Texture evolution during hot deformation of Moly-TZM

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Abstract. Moly-TZM was deformed at constant strain rate of 1.0 s⁻¹ to investigate the high strain rate deformation behaviour by microstructural and stress response change within a temperature range of 1400-1700 °C. To correlate the deformation behaviour with orientational change, recrystallization and recovery of the material, the microstructural investigation was undertaken using scanning electron microscopy (SEM) of electron back scattered diffraction (EBSD). Depending on the grain size and orientation spread recrystallized grains were identified and texture was calculated. Change in grain boundary characteristics with increasing temperature was determined by the misorientation angle distribution for the deformed and recrystallized grains. Subgrain coalescence and increase in subgrain size with increasing temperature was observed, indicating recrystallization not only occurred from the nucleation of the dislocation free grains in grain boundaries but also from the subgrain rotation and merging of the subgrains by annihilation of the low angle grain boundaries. Detailed studies on the evolution of texture of recrystallized grains showed continuous increase in <001> fiber texture in recrystallised grains, in contrast to a mixed fiber <001> +<111> for the deformed grains.

1. Introduction

Moly-TZM is well known for its superior strength and high temperature creep resistance [1-3]. Due to its low irradiation effect at high temperature range (≥800°C), Mo-TZM is a potential material for its structural application in nuclear reactors [4]. The overall composition of the alloy in wt% is 0.5-0.8%Ti, 0.08-0.1%Zr, and 0.016-0.02%C, and balance Mo. The superior strength of this alloy is attributed to the precipitation hardening from the formation of carbide precipitates of titanium (Ti) and zirconium (Zr). However in an earlier study it was revealed that the strength does not come from the pinning of the dislocation, rather the strengthening occurs by the pinning of dislocation ensembles which results in formation of substructure [1,5]. This substructure (cells/subgrains) works as dislocation barriers and hence rendering a high yield strength of Mo-TZM.

Previous studies showed that the Mo-TZM has a low strain rate sensitivity upto 900°C [3,6]. It has been predicted that at 900°C, when the material is deformed at high strain rate, some localised recrystallisation occurs [2]. A similar study of the deformation behaviour and microstructural evolution in the temperature of the order of ≈0.5Tm of Mo-TZM is lacking. In the present investigation, Mo-TZM alloy has been deformed in the temperature range of 1400-1700 °C at a constant strain rate 1.0 s⁻¹. The evolution of deformation microstructure has been investigated and analysed to elucidate the mechanism of texture formation in this material.

2. Experimental details

Cylindrical samples of dimension of 15 mm height and 10 mm diameter were prepared using EDM machining. The specimens were then annealed at 1350°C for 6 hours in vacuum (~10⁻⁵ millibar pressure) to obtain equiaxed microstructure. However the microstructure is not fully recovered after annealing, as deformed grains are observable in figure 1. Compression testing of the specimens was done at temperature range of 1400 to 1700°C with 100°C temperature interval at a constant strain rate of 1.0 s⁻¹. The samples were deformed up to 0.7 true strains. To retain the deformed microstructure the specimens were first cooled with high speed argon jet. The cooling rate of specimens after deformation with argon jet is typically 60-70 °C s⁻¹. The control of sample temperature was through a thermocouple is spot welded to the sample at the centre. The temperature was controlled to within ±1°C of the deformation temperature (1400, 1500, 1600 and 1700°C). Resistance heating was used with heating rate of 5°C s⁻¹. Before deformation specimens were kept at experimental temperature for...
5 min for uniform heating. Samples were sectioned along the compression axes for metallographic characterisation. After conventional metallographic polishing, the specimens were further polished using 1µm and 0.3µm alumina slurry for mirror finishing. A solution consisting of nitric acid, hydrofluoric acid and distilled water in the ratio 2:2:5 was used for chemical etching in order to reveal optical and SEM structures. For orientation imaging, the samples were electropolished using a solution of 5% perchloric acid in methanol. Optical microstructures were taken using Carl-Zeiss microscope, while orientation imaging was done on a FEI-250 scanning electron microscope attached with TSL-OIM EBSD camera. Texture was calculated from the EBSD scans.

3. Results and discussion

Fig. 2 displays the EBSD generated microstructures represented by Inverse Pole Figure (IPF) map for the deformed materials. From the change in orientation of grains it is anticipated that apart from dynamic recovery of the grains during deformation some localised recrystallisation had occurred. In IPF image displayed in Fig 2(a-d), recovered microstructure with small dynamically recrystallised grains surrounding the deformed grains are clearly seen (subset of Figs. 2a and b). In higher temperature deformed materials, that is, the ones deformed at 1600 and 1700°C, occurrence of grain growth is very prominent, which has presumably occurred during holding the samples at deformation temperature before deformation for the uniformity of temperature throughout the specimens.

