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# RESEARCH MEMORANDUM

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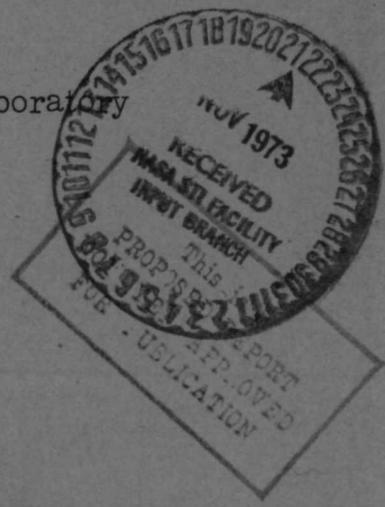
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9/29/54 Date from 12/2/54

FABRICATION AND PROPERTIES OF HOT-PRESSED  
MOLYBDENUM DISILICIDE

By Roger A. Long

Lewis Flight Propulsion Laboratory  
Cleveland, Ohio

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To Unclassified  
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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

RESEARCH MEMORANDUM

FABRICATION AND PROPERTIES OF HOT-PRESSED

MOLYBDENUM DISILICIDE

By Roger A. Long

SUMMARY

Hot-pressed molybdenum disilicide bodies produced by industrial processes at a temperature of  $2950^{\circ} \pm 100^{\circ}$  F and a pressure of 3000  $\pm$  500 pounds per square inch were investigated. The following characteristics and properties were observed:

Short-time tensile strength, lb/sq in.	
( $^{\circ}$ F)	
1800 . . . . .	40,000
2000 . . . . .	42,160
2200 . . . . .	42,800
2400 . . . . .	41,070
Preliminary 100-hour rupture stress	
at 1800 $^{\circ}$ F, lb/sq in. . . . .	above 30,000
Compression strength at 80 $^{\circ}$ F, lb/sq in. . . . .	350,000
Air-corrosion resistance at 100 $^{\circ}$ to 2850 $^{\circ}$ F,	
gram/(sq cm)(hr) . . . . .	maximum weight change, 3.1x10 $^{-6}$
Rockwell hardness at room temperature. . . . .	A-80 to A-87
Knoop hardness at room temperature,	
(1 kg load). . . . .	850 to 870
Average coefficient of linear thermal expansion	
at 80 $^{\circ}$ to 2732 $^{\circ}$ F, in./(in.)( $^{\circ}$ F) . . . . .	5.1x10 $^{-6}$
Thermal conductivity, watts/(sq cm)(cm)( $^{\circ}$ C)	
( $^{\circ}$ F)	
300 . . . . .	0.54
1000 . . . . .	.39
Brittleness at room temperature. . . . .	very high compared with heat-resisting alloys; comparable to ceramics and ceramals

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## INTRODUCTION

In the NACA research on materials for high-temperature applications, the intermetallic compound molybdenum disilicide  $\text{MoSi}_2$  is being investigated at the Lewis laboratory. The fabrication of molybdenum disilicide specimens by hydrostatic cold-pressing and sintering is described in reference 1 and data are presented describing physical characteristics of the material so prepared. These molybdenum disilicide specimens were prepared in the laboratory in an effort to eliminate impurities. The present investigation was conducted to determine hot-pressing conditions that might produce satisfactory hot-pressed molybdenum disilicide bodies and to evaluate the properties of the bodies produced by use of these conditions.

Inasmuch as the material characteristics obtainable under industrial conditions of powder and body preparation are of importance, the material investigated was in large part prepared by industrial methods. Preliminary investigations at the Lewis laboratory served as the basis for specifications to which bodies were to be made. Molybdenum disilicide powder for the experiments was compounded by the Electro Metallurgical Division of the Union Carbide and Carbon Corporation and hot-pressed into bars by the Metals Carbides Corporation. The bars were ground into shapes suitable for evaluating the various properties of the material.

The methods of powder and body preparation and the following characteristics of the hot-pressed bodies observed at the Lewis laboratory are presented: high-temperature short-time tensile strength and stress-rupture strength, high-temperature air-corrosion resistance, hardness, thermal conductivity, and density. Thermal-expansion data obtained by the National Bureau of Standards are also included. The effects of variations in several of the factors involved in fabrication of the material are discussed in the appendix.

Insufficient data are available to compare the strengths of bodies prepared by cold-pressing and sintering and by hot-pressing. A comparison is made, however, of the hardnesses and the densities of bodies prepared by the two methods.

## SPECIMEN PREPARATION

The various steps detailed in this section are, in general, presented singularly and represent the conditions that produced the

specimens for which data are given. Variations in these steps and the effects of such variations are discussed in the appendix with one exception, which is variation in particle size and particle-size distribution. The fine-powder materials produced by the use of two particle-size distributions are discussed in this section and are referred to as "uniform-powder and nonuniform-powder molybdenum disilicide."

### Molybdenum Disilicide Compounding

In the laboratory direct-reaction process used in preliminary experiments, an intimate near-stoichiometric mixture (a gram-atomic-weight ratio of 2.01 parts of silicon to 1 of molybdenum) of fine (through 325 mesh), 99.85 percent pure elemental powders was heated to the reaction temperature of approximately 1900° F in an argon or helium atmosphere at an absolute pressure of about 1/2 atmosphere. The product of the laboratory reaction, identified as molybdenum disilicide by X-ray diffraction and chemical-analysis techniques, was similar to the commercial powder prepared afterwards.

The process by which the molybdenum disilicide powder was commercially compounded is a modification of the direct-reaction process used at the Lewis laboratory for making small quantities of powder. The commercial powder, composed of 63.44 ±0.20-percent molybdenum, 36.45 ±0.20-percent silicon, 0.07-percent iron, and 0.10-percent other impurities by weight, was furnished in dry granular form with 100 percent of the particles passed through a 100-mesh screen and 77 percent through a 325-mesh screen.

### Body Fabrication

Bars 0.520 inch square and  $3\frac{1}{2}$  to 7 inches long were formed from the commercial powder according to the following procedure:

Milling - Molybdenum disilicide powder suspended in methanol was comminuted in a 6-liter chrome-steel ball mill to produce the particle-size distributions shown in the second and third columns of table I. The ball-mill charge comprised approximately 25 pounds of 7/16-inch chrome-steel balls, 10 pounds of powder, and  $1\frac{1}{2}$  quarts of methanol. Comminution time was approximately 24 hours to produce the nonuniform powder and approximately 48 hours to produce the uniform powder.

Chemical analysis of the molybdenum disilicide powder after the comminution process showed the only metallic "impurity" introduced by the milling process to be 0.5- to 1.0-percent iron.

Drying - The milled powder was partly dried at room temperature with evaporation of the methanol completed by application of radiant heat at powder temperatures below 140° F.

Hot-pressing - The dry powder was pressed in molded fine-grain graphite dies at a constant pressure of  $3000 \pm 500$  pounds per square inch and a maximum temperature of  $2950 \pm 100^\circ$  F. The pressing sequence comprised the following steps: application of pressure; heating, approximately 8 minutes; heating at maximum temperature, approximately 1/2 minute; release of pressure; cooling (in die), approximately 30 minutes.

