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**DYNAMIC PROPERTIES LABORATORY**

THE INITIATION OF DISCONTINUOUS YIELDING  
IN DUCTILE MOLYBDENUM

by

J. A. Hendrickson, D. S. Wood, and D. S. Clark

62

A REPORT ON RESEARCH CONDUCTED UNDER  
CONTRACT WITH THE OFFICE OF NAVAL RESEARCH

October 1954

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THE INITIATION OF DISCONTINUOUS YIELLING  
IN DUCTILE MOLYBDENUM

By

J. A. Hendrickson, D. S. Wood, and D. S. Clark

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THE INITIATION OF DISCONTINUOUS YIELDING  
IN DUCTILE MOLYBDENUM

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### ABSTRACT

This report presents the results of an experimental investigation of the initiation of yielding in fine grained ductile molybdenum under rapidly applied constant stress. Two lots of material, one produced by arc-casting and the other by powder metallurgy methods were investigated. The results show that both materials exhibit the phenomenon of delayed yielding as well as distinct yield points in their static stress-strain relations. The delay time for yielding was determined as a function of stress at temperatures of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ),  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ), and  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ), and measurements of pre-yield plastic strain were obtained.

The stress vs. delay time relations obtained do not exhibit a lower limiting stress (constant stress portion at low stress and long delay time) such as that exhibited in normal low carbon steel. This behavior of the molybdenum is attributed to the rather low concentrations of carbon and nitrogen in the material tested, and is consistent with the previously observed behavior of steel containing comparably low concentrations of these elements. An upper limiting stress (constant stress portion at high stress and short delay time) is observed in one of the stress vs. delay time relations found for molybdenum. For a given general range of delay times this is observed at a higher temperature than that at which the same behavior has been found in steel in previous investigations. This result is consistent with the higher melting point and higher ductile to brittle transition temperature of molybdenum as compared to iron.

These results, together with others obtained in this investigation, indicate that the mechanism of the initiation of yielding in ductile molybdenum is substantially the same as for annealed low carbon steel. This lends further support to the dislocation theory of yield point phenomena which is based largely upon the concept of the anchoring of dislocations in body-centered cubic metals by interstitial solute atoms such as carbon and nitrogen.

## INTRODUCTION

An experimental technique has been developed in this laboratory which is particularly suited for the determination of the length of time during which a material may behave elastically at stresses in excess of the static upper yield stress. In this method a constant tensile stress is applied to a specimen and the deformation is measured as a function of time. The stress is increased from zero to its final value in a continuous manner within a total time of from 5 to 10 millisecc. The stress is then held constant for the remainder of the test.

Using this type of rapid loading, tests have been made previously on annealed low carbon steel (1)\*, (2), and (3). The results showed that a well defined period of time was required for the initiation of plastic deformation at stresses exceeding the static upper yield stress. This observed period of time has been defined as the "delay time for yielding".

The delay time for a given material depends upon the temperature of the material and the applied stress. For mild steel it is found that at any given temperature, the relation between the logarithm of the delay time and the applied stress may be represented by a straight line along which the logarithm of the delay time decreases as the stress is increased to values above the static upper yield stress. The logarithm of the delay time at a constant stress level is found to vary approximately as the inverse of the absolute temperature.

Delayed yielding has been shown previously to exist in single crystals of the body centered cubic metal beta brass as well as in mild steel (4). There has not been any evidence of delayed yielding in single crystals of the face centered cubic metals, alpha brass and aluminum (4).

---

\*Figures appearing in parenthesis refer to references listed at the end of this report.

A very small amount of inelastic strain has been found to occur in rapid load tests on annealed mild steel during the period of delay before yielding (5) (6). The rate at which this inelastic microstrain develops is greatest at the beginning of constant load and decreases until a microstrain of about  $30 \times 10^{-6}$  in./in. is present. After the strain has reached a value of this order of magnitude, yielding occurs. Such microstrains also occur when the test stress is less than the static upper yield stress but greater than about one-half that value. When stresses in this range are applied the microstrain increases with time and asymptotically approaches an equilibrium value which is a function of the applied stress and test temperature.

A theoretical model of a yield nucleus has been proposed (6), (7), which is capable of quantitatively describing the experimental observations of delayed yielding and pre-yield inelastic microstrain in terms of the generation and motion of dislocations. This model is based upon the concept of the thermally activated release of dislocations from carbon and nitrogen "atmospheres" as proposed by Cottrell and Bilby, (8), the Frank-Read dislocation source (9), and the obstruction of dislocation motion by grain boundaries proposed by Cottrell (10).

Carbon and nitrogen atoms in interstitial solid solution are believed to produce a shear distortion as well as a simple volume expansion of the body-centered cubic iron lattice. For this reason these atoms are capable of forming relatively tightly bound "atmospheres" around screw dislocations (11), (12), as well as edge dislocations in iron and steel, and thus give rise to the yield point phenomena. On the other hand solute atoms are not expected to produce appreciable shear distortions in face-centered cubic lattices, and hence will not form tightly bound "atmospheres" around screw dislocations. This then explains the fact that marked yield point phenomena are not observed in the face-centered cubic metals.

These theoretical considerations thus indicate that the phenomenon of delayed yielding should always be found for materials

that have the body-centered cubic crystal structure and that exhibit distinct upper and lower yield points in their static stress-strain relations. Therefore, it is of some interest to determine whether materials of the body-centered cubic type, other than iron and beta brass, exhibit delayed yielding and the associated pre-yield inelastic microstrain effects. One such material is molybdenum. Bechtold and Scott (13) have shown that fine grained ductile molybdenum exhibits distinct upper and lower yield points in its static stress-strain relation. The purpose of the present investigation is to determine if this material also exhibits delayed yielding under rapidly applied loads, and to compare its behavior with that of low-carbon steel over suitable ranges of applied stress and temperature.

#### MATERIAL TESTED AND TEST SPECIMEN

The material used in this investigation is high purity molybdenum, cold worked and completely recrystallized to obtain a uniform, fine grain structure. The investigations were made on molybdenum produced by two different processes. One type of molybdenum was produced by arc-casting and the other was produced by powder metallurgy methods, hereafter referred to as sintered molybdenum.

The arc-cast molybdenum was obtained from the Climax Molybdenum Company, who supplied the following chemical analysis:

##### Heat No. 4-1050

|          |                 |
|----------|-----------------|
| Carbon   | 0.033%          |
| Oxygen   | 0.003% (max.)*  |
| Nitrogen | 0.0002% (max.)* |

\*Based on actual analysis of other similar heats.

The sintered molybdenum was obtained from the Westinghouse Electric Corporation who supplied the following chemical analysis:

##### Lot No. WNS-8000S

|          |                   |
|----------|-------------------|
| Carbon   | 0.006 - 0.007%    |
| Oxygen   | 0.0019% - 0.0023% |
| Nitrogen | 0.011% - 0.013%   |

The molybdenum was received in the form of 5/8 in. diameter bar rolled at a temperature below the recrystallization temperature. Specimen lengths were cut from the bar stock, machined to approximate dimensions, and finish ground.

Two types of specimens were used in this investigation. The design of the first type of specimen in which threads were used to transmit the applied force to the specimen is shown in Fig. 1. Specimens of this type are the same as those used in previous investigations (1). The stress concentration caused by the threads in rapid load tests on arc-cast molybdenum at stresses above 95,000 lb/in.<sup>2</sup> produced fracture in the thread reliefs of the specimens. The specimen design designated as type 2 and shown in Fig. 2, which eliminates the

**Fig. 1. Test Specimen, Type 1.**

6.

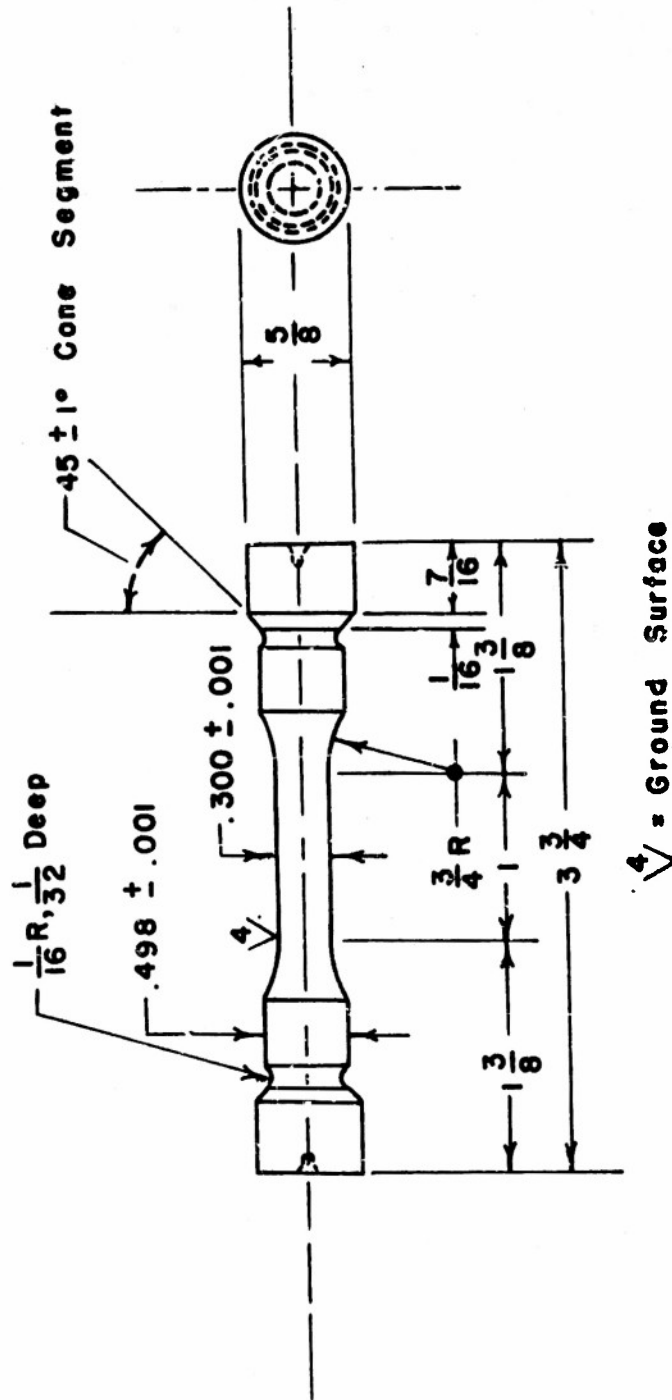


Fig. 2. Test Specimen, Type 2.

need for threads on the specimen, was used for rapid load tests at stresses above 95,000 lb/in.<sup>2</sup> on arc-cast molybdenum and for all the tests on sintered molybdenum. Both types of specimens have a round gage section 0.300 in. in diameter and 1 in. in length.

The specimens were recrystallized in a purified dry hydrogen atmosphere after finish grinding. The furnace schedule for the arc-cast molybdenum specimens was as follows:

- 1) heat to 2350°F at normal furnace rate under full power,
- 2) hold at 2350°F for one hour,
- 3) cool from 2350°F to room temperature at normal furnace rate with power off.

The furnace schedule for the sintered molybdenum specimens was as follows:

- 1) heat to 2100°F at normal furnace rate under full power,
- 2) hold at 2100°F for 1 hour,
- 3) cool from 2100°F to room temperature at normal furnace rate with power off.

These recrystallization treatments are essentially the same as those which were found by Bechtold and Scott (13) to give completely recrystallized uniform fine grain structures. The photomicrographs presented in Figs. 3, 4, and 5 show that such structures are present in the specimens employed in the present investigation.

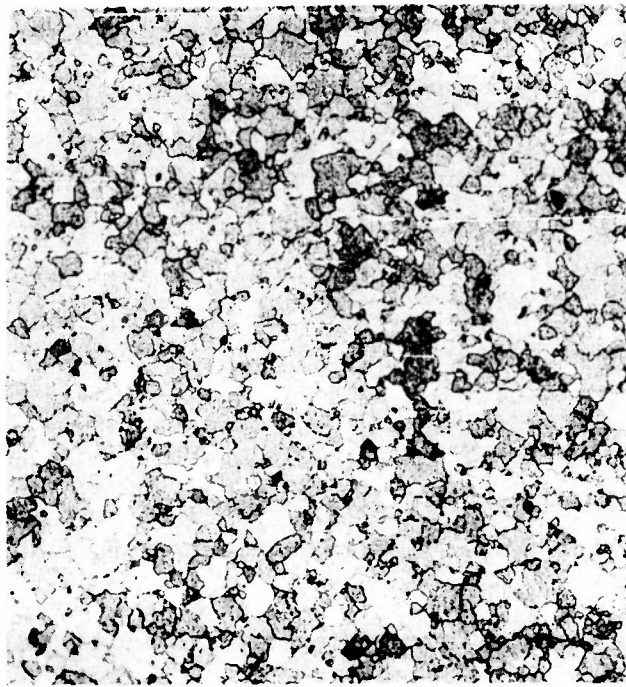


Fig. 3. Metallographic Structure of Sintered Molybdenum (X 100)

