HOT DEFORMATION OF THE Ti-6Al-4V ALLOY PREFORM OBTAINED BY SPARK PLASMA SINTERING OF POWDERS

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Abstract. *Titanium and its alloys are well-known as hard-to-be sintered due to the stability of their oxides. Thus, high temperatures and a long time are required to obtain a bulk material from powders. Spark Plasma Sintering (SPS) is a relative new sintering technique able to sinter these alloys in a shorter time and lower temperature allowing to achieve tailored microstructures. Therefore, SPS was elected to sinter the Ti-6Al-4V powder in a bulk preform. To achieve an lamellar alpha/beta microstructure free of pores the sintering cycle was conducted at isothermal 950°C (under beta-transus). Hot deformation tests were carried out at three levels of strain rate (0,01 s^{-1}, 0,1 s^{-1}, 1 s^{-1}) and at three temperatures (850°C, 950°C, 1050°C) in order to cover typical hot forging conditions of this alloy. The hot compressibility behavior was analyzed and metallographic examinations were performed to assess the microstructure changes. The experiments were carried out in a horizontal quenching and deformation dilatometer in vacuum atmosphere. As result, an alpha globular microstructure was achieved when deformation was performed at lower strain rate and intermediate temperature. Additionally, some instability phenomena of the plastic flow were observed at higher strain rate.*

Keywords*: Spark Plasma Sintering, Ti-6Al-4V, Hot Deformation.*

1. INTRODUCTION

Among the alpha/beta alloys, Ti-6Al-4V is by far the most popular titanium alloy. Recently, Leyens and Peters (2003) projected that more than 50% of all Ti alloys in use nowadays are of this composition. Beyond the well-known properties, which are mainly high specific strength and high corrosion resistance, the possibility of modifying the microstructure by thermomechanical treatment to obtain a preferred mechanical property regards the massive application of the this alloy. As demonstrated by Semiatin *et al* (1999), Shell *et al* (1999), Seshacharyulu *et al* (2002), Ding *et al* (2002) and Bruschi *et at* (2004), in fact, by choosing suitable parameters of the thermomechanical treatment, such as temperature, amount of strain, strain rate, cooling rate and primarily the starting microstructure, many different distribution and features of the alpha/beta phases can be attained and consequently different mechanical properties can be matched.

Lütjering and Williams (2003) stated that forging is the principal shaping process used for making titanium alloy components. When compared with machining, the forging process presents the benefit of not requiring metal removal and therefore, is in the most cases cheaper than. In the same way, common problems found in casting, like voids, inclusions and segregation must be avoided. Anyway, the forging process is based on the transformation of a simple geometry in a fairly complex shape in combination with a high microstructural control. At this point, the starting microstructure obtained by powder metallurgy of titanium may be a key of productive and cheaper thermomechanical treatment.

German and Bose (1996) have worked with powder metallurgy of titanium using a process usually called near-net-shape (NNS). Nevertheless, in such process a long time and high temperature are needed to reach fully dense materials and consequently, typically these materials hardly attained the mechanical standard requirements. On the other hand, Spark Plasma Sintering (SPS) is a sintering process which makes use of pulsed DC electric current and among other features is the unique sintering technique that allows a sintering soaking time in about minutes, preserving the fine microstructure existing in the powder. Near full density sintering of the titanium grade 1 and 3 were carried out by means of SPS by Zadra *et al* (2008). Mechanical properties of the materials fully matched the ASTM standards as well as the amount of the interstitial elements were found in agreement of them relative standards.

In this study the Ti-6Al-4V alloy was produced by SPS at temperature under beta transus to achieve an alpha/beta lamellar microstructure. Subsequently, hot deformation tests were conducted in order to assess the response of the material to the hot working. Furthermore, the achievement of a globular alpha phase is the main aim of the thermomechanical treatment.