#### Specimen Fabrication

Specimens of the following types were ground from the hot-pressed bars with diamond abrasive wheels:

Specimen	Type
Tensile, stress-rupture	Cylindrical; test-section diameter, 1/4 in.; conical ends (fig. 1)
Compression	Cylindrical; diam., 3/8 in.; length, $1\frac{1}{8}$ in.
Air-corrosion resistance, hardness, density	Rectangular; 1/2 by 1/2 by 1/4 in.
Thermal conductivity	Cylindrical; diam., 1/2 in.; length, 7 in.
Thermal expansion	Equilateral prism; sides, 0.4 in.; length, 2 in.

#### APPARATUS AND PROCEDURE

Metallographic specimens of uniform-powder and nonuniform-powder hot-pressed materials, etched with a solution containing 17-percent hydrochloric acid and 8-percent nitric acid, were photomicrographed for metallurgical examination.

Short-time tensile strength - Tensile specimens were pulled to fracture under the following conditions:

Tensile machine

Type . . . . . universal; hydraulic  
 Capacity, lb. . . . . 120,000  
 Low scale reading, lb. . . . . 0-6000  
 Loading rate  
 lb/min. . . . . 3500  
 lb/(sq in.)(min)(nominal) . . . . . 71,000

Specimen heating

Heat supply . . . . . platinum resistance-wire furnace  
 Temperature control . . . . . automatic; series resistor  
 discontinuously shunted  
 by temperature controller

Specimen temperature

measurement . . . . . thermocouples at both ends of gage  
 length; bead against specimen

Time at evaluation temperature

before application of load, hr. . . . . 1/2  
 Evaluation temperatures, °F . . . . . 80, 1800, 2000, 2200, and 2400

Preliminary experiments dictated the need of accurate specimen alinement. Special grips, held by extension arms designed to be self-aligning, were therefore devised (fig. 2). The grip inserts were made of Inconel "X" alloy or Haynes Stellite Alloy No. 25 depending on the application, and the grip housings and the extension arms of Inconel "X" alloy. The inserts were machined to close tolerances to match the conical portions of the tensile specimen, but local deformation of the inserts was intended to provide final specimen alinement.

Permanent deformation of specimens was determined by measuring the length between specimen ends before and after evaluation. Measurements after failure were made on those specimens that fractured in such a manner as to permit reassembly of the fragments.

Stress-rupture strength - The equipment (fig. 3) for the determination of stress-rupture data includes grips similar to those used in the tensile-strength evaluations. Specimen-alinement procedure was similar to that of the tensile-strength evaluation. The hydraulic-type rupture machine used was designed for the testing of brittle materials.

A preliminary evaluation was made by stabilizing specimen temperature at 1800° F; after stabilization, a loading sequence comprising 100-hour periods at loads successively higher by increments of 5000 pounds per square inch was followed until specimen fracture. A later evaluation, also at 1800° F, was made by holding load constant until failure resulted.

Compression strength - Room-temperature compression strength was determined by applying an axial compressive load to a cylinder of nonuniform-powder material having a length-diameter ratio of 3. The load was applied with the tensile machine previously described.

Air-corrosion resistance - The elevated-temperature air-corrosion resistance of the material was evaluated by determining the weight change produced during exposure of specimens to high-temperature freely circulating air in an open-tube resistor-element-type furnace for successive periods of time. The specimens were edge-mounted on alundum boats and evaluated under the following conditions:

Specimen surface finish, microin., rms . . . . .	5 to 15
Evaluation temperatures, °F. . . . .	2200, 2450, and 2850
Cumulative time at stabilized	
temperature, hr. . . . .	100, 135, 200, and 300

Hardness - Specimen macrohardness was measured with a standard Rockwell hardness tester using a diamond indenter and a 60-kilogram load. This procedure gives a value on the Rockwell A scale, which is commonly used in the cemented-carbide industry.

Microhardness determinations were made on polished and partly etched metallographic specimens by the use of a Knoop indenter with a Tukon hardness tester at a load of 1 kilogram.

Coefficient of linear thermal expansion - Linear thermal expansion of a nonuniform-powder prism specimen was determined by the National Bureau of Standards, using a unique dilatometer that is described in reference 2. The temperature range covered was 26° to 1500° C (80° to 2732° F).

Thermal Conductivity - Thermal conductivity was determined by the method of reference 3 from measurements of axial temperature distribution and axial heat flow along a cylinder .7 inches long and 1/2 inch in diameter pressed from nonuniform powder. Axial temperature distribution was measured by equally spaced thermocouples silver-soldered to the cylinder surface; heat flow was determined from measurements of temperature gradient along a similar cylinder

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of nickel, having a known thermal conductivity, in thermal series with the molybdenum disilicide cylinder. Radial heat loss from the cylinders was prevented by the method described in reference 3.

Density - Density was determined by calculations based on measured weights and computed volumes of specimens.

RESULTS AND DISCUSSION

Short-time tensile strength - The observed values of elevated-temperature short-time tensile strength are presented in figure 4 and compared with those of other high-temperature materials obtained from references 4 to 8. The comparison shows molybdenum disilicide to have superior short-time tensile strength at temperatures above approximately 1800° F.

The curves show that the strength of nonuniform-powder bodies is approximately constant at 42,000 pounds per square inch as temperature varies from 2000° to 2400° F. The strength of uniform-powder bodies remains constant at about 40,000 pounds per square inch from 1800° to about 2200° F; above 2200° F the strength decreases, being about one-half as great at 2400° F. The observed values of room-temperature strength were probably lower than the actual total stresses present because of bending stresses set up by noncollinearity of the grips. These bending stresses were reduced at the higher temperatures by local plastic deformation of the grip inserts that permitted uniaxial loading of the specimen.

A view of a specimen after a tensile evaluation, presented in figure 5, is indicative of the brittleness of the material. The measurements of specimen lengths before experiment and after fracture (by reassembly of the parts) gave the following values of permanent elongation:

Evaluation temperature (°F)	Permanent elongation (in.)/(in. total) specimen length	
	Nonuniform powder	Uniform powder
80	Undetectable	Undetectable
1800	Undetectable	Undetectable
2000	Undetectable	Undetectable
2200	Undetectable	0.001
2400	Undetectable	.005

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It is possible that the high rate of loading used may have caused observed values of elongation to be low. The decrease in strength of the uniform-powder material at temperatures above 2200° F is apparently associated with the plastic deformations observed, which were small but definite. The brittleness of the bodies is comparable to that of a ceramic or a ceramal.

Photomicrographs (fig. 6) show hot-pressed molybdenum disilicide to be a fine-grained material containing a definite amount of intergranular spheroidal secondary phase, which has not yet been identified. At high temperatures, the ductile behavior of the uniform-powder material and the nonductile behavior of the nonuniform-powder material may be a function of dispersion of the intergranular secondary phase. This phase is present in both materials but is dispersed differently in the two materials because of the difference in particle size, as shown in figure 6(b). The secondary phase seems to be a product of reaction between the methanol and the disilicide during the milling action. Later experiments (see appendix) showed that the phase is formed in lesser quantity, but not eliminated, by the use of benzene as the carrier medium during milling.

A direct comparison between the tensile strength of hot-pressed molybdenum disilicide, which is reported herein, and the modulus-of-rupture strength of low density, cold-pressed and sintered molybdenum disilicide, which is reported in reference 1, cannot be made because of the following reasons:

(1) Available information indicates that, for a given material, modulus-of-rupture values may be from one to three times as great as tensile values.

(2) Rate of loading has an appreciable effect on the strength values obtained. Inasmuch as the stress rates used in the modulus-of-rupture and the tensile evaluations were widely different, comparisons would be influenced by this effect.

