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The recrystallized volume fraction of Ti 3Al 2.5V seamless tubes is measured using Electron BackScatter Diffraction (EBSD) after annealing under various conditions. Standard tensile tests and Contractile Strain Ratio (CSR) measurements are carried out in order to analyze the effect of recrystallization on the tensile behavior and the anisotropy of the tubes. The tensile tests show anomalous yield-point phenomena, which become stronger when the recrystallized fraction is increased. CSR value changes through recrystallization, from 0.8 in the Cold Worked Stress Relieved (CWSR) state to 1.1 in the fully recrystallized structure. Orientation Distribution Functions (ODF) calculated from X-ray data reveal a decay in the intensity of the crystallographic texture as recrystallization advances. This can explain the tendency towards isotropy when complete recrystallization is achieved.

Ti-3Al-2.5V alloy is a near alpha titanium alloy widely used for the production of seamless tubes destined for the aeronautic industry. The predominant alpha phase has a hexagonal structure and can be textured through processing. The cold pilgering process is a widespread seamless tube forming operation in which the tube wall thickness and the inner diameter are reduced simultaneously. Tailoring of the mechanical properties in cold pilgered tubes of Titanium-base and Zirconium-base alloys is mainly achieved through control of the strain paths during the cold working stages. It is now widely accepted that forming operations with a high ratio of wall thickness to diameter reduction produce a preferential orientation of the basal poles nearly parallel to the radial direction of the tubes which benefits the final mechanical properties.[1,2] This fact has promoted the use of Kearns factors as a common industrial parameter to quantify the anisotropy of cold-pilgered tubes.[3] Nevertheless, Ti-3Al-2.5V seamless tubes are mostly employed in the CWSR condition. In industrial practice, some recrystallization might take place during the final heat treatment, which has a major effect on adjusting the mechanical properties (as the yield stress, the mechanical strength or the total relative elongation) to the user requirements. The purpose of this work is to study and explain the impact of the parameters of the final heat treatment on the microstructure and crystallographic texture of the tubes, and consequently, on their final mechanical properties and anisotropy.

Experimental

The Ti-3Al-2.5V (ASTM grade 9) tubes were formed by several cold pilgering passes with intermediate heat treatments. After the final pilgering pass, which involved 66% equivalent strain, the cold worked tubes were annealed for 120 minutes at different temperatures listed in **Table 1**. In this work, the effect of the heat treatment temperature was characterized through the recrystallized volume fraction (Rx) at the end of the heat treatment. Then, the effect of recrystallization over the evolution of microstructure, texture, tensile properties and anisotropy was analyzed.

Table 1. Recrystallized volume fraction, Kearns factors, and texture index values obtained after final annealing for 2h at the displayed temperatures.

Annealing temperature [K]	Recrystallized vol. fraction [%]	f_{RD} [a]	f_{TD} [a]	f_{ED} [a]	Texture index J
-	0 (CW)	0.4354	0.4405	0.1241	3.188
838	7	0.4528	0.4337	0.1134	3.604
863	21	0.4408	0.4693	0.0898	4.142
883	46	0.4381	0.4410	0.1209	2.847
933	90	0.4699	0.3914	0.1387	1.959
993	100	0.4356	0.4074	0.1570	1.817

[a] f_{RD}, f_{TD} and f_{ED} are the Kearns factors,[3] calculated for: RD, the radial direction, TD, the tangential direction and ED, the elongation direction.

For texture characterization, the tubular samples were cut to appropriate size (2 cm long), and then chemically thinned to achieve the final thickness allowing elastic flattening. The macroscopic coordinate system is then: ED –elongation direction, TD – tangential direction, and RD – radial direction. All the textures were determined from 5 incomplete pole figures measured by X ray diffraction using Co-Kα radiation. The Orientation Density Function (ODF) was computed using the MTEX method proposed by Hielscher and Schaeben.[4]

Microstructures were characterized using Electron BackScatter Diffraction (EBSD) in particular to assess the recrystallized volume fraction. EBSD samples were cut along the longitudinal direction and through one of the tube diameters. The surface normal to the circumferential direction was then mechanically polished and finally electropolished using a 10% $HClO_4$-90% CH_3OH solution at 17 V and 10°C. EBSD mapping was performed with a step size of 0.2 µm in a JEOL 6500F FEGSEM equipped with a HKL EBSD system. To ensure the statistical relevance of the recrystallized volume fractions, a minimal area of 18000 µm² was scanned for each sample.

