THERMAL EXPANSION OF MOLYBDENUM.

By Peter Hidnert and W. B. Gero.

ABSTRACT.

Expansion tests were made for various temperature ranges between room temperature and 750° C. on molybdenum ingots prepared from fine and coarse grained molybdenum powders and on samples swaged to various diameters. The results are presented in figures and tables.

SERIES 1 TO 4 (MOLYBDENUM SAMPLES SWAGED TO VARIOUS DIAMETERS).—Some of the expansion curves on heating show marked changes in the rate of expansion at about 350° C. In most cases the plotted observations on cooling lie above the expansion curves on heating, so that after the expansion tests the specimens were longer than before these tests. For the samples prepared from fine-grained molybdenum powder the average coefficients of expansion between 25 and 500° C. vary from 5.4×10^{-6} to 5.8×10^{-6} per degree centigrade, and for the samples prepared from coarse-grained powder the coefficients vary from 4.7×10^{-6} to 5.7×10^{-6}.

SERIES 5 (SWAGED AND ANNEALED SAMPLES).—The annealing was done in hydrogen gas at 1,500° C. The coefficients of expansion of the annealed samples of molybdenum are larger than the coefficients of the worked specimens. Several photomicrographs showing the structural changes that occurred in the samples of this series are included.

SERIES 6 (TESTS AT HIGH TEMPERATURES).—Forming gas was used for a neutral atmosphere in a high-temperature furnace in two expansion tests on a sample of molybdenum. The annealing incident to the first test had no appreciable effect on the coefficients of expansion of the second test.

CONTENTS.

I. INTRODUCTION.

Molybdenum and tungsten are both used for the manufacture of seals through hard glass of the pyrex variety. The fact that molybdenum can be readily machined while tungsten can not

makes the former the obvious choice for many purposes. The properties of molybdenum are such that it is definitely associated with high-temperature work. Examples of its uses are parts of radio tubes, terminal seals for mercury-arc rectifiers, and supports for filaments of incandescent lamps. In all these uses expansion is an important consideration.

In 1919 some preliminary results were published [1] on the thermal expansion of molybdenum (99.85 per cent). Work done at the Westinghouse Lamp Co. indicated that the different methods of preparing molybdenum ingots gave a wide variation in expansion of the finished product, and that the heat treatment had little or no effect on the expansion of molybdenum. The factors which seemed to influence this expansion are rate of reduction of the oxide and method of preparing the molybdenum ingots.

In the present investigation thermal-expansion tests were made on molybdenum ingots (0.625 inch diameter) prepared from fine-grained powder and coarse-grained powder, respectively. After tests on these ingots they were reduced to 0.250, 0.175, and 0.100 inch. Thermal-expansion tests were made after each reduction. Most of the samples were examined from room temperature to about 500° C. In expansion tests exceeding 500° C. a neutral atmosphere was used to prevent oxidation.

The samples and photomicrographs were prepared at the Westinghouse Lamp Co., Bloomfield, N. J., and the thermal expansion determinations were made at the Bureau of Standards.

The authors are indebted to Dr. W. Souder, L. W. Schad, and H. S. Rawdon for valuable suggestions; to W. T. Sweeney for assistance in the tests, and to E. S. Davenport for the preparation of the micrographs.

II. MATERIALS INVESTIGATED.

The samples investigated are grouped into six series, as given in the following table:

[1] B. S. Sci. Paper, No. 332.

TABLE 1.—Classification of Materials.

Series.	Laboratory number.	Description.
1	S516	Hexagonal molybdenum ingot, 0.625 inch diameter, prepared from fine-grained molybdenum powder.
	S516A	S516 after the thermal-expansion test and swaged to 0.250 inch diameter.
	S516B	S516A after the thermal-expansion test and swaged to 0.175 inch diameter.
	S516C	S516B after the thermal-expansion test and swaged to 0.100 inch diameter.
2	S515[1]	Hexagonal molybdenum ingot, 0.625 inch diameter, prepared from coarse-grained powder.
3	S540	Hexagonal molybdenum ingot, 0.625 inch diameter, prepared from coarse-grained powder.
	S540A	S540 after the thermal-expansion test and swaged to 0.250 inch diameter.
	S540B	S540A after the thermal-expansion test and swaged to 0.175 inch diameter.
	S540C	S540B after the thermal-expansion test and swaged to 0.100 inch diameter.
4	S603[2]	Hexagonal molybdenum ingot, 0.625 inch diameter, prepared from coarse-grained powder.
	S603A	S603 swaged to 0.250 inch diameter.
	S603B	S603A after the thermal-expansion test and swaged to 0.175 inch diameter.
	S603C	S603B after the thermal-expansion test and swaged to 0.100 inch diameter.
5	S1052	Specimen, 0.100 inch diameter, prepared from fine-grained powder.
	S1053	Specimen, 0.100 inch diameter, prepared from coarse-grained powder.
6	S516A′	Sample, 0.250 inch diameter, cut from same rod as S516A (series 1).

[1] After the expansion test sample failed to work down to 0.250 inch diameter.
[2] Not tested for expansion.

The preparation of the hexagonal molybdenum ingots of series 1 to 4, inclusive, was as follows: The ingots, 8 inches in length, were prepared by compressing molybdenum powder, resulting from the reduction of oxide under a pressure of about 30,000 lbs./in.^{2} After heating for 15 or 20 minutes at 1,500° C. in hydrogen for sintering the bars each ingot was then treated in an atmosphere of hydrogen at 80 per cent of the fusion current for 15 minutes, which caused the ingot to shrink about 25 per cent in the longitudinal direction and approximately 20 per cent in cross section. In this condition it exhibits properties of a brittle metal, but it is ductile enough to be machined.

After determination of the expansion from room temperature to 500° C. each ingot was reduced by swaging to a diameter of 0.250 inch. After determination of the expansion of the resulting bars they were further reduced by swaging to a diameter of 0.175 inch. This process was repeated, as shown in Table 1.

The temperatures to which the rods were heated before swaging for each size tested were approximately as follows:

TABLE 2.—Working Temperatures of Molybdenum.

