Creep of an Oxidation Resistant Coated Mo-9Si-8B Alloy

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Abstract

In order to evaluate the impact of an oxidation resistant coating on the structural performance of a Mo-9Si-8B alloy tensile creep experiments were conducted at 1200 °C. After a plastic strain of 6 % the creep rates of the coated samples compared favorably with the reported values for uncoated samples. Moreover, the coating structure was maintained during creep deformation and the coating exhibited a self-healing capability.

Introduction

The current paradigm of nickel-based superalloys has approached the very limits of high-temperature strength and stability, often operating at 0.9 T_m, of the incipient melting temperature [1]. In order to realize an increase in hot section intake temperatures alternative material systems must be explored. The Mo–Si–B alloy system is a promising candidate due to a number of exceptional properties possessed by the multiphase microstructures. For example, at 1200 °C the flow stress at 4 % strain in a three phase Mo-9Si-8B alloy is more than twice that for the commercial TZM alloy [2]. In earlier studies, oxidation-prone molybdenum was shown to be protected from oxidizing environments by the Mo–Si–B based coating structure to temperatures as high as 1600 °C [3]. In order for such a coating to be implemented into a gas turbine engine, it must demonstrate stability and robustness in a wide variety of environmental and operation conditions.

Numerous studies on high temperature oxidation behavior in Mo-Si-B alloys reveal that the alloy compositions with the best performance do not have satisfactory mechanical properties (notably low temperature ductility and fracture toughness) due to the high volume fraction of silicide phases needed for the oxidation resistance [4]. Similarly, alloy compositions with satisfactory mechanical properties usually exhibit poor oxidation resistance due to the continuous Mo_{ss} phase [5]. One strategy to address this dilemma is to apply an oxidation resistant coating. A successful coating has been developed based upon a co-deposition of B and Si by pack cementation that exhibits thermodynamic and mechanical compatibility and self-healing [6]. The coating has been

demonstrated to provide a robust environmental resistance to attack by oxidation, CMAS, hot corrosion by molten salt and water vapor [3]. Besides the environmental resistance it is important to assess the influence of the coating on the mechanical behavior. In this work it is demonstrated that the high temperature creep performance of a Mo-9Si-8B alloy is essentially unaffected by the coating, proving the protective capability of the coating under application relevant conditions with superimposed plastic deformation.

Experimental Procedures

The Mo-9Si-8B alloy (in at.%) was manufactured from elemental powders Mo, Si and B with purities of 99.95%, 99.9% and 98% respectively. Mechanical alloying was carried out using a planetary ball mill (Retsch PM 400) with WC balls, a powder to ball ratio of 1:12 and a speed of 200 rpm [5]. Compaction was carried out using the field assisted sintering technique (FAST) at 1600 °C and 50 MPa for hold times of 15 min. As a result, buttons with a diameter of 50 mm and a height of 10 mm and a residual porosity of < 2 % were produced [7].

After homogenization treatment at 1600 °C for 100 h, dogbone shaped tensile samples with a total length of 35 mm, a gage length of 15 mm and a square cross-section of 3 x 4 mm^2 were manufactured from above buttons by EDM (see insert in Fig. 3). Prior to pack cementation the gage section of these samples was ground to a 2500 grit finish. The tensile samples were then embedded in a powder mixture consisting of 62.5 wt.% Al_2O_3, 35 wt.% Si and B mixture (Si/B = 35/1) and 2.5 wt.% NaF for pack cementation in an Ar atmosphere at 1000 °C for 40 h to co-deposit the Si and B. A detailed description of the pack cementation process can be found elsewhere [8,9].

Tensile creep tests were on the one hand carried out under constant true stresses at 1200 °C in a Zwick universal testing device equipped with a Maytec vacuum furnace (<10^{-4} Pa). The creep strain as by inductive displacement transducers attached to the sample gage section was continuously monitored to test that the coating does not affect the creep behavior. For further details see [10]. On the other hand, further creep tests were performed in air in order to verify the adhesion and self-healing capability of the coating.

Results

The initial coating structure after pack cementation is displayed in Fig. 1a where (from left to right) an outer amorphous borosilica layer is in contact with a $MoSi_2$ layer which in turn is in contact with a mixed MoB and Mo_5SiB_2 (T_2) layer before reaching the three phase Mo_{ss}+Mo_3Si+T_2 substrate. After conditioning at 1400 °C for 10 h in air the coating structure evolves into the structure shown in Fig.1b where the top and bottom portion of the $MoSi_2$ layer converts to Mo_5Si_3 which is followed by a MoB/T_2 layer before reaching the substrate. Exposure of samples with this coating at 1700 °C for periods of 25 hours (without mechanical loading) indicated a negligible mass change of less than 1.7 mg/cm^2 [11].

