inch for the 100-hour rupture life at 1800° F of this lot of material was taken from the logarithmic plots (fig. 7) of a number of tests at specified loads

and could easily vary ±300 pounds per square inch because of the shallow slope of the curve and because close reproduction of rupture life at any one stress could not be obtained. Table II shows that these scattered values of rupture life occurred at all loads for all metal evaluated. A metallographic examination was made of all ruptured specimens and did not reveal any appreciable deviation in crystal structure from that in figure 12(a).

The 100-hour recrystallization temperature was in excess of 1800° F; therefore, this variable did not affect the evaluation of as-swaged metal in the long heating period required.

The rupture values reported for recrystallized material were obtained from specimens that were fully recrystallized in a hydrogen atmosphere (fig. 12(b)). Attempts were made to recrystallize this material fully in atmospheres of both helium and argon, and also in a vacuum (0.5 micron) but were unsuccessful as to the outer edges of the material. This fringe of as-swaged material (10 to 20 percent of the area of the test section) had a noticeable effect on the rupture life.

The excellent ductility and the lower strength of recrystallized molybdenum at 1800° F were much more evident in the rupture tests than in the short-time tensile tests. A rupture specimen had a 100-hour rupture strength of approximately 10,000 pounds per square inch and elongated about 100 percent before failure, with most of the elongation occurring within the 1/2-inch-long test section. This elongation and the resulting strain during evaluation were sufficient to hot-cold work the recrystallized grains into a grain structure similar in appearance to metal that had been subjected to 50 or more percent reduction by swaging during manufacture (fig. 12(c) and 12(d)). Probably this structure was formed after the test section had been appreciably reduced in diameter and therefore the rupture specimen was subjected to a much higher unit stress.

A third lot of 1/2-inch-diameter molybdenum, from another sintered and swaged ingot (source A), was also evaluated in rupture. This material was found to be extremely heterogeneous and unlike other material that had received an approximately equivalent amount of swaging. Many fractured rupture specimens were metallographically examined and found to contain hollow pipes and cold shuts in the test section. The data obtained from sound bars of this material (lot number 3, table II) have been included, however.

The test section of these specimens contained from 0 to 100 percent of very small fragmented grains (fig. 13), perhaps 1/100 the size of a typical grain, and the balance of the area was made up of the

typical, average-sized grains. The fine-grained material was found to be centrally located in the bar with a few streaks of typical material interspersed in some specimens. Table II lists the approximate percentage of fine-grained material in the test-section cross-sectional area of the specimens. No evident relation exists between the amount of fine grains present and the rupture life; however, the 100-hour rupture strength of this lot (approximately 19,700 lb/sq in.) is higher than that of the other lot containing no fine-grained material.

Types of Fracture

As-swaged and recrystallized molybdenum exhibited six distinct macro types of fracture at room temperature and from 1500° to 2400° F (fig. 14). A general description of the different types and their reductions in area is as follows:

1. Pin point; transgranular with 70- to 90-percent reduction in area

2. Approximately 45° shear either transgranular or intergranular with 10- to 40-percent reduction in area

3. Honeycombed tear; predominately transgranular with 50- to 99-percent reduction in area; torn or honeycombed surface along test-section length

4. Tear; predominately transgranular with approximately 40-percent maximum reduction in area

5. Brittle; transverse; transgranular or intergranular with essentially no reduction in area

6. Ductile transverse-transgranular with 0- to 25-percent reduction in area.

Molybdenum from both sources showed similar types of fracture for both as-swaged and recrystallized material. The as-swaged metal, worked sufficiently to give a fairly uniform grain structure from the circumference to the center of the bar, had a transgranular fracture with good elongation at all temperatures up to the recrystallization temperature. That material which was only partly recrystallized before or during testing had a torn fracture that was a mixture of both transgranular and intergranular failure similar to types 3 and 4 at temperatures above 2000° F. Type 3, with the honeycombed

surface, appeared to be an intergranular separation of the grains on the surface, as shown in the photomicrograph in figure 14(c), but was predominately transgranular.

When molybdenum is fully recrystallized, the grain boundaries are weaker than the grains at both room temperature and at temperatures above 2000° F; this boundary weakness results in an intergranular failure with low elongation. At temperatures from 1500° to 2000° F, the metal showed ductility as great as or greater than the as-swaged material. Figure 15(a) shows that in this temperature range the fracture was predominately transgranular. The grain boundaries apparently possessed sufficient strength and ductility to allow transgranular rupturing. Comparison of the photomicrographs (figs. 14(b) and 15(b)) of the test section, remote from the fracture zone, shows greater grain strain and deformation at 1800° F and shows higher elongation before fracturing. This change from a brittle, intergranular fracture at room temperature to a ductile, transgranular fracture from 1500° to 2000° F, reverting again to a much less ductile, intergranular fracture above 2000° F, seems rather odd, but is borne out in part by the work reported in reference 4. This work on recrystallized 0.025-inch-diameter wire showed an intergranular fracture at room temperature and a transgranular fracture at 572° F.

Directional Properties of Rolled Plate

Longitudinal rolling of molybdenum plate produces an anisotropic material, which is still strong and ductile in both the transverse and longitudinal directions (fig. 8). The material at room temperature is about 10,000 pounds per square inch stronger in tension in the transverse direction and at 1800° F about 5000 pounds per square inch stronger. There is little difference in ductility regardless of direction, at room temperature, but at 1800° F the transverse percentage elongation is about 50 percent of that in the longitudinal direction. The ductility in a direction normal to the surface, although not determined in this investigation, is known to be very low because of the brittle planes of weakness parallel to the rolling-surface plane.

Because of the rolling, the tensile fractures were wedge-shaped below the recrystallization temperature, the sharp edge of the wedge being parallel to the rolling surface. Above the recrystallization temperature, the fracture changed to a rough tear type.

Photomicrographs of the tensile specimens fractured at 1800° F (figs. 16 and 17) show that the rolling produced thin, flat grains with a longitudinal dimension of approximately one and one-half times

to twice the length of the transverse dimension. The greatest elongation would be expected to occur along the axis of the long dimensions of the grains. This expectation was borne out by the elongation measurements (table I). A photomicrograph of a plane parallel to the rolled surface of a longitudinal tensile specimen (fig. 16(b)) shows a greater elongation of the grains at the fracture than the same view of a transverse specimen (fig. 17(b)) in which the tension force was along the short dimension of the grains.

Manufacturing Variables

In the evaluation and comparison of the two manufacturers' products, a considerable variation was found to exist in properties, particularly at elevated temperatures. Because different ingot sizes were used by the two manufacturers, a comparison of material on the basis of finished swaged bar size could not be made. Therefore, a comparison was attempted between material that had received approximately equivalent amounts of ingot-area reduction.

Molybdenum from source A had a fairly clean, uniform, fibrous grain structure in all the bar sizes except the 1-inch-diameter size. Sufficient fabrication uniformity was maintained to produce a product that possessed a progressively lower recrystallization temperature and a tendency toward higher strengths with increasing amounts of swaging. Molybdenum from source B did not show any similar systematic change in properties for equivalent amounts of swaging, nor did it possess nearly the uniformity of grain structure.

The heavily swaged metal of both manufacturers had a rather uniform, fibrous, grain structure, but the material from source B had a recrystallization temperature approximately 125° F higher than that of A and recrystallization occurred over a greater range of temperatures.

Material from source B that was lightly swaged (44-percent reduction in area) had a very nonuniform grain structure, unlike any other material examined. The grains varied considerably in size (figs. 10(a) and 10(b)) and appeared to have some porosity. The strength of the material below the recrystallization temperature was 20 to 35 percent higher than material from source A that had received an equivalent amount of swaging. Material from source B, moreover, had a recrystallization temperature approximately 600° F lower than source A material. This recrystallization temperature, strangely, was about the same as for other molybdenum from source B that was swaged about twice as much.

The lightly swaged molybdenum from source B (figs. 10(a) and 10(b)) had a grain structure that resembled recrystallized material much more closely than the fibrous as-swaged structure found in other material. This material was probably subjected to a temperature in excess of the recrystallization temperature of the metal during the latter part of the swaging treatment for a sufficient length of time to cause full or partial recrystallization to occur. Further swaging at lower temperatures might then produce some smaller fragmented grains as evident in figure 10(a). Possibly a large amount of previous effective swaging was lost when recrystallization occurred. It is difficult to explain why the material still possesses excellent strength below the recrystallization temperature for the small amount of total ingot reduction. Therefore, total ingot reduction is not considered to be a criterion by which mechanical properties can be predicted.

Material Comparison at Elevated Temperatures

Molybdenum, as-swaged or recrystallized, has tensile properties from 1800° to 2400° F equivalent to or higher than other high-temperature materials, as shown by figure 18. Figure 19 is a comparison with these same high-temperature materials on a strength-to-weight-ratio basis.

The 100-hour stress-rupture strength at 1800° F is shown in figure 20 to be higher than several other high-temperature materials.

SUMMARY OF RESULTS

The following results were obtained from an investigation of the properties of high-purity sintered wrought molybdenum metal at temperatures up to 2400° F:

1. Commercially pure sintered wrought ("as-swaged") molybdenum possessed tensile strength and ductility in the temperature range of 1800° to 2400° F comparable with or higher than other proposed high-temperature materials. At a temperature of 1800° F, the short-time tensile strength varied between 25,310 and 33,670 pounds per square inch. At 2400° F, the tensile strength varied between 13,070 and 26,100 pounds per square inch, depending upon manufacturing swaging variables and whether the evaluation temperature was above or below the recrystallization temperature of the specimen evaluated.

2. At 1800° F, the stress for the 100-hour stress-rupture life of as-swaged molybdenum was approximately 19,300 ±300 pounds per

square inch, which is higher than that of other proposed high-temperature alloys. The stress for 100-hour life of recrystallized molybdenum at 1800° F was approximately 10,000 pounds per square inch.

3. Recrystallization lowered the tensile strengths of molybdenum at all temperatures. Recrystallized metal was quite ductile at elevated temperatures but extremely brittle at room temperature.

4. Swaging, from 38- to 96-percent ingot-area reduction, has little effect on increasing the room-temperature and the elevated-temperature tensile strength, but had a pronounced effect upon lowering the recrystallization temperature.

5. The effect of tensile-specimen "test-section" cross-sectional area upon the ultimate tensile strength was negligible.

6. As-swaged molybdenum exhibited a transgranular failure under tension at all temperatures at which "effective" recrystallization did not occur.

7. Recrystallized metal exhibited an intergranular failure at room temperature; from 1500° to 2000° F the fracture was predominately transgranular; and above 2000° to 2400° F the fracture reverted to the intergranular type.

8. Rolled plate 1/4 inch thick possessed definite directional properties. The tensile strength in the transverse direction was 10 to 15 percent higher than that in the longitudinal direction at all evaluation temperatures below the recrystallization temperature. Ductility in the longitudinal direction was almost double that in the transverse direction at elevated temperatures.

9. Molybdenum with similar amounts of ingot reduction but from two manufacturers possessed considerably different strength and recrystallization properties.

Lewis Flight Propulsion Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, December 10, 1950.

APPENDIX - METALLOGRAPHY OF SINTERED AND SWAGED MOLYBDENUM BARS

Metallographic Techniques

Polishing. - Conventional metallographic polishing methods proved inadequate for the preparation of molybdenum because of a worked surface layer that resisted final polishing methods.

Rough-polishing procedures were standard to 2/0 metallographic polishing papers and were extended to include a 25- and a 14-micron diamond-paste lap. Final polishing was accomplished by the use of electrolytic methods.

Electrolytic polishing methods, described in reference 11, gave excellent results. The process required careful control, and therefore added details and method deviations are itemized as follows:

(1) A current density of 21 amperes per square inch at a $3\frac{1}{2}$-inch electrode separation gave the most consistent results. A source of steady current was a prime requisite. Too high a current resulted in specimen pitting and too low, resulted in etching.

(2) Specimens during electro polishing were agitated in a vertical, in preference to a horizontal, position, which provided easier escape of gas bubbles from the specimen surface.

(3) The solution of 35 millimeters of concentrated sulphuric acid in 140 millimeters of distilled water gave the best results.

(4) Recommended polishing times were reduced to 15 to 20 seconds in order to obtain minimum polishing relief. When porosity was present in the molybdenum, a 6-micron diamond finish and a 5- to 10-second polish were used.

(5) As many as three specimens were polished in one Lucite mount with good results. When the area of one single specimen exceeded 0.300 square inch, however, pitting along the specimen edge and etching in the center resulted from unequal current distribution.

Etching. - Macro observations required a 1:1 nitric acid etch for 30 to 60 seconds followed by a concentrated hydrochloric acid rinse. This procedure gives a brilliant and very detailed etch, but one that lacks grain contrast for photography. After the macro etch with these solutions, swabbing with one part of 10-percent sodium

hydroxide in one part of 30-percent potassium ferrocyanide ($K_3Fe(CN)_6$) improves the grain contrast. For micro observations, the caustic-cyanide etchant was the most satisfactory of the standard molybdenum etchants and gave excellent detail and contrast for microscopic examination and photography. Etching was accomplished by immersion for 35 to 60 seconds, recrystallized structures requiring the longer times. This etchant gives a very light preferential stain depending upon grain orientation (see figs. 22(b) and 26(e)), which can be used to advantage or can be removed as desired by a few seconds rinse in concentrated hydrochloric acid.

As-Swaged Structures

A study of the metallography of sintered and swaged molybdenum was was confined to 1-inch- and 1/2-inch-diameter bars swaged from a rectangular sintered ingot. (See "MATERIALS" section of the report.)

Initial swaging of molybdenum bars produced a superimposition of hot and cold working resulting in complex structures. Although initial swaging is a high-temperature, manually operated, forging operation with a reheat after each 10 to 15 percent reduction and each reheat is at a progressively lower temperature, a limited amount of actual cold work occurs.

Continued working in the reduction of the 1-inch- to the 1/2-inch-diameter bars greatly improved the homogeneity of the molybdenum; however, structure variables still persisted and it was evident that initial structure patterns were not completely eliminated.

1-inch-diameter bars. - Figure 21 shows typical as-swaged structures of the 1-inch-diameter bars. Because swaging imparts greater deformations near the surface than at the center, recrystallization probably occurred near the outer part of the bar during one or more of the reheats. Further swaging reduction instigated a breakdown of these reformed grains, whereas in the bar center area the deformation was accumulative.

In figure 21(a) is shown a 6-inch length of 1-inch-diameter bar prepared in 0.020-inch steps showing the structure from bar center to bar circumference. An outside layer of large grains surrounds a fine-grained center or core.

A transverse view (fig. 21(b)) shows the core to be noncircular because the bar was swaged from a rectangular ingot. The forging flow lines are evident and locate the corners of the original ingot shape.

An example of a strain gradient is shown along the line A-C of figure 21(b). During swaging no recrystallization occurred from A to B. The very large grains at B indicate critical strain with limited deformation after recrystallization. The fine grains near C were not core-type grains, for higher magnification indicated severe fragmentation after recrystallization.

Detail of the large-grained area is shown in photomicrograph 21(c). Adjacent grains that received similar amounts of deformation show considerable variance in strain markings, which probably originated from prior grain orientation.

The detailed core structure (fig. 21(d)) is the smallest, most uniform, and least fibrous as-swaged grain structure encountered in this investigation. This core structure is close to the "as-sintered" structure. The black circular spots are porosity, which varied between bars, always decreasing from bar center to bar circumference.

Exceptions were found where the core area did not have the uniform small-grained structure shown in figures 21 and 22. These were cores intermixed with both large and small grains or an extension of the larger grains into the normally small-grained area, as shown in figure 23. These structure deviations are due to manufacturing variables that were not investigated. A reasonable surmise for these structure variables is that the large molybdenum bars during swaging were not uniformly worked along their length or were not uniformly heated, which resulted in varying depths of recrystallization. The structures in the insets of figure 23(a) are very similar to figures 21(c) and 21(d), which seems to substantiate the belief that the typical and nontypical structures encountered are a matter of depth of recrystallization during fabrication. These core variables would influence the correlation of the properties obtained on this large bar size and also the structure of smaller-diameter bar sizes.

1/2-inch-diameter bars. - As-swaged structures of the 1/2-inch-diameter bars were of two general types, and neither contained a core area. One bar type (fig. 24) was small grained throughout, whereas the other bar type (fig. 25) was large grained throughout. Both types, however, were similar in that the larger grains were found near the bar surface. The bars of each lot, or shipment, received fell into one of these two general types, although there were variations and deviations in each type and within each lot. Variations of structures are believed to have originated in the original powder-particle size, or to have been introduced by the swaging variables, as previously discussed.