Diameter in inches.	Temperatures.
	° C.
0.250	1,125 to 1,150
.175	1,050 to 1,100
.100	1,000 to 1,150

The actual temperature of working was somewhat below these temperatures due to the heat loss, the size of the rod, and the speed of the machine. In any case it is safe to assume that the actual working temperature was about 900° C. even for the smallest size.

The samples of series 5 (S1052 and S1053) were prepared relatively late in the investigation. The preparation which was slightly different from that outlined above, was as follows:

Three hundred and fifty grams of the powdered metal was pressed in a mold having an opening 8 by 0.75 inch square, at a pressure of 16 tons/in.[2] The approximately square ingot so formed was heated for 30 minutes at about 1,600° C. This treatment caused the ingot to shrink and made it more resistant to breakage by handling. The ingot was then placed in an atmosphere of hydrogen, and a current of 3,700 amperes was passed through it for 15 minutes, which caused an additional shrinkage. The ingots were then found to have the following dimensions: S1052, 6.40 by 0.598 by 0.599 inches; S1053, 6.80 by 0.540 by 0.626 inches. The working temperatures were as follows:

TABLE 3.—Working Temperatures of Molybdenum.

Size (diameter in inches).	Temperature. range.	Number of dies.
	° C.	
Ingot to 0.360	1,150 to 1,120	8
0.340 to 0.220	1,050 to 1,020	7
0.190 to 0.160	800 to 650	3
0.145 to 0.100	600 to 500	4

For sizes from the maximum to 0.220 inch diameter a Leeds and Northrup optical pyrometer was used to determine temperatures. Since this instrument was found impractical for smaller sizes under the prevailing conditions, the temperatures of the samples below 0.220 inch diameter were estimated by color.

The molybdenum ingots of series 1 to 4 indicated a Rockwell hardness[2] between B–75 and B–80. In series 5, specimen 1,052 (prepared from fine-grained powder) indicated a Rockwell hardness of B–82, and sample 1,053 (prepared from coarse-grained powder) showed a hardness of B–79 on the same scale.

It should be noted that the terms "fine grained" and "coarse grained" refer not to the sintered ingot, but to the state of the molybdenum metal powder after reduction from its oxides and before pressing into ingots. The material is usually "coarsened" in the process of reduction from molybdenum oxide by raising the temperature too rapidly during reduction, or by limiting the

[2] Rockwell, Trans. Am. Soc., Steel Treating, 2, p. 1013; 1922.

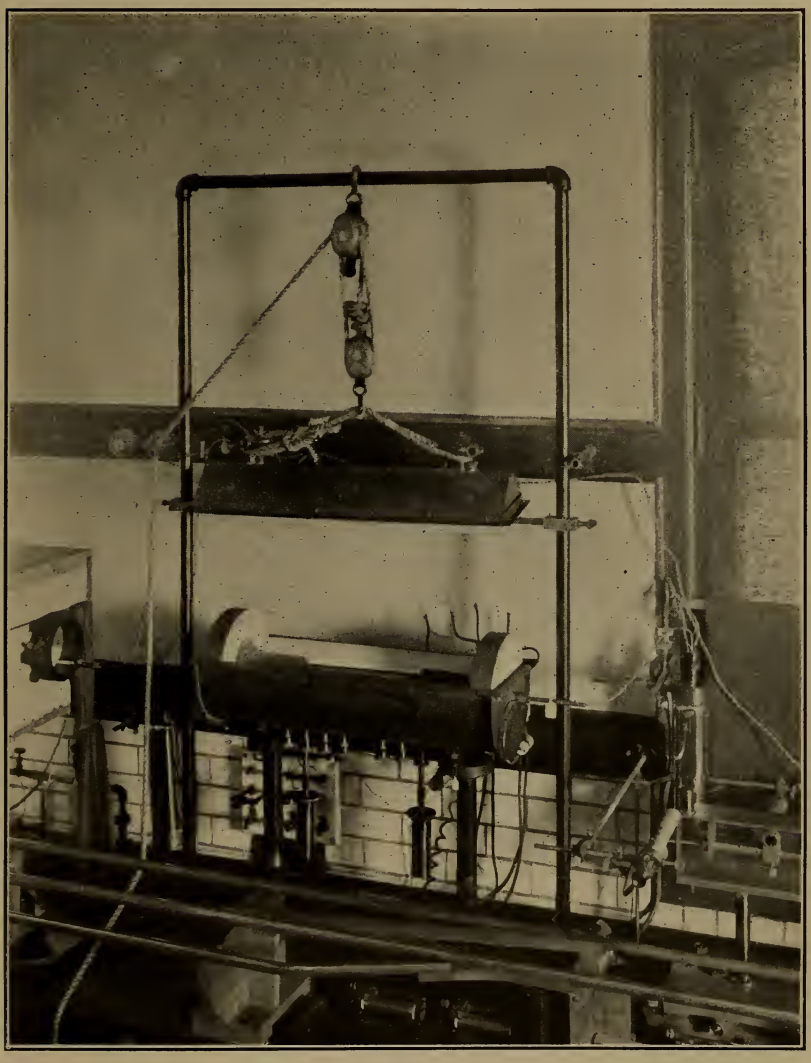

FIG. 1.—*Electrically heated furnace.*

supply of hydrogen gas or other reducing agent, or by certain impurities which may be present. After such "coarsening" takes place, although its apparent effects may be removed by later reduction operations, it is always a very difficult material to handle and can not be easily drawn to fine wire or rolled into sheet.

The molybdenum ingots of series 2 and 4 were prepared from the same coarse-grained powder. The ingot of series 3 was prepared from the same batch of oxide as series 2 and 4, but was reduced at a different time. The ingot of series 1 and the ingots from which the samples of series 5 were obtained were prepared from different powders.

Table 4 gives the chemical composition [3] of some of the specimens of molybdenum.

TABLE 4.—Constituents [1] of Molybdenum Samples.

