

## TECHNICAL PROGRESS REPORT

**PROJECT TITLE:** PROCESSING, MICROSTRUCTURE AND CREEP BEHAVIOR OF Mo-Si-B-BASED INTERMETALLIC ALLOYS FOR VERY HIGH TEMPERATURE STRUCTURAL APPLICATIONS

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**PRINCIPAL INVESTIGATOR:** Professor Vijay K. Vasudevan

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**INSTITUTION:** The University of Cincinnati  
Department of Chemical and Materials Engineering  
501D ERC, Cincinnati, OH 45221-0012

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## ABSTRACT

This research project is concerned with developing a fundamental understanding of the effects of processing and microstructure on the creep behavior of refractory intermetallic alloys based on the Mo-Si-B system. During this year, the compressive creep behavior of a Mo-3Si-1B (in wt.%) alloy at 1100 and 1200°C were studied and related to the deformation mechanisms through electron microscopy observations of microstructural changes and deformation structures. The microstructure of this alloy was three-phase, being composed of  $\alpha$ -Mo, Mo<sub>3</sub>Si and T2-Mo<sub>5</sub>SiB<sub>2</sub> phases. Results of compressive creep tests at 1200 and 1100°C showed that the creep rates were quite high at stress levels between 250 and 500 MPa. Two minima in the creep strain rate versus strain data were noted, one at small strain values and the second at much larger strains. A stress exponent of 4.26 was obtained upon plotting the strain rate corresponding to the first minima versus stress, which suggests that dislocation climb and glide dominate the creep process in the early stages. On the other hand, the large strain, minimum creep rate versus stress data gave a stress exponent of  $\sim 1.18$ , which indicates diffusional mechanisms and recrystallization dominate the later stages of the creep process. At 1100°C, a stress exponent of 2.26 was obtained, which suggests that both diffusional and dislocation mechanisms contribute to the creep strain. Based on the minimum creep rate data at 1100°C and 1200°C, the activation energy for creep was determined to be 525 kJ/mole, which is somewhat higher than that reported for self diffusion in  $\alpha$ -Mo. Microstructural observations of post-creep samples indicated the presence of many voids in the  $\alpha$ -Mo grains and few cracks in the intermetallic particles and along their interfaces with the  $\alpha$ -Mo matrix. In addition, TEM observations revealed the presence of recrystallized grains and sub-grain boundaries composed of dislocation arrays within the grains, which suggests that climb and recrystallization processes are active in the late stages of creep. These results are presented and discussed.

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## 1. INTRODUCTION

This research project combines a novel processing, experimental and modeling approach, with detailed quantitative analysis of the influences of microstructure, in a basic study of the creep behavior of the next generation of refractory alloys based on the Mo-Si-B system. Through these studies, we will gain insight into the high-temperature creep behavior of these materials, including the effects of microstructure and the associated deformation, damage and failure features. A model Mo-rich Mo-Si-B alloy has been chosen for study, since it is representative of the new class of ductile-brittle systems, which, owing to their promising properties, are receiving evaluation for very high temperature structural applications. Further development of these multiphase materials hinges on having a solid understanding of composition-processing-microstructure-mechanical property relations, including creep, to which the present effort is directed. This project, which is closely coupled with ongoing activities at the Air Force Research Laboratory (AFRL) and Oak Ridge National Laboratory (ORNL) on these materials, focuses on three key areas to address issues related to the creep behavior: 1) basic materials processing and microstructural studies; 2) evaluation of the creep phenomenology and establishment of the constitutive behavior of the three-phase alloy and the individual phases within the alloy, including the effects of microstructure; and 3) theoretical modeling of the creep behavior based on analysis of associated creep data, creep mechanisms, damage processes and microstructural parameters. An important component of this research is to perform a detailed analysis of microstructures utilizing modern electron optical techniques, to provide quantitative information on aspects such as volume fraction, length scales, morphology and distribution of the second-phase intermetallics, the nature of their interfaces with the matrix, and the deformation, damage and failure processes during creep. This information, together with those of the creep properties and phenomenology will be utilized to rationalize and theoretically model the observed creep behavior.

## 2. EXECUTIVE SUMMARY

This research project is concerned with developing a fundamental understanding of the effects of processing and microstructure on the creep behavior of refractory intermetallic alloys based on the Mo-Si-B system. These materials are representative of the next generation of refractory alloys being considered for very high temperature structural applications. During this period of performance, we have studied the microstructure and creep properties of a three-phase Mo-3 wt.% Si-1 wt.% B (Mo-8.9Si-7.71B in at.%) alloy that was produced using a powder metallurgy and supplied by UES/AFRL. Microstructural observations using back-scattered electron imaging in a scanning electron microscope revealed that this alloy was three-phase, consisting of  $\alpha$ -Mo,  $\text{Mo}_3\text{Si}$  and T2- $\text{Mo}_5\text{SiB}_2$  phases, with the combined volume fraction of the latter two being  $\sim 30\%$ . Compression creep tests were conducted under inert atmosphere at  $1200^\circ\text{C}$  and  $1100^\circ\text{C}$  at stress levels ranging from 100 to 500 MPa in an Applied Test Systems creep frame equipped with computerized data acquisition. The results showed that the creep rates at  $1200^\circ\text{C}$  were quite high and the data of creep strain rate versus time or strain displayed two minima, one at small strains and the second at much larger strains. The former minima varied linearly from a value of  $4.23 \times 10^{-5} \text{ min}^{-1}$  at 250 MPa to  $7.95 \times 10^{-4} \text{ min}^{-1}$  at 500 MPa, whereas the latter were  $3.58 \times 10^{-5} \text{ min}^{-1}$  and  $8.04 \times 10^{-5} \text{ min}^{-1}$  at the same stress levels, respectively. The stress exponent from the data corresponding to the first minima was determined to be  $\sim 4.26$ , which suggests that dislocation climb-glide creep dominates the creep process in the early stages. This value of the stress exponent is in the range of 2-7 reported for similar alloys by other investigators. The stress exponent determined from the second minimum creep rate data was  $\sim 1.18$ , which is near the value of 1 expected for diffusional creep and recrystallization mechanisms. At  $1100^\circ\text{C}$ , the minimum creep rates varied linearly with stress from  $1.66 \times 10^{-7} \text{ min}^{-1}$  at 100 MPa to  $2.13 \times 10^{-6} \text{ min}^{-1}$  at 300 MPa; the corresponding stress exponent was determined to be  $\sim 2.26$ , which suggests that both diffusional and dislocation mechanisms contribute to the overall creep process. The activation energy,  $Q$ , for creep was determined to be  $\sim 525 \text{ kJ/mole}$ , which is somewhat higher than the value of  $400 \text{ kJ/mole}$  reported for self diffusion in  $\alpha$ -Mo. Microstructural observations of post-creep samples indicated shape change and deformation of the  $\alpha$ -Mo grains, together with the presence of many voids in these grains. A few cracks in the intermetallic particles and along their interfaces with the  $\alpha$ -Mo matrix were also observed. TEM