As-swaged structures are shown in detail in figures 26(a) and 26(b). As deformation increased, grain strain and fragmentation became greater although most of the grains did not lose their identity and grain boundaries were still apparent. Strain markings are very similar in both the small-grained (fig. 26(a)) and large-grained (fig. 26(b)) types with little evident porosity.

Recrystallization Studies

Preliminary recrystallization studies were limited to 1-hour heating periods at temperature. The temperatures were lowered and heating periods lengthened to a maximum of 74 hours. These extended times were used to simulate possible high-temperature service conditions for molybdenum when the mechanical properties would be adversely affected by recrystallization. Recrystallization was sluggish in this metal because the degrees of "effective" cold-work varied considerably.

1-inch-diameter bars. - The over-all uniformity and small grain size of a typical recrystallized core area of the 1-inch diameter bars is shown in figure 22. Structure details and recrystallization studies are summarized as follows:

(1) This grain structure (fig. 22(b)) was more equiaxed, slightly smaller, and appreciably more uniform than others encountered in these studies.

(2) Recrystallization temperatures of the core could be more closely defined than the material in bars of smaller diameter because of more uniform and constant recrystallization characteristics.

(3) Partial recrystallization in varying amounts was attained at a temperature of 2800° F in 1 hour. Total recrystallization was achieved within 24 hours at 2800° F. At 2700° F for periods of 72 hours, total recrystallization was obtained in only a few samples.

Recrystallization characteristics of the large grains within the general core area are shown in figure 23.

Figure 23(b) is the other half of the as-swaged specimen shown in figure 23(a) after being heated 24 hours at 2800° F. Both specimens were mounted together to assure identical polishing and etching treatments. The effect of heating is shown by comparison of the microstructures in the left insets. Some of the severe strain markings so prominent in the as-swaged grains have almost disappeared.

The absence or near absence of these markings indicated that either recrystallization or stress relief had taken place. Recrystallization is always evident on microscopic examination by grain reformation; however a comparison of the X13 macrographs and the insets indicates no such reformation. In a further attempt to classify these large heated grains, it was felt that a microhardness survey and a comparison with hardnesses of known recrystallized grains would give further evidence as to their nature. The following microhardness results when compared to the recrystallized grain hardness indicate that no recrystallization had occurred, and that the left inset photomicrographs do show some degree of recovery or stress relief:

	Knoop hardness number 2 kg load
(a) Severly strained as-swaged grains	220
(b) Smooth as-swaged grains	209
(c) Grains (a) after 24 hours at 2800° F	212
(d) Grains (b) after 24 hours at 2800° F	213
(e) Recrystallized grains	184

1/2-inch-diameter bars. - A rather complete recrystallization study of the 1/2-inch-diameter bars is presented in figures 24 and 25. Recrystallization consistently began in the bar center and progressed to the surface. Figures 24 and 25 therefore contain photomicrographs from both the bar edge and the center. This recrystallization behavior is attributed to the difference in the amount of effective strain hardening during the swaging operation.

Figures 24(b) and 25(b) show the difference in time periods required to induce approximately equivalent amounts of initial recrystallization. Complete recrystallization except at the edges (figs. 24(c) and 25(c)) was obtained after heating 20 hours at 2000° F for the small-grained type, whereas a temperature of 2200° F for 20 hours was required for the large-grained type. Although the percentage reduction was the same for both types of structure, the larger as-swaged grain size had a 200° F higher recrystallization temperature, for the same time period, than the small-grained type.

Grain growth, in this material, was not observed even at temperatures 300° to 400° F higher than those used for the previous recrystallization studies. Comparison of figures 24(c) with 24(d) and 25(c) with 25(d) shows no detectable grain growth.

Figures 26(c), 26(d), and 26(e) are supplementary to figures 24 and 25 and show added structure details. Recrystallization seemed to begin in the most severly fragmented areas. The two recrystallized grains in figure 26(c) appear to have started in such an area.

The recrystallized structures of the small- and large-grained types (figs. 26(d) and 26(e)) show how the recrystallized grain sizes are closely similar to, and also retain some of the directionalism of, the as-swaged grain sizes (figs. 26(a) and 26(b)).

Often a small but varying amount of these similar fragmented areas did not recrystallize at the temperatures and times used. Because such areas (fig. 26(d)) appear to have been severly fragmented, it would seem that they should show early reformation characteristics. Much higher temperatures and soaking times, however, were necessary to recrystallize these areas.

It is suspected that the small, deeply etched areas in figure 26(d) result from impurities, such as oxides, surrounding the molybdenum grains which did not recrystallize.

Summary of recrystallization determinations. - The following table is a summary of all the recrystallization temperature-time determinations of all the material evaluated in this report:

Source	Bar diameter (in.)	Grain type	Recrystallization temperature (°F)[a]		
			Time, hr		
			1	20	75
A	1/4		----	----	1800
A	1/2	Small	2150	2000	----
		Large	2300	2200	1900
A	0.698		2500	----	----
A	1		2800-2850	2775-2800	2700-2750
B	1/4		2350	2200	----
B	1/2	Small	2150	2000	----
		Large and small mixed	2250	2150	----

[a] ±50° F.

Microhardness Studies

Microhardness readings were made on a Tukon tester using the Knoop indenter with an applied load of 2 kilograms. The size of the pyramidal indentations can be obtained by referring to figures 24(a) and 25(a). Hardness results are summarized as follows:

Bar diameter (in.)	Bar condition	Average K.H.N./ 2 kg
1	As-swaged, large outside grains	[a]212
1	As-swaged, core area	[a]219
1/2	As-swaged, large-grained type	[b]222
1/2	As-swaged, small-grained type	[b]222
1	Recrystallized, core area	184
1/2	Recrystallized, large-grained	[c]170
1/2	Recrystallized, small-grained	180

[a]Average of hardness values taken at 0.050-in. intervals across center step of fig. 21(a).

[b]Average of hardness values taken at 0.036-in. intervals across bars of figs. 24(a) and 25(a).

[c]True grain hardness because indentations did not cross any grain boundaries.

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TABLE I - SHORT-TIME TENSILE STRENGTH OF SINTERED WROUGHT MOLYBDENUM BARS

Test temperature (°F)	Ultimate tensile strength (lb/sq in.)	Test-section length (in.)	Elongation in test-section length (percent)	Test-section diameter (in.)	Type of fracture (a)	Remarks
1/4-in. diameter; source A.						
Room	89,550	0.750	26.3	0.1254	6	
1800	32,270		18.1	.1249	-	
2000	28,940		17.2	.1250	1	
2000	30,400		22.4	.1253	1	
2200	20,080		29.1	.1248	3	
2200	20,400		34.2	.1250	3	
2200	21,770		35.3	.1255	1	
2400	18,460		35.3	.1255	3	
2400	19,180		20.8	.1248	3	
2400	19,260		28.2	.1246	3	
1/2-in. diameter; source A.						
Room	83,450	1.500	0.4	0.2500	5	
Room	89,640		1.2	.2504	5	
Room	62,900		0	.2509	5	(b)
Room	70,550		0	.2508	5	(b)
Room	86,900		.3	.2508	5	(c)
1800	33,250		26.6	.2515	1	
2000	29,460		28.6	.2506	1	
2000	28,170		27.5	.2502	1	
2000	24,920		38.4	.2507	1 and 4	(b)
2000	28,680		25.3	.2497	4	(c)
2200	20,140		42.7	.2500	4	
2200	17,220		21.4	.2500	3	
2400	14,760		8.4	.2502	4	
2400	15,280		7.3	.2503	2	(d)
0.698-in. diameter; source A.						
Room	90,170	0.750	2.1	0.1246	5	
Room	90,550	.750	.8	.1255	5	
1800	32,760	.750	21.1	.1252	1	
2000	26,690	.750	16.9	.1258	1	
2000	27,900	.750	19.8	.1254	1	
2200	25,640	.750	13.3	.1254	1	
2200	26,860	.750	14.6	.1255	1	
2400	25,800	.750	11.1	.1254	1	
2400	26,100	.750	7.2	.1249	1	
Room	89,750	1.500	5.6	.2472	5	
1500	40,740	1.500	16.1	.2450	1	
1800	33,640	1.500	23.7	.2454	1	
2000	29,170	1.500	23.2	.2468	1	
2200	26,100	1.500	17.7	.2466	1	
2200	28,480	1.500	20.4	.2470	1	
2400	23,720	1.500	16.9	.2486	1	
2400	23,960	1.500	19.3	.2470	1	
Room	87,680	1.625	1.3	.3658	5	
1800	32,500	1.625	29.9	.3754	1	
1800	30,260	1.625	34.7	.3703	1	(e)
1800	30,500	1.625	29.3	.3693	1	(f)
2000	29,700	1.625	27.4	.3760	1	
2000	27,310	1.625	21.3	.3730	1	(g)
2200	25,840	1.625	21.5	.3680	1	
2400	24,100	1.625	26.7	.3704	1	
2400	23,680	1.625	23.1	.3730	1	(g)
2600	18,520	1.625	19.3	.3690	1	(g)
2000	29,490	1.250	44.8	.4990	1	(g)(h)
2400	27,330	1.250	41.2	.4997	1	(g)(i)
2600	17,380	1.250	37.1	.4970	1	(g)(j)
1-in. diameter; source A.						
Room	70,820	1.250	1.3	0.4976	5	
Room	75,180		1.1	.5050	5	
Room	31,370		0	.5037	5	(k)
1800	25,310		61.9	.5032	1	
1800	22,270		40.0	.5044	4	(k)
2000	23,850		----	.5013	1	
2200	21,310		48.6	.5041	1 and 3	
2200	23,500		21.2	.5044	2	(m)
2400	19,940		35.1	.5040	3	
2400	20,880		31.7	.5044	3	
1/4-in. diameter; source B.						
Room	92,300	0.750	16.9	0.1249	6	
Room	92,770		17.6	.1247	6	
Room	94,450		24.6	.1247	6	
1500	33,280		21.9	.1245	1	
1800	28,530		17.7	.1250	1	
1800	28,880		20.7	.1255	1	
2000	25,740		22.1	.1246	1	
2200	22,040		25.4	.1248	1	
2400	18,030		18.2	.1248	3	
2400	18,030		35.3	.1245	3	
1/2-in. diameter; source B.						
Room	86,040	1.500	6.3	0.2498	5	
Room	89,700		15.1	.2497	6	
Room	36,010		.5	.2500	5	(n)
Room	40,080		.5	.2510	5	(n)
1500	38,320		19.3	.2504	1	
1500	28,560		27.8	.2490	2	(n)
1800	31,950		19.7	.2505	1	
1800	33,670		17.5	.2503	1	
1800	26,070		29.0	.2510	2	(n)
2000	29,600		16.1	.2509	1	
2000	28,960		17.3	.2495	1	
2000	22,030		19.8	.2492	2	(n)
2200	22,700		----	.2500	3	
2200	17,810		8.4	.2503	2	(n)
2400	16,280		----	.2503	3	
2400	15,510		40.7	.2502	3	
2400	13,070		4.1	.2510	2	(n)

[a] For explanation of numbers, see "Types of Fracture" in DISCUSSION OF RESULTS.
[b] Heated 75 hours at 1900° F (partly recrystallized).
[c] Heated 75 hours at 1800° F (no recrystallization).
[d] Recrystallized before testing.
[e] Heated 48 hours at 1800° F before testing (no recrystallization).
[f] Heated 48 hours at 2200° F before testing (no recrystallization).
[g] Brought up to temperature in 5 minutes by induction heating (loss of metal by oxidation was estimated).
[h] 0.001 in. of diameter subtracted because of oxidation.
[i] 0.0015 in. of diameter subtracted because of oxidation.
[j] 0.002 in. of diameter subtracted because of oxidation.
[k] Heated 20 hr at 2800° F (recrystallized).
[m] 45° shear fracture.
[n] Heated 48 hr at 2400° F (recrystallized).

TABLE II - STRESS-RUPTURE STRENGTH AT 1800° F OF 1/2-INCH-DIAMETER SINTERED WROUGHT MOLYBDENUM BARS

Rupture stress (lb/sq in.)	Rupture life (hr)	Test-section diameter (in.)	Fine grain in test section (percent)	Remarks
Lot number 2				
20,000	46.0	0.2440		
20,000	23.2	.2484		
20,000	8.6	.2518		
19,500	85.8	.2512		
19,500	44.5	.2510		
19,500	42.2	.2510		
19,500	115.7	.2503		
19,000	267.1	.2488		
19,000	141.4	.2516		
19,000	36.7	.2512		
20,000	1.5	.2507		Recrystallized (1 hr at 2500° F in argon)
20,000	.8	.2495		Recrystallized (1 hr at 2500° F in helium)
19,000	.2	.2512		Recrystallized (1 hr at 2300° F in hydrogen)
15,000	2.5	.2506		Recrystallized (1 hr at 2300° F in hydrogen)
10,000	98.7	.2512		Recrystallized (1 hr at 2300° F in hydrogen)
Lot number 3				
21,000	56.2	0.2503	0	
21,000	45.2	.2503	90	
20,000	151.9	.2514	30	
20,000	140.7	.2500	100	Test stopped; no fracture
20,000	90.4	.2510	80	
20,000	78.8	.2506	0	
20,000	64.3	.2505	40	
20,000	60.6	.2504	80	
20,000	56.7	.2505	50	
19,500	208.5	.2508	0	Test stopped; no fracture

TABLE III - SHORT-TIME TENSILE STRENGTH OF 1/4-INCH SINTERED ROLLED MOLYBDENUM PLATE

Direction	Test temperature (°F)	Ultimate tensile strength (lb/sq in.)	Test-section length (in.)	Elongation in test-section length (percent)	Test-section diameter (in.)
Longitudinal direction of rolling	Room	88,730	0.750	1.1	0.1246
	Room	85,950		3.6	.1251
	1800	35,190		30.1	.1250
	2000	33,780		28.5	.1247
	2000	30,990		17.7	.1245
	2200	29,270		19.4	.1254
	2200	28,200		16.3	.1247
	2400	22,350		9.6	.1253
	2400	22,170		11.4	.1255
Transverse to direction of rolling	Room	96,300	0.750	1.0	0.1246
	Room	99,500		2.7	.1202
	1800	40,650		14.5	.1246
	2000	34,960		11.8	.1250
	2000	35,040		15.0	.1251
	2200	27,290		9.2	.1246
	2200	26,720		14.5	.1244
	2400	24,310		----	.1249