2. MATERIALS AND METHODS

Commercial grade Ti-6Al-4V gas atomized powder supplied by TLS was used to obtain the bulk preform. The powder particles has a spherical shape and size under 45 µm. The chemical composition of the powder, certificated by the supplier, is reported in Tab. 1. A SPS device Sumitomo Dr. Sint. 1050, using graphite dies, was used to sinter a disk of 15 mm height and 30 mm diameter per cycle. The sintering cycle was conducted as following: heating rate of

100°C/min from room temperature to 900°C, then reduced to 50°C/min from 900 to 950°C; lastly 5 minutes at isothermal holding at 950°C, followed by free cooling within the die. A compaction pressure of 60 MPa was applied at 600°C and kept up to the end of the cooling. Vacuum was imposed, but atmosphere pressure varied up to 15 Pa during the sintering cycle.. From each disk were electro-eroded (electrical discharge machining) 10 compression specimens of 10 mm height and 5 mm diameter. The axis of the specimen coincides with uniaxial compaction pressure axis on the disk built-up. A horizontal quenching and deformation dilatometer Bahr 805A/D was used to perform the isothermal hot deformation tests. A flat disk 0,1 mm thick and 8 mm of diameter made of 99,95% molybdenum metal basis was placed on the contact zone between the specimen and the punch to improve the contact conditions. The temperature of the specimens were monitored using a Platinum:Platinum-Rhodium (Pt:Pt-Rh) thermocouple. All tests were conducted in high vacuum. The specimens were maintained about five minutes at isothermal temperature before the starting of the tests. Three temperatures were selected to perform the compression: 850°C, 950°C and 1050°C; and three constant strain rates: 0,01 s^{-1}, 0,1 s^{-1} and 1 s^{-1}, were investigated. For each condition three tests were carried out. The specimens were deformed to half their initial height to impose a true strain of about 0.8 and were cooled at 25°C/s to room temperature after deformation in order to inhibit further microstructural changes. The deformed specimens were sectioned parallel to the deformation axis and the cut surface was prepared for metallographic examination. To reveal the microstructure Kroll's reagent (2%HF + 4HNO3) was used to etch the prepared surface. Light Optical Microscopy (LOM) was conducted for microstructure examination. Density measurements were carried out by water displacement method (Archimedes' principle).

Table 1. Chemical composition of the Ti-6Al-4V powder.

Chemical Composition	Al	V	C	Fe	O	N	Y	Zn	Mg	Ti
Powder [(1)]	5,91	4,20	0,008	0,032	0,15	0,003	<0,01	<0,002	<0,002	Bal.

[(1)] Suppliers' certification.