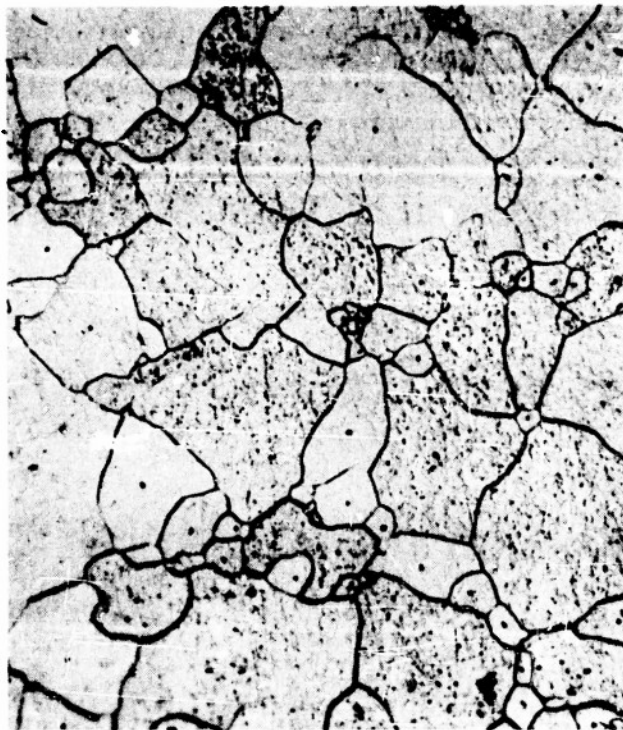


Fig. 4. Metallographic Structure of Sintered Molybdenum (X 500)

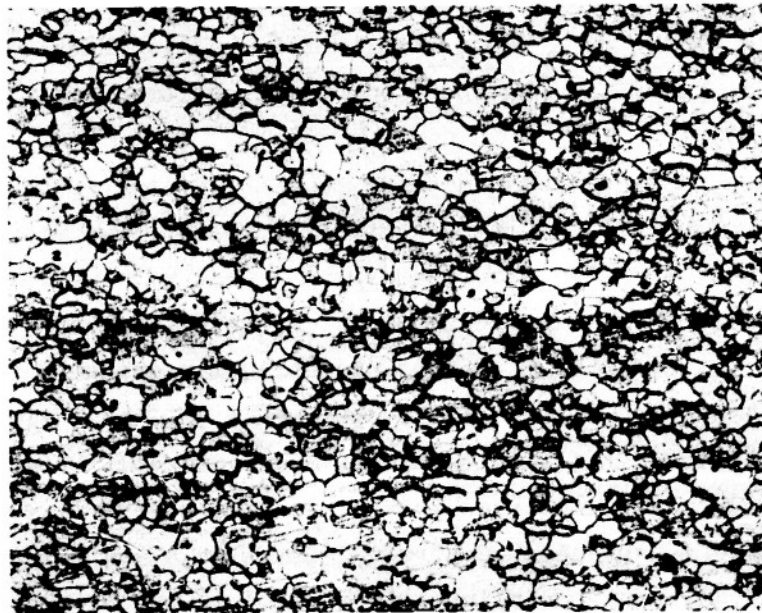


Fig. 5. Metallographic Structure of Arc-Cast Molybdenum (X 100)

### EQUIPMENT AND PROCEDURE

The rapid load tests in this investigation were made by rapidly but smoothly applying the load to the specimen within a period of about 10 millisec, and maintaining the load constant thereafter until the test was terminated by yielding and/or fracture. This was accomplished by use of a rapid load tensile testing machine of special design, hydro-pneumatically operated, which has been described previously (1).

The applied load and the extension of the specimen were determined as functions of time during the tests. Two means of detecting the extension of the specimen during a test were used simultaneously. Tensile strains were measured by means of two type A-5 SR-4 resistance sensitive wire strain gages bonded diametrically opposite to one another on the gage section of the specimens. Suitable electrical connections between the specimen gages and temperature compensating dummy specimen gages completed the strain gage bridge circuits. A less sensitive measurement of strain, giving an overall indication of the specimen deformation during a test was obtained by means of two extensometers, diametrically opposite to one another and attached to the specimen grips. The extensometers are the same ones previously used in work of this type (1). The load acting on the specimen was measured by means of a dynamometer, described previously (1), employing type AB-14, SR-4 strain gages.

The signals from the extensometers, strain gages, and dynamometer were recorded on photographic paper by a recording oscillograph manufactured by the Consolidated Engineering Corporation, Pasadena, California. A 3,000 cycle/sec. carrier bridge amplification system was used. The natural frequency of the fluid damped galvanometer elements used in the oscillograph is 835 cycles/sec. Timing lines at intervals of 0.1 or 0.01 sec. are projected onto the test record to provide a time base.

The load acting on the specimen was determined within an

estimated overall accuracy of 4 per cent. The output of the extensometer and strain gages was determined within an accuracy of about 5 per cent.

The arc-cast and sintered molybdenum specimens were tested at temperatures of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ),  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ), and  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ). Thermally insulated specimen grips were used in order to reduce the heat flux into and out of the specimen to a reasonable value. Hard rubber was chosen as the thermal insulation in the grips because it exhibits a desirable combination of thermal and mechanical properties.

The specimen grips used for tests on arc-cast molybdenum at a temperature of  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ) are the same as described previously (1) for tests on mild steel. The specimen grips incorporate fluid headers. A bellows over the specimen connects to these two headers and the specimen is heated by circulating hot oil from one header through the bellows to the other header. An oil heating and circulating unit, as previously described (1), is used for this purpose.

The new specimen and grip design, shown in Fig. 6 in their assembled form, were used to eliminate the fracturing tendency occurring at the threads of the specimen. Such fractures were found to occur in tests on arc-cast molybdenum at high stress levels at room temperature. The new grips and specimens employ a toroidal seat which transmits the applied force to the conical section of the specimen, eliminating the need for threaded specimens. This grip and specimen design was used for all the tests on sintered molybdenum, for all the tests at  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ) on arc-cast molybdenum, and for the tests at  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ) on arc-cast molybdenum at high stress levels.

The tests on the sintered molybdenum at a temperature of  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ) were accomplished by inserting a flat, round Chromalox 115 volt, 125 watt heater within the hard rubber container shown in Fig. 6. An SAE 10 oil was used as the thermal conducting fluid for high temperature tests. The oil was put in the hard rubber container and its temperature controlled by regulating the heat output of the Chromalox

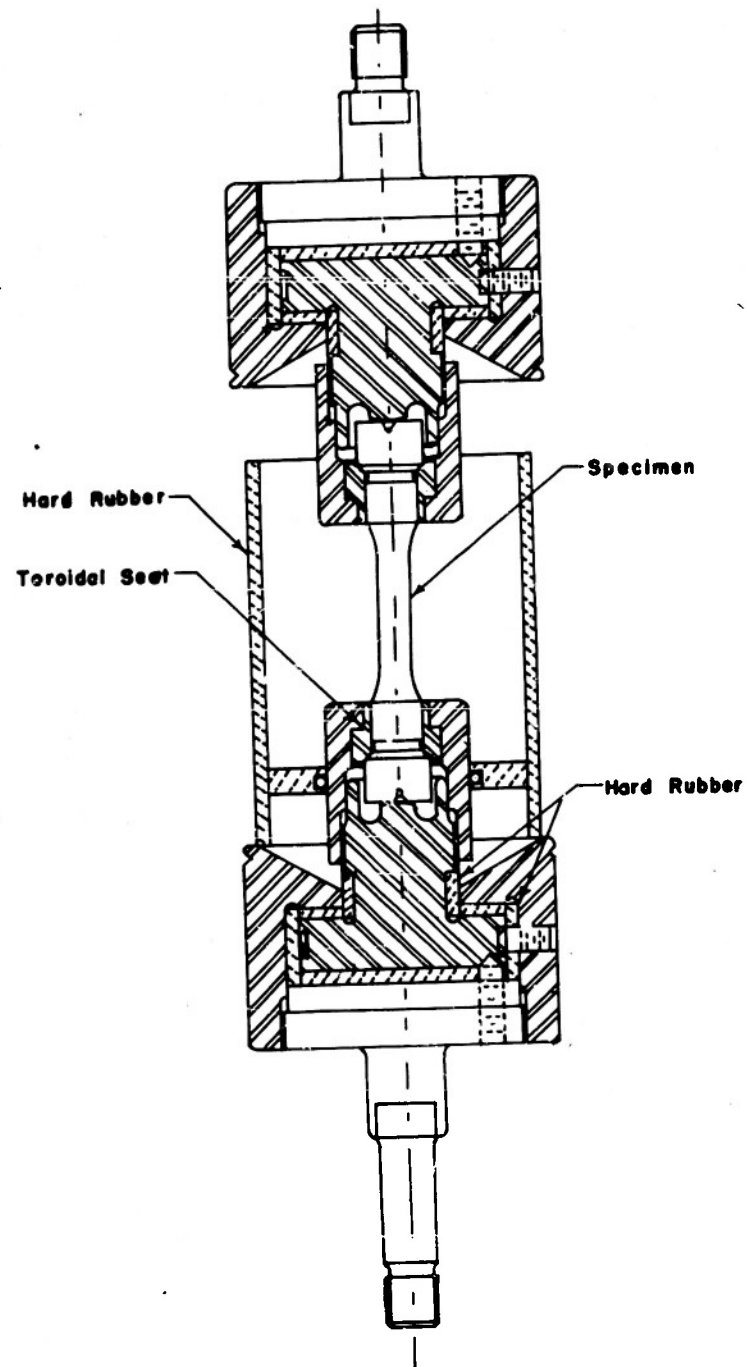


Fig. 6. Test Specimen and Grip Assembly

heater with a variac voltage control

The tests at a temperature of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ) were accomplished by means of bubbling liquid nitrogen through kerosene which surrounded the specimen. The liquid nitrogen was introduced by means of an 8 turn helix of  $1/8$  in. I.D. copper tubing, with the only outlet being a series of small holes at the bottom of the last coil. The helix was placed inside the hard rubber container shown in Fig. 6, and the temperature of the specimen was controlled by adjusting the flow of liquid nitrogen through the copper tubing.

The room temperature tests at  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ) were made while the specimen was exposed to the surrounding atmosphere. The temperature variation during room temperature tests was within  $\pm 1^{\circ}\text{F}$ , making the need for temperature control unnecessary.

The temperature of the specimens was determined by means of a copper-constantan thermocouple secured to the specimen gage section by means of electricians' rubber tape. Preliminary tests at temperatures of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ), and  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ) were made with a thermocouple on each of the fillets of the specimen and one on the gage section. These tests indicated that the total temperature variation across the gage length was within  $\pm 10^{\circ}\text{F}$  for the tests at a temperature of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ), and  $\pm 1^{\circ}\text{F}$  for the tests at a temperature of  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ).

The output of the thermocouples was measured with a Leeds and Northrup portable precision potentiometer. The potentiometer readings were converted to temperature readings with an accuracy of  $\pm 1/2^{\circ}\text{F}$ . The temperature variations during a test and from test to test were about  $\pm 5^{\circ}\text{F}$ , and  $\pm 1^{\circ}\text{F}$  at temperatures of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ) and  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ), respectively.

The upper limit of the stress employed at any given temperature was taken to be that stress at which the delay time was about 10 millisecon. A characteristic of the rapid load machine is such that a period of about 10 millisecon is required for the load to reach a constant value. Hence any tests in which the delay times are shorter

than 10 millisecc cannot be considered to be performed at constant stress.

The lower limit of stress was taken to be that stress at which the delay time was about 1 hour. Stresses corresponding to delay times greater than about 1 hour were not used since the temperature variations over such a length of time would be unreasonable.

The stresses at which the tests were made were chosen to obtain a reasonably uniform distribution of the test points in the plot of delay time vs. stress. A limited amount of molybdenum available for this investigation restricted the number of specimens and hence the total number of tests that could be made at a given temperature.

Static tension tests were performed for both arc-cast and sintered molybdenum at temperatures of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ),  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ), and  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ). The static tension tests were made in the rapid load machine, using the same grips and temperature control arrangements as for rapid load tests. The static tests were performed by controlling the applied load with the various manually operated valves associated with the machine in such a manner that the load was applied in steps. A short length of record was taken on the oscillograph after the stress and the strain attained equilibrium values. This operation was performed after each load increment was applied. The maximum strain to which the specimens could be subjected was 5 per cent since this was the maximum range of the extensometer. Two static tests were made for each temperature investigated for both sintered and arc-cast molybdenum.

### EXPERIMENTAL RESULTS

The values of upper yield stress and yield elongation obtained in the static tests at temperatures of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ),  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ), and  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ) are given in Tables I and II for arc-cast and sintered molybdenum respectively. The static stress vs. strain relations obtained are given in Figs. 7, 8, 9, 10, 11 and 12. The static stress-strain relations are plotted on two scales of strain. The dashed curves, plotted to the fine strain scale, correspond to strains indicated by SR-4 strain gages cemented to gage sections of the specimens. The solid curves, plotted to a coarse strain scale, were obtained from the extensometer measurements.

Considerable differences between stress-strain relations in the plastic range for a given material and test temperature may be noted, particularly in the curves for arc-cast molybdenum tested at  $76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ). These differences may be attributed to variations in average strain rate between tests.

Fractures occurred in two of the four static tests performed at  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ), one fracture in an arc-cast and the other in a sintered molybdenum specimen, as indicated in Figs. 7 and 10. The fracture surfaces of these specimens indicated a brittle type of failure even though some plastic deformation was recorded prior to rupture.

Two typical oscillograph records of stress, strain in gage section, and overall specimen extension as functions of time during rapid load tests are shown in Figs. 13 and 14. The manner in which the delay time for yielding is obtained from the extensometer trace is indicated on these records. The traces obtained from the SR-4 strain gages show the pre-yield plastic strains which occur in the two types of molybdenum tested.