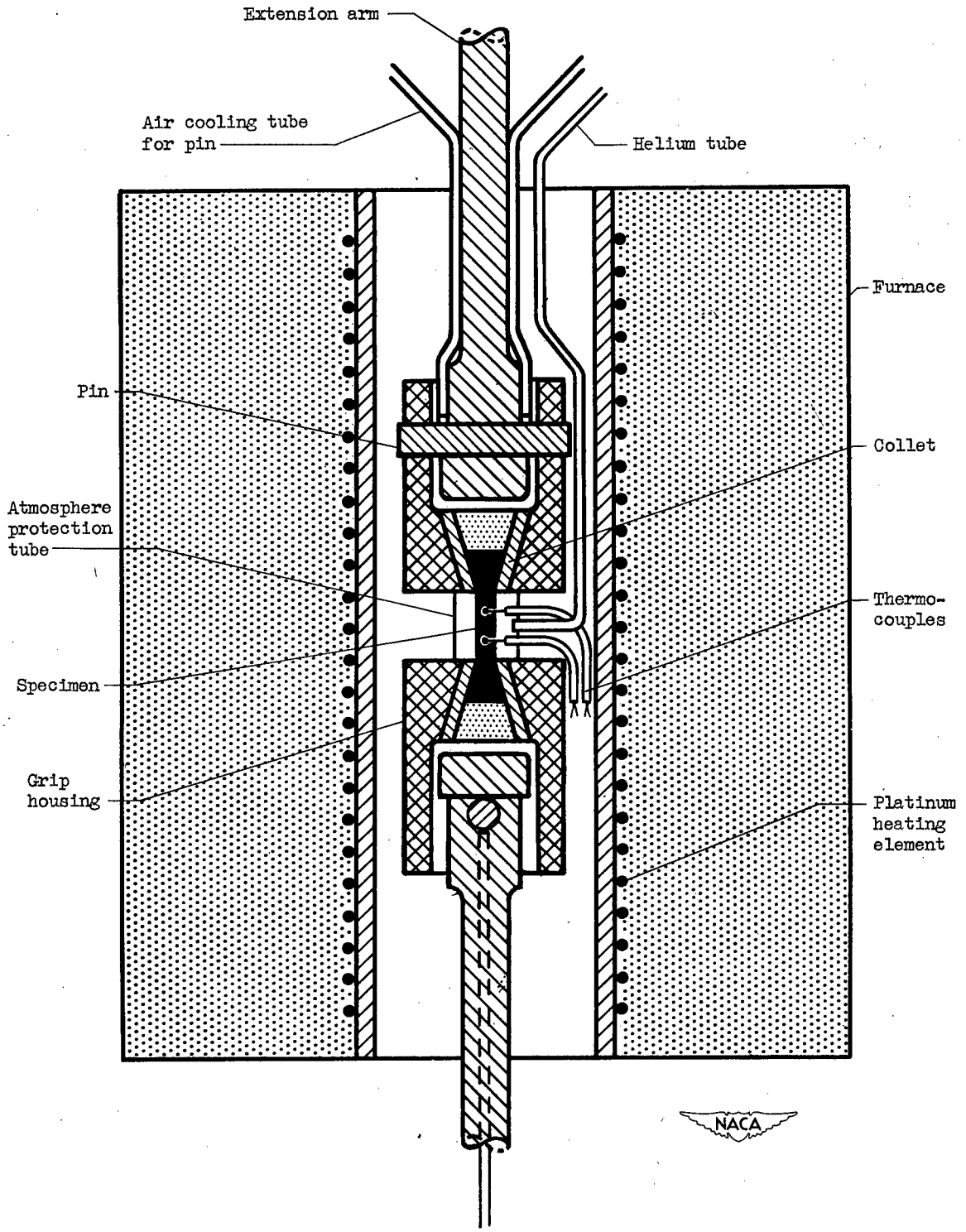
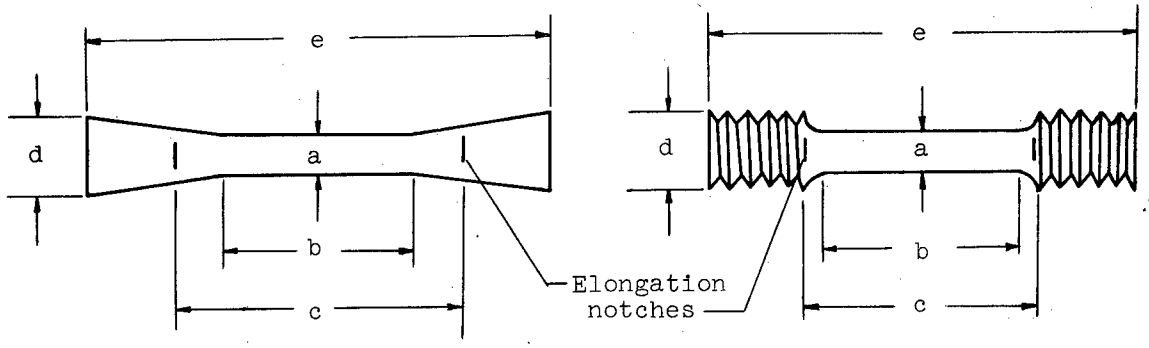
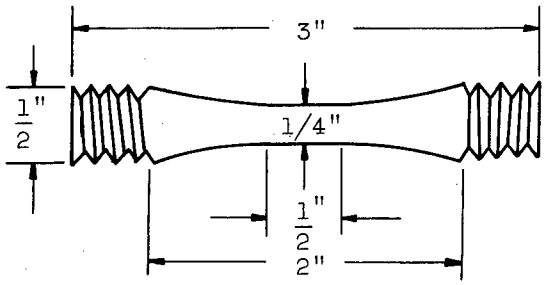


Figure 1. - Apparatus for high-temperature tensile tests.

Type	a (in.)	b (in.)	c (in.)	d (in.)	e (in.)
Threaded	0.125	0.750	1.000	0.250	1.750
Tapered	.250	1.500	1.875	.500	3.000
Tapered	.375	1.625	2.250	.687	3.500
Tapered	.500	1.250	2.000	1.000	4.250



(a) Short-time tensile specimens.



(b) Stress-rupture specimen.

Figure 2. - Short-time tensile and stress-rupture specimens.

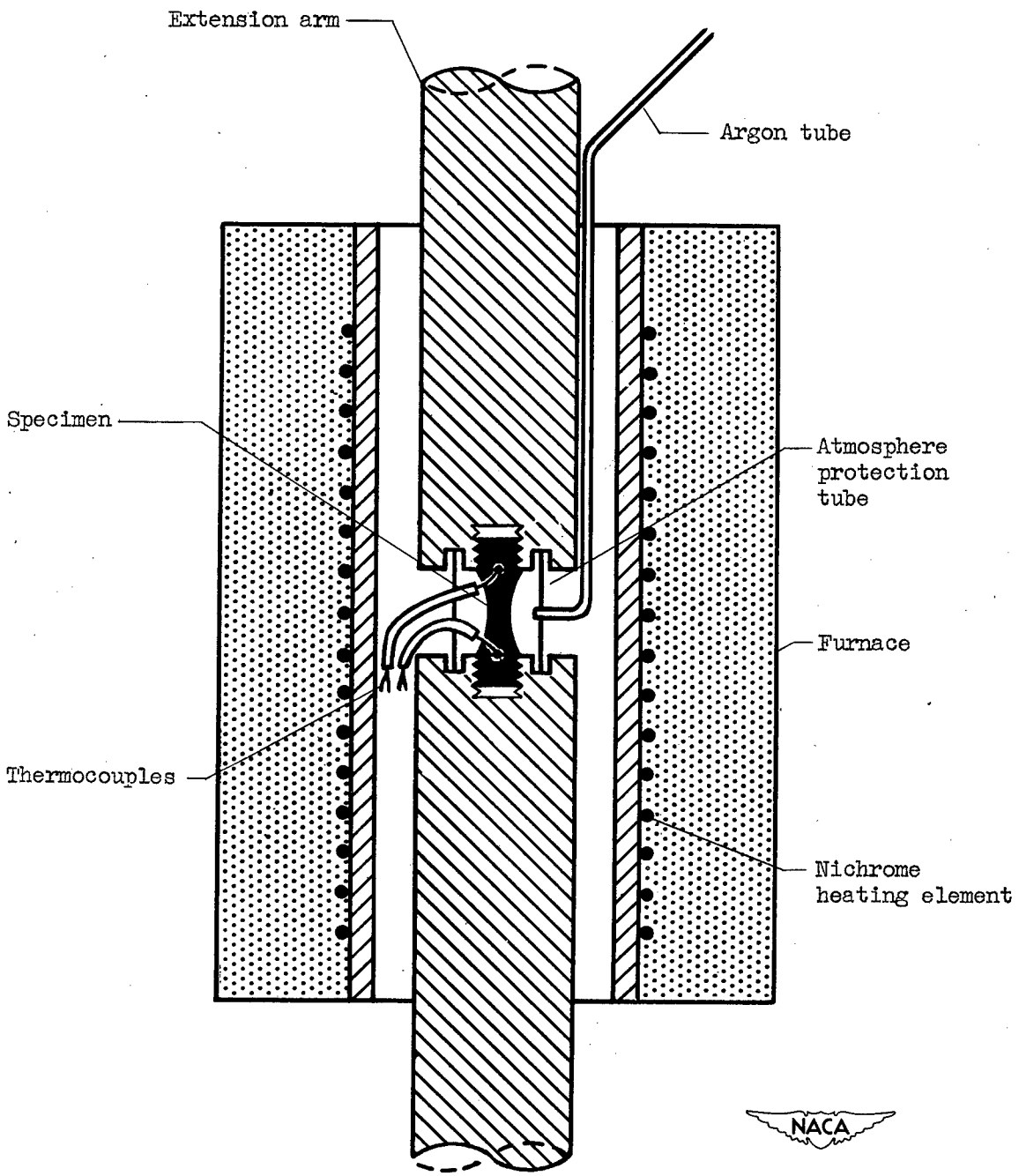


Figure 3. - Apparatus for high-temperature stress-rupture testing.

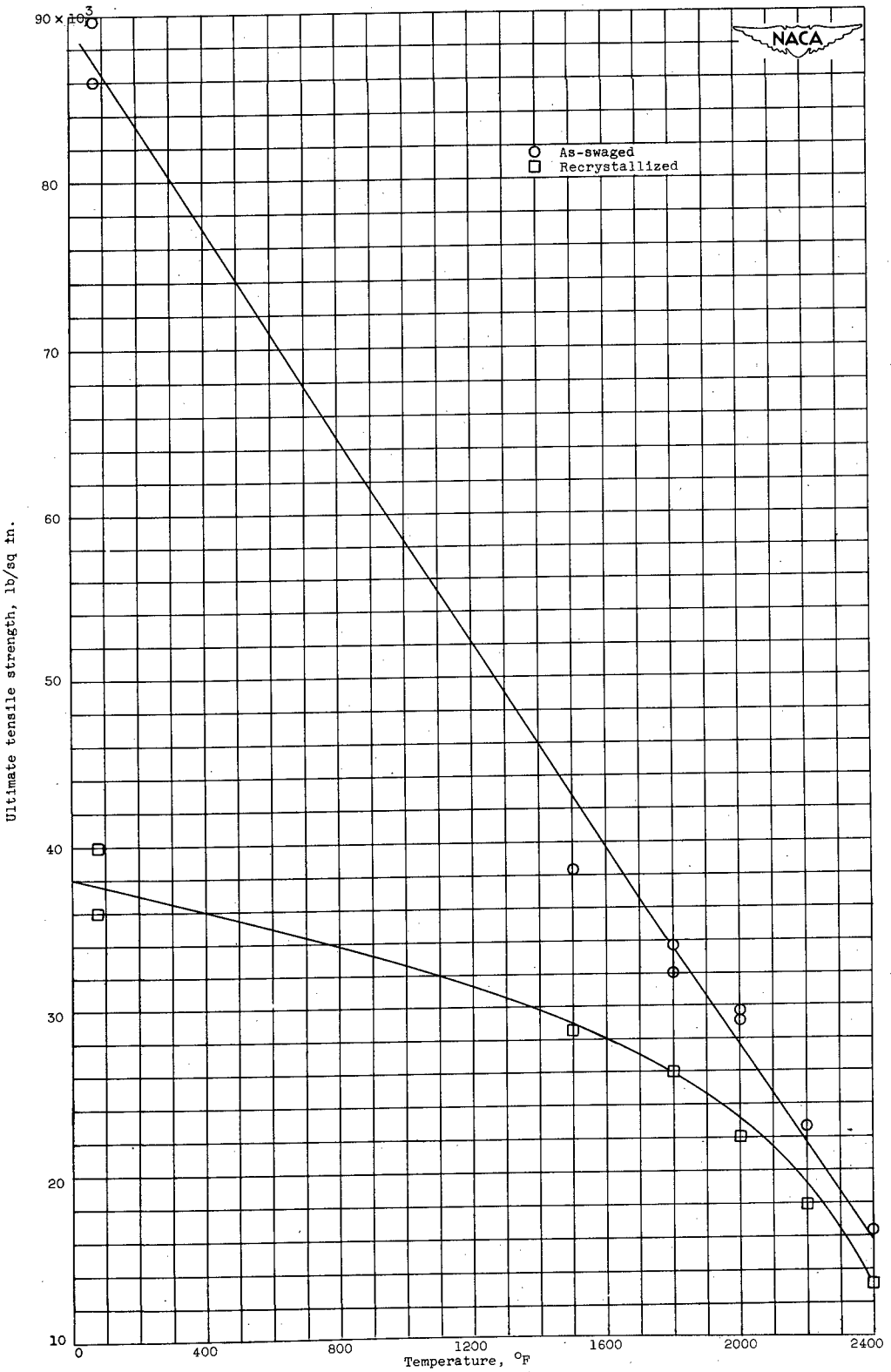


Figure 4. - Effect of recrystallization on ultimate tensile strength of 1/2-inch-diameter molybdenum bar (source B).

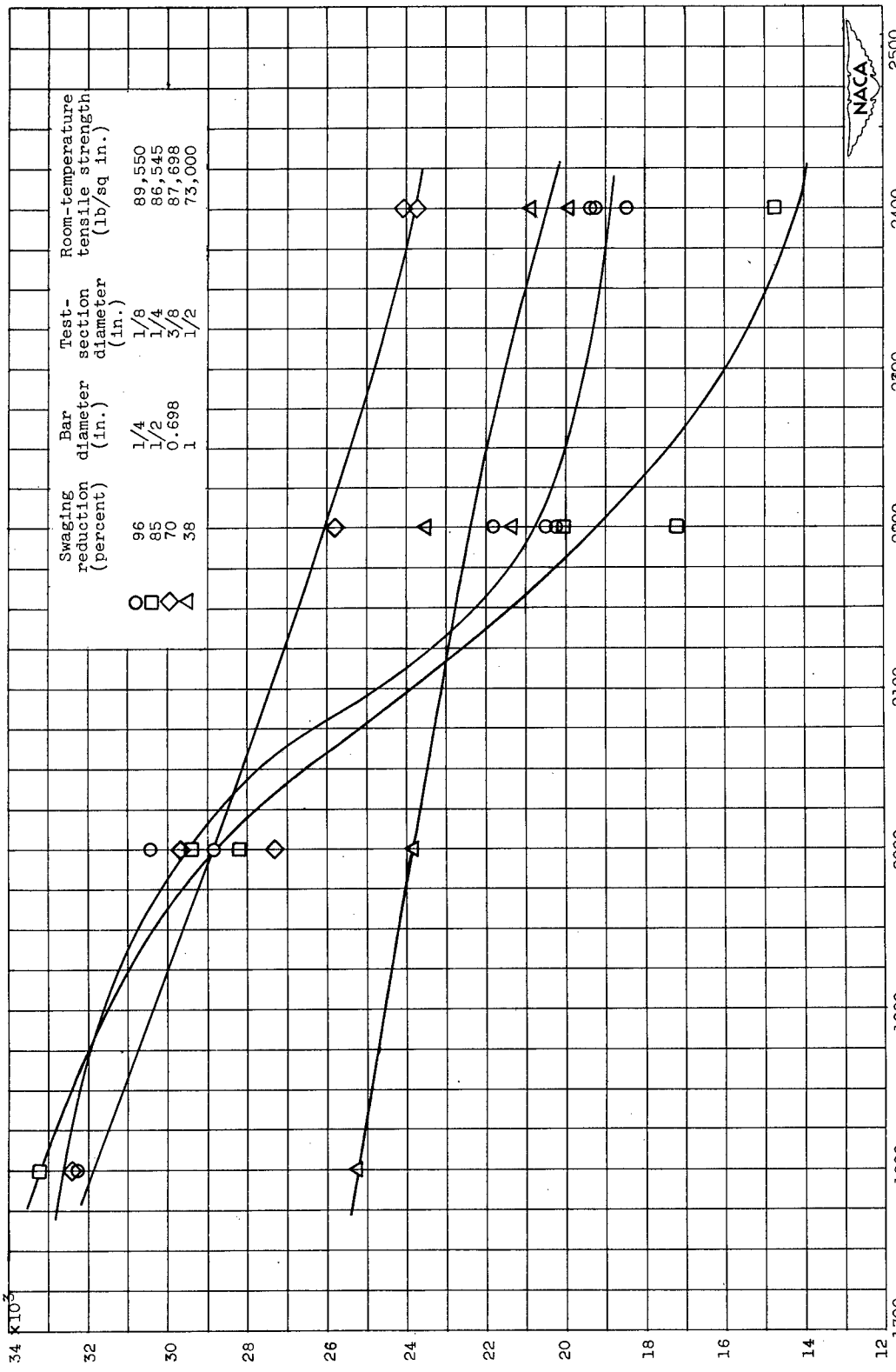


Figure 5. - Effect of swaging on ultimate tensile strength of molybdenum.