3. RESULTS AND DISCUSSIONS

3.1. Starting Microstructures

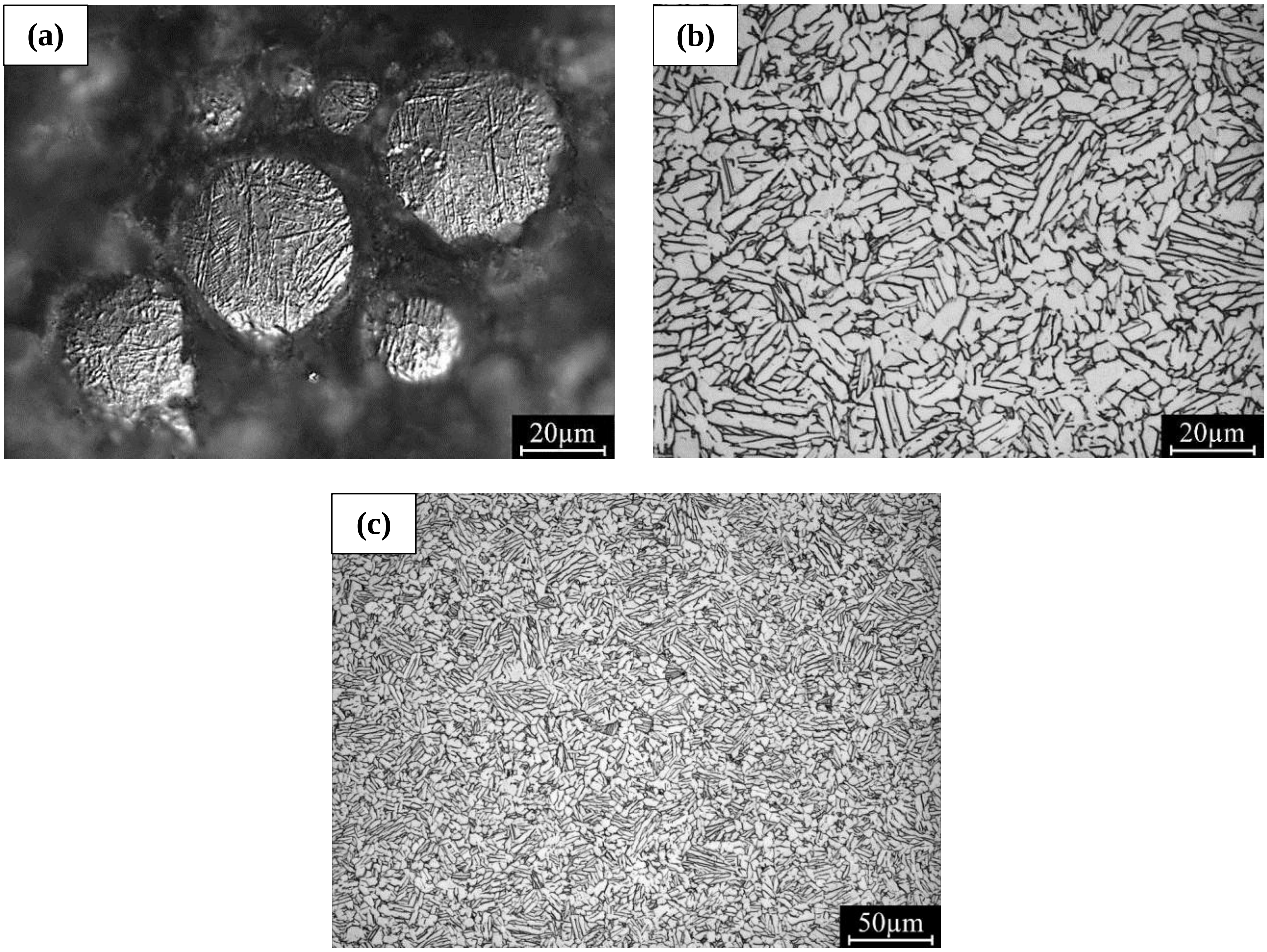

Figure 1. Microstructures of (a) pre-alloyed powder of Ti-6Al-4V (polarized light), (b) the Ti-6Al-4V alloy used in this investigation and (c) the Ti-6Al-4V alloy used in this investigation at lower magnification.

The microstructures of the powder and the preform are shown in Fig. 1. Since the powder was obtained by atomization, the rapid cooling rate intrinsic of this process resulted in a microstructure consisting in a martensitic alpha phase (needle-like α phase).

The oxygen (O_2) and nitrogen (N_2) contents in the sintered samples (0,22% ± 0,01) and (0,037% ± 0,01), respectively, are slightly higher than that contained in the supplier certification (Table 1). Nevertheless, similar higher amounts were also found in a powder sample just before the sintering and therefore they can be considered surface contaminations. Lütjering and Williams (2003) mentioned that at about 0,20% of oxygen, the beta transus temperature of this material is around 995°C, then sintering at 950°C followed by free cooling (medium cooling rate) resulted in an alpha + beta phase microstructure consisting in a thin lamellar alpha colonies and interlamellar beta phase. Additionally, globular alpha revolving the alpha colonies can be seen. A similar microstructure obtained using Hot Isostatic Pressing (HIP) is cited by di Ioro *el at* (2007) as a lamellar alpha grain gathered in colonies plus globular alpha grains. In the material under investigation the grain size of the lamellar alpha colony is about 50 µm or less, such dimension is comparable with the powder particle size (under 45 µm), leading to consider those colonies as the prior powder particle microstructure with tittle changes.

The sintered material attained a density of 4,41 g/cm^3, corresponding to about 99,8% pore-free, confirmed by the metallographic examination.

3.2. Flow Curves

True stress-true strain curves at temperatures under and over beta transus at 1 s^{-1} strain rate are shown in Fig. 2. It is clearly visible the influence of the temperature on the hot working behaviour, decreasing over than five times the flow stress from 850°C to 1050°C.