The corresponding values of stress and delay time for yielding obtained in the rapid load tests are given in Tables III and IV, and in Figs. 15 and 16, in which the experimental points of stress vs. logarithm of the delay time for yielding are plotted. The average

TABLE I  
Results of Static Tests  
Arc-Cast Molybdenum

| <u>Temperature</u> | <u>Upper Yield Stress<br/>10<sup>3</sup> lb/in.<sup>2</sup></u> | <u>Yield Elongation<br/>Per Cent</u> | <u>Specimen<br/>Number</u> |
|--------------------|-----------------------------------------------------------------|--------------------------------------|----------------------------|
| -74°F (-59°C)      | 87.5                                                            | 2.0                                  | 47                         |
|                    | 89.0                                                            | 1.8                                  | 46                         |
| +76°F (24°C)       | 40.4                                                            | 0.30                                 | 1                          |
|                    | 40.2                                                            | 2.05                                 | 44                         |
| +200°F (93°C)      | 20.0                                                            | 0.40                                 | 23                         |
|                    | 16.5                                                            | 0.30                                 | 24                         |

TABLE II  
Results of Static Tests  
Sintered Molybdenum

| <u>Temperature</u> | <u>Upper Yield Stress<br/>10<sup>3</sup> lb/in.<sup>2</sup></u> | <u>Yield Elongation<br/>Per Cent</u> | <u>Specimen<br/>Number</u> |
|--------------------|-----------------------------------------------------------------|--------------------------------------|----------------------------|
| -74°F (-59°C)      | 84.8                                                            | 1.10                                 | 026                        |
|                    | 84.9                                                            | 1.50                                 | 030                        |
| +76°F (24°C)       | 48.5                                                            | 1.90                                 | 01                         |
|                    | 46.2                                                            | 1.90                                 | 028                        |
| +200°F (93°C)      | 34.8                                                            | 0.90                                 | 029                        |
|                    | 34.0                                                            | 0.90                                 | 031                        |

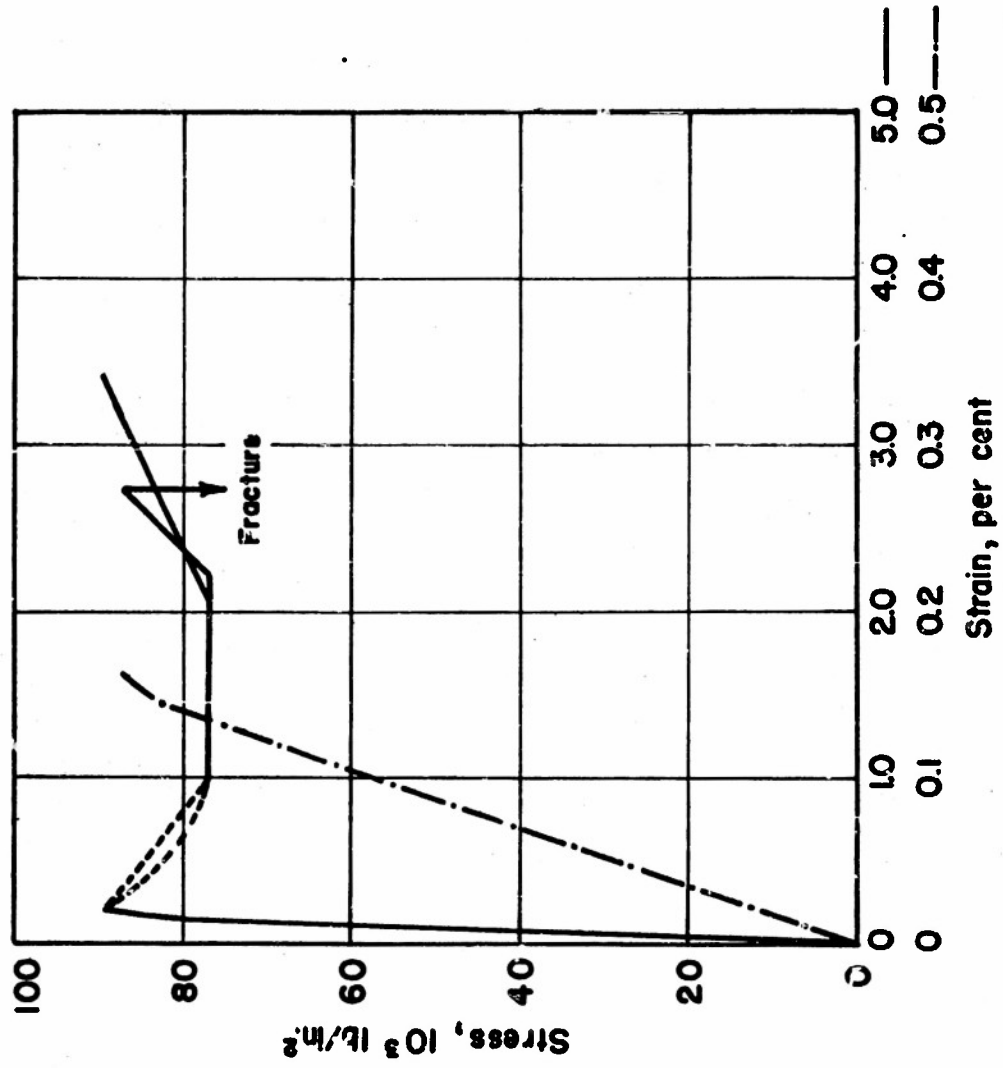


Fig. 7. Static Stress vs. Strain for Arc-Cast Molybdenum at  $-74^\circ\text{F}$  ( $-59^\circ\text{C}$ ).

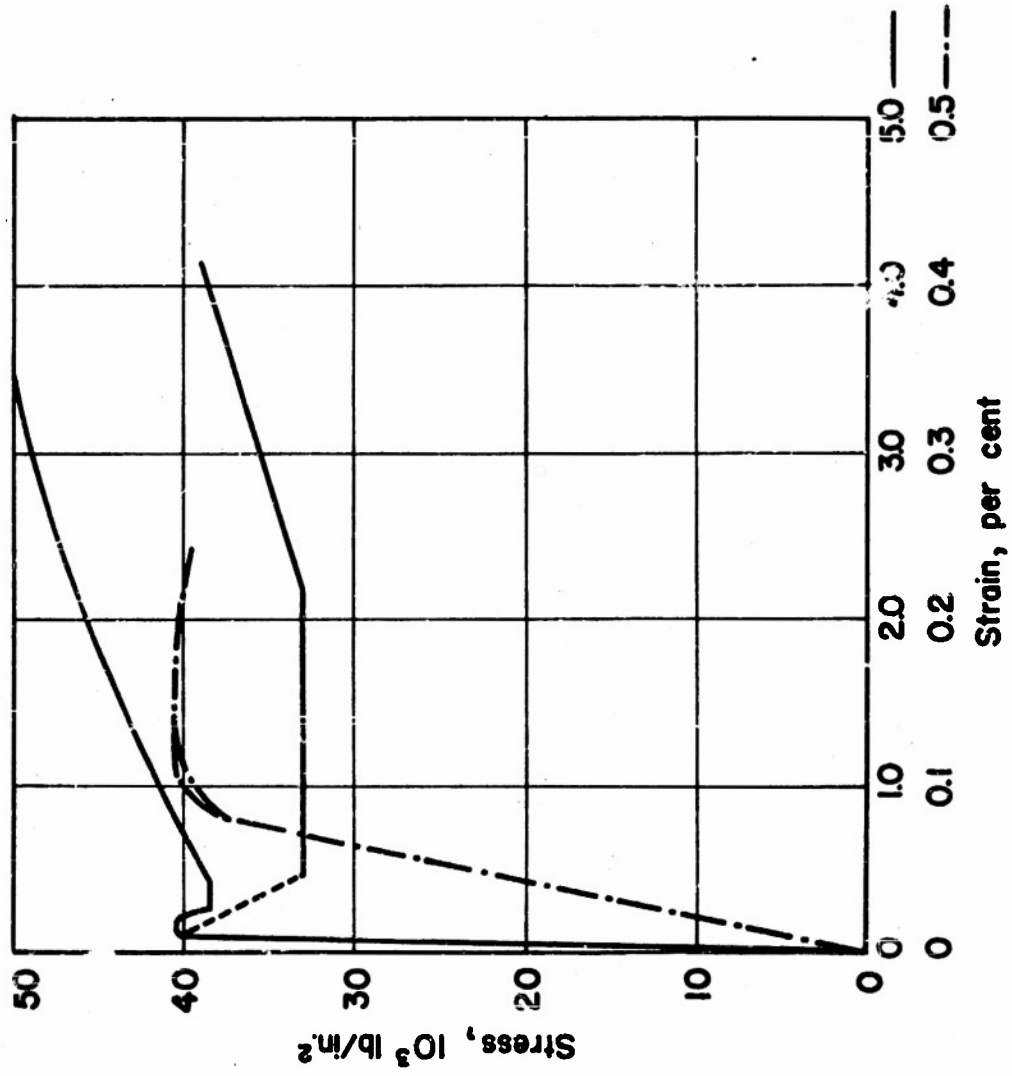


Fig. 8. Static Stress vs. Strain for Arc-Cast Molybdenum at  $+76^\circ\text{F}$  ( $24^\circ\text{C}$ ).

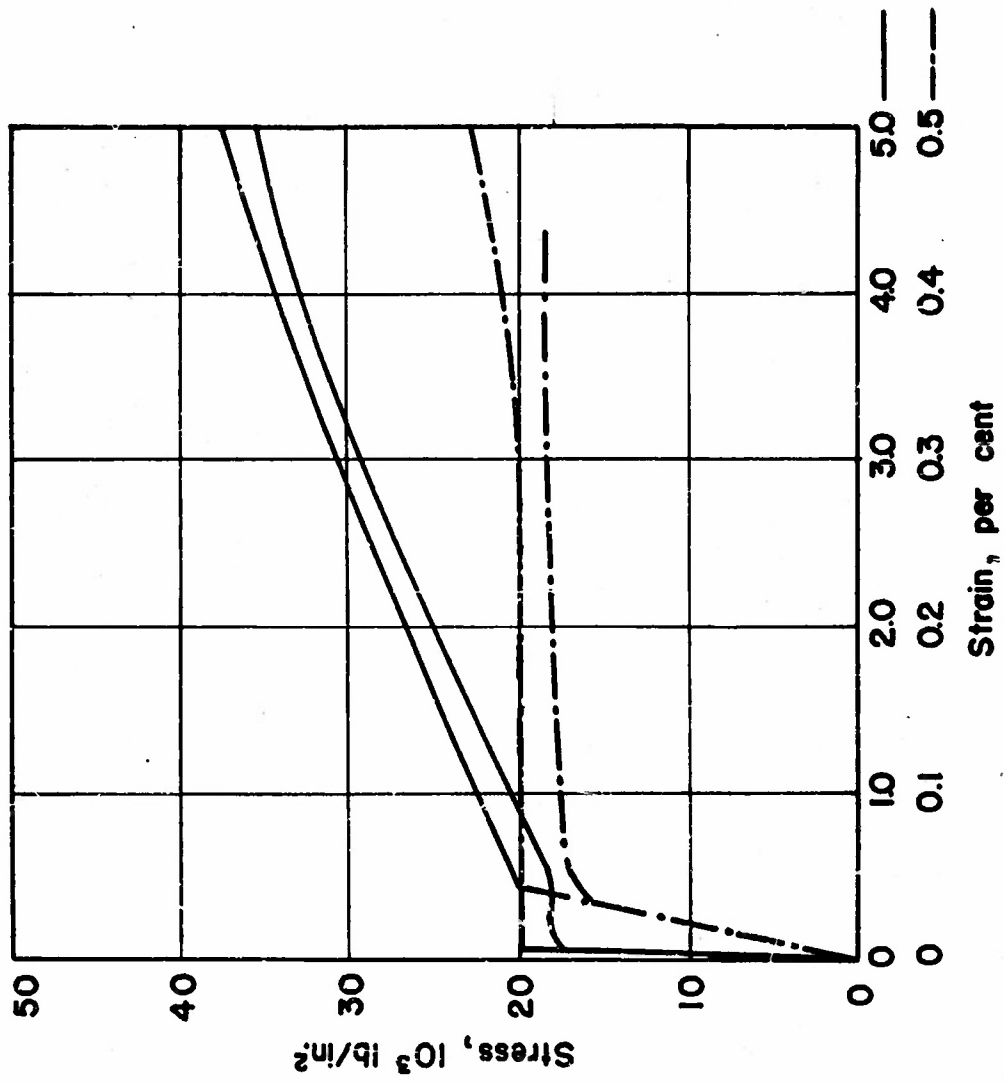


Fig. 9. Static Stress vs. Strain for Arc-Cast Molybdenum at +200°F (93°C).

