

# Kinetics of Recrystallization and Grain Growth in Ti-17 Alloy

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The aim of this research is to simulate the forging step in the two phase  $\alpha/\beta$ -range followed by recrystallization in the  $\beta$  field during which microstructure evolves. This involves the following phenomena: grain deformation, recrystallization and grain growth for the  $\beta$  matrix and disorientation/fragmentation for the  $\alpha$  Widmanstätten platelets. More specifically, this work analyzes the effects of a deformation in the  $\alpha/\beta$  field on the subsequent  $\beta$  grain growth during a heat treatment at higher temperatures for a Ti-17 titanium alloy. The experimental part is based on uniaxial compression tests in order to simulate the forging step. The complete thermomechanical treatment starts with a temperature homogenization step (10min) followed by uniaxial compression with a strain rate of  $10^{-2} \text{ s}^{-1}$  in the  $\alpha/\beta$ -phase field and a water quenching; the samples are then subjected to a recrystallization / grain growth step at higher temperatures (in the  $\beta$ -phase domain). The evolution of grain structures and substructures were investigated by optical and scanning electron microscopy (SEM) and electron backscattering diffraction (EBSD). The influence of strain and heat treatments time on the microstructure is described. The kinetics of recrystallization and grain growth in the alloy have been determined.

**Keywords:** Titanium (Ti), Ti-17 alloy, dynamic recrystallization (DRX), microstructure, grain growth

## 1. Introduction

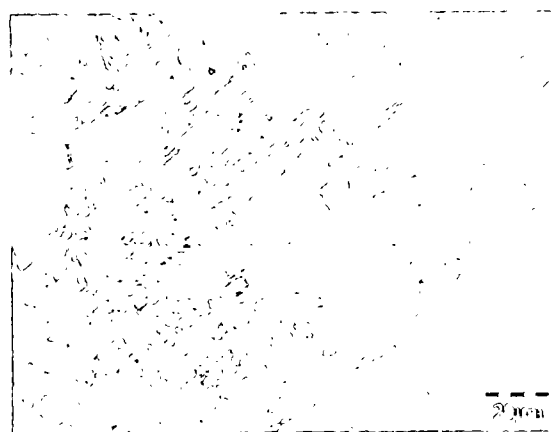
Hot forming is a major stage in the processing route of titanium alloys<sup>1,2</sup>. During forging operation, the conditions of strain and of subsequent heat treat cycle have a significant influence on the structure and properties of the final product. This is the result of the interaction of a certain number of physical phenomena concerning titanium: morphology and work state of the grain, recovery, static or dynamic recrystallization, allotropic transformation and precipitation. The laws and kinetics which govern these phenomena must therefore be known in order to optimize the microstructure and the mechanical properties of titanium alloys. A key issue is to predict the influence of the microstructure resulting from a given operation on further microstructure evolutions during the subsequent stages of the process. More precisely, the effects of a deformation in the  $\alpha/\beta$  field on the subsequent  $\beta$  grain growth during a heat treatment at higher temperature have been studied. The kinetics of the  $\beta$  grain growth is mainly influenced by the generation of new sub-grains and grains by dynamic recovery and recrystallization during the deformation of the two-phase microstructure. Such studies have already been investigated on titanium alloys, especially on Ti 64 (Ti-6Al-4V)<sup>3-5</sup>. However, for Ti-17 alloy which is a beta-metastable alloy with beta transus between 880°C and 900°C, used in high pressure compressor disks, there is little work focusing on the  $\alpha/\beta$  forging step, microstructural evolution during  $\beta$  heat treatment and their relationship.

The objective of the present study is to analyze the microstructure evolution during heat treatments in the  $\beta$  field of a Ti-17 alloy initially hot deformed in the  $\alpha/\beta$  field. Hot compression behavior of Ti-17 alloy was examined by compressive tests over a wide range of

strains. The strain rate and the temperature were chosen in order to be line up with the standard forging conditions of Ti-17 alloys (with hydraulic presses). The relationships between deformation parameters and correlated microstructure evolutions were investigated.

## 2. Material and Procedure

Ti-17 samples have the chemical composition of Ti-2.01Sn-4.07Mo-2.06Zr-0.04Fe-5.07Al-0.01C-0.11O-0.004N-4.07Cr (wt-%). The microstructure of the starting material is shown in Figure 1.



**Figure 1.** Microstructure (optical microscopy) of as received Ti-17 alloy with Widmanstätten lamellae

As seen in Figure 1, the initial alloy has a typical lamellar microstructure with  $\sim 830 \mu\text{m}$  former  $\beta$  grain size and  $20\text{-}40 \mu\text{m}$  length,  $\sim 1\text{-}2 \mu\text{m}$  thick  $\alpha$  lamellae.