The following EBSD data processing procedure was designed using the Channel5 software. A grain was defined as a group of at least 5 neighboring indexed points having the same crystal orientation within a tolerated angular deviation of 5°. Also, any given grain was considered as recrystallized if its internal mean misorientation was less than or equal to 1.5°, which is the maximum measured value of intragranular mean misorientation for the fully recrystallized sample. The recrystallized volume fraction was then calculated as the fraction of points in an EBSD orientation map belonging to the recrystallized grains.

Tensile tests were conducted at ambient temperature with a cross-head speed maintained at 5 mm/min, so that the initial strain rate was 2.4×10^{-4} s^{-1}. CSR measurements were carried out in order to evaluate the anisotropy of the tubes. The CSR is defined as the ratio of circumferential to radial strain after a given tensile strain (3.5%) as described in the SAE AS 4076 specification.[5]

Results and Discussion

The estimated recrystallized volume fractions obtained after the heat treatments are displayed in **Table 1**.

Microstructure Evolution. EBSD pattern quality maps (as evaluated here by the band contrast value provided by the Channel 5 software) allow one to visualize the microstructural changes that occur with recrystallization. **Figure 1** shows the pattern quality maps for the 7% Rx, 46% Rx and fully recrystallized conditions. In the 7% Rx condition, the pattern quality is low because of the crystal distortions caused by the forming process. Nevertheless, a few recrystallized grains can be recognized. At the 46% Rx condition, some grains have nearly achieved the final grain size, while other grains are smaller. The size distribution may be explained by the nucleation kinetics, new grains appearing sooner or later according to the local stored energy level. In the fully recrystallized condition, the microstructure is nevertheless quite homogeneous, with an average grain diameter of 4 μm.

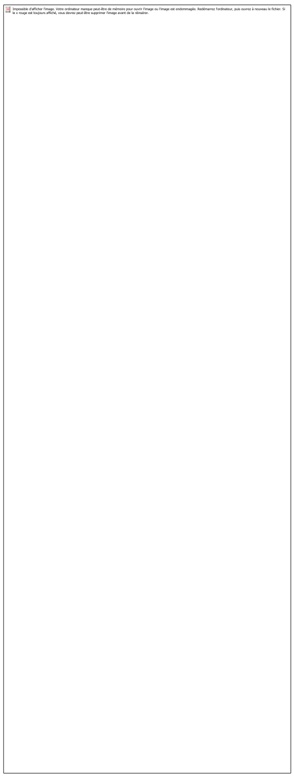

Fig. 1. EBSD Pattern quality maps of (a) 7% Rx, (b) 46% Rx and (c) fully recrystallized Ti-3Al-2.5V tubes.

Tensile Behavior. Examples of the true stress - true strain curves plotted for each of the recristallization conditions are shown in **Figure 2a**. For tubes with Rx over 20% a marked yield point phenomenon (plateau) was observed. This can be explained by the advance in recrystallization and the consequent decrease of the initial dislocation density, which is then suddenly augmented at the beginning of plastic deformation. In order to accommodate the imposed strain rate, the speed of the mobile dislocations, which is a strong function of the stress, must decrease, and the yield point phenomenon appears. The so-called Lüders elongation increased with the recrystallized volume fraction (**Figure 2b**).

Fig. 2. a) Examples of the true stress–true strain curves obtained for the different recrystallization conditions. b) Zoom to the yield point phenomena, observed for samples with more than 20% recrystallized volume fraction.

Because of the reduced ductility, CSR measurement was not possible in the cold worked condition. The CSR values for the other conditions show an increase of 30% between the least (7%) and the greatest (100%) recrystallized specimens, as shown in **Figure 3a**.