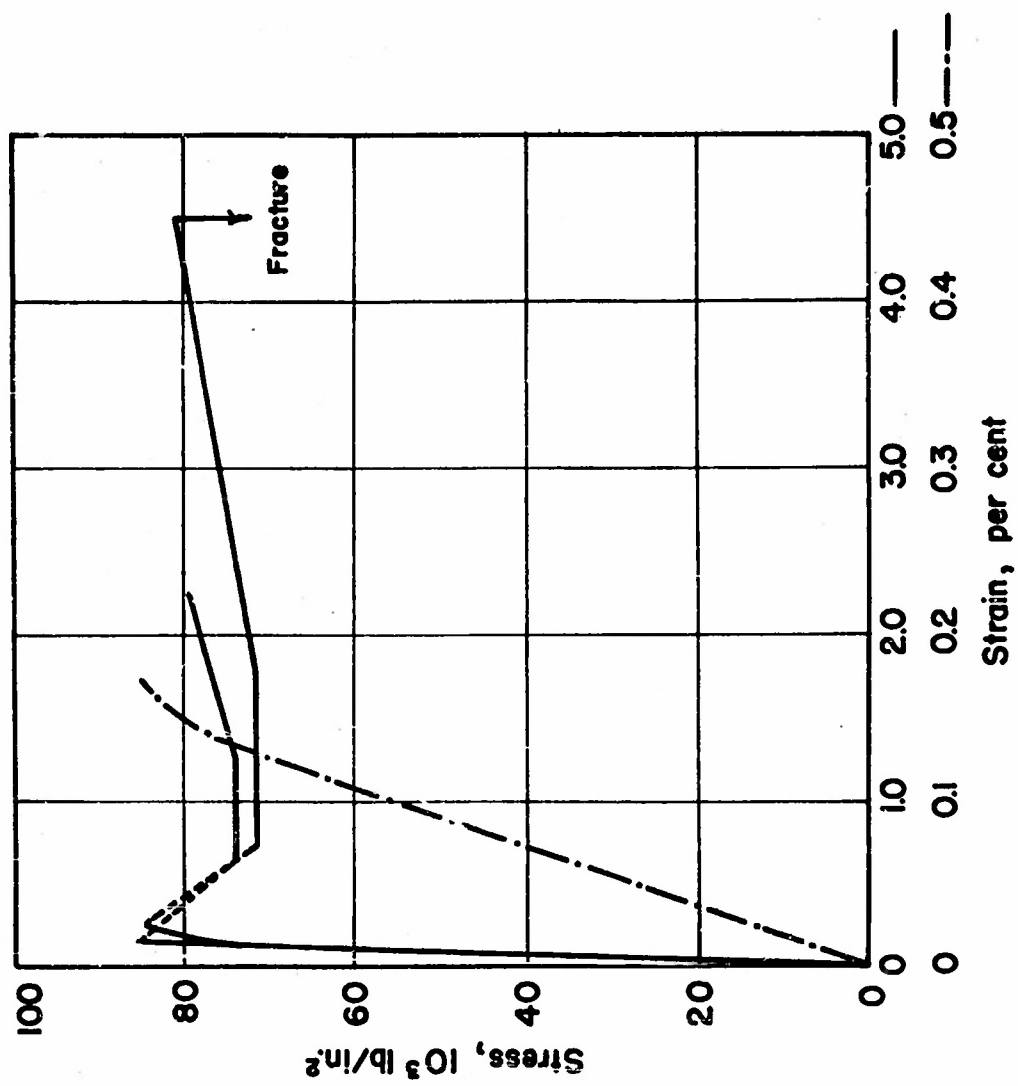


Fig. 10. Static Stress vs. Strain for Sintered Molybdenum  
at  $-74^\circ\text{F}$  ( $-59^\circ\text{C}$ )

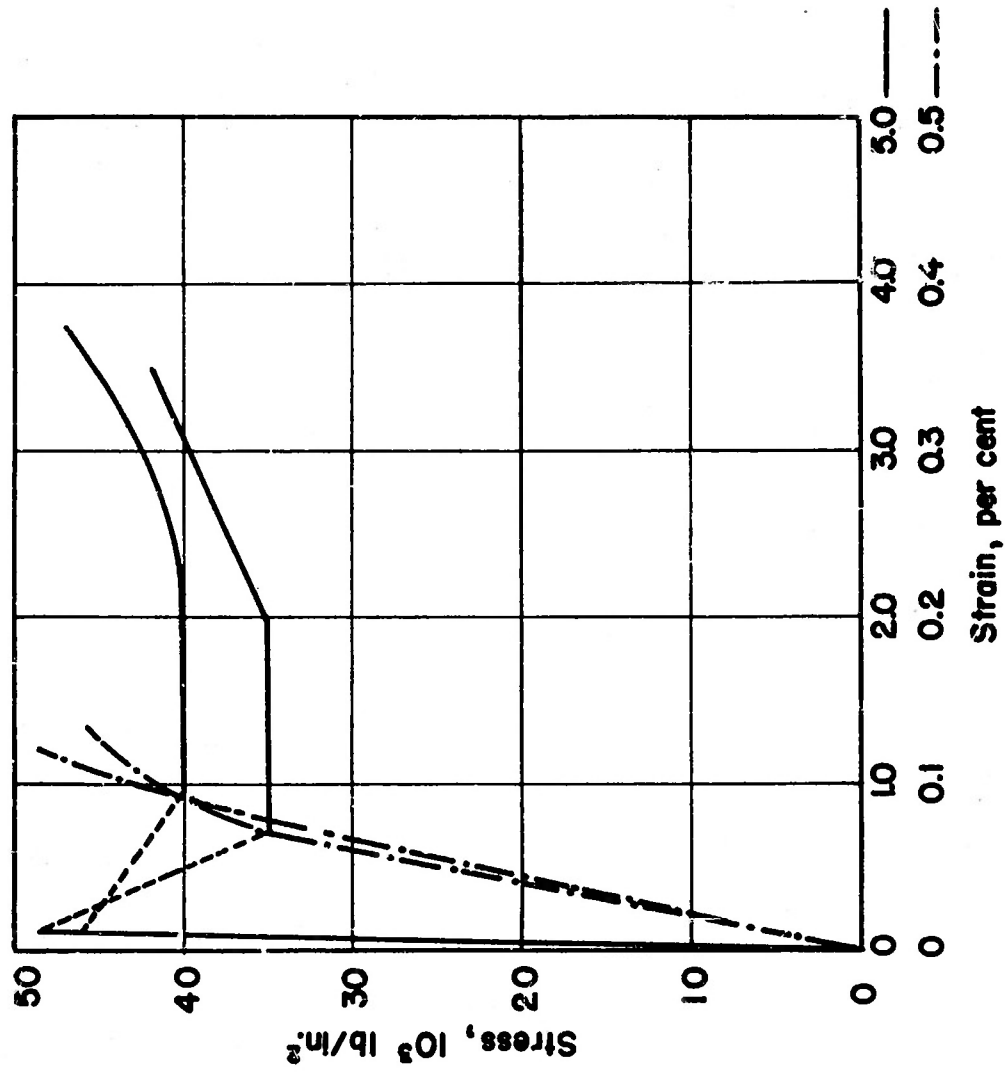


Fig. 11. Static Stress vs. Strain for Sintered Molybdenum at  $+76^\circ\text{F}$  ( $21^\circ\text{C}$ )

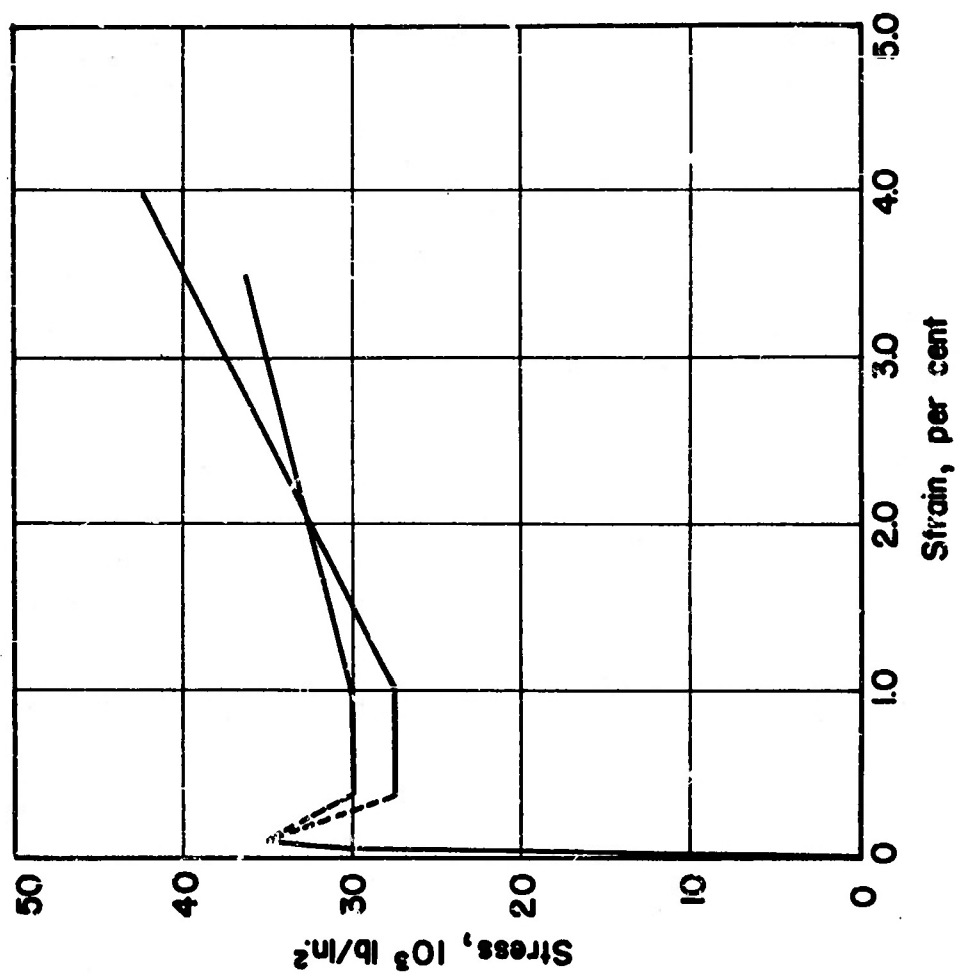


Fig. 12. Static Stress vs. Strain for Sintered Molybdenum at +200°F (93°C).

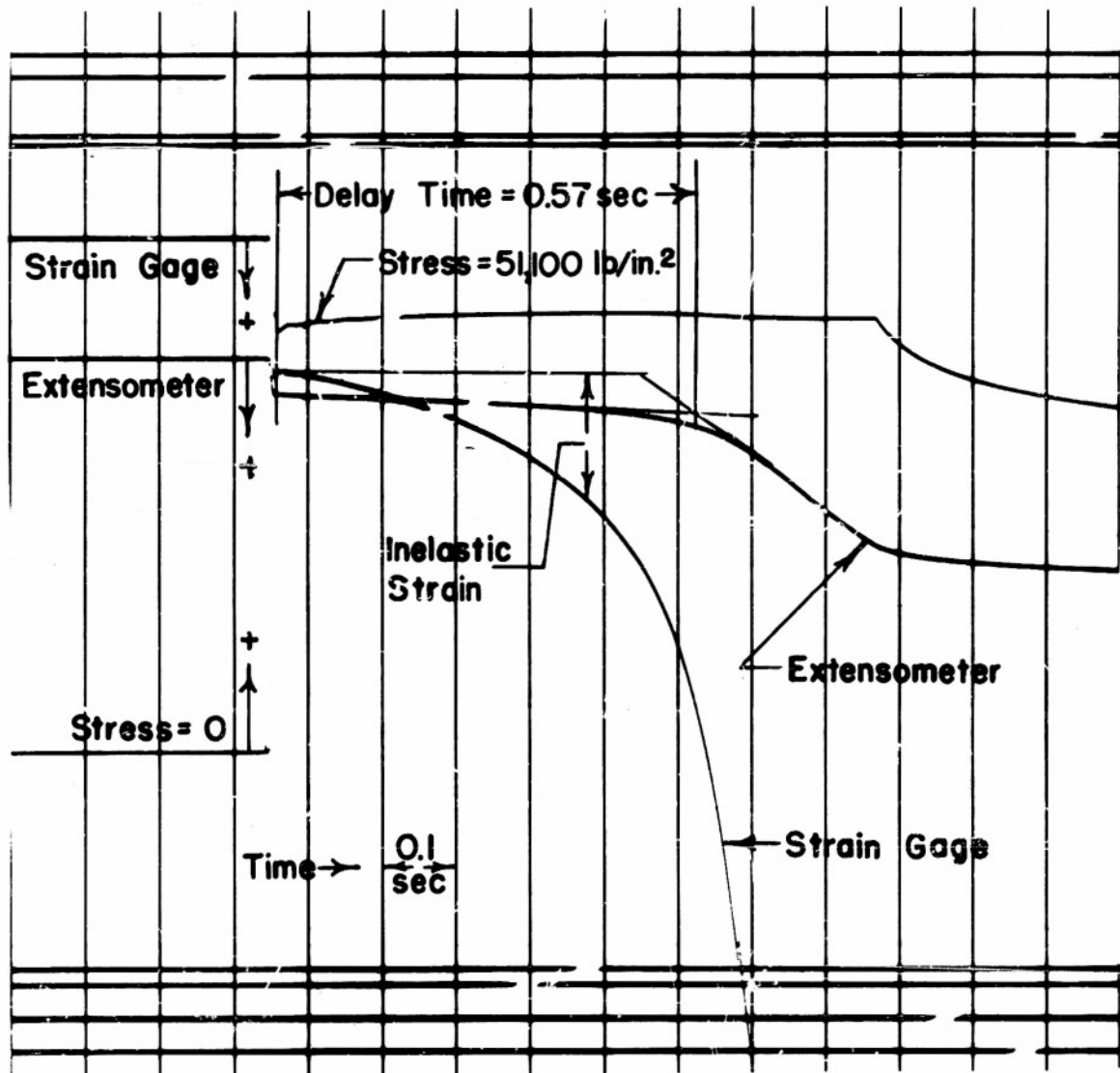


Fig. 13. Rapid Load Test Record for Arc-Cast Molybdenum at +200°F (93°C), Specimen 15.

