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Microstructure and thermal stability in CP titanium processed by electroplastic rolling

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Abstract. Ultrafine-grained (UFG) CP titanium (Grade-4) sample was processed by electroplastic rolling (EPR) at room temperature which was compared to a specimen processed by conventional cold rolling (CR). EPR was performed using pulsed unidirectional current with a current density of 95 A/mm^2, pulse duration of 10^{-4} s and frequency of 1000 Hz. It was found that the sample processed by EPR has slightly higher dislocation density and smaller crystallite size than for the CR specimen resulting in a higher tensile strength for the former specimen. In the case of EPR sample, the relative fraction of <c+a> dislocations is lower than for CR specimen. During annealing the relative fraction of <c+a> dislocations decreased for both samples which can be explained by the fact that the <c+a> dislocations have larger Burgers-vector and consequently higher formation energy than the other two types.

Introduction

Bulk ultrafine-grained (UFG) materials are currently a major focus in materials science due to their unique properties by comparison with their coarse-grained counterparts [1]. Porosity- and contamination-free bulk UFG metals and alloys can be produced by severe plastic deformation (SPD) [1]. Recently, novel SPD methods were elaborated where the efficiency of plastic deformation in grain refinement was increased by a simultaneous application of pulsed electric current [2]. These procedures are called as electroplastic deformation methods. The major effect of the current pulses was to reduce the flow stress [3]. The plasticity of materials had been significantly improved concurrently [4]. These effects are most probably caused by the enhanced migration of vacancies as well as the easier cross-slip and climb of dislocations due to current pulses [3]. In this work, the microstructure and the thermal stability of commercially pure (CP) titanium processed by electroplastic rolling are studied in comparison with a conventionally cold rolled sample.

Experimental materials and procedures

Commercially pure (Grade-4) UFG titanium sample was processed by electroplastic rolling (EPR) at room temperature which was compared to a specimen processed by conventional cold rolling (CR). CR and EPR were carried out on strips with coarse-grained structure reducing the initial thickness of 2.35 mm to final thicknesses of 0.32 and 0.24 mm which correspond to the true strains of 2.0 and 2.3 for CR and EPR, respectively. EPR was performed using pulsed unidirectional current with a current density of 95 A/mm^2, pulse duration of 10^{-4} s and frequency of 1000 Hz. After each pass the sample was cooled in water to avoid a significant heat effect.
The microstructures of Ti samples were investigated by X-ray line profile analysis. The X-ray line profiles were measured by a special high-resolution diffractometer (Nonius FR591) using CuKα_1 radiation (λ = 0.15406 nm). The line profiles were evaluated by the extended Convolutional

Multiple Whole Profile (CMWP) fitting procedure described elsewhere [5]. The CMWP procedure gives seven fitting parameters for hexagonal crystals: the median and the variance, m and σ, of the log-normal size distribution function; the density and the arrangement parameter of dislocations, ρ and M; the q_1 and q_2 parameters in the contrast factors of dislocations and the twin frequency, β. The latter quantity is defined as the relative fraction of twin boundaries among {111} lattice planes. At ambient temperature, twinning occurs mainly on {102}, {111} and {112} planes [6], therefore in the line profile analysis we assume the existence of these twin families with equal weights. The area-weighted mean crystallite size, $\langle x \rangle_{area}$, was calculated from m and σ using the formula given in reference [7]. The q_1 and q_2 parameters of the contrast factors depend on the character of dislocations and therefore enable the determination of the fractions of <a>, <c> and <c+a> type dislocations in the specimen. The detailed description of this procedure is given in reference [8]. The thermal stability of Ti samples processed by CR and EPD was investigated by differential scanning calorimetry (DSC) at a heating rate of 40 K/min using a Perkin-Elmer DSC2 calorimeter.