Laboratory number.	Constituents.				
	Fe	Si	Ca	Cu	W
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
S516A	0.03	0.03	.....	.....	1.85
S516B	.05	.014	<0.005	.....	1.80
S515	.04	.003	.....	.....	.....
S540B	.11	.003	.....	0.014	.....
S540C	.03	.004	.....	.006	.....
S603A	.10	.017	<.005	.....	.....
S603C	.11	.007	.010	.005	.....
S1052	.015	.002	.....	.....	.....
S1053	.010	.004	.....	.....	.....

[1] Sulphur not detected in any of the samples; test sensitive to 0.003 per cent; check determinations on total molybdenum agreed within the experimental limits with the molybdenum values obtained by difference.

III. APPARATUS.

The electrically heated furnace (with the top raised) shown in the accompanying figure was used in making the thermal-expansion tests from room temperature to about 500° C. A detailed description of the construction of this furnace was given in Scientific Paper of the Bureau of Standards No. 219. The furnace has been modified somewhat since this paper appeared.

For temperatures exceeding 500° C. the furnace illustrated in Scientific Paper of the Bureau of Standards No. 352 was employed. This paper also gives the method used in making observations on thermal expansion.

Pt–PtRh thermocouples were employed in determining the temperatures at both ends and at the center of each sample of molybdenum.

[3] Determined by H. A. Bright and W. C. Fedde, of this bureau.

IV. EXPERIMENTAL AND DERIVED RESULTS.

1. EXPANSION OF MOLYBDENUM SWAGED TO DIFFERENT DIAMETERS (SERIES 1 TO 4).

In these series (1 to 4) the specimens were prepared from fine and coarse grained molybdenum powders, respectively, and were investigated from room temperature to about 500° C. The expansion observations obtained on all the samples heated to about 500° C. are represented graphically in Figures 2 to 5, inclusive.

The expansion curves of S516A, S516C, S540, S540A, S540C, and S603A on heating show marked changes in the rate of expansion at about 350° C. and the curves of S515, S540B, and S603C are irregular. In most cases the plotted observations on cooling lie above the expansion curves on heating, so that after the thermal-expansion tests the specimens were longer than before these tests. The maximum deviation between the curves on heating and cooling was found to be 450 millionths per unit length. In general, the curves on cooling are more regular than those on heating.

From the expansion curves shown in Figures 2 to 5, inclusive, the average coefficients of expansion given in Table 5 were computed for various temperature ranges. This table also shows the changes in length from the original lengths after the thermal-expansion tests. The plus (+) sign indicates an increase in length and the minus (−) sign a decrease in length.

TABLE 5.—Average Coefficients of Expansion and Changes in Length.

Series.	Laboratory number.	Average coefficients of expansion per degree centigrade.								Change in length after test.
		25 to 100°C.	100 to 200°C.	200 to 300°C.	300 to 400°C.	400 to 500°C.	25 to 250°C.	250 to 500°C.	25 to 500°C.	
		×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	Per ct.
[1]	S516	5.4	5.1	5.2	5.6	5.7	5.3	5.5	5.4	−0.010
	S516A	4.9	5.1	5.4	5.7	7.4	5.1	6.3	5.8	−.001
	S516B	4.9	5.1	5.2	5.4	6.2	5.1	5.7	5.4	−.007
	S516C	4.6	4.8	5.2	5.9	6.4	4.8	6.0	5.4	+[2].022
[3]	S515	3.7	5.5	4.8	5.8	5.8	4.6	5.7	5.2	−[2].009
[3]	S540	5.0	5.0	5.2	5.0	3.4	5.1	4.4	4.7	−.036
	S540A	4.8	4.8	5.2	6.4	7.0	4.9	6.4	5.7	+.009
	S540B	4.5	4.6	5.1	5.1	6.5	4.7	5.6	5.2	+.017
	S540C	4.5	4.3	4.4	6.1	7.2	4.4	6.2	5.3	+.012
[3]	S603 [4]									
	S603A	4.6	4.9	5.2	6.5	7.0	4.8	6.5	5.7	+.026
	S603B	4.8	5.1	5.4	5.9	6.7	5.0	6.1	5.6	+.002
	S603C	3.7	4.7	4.9	4.9	5.8	4.5	5.2	4.9	+.043

[1] Prepared from fine-grained molybdenum powder.
[2] At any given temperature above room temperature the deviation between the expansion curves on heating and cooling did not exceed this value.
[3] Prepared from coarse-grained molybdenum powder.
[4] Sample S603 not tested.