Neither of these materials is considered to be the best that can be produced by the respective methods.

Stress-rupture data - Specimens of uniform-powder material broke in tension after the following cumulative applications of load at 1800° F:

Load (lb/sq in.)	Time (hr)	
	Specimen	
	A	B
20,000	100	---
25,000	100	---
30,000	100	---
35,000	3	3

The data obtained indicate that the 100-hour rupture life is somewhat greater than 30,000 pounds per square inch. This value is approximately three times that for high-temperature alloys reported in references 4 and 5, and approximately twice as great as published values for ceramic materials (reference 7).

Compression strength - Room-temperature compression strength of the single specimen evaluated was determined as 350,000 pounds per square inch. At failure the specimen shattered suddenly, breaking into numerous very small pieces.

Air-corrosion resistance - The following weight changes were caused by successive exposures to high-temperature freely circulating air:

Temperature (°F)	Cumulative weight gain, (grams/(sq cm)(hr))			
	Time (hr)			
	100	135	200	300
2200	$0.65 \times 10^{-6}$	- -	$1.0 \times 10^{-6}$	$0.70 \times 10^{-6}$
2450	3.00	- -	.96	.64
2850	-3.67	$-3.10 \times 10^{-6}$	- -	- -

The weight gains at temperatures of 2200° and 2450° F were probably caused by the formation of molybdenum disilicide derivatives on the exposed surfaces. Metallographic examination showed (fig. 7) a definite migration of the secondary phase from the core to the surface of the specimen exposed 135 hours at 2850° F that was not apparent in specimens exposed at the lower temperatures. Electron-diffraction examination indicated the surface material to be amorphous.

The weight loss that occurred at 2850° F may be attributable to sublimation of this phase or to wetting of the supporting boat by this phase.

Hardness - Rockwell hardnesses of both uniform-powder and nonuniform-powder materials were determined as A-80 to A-87 and Knoop hardnesses, using a 1-kilogram load, as 850 to 870. The Knoop values of microhardness are considered more representative than the Rockwell values of macrohardness; because of the high brittleness of the specimens, the Rockwell indenter caused some indentation cracking, whereas the Knoop indenter did not.

The Rockwell hardness of molybdenum disilicide specimens prepared by cold-pressing and sintering is reported in reference 1 as C-57.

Coefficient of linear thermal expansion - The average coefficient of linear expansion of the material, calculated from data supplied by the National Bureau of Standards (fig. 8), is  $5.1 \times 10^{-6}$  inch per inch per °F over the temperature range 80° to 2732° F. The linear thermal expansion is almost identical with that reported in reference 2 for corundum ( $Al_2O_3$ ) parallel with the rhombohedral axis. Approximate uniformity of thermal expansion over the range investigated indicates no phase transformations.

Thermal conductivity - The values presented in figure 9 show that thermal conductivity of molybdenum disilicide decreases with increase in temperature. This trend is opposite to that reported for high-temperature alloys in references 4 and 5. The thermal conductivity varies approximately linearly with change in temperature and is 0.54 watt per square centimeter per centimeter per °C at 300° F and 0.39 watt per square centimeter per centimeter per °C at 1000° F.

Density - Density of the uniform-powder material was determined as 5.87 grams per cubic centimeter and that of the nonuniform-powder material as 5.90 to 5.92 grams per cubic centimeter. These values are 94.07 and 94.53 percent, respectively, of the theoretical density of molybdenum disilicide, 6.24 grams per cubic centimeter, reported in reference 1. The observed densities are about 70 percent of the densities of high-temperature alloys in current use.

The density of molybdenum disilicide specimens prepared by cold-pressing and sintering is reported in reference 1 as 5.68 grams per millimeter.

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### SUMMARY OF RESULTS

Bodies were satisfactorily hot-pressed from uniform and from nonuniform molybdenum disilicide powders at a temperature of  $2950^{\circ}$   $\pm 100^{\circ}$  F and a pressure of  $3000 \pm 500$  pounds per square inch. Evaluation of the properties of the bodies may be summarized as follows:

1. At temperatures above  $1800^{\circ}$  F, hot-pressed molybdenum disilicide had outstanding short-time tensile strength. Bodies made of powder of nonuniform particle size were stronger at temperatures above about  $2200^{\circ}$  F than bodies made of powder of uniform particle size. Bodies made of powder of nonuniform particle size had short-time tensile strengths at temperatures between  $2000^{\circ}$  and  $2400^{\circ}$  F of approximately 42,000 pounds per square inch. Specimens of uniform-particle-size powder showed evidence of ductility above approximately  $2200^{\circ}$  F.

2. Preliminary data indicated that the 100-hour rupture stress of the material at  $1800^{\circ}$  F was in excess of 30,000 pounds per square inch, which is approximately three times that of the strongest high-temperature alloys now in use.

3. Evaluation of a single specimen indicated a room-temperature compression strength of 350,000 pounds per square inch.

4. Air-corrosion resistance of the material was extremely high; the weight of small rectangular specimens changed at a maximum rate of only  $3.10 \times 10^{-6}$  grams per square centimeter per hour during various exposures of 300 hours at  $2200^{\circ}$  F, 300 hours at  $2450^{\circ}$  F, and 135 hours at  $2850^{\circ}$  F.

5. The material had a measured Rockwell hardness of A-80 to A-87 and a Knoop microhardness under a load of 1 kilogram of 850 to 870.

6. Average coefficient of linear thermal expansion of the material was determined as  $5.1 \times 10^{-6}$  inch per inch per  $^{\circ}$ F over the temperature range of  $80^{\circ}$  to  $2732^{\circ}$  F.

7. Thermal conductivity of the material was determined as 0.54 watt per square centimeter per centimeter per  $^{\circ}$ C at  $300^{\circ}$  F and 0.39 watt per square centimeter per centimeter per  $^{\circ}$ C at  $1000^{\circ}$  F.

Lewis Flight Propulsion Laboratory,  
National Advisory Committee for Aeronautics,  
Cleveland, Ohio, May 26, 1950.

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

FABRICATION AND PROPERTIES OF HOT-PRESSED

MOLYBDENUM DISILICIDE

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## APPENDIX

EFFECTS OF FABRICATION VARIABLES ON PROPERTIES OF  
HOT-PRESSED MOLYBDENUM DISILICIDE

The effects of change in each fabrication factor were not determined for each property of the material, but sufficient information is available to indicate practical limits of many of the factors.

## Preparation of Molybdenum Disilicide Powder

Molybdenum disilicide is formed with an evolution of heat at a particular activation-reaction temperature. In the reaction of the molybdenum and silicon powders, an excess of silicon (0.5 percent of the stoichiometric proportion) was used because an excess of one of the elements would tend to produce reaction completeness. Silicon was chosen as the excessive element because of its smaller atomic radius and its superior oxidation properties.

Purity of the material produced by the direct-reaction process would depend in large part on initial purity of the elemental powders. Completeness of the reaction is dependent on prior intimate mixing of the elemental powders.

In initial experiments, the powder mixture was heated to the reaction temperature in an evacuated tube at an indicated pressure of 1 micron. This procedure resulted in a small amount of silicon-metal volatilization and later reactions were therefore conducted in an atmosphere of argon or purified helium at an absolute pressure of about  $1/2$  atmosphere. The inert atmosphere was maintained during cooling to prevent possible air corrosion of the compound.

