High-Pressure Torsion of Ti: Synchrotron characterization of phase volume fraction and domain sizes

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Abstract. Rods of grade 2 Ti were processed by Equal-Channel Angular Pressing (ECAP) (φ = 120º at 573 K) employing 2, 4 and 6 passes. The same billets were further deformed by High-Pressure Torsion (HPT) at room temperature, varying both the hydrostatic pressure (1 and 6 GPa) and the number of rotations (n = 1 and 5). The ECAP and HPT samples were studied by synchrotron radiation at DESY-Petra III GEMS line. On the ECAP samples, textures were thus determined while for both ECAP and HPT samples the measurements were further analyzed by MAUD. Domain sizes and phase volume fractions were determined as a function of the radial direction of the samples. Alpha and Omega phases were detected in different amounts depending mostly on hydrostatic pressure and shear deformation. These transition phases can be pressure-induced during HPT processing and the results of Vickers microhardness measurements were related to the processing parameters and to the amounts of these phases.

Keywords: SPD, HPT, ECAP, ω-Ti, nanostructure.

1. Introduction

It is well known that severe plastic deformation (SPD) has excellent capability for grain refinement, thereby enhancing the mechanical properties of metals and alloys [1, 2]. Among the various SPD techniques recently developed, Equal-Channel Angular Pressing (ECAP) and High-Pressure Torsion (HPT) have received a great deal of attention: in the former, pure shear deformation can be repeatedly applied with no change in the cross-sectional dimensions of the sample, while the latter consists of the simultaneous application of pressure and shear. It has also been demonstrated that by combining SPD processes with more conventional deformation modes, ultrafine-grained microstructures useful for technological applications can be obtained [3-6]. Ti occupies a special place among the light metals since it combines high strength with low density and high corrosion resistance; consequently, its alloys find a number of applications in the aerospace industry, chemical equipment and medical implants. Regarding this last application, SPD techniques have been successfully applied to commercially pure Ti as a strategy for increasing its tensile strength.
up to the level of more complex Ti alloys, for example, the Ti-6Al-4V, customarily employed for biological implants or, more recently, the Ti-Nb-Ta-Zr alloys [7-9]. Besides the strengthening effect promoted by grain refinement, it has been observed that under certain circumstances, such as room temperature SPD and high pressures of 5 – 8 GPa, commercially pure Ti and Zr can form a phase known as \( \omega \)-phase [10-11]. Such pressure-induced \( \alpha(hcp) \rightarrow \omega \)(hexagonal) transformation has been reported for pure Ti under uniaxial pressure stresses higher than ~5 GPa [12]. Therefore, the high pressures involved in the HPT process can induce the \( \alpha \) to \( \omega \) transformation in Ti and Zr alloys, and recent investigations report that the fraction of \( \omega \)-phase increases with HPT strain, thereby promoting an increase in hardness and loss of ductility [13-16]. It is important to note that studies on the phase transformation and its relationship with the HPT process are relatively scarce.

Synchrotron radiation allows material characterization, with high accuracy, on many aspects such as the composition (volume fraction of each phase), domain sizes, defects such as twins, stacking faults and dislocations, textures and residual strains of many kinds. When high energy synchrotron radiation is used, its high penetration allows the potential for experiments that are usually far beyond the capabilities of any other technique. Besides, micrometer-sized beams allow the exploration of well localized very small amounts of materials for inspecting heterogeneity of the properties of concern, including volume fraction of phases. ECAP and, in particular, HPT samples usually provide only a small amount of material, having heterogeneous microstructure and properties as a result of the characteristic strain heterogeneity of the process. Such heterogeneity is difficult to evaluate because of many varying interaction effects between the anvil and the samples and the sample size effects.

The current paper describes a study of the correlations between local mechanical properties, indirectly measured by microhardness, and synchrotron radiation taken along the radii of the various ECAP + HPT severely deformed samples.