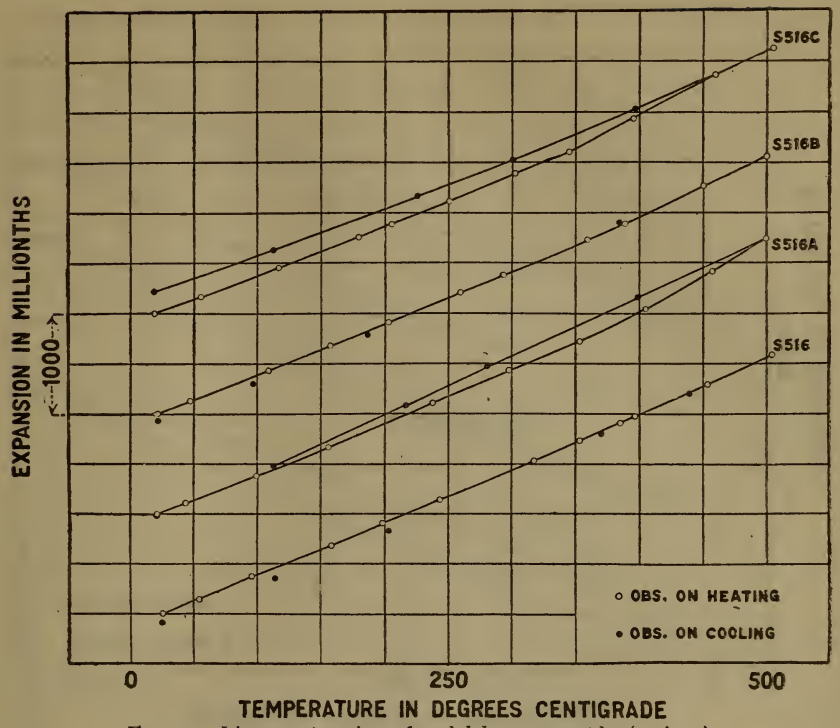

FIG. 2.—*Linear expansion of molybdenum samples (series 1).*

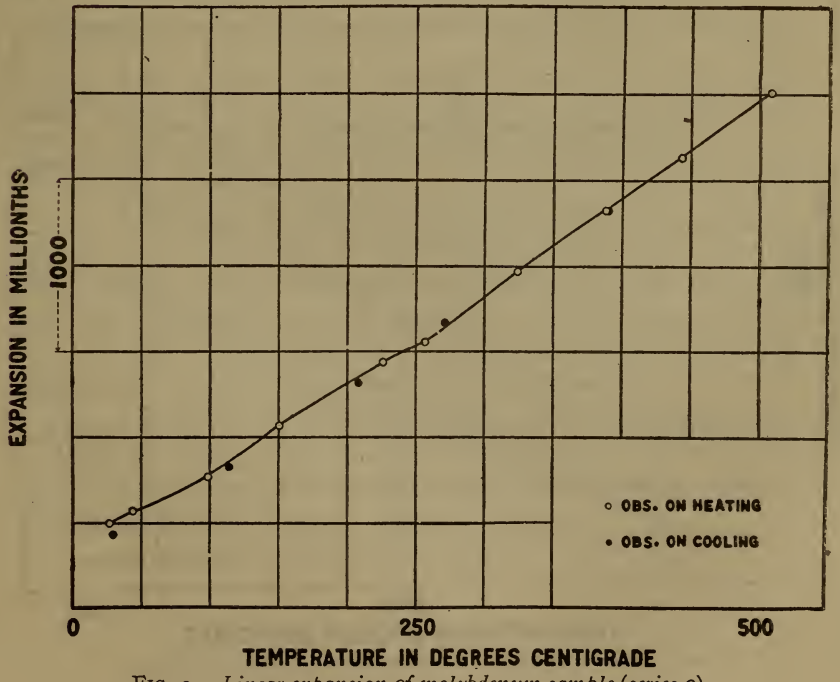

FIG. 3.—*Linear expansion of molybdenum sample (series 2).*

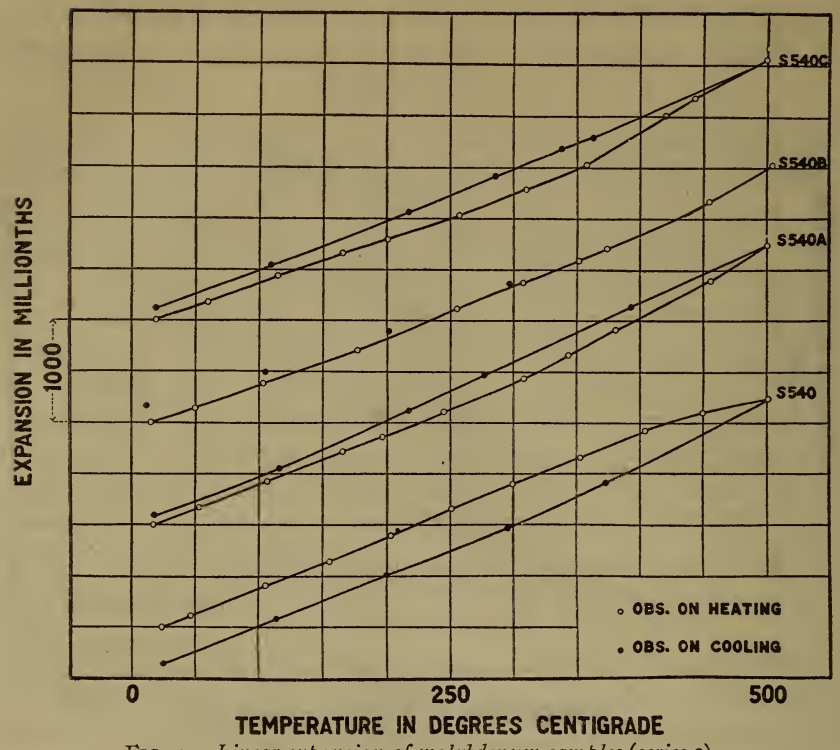

FIG. 4.—*Linear expansion of molybdenum samples (series 3).*

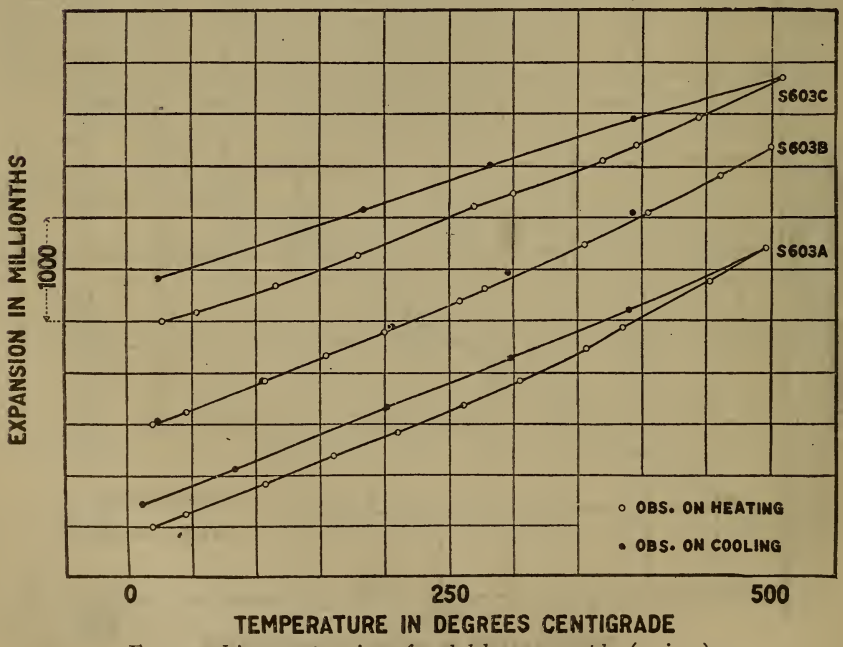

FIG. 5.—*Linear expansion of molybdenum samples (series 4).*

From an examination of the expansion curves and the preceding table it is evident that the coefficients of expansion do not increase regularly with temperature. For the samples prepared from fine-grained molybdenum powder the average coefficients of expansion between 25 and 100° C. vary from 4.6×10^{-6} to 5.4×10^{-6} per degree centigrade, and for the samples prepared