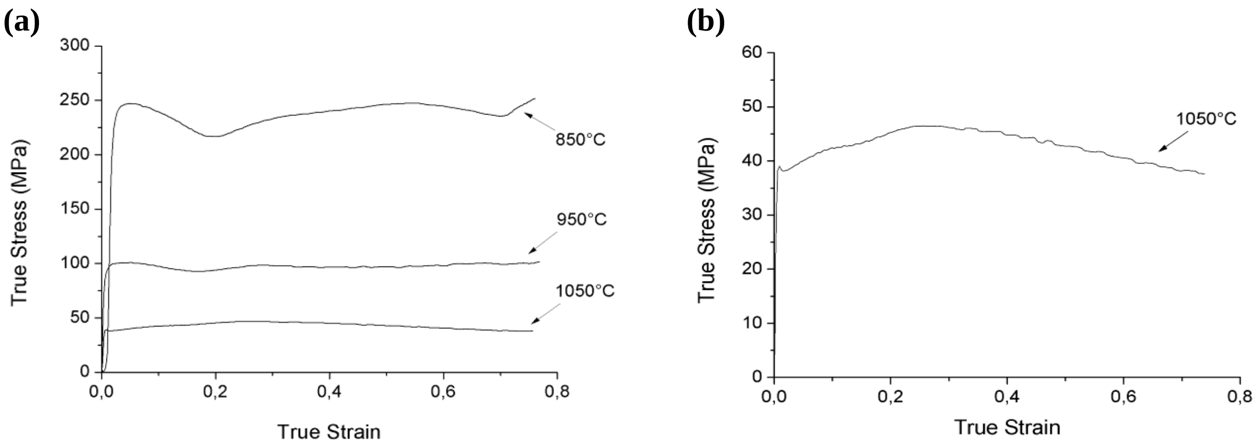

Figure 2. True stress-strain curves of Ti-6Al-4V at (a) different temperatures and strain rate of 1 s^{-1} and, at (b) 1050 °C and 1 s^{-1}, with adjusted scale.

The stress-strain curve recorded at 850°C shows a peak flow stress followed by a softening and later an oscillation. This behaviour can be considered an indication of a localized or unstable plastic flow (Seshacharyulu *et al,* 2002). In fact, microstructural examination confirmed the presence of marked shearbands at 45°, as can be seen in the Fig. 3. Hot deformation at 950°C is still carried out into the alpha/beta field, displaying a stress-strain curve tendencially similar to that performed at 850°C, but at lower level of stress. Furthermore, a clear steady-state behaviour after a slight stress oscillation is attained.

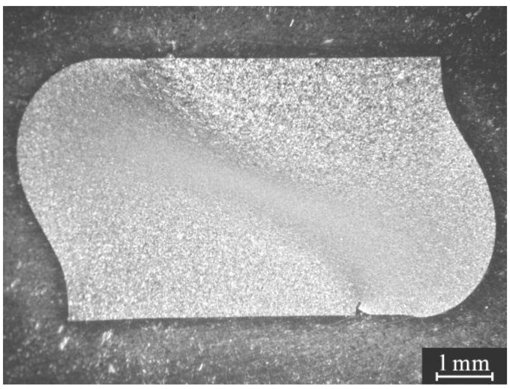

Figure 3. Macroscopic shearband at 45° on the specimen deformed at 850°C and 1 s^{-1}

The curve at 1050°C shows a strain hardening behaviour followed by a softening. Such occurrence is shown with adjusted scale in Fig. 2b. Likely, taking account the temperature over beta transus, the explanation can bethat a given amount of strain have to be attained to take place adiabatic heating, promoting thus the observed softening.

The stress-strain curves recorded at 950 °C at three different strain rates are shown in Fig. 4. It is noted that the stress increases with strain rate. As discussed before, the curve at 1 s^{-1} shows a slight oscillation in the flow stress, but microstructural evidence of unstable plastic flow was not found. The softening phenomenon observed in the curve at 0,1 s^{-1} may be attributed to adiabatic heating, which raises the actual temperature of the sample and also the proportion of the soft beta phase during deformation (Ding *et al*, 2002). Moreover, when the hot deformation was carried out at lower strain rate just a little softening may be observed.

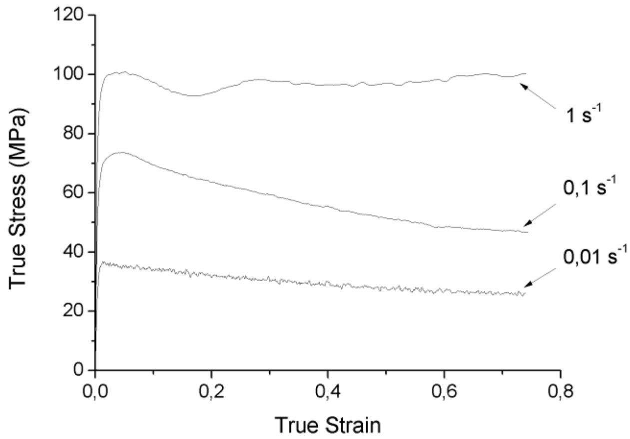

Figure 4. True stress-strain curves of Ti-6Al-4V at different strain rates and same temperature: 950 °C.