Results and discussion

The characteristic parameters of the microstructure obtained by X-ray line profile analysis are listed in Table 1. The sample processed by EPR has slightly higher dislocation density and smaller crystallite size than for CR specimen which can be explained by the larger strain for EPR sample. Table 1 suggests the abundance of <a> dislocations for EPR sample which can be explained by their smallest Burgers-vectors compared to the other two types of dislocations. At the same time, in the case of CR sample the relative fraction of <c+a> dislocations is comparable with that of <a> dislocations. This difference between the composition of the dislocation structure can be explained as follows. During plastic deformation of Ti, the plastic strain in the hexagonal <c> direction is usually resulted by twinning or <c+a> dislocations. At room temperature the critical resolved shear stress for <c+a> dislocations in pyramidal slip planes is about five times larger than that for <a> type dislocations in basal planes [9]. Therefore <c+a> type dislocations are activated at locations where the stresses are very high. At the same time, it was shown in previous papers [3,4,10] that current pulses reduced the flow stress during plastic deformation. Most probably, the reduction of the flow stress is accompanied by the decrease of the internal stresses developed during electroplastic straining therefore the probability of activation and multiplication of <c+a> type dislocations are lower during EPR process than in the case of CR procedure. This may explain the smaller fraction of <c+a> dislocations and the abundance of <a> dislocations after EPR. In the case of EPR, the strain in the hexagonal <c> direction may be partially produced by the slide of fine grain boundaries beside the activity of a smaller fraction of <c+a> dislocations [10]. The grain boundary sliding is facilitated by the enhanced diffusivity of vacancies due to electric current [3]. As the resistivity of grain boundaries is higher than the grain interiors, the electric pulses may cause a heating of the grain boundary regions thereby causing faster diffusion along grain boundaries.

Table 1: Parameters of the microstructure obtained by X-ray line profile analysis for the samples processed by CR and EPR, as well as for the specimens annealed in a DSC scan up to 809 K. $\langle x \rangle_{area}$ is the area-weighted mean crystallite size, ρ is the dislocation density, YS is the yield strength and UTS is the ultimate tensile strength.

sample	$\langle x \rangle_{area}$ [nm]	ρ [$10^{14}m^{-2}$]	YS [MPa]	UTS [MPa]	<a> [%]	<c> [%]	<c+a> [%]
CR	40±5	15±2	920	992	48±6	8±4	44±6
CR, 809K	71±9	5±1	-	-	84±10	8±4	8±4
EPR	29±4	21±2	950	1015	74±10	10±8	16±4
EPR, 809 K	87±9	9±1	-	-	80±10	8±4	12±4

The twin frequency is practically zero for both samples. Table 1 shows that both the yield strength (YS) and the ultimate tensile strength are slightly larger for EPR-processed sample than for CR specimen which can be explained by the higher dislocation density and the smaller crystallite size.
Figure 1 shows the DSC thermograms obtained for CR and EPR samples at a heating rate of 40 K/min. An exothermic peak appears for both CR and EPR specimens between 800 and 900 K which is most probably related to the recovery and recrystallization of the severely deformed microstructures. The parameters of the microstructure were determined at the beginning of the DSC peak by quenching the samples when their temperature reaches 809 K. These parameters are also listed in Table 1. It can be seen that the dislocation density decreased by a factor of 2-3 compared to the as-received state for both samples. The reduction of the dislocation density before the exothermic DSC peak observed for Ti differs from the behavior of severely deformed Cu [11]. It has been shown that in the latter case, the dislocation density and the crystallite size remained unchanged till the beginning of the exothermic DSC peak, indicating that the recovery and recrystallization starts only at the temperature corresponding to the beginning of the DSC peak. At the same time, for the present Ti samples, the decrease of the dislocation density shows that the recovery starts before the observed DSC peak. Most probably, the recovery spreads in a wide temperature range before the peak, therefore the released heat cannot be recognized below 800 K. The reduction of the dislocation density is accompanied by the increase of the crystallite size and the decrease of the dislocation arrangement parameter, *M* (not shown here). The latter observation indicates that the remaining dislocations in the recovered microstructure are arranged into a more stable dipole configuration where the strain field of dislocations is screened.