observations of the crept samples revealed the presence of recrystallized  $\alpha$ -Mo grains containing small-angle sub-grain boundaries made up of dislocation arrays. A few dislocations and sub-grains were also noted within the Mo<sub>3</sub>Si and T2 grains. These observations suggest that the bulk of the deformation and strain during creep is carried by the  $\alpha$ -Mo.

### 3. TECHNICAL APPROACH

The basic strategy being utilized is to first establish the creep phenomenology and constitutive behavior under compression of a selected three-phase  $\alpha$ -Mo + T2-Mo<sub>5</sub>SiB<sub>2</sub> + Mo<sub>3</sub>Si material and of each of the three constituent pure phase materials. (Data for single-phase T2 is available in the literature, whereas those for the  $\alpha$ -Mo and Mo<sub>3</sub>Si have to be generated experimentally). The second step is to arrive at suitable methods for representing microstructure of these materials in three-dimensions. The third step is to model compressive creep by time-dependent finite element modeling (FEM) from constituent phase properties and appropriate representation of microstructure as a mesh. The fourth step will be to determine the creep behavior under tensile conditions and evaluate the damage processes. Tensile creep testing of these materials is a non-trivial problem because of the high strengths of these materials and high temperatures (1000-1400°C) involved, and will require special test sample designs. Lastly, another three-phase alloy composition will be evaluated to study volume fraction/microstructure effects. The aim of the modeling effort is to predict material behavior and that of the experiment to validate the predictions.

### 4. EXPERIMENTAL DETAILS

A three-phase  $\alpha$ -Mo + T2 + Mo<sub>3</sub>Si Mo-3 wt.% Si-1 wt.% B (Mo-8.9Si-7.71B in at.%) alloy was received from UES/AFRL (Dayton, OH). A powder metallurgy route was used by Plansee (Austria) to process this material into bulk form. Thermal influences on microstructure evolution were studied and conditions were established to obtain a well-defined three-phase microstructure. Samples were cut from the ends (undeformed parts) of the bend test bars, prepared metallographically and fine polished with colloidal silica prior to observations of the microstructures. The microstructures were observed in a FEI XL30 FEG-ESEM using back-scattered electron (BSE) imaging. The chemical compositions of phases present were analyzed by energy dispersive x-ray spectroscopy (EDS) using an EDAX Genesis 4000 EDS system attached to the SEM. Samples measuring 5x5x10 mm were sectioned using electro discharge machining (EDM) for the creep tests. Considerable effort was directed toward getting the creep testing equipment and associated hardware/software at AFRL operational. The creep tests were conducted on a creep frame constructed by Applied Testing Systems. The creep frame consists of lever arm system for loading with an automated ram system that allows for maintaining constant stress or strain rate, a furnace for elevated temperature testing, and an environmental chamber for testing in controlled atmospheres. During experiments, time, displacement, temperature, and applied load measurements are performed by a data acquisition system and collected by a PC. Compression creep tests were conducted on the Mo-3Si-1B alloy samples at 1100 and 1200°C and stress levels ranging from 100 to 500 MPa. Data on creep strain and creep rate with time under these conditions has been obtained.

The microstructures of the crept samples were observed using BSE imaging in the FEI XL-30 ESEM. In addition, thin foils for transmission electron microscopy (TEM) were prepared by sectioning thin strips from the crept samples, then punching 3-mm diameter disks, followed by dimpling using a Fischione Model 150 dimple grinder and then ion-milling to perforation using a Fischione Model 1010 low-angle ion milling system. The foils were observed in a Philips CM20 TEM operated at 200 kV and photographs under bright field (BF), dark field (DF), weak-beam dark field (WBDF) and selected area diffraction (SAD) modes were recorded. The chemical composition of phases present was also analyzed using an EDAX Genesis 2000 energy dispersive x-ray spectroscopy (EDS) system attached to the same TEM.

## 5. RESULTS AND DISCUSSION

### 5.1 Microstructure of Heat Treated, Undeformed Material

As reported in last year's report, the microstructure of the thermo mechanically processed and heat treated Mo-3Si-1B alloy (Figure 1) was composed of three phases appearing in bright, gray and dark contrast, which were confirmed by EDS analysis as being the  $\alpha$ -Mo, Mo<sub>3</sub>Si and T2-Mo<sub>5</sub>SiB<sub>2</sub> phases, respectively. Some grain alignment of the  $\alpha$ -Mo and intermetallic particles is evident as a result of extrusion. The intermetallic particles showed a tendency for clustering together and bands of these were separated by the  $\alpha$ -Mo regions. The average size of Mo<sub>3</sub>Si and T2 particles was  $\sim 10$  nm and their total volume fraction was estimated to be  $\sim 30\%$ .