Isothermal compression tests were conducted on Ti-17 alloy samples with initial lamellar microstructures in order to simulate the forging step in the  $\alpha/\beta$ -range. For this purpose, cylindrical specimens measuring 10 mm diameter and 15 mm in height were machined by

electroerosion. The cylinder ends were grooved for retention of the glass lubricant used during compression tests ( $C_{\text{Graphite}}$ ). Specimens were heated to test temperature, soaked for 10 min, and then upset under constant axial strain rate to a height reduction range 0%-100% with 25% intervals. The strain rate was  $10^{-2} \text{ s}^{-1}$ . The stress/strain curve corresponding to this compression test is shown in Figure 2. After hot compression, the specimens were water-quenched immediately, to preserve the hot deformed structures. At the end, a set of heat treatments (in the  $\beta$  field) were conducted on the samples. The thermomechanical sequence is described in Figure 3.

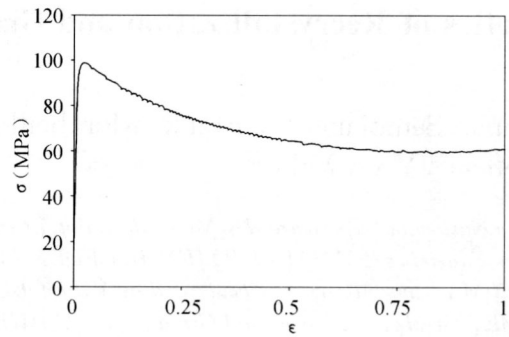


Figure 2. stress/strain curve of the compression test in the alpha/beta field

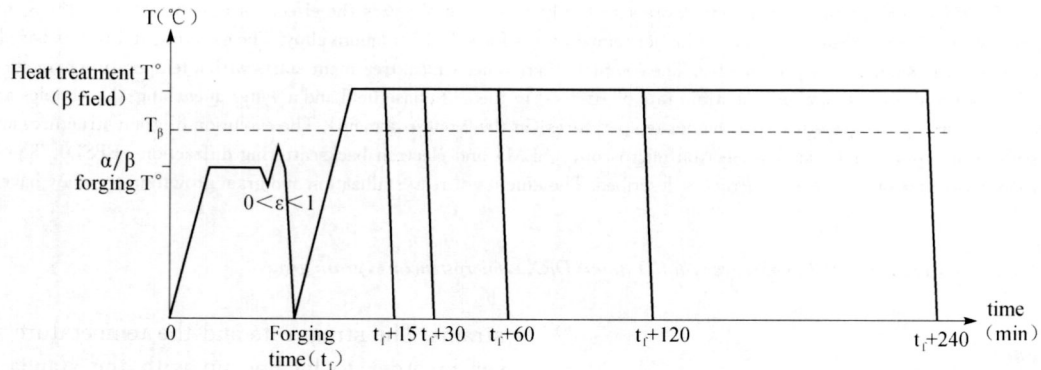


Figure 3. Thermomechanical sequence

Treated specimens were axially sectioned parallel to the compression axis and the cut surface was prepared for metallographic observation in the center zone. The specimens observed by optical microscopy were etched by the following solution: 8% HF + 40% HNO<sub>3</sub> + 52% H<sub>2</sub>O. Samples for SEM were prepared by electropolishing in a solution of 6% HClO<sub>4</sub> + 35% CH<sub>3</sub>(CH<sub>2</sub>)<sub>3</sub>OCH<sub>2</sub>CH<sub>2</sub>OH + 59% CH<sub>3</sub>OH and observed on a JEOL 6500 microscope.

### 3. Microstructure Evolutions During the Thermomechanical Sequence

The evolution of grain size of the Ti-17 alloy hot compressed to the strain of 25%, 50% and 100% in the  $\alpha/\beta$  field at  $10^{-2} \text{ s}^{-1}$  is shown in Figure 5 and 6. All curves showed that during the post-forging heat treatment, there are two successive stages. It starts with a recrystallization step during which the grain size is divided by two; then a grain growth phenomenon occurs and, if the heat treatment is long enough, the final grain size may exceed the grain size just after the  $\alpha/\beta$  forging. Therefore, it seems possible to refine the grains through a sequence of forging steps followed by a  $\beta$  heat treatment. Indeed, static recrystallization takes place during the early stand; it is to reorganize the sub-grain boundaries (that appeared during forging) and create new grain boundaries that, overall, results in refining and homogenizing the grain size. The schematic evolution of the microstructure is described in Figure 4<sup>6)</sup>.