Methanol was first used as the liquid carrier in the milling of the disilicide compound to the desired particle size. A degree of gumminess and agglomeration of the dried particles was observed. Benzene was later substituted for the methanol, and the compound powders after being dried showed no evidence of gumminess or agglomeration. These results indicated a possible reaction between methanol and molybdenum disilicide. This reaction was further indicated by metallographic examination that showed the presence of greater amounts of intergranular secondary phase in material

prepared with methanol than in material prepared with benzene. Complete evaporation of the benzene was accomplished by maintaining the powder at temperatures not exceeding 140° F.

### Fabrication of Bodies

The data presented indicate that a powder of fine particle size produces fabricated bodies of high tensile strength. Bodies were fabricated from coarse powder (table I) by the procedures used in fabricating the uniform- and the nonuniform-powder bodies. The following values were obtained with coarse-powder bodies:

Evaluation temperature (°F)	Short-time tensile strength (lb/sq in.)	Compression strength (lb/sq in.)
80	17,350; 18,780; 19,130	210,200
2000	20,430	
2200	25,000; 25,800	
2400	21,500	

No deformation of the specimens was observed. Bodies of coarse-particle-size powders can be satisfactorily fabricated, but the high-temperature strength of such bodies is about one-half that shown in figure 4 for bodies of fine-particle-size powders.

Molded graphite or graphite-clay dies were used successfully in the laboratory and in commercial hot-pressing of molybdenum disilicide powder. Microscopic examination of specimens showed that the graphite reacted with the bodies to a depth of about 1 mil below the surface. Chemical analysis indicated that carbon content of the interior portions of the pressed bodies had not increased during hot-pressing.

The possible number of combinations of heat, pressure, and time can be great, but the following ranges and combinations seem to be the most applicable at present. At low plunger-die pressures (1000 to 2500 lb/sq in.), pressing temperatures of 3100° to 3200° F gave satisfactory dense bodies. This temperature range is, however, at the high limit of furnace equipment used in the commercial production of cemented carbides. Use of such high temperatures is undesirable. A temperature as low as 2600° F could be used satisfactorily in conjunction with high pressure, the pressure being

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limited by die-material strength. The temperature range currently usable for hot-pressing molybdenum disilicide therefore appears to be that between 2700° and 3000° F.

The pressure required for hot-pressing would be that giving a high degree of compactness within the previously specified temperature range. Investigation showed that die pressure, which can be varied between 1000 and 5000 pounds per square inch in order to give acceptably dense bodies, should increase as temperature is decreased.

The maximum density to which molybdenum disilicide can be hot-pressed is of some interest. The highest density produced in these experiments was 6.16 grams per cubic centimeter (98.7 percent of the reported theoretical density of 6.24 grams/cm<sup>3</sup>) and resulted from use of powder of nonuniform particle size at high values of pressing temperature and pressure (3100° F and 4480 lb/sq in.).

Density of a hot-pressed body is not at present a specification that will establish the properties or characteristics of molybdenum disilicide bodies. Establishment of definite relations among body properties, fabrication variables, and density will, however, make density a prime criterion in future specifications. The density to be specified will be that to which bodies can be hot-pressed to give the desired compromise among the properties of the material.

#### Swaging

In order to investigate the possibility of working hot-pressed molybdenum disilicide, attempts were made to swage rods 0.372 inch in diameter and 7 inches long that had been ground from bars 3/8 by 3/8 by 8 inches. The rods, heated to approximately 3100° F, were inserted in a rotary swaging machine set to reduce rod diameter 2.75 percent. The swaging action was so severe that the rods were instantly "chewed" into many pieces. Examination of the fracture faces showed them to have the appearance characteristic of fracture surfaces of brittle materials. The failure in swaging may have been induced by (1) thermal shock of the hot rod by the cooler dies, (2) insufficient ductility of the material to withstand the heavy swaging action, or (3) cleavage action of the intergranular phase.

#### REFERENCES

1. Maxwell, W. A.: Properties of Certain Intermetallics as Related to Elevated-Temperature Applications. I - Molybdenum Disilicide. NACA RM E9G01, 1949.

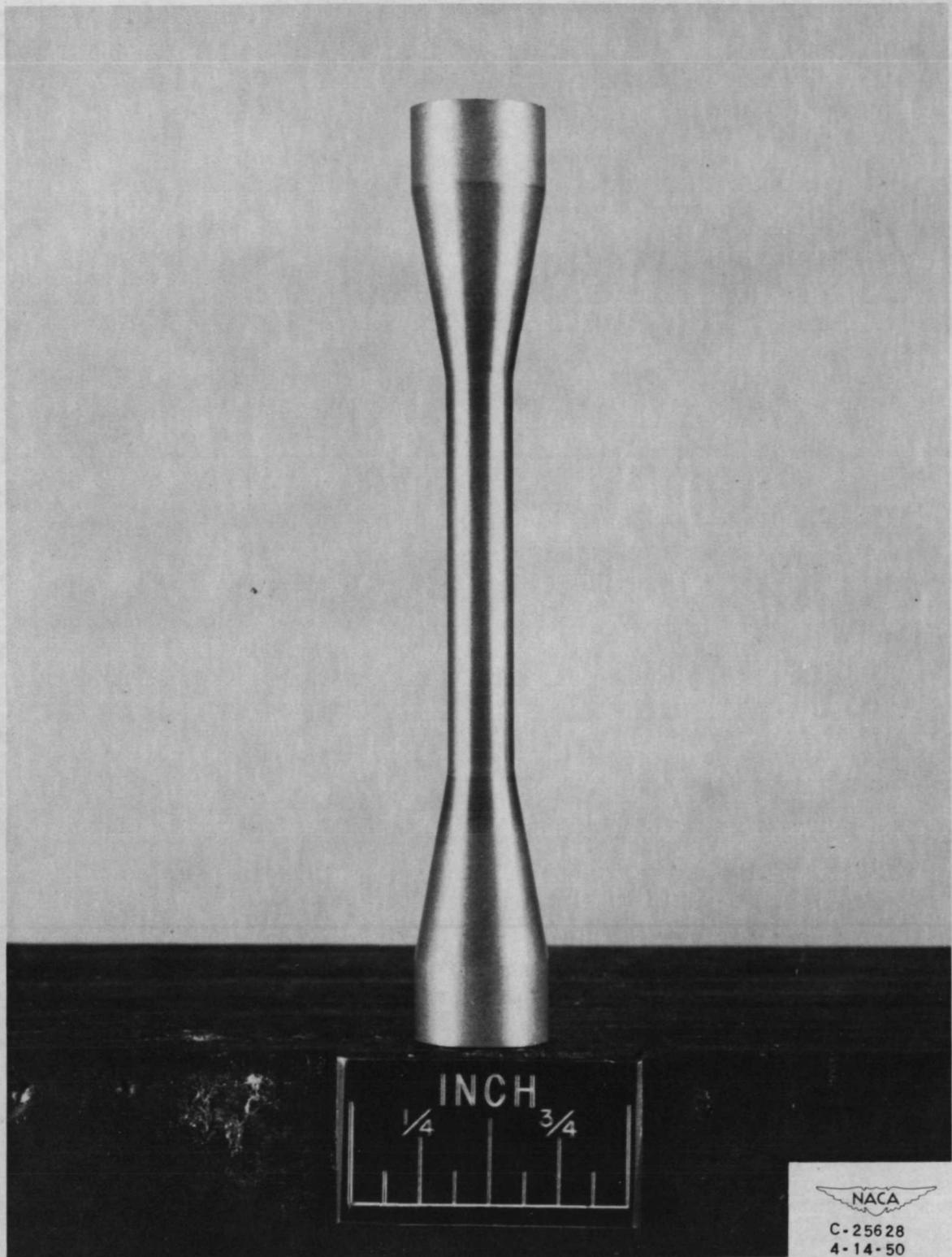
- 1360
2. Geller, R. F., and Yavorsky, Paul J.: Effects of Some Oxide Additions on the Thermal Length Changes of Zirconia. RP1662, Nat. Bur. Standards Jour. Res., vol. 35, no. 1, July 1945, pp. 87-110.
  3. Van Dusen, M. S., and Shelton, S. M.: Apparatus for Measuring Thermal Conductivity of Metals up to 600° C. RP 668, Nat. Bur. Standards Jour. Res., vol. 12, no. 4, April 1934, pp. 429-440.
  4. Anon.: Haynes Alloys for High-Temperature Service. Haynes Stellite Co. (Kokomo, Ind.), 1948.
  5. Anon.: Inconel "X", Data and Information. Development & Res. Div., The International Nickel Co., Inc., rev., Jan. 1949.
  6. Deutsch, George C., Repko, Andrew J., and Lidman, William G.: Elevated-Temperature Properties of Several Titanium Carbide Base Ceramals. NACA TN 1915, 1949.
  7. Burdick, M. D., Moreland, R. E., and Geller, R. F.: Strength and Creep Characteristics of Ceramic Bodies at Elevated Temperatures. NACA TN 1561, 1949.
  8. Gangler, James J., Robards, Chester F., and McNutt, James E.: Physical Properties at Elevated Temperature of Seven Hot-Pressed Ceramics. NACA TN 1911, 1949.