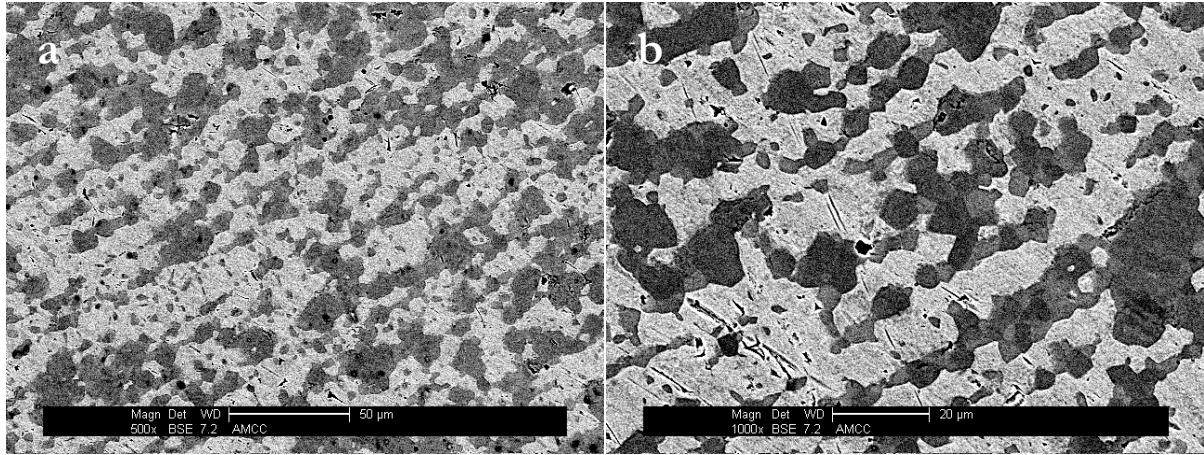


Figure 1. BSE micrographs showing microstructure of Mo-3Si-1B alloy. (a) 500X and (b) 1000X.

### 5.2 Compression Creep Test Results

The results of compression creep tests on the Mo-3Si-1B alloy at 1200°C at stress levels varying from 250 to 500 MPa are shown in Figure 2 in the form of a creep strain rate versus strain plot. As can be seen in Figure 2, the creep curves show three distinct stages, an initially non-linear part with a greater slope/rate followed by a minimum at small strains, then an increase, followed by a second minimum at much larger strains. These features are particularly evident at the higher stress levels. Furthermore, with an increase in the applied stress both the rate of strain and magnitude of strain increases appreciably, with values of the latter of  $\sim 50\%$  at 500 MPa at the end of the test. The minimum creep rate corresponding to the first and second minima determined from these data are shown as a function of stress in Table 1 and plotted in Figure 3.

Table 1. Minimum Creep Rate as a Function of Stress at 1200°C for the Mo-3Si-1B Alloy.

| Stress (MPa) | Minimum Creep Rate (min <sup>-1</sup> ) |                         |
|--------------|---|-------------------------|
|              | First Minimum                           | Second Minimum          |
| 250          | 4.23 x 10 <sup>-5</sup>                 | 3.38 x 10 <sup>-5</sup> |
| 300          | 8.39 x 10 <sup>-5</sup>                 | 4.86 x 10 <sup>-5</sup> |
| 400          | 2.97 x 10 <sup>-4</sup>                 | 6.07 x 10 <sup>-5</sup> |
| 500          | 7.95 x 10 <sup>-4</sup>                 | 8.04 x 10 <sup>-5</sup> |

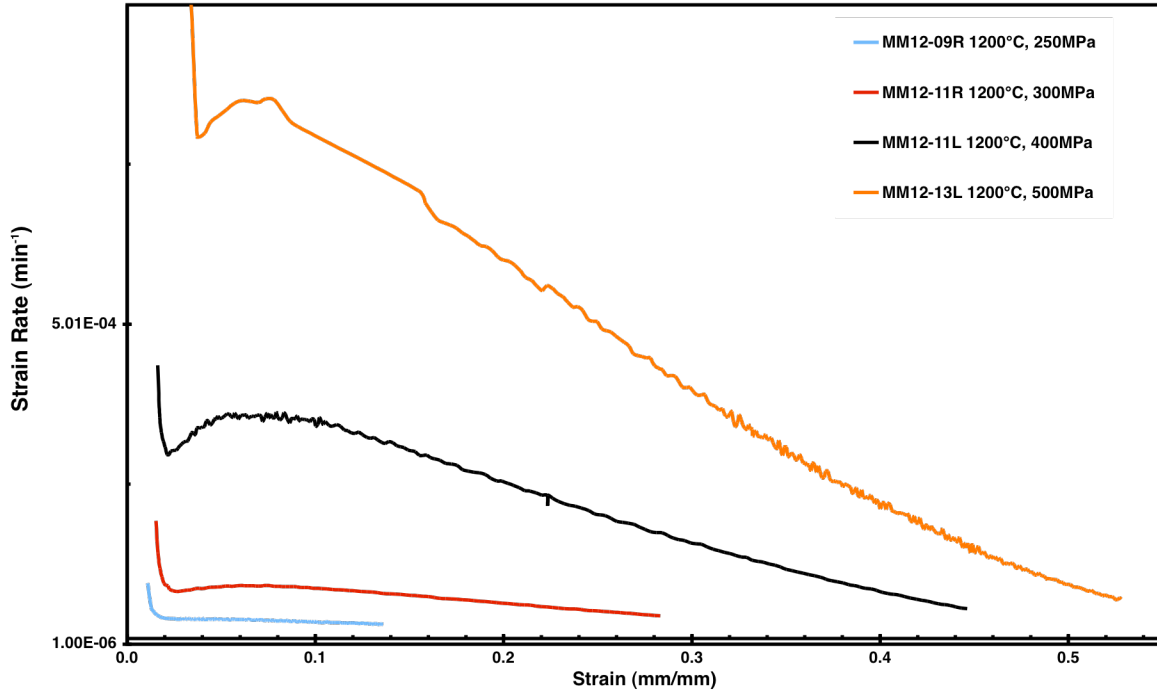


Figure 2. Plot of compressive creep rate versus strain at various stress levels for the Mo-3Si-1B alloy tested at 1200°C.