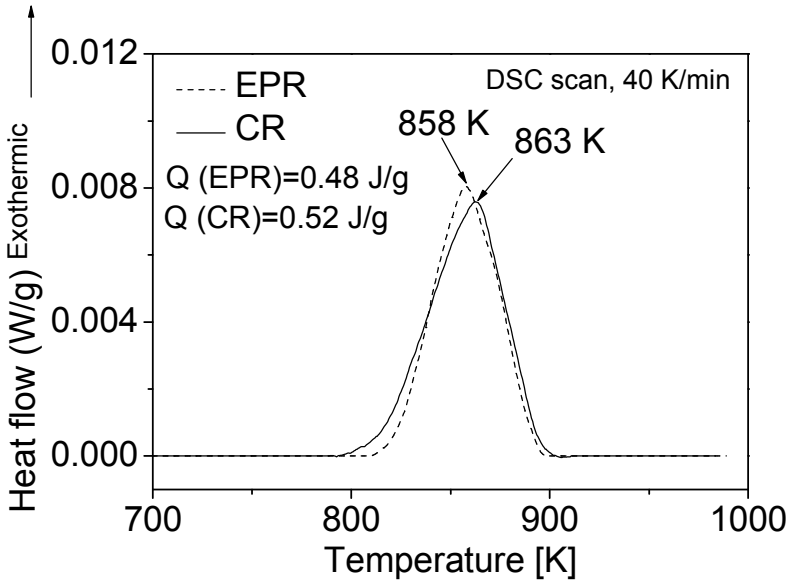

Figure 1: DSC thermograms taken at a heating rate of 40 K/min for Ti samples processed by cold rolling (CR) and electroplastic rolling (EPR). The heat released in the exothermic peak (Q) is also shown in the figure.

Table 1 shows that as a result of annealing before the beginning of the DSC peak, the relative fraction of the <c+a>-type dislocations decreased especially for CR sample indicating that these dislocations disappeared faster than the <a> or <c>-type ones. This can be explained by the fact that the <c+a> dislocations have larger Burgers vector and consequently higher formation energy than the other two types thereby increasing the driving force for annihilation. The relative fraction of the dislocation density annihilated before the DSC peak is larger for CR sample most probably due to the higher fraction of <c+a>-type dislocations compared to EPR specimen. This may be the reason of the earlier beginning of the DSC peak for CR sample. The areas under the DSC peaks for the two

samples (0.52 and 0.48 J/g for CR and EPR specimens, respectively) agree well despite the smaller dislocation density for CR sample at 809 K. This apparent dichotomy can be explained partially by the fact that the DSC peak starts at smaller temperature than 809 K for CR sample therefore the dislocation density at the beginning of the peak is most probably larger than that measured at 809 K. Moreover, it is also noted that for severely deformed metals a significant portion of the exothermic DSC peak can be attributed to point defects (e.g. vacancies) as it has been shown in previous papers (e.g. [12]). The vacancy concentration may be also different in CR and EPR samples which can not be measured directly by X-ray line profile analysis.

Summary

It was found that the sample processed by EPR has slightly higher dislocation density and smaller crystallite size than for the CR specimen resulting in a higher tensile strength for the former specimen. In the case of EPR sample, the relative fraction of <c+a> dislocations is smaller than for CR specimen. Most probably, the electric pulses reduced the internal stresses and therefore the probability of activation and multiplication of <c+a> type dislocations in EPR sample. In this sample the strain in the hexagonal <c> direction may be partially produced by the slide of fine grain boundaries which is facilitated by the enhanced diffusivity of vacancies due to electric current. It was found that the dislocation density was reduced before the exothermic DSC peak for both samples indicating that the recovery started before the appearance of the DSC peak. The relative fraction of the dislocation density annihilated before the DSC peak is larger for CR sample, most probably due to the higher fraction of <c+a>-type dislocations compared to EPR specimen.

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