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EVIDENCE OF THE SEMI-SOLID FORMATION IN THE MEDICAL GRADE TI6AL4V ALLOY USING INDUCTION HEATING

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Abstract. *One alternative for processing cost reduction with simultaneous improvement of the mechanical properties of the Medical Ti6Al4V alloy is to get its semi-solid feedstock with a non-dendritic microstructure for further processing. The purpose of the present work is to evaluate the possibility of obtaining a semi-solid Ti6Al4V alloy by heating it up from the room temperature to the range temperature between the lines solidus and liquidus, using induction heating. The Ti6Al4V billets underwent heat treatment and quenching for semi-solid formation using a designed device and specific time pulsed profile. The billet temperature reached 1630 °C, and after the cooling rate of 54 °C/s, some samples formed a globular phase characteristic of the semi-solid alloy. This study shows that it is possible to get a semi-solid microstructure of this alloy starting from its solid state.*

Key Words: *Ti6Al4V ELI, Semi-solid State, Induction Heating, Semi-solid Ti Alloy*

1. INTRODUCTION

Titanium alloys are widely used in various fields of engineering and medicine due to their high tensile strength, biocompatibility and corrosion resistance [1]. The Medical Ti6Al4V alloy is the biomaterial of choice for many orthopedic applications as implants and prostheses, manufactured by conventional metallurgical processes and posterior heat treatments for stress relief [2]. Hot forging, for instance, is required for the Ti6Al4V alloy that involves heating of the die to usually about 200 °C, and of the workpiece between 900 °C and 1000 °C to avoid residual stress and defects in the product. Moreover, the processing must be fast to prevent cooling of the workpiece with a consequently increasing forging force and risk of damaging of the die [3]. Because of the high production costs of the Ti6Al4V alloy and the difficulties to produce a material with

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predetermined service properties [4], new processing methods have been suggested and studied. The conventional Ti6Al4V has the Young modulus between 105 – 120 GPa, elongation about 8% and hardness of about 399 HV [5]. The microstructure, and consequently, the mechanical properties of Ti alloys are very sensitive to the thermo-mechanical processing parameters. At room temperature the Ti6Al4V is a two-phase (α + β) alloy, and although the body-centered cubic (BCC) β phase is desirable to aid deformation of the alloy (Young modulus about 73 GP, elongation between 45% and 95%, and 336 HV[5]) the β grains are not abundant (< 10%). Therefore, the plastic strain is limited by the slip systems of the hexagonal close-packed (HCP) α phase and the mechanical properties of the alloy reflect the α phase properties. At high temperatures, however, especially above 900 °C, the amount of β phase increases abruptly [6]. The high temperature phase distribution of the Ti6Al4V alloy is, therefore, interesting to improve its formability. One alternative for processing cost reduction with simultaneous improvement of the mechanical properties of metal alloys is developed by Flemings and co-workers at the MIT in the 1970s called semi-solid metal forming (SSF). The process was originally developed using lightweight aluminum alloys for further industrial applications and, since then, thixoforming and rheocasting became an alternative to the traditional casting. The main purpose of the process is to get a semi-solid alloy feedstock with non-dendritic microstructure [7]. Induction melting process has been widely used as a method for melting high-purity metals. The heating occurs via electromagnetic field that induces an eddy current resulting in the Joule heating of the metal. The electromagnetic field is imposed by a coil [8]. Until now, the induction heating has been suggested for semi-solid preparation starting from the melted metal and only for aluminum alloys. However, for application of this technology in Ti alloys, the melting temperature of about 1670 ^{o}C [9] is a limiting factor. The purpose of the present work is to evaluate the possibility of obtaining a semi-solid Ti6Al4V alloy by heating it up from the room temperature to the range of temperature between the lines *solidus* and *liquidus* using induction heating.