The data in Table 1 and Figure 3 show that the minimum creep rate corresponding to both minima increases with stress and that the creep behavior obeys the Mukherjee-Sherby-Dorn creep law of the type:

$$\dot{\epsilon}_{\min} = A\sigma^n \exp\left(-\frac{Q}{RT}\right) \quad (1)$$

Furthermore, a natural log-log plot of the minimum creep rate versus stress data corresponding to the first minimum, Figure 3(a), gives an excellent straight line fit, yielding a stress exponent of  $\sim 4.26$ . This value is in the range of 3-5 expected for power law/dislocation creep, suggesting that this (i.e., dislocation climb-glide) is the dominant creep mechanism in the initial stages of creep in this alloy. This is in agreement with previous studies on compressive deformation/creep of three-phase  $\alpha$ -Mo + Mo<sub>3</sub>Si + T2 alloys, where stress exponents ranging from 2.1-3.9 [1] and from 2.5-7.1 [2] have been reported, though the compositions and hence volume fraction of the intermetallic phases of the alloys studied differ from that of the present work. On the other hand, plotting the minimum creep rate corresponding to the second minimum versus stress, Figure 3(b) and an excellent power law fit to this data yields a stress exponent of 1.18, which is close to the value of 1 expected for diffusional (Nabarro-Herring or Coble) creep and/or recrystallization. These results suggest that diffusional and recrystallization processes dominate creep at the later stages.

The compression creep curves of the samples tested at 1100°C at 100, 150 and 300 MPa are shown in Figure 4. Only a single minimum in the creep rate versus strain plot was detected, with the corresponding values shown in Table 2. A natural log-log plot of minimum creep rate versus stress is shown in Figure 5 and a straight line fit to this data yields a stress exponent of 2.26. This value is in the range reported for other Mo-Si-B alloys by previous investigators [1,2] and suggests that both diffusional and dislocation creep processes contribute to the overall creep rate at this temperature over the stress range studied. Furthermore, from a plot of the  $\ln(\text{minimum creep rate})$  at 1100 and 1200°C vs  $10000/T$ , Figure 6, the activation energy for creep is obtained as 525 kJ/mole. This value is somewhat higher than that reported for other Mo-Si-B alloys by previous investigators [1,2] and for self-diffusion in  $\alpha$ -Mo (400 kJ/mole).

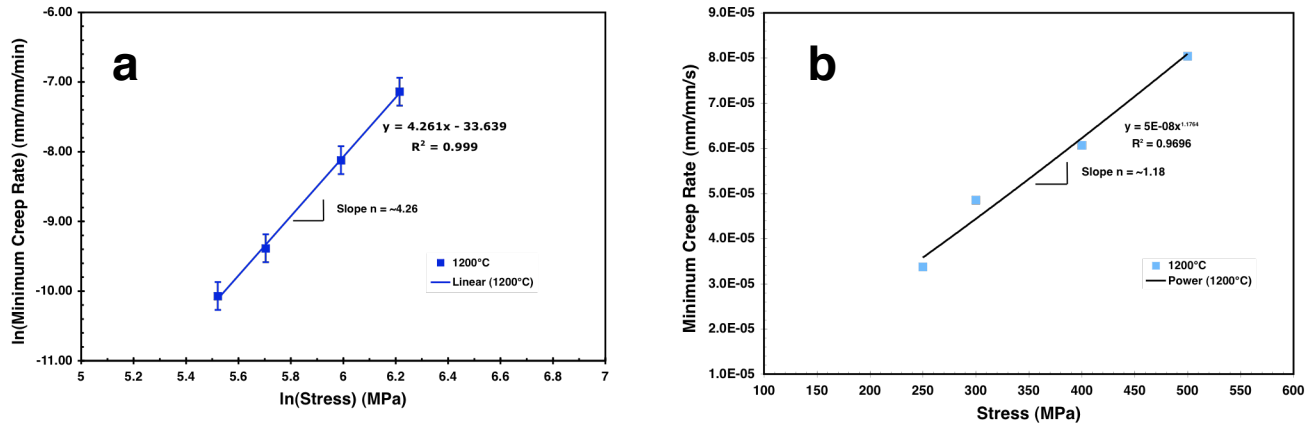


Figure 3. Minimum creep rate versus stress at 1200°C for the Mo-3Si-1B alloy. (a) First minimum and (b) second minimum.