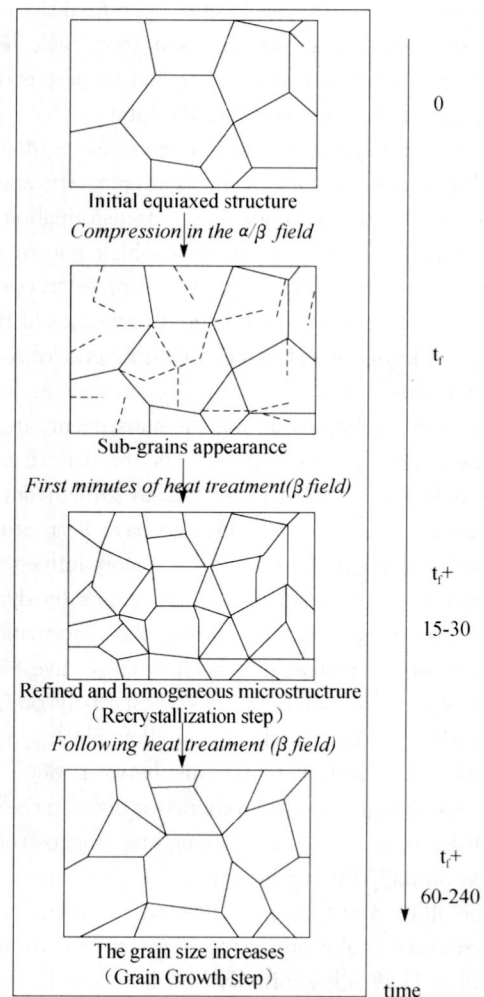
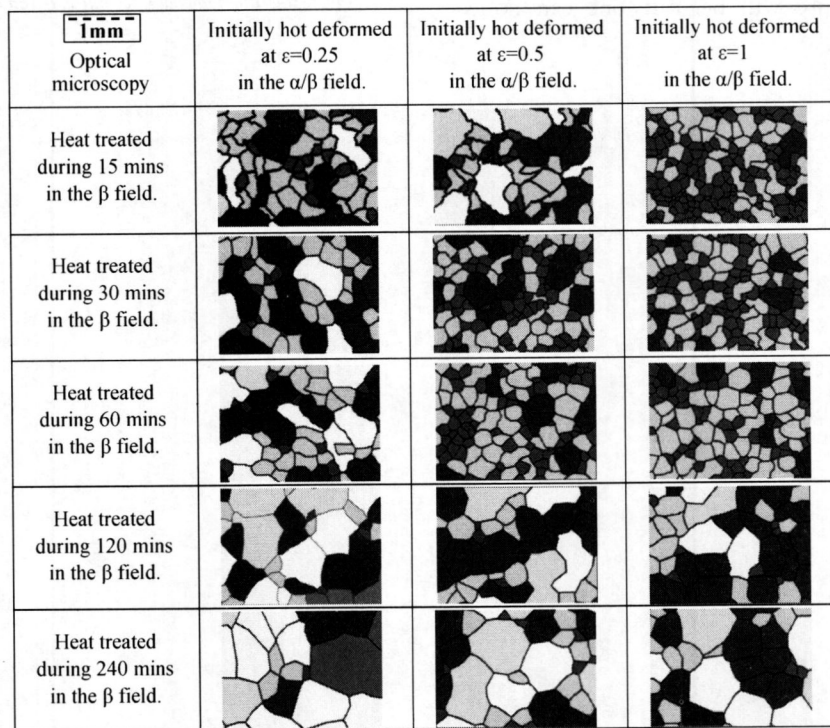


Figure 4. Microstructure evolution during thermomechanical treatment ( $\alpha+\beta$  compression and  $\beta$  heat treatment)

