High temperature deformation behavior of the molybdenum alloy TZM

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Abstract. The molybdenum alloy TZM (Mo-0.5wt%Ti-0.08wt%Zr) is a commonly used constructional material for high-temperature applications. It is well known that molybdenum and its alloys develop a distinct subgrain structure and texture during hot deformation. These microstructural aspects have a significant effect on strength at elevated temperatures. It was observed that with proceeding primary recrystallization and therefore with disappearance of subgrains the yield strength drops almost to the level of pure molybdenum. The aim of the present work was to investigate and describe the strain hardening of hot deformed TZM on a microstructural basis. For this purpose sintered and prerolled TZM rods were recrystallized and each of them deformed to a specific degree of deformation afterwards. Especially the evolution of disorientation distributions was analyzed by electron backscattering diffraction (EBSD) and used to describe the work hardening effect. The yield strength was determined by tensile tests between room temperature and 1473 K. By analyzing disorientation profiles the formation and evolution of geometrically necessary and incidental dislocation boundaries could be observed. A model developed by Pantleon was used to describe the work hardening of TZM.

1. Introduction
The molybdenum alloy TZM contains 0.5 wt.% Ti, 0.08 wt.% Zr and approximately 200 ppm C. It is commonly used as structural material in thermally loaded applications or as plate material in rotary X-ray tubes. Compared to pure molybdenum TZM exhibits a higher strength and creep resistance. Especially at elevated temperatures the primary strengthening mechanism was attributed to carbide particles. An earlier study [1] revealed that these carbides do not increase the strength by an Orowan-like dislocation pinning mechanism but are able to pin dislocation ensembles like cell walls or subgrain boundaries. Furthermore the particles help to increase the recrystallization temperature. Dislocation arrangements like cell walls or subgrain boundaries are able to act as glide barriers even at high temperatures and hence increase the yield strength of the material until recrystallization occurs.

A strong work hardening effect in TZM was reported earlier [2]. The tendency of pure molybdenum to form dislocation networks during hot deformation is well known. Results for Mo
single and polycrystals were reported e.g. in [3]. The aim of this work is to provide a better microstructure-based understanding of the work hardening behaviour of TZM.

Work hardening is commonly expressed by plotting the work hardening rate versus the flow stress [4-6]. Normally a so called stage-III behaviour is observable. Depending on the strain path the stages IV and V may be present too [6]. For stage III there is the common understanding that hardening is caused by generation and storage of dislocations resulting in the formation of dislocation walls. Kocks developed a model for stage III which needs just one microstructural parameter, namely the overall dislocation density [6]. Unfortunately stage IV cannot be described by this model. Pantleon however picked up this approach incorporating the existence of excess dislocations [7]. With this modification the model is able to explain the formation and evolution of dislocation boundaries and describe work hardening up to stage IV. This approach was used to model the work hardening of TZM. For a detailed description of the model the interested reader is referred to [7,8,9].

The flow stress of molybdenum and its alloys below the transition temperature $T_0$ is governed by a high Peierls-Nabarro-potential [10,11]. This thermal part of flow stress has to be separated from the athermal contributions like work hardening. The description of the used procedure would exceed the scope of this paper and is given elsewhere [12].

2. Experimental

The investigated material was produced via a standard PM route. After sintering the alloying contents were 0.49 wt% Ti, 0.071 wt% Zr, 180 ppm carbon and 150 ppm oxygen. Afterwards, the sintered material was forged to a (logarithmic) strain of $\varphi = 0.5$ and completely recrystallized at 1900 K. This condition defines the non-deformed state and acts as a base line for the following experiments. It was named initial state (IS). Samples with increasing degree of deformation $\varphi$ were produced from the IS material by hot rolling at 1470 K. Finally, samples for tensile tests and orientation mappings were machined from all states. All tensile tests were done at a constant strain rate of $2 \cdot 10^{-3} \, s^{-1}$ between room temperature and 1473 K.

In order to determine the microstructural parameters which characterize the hardening behavior the samples were investigated by EBSD. To achieve a sufficient success rate (fraction of indexed Kikuchi Maps) of more than 85% of the measured points, the specimens were electro polished. The measurements were done on a LEO Gemini 1530 SEM with an HKL Nordlys EBSD detector. Due to the small cross section of the acquisition area each sample was measured at three different points to ensure the representativeness of the measurement. The texture effect was determined by calculating the inverse pole figures and Taylor factors in tensile direction. The disorientations were calculated from the orientation mappings.

