## Study of hot multiaxial processing of titanium alloy

## Margaux Saint Jalme<sup>a</sup>, Julien Favre<sup>b</sup>, Christophe Desrayaud<sup>c</sup>, Damien Fabregue<sup>d</sup>, Sylvain Dancette<sup>e</sup>, Etienne Archaud<sup>f</sup> and Christian Dumont<sup>g</sup>

- (a) Ecole des Mines de Saint-Etienne (EMSE) margaux.saint-jalme@emse.fr
  - (b) Ecole des Mines de Saint-Etienne (EMSE) julien.favre@emse.fr
  - (c) Ecole des Mines de Saint-Etienne (EMSE) cdesray@emse.fr
- (d) MATEIS Laboratory -INSA Lyon (MATEIS) damien.fabregue@insa-lyon.fr
- (e) MATEIS Laboratory -INSA Lyon (MATEIS) sylvain.dancette@insa-lyon.fr
  - (f) Aubert & Duval etienne.archaud@eramet.com
  - (g) Aubert & Duval christian.dumont@eramet.com

## **Résumé :**

Une procédure originale permettant d'exploiter des gradients de température et de déformation proches de ceux rencontrés lors du forgeage multiaxial à chaud des alliages de titane est présentée. Le module de déformation multiaxial MaxStrain du système de simulation physique « Gleeble 3500 » permet de créer des chemins de déformation complexes proches de ceux rencontrés pour les gammes industrielles. Les gradients seront identifiés en croisant les champs de microstructure expérimentaux avec les gradients thermomécaniques simulés par éléments finis, afin de comprendre l'effet de tels gradients sur le comportement rhéologique du matériau et sur les transformations de la microstructure.

#### Mots clefs : forgeage multiaxial à chaud, alliages de titane, Gleeble MaxStrain, gradients thermomécaniques

## Abstract:

An original procedure is presented to take advantage of temperature and strain gradients similar to the ones encountered in hot open-die forging of titanium alloys. The multi-axial deformation module MaxStrain of the physical simulation system "Gleeble 3500" is used to create complex deformation paths close to industrial ones. The gradients will be identified by crossing microstructure fields and finite element simulation of thermomechanical gradients, in order to understand the effect of such gradients on the material flow and on the microstructural transformations.

# Key words: hot open-die forging, titanium alloys, Gleeble MaxStrain, thermomechanical gradients

#### **1** Introduction

Ti-6Al-4V (TA6V, grade 5) is the most common alloy used in aeronautic industry due to excellent service performance (high specific strength and good corrosion resistance) [1]. It is usually formed by high temperature processing. The industrial forging processes involve a long series of plastic deformation followed by rotations to decrease the section of the bars down to obtain long products. The last step of the process aims at obtaining a globularized structure of alpha phase. Empirical or semi-empirical constitutive models considering thermomechanical variables are widely used to predict microstructure evolutions and plastic flow behaviour during hot forging process [2]–[4]. Nevertheless, the effect of multiaxial forging combined with heterogeneous temperature and intermediate delay between deformation steps on the microstructure transformations has not been yet reported in literature. This paper presents an experimental procedure to reproduce such thermomechanical conditions in laboratory to understand better the evolution of microstructure and mechanical properties during the industrial process of TA6V. A first series of uniaxial hot isothermal compression tests are carried out to extract the constitutive law of the flow stress for this two-phased alpha-beta titanium alloy. These results will feed our finite element model of Gleeble MaxStrain tests, designed in order to be representative of the industrial operating ranges.

## 2 Material and experimental procedure

Two-phase alpha-beta titanium alloy Ti-6Al-4V chemical composition is given in Table 1. The alpha phase estimated volume fraction at room temperature is 92 % [5] and the measured transus temperature  $(\alpha + \beta \leftrightarrow \beta)$  is  $T_{\beta} = 995$  °C.

|           |            |             | a            |             |        |
|-----------|------------|-------------|--------------|-------------|--------|
| Tahlo 1 - | Chemical   | composition | of Ti-6AI-4V | in weight r | prcont |
| I UDIC I  | Gilcinicul | composition |              | in weight p |        |

| Al   | V    | Fe     | 0      | С      | Ν      | Ti   |
|------|------|--------|--------|--------|--------|------|
| 6.00 | 4.00 | < 0.30 | < 0.20 | < 0.08 | < 0.07 | Base |

Testing temperatures are chosen between 750 °C and 1100 °C in order to scan temperatures below and above the beta transus. Classical strain rate for open-die forging are set between 0.001 s<sup>-1</sup> and 0.1 s<sup>-1</sup>.