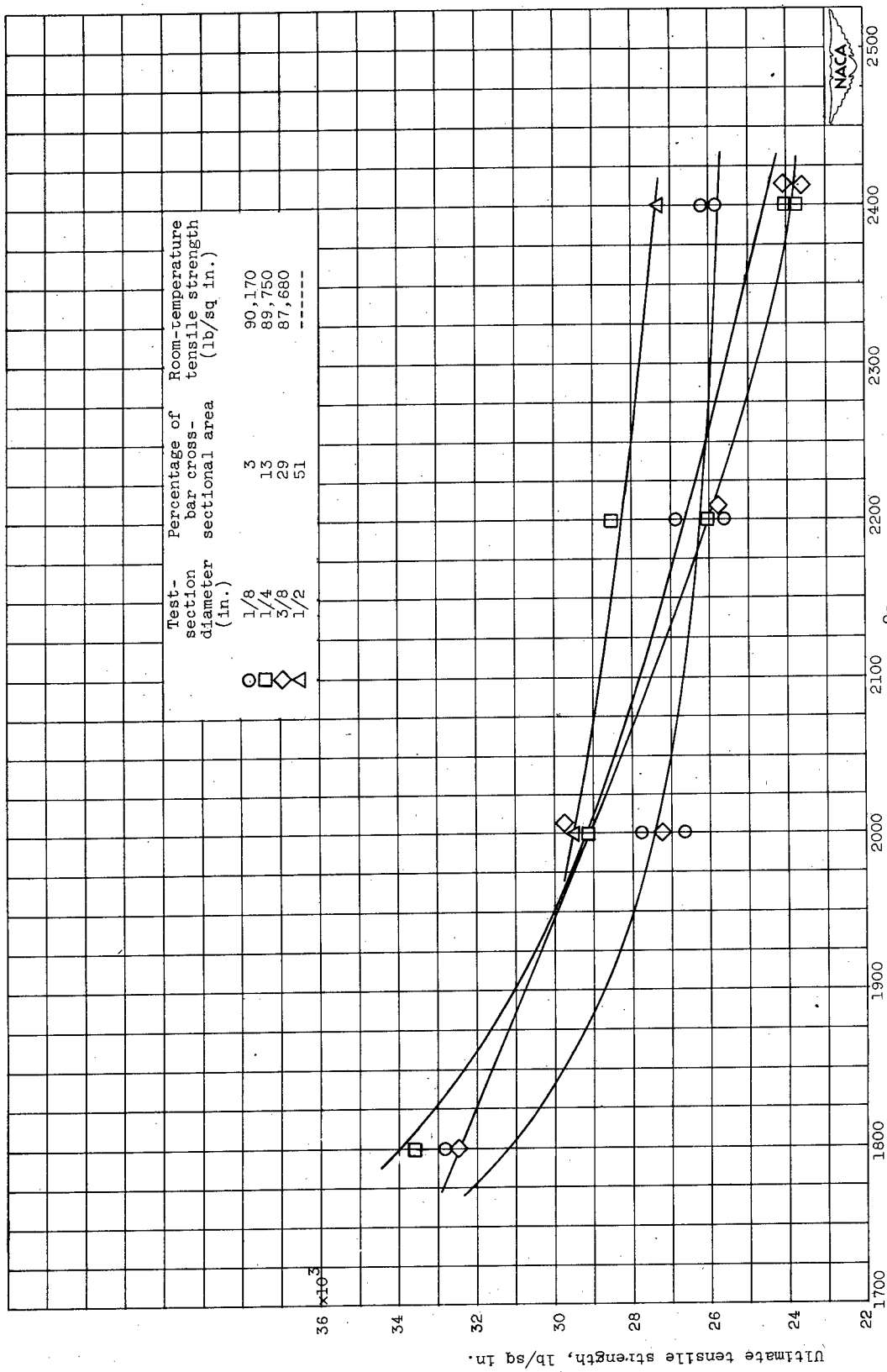


Figure 6. - Effect of tensile test-section area on evaluation of ultimate tensile strength of molybdenum, 0.698-inch bar.

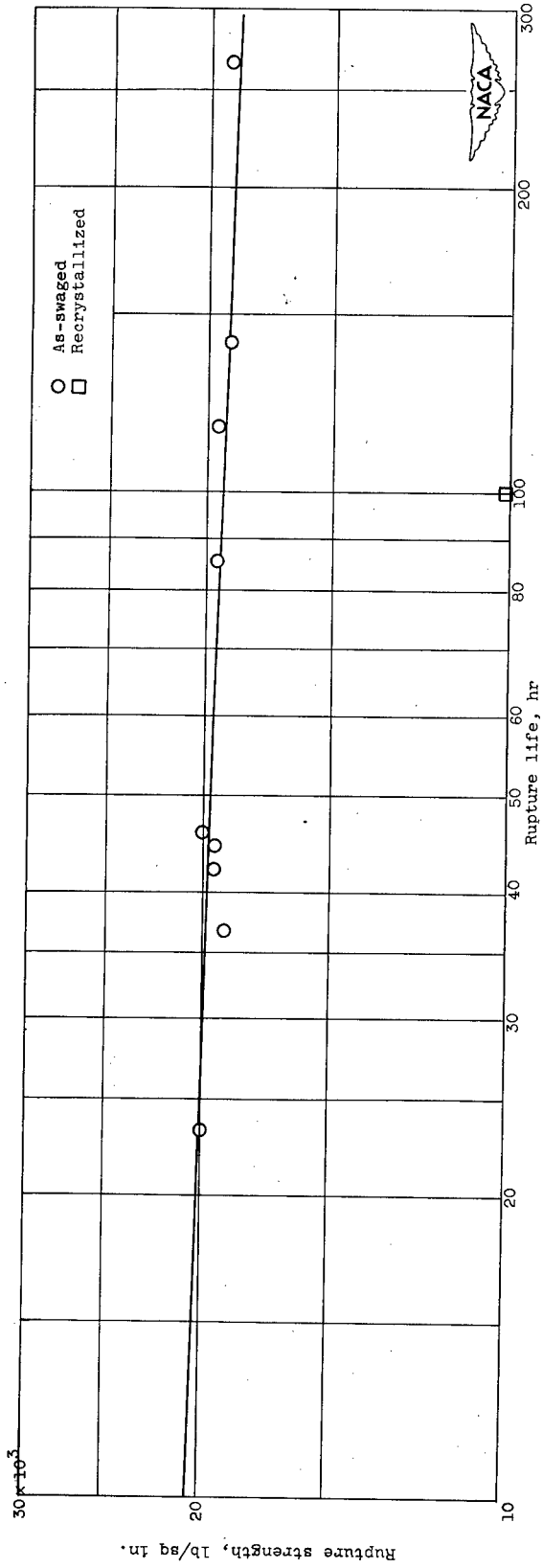


Figure 7. - Stress-rupture life of 1/2-inch-diameter wrought molybdenum bars at 1800° F.

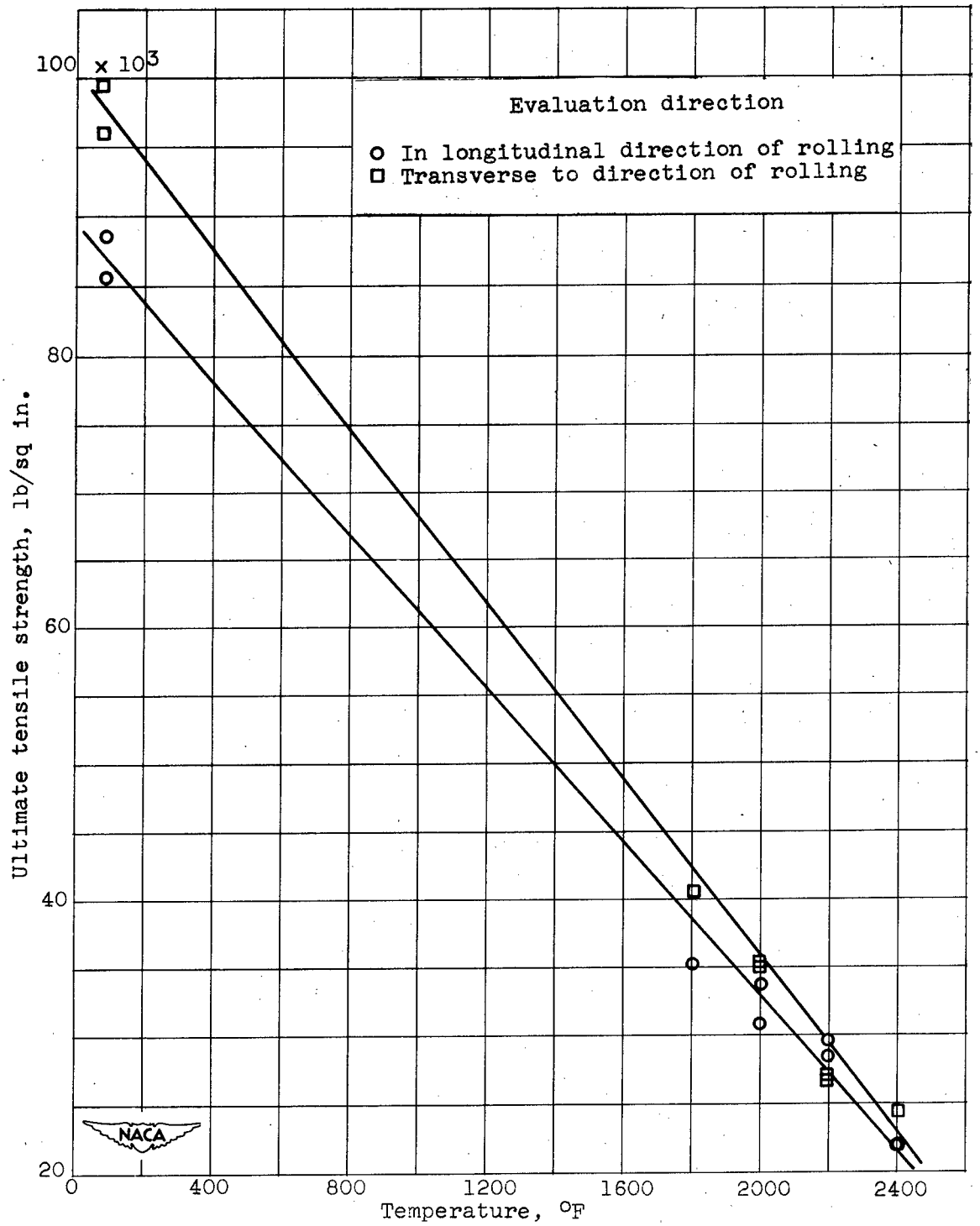
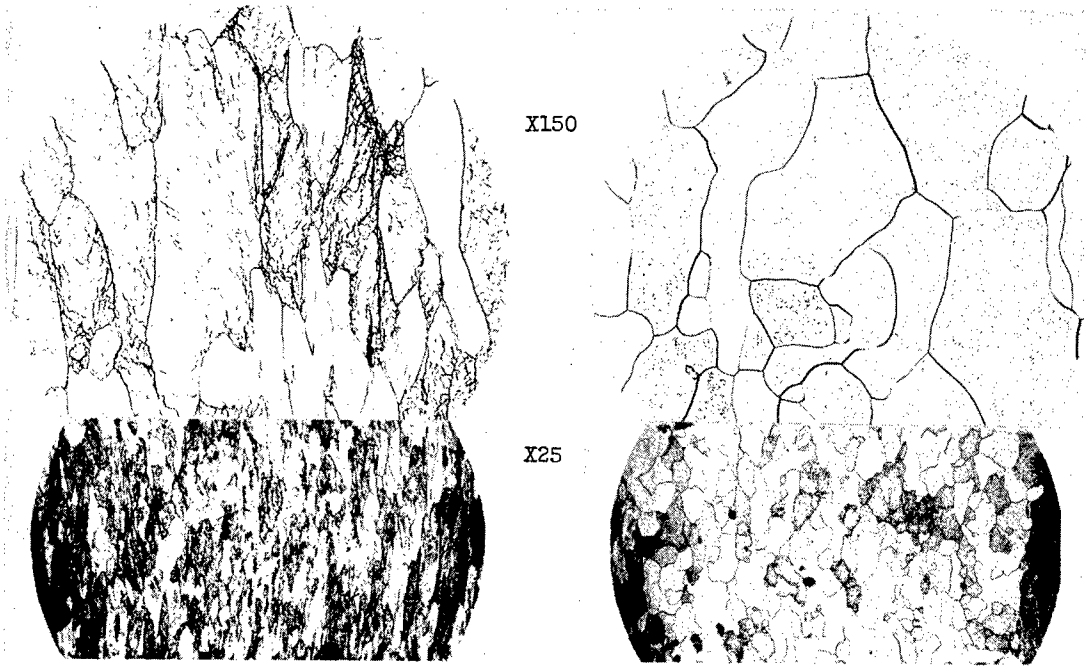
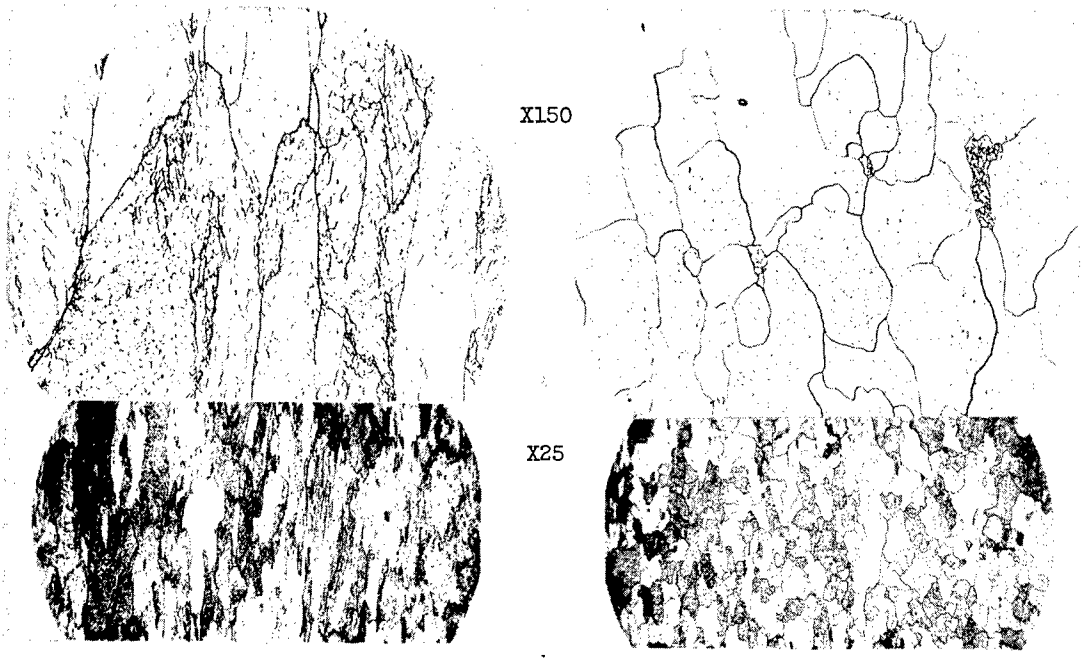


Figure 8. - Directional properties of 1/4-inch rolled molybdenum plate.



(a) 1/4-inch-diameter bar; fractured at 1800° F.

(b) 1/4-inch-diameter bar; fractured at 2200° F.



(c) 1/2-inch-diameter bar; fractured at 1800° F.

(d) 1/2-inch-diameter bar; fractured at 2200° F.