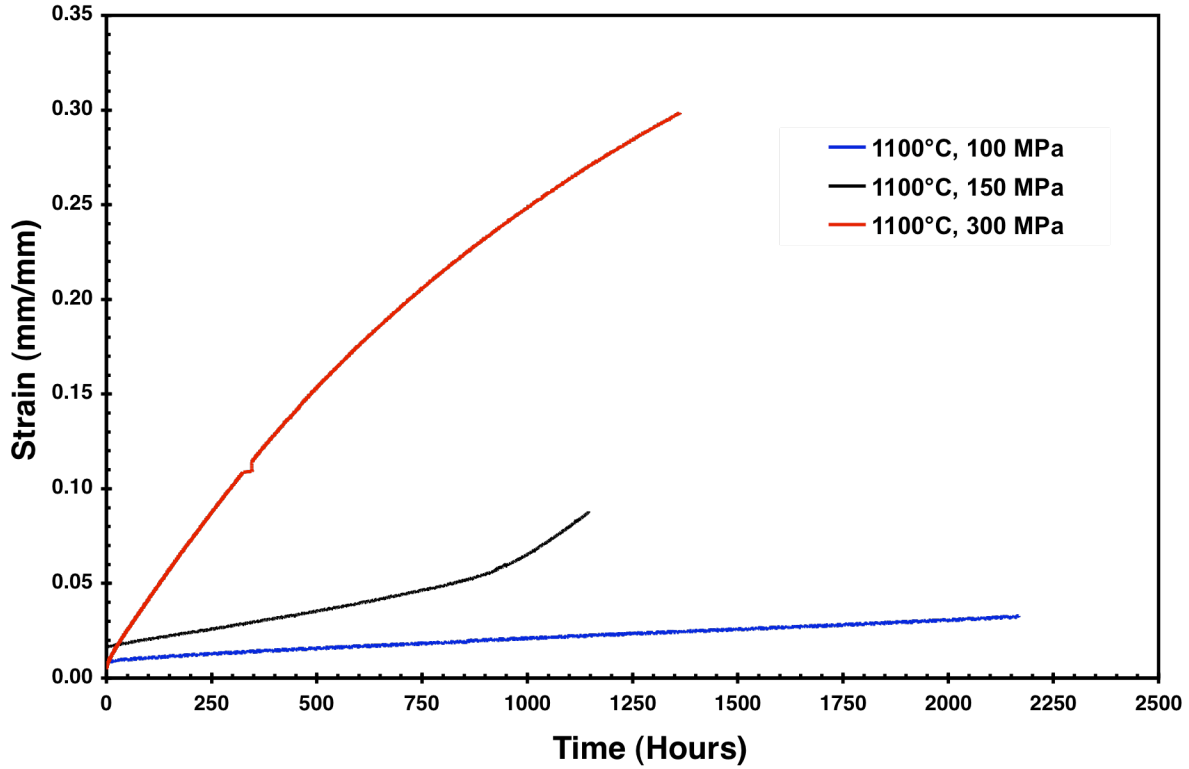


Figure 4. Plot of compressive creep strain versus time at various stress levels for the Mo-3Si-1B alloy tested at 1100°C.

Table 2. Minimum Creep Rate as a Function of Stress at 1100°C for the Mo-3Si-1B Alloy

| Stress (MPa) | Minimum Creep Rate ( $\text{min}^{-1}$ ) |
|--------------|--|
| 100          | $1.66 \times 10^{-7}$                    |
| 150          | $6.36 \times 10^{-7}$                    |
| 300          | $2.13 \times 10^{-6}$                    |

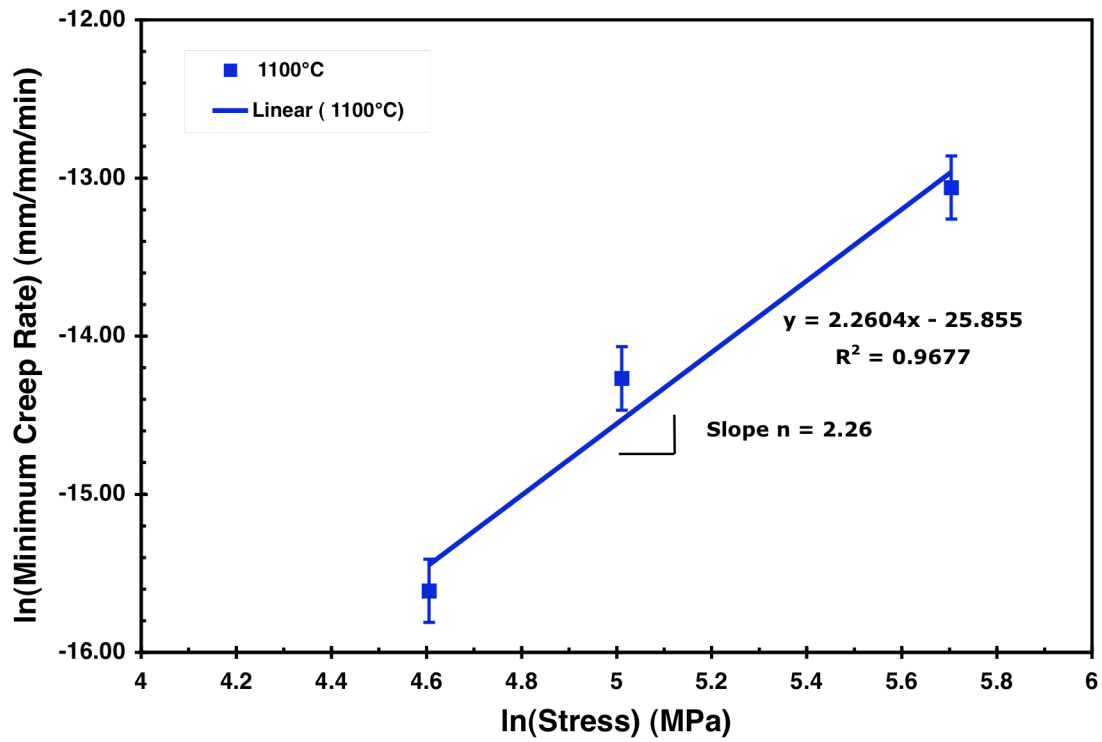


Figure 5.  $\ln$ - $\ln$  plot of Minimum creep rate versus stress for the Mo-3Si-1B alloy tested at 1100°C.