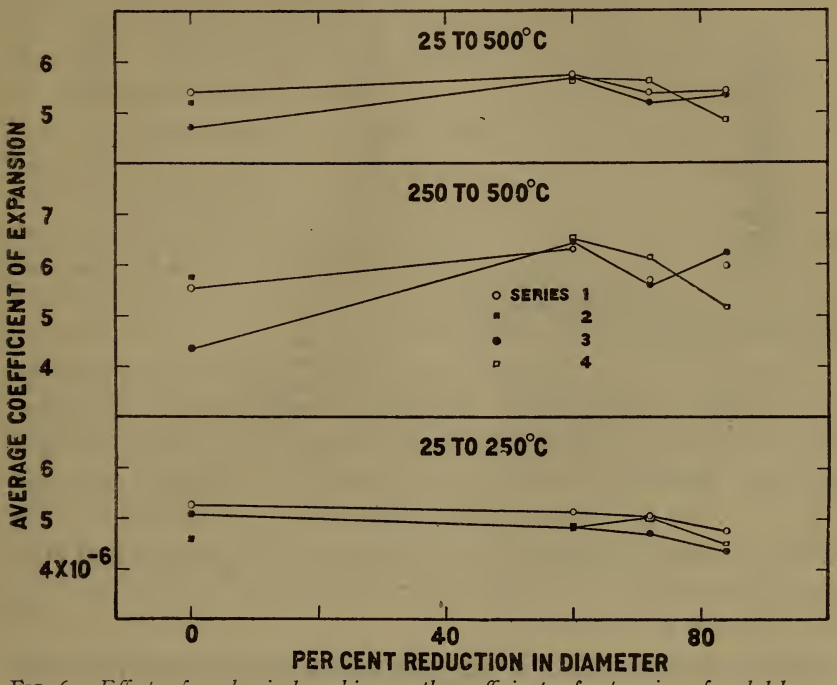

FIG. 6.—*Effects of mechanical working on the coefficients of expansion of molybdenum.*

from coarse-grained powder the coefficients vary from 3.7×10^{-6} to 5.0×10^{-6}. For the range from 25 to 500° C. the coefficients of the former samples vary from 5.4×10^{-6} to 5.8×10^{-6}, and the coefficients of the latter vary from 4.7×10^{-6} to 5.7×10^{-6}. Comparisons at other temperature ranges may easily be made from the data given in the preceding table.

Figure 6 shows the relations between the percentage reduction in diameter of the molybdenum samples and the average coefficients of expansion for several temperature ranges. The initial diameter (0 per cent reduction) is 0.625 inch.

The following table gives a comparison of the average coefficients of expansion and contraction for several temperature ranges:

TABLE 6.—Comparison of Coefficients on Heating and Cooling.

Series.	Laboratory number.	Average coefficients of expansion per degree centigrade.			Average coefficients of contraction per degree centigrade.		
		25 to 250° C.	250 to 500° C.	25 to 500° C.	500 to 25° C.	500 to 250° C.	250 to 25° C.
		$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$
1 [1]	S516	5.3	5.5	5.4	5.6	5.8	5.3
	S516A	5.1	6.3	5.8	5.8	5.9	5.6
	S516B	5.1	5.7	5.4	5.5	5.8	5.3
	S516C	4.8	6.0	5.4	5.0	5.3	4.6
2 [2]	S515	4.6	5.7	5.2	5.4	5.7	5.1
3 [2]	S540	5.1	4.4	4.7	5.5	6.0	4.9
	S540A	4.9	6.4	5.7	5.5	5.7	5.3
	S540B	4.7	5.6	5.2	4.8	5.5	4.1
	S540C	4.4	6.2	5.3	5.1	5.3	4.8
4 [2]	S603 [3]						
	S603A	4.8	6.5	5.7	5.2	5.3	4.9
	S603B	5.0	6.1	5.6	5.6	6.0	5.1
	S603C	4.5	5.2	4.9	4.0	3.8	4.2

[1] Prepared from fine-grained mlybdenum powder.
[2] Prepared from coarse-grained molybdenum powder.
[3] Sample S603 not tested.

In most cases the coefficient of contraction is less than the corresponding coefficient of expansion. This is to be expected, since it was found previously that the curve on cooling in most cases lies above the corresponding curve on heating (see figs. 2 to 5). For the temperature range from 25 to 250° C., the average variation between the coefficients of expansion and contraction is $\pm 0.3 \times 10^{-6}$, and for the range from 250 to 500° C., the variation is $\pm 0.6 \times 10^{-6}$.

2. EFFECT OF ANNEALING ON EXPANSION OF SWAGED MOLYBDENUM (SERIES 5).

This series gives data on the expansion of cold-worked and annealed samples of molybdenum prepared from fine-grained powder and coarse-grained powder, respectively. The cold-worked specimens (S1052 prepared from fine-grained powder and S1053 prepared from coarse-grained powder) were 0.100 inch in diameter and prepared at the same time under similar conditions.

The method of procedure in the expansion-tests was as follows: (1) Expansion tests of the cold-worked samples from room temperature to 300° C. (below annealing temperature). (2) Expansion determinations on same specimens from room temperature to a temperature (500° C.) sufficiently high so that the samples might be partially annealed by the heat incidental to the tests. (3) Expansion tests from room temperature to 500° C. on same specimens after annealing at about 1,500° C.

For annealing each sample an electric furnace was brought to temperature and the specimen inserted. Hydrogen gas was employed as a neutral atmosphere. The specimen was first held at approximately 1,150° C. for 15 minutes and then pushed into a cooling chamber. The microstructure of the sample was examined after cooling. This procedure was repeated at 1,280° C. for 15 minutes, at 1,320° C. for 30 minutes, and finally at 1,500° C. for 30 minutes. Annealing started at the first treatment, but was not complete until a temperature of approximately 1,500° C. had been reached. Doubtless, if sufficient time had been given at lower temperatures the same results would have been accomplished.