2. Experimental material and procedures

Samples of commercially pure grade 2 Ti (99.2 % purity) were annealed at 983 K for 2 h, resulting in an \( \alpha \)-phase microstructure with an average grain size of ~15 \( \mu \)m. Billets of 10 mm diameter and 70 mm length were then processed employing 2, 4 and 6 ECAP passes at 573 K, following route Bc [17] in a die with the angle between the channels equal to 120\(^\circ\), and under a ram speed of 5 mm min\(^{-1}\). Such process reduced the grain size down to ~150 – 200 nm. Further information on ECAP processing and the resulting mechanical properties of this material are available in an earlier report [7]. The ECAP-deformed samples were machined into disks having thicknesses of ~1.1 mm, which were carefully polished to give HPT disk samples of 0.82 ± 0.02 mm thickness and 10 mm diameter. HPT processing was performed at room temperature using a quasi-constrained facility [18], operating at a rotational speed of 1 rpm and varying both hydrostatic pressure (P = 1 and 6 GPa) and the number of rotations (N = 1 and 5). The samples were identified as ExPyTz, were x denotes the number of ECAP passes and y and z indicate, respectively, the HPT pressure and the number of turns. Vickers microhardness (Hv) was measured along the radial distance from the centers to the edges of the disks. An FM–800 microhardness tester was used with a load of 1 kgf and a dwell time of 15 s.

The samples were set on a programmable rotating and displacing stage for measuring each Debye-Scherrer diagram with a typical exposure time of 100 s. Single Debye-Scherrer diagrams from the center to the periphery were taken at every 1 mm displacement. ECAP 4-passes (4X) sample and ECAP 4X + 70 % rolling reduction samples were rotated around a vertical axis in order to characterize the complete texture and microstructure.

3. Experimental results

3.1 Microhardness measurements

The microhardness is shown in Table 1 as the maxima achievable at the edges of the disks after HPT processing. The hardness values measured along the radii of the disks are shown in
Fig. 1 as the variations of hardness with equivalent von Mises strain for the processed disks where the equivalent von Mises strain, $\varepsilon_{vM}$, is given by a relationship of the form:

$$
\varepsilon_{vM} = \frac{2 \pi X r}{h\sqrt{3}} 
$$

where $X$ is the number of torsion turns, $r$ is the radius and $h$ is the thickness of the sample.

Table 1: Notation, processing conditions and measured microhardness value of each sample.

| Sample      | ECAP ($\phi = 120^\circ/573$ K) | HPT       | Max. Hardness (*) |
|-------------|----------------------------------|-----------|-------------------|
|             | Passes | Pressure (GPa) | Turns | (Hv)       |
| E0P6T5      | 0      | 6              | 5     | 400        |
| E2P6T5      | 2      | 6              | 5     | 390        |
| E6P6T5      | 6      | 6              | 5     | 415        |
| E4P6T1      | 4      | 6              | 1     | 350        |
| E4P1T5      | 4      | 1              | 5     | 300        |
| 4X          | 4      | -              | -     | 245        |
| 4X+CR       | 4+70% CR | -              | -     | 305        |
| Annealed    | -      | -              | -     | 167        |

(*) – Maximum hardness, measured close to the edge of the disk.

Figure 1. Vickers microhardness measured along the radius of each sample as a function of the equivalent von Mises strain. E4P1T0 curve shows only the variation along the radius, without strain change due to torsion.

3.2 Synchrotron measurements.

The GEMS line at Petra III, DESY, allows the operation of a highly collimated, low divergence and intense x-ray beam with micrometer sized beams [19-20]. All samples processed by HPT were measured on a single shot approach in a direction perpendicular to the disk surface, going through the 1.1 mm thickness with a square 200 $\mu$m $\times$ 200 $\mu$m beam size. Measuring positions that approximately coincide with the measuring positions for microhardness are shown in Fig. 2. The experimental results were analyzed by MAUD by following the generally
accepted rules for reaching convergence after a few iterations for each parameter set. Peak broadening was analyzed by the Delf isotropic model, as implemented in MAUD [21-22]. Textures were also calculated by the E-WIMV algorithm. Although the results coincide with previous literature data, they are certainly not conclusive for the HPT samples because of the single Debye-Scherrer used for the calculations. Thus, more reliable results were obtained for the ECAP 4X and ECAP 4X + 70% rolled samples. A typical Debye-Scherrer image is shown in Fig. 3, where evidence for the presence of both α and ω phases, texture and peak broadening is visible.

Fig. 2. Measurement positions for synchrotron beam impingement. Fig. 3. Typical Debye-Scherrer image showing both phases and the presence of texture.

Fig. 4 shows the evolution of the ω-phase volume fraction along the radius of each HPT sample, with the horizontal axes displaying the von Mises strain calculated by eq (1) for each measurement point.