The grain sizes of the deformed and recrystallised portions of the microstructure are plotted in Fig 3(a) as a function of temperature. The recrystallised grains have been identified by the grain orientation spread (GOS) criterion. The grains with GOS<2°, have been identified as recrystallized grains. The plot indicates that the effect of temperature on the size of the recrystallised grains is minimal as the change in size of the dynamically recrystallised grains is very small. However, the size of the deformed grains increases with increasing temperature. Grain boundary character distribution with high angle boundaries (>15°), low angle boundaries (<15°) and coincide site lattice (CSL) boundaries are plotted in Fig. 2(e) after normalisation as a function of temperature. The high angle grain boundary fraction decreases for 1600 and 1700°C. The low angle grain boundary fractions in the 1600 and 1700°C deformed samples were higher than 1400 and 1500°C, possibly due to the higher fraction of subgrains within the deformed grains. The CSL boundary fraction is high for 1400 and 1500 °C. It is clear from Fig. 2 that within the temperature range of 1500- 1600 °C there is a transition in the restoration mechanism during the deformation. To observe the effect of dynamic recrystallisation on deformation, number fraction of the recrystallised grains obtained after partitioning (using grain orientation spread) are plotted in Fig. 3(b) as a function of temperature. The fraction of recrystallised grains decreases with increasing temperature.

From Fig. 3(a), it is observed that the fraction of the recrystallised grains is almost constant with temperature. However, prior grain growth is indicated for the deformed grains. Evidently at higher temperatures, grain growth is predominant in the as-received material, whereas at the lower temperatures 1400 and 1500 °C, dynamic recrystallisation is the dominating process. Possibly for the higher temperature of 1600 °C and 1700 °C the recrystallised grains were contributing to the average size growth of the pre-existing grains which were later identified as primarily deformed grains.
Fig. 2 (a-d): IPF maps of the Moly-TZM samples deformed at strain rate $1.0 \text{s}^{-1}$ at different temperatures: (a) 1400 °C (b) 1500 °C (c) 1600 °C (d) 1700 °C. In the subset of each micrograph, at the right bottom corner, the texture is represented through inverse pole figures. Fig. 2(e): Grain boundary characteristics distribution (GBCD) for all the samples showing distinctive change in the grain boundary distribution between 1400°C, 1500°C and 1600°C, 1700°C. For Figs. (a) and (b), the magnified view of selected regions after partitioning the dynamically recrystallised grains using GOS<2° are also given at the top right corner.

Since the tests were done in the uniaxial loading condition, the textures have been depicted by inverse pole figures (IPF). The IPF are presented in the subset of Fig. 2(a-d). IPFs have been calculated on the compression plane. In all the cases, the pole density at <001> location is higher than at <111> location. Further, the pole density at <001> location is relatively higher for the samples deformed at 1400 and 1500°C. The change in ratio of pole density at <001> and <111> locations with temperature is plotted in Fig. 3(c). The ratio $<001>/<111>$ decreases with increasing temperature, due to increase in pole density at <111>. The increase in pole density at <111> suggests the occurrence of pencil glide, where <111> is the principle direction of slip consisting of number of planes. The IPFs exclusively calculated from the recrystallised grains (partitioned using the criterion GOS<2°) are shown in Fig.4. It can be seen that most of the recrystallised grains have <001> axis parallel to the compression axis. Further, from the values of pole density, it can be observed that the texture weakens with increase in the deformation temperature.
Fig. 3(a): Grain size distribution for deformed and recrystallised grains showing the effect of temperature; (b): Variation of number fraction of the recrystallised grains with temperature; and (c) change in the intensity strength ratio of <100> and <111> component with increasing temperature.

![Graphs showing grain size distribution and intensity strength ratio](image)

**(a) 1400°C SR 1.0s⁻¹**

max = 9.507

**(b) 1500°C SR 1.0s⁻¹**

max = 7.841

**(c) 1600°C SR 1.0s⁻¹**

max = 6.395

**(d) 1700°C SR 1.0s⁻¹**

max = 5.925

Fig. 4 Inverse pole figures calculated from the recrystallised part of the samples deformed at strain rate of 1.0 s⁻¹ at temperatures: (a) 1400 °C, (b) 1500 °C, (c) 1600 °C, and (d) 1700 °C.

### 4. Conclusions

It is possible to carry out deformation Molybdenum–TZM alloy in the temperature range 1400-1700°C at the strain rate 1 s⁻¹ without developing any instability. Microstructural analyses reveal that the dynamic restoration mechanisms (dynamic recrystallisation and recovery) are most prominent for the temperatures 1400 and 1500 °C, whereas for the temperatures 1600 and 1700°C grain growth controls the dissipation of stored energy. Texture analyses indicate that deformation mostly occurs by the activation of pencil glide with <111> direction as principle slip direction.

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