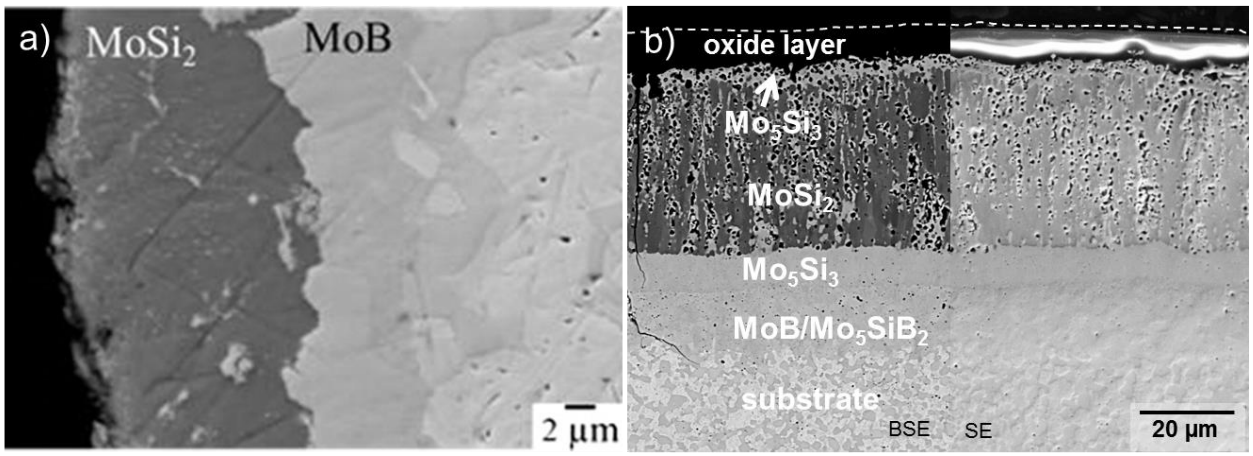

Fig. 1 a) BSE-micrograph of the as-packed sample [3], b) BSE-micrograph left and SE-micrograph right of the conditioned coated Mo-9Si-8B sample

Following exposure at 1200 °C in air and under an applied creep stress of 50 MPa the sample exhibited a full retention of the coating after 6 % plastic strain as shown in plan view in Fig. 2a and in cross section in Fig. 2b. There was no evidence for coating spallation or oxidation of the substrate. Some crack development was detected normal to the tensile direction (Fig. 2b) but it is evident that the borosilica was able to form and flow into the cracks as a self-healing process (white arrows in Fig. 2b II). In essence the same observations were made for creep tested samples at 1200 °C under a stress of 100 MPa.

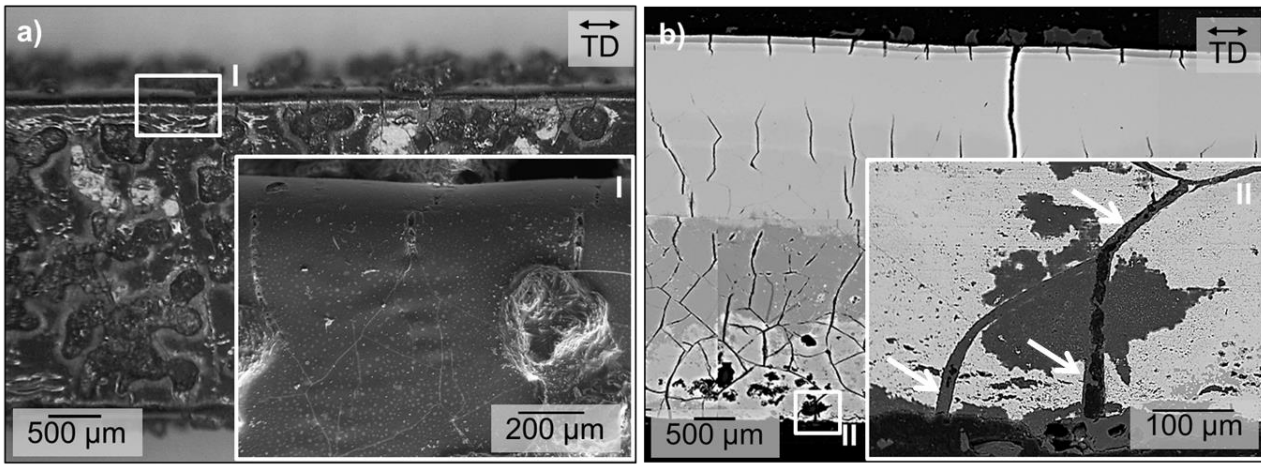

Fig.2 a) In plan view of coated sample surface after 6 % plastic strain, b) cross section of coated sample after 6 % plastic strain illustrating the flow of borosilica to fill cracks (TD refers to tensile direction)