No evidence of ω-phase was found in the ECAP 4X and 4X + 70% rolled samples. The correlation ω volume fraction / microhardness, suggests that the main source for hardness is the presence of this phase. The other two microstructural features probably influencing the hardness are the grain size and micro-strains, which accumulated deformation energy mainly as dislocation arrays. They were calculated by the Delf model, as implemented in MAUD software, and are shown in Figs. 5 and 6. However, both quantities do not evolve much beyond 2 mm from the center of the disks, except for E4P6T1 which shows a high increment of the accumulated micro-strain on the ω–phase together with a decrement of the same variable for the phase α. This shows that the ω–phase is taking most of the deformation and increases when moving radially from the disk center. Also the data for ECAP 4X and 4X + 70% rolling are shown in Figs. 5 and 6. They are placed in a radial position where they would experience the same von Mises equivalent strain after ECAP (Null for 4X and 1.39 for 70% rolled).

4. Discussion

The current results show that a transition from α to ω phase is not only favored by high pressures but also by the shearing strain. In the semi-constrained arrangement for HPT, along the radii the pressure decreases and the shearing strain increases, so that it is obvious that a high pressure is not sufficient for inducing the phase transition at the largest volume fractions. Moreover, as shown by the absence of ω-phase in sample E4P1T5, the sole presence of shearing, 5 turns in this case, was not sufficient to induce the phase transition, and the same conclusion applies when large shear deformations are applied by the ECAP process.
Fig. 4. Omega phase volume fraction vs. von Mises strain developed along the sample radii.

As shown, domain sizes and micro-strains stabilize immediately beyond 1 mm from the center of the samples. Exception is made for the E4P6T1 sample, in which the α-phase domain size decreases at the edge of the disk, whilst the ω-phase domain size is kept almost constant along the total diameter, although it is close to the values of the other samples when plotted in terms of the von Mises strain.

While the micro-strain also stabilizes beyond 2 mm away from the center of the disks, sample E4P6T1 shows a preferential accumulation of micro-strains on the ω-phase on the rim of the disk. This higher accumulated microstrain could explain the microhardness, which is higher than on positions with similar von Mises strains on the samples subject to 5 turns of torsion. It can be inferred that the total deformation process can be understood as a competition between the phase transition and slip as mechanisms for carrying the plastic deformation. The complete interaction should be studied more carefully by inspecting intermediate numbers of turns for varying pressures. Finally, the reasonable correlation between microhardness and the ω-phase volume fraction can be explained by the combined effect of precipitation hardening and residual stresses. With regard to the former, it has been reported that the lattice misfit between Ti and ω is less than 4% [10], the figure which normally marks the transition between coherent and incoherent interface, hence precipitation hardening is to be expected in the present case. Second, a comparison of the unit cell volume for ω and Ti-α
shows that the former is approximately 50% larger and consequently residual stresses will build up and affect the hardness.

5. Summary and conclusions

1. The $\alpha \rightarrow \omega$-phase transition is favored by shearing strain combined with high pressures. By contrast, large shear strain does not produce the phase transition in the absence of sufficient hydrostatic pressure.

2. High-Pressure Torsion decreases the domain sizes to almost half of that obtained by ECAP processing at 4X. However it is almost as effective as ECAP + 70% rolling in decreasing the domain sizes to an average of 50 nm. As a general rule, after 5 turns at 6 GPa pressure the domain size does not depend significantly on the number of ECAP passes and the $\omega$-phase domain sizes are smaller than those of the $\alpha$-phase.

3. Microstrains, accumulated as dislocation arrays, are typically between $2 \times 10^{-3}$ and $3 \times 10^{-3}$. Both phases do not differ much in their capacity to carry plastic deformation, except in the E4P6T1 sample which shows a larger energy accumulation of the $\omega$-phase to the detriment of the plastic strain for the $\alpha$-phase.

4. Microhardness is well correlated with the $\omega$-phase presence and only secondarily with domain sizes and microstrains. For instance, there is a slight trend to accumulate less microstrain for the highest volume fraction of the $\omega$-phase, as if a large part of the deformation were carried by the phase transition itself. However the opposite appears to happen in sample E4P6T1: when the $\omega$-phase presence increases the same occurs with the accumulation of plastic deformation and also in the same phase.

5. It can be suggested that the evolution of hardness is linked to both precipitation hardening and residual stresses caused by the large unit cell volume differences of Ti-$\alpha$ and the $\omega$-phase.

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