The expansion curves of the worked samples of molybdenum were found to be fairly regular, except the curve of S1053 during the second test (room temperature to 500° C.), which showed an irregularity at about 350° C. In the annealed specimens the expansion curves are also regular, but the rates of expansion are generally greater than for the worked samples. In all tests (worked or annealed specimens) the plotted obsevations on cooling were found to be near the expansion curves on heating. The maximum deviation between the curves on heating and cooling does not exceed 40 millionths per unit length.

From the expansion curves the average coefficients of expansion given in the following table were derived for various temperature ranges:

TABLE 7.—Average Coefficients of Expansion of Worked and Annealed Molybdenum.

Temperature range (° C.).	Average coefficients of expansion per degree centigrade.					
	S1052 (prepared from fine-grained powder).			S1053 (prepared from coarse-grained powder).		
	Cold worked.		Annealed.[1]	Cold worked.		Annealed.[1]
	First test.	Second test.		First test.	Second test.	
	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶
25 to 100	5.0	4.3	5.2	4.7	4.8	5.3
100 to 200	5.3	5.1	5.4	5.0	5.0	5.6
200 to 300	5.3	5.3	5.5	5.0	5.1	5.6
300 to 400	.....	5.5	5.6	.....	4.9	5.8
400 to 500	.....	6.1	5.9	.....	5.8	6.7
25 to 250	5.2	4.9	5.3	4.9	5.0	5.5
250 to 500	.....	5.7	5.7	.....	5.3	6.1
25 to 500	.....	5.3	5.5	.....	5.1	5.8

[1] Expansion data obtained by Fizeau-Pulfrich method.

Before the data on the annealed samples given in the preceding table were obtained the annealed specimens were heated to 500° C. and showed indications of bending when the 300 mm samples were supported horizontally in the usual manner during an expansion test. Expansion tests on small sections of these specimens were, therefore, made [4] by a different method—namely, the Fizeau-Pulfrich method [5]—and the results secured are included in the preceding table.

The coefficients of expansion of the annealed samples of molybdenum are larger than the coefficients of the worked specimens. The coefficients of expansion of the annealed sample prepared from coarse-grained powder are greater than the coefficients of the annealed specimen prepared from fine-grained powder.

MICROSTRUCTURAL CHANGES.

The accompanying photomicrographs (figs. 7 and 8) show the microstructural changes that occurred in the samples of this series. Sections of these samples were polished in a plane parallel to the longitudinal axis of the rod. Each specimen was ground down to about the middle of the rod. The polished specimen was electrolytically etched in a 2.5 per cent aqueous NaOH solution to which had been added a few drops of H_2O_2. The specimen was used as anode, while a tungsten ingot served as cathode. A current of about 0.5 ampere for 10 to 20 seconds usually brought out the structure.

Figures 7(*a*) and 8(*a*) show that the swaged samples of molybdenum prepared from fine and coarse grained powders, respectively, were extremely cold worked before the expansion tests. The heat treatment incidental to the two expansion tests, from room temperature to 300° C. and then from room temperature to 500° C., caused no structural alteration in either material (figs. 7(*b*) and 8(*b*)). Before the next expansion test the samples were completely annealed, as shown by Figures 7(*c*) and 8(*c*). After the expansion tests on the annealed samples no appreciable change in structure is seen (figs. 7(*d*) and 8(*d*)).

After annealing a marked structural difference between the specimens manufactured from coarse and fine grained powders was observed. Later trials when both materials were annealed at the same time in the same furnace confirmed this phenomenon.

[4] By G. E. Merritt of this bureau.
[5] Described in B. S. Sci. Paper, No. 393.