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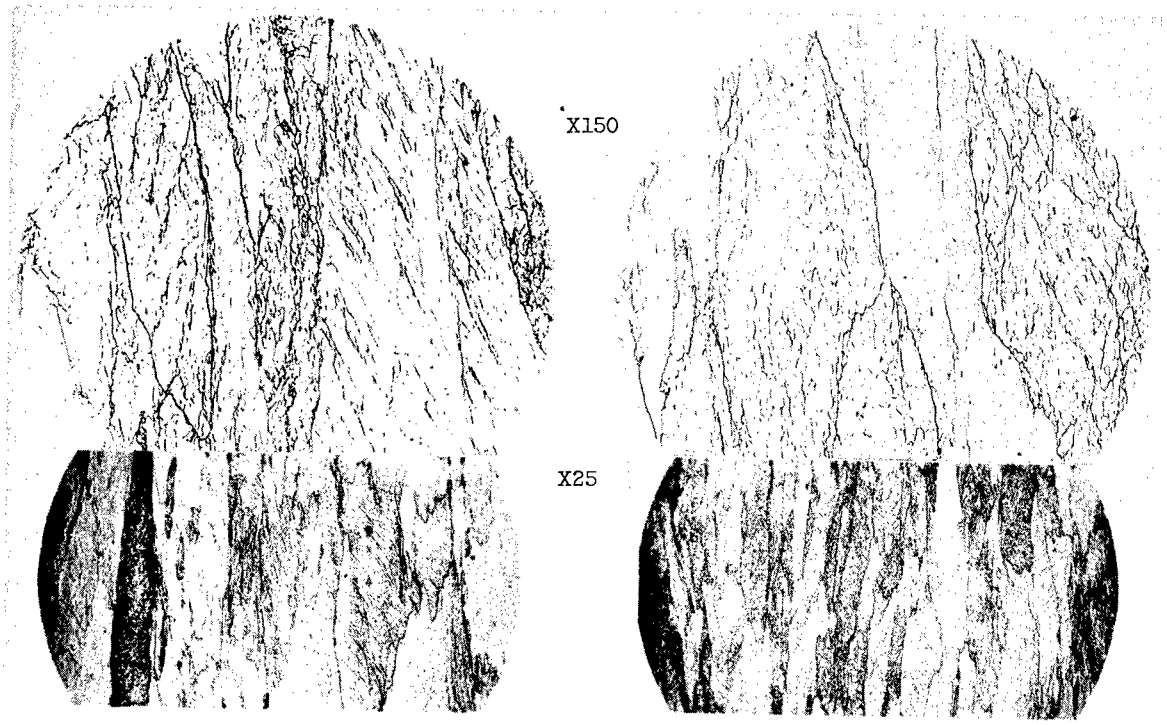
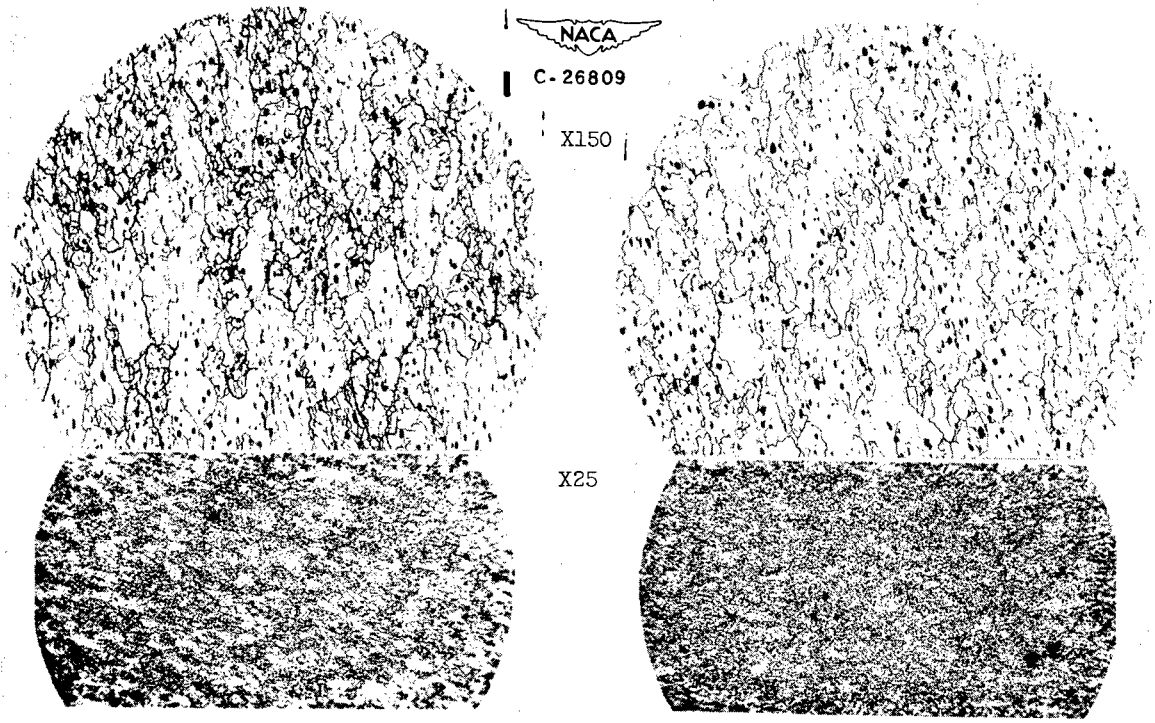
Figure 9. - Effects of swaging as shown by longitudinal views remote from fracture area through center axis of molybdenum tensile specimens. X150 and X25 (reduced 15 percent).



(e) 0.698-inch-diameter bar; fractured at 1800° F.

(f) 0.698-inch-diameter bar; fractured at 2200° F.

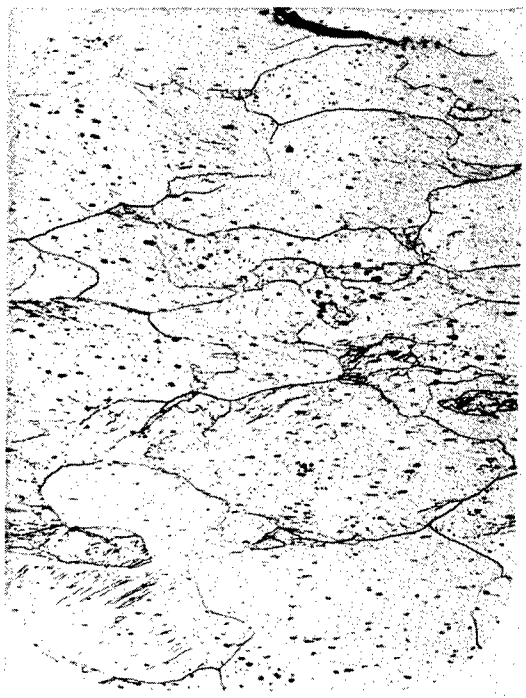


(g) 1-inch-diameter bar; fractured at 1800° F.

(h) 1-inch-diameter bar; fractured at 2200° F.

Figure 9. - Concluded. Effects of swaging as shown by longitudinal views remote from fracture area through center axis of molybdenum tensile specimens. X150 and X25 (reduced 15 percent).

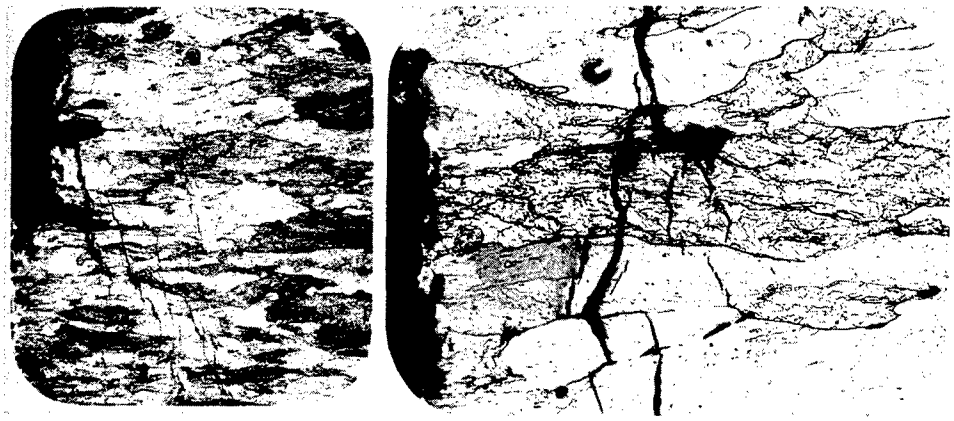
(a) As-swaged; small grain.



(b) As-swaged; large grain.

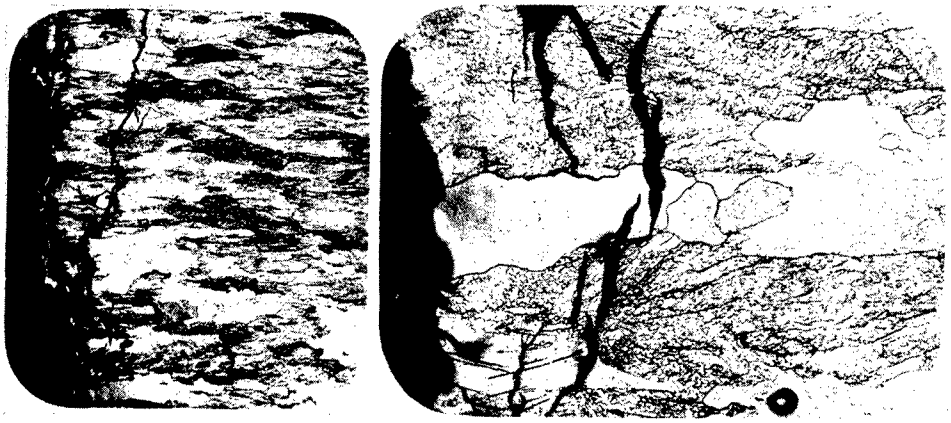
(c) Recrystallized; intergranular failure.

Figure 10. - As-swaged and recrystallized molybdenum as shown by longitudinal views of tensile specimens from source B fractured at room temperature. 1/2-inch-diameter bars. X150 (reduced 15 percent).



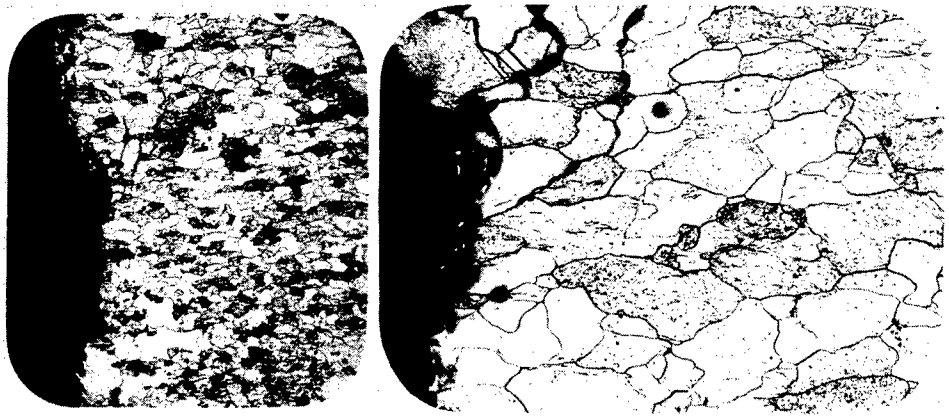
X25 X150

(a) As swaged; no recrystallization.



X25 X150

(b) Heated 75 hours at 1800° F; recrystallization beginning.



X25 X150

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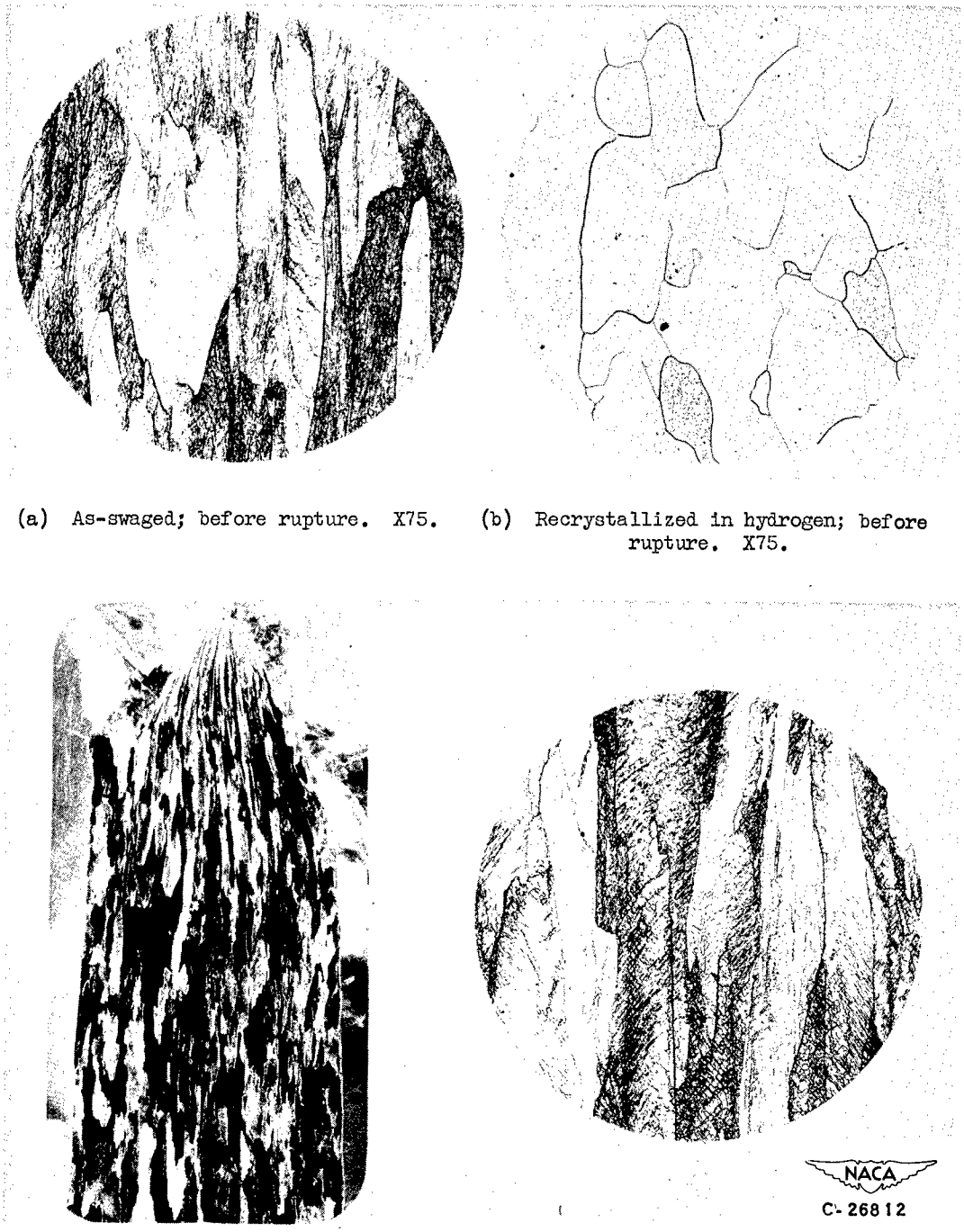
(c) Heated 75 hours at 1900° F; recrystallization almost complete.

Figure 11. - Effects of recrystallization as shown by longitudinal views of molybdenum tensile specimens fractured at room temperature. 1/2-inch-diameter bars. X25 and X150 (reduced 15 percent).



(a) As-swaged; before rupture. X75.

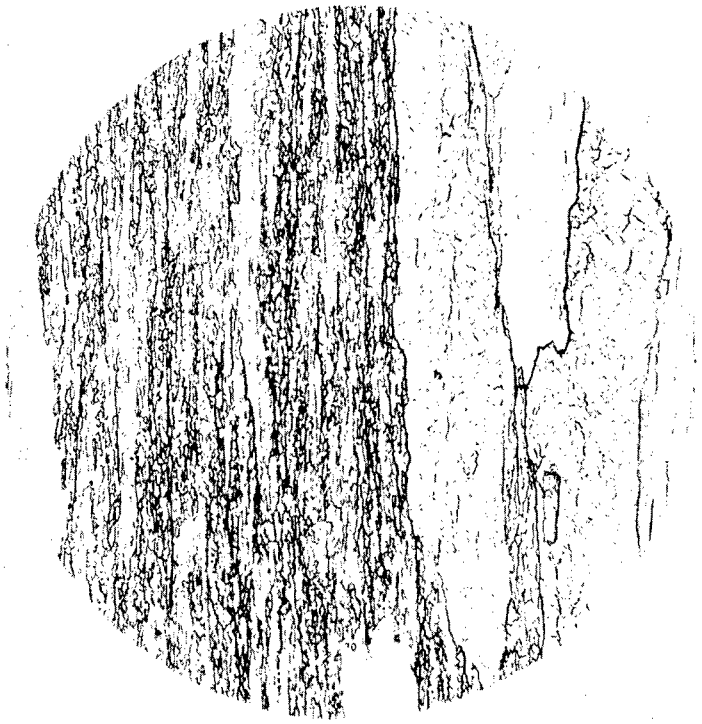
(b) Recrystallized in hydrogen; before rupture. X75.

(c) Recrystallized specimen ruptured at 1800° F and 15,000 pounds per square inch; $2\frac{1}{2}$-hour life. X8.