TABLE I - PARTICLE SIZE AND PARTICLE-SIZE DISTRIBUTION OF  
MOLYBDENUM DISILICIDE POWDERS USED IN  
HOT-PRESSED BODIES

[Particle sizes determined by microscopic method based on  
ASTM tentative standard 1614 (1938)]

Upper limit (microns)	Percentage of total smaller than upper limit		
	Fine powder		Coarse powder
	Nonuniform	Uniform	
25	100	100	100
16	98 to 99	100	100
6	90 to 95	100	50
3	---	98 to 99	---





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Figure 1. - Tensile specimen.

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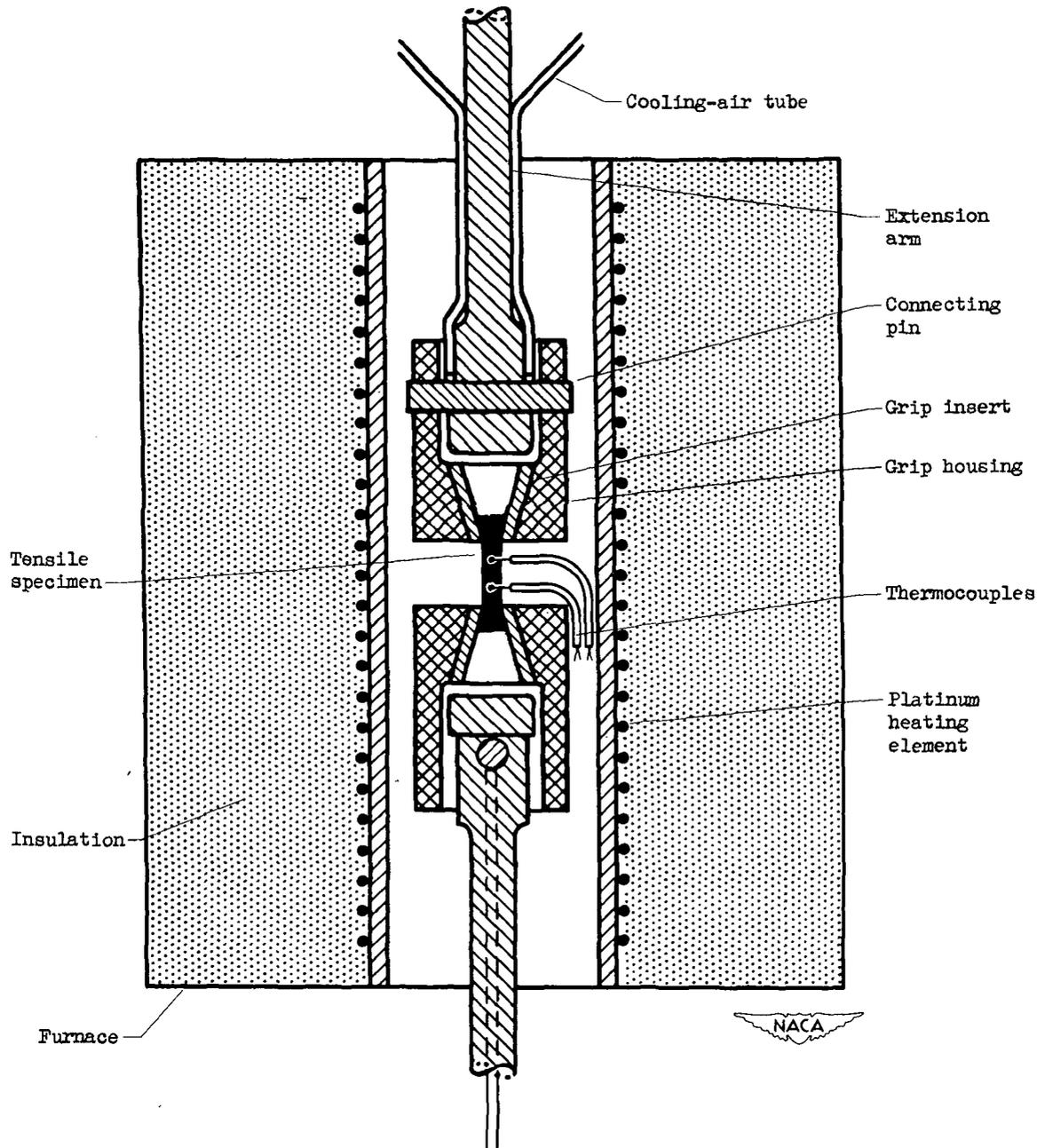
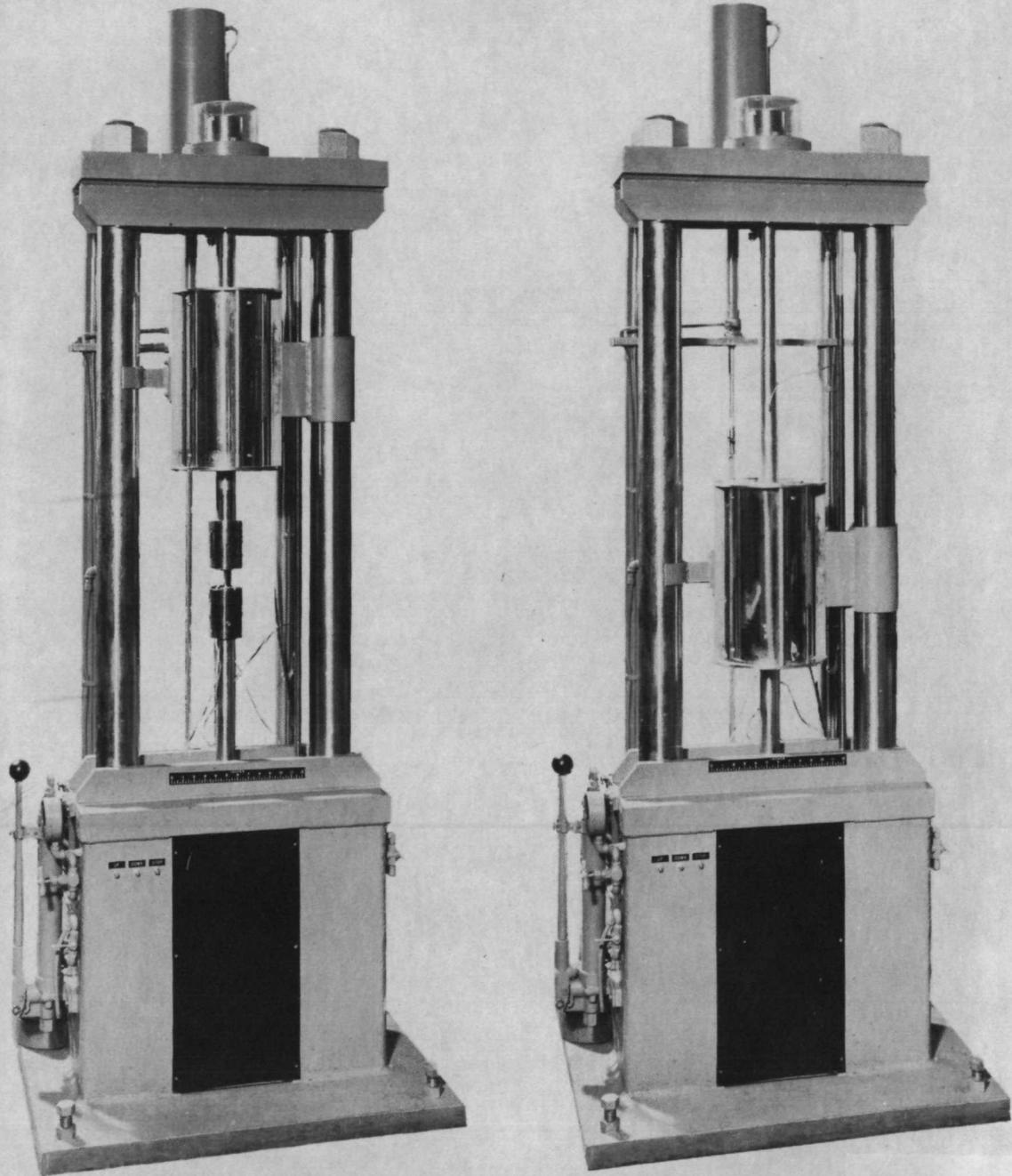


