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Texture and microstructure of high purity tantalum

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ABSTRACT

The texture and microstructure of rolled and annealed pure Ta was investigated by means of quantitative texture analysis and metallography. The rolling textures were discussed using simulations according to Relaxed Constraints Taylor Theory. The annealed samples revealed recovery and continuous recrystallization for weak rolling degrees and low annealing temperatures and discontinuous recrystallization for high deformations and high annealing temperatures.

INTRODUCTION

The investigation of Ta is of interest mainly for two reasons. First Ta is suitable for the examination of basic aspects of deformation, recovery and recrystallization of bcc metals over a wide temperature range, since it does not undergo phase transformation during annealing. Second Ta is of industrial importance due to its high melting point, low vapour pressure, good thermal and electrical conductivity and biocompatibility. Texture analysis and metallography represent a sensitive tool for the investigation of rolled and annealed Ta, since the corresponding physical mechanisms, i.e. dislocation glide and recrystallization, lead to characteristic changes of the orientation distribution of the crystallites.

EXPERIMENTAL PROCEDURE

Cold rolling of the initial Ta sheet was carried out on a laboratory rolling mill. Samples were prepared after a deformation of $\epsilon = 70\%$, 80% , 90% and 95% . The subsequent heat treatment was executed under vacuum at 1000°C , 1100°C , 1200°C and 1300°C , respectively. Optical microscopy of longitudinal sections was carried out after etching in a solution of 100 ml H_2O , 80 ml HF and 50 ml HNO_3 for 500s. The textures were examined by measuring four incomplete pole figures in the center layer of the samples. Subsequently the orientation distribution function (ODF) was calculated (1).

EXPERIMENTAL RESULTS

The texture of the 70% rolled Ta reveals a high intensity between $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$ (α -fibre) (Fig.1). After annealing at 1000°C , $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$ are sharpened whereas $\{112\}\langle 110 \rangle$ is weakened. After annealing at 1100°C the same tendency is revealed. After heat treatment at 1200°C and 1300°C the intensity of the α -fibre is reduced and the texture exhibits a

maximum at $\{111\}\langle 112 \rangle$. The texture after 80% rolling corresponds to that of the 70% rolled specimen. The heat treatment leads to similar texture changes as for the 70% rolled sample (Fig.2). After annealing at 1000°C the α -fibre is sharpened at $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$. The $\{112\}\langle 110 \rangle$ component is decreased. After 90% and 95% the α -fibre has become sharper than for lower rolling degrees. Here the annealing treatment leads to a texture change. The orientations on the α -fibre are vanished and $\{111\}\langle 112 \rangle$ is enhanced (Fig.2). The longitudinal micrographs of the rolled samples expose a flat grain morphology (Figs.3a,4a). After annealing at 1000°C , in some regions new grains are formed, whereas adjacent zones exhibit residual deformation structure. The new grains typically have an elongated instead of an equiaxed shape.

DISCUSSION

The rolling texture is discussed in terms of "Relaxed Constraints Taylor Theory" (2). Two specific assumptions were made. First, the ϵ_{13} and the ϵ_{23} tensor components were both relaxed since the "pancake" like shape of the grains admits shears in rolling- and in transverse direction. Second, dislocation glide on $\{110\}$ planes (case A), on $\{110\}$ plus $\{112\}$ planes (case B) and on $\{110\}$, $\{112\}$ plus $\{123\}$ planes (case C) was considered. Simulation A (Fig.5a) leads to a texture with $\{112\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$. The texture of the rolled Ta is not adequately simulated (Fig.1). Model B (Fig.5b) yields two maxima at $\{001\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$. Although the simulated texture has changed to a more fibre like shape, the contour of the measured α -fibre is not convincingly reproduced. Simulation C (Fig.5c) shows a uniformly shaped α -fibre, i.e. a good accordance with the measurements. It follows, that dislocation glide on $\{110\}$, $\{112\}$ and $\{123\}$ planes has taken place during rolling.

The annealing of rolled Ta basically generated two kinds of textures. First, the enhancement of $\{001\}\langle 110 \rangle$ accompanied by the decrease of $\{112\}\langle 110 \rangle$ for moderate deformations and temperatures (Fig.2). Second, the decrease of the α -fibre accompanied by the sharp increase of the $\{111\}\langle uvw \rangle$ -fibre (γ -fibre) for higher deformations and temperatures.