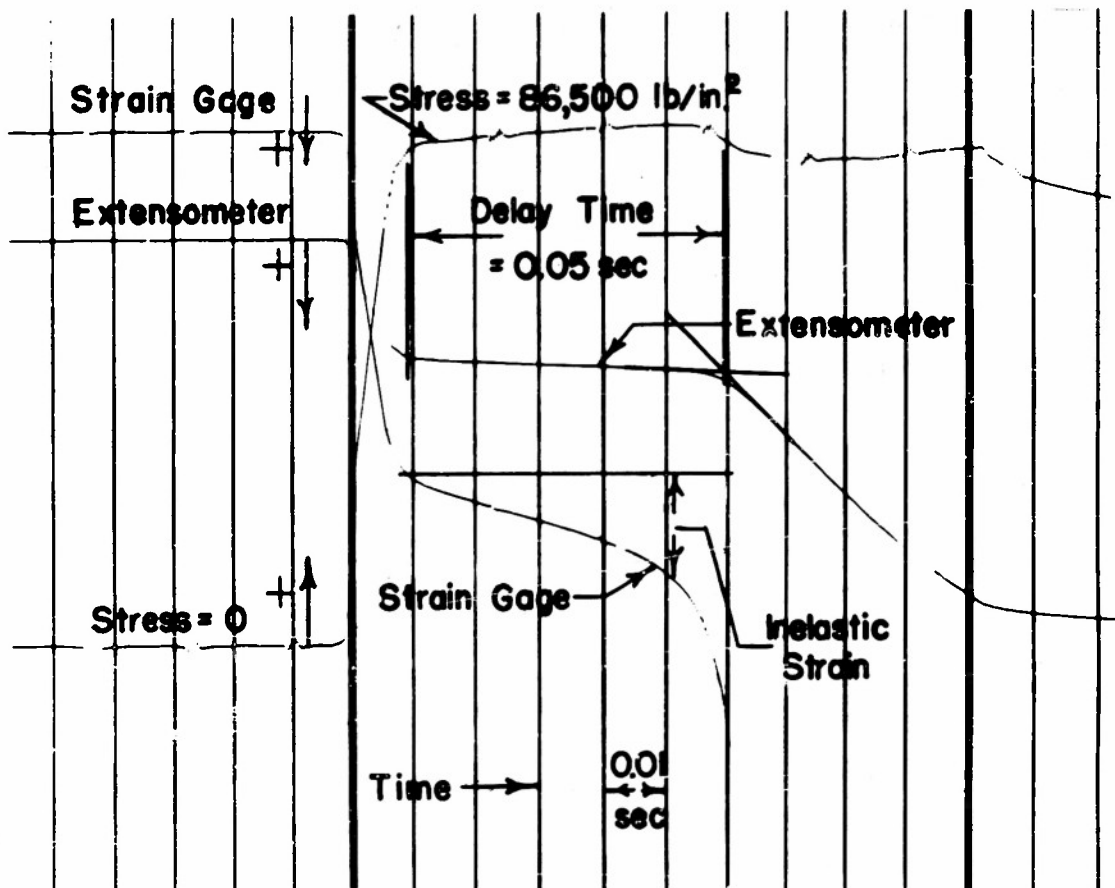


Fig. 14. Rapid Load Test Record for Sintered Molybdenum  
at +76°F (24°C), Specimen 013.

TABLE III  
Results of Rapid Load Tests  
Arc-Cast Molybdenum

| <u>Temperature</u> | <u>Stress</u><br><u>10<sup>3</sup> lb/in.<sup>2</sup></u> | <u>Delay Time</u><br><u>Sec</u> | <u>Specimen</u><br><u>Number</u> |
|--------------------|-----------------------------------------------------------|---------------------------------|----------------------------------|
| -74°F (-59°C)      | 79.5                                                      | 3000 ± 1000                     | 38                               |
|                    | 84.8                                                      | 590                             | 41                               |
|                    | 88.7                                                      | 150                             | 42                               |
|                    | 95.1                                                      | 79                              | 34                               |
|                    | 98.6                                                      | 7.8                             | 37                               |
|                    | 102.0                                                     | 3.9                             | 35                               |
|                    | 107.0                                                     | 1.3                             | 30                               |
|                    | 117.5                                                     | 0.87                            | 40                               |
|                    | 120.0                                                     | 0.06                            | 39                               |
| 76°F (24°C)        | 48.0                                                      | 3600                            | 5                                |
|                    | 51.3                                                      | 48                              | 10                               |
|                    | 58.0                                                      | 18                              | 12                               |
|                    | 63.3                                                      | 9.2                             | 16                               |
|                    | 68.0                                                      | 3.8                             | 4                                |
|                    | 76.8                                                      | 1.2                             | 26                               |
|                    | 87.0                                                      | 0.15                            | 2                                |
|                    | 95.2                                                      | 0.09                            | 3                                |
|                    | 102.0                                                     | 0.011                           | 27                               |
| 200°F (93°C)       | 20.8                                                      | 280                             | 22                               |
|                    | 26.2                                                      | 39                              | 20                               |
|                    | 33.7                                                      | 12                              | 19                               |
|                    | 36.2                                                      | 9.0                             | 18                               |
|                    | 45.7                                                      | 1.4                             | 17                               |
|                    | 51.1                                                      | 0.57                            | 15                               |
|                    | 64.5                                                      | 0.12                            | 13                               |
|                    | 66.5                                                      | 0.043                           | 21                               |
|                    | 75.6                                                      | 0.016                           | 11                               |

TABLE IV  
Results of Rapid Load Tests  
Sintered Molybdenum

| <u>Temperature</u> | <u>Stress<br/>10<sup>3</sup> lb/in.<sup>2</sup></u> | <u>Delay Time<br/>Sec</u> | <u>Specimen<br/>Number</u> |
|--------------------|-----------------------------------------------------|---------------------------|----------------------------|
| -74°F (-59°C)      | 72.3                                                | 3600                      | 018                        |
|                    | 76.5                                                | 800                       | 014                        |
|                    | 83.6                                                | 200                       | 019                        |
|                    | 87.1                                                | 120                       | 016                        |
|                    | 92.5                                                | 66                        | 09                         |
|                    | 96.0                                                | 30                        | 017                        |
|                    | 102.0                                               | 0.20                      | 011                        |
|                    | 104.0                                               | <0.01                     | 05                         |
| 76°F (24°C)        | 47.5                                                | 160                       | 010                        |
|                    | 50.2                                                | 85                        | 07                         |
|                    | 52.7                                                | 330                       | 04                         |
|                    | 58.6                                                | 30                        | 03                         |
|                    | 62.2                                                | 16                        | 012                        |
|                    | 69.0                                                | 0.86                      | 02                         |
|                    | 77.1                                                | 0.20                      | 06                         |
|                    | 86.5                                                | 0.050                     | 013                        |
|                    | 90.5                                                | 0.020                     | 08                         |
| 200°F (93°C)       | 34.9                                                | 1100                      | 020                        |
|                    | 40.8                                                | 140                       | 023                        |
|                    | 44.5                                                | 18                        | 025                        |
|                    | 47.5                                                | 0.40                      | 027                        |
|                    | 51.2                                                | 1.3                       | 024                        |
|                    | 59.1                                                | 0.22                      | 021                        |
|                    | 62.5                                                | 0.067                     | 015                        |

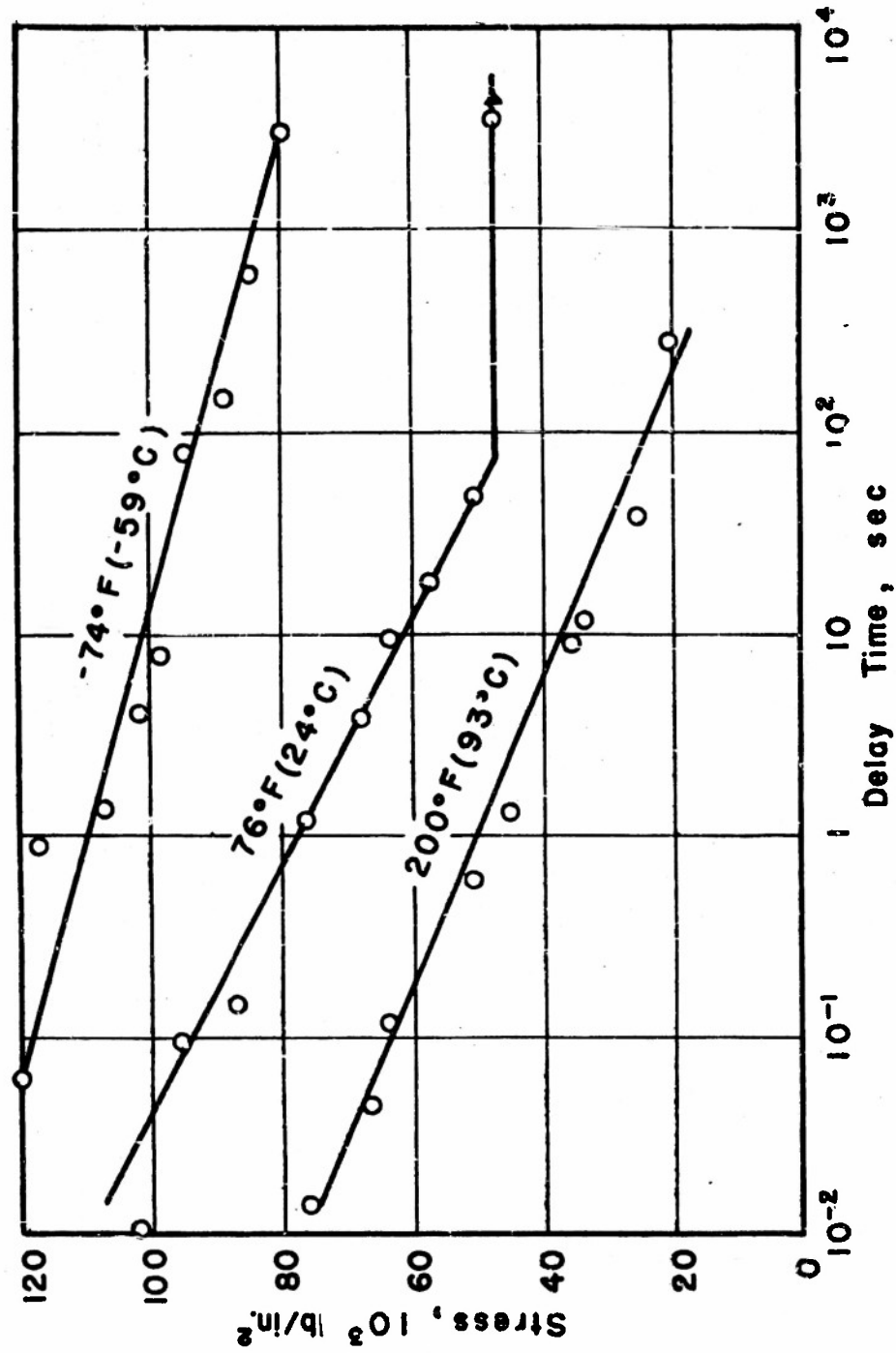


Fig. 15. Stress vs. Delay Time for Arc-Cast Molybdenum.

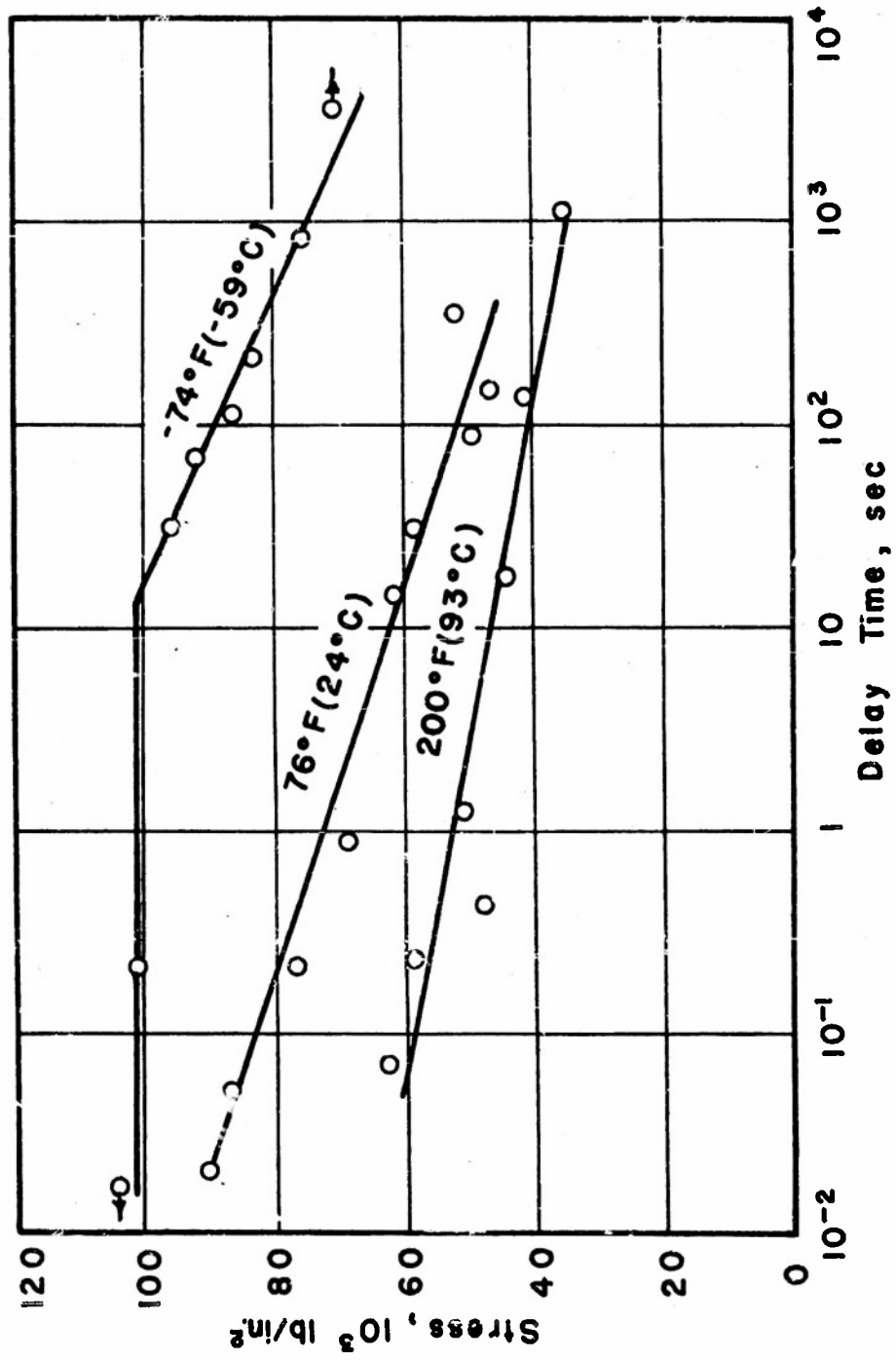


Fig. 16. Stress vs. Delay Time for Sintered Molybdenum.

trends of the experimental points of Figs. 15 and 16 are represented by straight lines.