The values of yield point, mechanical strength and relative total elongation, measured at different degrees of recrystallization, are plotted in **Figure 3b.** As expected, the mechanical strength and the yield point of the tubes decreased with increasing recrystallization. Both values were found to be strongly correlated with the recrystallized volume fraction, showing a very good reproducibility of the tensile flow stresses on tubes over tubes after same heat treatment. As for the total relative elongation, the results showed a greater dispersion, this might be explained by complications related to the tensile test; because no notch was made on the tubular samples, necking did not always occur in the gauge length measured by the extensometer. Nevertheless, the observed tendency is the usual increase in ductility which comes with recrystallization, as a consequence of the lower dislocation density.

Fig. 3. a) Evolution of the texture index and CSR during recrystallization. b) Elongation (El), mechanical strength (Rm) and yield point (Re) for the tensile-tested samples with different recrystallized fractions.

Texture evolution. Figure 4 shows that the textures of all investigated samples (cold worked and annealed) have the orientation density maxima at the same position. The basal poles are spread in the plane normal to the axial direction, with a higher density at 60° from the radial direction, and crystals preferentially exhibit a <10-10> direction aligned with the axial direction. Only the texture strength decreases when recrystallization progresses over 45% Rx.

Fig. 4. $(10\bar{1}0)$ *And* (0001) *pole figures of the a) cold worked, b) 7% Rx, c) 21% Rx, d) 46% Rx, e) 90% Rx and f) fully recrystallized conditions. The same grey-scale applies for all pole figures.*

The texture index gives a description of the sharpness of the texture, it is defined as follows,[6]

$$J = \oint [f(g)]^2 dg \qquad (1)$$

Where *f(g)* is the orientation density function and *g* the orientation. The evolution of the texture index during recrystallization is shown in **Figure 3a** and the calculated values are specified in **Table 1**. *J* increases at the very beginning of recrystallization, and then decreases steadily as recrystallization progresses. This result shows that *J* is a suitable parameter to evaluate the advancement of the recrystallization process in Ti-3Al-2.5V tubes, at least in the particular situation where the texture type does not change.

As illustrated by **Figure 4b and c**, the rolling texture is reinforced at 7% Rx and 21% Rx which is in agreement with the texture evolution observed in commercially pure titanium at the beginning of recrystallization.[7] Further recrystallization leads on the contrary to a texture strength decrease (**Figure 4d, e** and **f)**, but the final texture remains close to the deformation texture. In particular, the well-known 30°<0001> rotation that describes the texture change which is classically observed during annealing of hcp metals, does not occur here. It has been shown that this texture evolution during annealing of strongly cold rolled low alloyed titanium sheets, leading to the improperly so-called “recrystallization texture”, is actually mainly controlled by grain growth.[8] In the present study, grain growth was very limited under the applied conditions, which probably contributes to the texture stability. In fact, Ti-3Al-2.5V contains some dispersed β phase, stabilized by the vanadium content. The β phase is known to reduce the rate of grain growth in alpha or near-α titanium alloys,[9] which may in part explain the low average grain size obtained in the fully recrystallized condition.

The Kearns factors, which give a description of the amount of basal poles aligned in each direction, were calculated from the (0002) pole figures according to Anderson et al.[10] Those calculated values, shown in **table 1**, do not vary much, and therefore cannot by themselves explain the observed CSR increase.

The low values of the CSR at the initial condition are a consequence of the inclination of the basal pole towards the tangential direction.[1,2] During recrystallization, the evolution of the CSR responds to the same phenomenon observed for the texture index *J*, also plotted on **Figure 3a**. The weak value of *J* in the fully recrystallized condition describes the smoothness of the final texture, which is equivalent to saying that the material has lost much anisotropy, and, consequently CSR comes closer to the isotropic value of 1. This explains the measured tendency of the material towards an isotropic behavior as recrystallization advances.

Conclusions

1. The variations in the CSR taking place during recrystallization are the result of the weakening of the crystallographic texture, which can be quantified by the texture index *J.*
2. Since the texture changes occurring during recrystallization in alpha titanium alloys do not affect the orientation distribution of basal poles, Kearns factors are not suitable to describe the observed evolutions.
3. Recrystallization is associated with the appearance of yield point phenomena and Lüders elongation in strongly annealed Ti-3Al-2.5V tubes.
4. Besides the reported yield point phenomena, the tendencies observed for tensile behavior respond to the usual consequences of recrystallization in alpha titanium alloys.

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