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A study of hot deformation behavior and microstructural characterization of Mo–TZM alloy

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ABSTRACT

Hot compression testing of Mo–TZM (Mo–0.5Ti–0.1Zr–0.02C) alloy was carried out between 600 and 900 °C employing strain rates from 0.001 s^{-1} to 1 s^{-1}. Both the constant strain rate and strain rate change test results showed that Mo–TZM possesses low strain rate sensitivity in this temperature range. Activation energy calculated by using strain rate change data and plotting temperature compensated strain rate (Z) vs. shear modulus corrected flow stress (σ/G) was found to be 290 kJ/mol. Electron back scattered diffraction (EBSD) and transmission electron microscopic (TEM) results obtained from the rapidly cooled deformed specimens revealed the formation of subgrains. Flow stress–plastic strain results and misorientation angles between subgrains showed an anomalous behavior at 800 °C.

1. Introduction

For the new generation high temperature nuclear reactors, there is a demand for the materials capable of withstanding the aggressive environment with respect to stress, radiation, liquid metal corrosion, etc. at high temperatures. Molybdenum and niobium base alloys are being considered as the most promising materials [1] for such applications where conventional nickel or cobalt base superalloys cannot be used. Mo possesses high strength and creep resistance at high temperatures, high thermal conductivity, low coefficient of thermal expansion and excellent corrosion resistance against liquid metals [2–7]. Mo–TZM is a potent Mo base alloy containing 0.5–0.8%Ti, 0.08–0.1%Zr, and 0.016–0.02%C (wt%). Due to solid solution strengthening and precipitation strengthening by complex carbides of titanium and zirconium, Mo–TZM possesses higher strength and creep resistance than unalloyed molybdenum at high temperatures [8–10]. The presence of carbides in the microstructure also increases the recrystallization temperature of Mo [8]. A large number of studies have been reported on the irradiation behavior of the alloy under different neutron energy, fluence, and irradiation temperatures [11–21]. Mo–TZM alloy shows void swelling and irradiation embrittlement behavior at temperatures below 800 °C. However, irradiation at temperatures greater than 800 °C results in little change in mechanical properties due to annihilation of point defects produced by neutron irradiation [4,21]. Thermally activated vacancy diffusion mechanism operates at these high temperatures causing reduction in defects and hence embrittlement. Mo–TZM, thus appears to be a suitable structural material for the nuclear reactors operated in the temperature range of 800–1000 °C.

Due to higher melting temperature, powder metallurgical techniques are preferred to produce Mo–TZM alloy [22]. The ductile–brittle transition temperature of the alloy decreases with progressive hot working of the sintered bars in the temperature range of 800–1400 °C [23,24]. Low strain rate sensitivity and occurrence of dynamic strain aging at the temperatures lower than 450 °C has been reported earlier [25,26]. Fracture toughness values and crack initiation and propagation behavior by monotonic and cyclic loading below and above DBTT have been identified [27,28,6,29]. Morito studied the tensile behavior of heat treated electron beam welded Mo–TZM and reported that the carbides precipitate along the grain boundaries and strengthen it, hence, suppressing intergranular embrittlement [30,31].

In the present investigation, hot compression tests were carried out on Mo–TZM alloy in its application temperature range of 600–900 °C at four different strain rates between 10^{-3} and 1 s^{-1}. Strain rate change tests were also conducted to compare with the tests at constant strain rate. The effect of deformation conditions on evolution of microstructure was studied by electron back scattered diffraction (EBSD) technique. Constitutive behavior $\sigma = f(\dot{\epsilon}, T, \epsilon)$ for assessment of deformation limits and deformation mechanisms responsible for evolution of microstructure were studied.

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2. Experimental procedure

Mo–TZM alloy was prepared by arc melting the electrode made by pressing and sintering. Table 1 represents the detailed chemical composition of arc-consolidated alloy. Cylindrical specimens of 5 mm diameter and 7 mm height were prepared from the alloy plate. Since the microstructure of the as-received materials was heterogeneous with highly elongated grains, the specimens were annealed at 1650 °C for 2 h under reducing atmosphere. Hot compression tests were conducted at constant true strain rate under argon atmosphere. The samples were heated to four different temperatures ranging from 600 to 900 °C and deformed up to the maximum strain of 0.6 at four different true strain rates of 0.001, 0.01, 0.1 and 1 s^{-1}. Constant true strain rate $\dot{\epsilon}$ for a sample of initial height h_o was achieved by varying the ram velocity v with time t as $v = h_o\dot{\epsilon}\exp(-\dot{\epsilon}t)$. In addition to these tests, strain rate change tests were also conducted by deforming the specimens incrementally by changing strain rates in steps from 1 to 0.001 s^{-1} and subsequently from 0.001 to 1 s^{-1}. At the starting strain rate of 1 s^{-1} the samples were deformed to true strain of 0.25, after which at each subsequent strain rate of 0.1, 0.01, 0.001, 0.01, 0.1 and 1 s^{-1}, the samples were deformed in steps of 0.05 true strain. After each hot compression tests, the specimens were quenched in water. A thermocouple was placed on to the sample and the temperature was controlled within ±2 °C. At each temperature a new sample was deformed. The deformed specimens were sectioned along the compression axis and prepared for metallographic examination using standard techniques that involve polishing of samples using different grades of emery papers (60–600 ASTM sizes) up to 1 μm diamond finish. The specimens were further electropolished using a bath consisting of 88% (by volume) Methanol and 12% H_2SO_4, applying a potential of 20 V for 30 s at 15 °C. Electron back scattered diffraction (EBSD) studies of the electropolished samples were carried out in an orientation imaging microscope (FEI make). Electron beam scanning speed was maintained at 0.5 μm. Grain size, grain orientation spread (GOS) and grain average misorientation (GAM) were calculated from the EBSD data. Transmission electron microscopic (TEM) studies were also carried out to characterize subgrain structure. Thin slices were also cut along the compression axis and 3 mm diameter TEM samples were prepared by optimizing jet thinning parameters as 30 V and −50 °C using the electrolyte consisting of 20 vol.% perchloric acid and methanol solution.