3.3. Peak Flow Stresses and Strain Rate Sensitivity (*m*)

From the stress-strain curves the peak flow stress was measured as maximum stress recorded at given temperature and strain rate. Table 2 reports these values.

Table 2. Peak flow stresses (in MPa) for different temperatures and strain rates.

σ (MPa)			
	Strain rate, $\dot{\varepsilon}$ (s^{-1})		
Temperature (°C)	0,01	0,1	1
850			248
950	36	74	101
1050			47

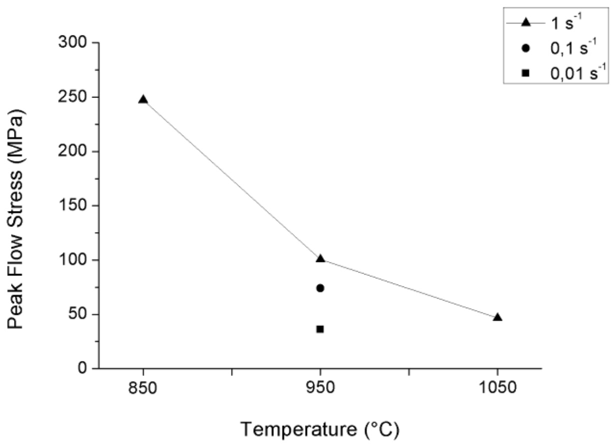

Figure 5. Peak flow stress *vs* deformation temperature.

The relationship between peak flow stress and temperature for different strain rates is shown in Fig. 5. It is clear that increasing the temperature the stress required to yield the material decreases, as before pointed out, but this behavior does not follow a linear trend. At 850°C the amount of alpha phase, which has a Hexagonal Close Packed (HCP) unitary cell, is larger than at 950°C and therefore the material is much harder to be deformed. Similarly, when deformed at 950°C the microstructure consists in majority amount of beta phase (BCC) due to the vicinity of beta transus temperature. Thus, peak flow stress at 950°C is closer to that at 1050°C (full beta phase) than that at 850°C (majority alpha phase).

To support the argument concerning the amount of phases present at different temperature of the hot deformation tests, the isothermal sections of the Ti-rich corner of the ternary Ti-Al-V phase diagram are shown in Fig. 6.

Through the lever rule the amount of the beta phase at 800 °C (Fig. 6a) can be rough estimated in about 14%, while the alpha phase accounts for 86%. In the same way, at 900 °C (Fig. 6b) the amount of the beta phase is about 50%, while the alpha phase accounts for another 50%. Interpolating the two estimated phase extents the amount of beta phase at isothermal 850 °C may be expected in about 32%, whilst the alpha phase would account for 68%. As mentioned before the beta transus temperature for this alloy is about 995 °C, such temperature corresponds to the full beta phase composition. Then, interpolating the estimated amounts of beta and alpha phases at 900 °C and 995 °C, it can be assess that at isothermal 950 °C the amount of beta phase is about 78%, whilst the alpha phase would account for only 22%. Finally, hot deformation tests at isothermal 1050 °C are performed at completely beta phase composition.