(d) Recrystallized ruptured specimen; view remote from fracture. X75.

Figure 12. - Structure of molybdenum stress-rupture specimens before and after testing as shown by longitudinal views. X75 and X8 (reduced 15 percent).

(a) Ruptured at 1800° F; 20,000 pounds per square inch. X8.



(b) Ruptured specimen; view remote from fracture. X75.

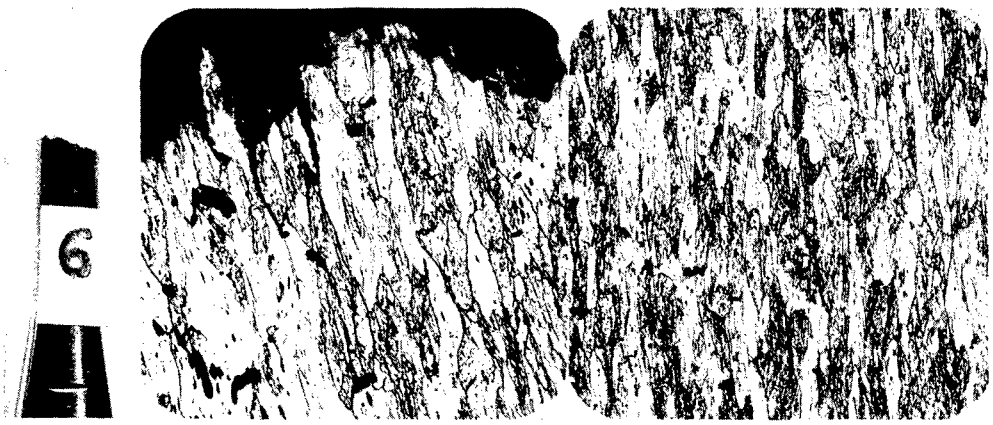
Figure 13. - Longitudinal views of heterogeneous stress-rupture specimen. X8 and X75 (reduced 15 percent).

X150 X100

(d) Type 4; as-swaged; fractured at 2200° F.

X150 X100

(e) Type 5; heated 48 hours at 2400° F; fractured at room temperature.

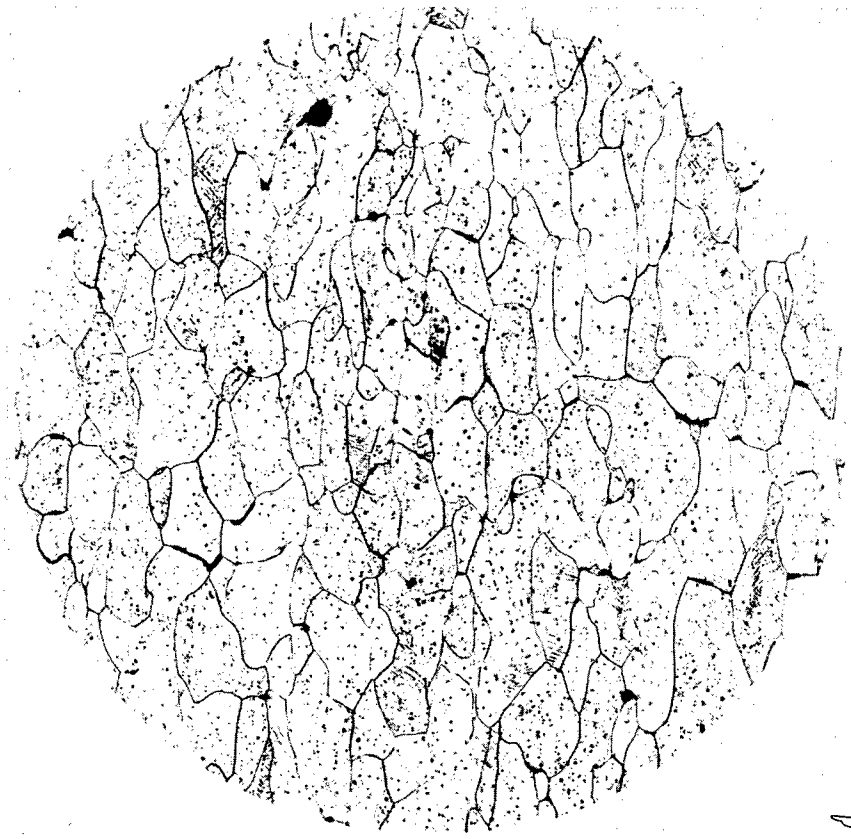


X150 X100 NACA

(f) Type 6; as-swaged; fractured at room temperature. C-26815

Figure 14. - Concluded. Types of fracture as shown by longitudinal views at fracture (left) and remote from fracture (right) of molybdenum tensile specimens. X150 and X100 (reduced 15 percent).

(a) At fracture. X150.



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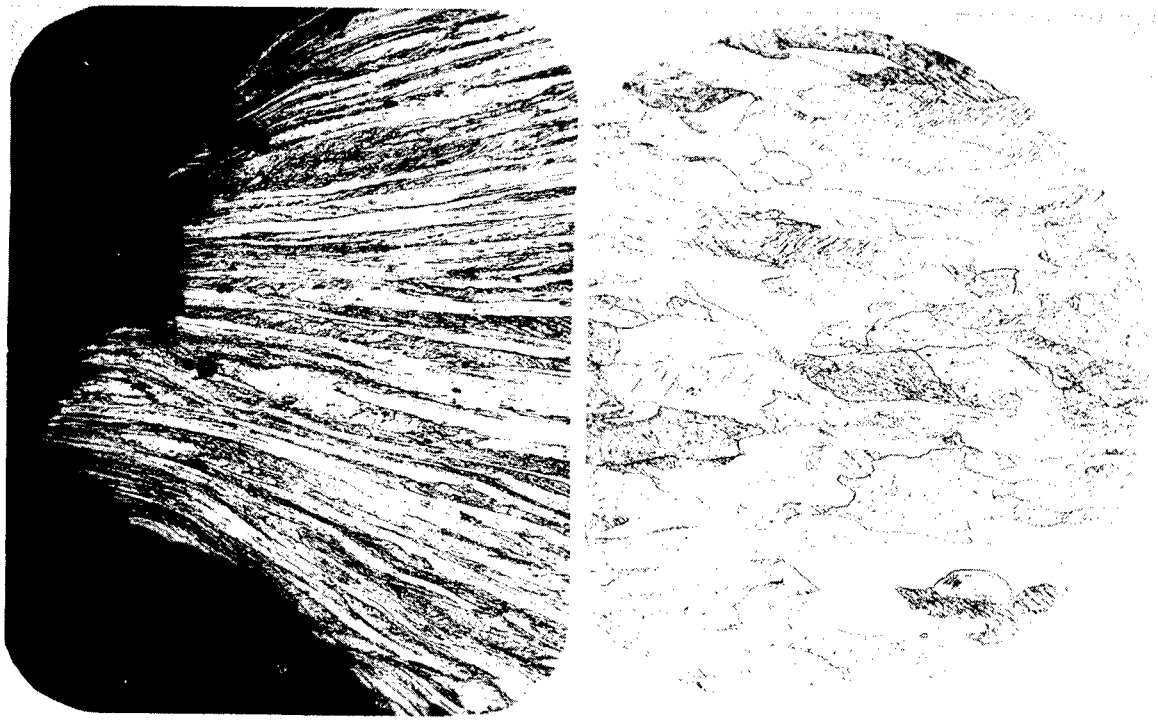
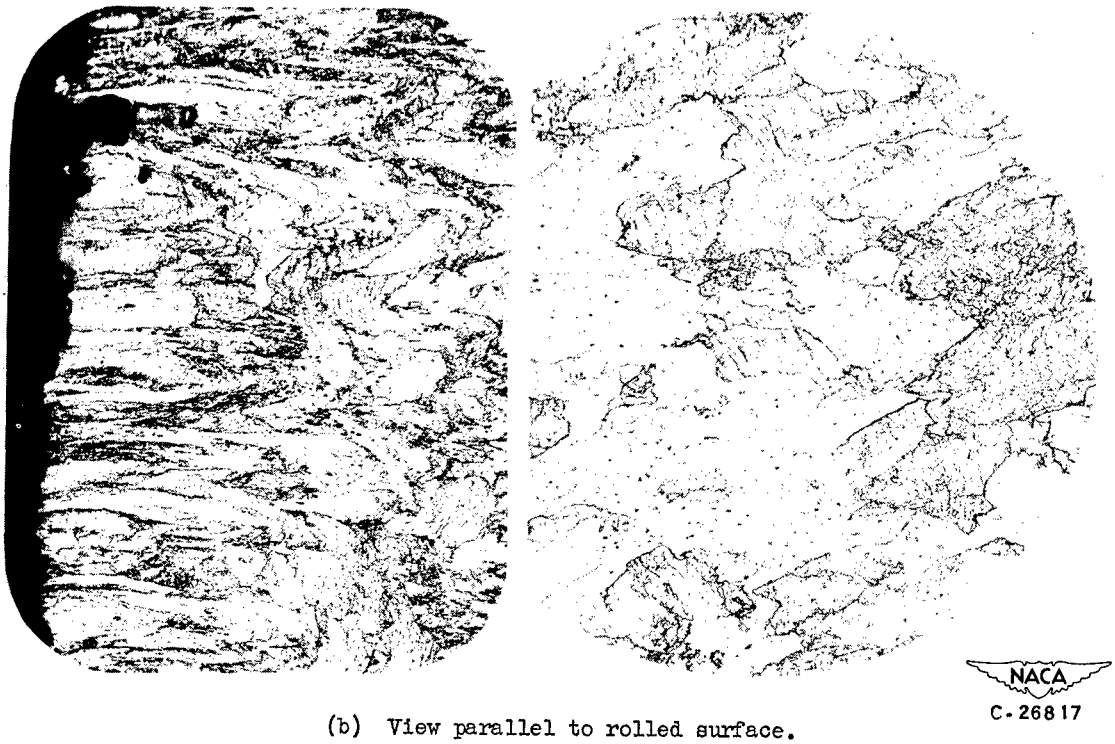
(b) Remote from fracture. X100.

Figure 15. - Ductile, transgranular fracture of recrystallized molybdenum as shown by longitudinal views of tensile specimen. Heated 24 hours at 2400° F; fractured at 1800° F. X150 and X100 (reduced 15 percent).

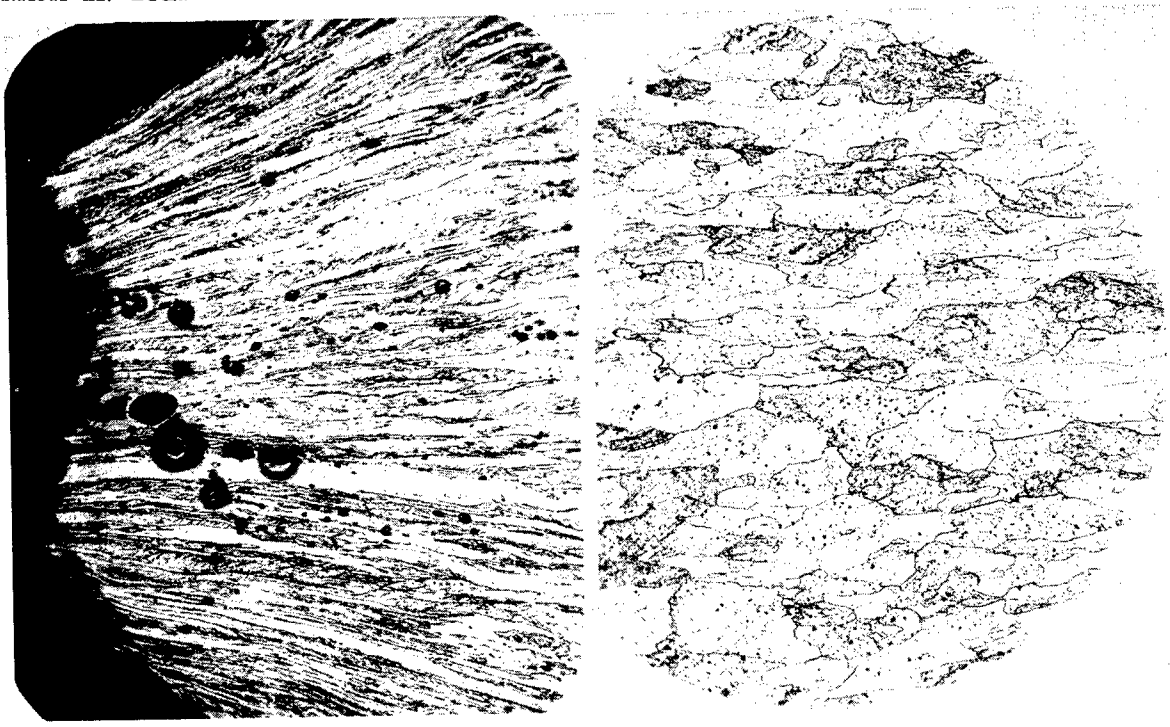


(a) View perpendicular to rolled surface.

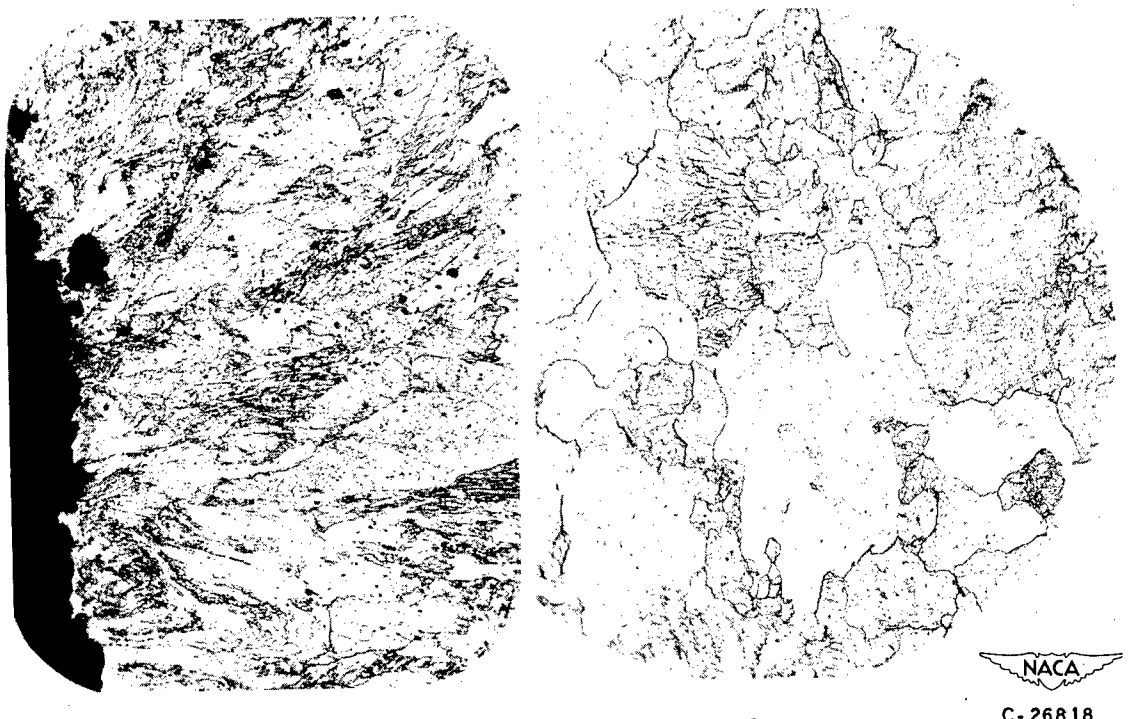


(b) View parallel to rolled surface.

Figure 16. - Views at fracture (left) and remote from fracture (right) of tensile specimens of 1/4-inch rolled molybdenum plate with long axis of test-section parallel to longitudinal direction of rolling. Fractured at 1800° F. X100 (reduced 15 percent).



(a) View perpendicular to rolled surface.



(b) View parallel to rolled surface.

Figure 17. - Views at fracture (left) and remote from fracture (right) of tensile specimens of 1/4-inch rolled molybdenum plate with long axis of test-section transverse to direction of rolling. Fractured at 1800° F. X100 (reduced 15 percent).