Figure 2. - Apparatus for high-temperature tensile evaluation.

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(a) Furnace in specimen-alignment position. (b) Furnace in evaluation position.  
Figure 3. - Stress-rupture machine.

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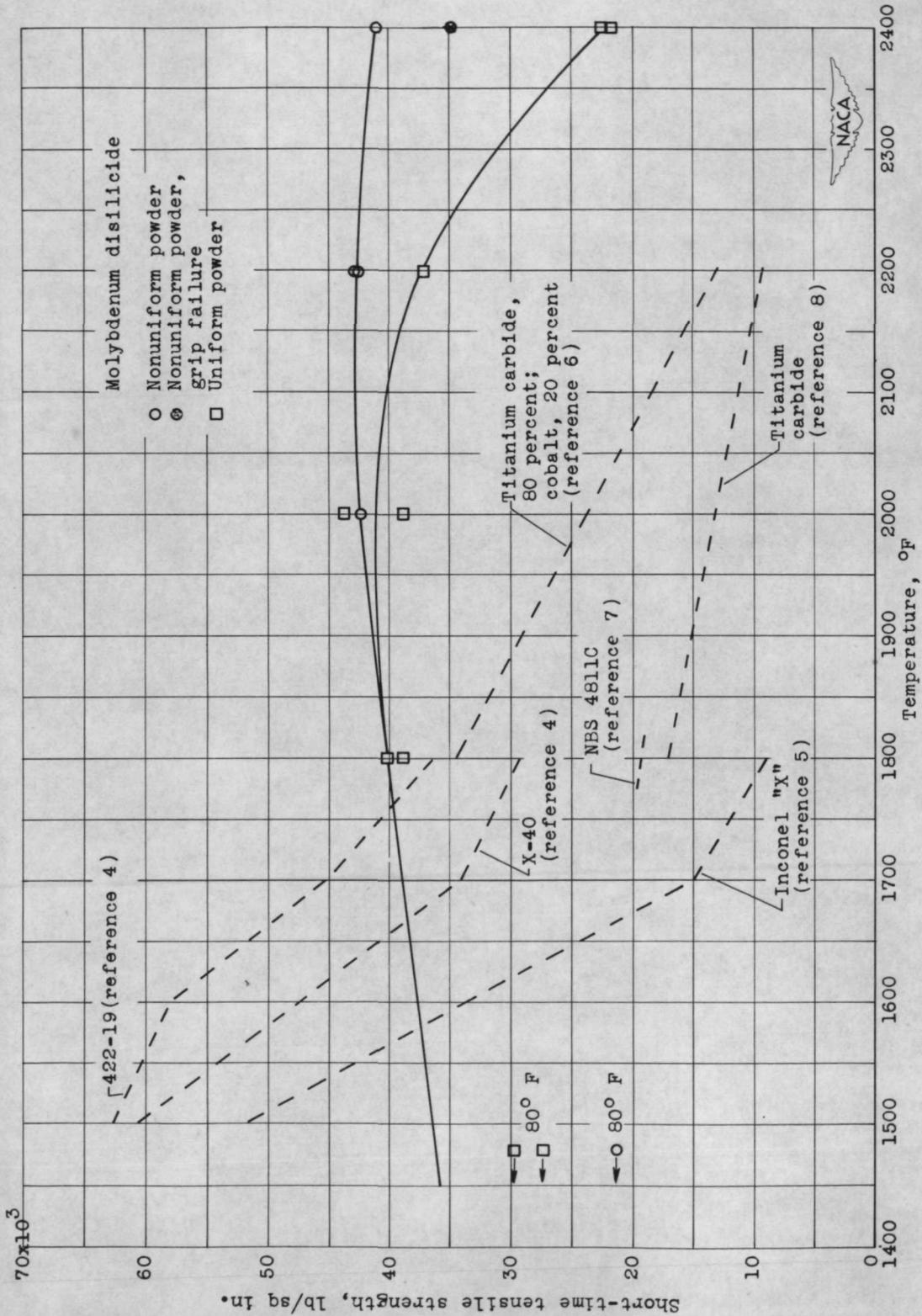


Figure 4. - Short-time tensile strengths of molybdenum disilicide and several other materials at elevated temperatures.