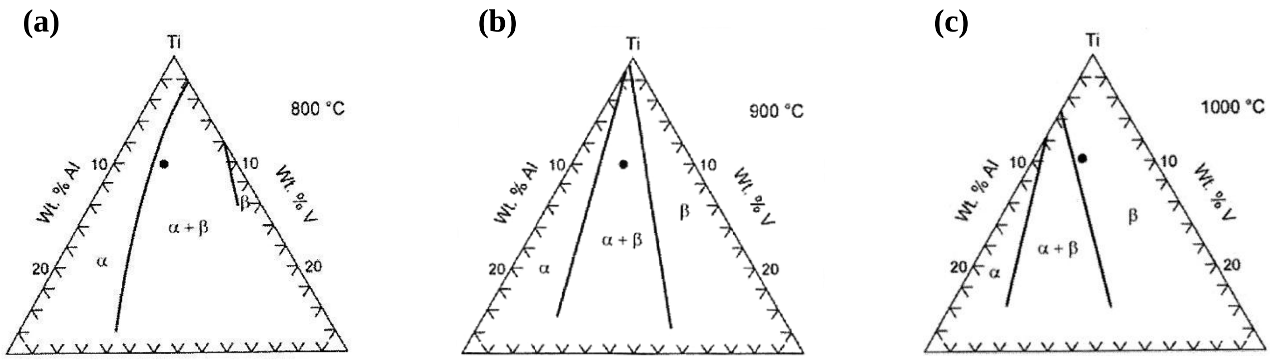

Figure 6. Isothermal section at (a) 800 °C, (b) 900 °C and (c) 1000 °C through the Ti-rich corner of the ternary Ti-Al-V phase diagram (solid point: the Ti-6Al-4V alloy) [Lütjering and J. C. Williams (2003)]

To determine the strain rate sensitivity (*m*) of the material at 950 °C the Eq. (1) has to be. It can be calculated as the slope of the curve when the peak flow stress versus strain rate is plotted in log(σ)-log($\dot{\varepsilon}$). Such graph is shown in Fig. 7.

$$m = f(\dot{\varepsilon}, T) = \frac{\partial log\sigma}{\partial log\dot{\varepsilon}} \quad (1)$$

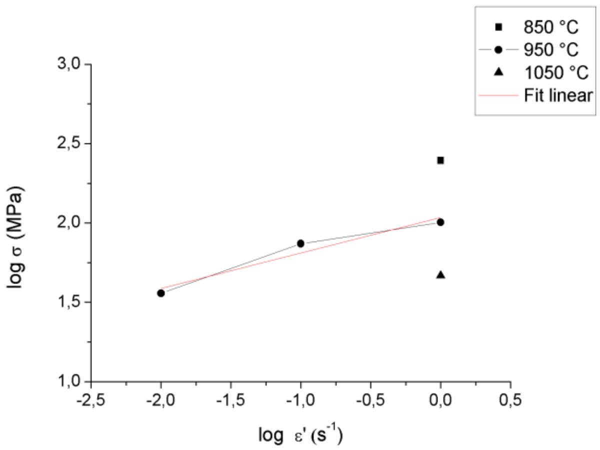

Figure 7. *log* peak flow stress *vs log* strain rate. The slope of these curves is the "*m".*

A linear fitting with poor correlation factor (R-square= 0,91) is obtained. The strain rate sensitivity (*m*) of the material was calculated as being 0,22. Semiatin et al (1999) reports this value of *"m"* at about 0.2 of true strain, however the starting microstructure is completely different.