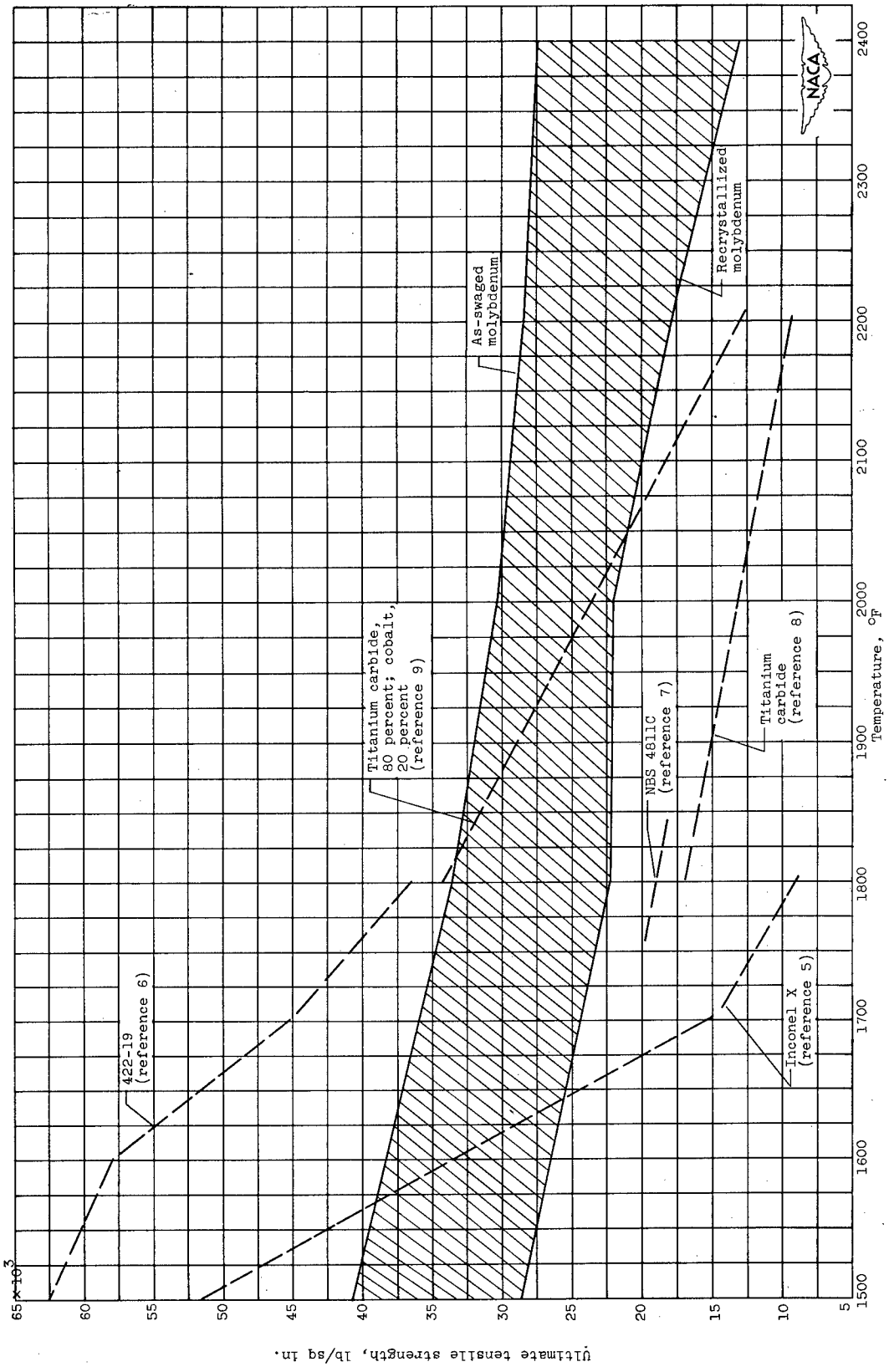


Figure 18. - Ultimate tensile strengths of sintered wrought molybdenum bars and several other materials at elevated temperatures.

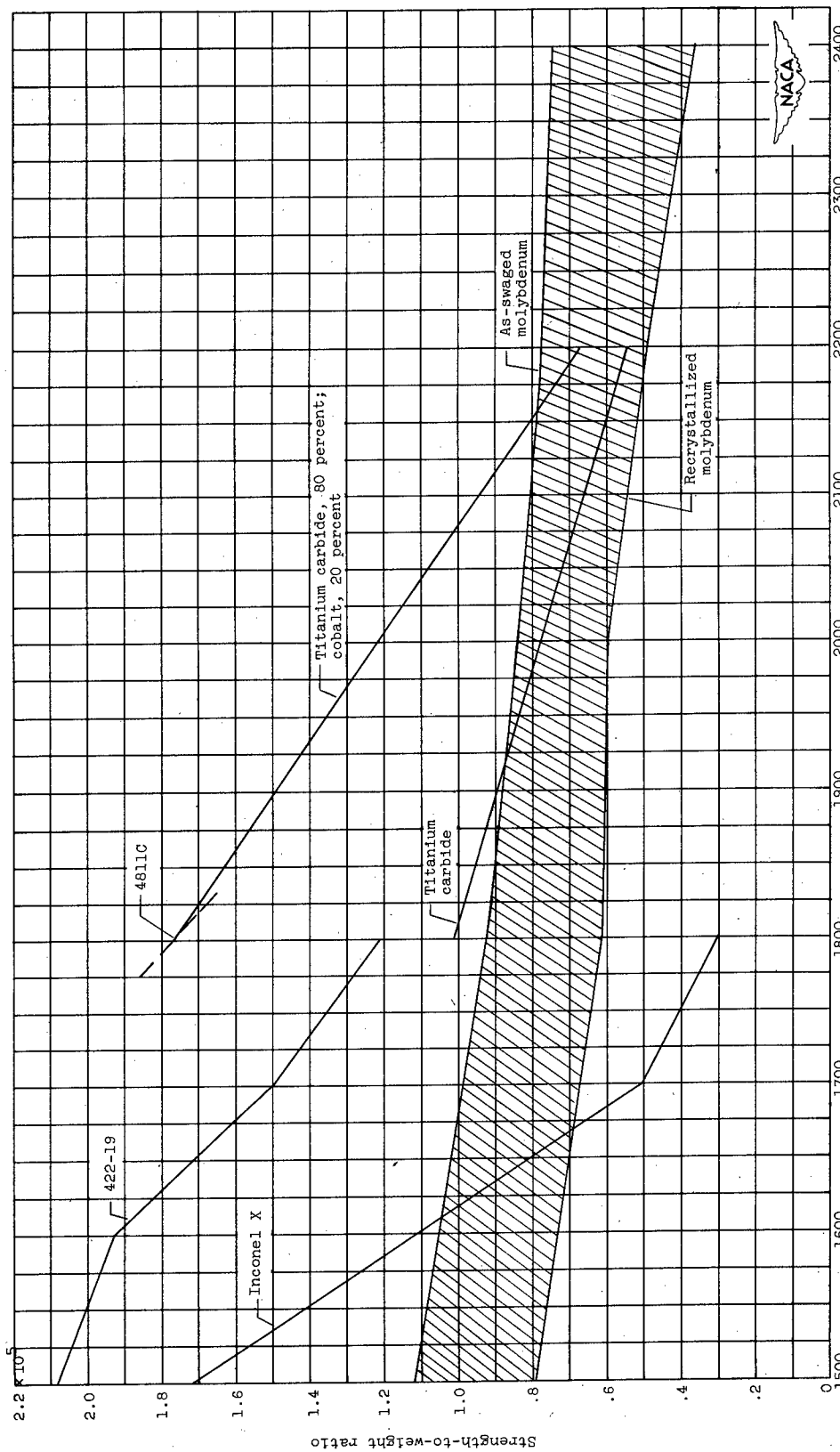


Figure 19. - Strength-to-weight ratios of molybdenum and several other materials at elevated temperatures.

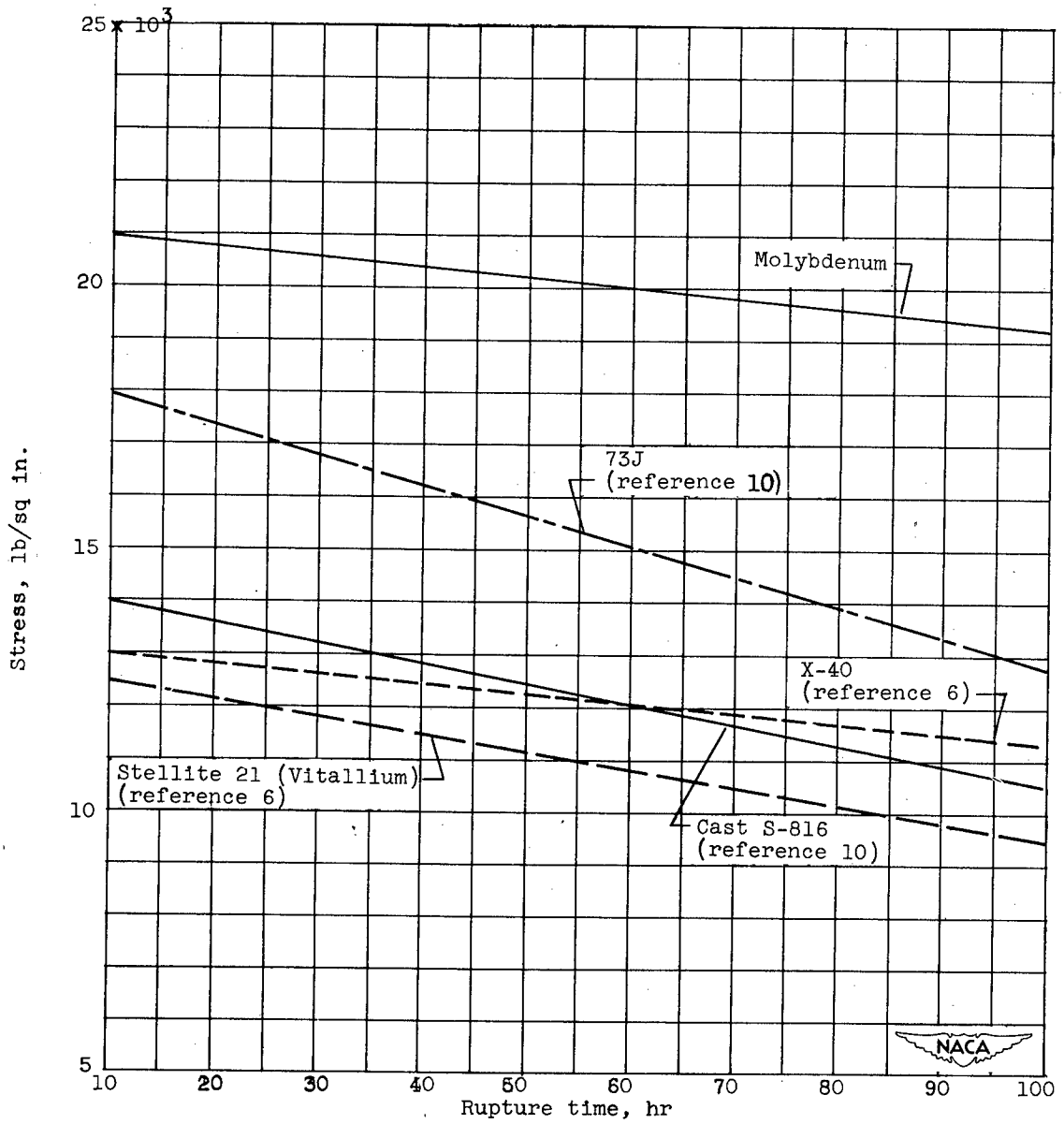
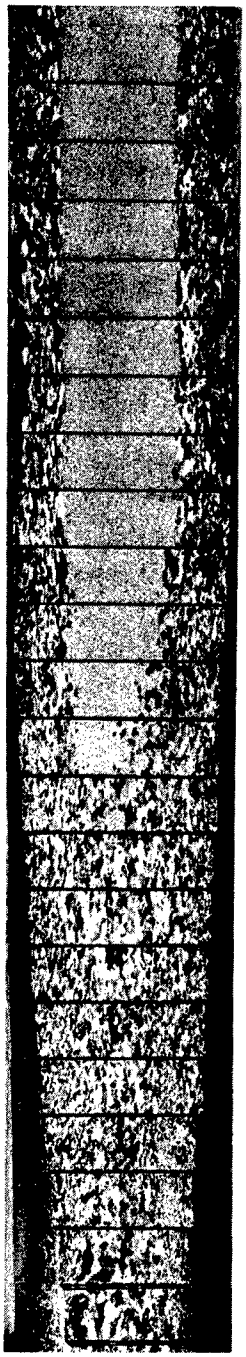
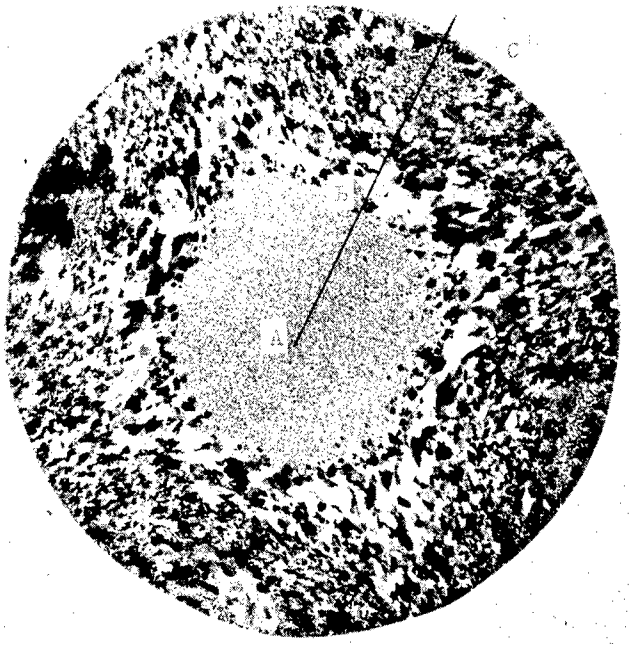


Figure 20. - Stress-rupture properties of 1/2-inch-diameter sintered wrought molybdenum bars and several other materials at 1800° F.

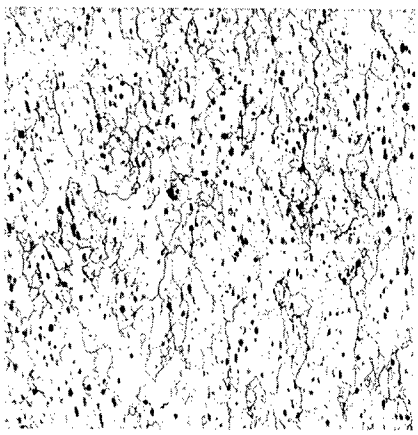


(a) Longitudinal stepped cuts showing core and circumferential structure. X1.5.



(b) Transverse view showing swaging flow lines. X4.

(c) Longitudinal structure detail in large-grained area. X100.