Table 1
Chemical composition of arc cast Mo–TZM alloy.

Material	Elements (ppm in wt%)							
	C	O	N	Ti	Zr	Fe	Ni	Si	Mo
Mo–TZM	220	20	10	5000	1100	<10	<10	<10	Rest

3. Results and discussion

3.1. Stress–strain behavior

The true stress σ vs. plastic true strain ε for this TZM alloy at different strain rates and temperatures are shown in Fig. 1. From the

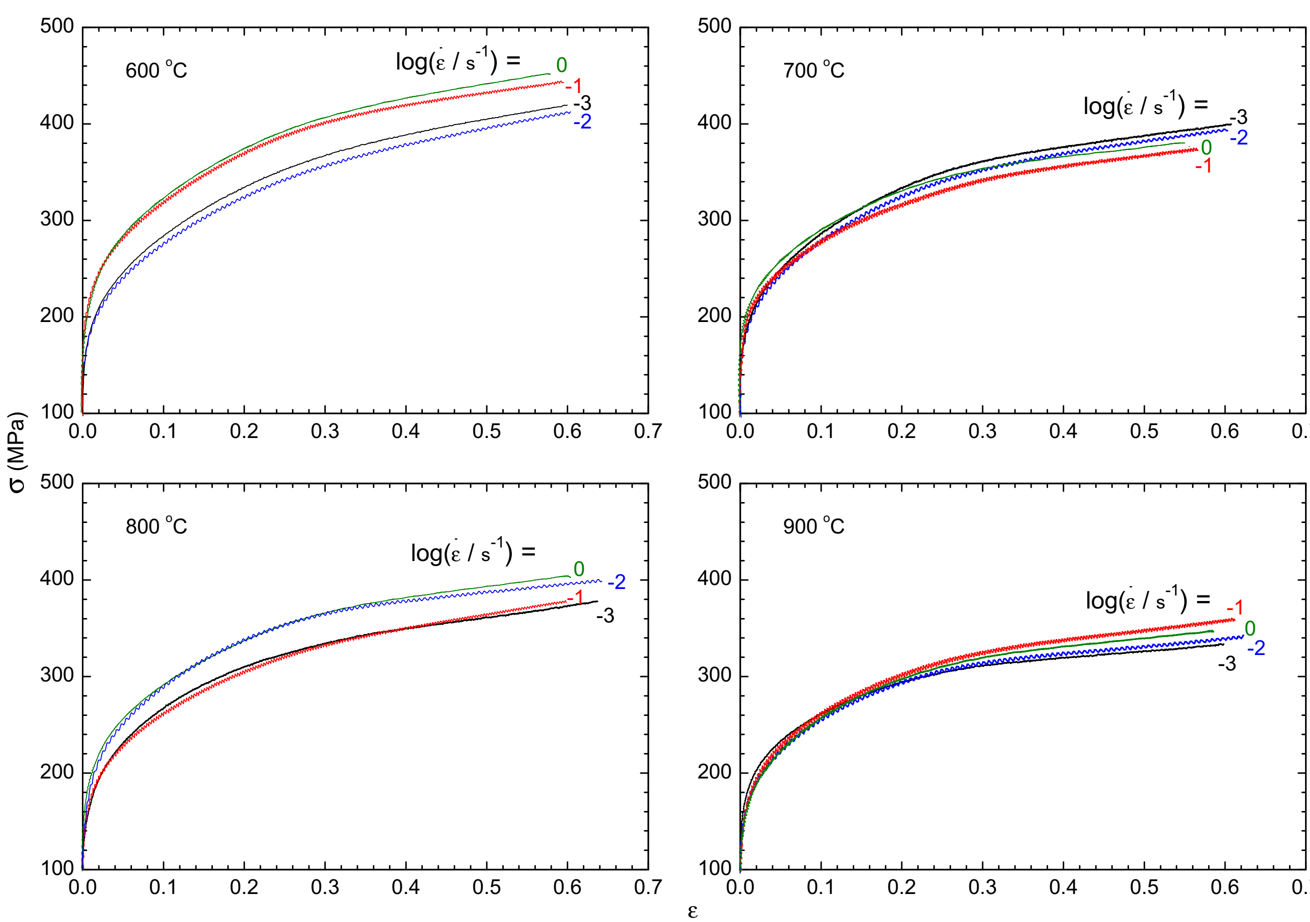

Fig. 1. True stress–true strain plots obtained from the compression tests conducted at 600, 700, 800 and 900 °C applying four strain rates 0.001, 0.01, 0.1 and 1 s^{-1}.