The as-received microstructure of the material is presented in Figure 1. This paper focuses on the axis equal to the billet axis, denoted 'axis 1'. Compression tests along the two other axis of the billet will be studied later to evaluate the effect of alpha lamellae orientations. Alpha platelets are observed in large equiaxed prior beta grain having a milimetric size. The alpha platelets have a marked orientation perpendicular to the analysis surface, creating white "flower" patterns. It is the typical microstructure before the globularization-forging step.



Figure 1 - Microstructure of the as-received material, along the billet axis

#### 2.1 Uniaxial hot isothermal compression tests

The specimens have a standard cylindrical shape (height 15 mm, diameter 10 mm) which allows to reduce strain heterogeneity due to friction [5]. To minimise friction, a coating of carbon powder is used. It is completed with a boron nitride coating for chemical inertia. Uniaxial hot isothermal compression tests are processed with a Schenck<sup>TM</sup> hydraulic machine [6].

Specimens are heated up using a lamp furnace in a quartz tube under argon atmosphere to prevent oxidation. They are held 10 minutes at the desired temperature before compression, to ensure temperature homogeneity, then water-quenched after deformation to fix the high temperature microstructure. For each specimen of this first series of tests, the strain is equal to 1.0. The tested strain rate are 0.1 s<sup>-1</sup>, 0.01 s<sup>-1</sup>, 0.001 s<sup>-1</sup>. The tested temperatures are 750 °C, 850°C, 950 °C, 1050 °C and 1100 °C.

For metallographic observation, the specimens are cut along the central axis. Sample preparation for optical microscopy (OM) includes mechanical grinding up to colloidal silica and Kroll chemical etching. Sample preparation for scanning electron microscope (SEM) includes mechanical grinding up to colloidal silica and finishing on Vibromet<sup>TM</sup>.

#### 2.2 Multiaxial hot cross-forging tests

Gleeble physical simulation system offers the opportunity to explore closer the effect of complex strain and temperature paths. With the MaxStrain module, it is now possible to carry out automatic cross forging of specimens under plane-strain compression with high reproducibility. Such experimental test generates thermomechanical gradients that we need to identify precisely for further understanding the effect of such gradients on microstructure evolution, and especially globularization of alpha lamellae. Figure 2 shows a film sequence of a MaxStrain test.





Figure 2 - Schematic drawing [7] (a) and a sequence (b) of the experimental setup of multiaxial forging technique in the MaxStrain System

Specimen are 196 mm long, with a square section of 15 mm, and the deformation is localized in a zone with a smaller section of 12 mm (Figure 8). The control thermocouple is deported near the deformation zone to avoid damage during the cross-forging. For the analysis of the thermal field, a secondary monitoring thermocouple is set on the deformation zone, and it is forged together with the workpiece. First tests were tested on carbon steel to evaluate the experimental setup. The deformation sequence is a series of 40 hits at 900°C, with a displacement of 10 mm and a speed of 50mm.s<sup>-1</sup>. The pressure is maintained 0.5 s after each hit, then the punch is retracted. Then the specimen is rotated of 90° (Figure 2), with a duration of 0.8 s (0.5 s delay, and 0.3 s for the rotation), and a new compression cycle is started. At the end of the sequence, the specimen is air cooled.

## **3** Results and discussion

### **3.1** Constitutive law of the flow stress of the alloy (axis 1)

This first set of curves obtained by uniaxial compression are illustrated on Figure 3. The curves show a work hardening stage for low strain (<0.1), followed by flow softening with more or less intensity, depending on temperature and strain rate. Semiatin [8] found that flow softening of TA6V with a colony microstructure is generated by deformation heating effect and platelet bending or kinking. Later, the same author [9] deduced that flow softening in flow stress-strain curves can be interpreted by the loss of alpha/beta interface strengthening. Nevertheless, one can observe a real controversy about flow softening mechanism in literature. For instance Roy [10] stated that flow softening was generated by globularization and dynamic recrystallization of alpha lamellae at higher temperature.