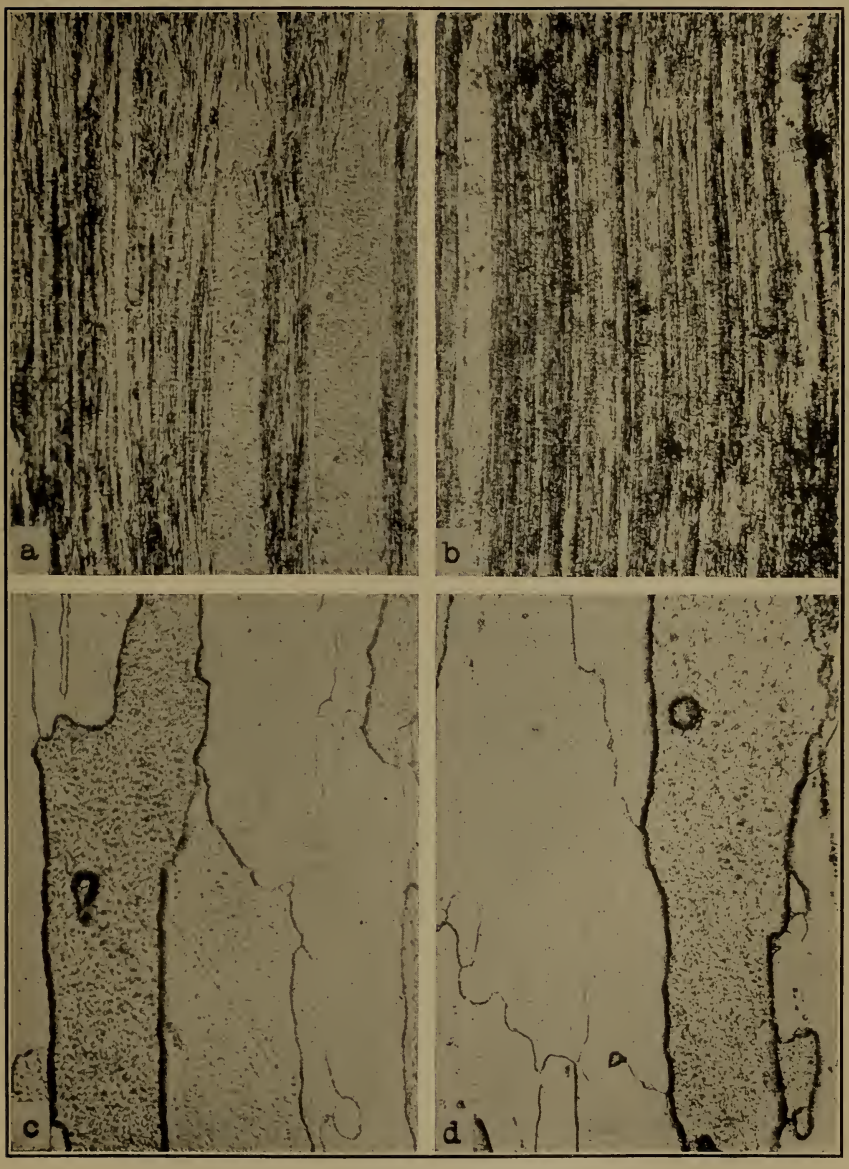

FIG. 7.—*Microstructure of molybdenum prepared from fine-grained powder (S1052).* × 200.

Iron 0.015, silicon 0.002, molybdenum 99.983 (by difference). (*a*) Swaged rod, (*b*) after two expansion tests to 500° C., (*c*) annealed at 1,500° C., (*d*) after final expansion test.

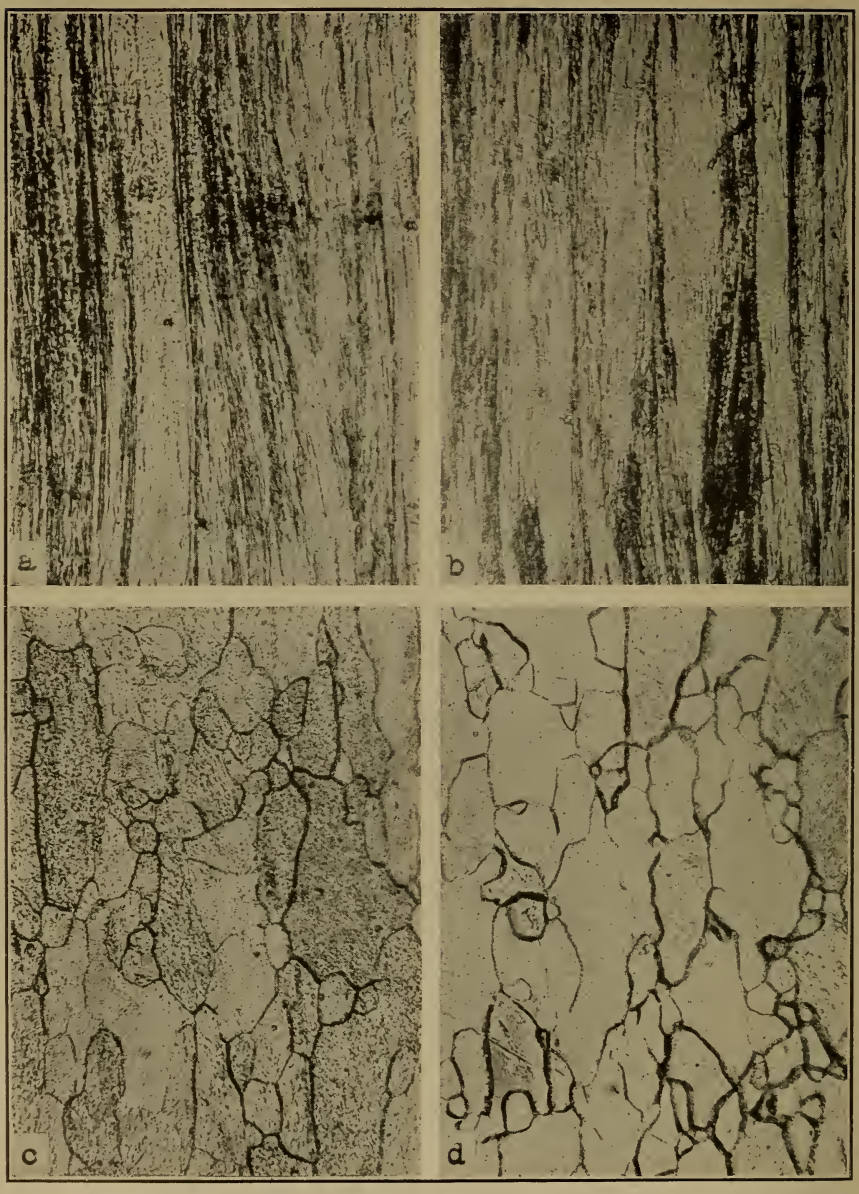

FIG. 8.—*Microstructure of molybdenum prepared from coarse-grained powder (S 1053).* × *200.*

Iron 0.010, silicon 0.004, molybdenum 99.986 (by difference). (*a*) Swaged rod, (*b*) after two expansion tests to 500° C., (*c*) annealed at 1,500° C., (*d*) after final expansion test.

3. EXPANSION MEASUREMENTS AT HIGH TEMPERATURES (SERIES 6).

In order to obtain some data on the thermal expansion of molybdenum above 500° C., additional tests were made on a specimen (S516A′) cut from the same rod as S516A prepared from fine-grained powder. Forming gas (approximately 93 per cent nitrogen and 7 per cent hydrogen) was used for a neutral atmosphere in the high-temperature furnace. Since this gas contained moisture and traces of oxygen, it was first passed over heated copper contained in an iron tube and then through two bottles of concentrated sulphuric acid.

The results of two thermal expansion tests on the molybdenum sample S516A′ are given in Figure 9 and the following table:

TABLE 8.—Average Coefficients of Expansion for Various Temperature Ranges Between 25 and 750° C.

Temperature range (°C.).	Average coefficient of expansion per degree centigrade.	
	First heating.	Second heating.
	×10⁻⁶	×10⁻⁶
25 to 100	5.1	5.2
100 to 200	5.4	5.5
200 to 300	5.5	5.5
300 to 400	5.6	5.9
400 to 500	6.0	5.9
500 to 600	6.0	6.2
600 to 700	6.4	6.4
25 to 250	5.3	5.4
250 to 500	5.7	5.8
25 to 500	5.5	5.6
500 to 700	6.2	6.3
500 to 750	.....	6.3

In the first test an observation wire broke at about 780° C., and in the second test both observation wires broke above 760° C., probably due to the effect of forming gas on these wires which were made from an alloy of platinum and osmium. In air it is possible to heat these wires to 1,000° C. without difficulty.