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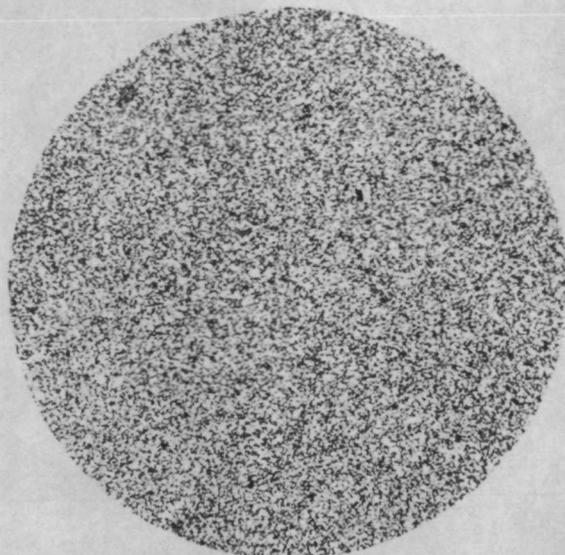
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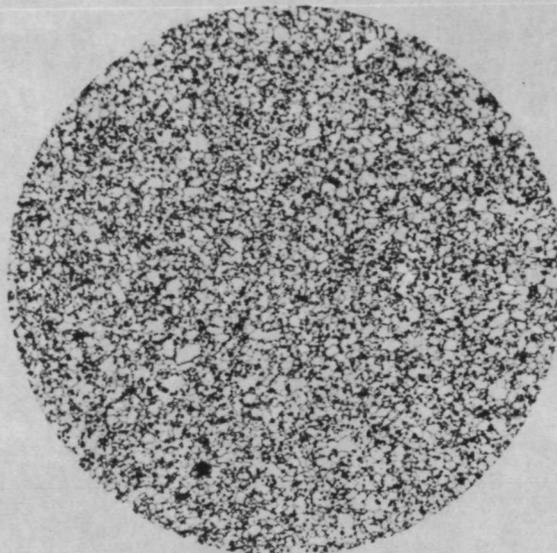
Figure 5. - Tensile specimen after fracture.

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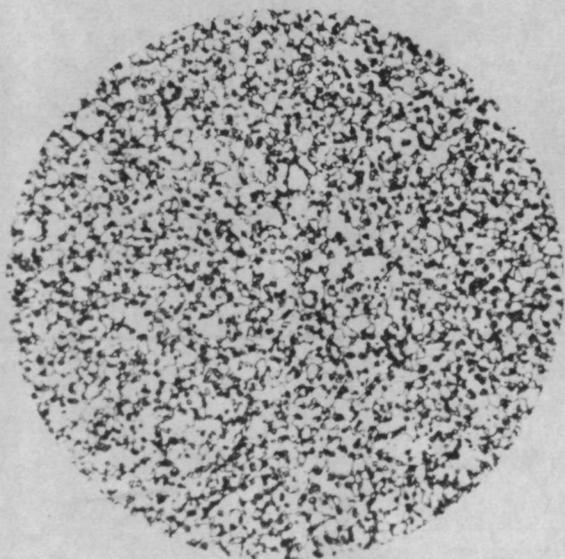


Uniform powder

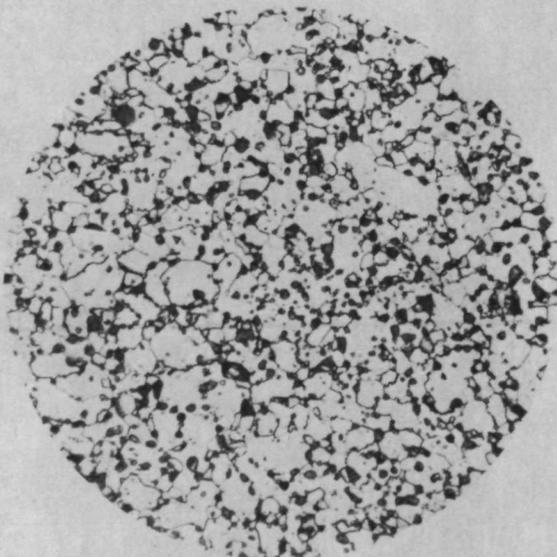


Nonuniform powder

(a) X150.



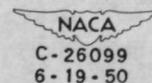
Uniform powder



Nonuniform powder

(b) X500.

Figure 6. - Hot pressed molybdenum disilicide bodies; etchant, 17-percent hydrochloric and 8-percent nitric acid; etching time, 70 minutes.

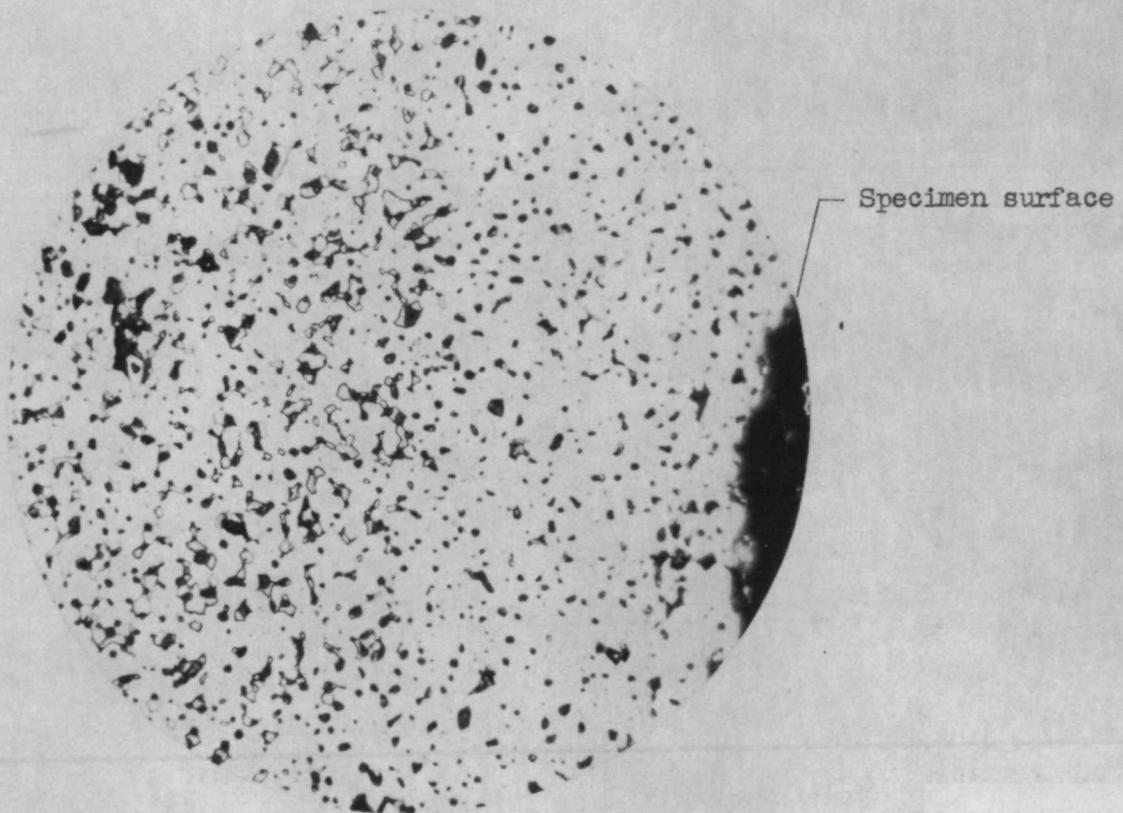


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Figure 7. - Phase migration in hot-pressed molybdenum disilicide after 135 hours at 2850° F. Echant, 10-percent sodium hydroxide, 30-percent potassium ferricyanide; etching time, 30 seconds. X500.