Figure 3 - Stress strain curve for different temperature and strain-rate test conditions

As Xu [11] observed it for alpha/beta Ti-17 alloy, all curves reach a peak stress for strains below 0.1. Maximum peak stress is registered for the lower temperature, 750 °C, and for the higher strain rate, 0.1 s<sup>-1</sup>. It reaches 350 MPa for {750 °C; 0.1 s<sup>-1</sup>} test conditions, compared to 10 MPa for {1100 °C; 0.001 s<sup>-1</sup>}. Same trend is observed for flow softening intensity. Temperature and strain rate have an important influence on peak stress and flow behaviour. According to the same authors, the deformation is assumed to be adiabatic for strain rate up to 0.1 s<sup>-1</sup>. They assume that 95% of the deformation work (area under the stress strain curve) is converted into heat:

$$\Delta T = \frac{0.95 * \int \bar{\sigma} * d\bar{\varepsilon}}{\rho * c}$$

With  $\rho$  the density and c the specific heat of TA6V.

They concluded that only a small portion of flow softening could be attributed to adiabatic heating. This is correlated with [11] conclusions : the bending, kinking and rotation of the lamellar alpha before the initiation of dynamic globularization are major characteristics of microstructure changes to explain the initial flow softening.

One can also notice that the alpha + beta microstructure stress-strain curves show more intense flow softening than pure beta microstructure, correlated to literature results [9]. It was shown by Colin [12] that the presence of a peak in flow stress tends to vanish in pure beta phase. Saby [4] studied the

individual influences of phase fraction and of temperature in the two-phases domain. It was shown that for TA6V at 955 °C, the flow stress of beta phase is about 60 % lower than that of alpha phase.

Microstructures of deformed specimens along the compression axis are presented in Figure 4. Depending on the alpha + beta phases fractions during the deformation, the morphology of prior beta grains and alpha lamellae after quenching is not the same for the different test conditions. For test temperatures close or above beta transus (Figure 4, 1050 °C and 950 °C) quenching led to the formation of microstructure composed solely of martensitic alpha prime. Elongated prior beta grains are wider than for tests at lower deformation temperature. At lower test temperatures (Figure 4, 850 °C), deformed alpha lamellae (kinking and bending) can be observed in more little elongated prior beta grains. Shear bands are also observed. A quantitative procedure to assess these microstructures is under development. Scanning Electron Microscope is also used with Electron Back Scattered Diffraction maps (EBSD). Two examples of band contrast maps obtained with EBSD are shown in Figure 5. Further analysis of crystallographic information from EBSD maps will help us to understand the effect of strain, strain rate, temperature and alpha lamellae initial orientation on the microstructure transformations.



*Figure 4 - Transverse sections of alpha + beta cylinders, for different temperature and strain rate compression conditions* 



Figure 5 - Band contrast map obtained with EBSD for two samples

These first experimental data were also used to calculate the rheological parameters (strain rate sensitivity m, the apparent activation energy for deformation Q and a constant A) assuming a Norton-Hoff like constitutive equation:

$$\dot{\varepsilon} = B \cdot \sigma^n \cdot e^{-\frac{Q}{RT}}$$
$$\sigma = A * (\dot{\varepsilon})^m * \exp\left(\frac{mQ}{RT}\right)$$

With A, B material constants, m=1/n the strain rate sensitivity parameter and Q the apparent activation energy in J/mol. In literature, m varies from 0.19 to 0.24 for strengthening state (epsilon < 0.1) and from 0.13 to 0.19 for stationary state [12]. Q varies from 380 to 750 kJ/mol for alpha + beta domain and from 130 to 270 kJ/mol for pure beta. Our first calculations from experimental data are reported in Table 2. The m parameter is estimated about 0.16, and Q is within the range 480-550 kJ/mol, in agreement with literature.

Table 2 - Rheological parameters of studied TA6V

| 3   | Α                      | m     | Q (J/mol)             |
|-----|------------------------|-------|-----------------------|
| 0.1 | 2.13 x10 <sup>-2</sup> | 0.156 | 5.48 x10 <sup>5</sup> |
| 0.8 | 4.06 x10 <sup>-2</sup> | 0.159 | $4.81 \text{ x} 10^5$ |

Maps of m depending on strain rate and temperature, for 0.1 and 0.8 strain are presented in Figure 6 and Figure 7 respectively. The m parameter seems to depend mostly on temperature, and increases with temperature. This may be associated to the change of the fraction of beta phase with temperature: the high values of m would be associated with pure beta phase, and the decrease with temperature would be associated to a progressive increase of alpha phase below 1000°C. Furthermore the softening observed at low temperature results in a dependance of m, A, and Q with strain. However to respect the elastoplastic formulation of the Norton-Hoff constitutive equations, constant average values of A m and Q will be chosen for future finite element calculations.



Figure 6 - m values with strain rate and temperature for 0.1 strain



Figure 7 - m values with strain rate and temperature for 0.8 strain

This results, correlated with literature data and completed with the calculation of the rheological parameters in the other direction of the billet (assumed to be axisymmetric), will feed an upcoming finite element simulation of the uniaxial compression test.