It is interesting to note that the coefficients of expansion on the second heating agree closely with those on the first heating. This indicates that the annealing incident to the first thermal-expansion test had no appreciable effect.

A comparison of the average coefficients of expansion of the first heating with those of S516A (series 1) shows fairly good

agreement, except for the temperature ranges between 400 and 500° C. and from 250 to 500° C. This indicates that these two samples, which were cut from the same rod, were probably not homogeneous, or, since the methods differ, the surrounding atmosphere might have caused an effect at the high-temperature range (400 to 500° C.).

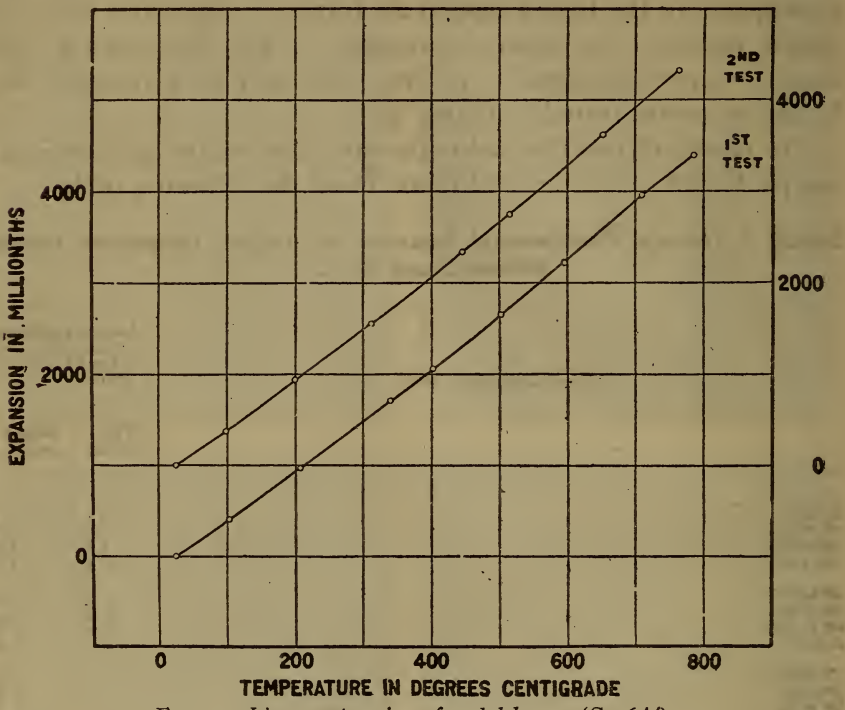

FIG. 9.—*Linear expansion of molybdenum (S516A').*

V. SUMMARY AND CONCLUSIONS.

A study was made of the linear thermal expansion of molybdenum prepared from fine and coarse grained molybdenum powder, respectively. Expansion tests were made on molybdenum ingots (0.625 inch diameter) and on samples swaged to 0.250, 0.175, and 0.100 inch diameter, respectively. Most of the specimens were examined from room temperature to about 500° C.

SERIES 1 TO 4 (MOLYBDENUM SAMPLES SWAGED TO VARIOUS DIAMETERS).—The expansion observations obtained on all the samples heated to about 500° C. are represented graphically in figures. Some of the expansion curves on heating show marked

changes in the rate of expansion at about 350° C. In most cases the plotted observations on cooling lie above the expansion curves on heating, so that after the expansion tests the specimens were longer than before these tests.

The coefficients of expansion do not increase regularly with temperature. For the samples prepared from fine-grained molybdenum powder the average coefficients of expansion between 25 and 100° C. vary from 4.6×10^{-6} to 5.4×10^{-6} per degree centigrade, and for the samples prepared from coarse-grained powder, the coefficients vary from 3.7×10^{-6} to 5.0×10^{-6}. For the range from 25 to 500° C., the coefficients of the former samples vary from 5.4×10^{-6} to 5.8×10^{-6}, and the coefficients of the latter vary from 4.7×10^{-6} to 5.7×10^{-6}.

The relations between the percentage reduction in diameter of molybdenum specimens and the average coefficients of expansion for several temperature ranges are shown in Figure 6. In most cases the coefficient of contraction is less than the corresponding coefficient of expansion. For the temperature range from 25 to 250° C., the average variation between the coefficients of expansion and contraction is $\pm 0.3 \times 10^{-6}$, and for the range from 250 to 500° C., the variation is $\pm 0.6 \times 10^{-6}$.

Series 5 (Swaged and Annealed Samples).—Data on the expansion of several cold worked and annealed samples (0.100 inch diameter) are presented. The annealing was done in hydrogen gas at 1,500° C. The coefficients of expansion of the annealed samples of molybdenum are larger than the coefficients of the worked specimens. Several photomicrographs (figs. 7 and 8) showing the structural changes that occurred in the samples of this series are included.

Series 6 (Tests at High Temperatures).—Additional tests were made on a specimen (0.250 inch diameter) of molybdenum from room temperature to about 750° C. Forming gas was used for a neutral atmosphere in a high-temperature furnace. The results of two expansion tests are given in Figure 9 and Table 8. The annealing incident to the first expansion test had no appreciable effect on the coefficients of expansion of the second test.

Since molybdenum manufactured from fine-grained powder is more easily fabricated by means of rolling, swaging, or drawing, it is probable that most commercial molybdenum will more nearly approach the "fine" rather than the "coarse" material in prop-

erties. The authors, therefore, give the following average values for the coefficients of expansion of commercial molybdenum:

4.9×10^{-6} for the range 25 to 100° C. and

5.5×10^{-6} for the range 25 to 500° C.

Variations from these values are to be expected, due to variations in grain size of the powder from which the samples are manufactured, degree of mechanical working, or annealing.

WASHINGTON, January 25, 1924.