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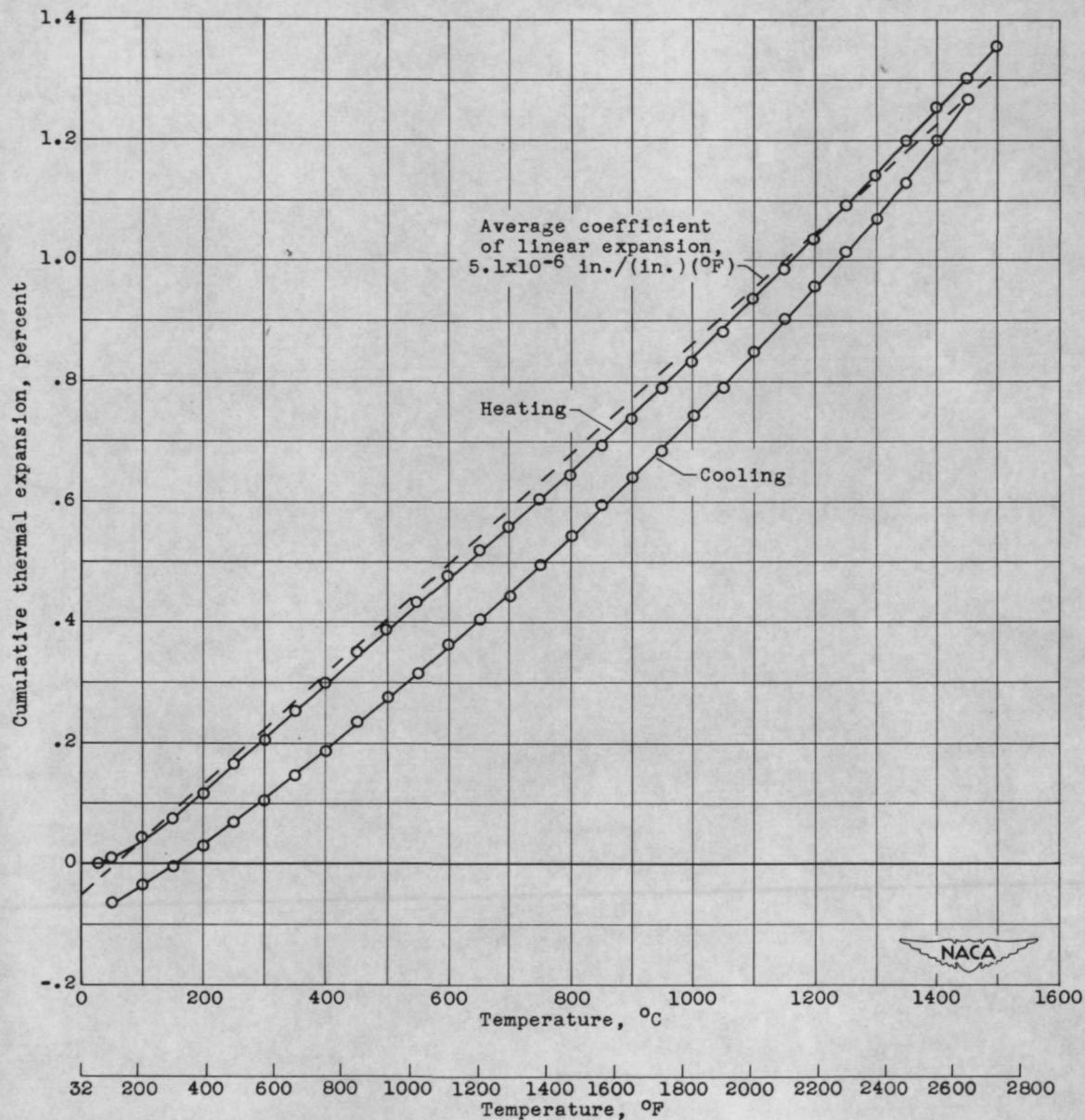


Figure 8. - Thermal expansion of hot-pressed molybdenum disilicide. Data furnished by National Bureau of Standards.

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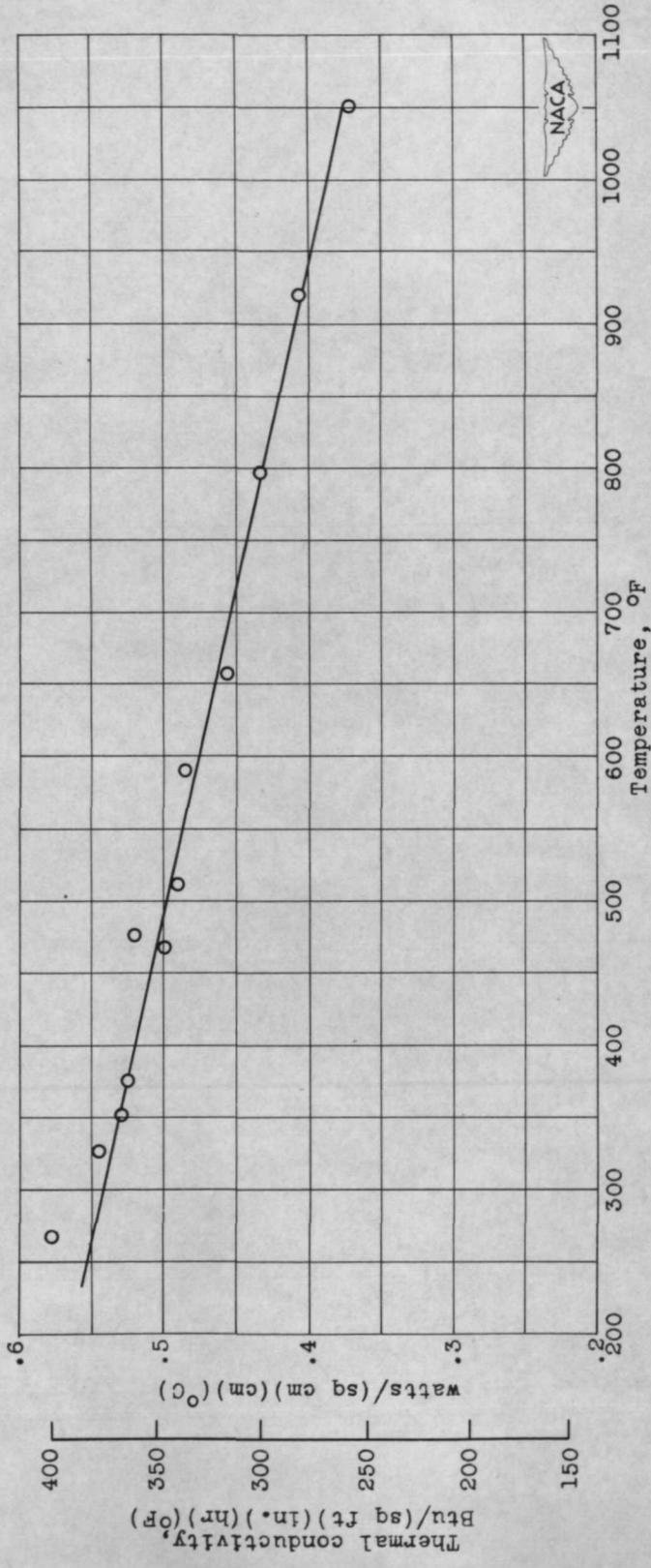


Figure 9. - Thermal conductivity of hot-pressed molybdenum disilicide at various temperatures.

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To Unclassified

By authority of Change No. 2327

7/29/54 Date same 12/2/54