3. Results and Discussion

The samples were characterized by SEM/EDX to verify the precipitation of particles. Carbide and oxidic particles were found. The carbide particles are mostly located at grain boundaries and appeared to be molybdenum carbides. Fine precipitated particles with potential to pin dislocations and cause a significant particle hardening effect were not found. Titanium is mostly soluted in the molybdenum matrix. Its hardening contribution is minor compared to the observed effects and will be neglected [13]. During the first stages of deformation the original grains were elongated and start to disorient. This may indicate the formation of dislocation boundaries (figure 1b). With increasing degree of deformation subgrains are formed within the original grains. According to literature [e.g. 14] the subgrain boundaries are of different type called incidental dislocation boundaries (IDB) and geometrically necessary boundaries (GNB) [14,15].
Figure 1. SEM micrographs of (a) IS and deformed states with (b) $\varphi=0.5$ and (c) $\varphi=1.5$, molybdenum carbides located at grain boundaries can be observed (b)

For a quantitative description of the substructure development the disorientation distributions of the different stages were calculated from the EBSD results. Even in the early stages of deformation the distributions reveal two maxima. A superposition of two lognormal distributions was fitted to the measured values. From the parameters of each function $i$ the global maximum $\theta_{i\text{max}}$ was calculated. The development of the values for $\theta_{i\text{max}}$ is shown in figure 2. One maximum is in the range of two degrees, the other develops depending on the degree of deformation between 3° to 8° (figure 2). It is assumed that the first peak represents the IDBs whereas the broad second peak represents the GNBs.

For modeling the disorientation evolution Pantleon assumes that a fluctuation in statistical trapping of dislocations and an imbalance in the activation of slip systems on both sides of a boundary leads to a bias of dislocation fluxes. The imbalance of slip activation is Gaussian distributed with a standard deviation $\sigma_{imb}$ [7,9]. The evolution of disorientations is described by:

$$\left\langle \theta^2 \right\rangle = \frac{Pb}{d^2} \gamma + \sigma_{imb}^2 \gamma^2$$

(1)

For each stage of deformation the shear strain $\gamma$ was calculated from $\varphi$ using the related Taylor factor which was determined from EBSD orientation measurements [2]. The probability $P$ of dislocation immobilization in a boundary is set equal to 1. Values for $d^*$ and $\sigma_{imb}$ were determined by fitting the right side of equation (1) to the square of $\theta_{i\text{max}}$.

Figure 2. Development of $\theta_{i\text{max}}$ with $\gamma$ indicating the evolution of IDBs and GNBs during rolling at 1473 K

Figure 3. Yield strength of TZM at different temperatures and $\varphi$, indicated values for 293 K are reduced by the thermal stress component [12]
From the tensile tests the yield strengths of the hot worked TZM at different degrees of deformation $\varphi$ and temperatures were obtained. The $\sigma_y - \varphi$ plots reveal a strong work hardening effect (figure 3). Certainly the thermal stress contribution below a transition temperature $T_0$ must not be neglected. The transition temperature $T_0$ was determined to be 233°C (506K) at a strain rate of $2 \times 10^{-3}$ s$^{-1}$ [12]. For this reason the stress values for $T < T_0$ were reduced by the thermal stress component (see figure 3) which was determined for this material as described in [12]. Using the model proposed by Pantleon for stages III and IV which is based on the development of GNBs and IDBs, the athermal shear stress $\tau_{\text{atherm}}$ at yielding as function of $\gamma$ can be described in good agreement with the experimental values (figure 4). Based on these findings and incorporating texture effects, the temperature dependence of the shear modulus [13] as well as the thermal stress part, the development of the yield strength $\sigma_y$ can be predicted for different temperatures and $\varphi$ (figure 5). It can be concluded that even at temperatures up to 1300 K work hardening strongly enhances the strength of TZM. The observed strength increase is in the range of 500 to 600 MPa. The effect is mainly carried by geometrically necessary and incidental dislocation boundaries and can be described by the model proposed by Pantleon in good agreement with experimental data.

References
[1] Mrotzek T, Hoffmann A and Martin U 2006 Int. J. Refract. Met. H. 24 298
[2] Mrotzek T, Hoffmann A, Martin U and Oettel H 2007 Mater. Sci. Forum 539-543 2725
[3] Luft A 1995 Prog. Mater. Sci. 35 69
[4] Nabarro F R N, Basinski Z S and Holt D B 1964 Adv. Physics 13 193
[5] Gil Sevillano J, van Houtte P and Aernoudt E 1981 Prog. Mater. Sci. 25 69
[6] Kocks U F and Mecking H 2003 Prog. Mater. Sci. 48 171
[7] Pantleon W 2004 Mater. Sci. Eng. A 387-389 257
[8] Holt D L 1970 J. Appl. Phys. 41 3197
[9] Pantleon W 2005 Mater. Sci. Eng. A 400-401 118
[10] Peierls R 1940 Proc. Phys. Soc. 52 34
[11] Nabarro F R N 1947 Proc. Phys. Soc. 59 256
[12] Schimpf C, Mrotzek T and Martin U 2009 Proc. 17th Int. Plansee Sem. (Reutte) vol 2
[13] Wesemann I, Hoffmann A, Mrotzek T and Martin U 2009 Proc.17th Int. Plansee Sem.(Reutte)
[14] Hansen N 2005 Adv. Eng. Mater. 7 815
[15] Kuhlmann-Wilsdorf D and Comins N R 1983 Mater. Sci. Eng. 60 7