3.4. Microstructures After Hot Deformation

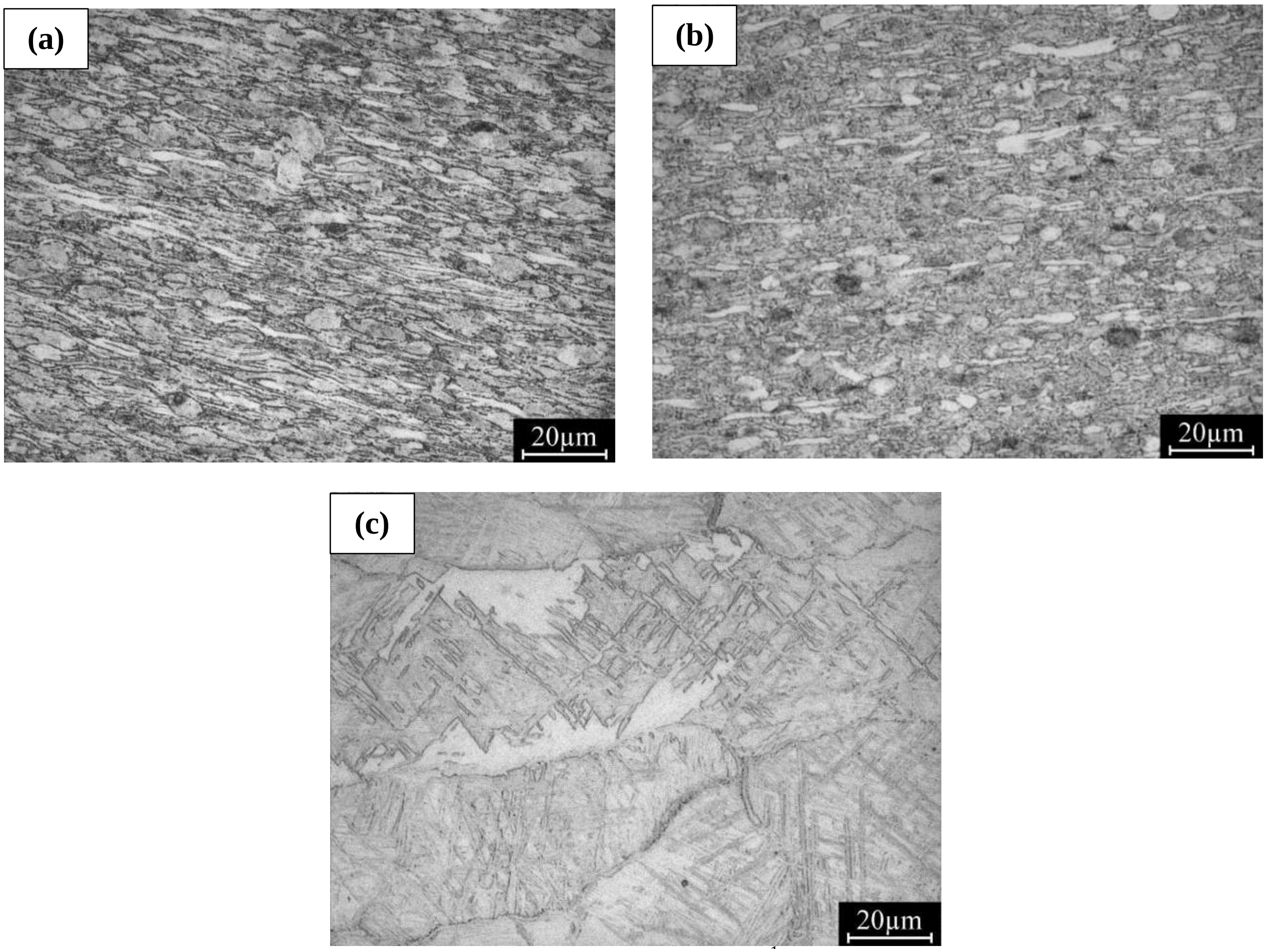

Figure 8. Microstructures of Ti-6Al-4V specimens deformed at 1 s^{-1} of strain rate and different temperatures: (a) 850 °C, (b) 950 °C and (c) 1050 °C;

The microstructures of the specimens deformed at 1 s^{-1} strain rate (flow curves in Fig. 2a) are shown in Fig. 8. The shearbands shown in Fig. 3 occurred in the specimen at 850 °C are shown in Fig. 8(a). No cracks or voids are identified. In Fig. 8(b) the microstructure resultant from the hot working at high temperature into the alpha/beta field shows some elongated grains of prior alpha phase in a matrix of fine transformed beta, not well resolved at LOM. Finally, elongated grains of alpha martensite in a beta matrix plus some alpha at prior beta grain boundaries is shown in Fig. 8(c), where grain grow is evident.

The microstructures of the specimens hot deformed at 950 °C at 0,1 s^{-1} and 0,01 s^{-1} are shown in Fig. 9(a) and (b), respectively. At intermediate strain rate (Fig. 9(a)) the presence of alpha lamellae can be still seen, but it seems to have been sheared. While at lower strain rate (Fig. 9(b)) a full alpha globular phase was attained.