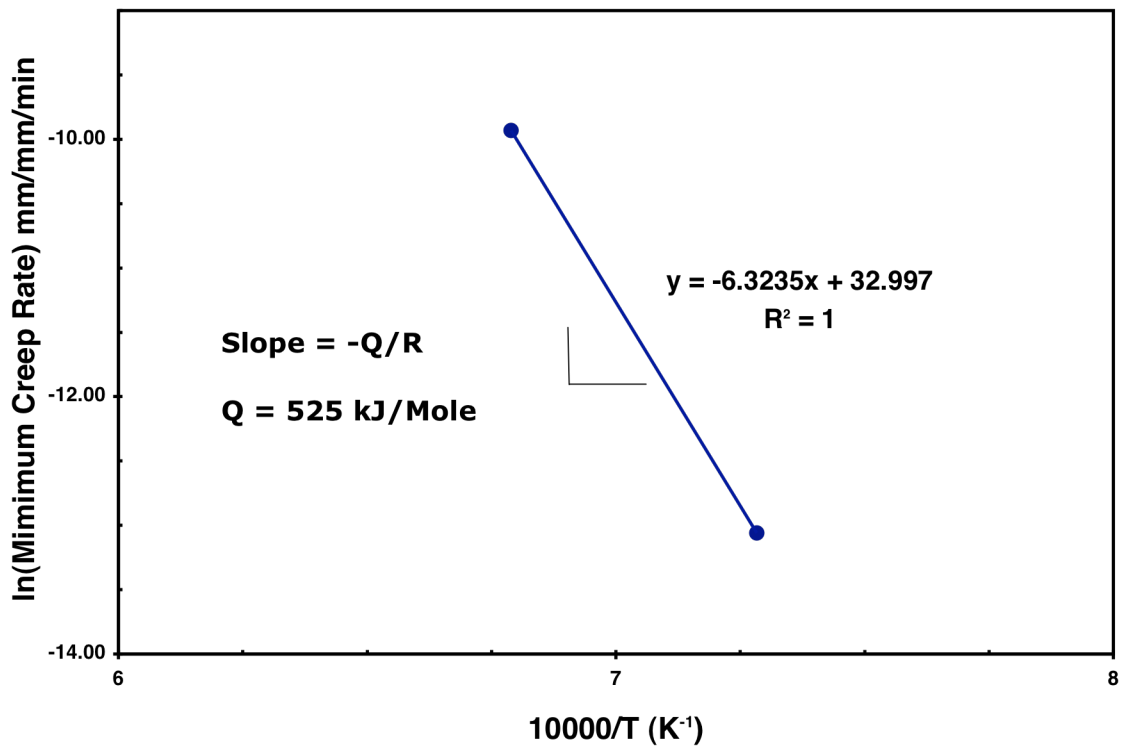


Figure 6.  $\ln(\text{Minimum Creep Rate})$  versus  $10000/T$  plot for the Mo-3Si-1B alloy tested at 1100 and 1200°C under a stress of 300 MPa.

### 5.3 Microstructure of Crept Samples

Visual examination of the crept samples showed evidence for barreling, but no macro cracks or other defects were visible. Specimens were sectioned from the crept samples in a direction normal to the compression axis, metallographically prepared and their microstructures observed using BSE imaging in the SEM. A representative BSE micrograph of the samples crept at 1200°C at a stress of 300 MPa is shown in Figure 7.

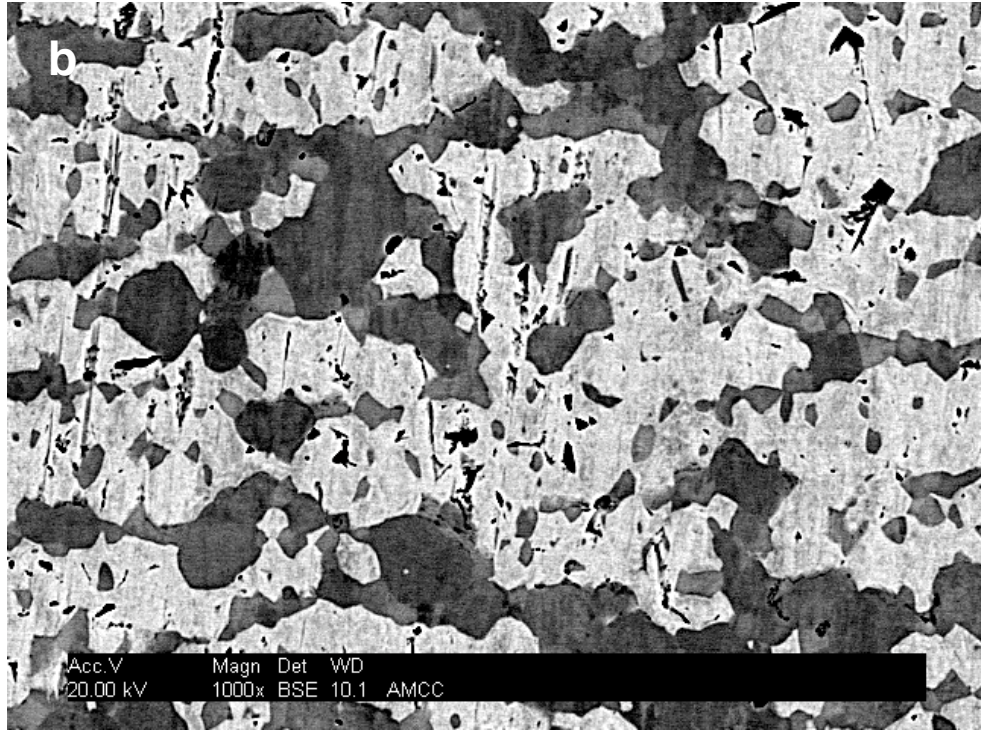


Figure 7. BSE micrograph showing microstructure of the Mo-3Si-1B alloy crept at 1200°C under a stress of 300 MPa.