#### 3.2 Preliminary results obtained on the Gleeble MaxStrain tests

Here are the results of simple pre-tests realized on MaxStrain module. Figure 9 shows two deformation steps with a holding time between. Measured temperature, with a welded thermocouple on the surface of the MaxStrain specimen set as seen on Figure 8, shows temperature drop during deformation. Moreover, a temperature gradient exists between the surface and the core of the sample.



*Figure 8 - MaxStrain specimen after deformation, with the thermocouple set close to the deformation zone* 



*Figure 9 - Plastic strain and temperature versus time for a hot multiaxial MaxStrain test on carbon steel* 

Figure 9 illustrates the evolution of plastic strain with time during a cross-forging sequence. The first step of the sequence is associated to a lower strain, because the sample has a square section. After the first hit, the section becomes rectangular due to the Poisson effect, and the strain level is larger during the next hits. The red curve on Figure 9 shows the decrease of temperature recorded by a secondary thermocouple located on the surface to be forged. The temperature falls to 400°C at the extreme surface due to the contact with cold anvils. Then when the anvils are retracted, the surface heats again due to the Joule heating system of the Gleeble.

The rheological parameters calculated in 3.1 will feed a Finite Element Model of the MaxStrain test, in order to identify with great accuracy the thermomechanical gradients involved.

#### 4 Conclusion

This paper is a teaser for upcoming original experimental procedure to make better use of thermomechanical gradients involved in Gleeble MaxStrain tests and to come closer to industrial multiaxial hot forging conditions.

## **Bibliography**

- [1] X. G. Fan *et al.*, « Acceleration of globularization during interrupted compression of a two-phase titanium alloy », *Mater. Sci. Eng. A*, vol. 720, p. 214-224, mars 2018.
- [2] S. L. Semiatin, S. L. Knisley, P. N. Fagin, D. R. Barker, et F. Zhang, « Microstructure evolution during alpha-beta heat treatment of Ti-6Al-4V », *Metall. Mater. Trans. A*, vol. 34, nº 10, p. 2377-2386, oct. 2003.
- [3] T. R. Bieler et S. . Semiatin, « The origins of heterogeneous deformation during primary hot working of Ti–6Al–4V », *Int. J. Plast.*, vol. 18, nº 9, p. 1165-1189, sept. 2002.
- [4] M. Saby, E. Massoni, et N. Bozzolo, « A metallurgical approach to individually assess the rheology of alpha and beta phases of Ti–6Al–4V in the two-phase domain », *Mater. Charact.*, vol. 89, p. 88-92, mars 2014.
- [5] R. Castro et L. Seraphin, « Contribution à l'étude métallographique et structurale de l'alliage de titane ta6v », *Mém. Sci. Rev. Métallurgique*, vol. 12, p. 1025–1058, 1966.
- [6] F. Montheillet et C. Desrayaud, « Essais rhéologiques à chaud », p. 20, 2009.
- [7] J. J. Jonas, R. A. Holt, et C. E. Coleman, « Plastic stability in tension and compression », Acta Metall., vol. 24, nº 10, p. 911-918, oct. 1976.
- [8] S. L. Semiatin, F. Montheillet, G. Shen, et J. J. Jonas, « Self-consistent modeling of the flow behavior of wrought alpha/beta titanium alloys under isothermal and nonisothermal hot-working conditions », *Metall. Mater. Trans. A*, vol. 33, nº 8, p. 2719-2727, août 2002.
- [9] S. L. Semiatin et G. D. Lahoti, « Deformation and unstable flow in hot forging of Ti-6Ai-2Sn-4Zr-2Mo-0.1Si », *Metall. Trans. A*, vol. 12, nº 10, p. 1705-1717, oct. 1981.
- [10] S. Roy, S. Suwas, «The influence of temperature and strain rate on the deformation response and microstructural evolution during hot compression of a titanium alloy Ti–6Al–4V–0.1B», Journal of Alloys and Compounds, vol. 548, p. 110-125, 2013
- [11] J. Xu, W. Zeng, D. Zhou, H. Ma, S. He, W. Chen, «Analysis of flow softening during hot deformation of Ti-17 alloy with the lamellar structure», Journal of Alloys and Compounds, vol. 767, p. 285-292, 2018

[12] Hétérogénéités de déformation au cours du forgeage d'aubes en alliage de titane TA6V, thèse Aurélie Colin, Mines Saint-Etienne