(d) Longitudinal structure detail in core area. X150

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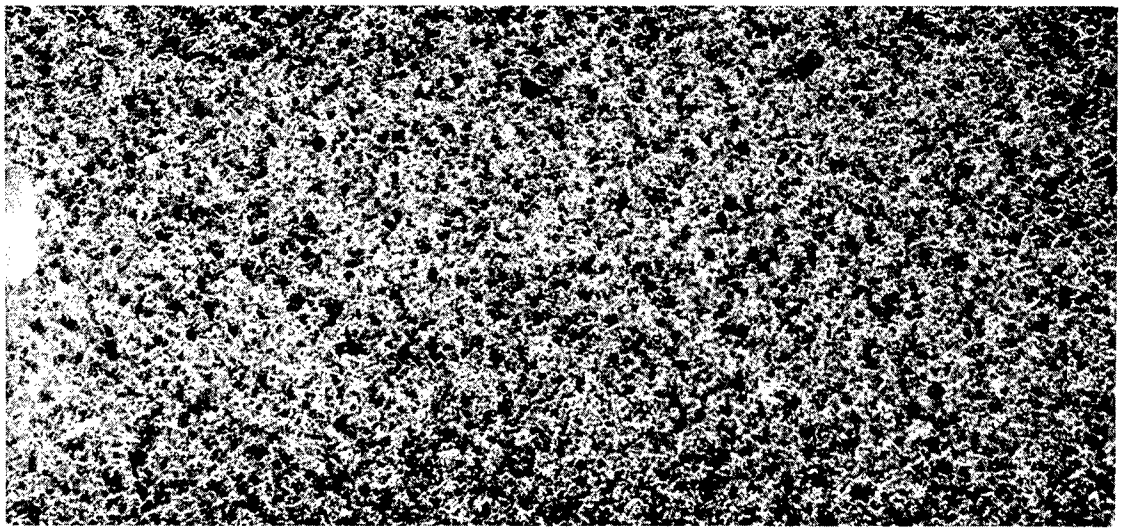
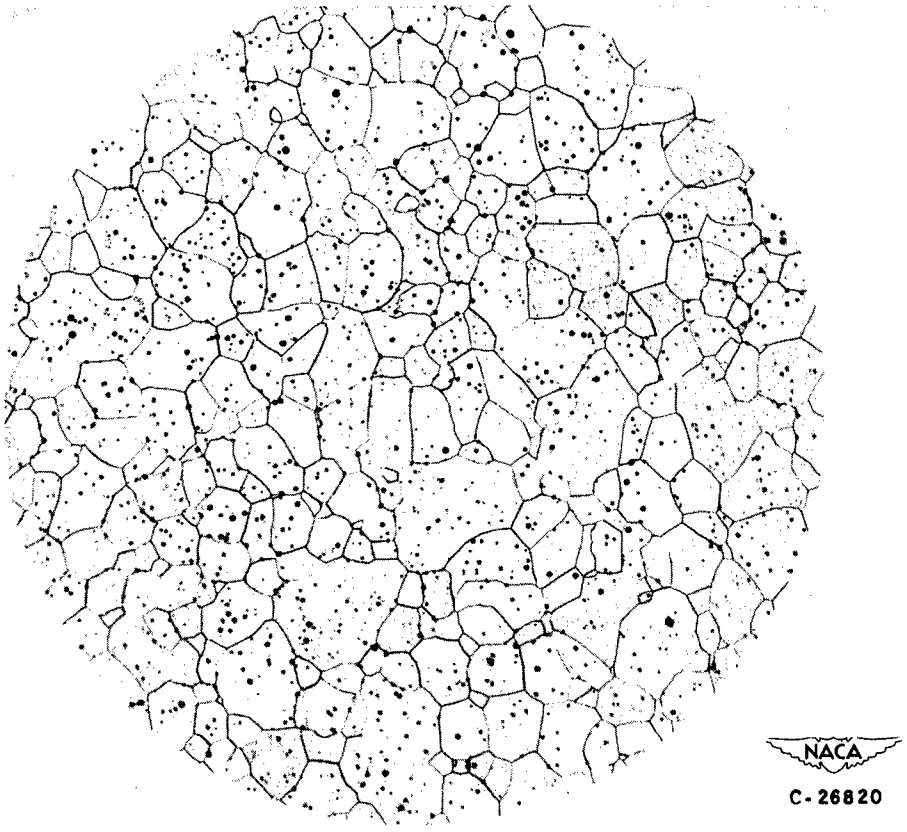
Figure 21. Typical structures of 1-inch-diameter as-swaged molybdenum bar. X1.5 to X150 (reduced 15 percent).

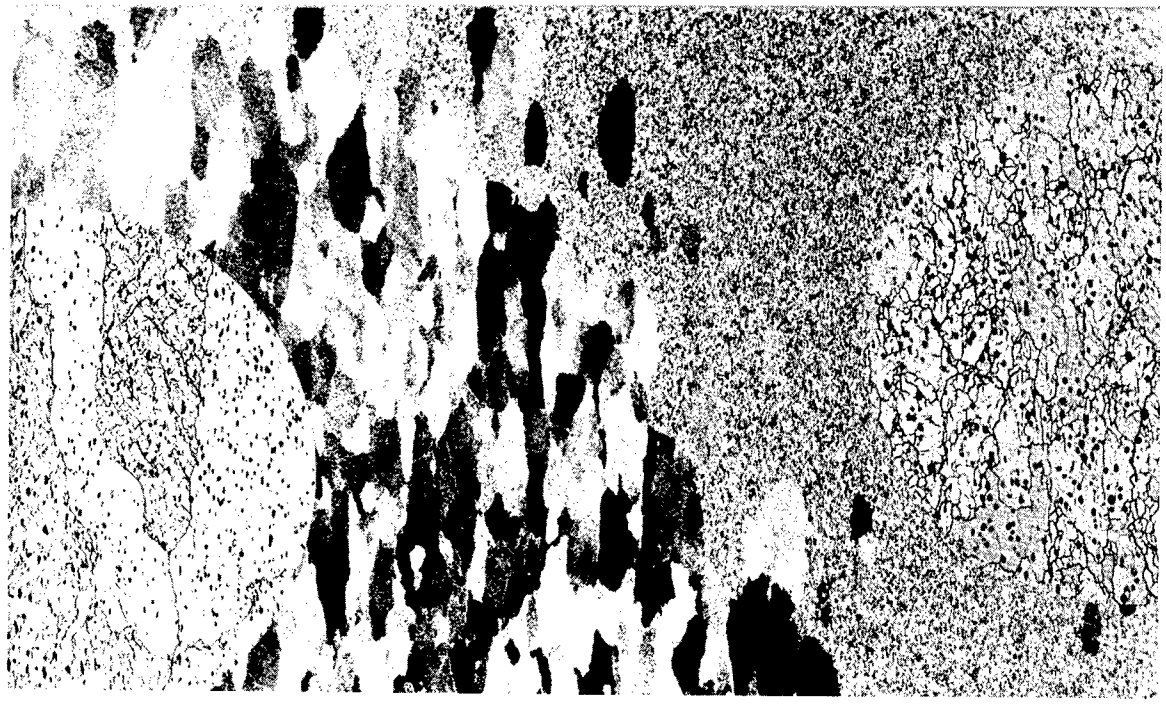


(a) Entire cross section showing small-grain uniformity. X13.

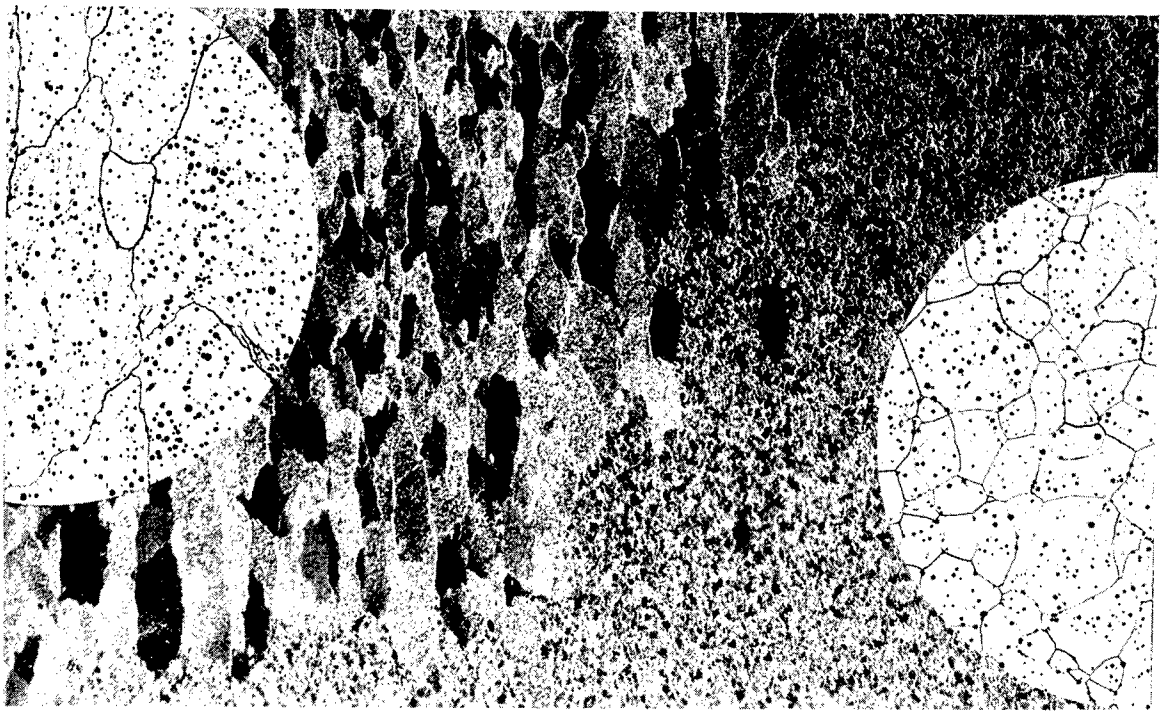


(b) Structure detail. X150.

Figure 22. Longitudinal views of recrystallized typical core area from 1-inch-diameter molybdenum bar after 72 hours at 2700° F. X13 and X150 (reduced 15 percent).



(a) Nonuniform core of "as-swaged" bar.



(b) Nonuniform core after 24 hours at 2800° F.

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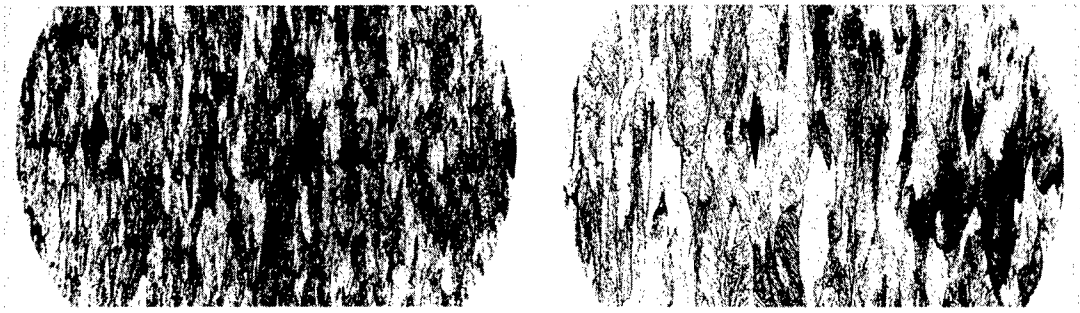
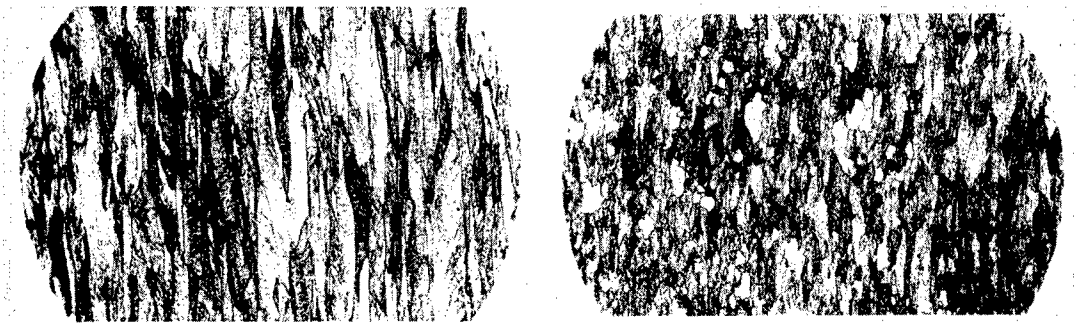
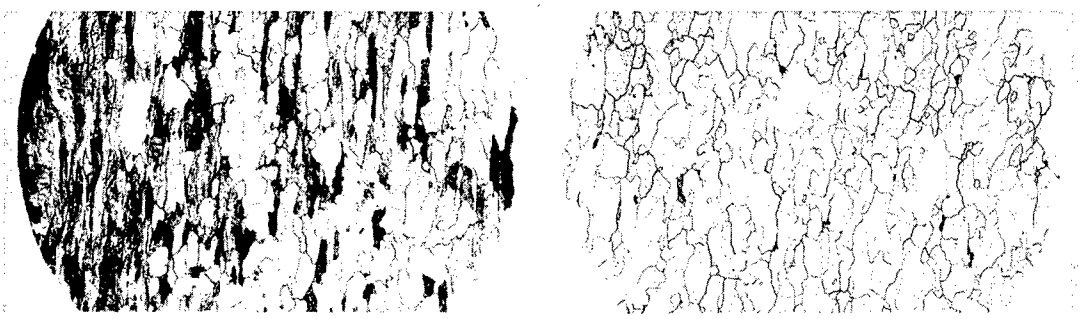
Figure 23. Example of deviation from uniform core structure of 1-inch-diameter molybdenum bar. X13; insets, X150 (reduced 15 percent).



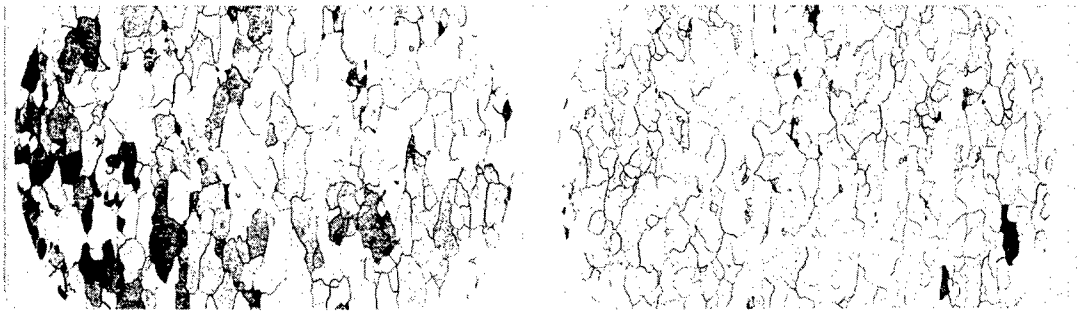
(a) As-swaged.



(b) Recrystallization beginning after 1 hour at 2000° F.



(c) Recrystallization after 20 hours at 2000° F.



(d) Grain structure after 20 hours at 2300° F.

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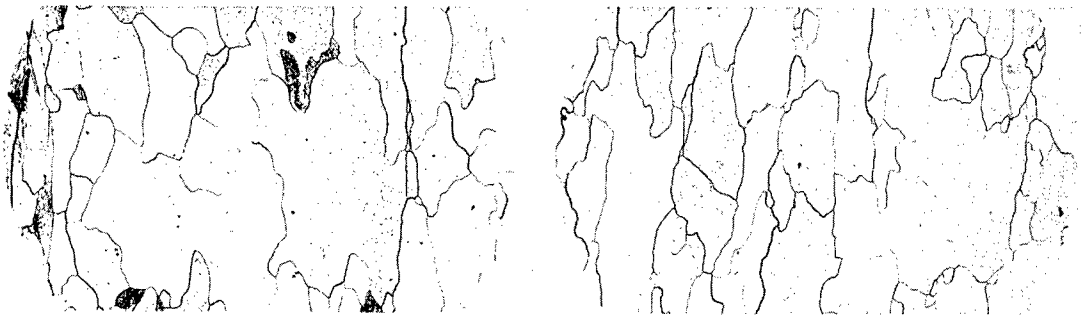
Figure 24. Recrystallization of small-grained 1/2-inch-diameter molybdenum bar. Longitudinal views at bar edge (left) and bar center (right). X35 (reduced 15 percent).

(a) As-swaged.

(b) Recrystallization beginning after 20 hours at 2000° F.

(c) Recrystallization after 20 hours at 2200° F.



(d) Grain structure after 3 hours at 2600° F.

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Figure 25. Recrystallization of large-grained 1/2-inch-diameter molybdenum bar. Longitudinal views at bar edge (left) and bar center (right). X35 (reduced 15 percent).

(a) As-swaged, small grained.

(b) As-swaged, large grained.

(c) Initial recrystallization as shown in figure 24 (b).

(d) Completely recrystallized, small grained.

(e) Completely recrystallized, large grained.

Figure 26. Structural detail at center of 1/2-inch-diameter molybdenum bars. X150 (reduced 15 percent).

Alloys, Heat-Resisting 5.1.4

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Some Properties of High-Purity Sintered Wrought Molybdenum Metal at Temperatures up to 2400° F

By R. A. Long, K. C. Dike, and H. R. Bear

NACA TN 2319

March 1951

(Abstract on Reverse Side)

Materials, Properties 5.2

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Materials, Properties, Tensile 5.2.1

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Materials, Properties, Stress-Rupture 5.2.4

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NACA TN 2319

March 1951

(Abstract on Reverse Side)

Abstract

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Outstanding mechanical properties reported are: tensile strengths from 1800° to 2400° F of 33,670 and 27,500 pounds per square inch; 100-hour strength at 1800° F of 19,300 ±300 pounds per square inch. The properties of materials evaluated varied with degree of "effective" cold working, recrystallization, temperature, and manufacturing source.

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