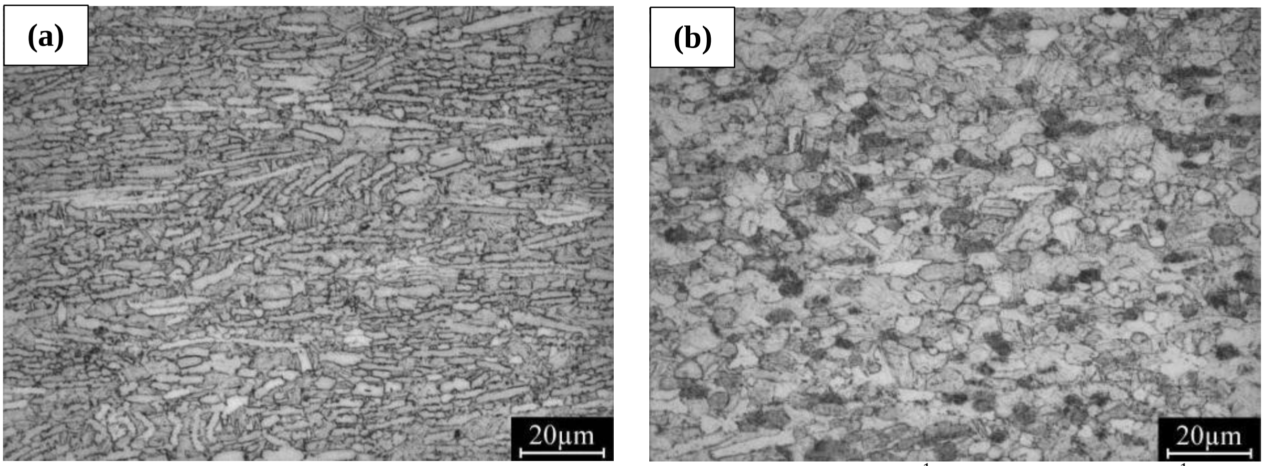

Figure 9. Microstructure of Ti-6Al-4V specimens deformed at (a) 950 °C/ 0,1 s^{-1} and (b) 950 °C/ 0,01 s^{-1}.

4. CONCLUSIONS

Sintering of a gas atomized Ti-6Al-4V pre-alloyed powder by means of Spark Plasma Sintering was carried out to produce preforms having a lamellar alpha phase microstructure. Such preforms were subjected to hot deformation in a dilatometer at three temperatures and three strain rates, at same 0.8 true strain. The results summarily as following:

(i) Temperature is more effective parameter than strain-rate.

(ii) Both temperature and strain rate have to be appropriate for attaining a globular alpha.

(iii) From alpha lamellar microstructure of Ti-6Al-4V preform obtained by SPS was possible to achieve a globular alpha phase by hot deformation at high temperature into alpha/beta field and lower strain rate.

5. REFERENCES

Bruschi, S., Poggio, S., Quadrini, F., Tata, M.E., 2004. *Workability of Ti–6Al–4V alloy at high temperatures and strain rates.* Materials Letters 58, 3622– 3629.

Ding, R., Guo, Z.X., Wilson, A., 2002. *Microstructural evolution of a Ti-6Al-4V alloy during thermomechanical processing*. Materials Science and Engineering A327, 233-245.

di Ioro, S., Briottet, L., Rauch, E.F., Guichard, D., 2007. *Plastic deformation, damage and rupture of PM Ti–6Al–4V at 20 K under monotonic loading*. Acta Materialia 55, 105-118.

German, R.M. and Bose, A., 1996. *Injection Moulding of Metals and Ceramics*. Metal Powder Industries Federation, Princeton, NJ.

Leyens, C. and Peters, M. Edited by, 2003. *"Titanium and Titanium Alloy".* Wiley-VCH.

Lütjering, G. and Williams, J.C., 2003. "*Titanium"*. Springer.

Semiatin, S.L., Seetharaman, V., Weiss, I., 1999. *Flow behavior and globularization kinetics during hot working of Ti–6Al–4V with a colony alpha microstructure*. Materials Science and Engineering A263, 257–271.

Seshacharyulu, T., Medeiros, S.C., Frazier, W.G., Prasad, Y.V.R.K., 2002. *Microstructural mechanisms during hot working of commercial grade Ti-6Al-4V with lamellar starting structure.* Materials Science and Engineering A325, 112-125.

Shell, E.B. and Semiatin, S.L., 1999. *Effect of Initial Microstructure on Plastic Flow and Dynamic Globularization during Hot Working of Ti-6Al-4V. Metallurgical and Materials Transactions A.* Vol. 30A, 3219-3229.

Zadra, M., Casari, F., Girardini, L., Molinari, A., 2008. *Microstructure and mechanical properties of cp-titanium produced by spark plasma sintering.* Powder Metallurgy, Vol. 51, No 1, 59-65.

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