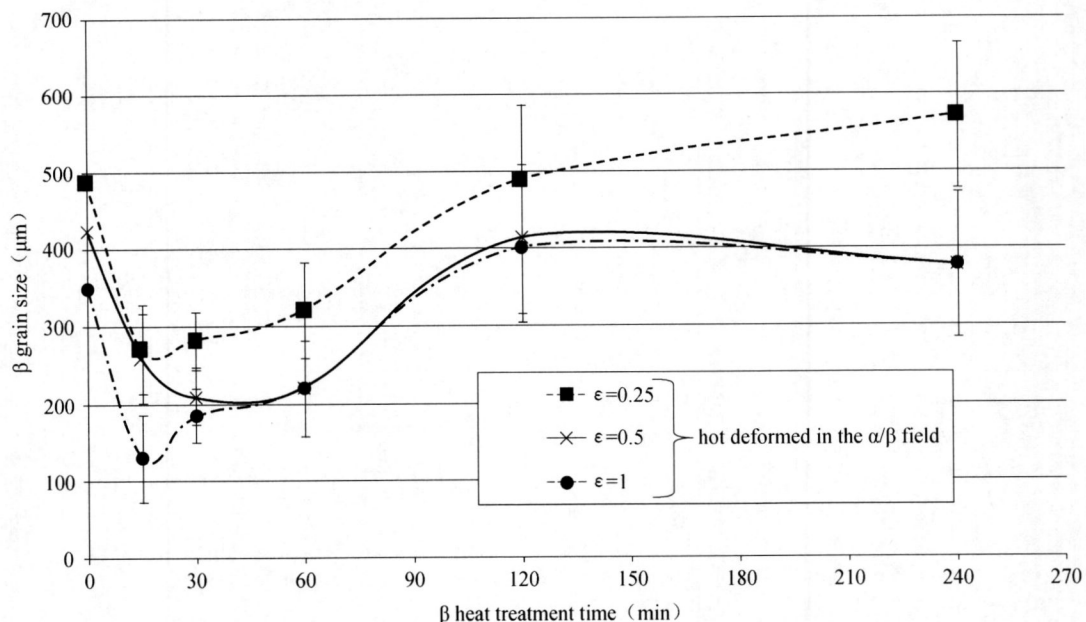
#### 4. Results: Grain Growth Kinetics

In order to determine the kinetics of recrystallization and grain growth, the microstructure of samples strained and heat treated in different conditions has been investigated with optical microscopy and SEM. The results concerning the grain size are shown in Figure 5 and Figure 6. It is remarkable that the kinetics of recrystallization is quite similar for all the samples hot deformed between 0.25 and 1.00; after a heat treatment of 15-30 minutes, the most homogeneous and refined microstructure can be observed. The minimal

grain size is obtained for the samples the most strained (less than  $200\mu\text{m}$  for the alloy hot deformed at  $\epsilon=1$  and heat treated during 15 minutes) but because the microstructure evolves significantly during the heat treatment, the relevant industrial values are the ones after several hours of heat treatment. Yet, after a few hours of  $\beta$  heat treatment, the grain size is finer for highly deformed samples than for lower deformed sample. Moreover, the grain size seems to stabilize for the high deformed samples whereas, the grain size keeps increasing for the alloy hot deformed to a strain of 0.25.



**Figure 5.** Images (obtained by optical microscopy and worked with Analysis software) of Ti-17 alloy deformed in the  $\alpha/\beta$  field, strain between 0.25 and 1 and heat treated in the  $\beta$  field between 15 and 240 minutes (each grey level corresponds to a grain size class)



**Figure 6.** Effect of the  $\beta$  heat treatment time on the microstructure evolution at constant strain

## 5. Conclusions

Isothermal hot compression tests in the  $\alpha/\beta$  field and heat treatment in the  $\beta$  field were conducted on Ti-17 alloy. The final microstructures were analyzed to quantify fragmentation and grain growth kinetics. The following conclusions were drawn from this work.

(1) During the  $\beta$  heat treatment, there is a step of recrystallization which occurs in a few minutes and which is followed by a very slow grain growth step.

(2) Initial strain has an influence on the final microstructure; the more strained the sample, the finer the final microstructure will be. But, one can notice

that a strain of 0.5 can achieve a fine structure.

## REFERENCES

- 1) C. Sauer and G. Luetjering, *J. Mater. Process. Technol.* 117 (2001), pp. 311-317.
- 2) I. Weiss and S. L. Semiatin, *Mater Sci Eng A* 243 (1998), pp. 46-65.
- 3) S. L. Semiatin, V. Seetharaman and I. Weiss, *Mater. Sci. Eng. A* 263 (1999), pp. 257-271.
- 4) S. Mironov, M. Murzinova, S. Zharebtsov, G. A. Salishchev and S. L. Semiatin, *Acta Mater.* 57 (2009), pp. 2470-2481.
- 5) J. H. Kim, S. L. Semiatin and C. S. Lee, *Mater. Sci. Eng. A* 394 (2005), pp. 366-375.
- 6) F. J. Humphreys, M. Hatherly, *Recrystallization and Related Annealing Phenomena (Second Edition)*, (2004).