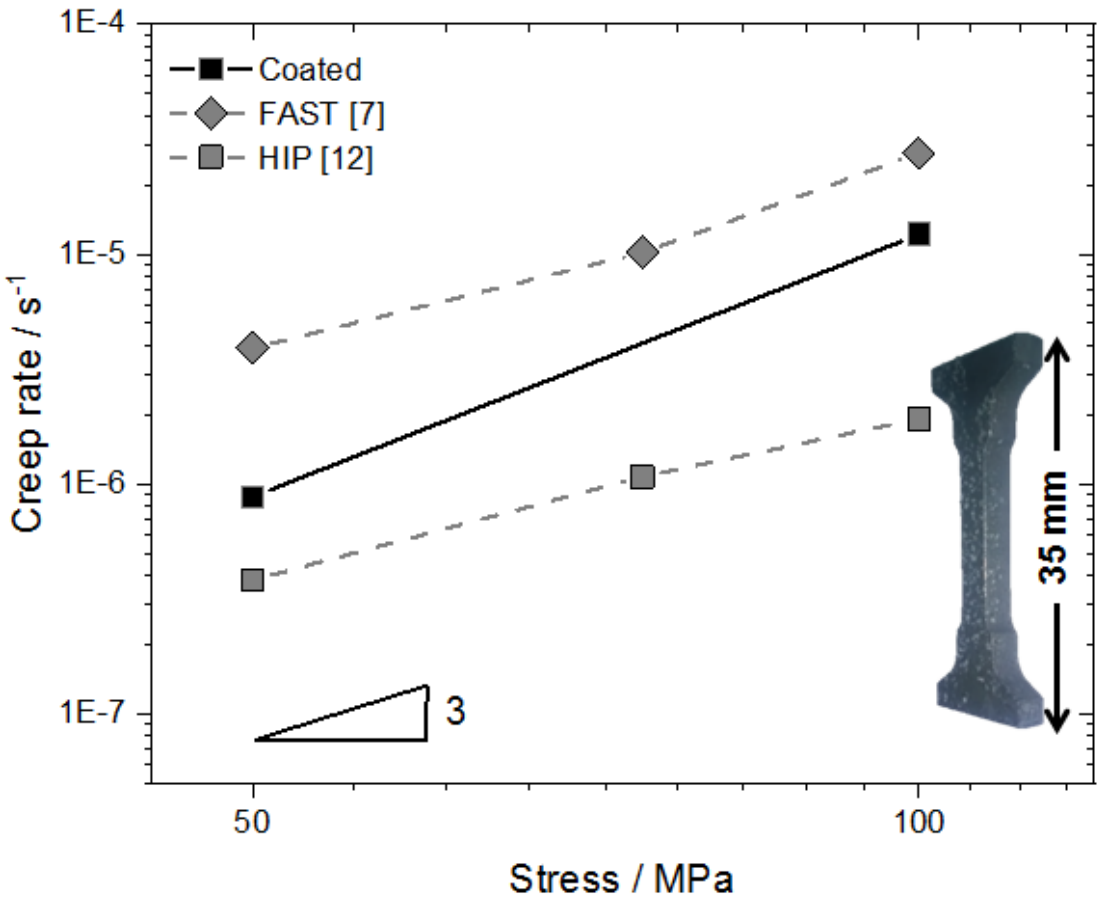

Fig. 3) Double logarithmic plot of the minimum strain rate vs. applied stress at 1200 °C for Mo-9Si-8B alloys with different grain sizes. The grain size for the uncoated FAST sample is 0.75 µm [7] and that for the uncoated HIP sample is 1.5 µm [12] while that for the coated sample has an intermediate value of about 1.3 µm.

The minimum creep rates for the coated samples were compiled with literature results obtained on uncoated material and are plotted in Fig. 3 versus the applied creep stress. Two conclusions can be drawn from this comparison: (i) as discussed in [7] for a series of Mo-9Si-8B alloys with different Zr additions, a clear tendency for increased creep rates with decreasing grain size is noted. This was explained in [7] by the increased contribution of grain boundary sliding processes and our coated samples fit very well into this trend; (ii) no obvious impact of the additional coating on the course of the minimum creep rates is found which expresses itself in the similar stress exponents ranging between 2.3 and 3.7. This supports the above observations in that the coating is well adherent and obviously able to protect the underlying substrate well.

Conclusions

The effect of an oxidation resistant Mo-Si-B based coating on the deformation of a Mo-9Si-8B alloy was evaluated during tensile creep experiments at 1200 °C. The observed creep rates for coated samples at stresses between 50 and 100 MPa are consistent with the reported values for uncoated samples demonstrating that the coating had a negligible effect on the creep behavior. At a plastic strain of 6 % some cracking was observed normal to the tensile direction, but the cracks were filled by flowing borosilica glass indicating a self-healing capability. The coating structure was maintained during creep deformation.

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