A change in shape of the  $\alpha$ -Mo grains can be clearly seen, whereas the  $\text{Mo}_3\text{Si}$  and T2 particles appear relatively unchanged compared with the uncrept material. Furthermore, many voids and cracks can be seen within the  $\alpha$ -Mo grains and a few are present within the intermetallic particles and along their interfaces with the  $\alpha$ -Mo grains.

TEM micrographs of the sample creep-tested at 1200°C under a stress of 300 MPa are shown in Figures 8 and 9. These micrographs indicate that the bright  $\alpha$ -Mo regions seen in the BSE micrograph in Figure 7 are actually composed of recrystallized grains of  $\alpha$ -Mo, 1-5  $\mu\text{m}$  in size. These grains also contain a relatively low dislocation density, as well as low-angle sub-grain boundaries made up of dislocation arrays (Figure 8) The  $\text{Mo}_3\text{Si}$  and T2 grains contain a few dislocations, Figure 9. In addition, the latter grains are composed of many subgrains. These various observations indicate that the majority of the strain during creep is carried by the  $\alpha$ -Mo grains and that at large strains recrystallization of the latter is the predominant creep mechanism, consistent with the observed stress exponent near 1.

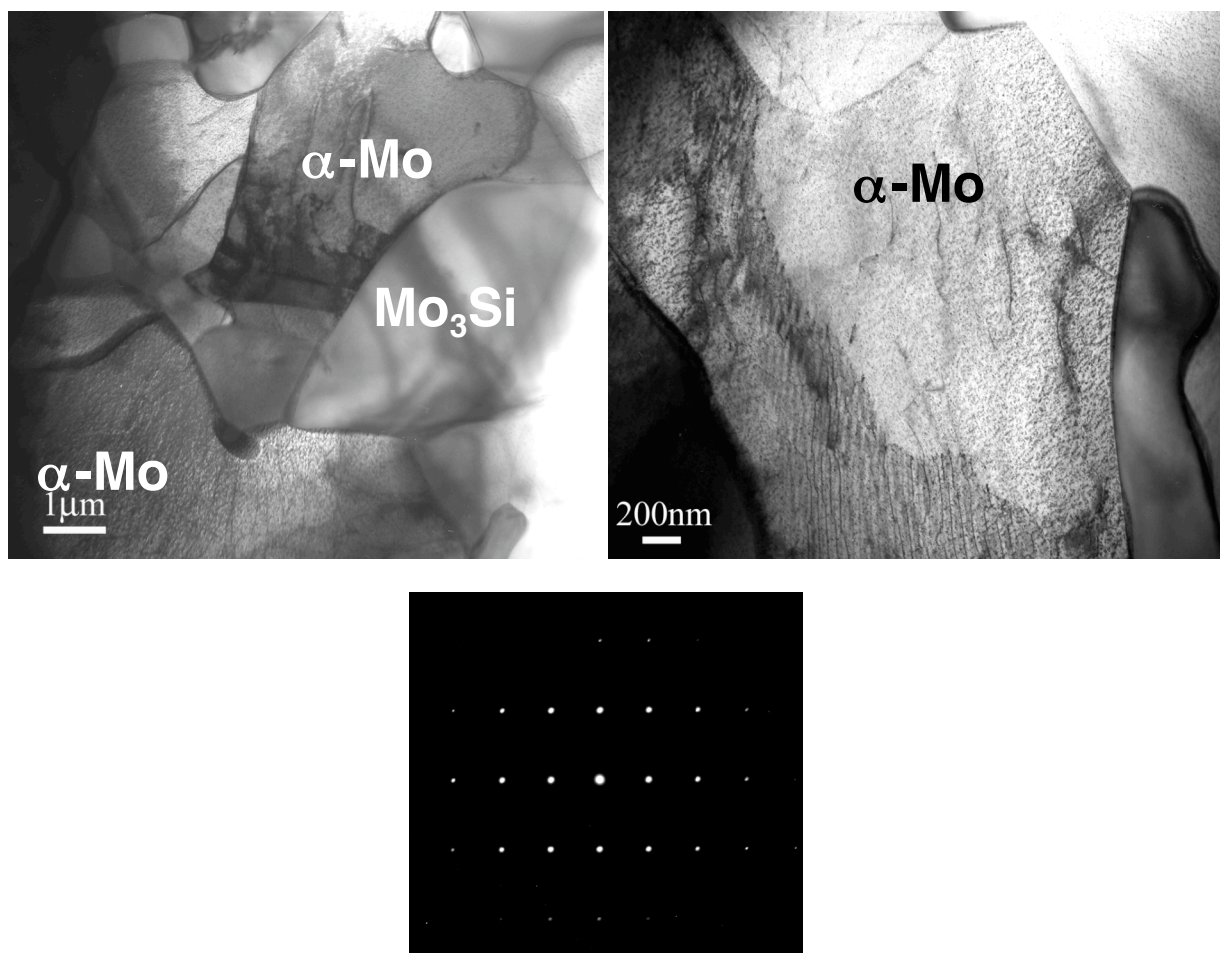


Figure 8. BF TEM micrographs (a,b) and  $[011]$   $\alpha$ -Mo SAD pattern (c) showing the microstructure of the Mo-3Si-1B alloy crept at  $1200^\circ\text{C}$  under a stress of 300 MPa.