The brittle type of fracture observed in the static tests at a temperature of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ) occurred in four of the eight rapid load tests performed on sintered molybdenum and in seven of the nine tests on arc-cast molybdenum at that temperature. There were no fractures in any of the rapid load tests at a temperature of  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ). Brittle type fractures occurred in one of the nine rapid load tests on sintered molybdenum and in two of the nine tests on arc-cast molybdenum at a temperature of  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ). The fact that such fractures were not observed in all tests at  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ) and in more if not all tests at  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ) is due to the limited extension (5 per cent or less) which the rapid load testing machine will permit.

Preyield plastic strain vs. time for a series of constant stresses applied to specimens of arc-cast and sintered molybdenum at temperatures of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ),  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ), and  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ) are shown in Figs 17 through 22. The applied stresses in each case were above the static upper yield stress. These values of preyield plastic strains are the differences between the total strain indicated by the SR-4 strain gages at any instant and the elastic strain. The elastic strain is substantially constant during any given test and is taken to be the total strain at the instant the stress attains the constant test value.

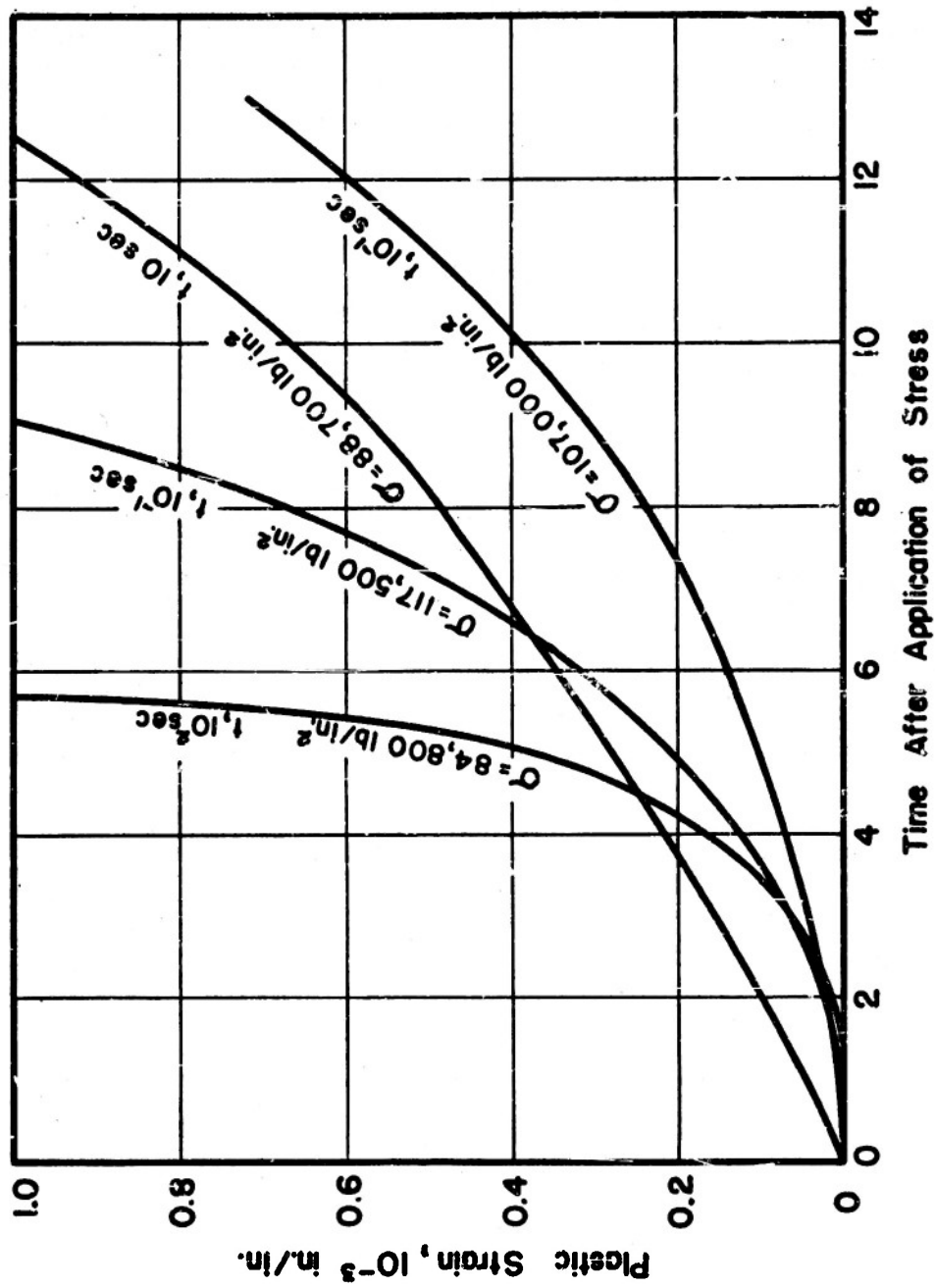


Fig. 17. Preyield Plastic Strain vs. Time for Arc-Cast Molybdenum at  $-74^\circ\text{F}$  ( $-59^\circ\text{C}$ ),  $\sigma$  and  $t$  values give the constant test stress and time scale multiplying factor respectively for each curve.

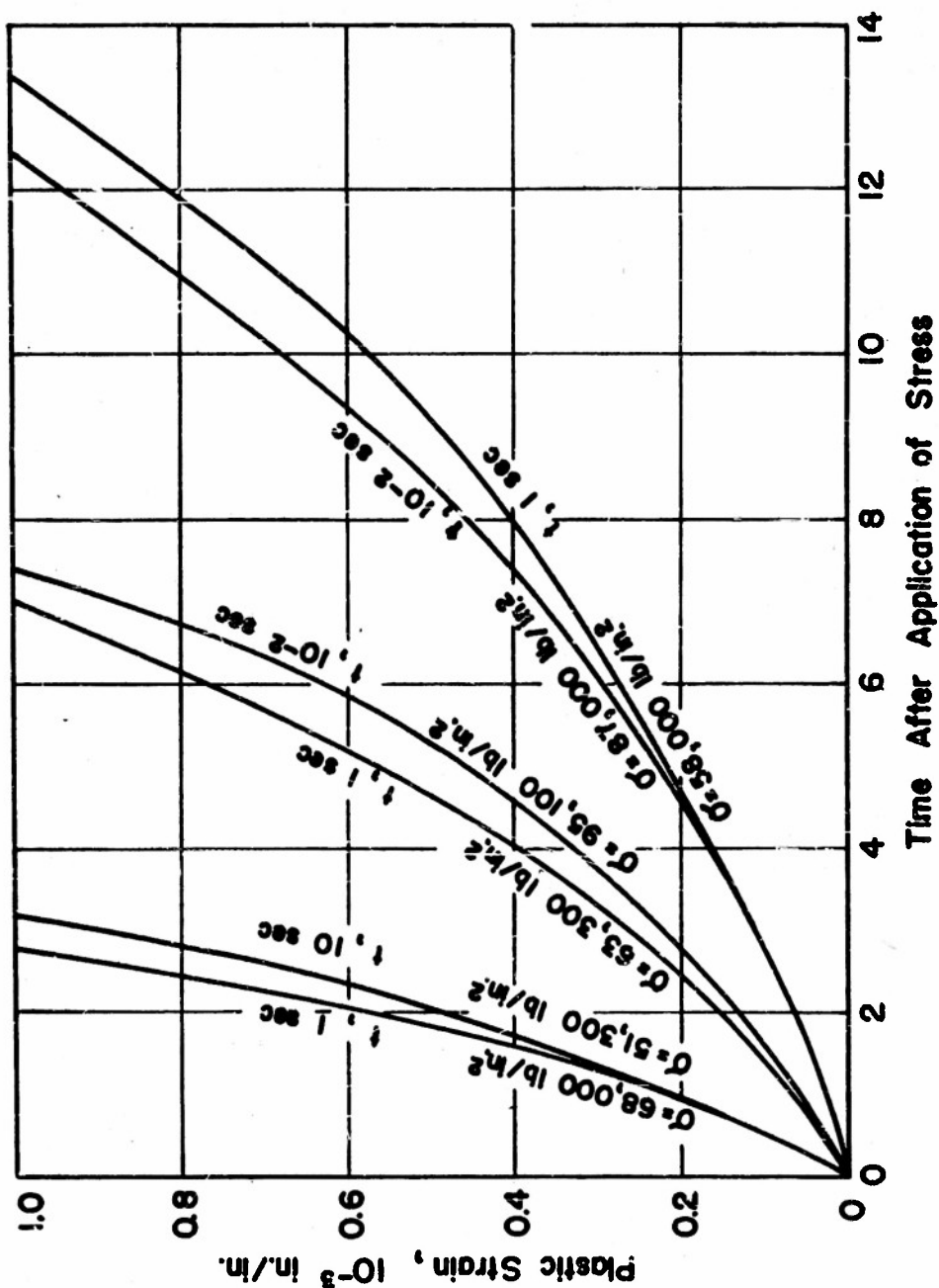


Fig. 18. Freyfeld Plastic Strain vs. Time for Arc-Cast Molybdenum at  $+75^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ),  $\sigma$  and  $t$  values give the constant test stress and time scale multiplying factor respectively for each curve.

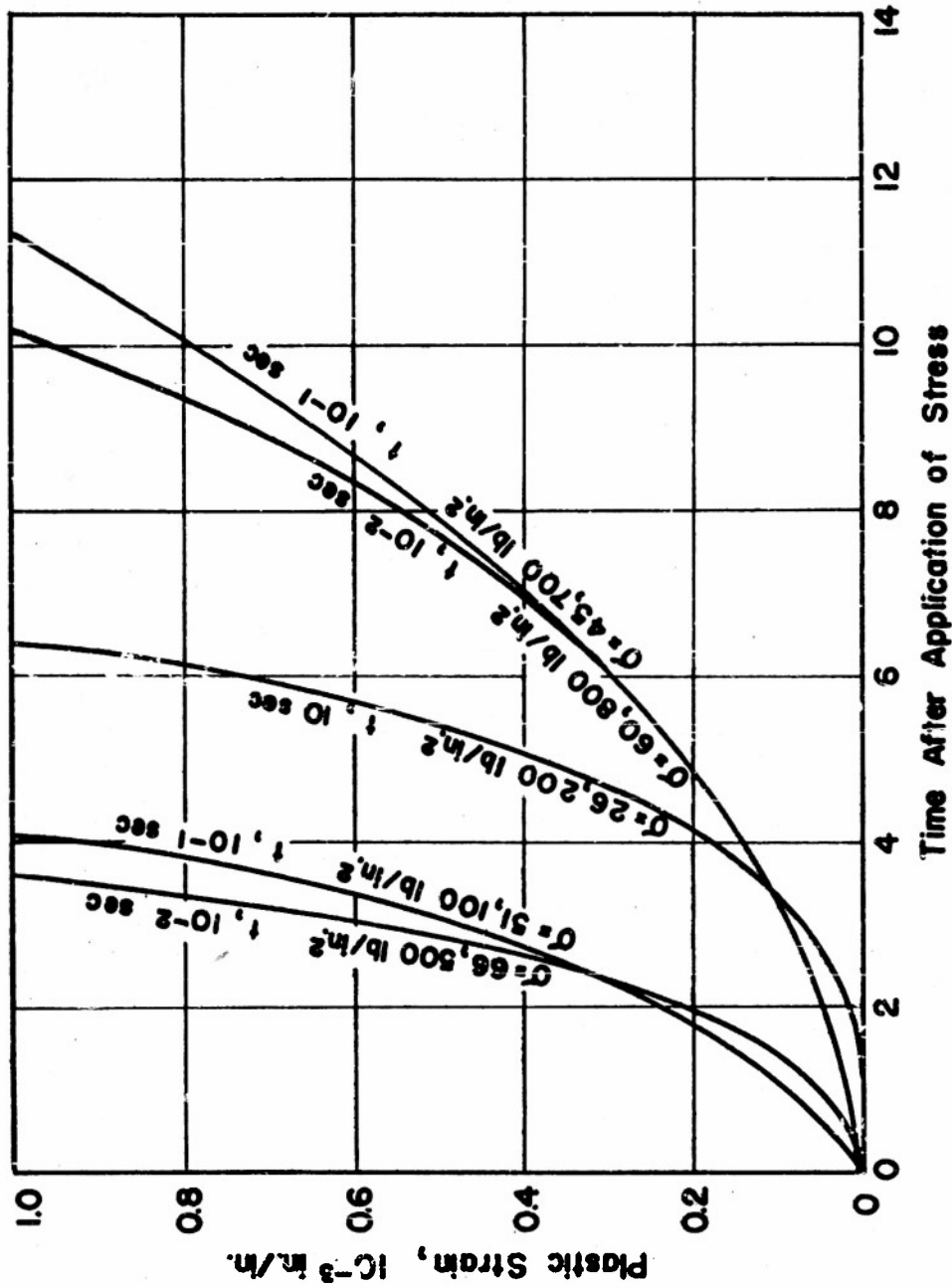


Fig. 19. Preyield Plastic Strain vs. Time for Arc-Cast Molybdenum at  $+200^\circ\text{F}$  ( $93^\circ\text{C}$ ).  $\sigma$  and  $t$  values give the constant test stress and time scale multiplying factor respectively for each curve.