The $\{001\}\langle 110 \rangle$ orientation reveals a low Taylor factor, i.e. low dislocation density. Also line broadening experiments have shown that the dislocation density in $\{001\}\langle uvw \rangle$ is smaller than in $\{112\}\langle uvw \rangle$ or $\{111\}\langle uvw \rangle$ (3). The average cell size was found to yield a maximum in grains with a $\{001\}\langle 110 \rangle$ orientation (4). It is thus followed that the $\{001\}\langle 110 \rangle$ oriented grains reveal a low dislocation density and hence a weak tendency to form nuclei during primary recrystallization (RX). This reluctance of $\{001\}\langle 110 \rangle$ against RX was also observed in pure iron by applying single orientation measurements (5).

The increase of $\{001\}\langle 110 \rangle$ during annealing can thus not be explained by RX. On the other hand it can also not be understood in terms of recovery. First the increase of $\{001\}\langle 110 \rangle$ is stronger than it would be caused by the decrease of diffuse scattering. Second the microstructure, especially of the 70% rolled and annealed samples, reveals a partial or even complete reformation of the grain morphology. Since no basic texture transition has taken place, i.e. $\{001\}\langle 110 \rangle$ has only increased during annealing and since new grains have been formed without preceding nucleation and thus without movement of large angle grain boundaries the observed texture is attributed to continuous recrystallization.

The second type of annealing texture reveals a strong $\{111\}$ -fibre, i.e. $\{111\}\langle 110 \rangle$ and $\{111\}\langle 112 \rangle$. The increase of $\{111\}\langle 112 \rangle$ during annealing corresponds to the decrease of $\{112\}\langle 110 \rangle$ (Figs.1,2). The $\approx 35^\circ \langle 110 \rangle$ rotation relationship between both orientations is close to $27^\circ \langle 110 \rangle$, i.e. to the $\Sigma 19a$ coincidence boundary. Grain boundaries with this rotation relationship are known to reveal high growth rates (6). It is thus assumed that growth selection plays the leading role in the development of the $\{111\}\langle 112 \rangle$ RX component. This is also valid for the $\{111\}\langle 110 \rangle$ RX orientation, which has a good growth relationship to the $\{113\}\langle 110 \rangle$ rolling component. On the other hand it reveals a very fine cell

structure (4) and a high Taylor factor, exceeding even that of $\{111\}\langle 112 \rangle$. This indicates a high nucleation tendency. It is thus assumed that the occurrence of a strong $\{111\}\langle 110 \rangle$ RX component results from both a high nucleation rate and growth selection.

CONCLUSIONS

1.) Rolling textures of Ta can be interpreted by means of Relaxed Constraints Taylor Theory. The simulations with relaxation of ϵ_{13} and ϵ_{23} on the one hand and dislocation glide on $\{110\}$, $\{112\}$ and $\{123\}$ planes on the other hand, yield good correspondence with the measured rolling textures.

2.) Samples with rolling degrees up to 80% which are annealed up to 1200°C reveal an inhomogeneous development of the texture and microstructure. Whereas the rolling component $\{112\}\langle 110 \rangle$ is consumed by discontinuous RX, grains with a $\{001\}\langle 110 \rangle$ orientation are rearranged by continuous RX. In samples with stronger deformation and / or higher annealing temperature a strong $\{111\}$ fibre texture is formed by discontinuous RX.

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FIGURES

Figure 1
Texture after various
heat treatments of 70%
rolled samples.

(a) α -fibre, (b) γ -fibre.

Figure 2
Annealing textures
for various rolling
degrees,
1 hour at 1000°C.
(a) α -fibre, (b) γ -fibre.

Figure 3
Longitudinal
micrographs,
70% rolled.
(a) 70% rolled,
(b) 70% rolled +
1 hour at
1000°C.

Figure 4
Longitudinal
micrographs,
90% rolled.
(a) 70% rolled,
(b) 70% rolled +
1 hour at
1000°C.

Figure 5 Simulations according to Taylor Relaxed Constraints Theory.