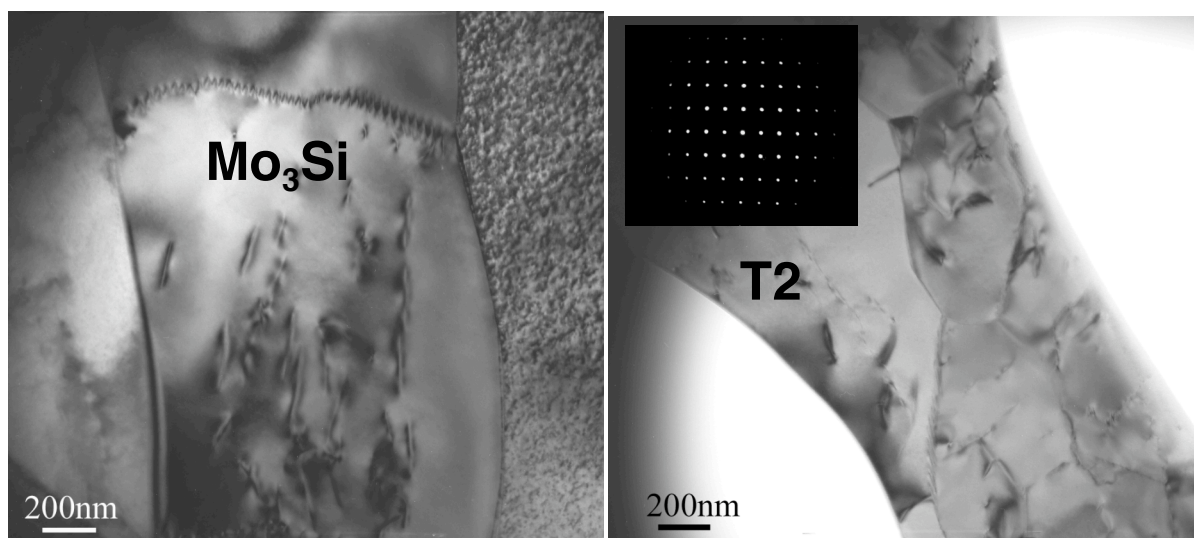


Figure 9. BF TEM micrographs showing (a)  $\text{Mo}_3\text{Si}$  grain and (b) T2- $\text{Mo}_5\text{SiB}_2$  grain (inset  $[100]$  T2 SAD pattern) in the Mo-3Si-1B alloy crept at  $1200^\circ\text{C}$  under a stress of 300 MPa.

## 6. CONCLUSIONS

The main conclusions arising from work on a Mo-3Si-1B alloy during this project period of performance from July 1, 2004 to June 30, 2005 are:

1. The microstructure of this alloy is three-phase, consisting of  $\alpha$ -Mo, Mo<sub>3</sub>Si and T2-Mo<sub>5</sub>SiB<sub>2</sub> phases, with the combined volume fraction of the latter two being ~30%.
2. Compression creep tests showed that the creep rates at 1200°C were quite high and the creep rate versus strain data revealed two minima, the first at small strains and the second at much larger strains.
3. From the creep rate data at 1200°C, the stress exponent corresponding to the first minima was determined to be ~4.26, which is in the range of 2-7 reported for other Mo-Si-B alloys by previous investigators and indicates that dislocation climb-glide dominates the creep process in the initial stages. The stress exponent corresponding to the second minima was ascertained to be ~1.18, which is near the value of 1 and indicates that diffusional creep and/or recrystallization mechanisms dominate the creep process in the later stages.
4. From the creep data at 1100°C over the stress range of 100-300 MPa, the stress exponent was determined to be ~2.26, which suggests that both diffusional and dislocation mechanisms contribute to the creep process at this temperature and stress range.
5. The activation energy for creep was determined to ~525 kJ/mole for the Mo-3Si-1B alloy between 100 and 1200°C. This value is higher than those reported for other Mo-Si-B alloys by previous investigators and for self-diffusion (400 kJ/mole) in  $\alpha$ -Mo.
6. Microstructural observations of post-crept samples indicated shape change and deformation of the  $\alpha$ -Mo grains, together with the presence of many voids in these grains. A few cracks in the intermetallic particles and along their interfaces with the  $\alpha$ -Mo matrix were also observed.
7. TEM observations of the crept microstructure gave evidence for recrystallized grains of  $\alpha$ -Mo of size 1-5 $\mu$ m, as well as the presence of low-angle, sub-grain boundaries made up of dislocation arrays within these grains. The Mo<sub>3</sub>Si and T2 grains contained a relatively low dislocation density. These various observations suggest that the bulk of the deformation and strain during creep is carried by the  $\alpha$ -Mo and that recrystallization of this phase is responsible for the large creep strain.

## 7. REFERENCES CITED

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2. A. P. Alur, N. Chollacoop and K. S. Kumar, *Acta Mater.*, **52**, 5571-5587 (2004).

## 8. PLANS FOR THE NEXT YEAR/PERIOD OF PERFORMANCE

- Continue TEM analysis of microstructure and damage processes in the crept samples
- Conduct compressive creep testing of pure phase  $\alpha$ -Mo and Mo<sub>3</sub>Si materials at selected temperatures and stresses matching those used for the three-phase alloy.
- Conduct compressive creep tests on Mo-Si-B alloys received from Dr. Joachim Schneibel at ORNL.
- Machine samples for tensile creep testing and complete this testing at selected temperatures and stresses.

- Complete modeling of creep in these alloys.

## **9. LIST OF PUBLICATIONS, PRESENTATIONS AND STUDENTS SUPPORTED FROM THE GRANT**

### ***9.1 Publications and Presentations***

A paper based on the results obtained thus far was presented at the 2005 TMS/ASM Fall Meeting in Pittsburgh, PA in October (2005). An article based on the results is being prepared for submission to the journal *Intermetallics*.

### ***9.2 Students Supported Under this Grant***

Brian Riestenberg, Ph.D. student in the Department of Chemical and Materials Engineering, University of Cincinnati.

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