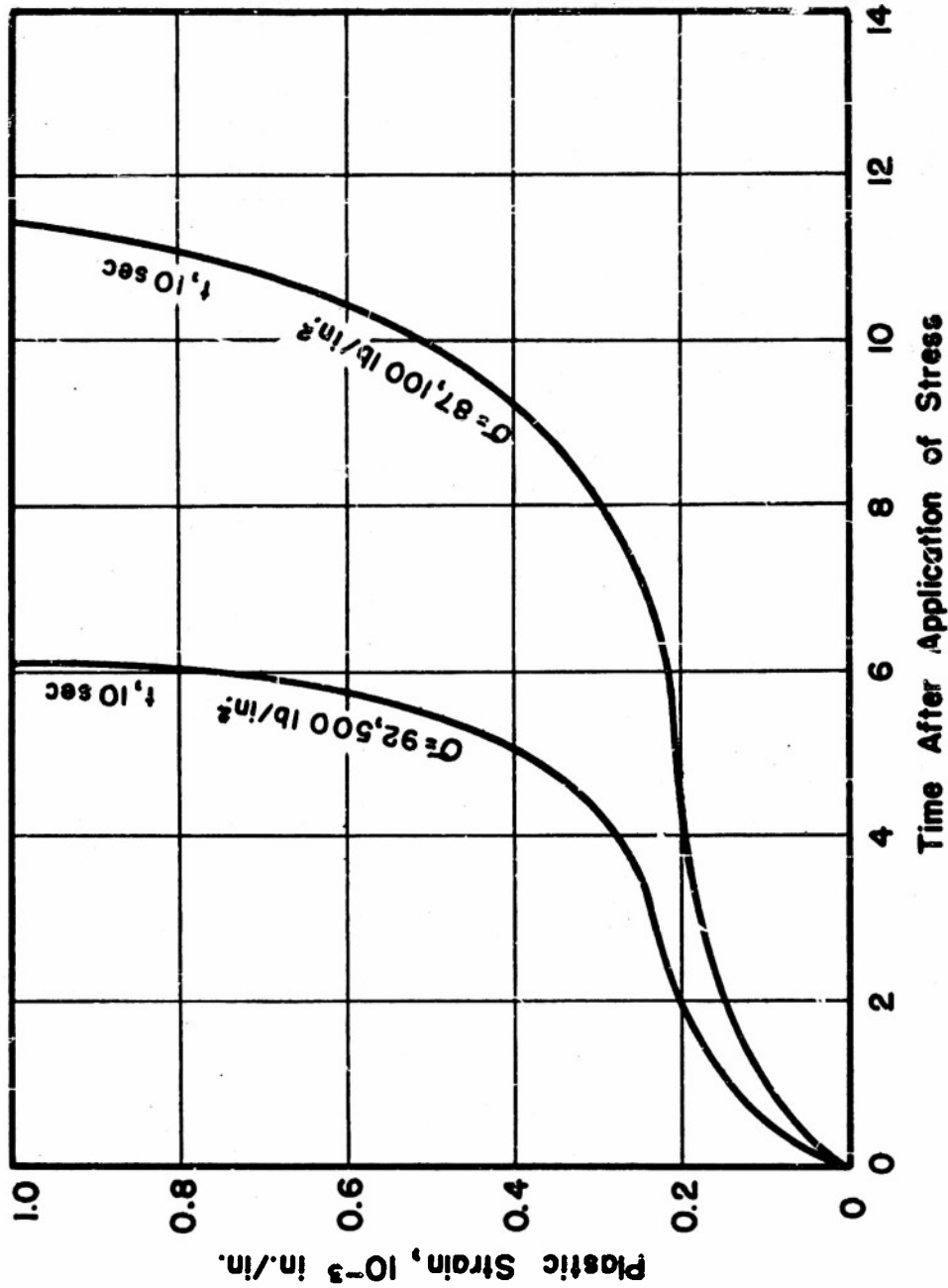


Fig. 20. Preyield Plastic Strain vs. Time for Sintered Molybdenum at  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ),  $\sigma$  and  $t$  values give the constant test stress and time scale multiplying factor respectively for each curve.

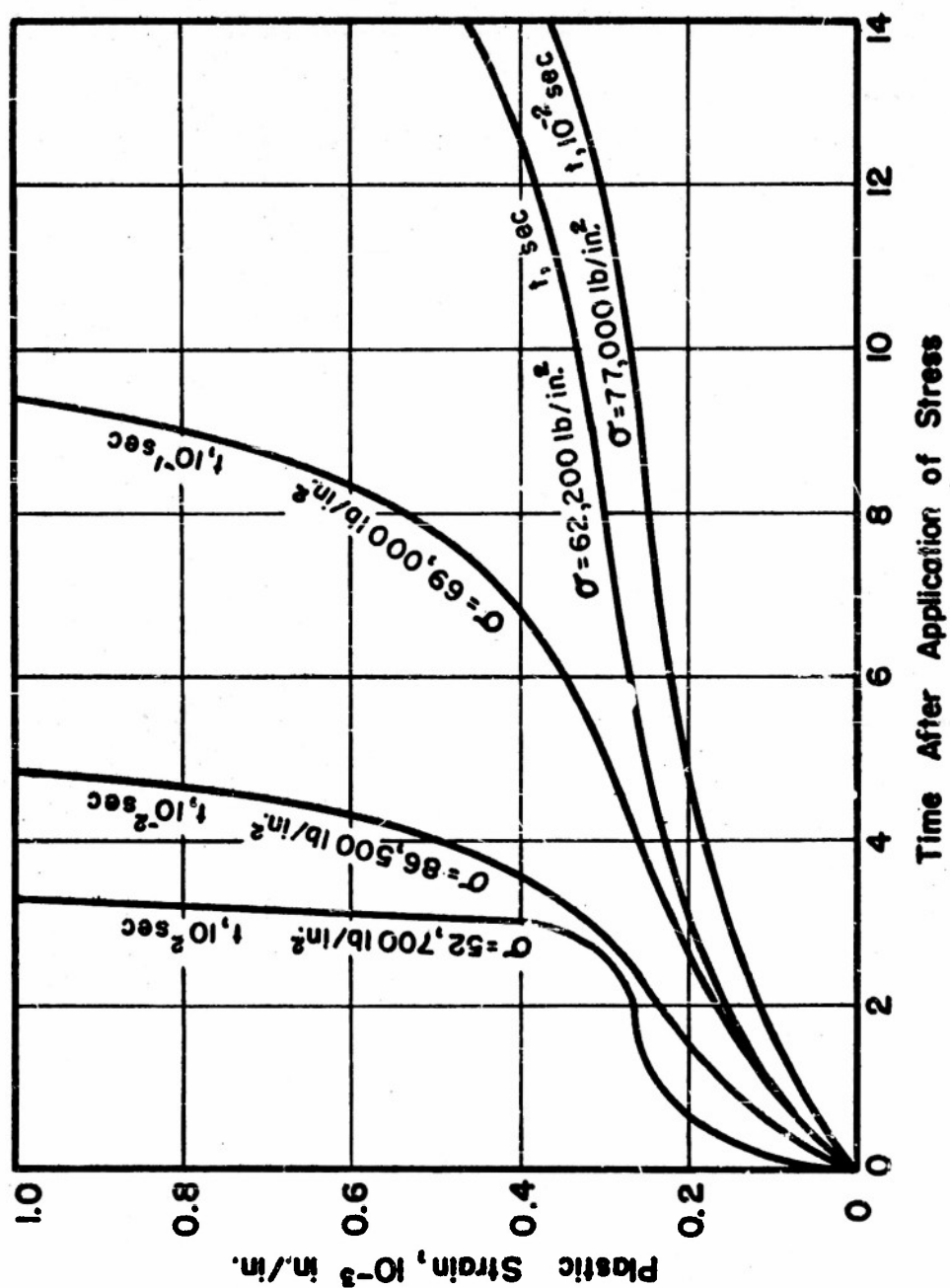


Fig. 21. Preyield Plastic Strain vs. Time for Sintered Molybdenum at  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ),  $\sigma$  and  $t$  values give the constant test stress and time scale multiplying factor respectively for each curve.

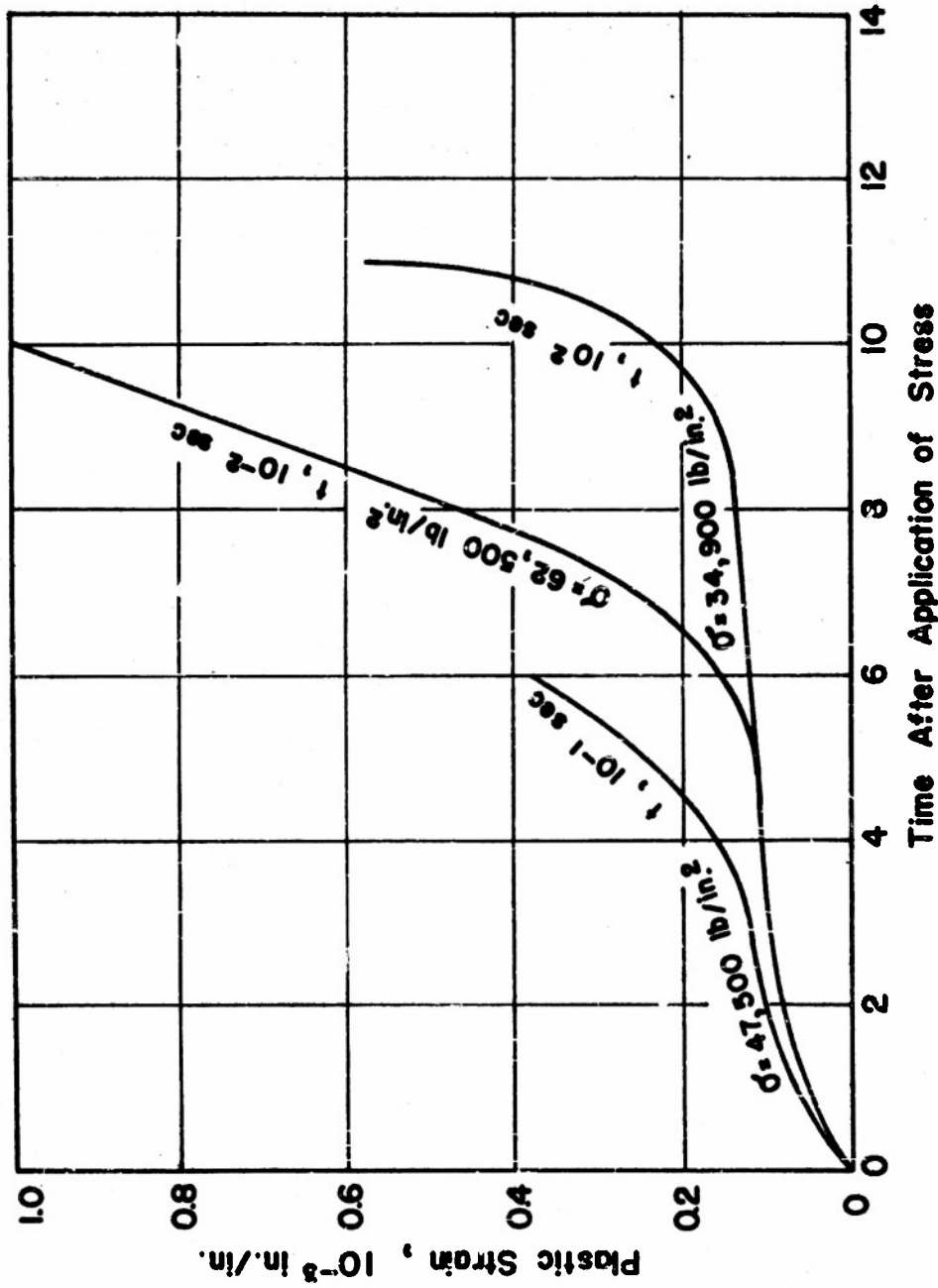


Fig. 22. Preyfeld Plastic Strain vs. Time for Sintered Molybdenum at +200°F (93°C),  $\sigma$  and  $t$  values give the constant test stress and time scale multiplying factor respectively for each curve.

### DISCUSSION OF RESULTS

The results of the static tension tests are substantially in agreement with those obtained by Bechtold and Scott (13) on similar materials. These show clearly that fine grained ductile molybdenum, produced by either the arc-casting or powder metallurgy processes, exhibits the phenomenon of a distinct yield point and that the yield point is quite temperature sensitive. Thus these materials exhibit a yielding behavior which is quite similar to that commonly observed in iron and low carbon steels. However, some quantitative differences between the molybdenum employed in this investigation and annealed low carbon steels previously investigated are indicated by the static tests. First, the upper yield stress of the molybdenum continuously decreases as the temperature is increased in the range from  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ) and  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ) and no multiple yield points are observed in molybdenum during static tests at  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ). Annealed low carbon steel on the other hand exhibits a decrease in upper yield stress with increasing temperature up to about room temperature after which the upper yield stress remains about constant up to at least  $+250^{\circ}\text{F}$  ( $121^{\circ}\text{C}$ ) (1). Also, at  $+150^{\circ}\text{F}$  ( $66^{\circ}\text{C}$ ) and  $+250^{\circ}\text{F}$  ( $121^{\circ}\text{C}$ ) annealed low carbon steels exhibit multiple yield points (1). These features of the behavior of low carbon steel may be attributed to the thermal diffusion of carbon and/or nitrogen to dislocations during testing and may be considered as strain aging effects. The absence of such behavior in molybdenum in the same temperature range may be due to lower diffusion coefficients of the interstitial atoms, or the lower concentrations of these atoms in the molybdenum tested, or both.

Second, the molybdenum exhibits larger deviations from linear elasticity just prior to yielding than does low carbon steel. Also, the arc-cast molybdenum tested at  $+200^{\circ}\text{F}$  ( $93^{\circ}\text{C}$ ) actually does not exhibit a drop in load at the yield point, although it shows a yield point elongation at constant stress. This is also probably due to the rather low concentration of carbon and/or nitrogen in the molybdenum. Similar

behavior has been observed in steel in which the carbon and nitrogen concentrations have been reduced to values comparable with those in the molybdenum (2).

The results of the rapid load tests show clearly that ductile molybdenum exhibits the phenomenon of delayed yielding under rapidly applied constant stress. The dependence of the delay time for yielding upon stress and temperature as shown in Figs. 15 and 16 is, on the whole, similar in form to that found for annealed low carbon steel (1).

The stress vs. delay time curves for the molybdenum tested, with the possible exception of the curve for the arc-cast material tested at  $76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ), do not appear to show a lower limiting stress (horizontal portion at a low stress and long delay times), such as that found in normal low carbon steel. However, a similar behavior has been observed in steel in which the carbon and nitrogen concentrations were exceptionally low (2). Thus the observed behavior of molybdenum in this respect is consistent with the relatively low concentrations of carbon and nitrogen in the material tested. The single case in which such a lower limiting stress is indicated for molybdenum is based upon only one experimental point and is, therefore, somewhat doubtful.

The stress vs. delay time curve for sintered molybdenum tested at  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ) exhibits an upper limiting stress (horizontal portion at high stress and short delay times). Similar behavior is exhibited by low carbon steel at  $-320^{\circ}\text{F}$  ( $-196^{\circ}\text{C}$ ) (3). The fact that such an upper limiting stress may be observed in molybdenum at a higher temperature than in low carbon steel may be related to its higher melting point and is consistent with Bechtold and Scott's observation (13) that the transition temperature in unnotched tensile specimens is considerably higher for molybdenum than for armco iron.

The absence of an upper limiting stress in the stress vs. delay time relation for arc-cast molybdenum tested at  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ) does not indicate that a fundamental difference exists between the two types of molybdenum. Rather, it indicates a quantitative difference in

their behavior in which the upper limiting stress for arc-cast molybdenum at a temperature of  $-74^{\circ}\text{F}$  ( $-59^{\circ}\text{C}$ ) occurs in a range of delay times which are shorter than those which can be investigated with the testing technique employed in the present investigation. This quantitative difference between the delayed yielding behavior of the two types of molybdenum tested is probably associated with the differences in carbon, nitrogen, or oxygen contents in these materials.

The measurements of pre-yield plastic strain in molybdenum given in Figs. 17 - 22 show that this material exhibits considerably larger pre-yield plastic strains than those which have been previously observed in low carbon steel (6). The latter material exhibits pre-yield plastic strains of about  $30 \times 10^{-6}$  in/in. whereas the molybdenum exhibits pre-yield plastic strains ranging from about  $100 \times 10^{-6}$  in/in. to about  $1000 \times 10^{-6}$  in/in. The arc-cast material exhibits particularly large strains of this type. Furthermore, in the arc-cast material the pre-yield plastic strain rate increases continuously with time during any particular test at constant stress. Conversely, in the sintered molybdenum, as in low carbon steel, the pre-yield plastic strain rate decreases with time at constant stress up until the time when macroscopic yielding begins. The latter behavior is consistent with the dislocation model of a yield nucleus which has been developed previously (6), (7), while the behavior of the arc-cast molybdenum is not consistent with this model.

This difference between the behavior of arc-cast molybdenum on the one hand and sintered molybdenum and low carbon steel on the other may be associated with the relative differences in the carbon and nitrogen contents of the materials. The arc-cast material contains considerably more carbon and less nitrogen than the sintered material. If the assumption is made that dislocations in molybdenum interact strongly with nitrogen atoms, but only relatively weakly with carbon atoms, then the arc-cast material might be expected to contain many more dislocations which are unanchored by Cottrell "atmospheres" than the sintered material. Hence the behavior of the yield nuclei would be different in the arc-cast material than is predicted by the theoretical dislocation model.

However, this point of view may be subject to criticism in that even the very small concentration of nitrogen present in the arc-cast material should be sufficient to provide complete "atmospheres" for all the dislocations which are expected to be present initially in the material. Thus if the initial density of dislocations is  $10^8/\text{cm}^2$  the concentration of nitrogen required to provide one nitrogen atom per atomic length along each dislocation is only about  $10^{-6}$  weight per cent.

An alternative hypothesis for the effect of different carbon and nitrogen contents upon the pre-yield plastic strains in molybdenum is that the grain boundaries in molybdenum are markedly strengthened by nitrogen but not by carbon. According to the theory, the ability of grain boundaries to impede the motion of dislocations plays an important part in the development of pre-yield plastic strain. Thus weaker grain boundaries in the arc-cast molybdenum, due to low nitrogen content, could explain the observed behavior. Since the saturation of all the grain boundaries would require larger amounts of nitrogen than the saturation of all the dislocations, this second explanation may be preferable to the first.

Since the pre-yield plastic strain in the sintered molybdenum occurs in a manner which is qualitatively in accord with the dislocation theory (7) of a yield nucleus, it may be compared with that theory quantitatively. Specifically, experimental values of the initial rate of the pre-yield plastic strain as a function of applied stress may be compared with the theoretical relationship between these two quantities. Sufficient reliable data for this comparison were only obtained in the case of the sintered molybdenum tested at  $+76^\circ\text{F}$  ( $24^\circ\text{C}$ ). These initial pre-yield plastic strain rates are found by determining the initial slopes of the experimental curves given in Fig. 21, and the values are plotted in Fig. 23 to a logarithmic abscissa scale versus the reciprocals of the corresponding values of the applied stress on the ordinate scale. The experimental points are seen to correspond to a straight line in this plot.

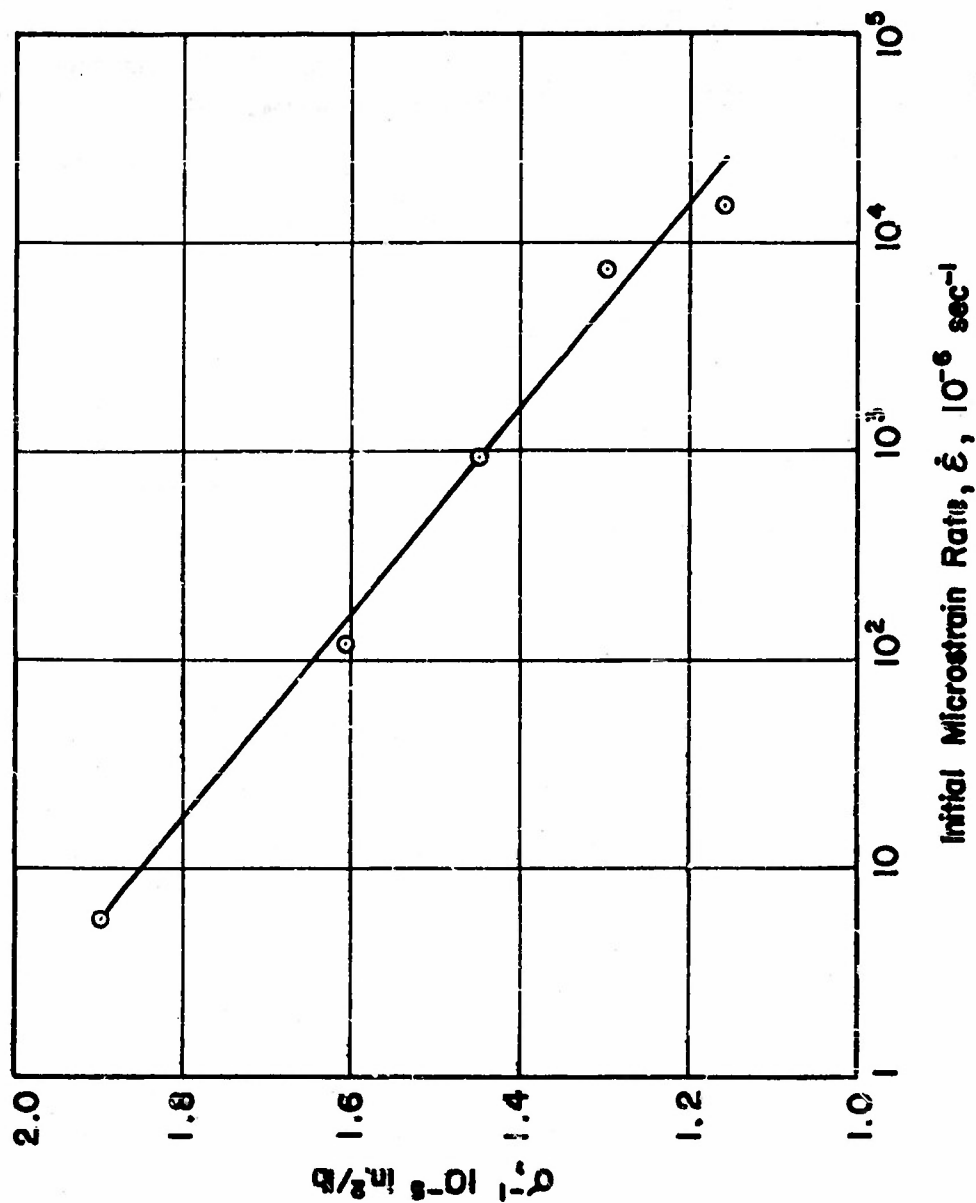


Fig. 23. Reciprocal of the Tensile Strain Rate vs. the Initial Preyield Plastic Strain Rate for Sintered Molybdenum at  $+76^{\circ}\text{F}$  ( $24^{\circ}\text{C}$ ).

The theoretical relationship between the initial pre-yield plastic strain rate and the applied stress is (7)

$$\ln \dot{\epsilon}_0 = - \frac{2\gamma_0^2 f(\gamma/\gamma_0)}{\sigma b k T} + C \quad (1)$$

Where  $\dot{\epsilon}_0$  = initial pre-yield plastic strain rate,  
 $\gamma_0$  = energy per unit length of a dislocation not bound by a Cottrell "atmosphere",  
 $\gamma$  = energy per unit length of a dislocation bound by a Cottrell "atmosphere",  
 $\sigma$  = applied tensile stress,  
 $b$  = the Burger's vector of a slip dislocation in molybdenum,  
 $k$  = Boltzmann's constant  
 $T$  = absolute temperature,  
 $C$  = an undertermined constant.

and  $f(\gamma/\gamma_0) = \cos^{-1}(\gamma/\gamma_0) - \gamma/\gamma_0 (1 - \gamma^2/\gamma_0^2)^{\frac{1}{2}}$

Thus the theory predicts a linear relationship between the logarithm of the initial strain rate and the reciprocal of the applied stress in agreement with experiment as shown in Fig. 23. The theoretical value of the slope of this relationship is

$$\frac{d(\ln \dot{\epsilon}_0)}{d(1/\sigma)} = - \frac{2\gamma_0^2 f(\gamma/\gamma_0)}{b k T} \quad (2)$$

and this may be compared quantitatively with the experimental value determined from the slope of the line in Fig. 23. The quantities in equation (2) are all accurately known except  $\gamma_0$  and  $\gamma$ . The energy per unit length of a free dislocation,  $\gamma_0$ , may reasonably be taken as

$$\gamma_0 = \frac{1}{2} G b^2 \quad (3)$$

where  $G$  is the shear modulus of molybdenum ( $G = 6.6 \times 10^{10}$  dynes/cm<sup>2</sup> according to A.S.M. Metals Handbook, 1948). This corresponds to 4.2 electron volts per atomic distance along the dislocation. Using this value of  $\gamma_0$  and the known values of  $b$ ,  $\kappa$  and  $\tau$  in equation (2), a value for the binding energy ( $\gamma_0 - \gamma$ ) of a dislocation in molybdenum with an "atmosphere" of interstitial solute atoms may be obtained by substituting the experimental value of the slope,  $d(\ln \dot{\epsilon}_0)/d(1/T)$  in the equation. This leads to a value of the binding energy of ( $\gamma_0 - \gamma$ ) = 0.022 electron volt per atomic distance along the dislocation line. This value is about three and one-half times as large as the value for dislocations in iron determined in the same manner. However, it is only about one-twentieth of the value estimated by Cottrell and Bilby (8). Thus the values of the binding energy determined in the above manner for dislocations in both iron and molybdenum deviate from the theoretical values by the same order of magnitude. A satisfactory resolution of this discrepancy has not yet been found.

### SUMMARY AND CONCLUSIONS

The initiation of yielding under rapidly applied constant stress has been investigated in two lots of fine grained ductile molybdenum. One lot was produced by the arc-casting method and the other by the powder metallurgy method. Both lots were worked at temperatures below the recrystallization temperature, followed by a recrystallization treatment which produced a rather uniform fine grained structure. These materials were found to exhibit distinct yield points in their static stress-strain relations, in agreement with the work of Bechtold and Scott (13).

The results of this investigation show that fine grained ductile molybdenum exhibits the phenomenon of delayed yielding in a form very similar to that observed in annealed low carbon steels. This result lends further support to the view proposed by Cottrell (10) on theoretical grounds that yield point and delayed yield phenomena are a consequence of the anchoring of dislocations in body centered cubic metals by interstitial solutes such as carbon and nitrogen. The present work gives a tentative indication that nitrogen may be more effective than carbon in anchoring dislocations or in strengthening grain boundaries in molybdenum.

A quantitative difference between the delayed yielding behavior of molybdenum and low carbon steel which is shown by the present work is that the upper limiting stress in the stress vs. delay time relations may be observed at higher temperatures in molybdenum than in steel. Thus experimental studies of this upper limiting stress phenomenon with a view toward its possible relationship to conventional transition temperatures might be more conveniently carried out with molybdenum than with iron or steel.

The initial rate of pre-yield plastic strain in the sintered molybdenum tested at room temperature is found to depend upon the applied stress in a manner which is in agreement with a dislocation theory of delayed yielding. Quantitative comparison of theory and

experiment in this respect yields a value for the binding energy of a dislocation in molybdenum with an "atmosphere" of interstitial solute atoms which is of the same order of magnitude as that found for iron.

#### ACKNOWLEDGMENT

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