2. MATERIALS AND METHOD

To evaluate the possibility of obtaining a semi-solid titanium alloy, the alloy with the following mass composition was used: 89.58% Ti; 6.14% Al; 4.00% V; 0.16% Fe; 0.11% O; 0.003% C; 0.005% N; 0.002% H. The raw material was provided in an extruded bar with 9.53 mm in diameter. Six billets were cut from the center of the bar, each one of 40 mm in length, using a metallographic precision cutter. Two billets were used for the measurement calibration and the electromagnetic induction equipment and discarded, another two were used as control specimens and for evaluation of the microstructure of the alloy without treatment, and the remaining two underwent a semi-solid treatment. To apply the electromagnetic induction to the billets a specific device was designed and built that allows the correct positioning of the billets within the induction coil without touching them. The device consists of an epoxy-painted stand made of carbon steel and the rubber feet that permit height and level adjustment to align the billet with the induction coil. Since, at high temperatures, titanium shows a close affinity to oxygen, the device contains a chamber for an internal atmosphere control with an injection of argon or some other inert gas. The chamber is constructed of 5 mm thick plates of an epoxy resin and fiberglass composite, non-metallic materials, to avoid the magnetic field to influence them. To allow for billet visualization, the upper part of the chamber has a 6 mm thick tempered glass window fixed and sealed with high temperature silicone. At the bottom of the chamber there is a trapdoor to permit the billet to fall quickly into a reservoir containing cold water as soon as the desirable surface temperature is reached. This procedure is necessary in order to freeze the metastable microstructure. The billets are positioned in the chamber as shown in Fig. 1 in order to obtain semi-solid microstructure. The reservoir beneath the chamber is loaded with 40 l of cool water (5 ± 1 °C) measured with an alcohol thermometer column. For the billet temperature measurement a laser pistol Raytek® model Raynger 3i Plus is used, with 1 ^{o}C accuracy and temperature range from 700 to 3000 ^{o}C calibrated with a platinum thermocouple and a temperature controller Minipa® model MT-510, suitable for direct reading of a specimen. The chamber is closed and argon is allowed to flow through the tube connecting the chamber to the gas cylinder. The programmable medium frequency induction heating JMMF®, 5 kW maximum power, is turned on and the billet temperature is monitored uninterruptedly with the laser pistol positioned vertically at 300 mm far from the upper surface of the billet and set to the center of it. The sequence of the heating treatment for the samples from billet 1 and billet 2 are presented in Table 1.

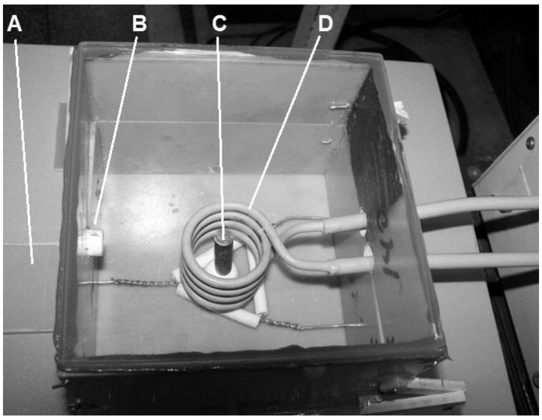

Fig. 1 Chamber sealed with silicone: (A) trapdoor; (B) argon ingate; (C) billet and (D) coil

The billet temperature rises rapidly to 1630 ^{o}C which is maintained for 3 s and the trapdoor is opened releasing the billet into the cold water reservoir and freezing the microstructure. The two billets prepared in this way together with the two control billets undergo the metallographic routine to allow the microstructure evaluation in the optical microscope: cutting with a Minitom precision cutter Struers® with diamond disc; embedding in the cold resin to avoid microstructural changes; grinding sequentially with paper grit 220, 400, 600 and 1200; polishing with soft cloth impregnated with alumina particles of 0.3 µm in diameter; and etching with the Keller's reagent. The microstructural evaluation is performed in an optical microscope (OM) Olimpus® model CX31RBSFA.

Table 1 Heating treatment sequence of the samples from billet 1 and billet 2

Machine power (%)	Pulse (on/off)	Time (s)	Initial temperature (^oC)	Final temperature (^oC)
75.0	Continue	90	32	1300
50.0	Intermittent 1s/1s	30	1300	1630
50.0	Intermittent 1s/2s	6	1630	1630

3. RESULTS

The parts cut transversally and longitudinally to the billet axis and embedded in the resin of the first billet are shown in Fig. 2. The OM images from the Ti alloy as supplied can be seen in Fig. 3.

a) b)

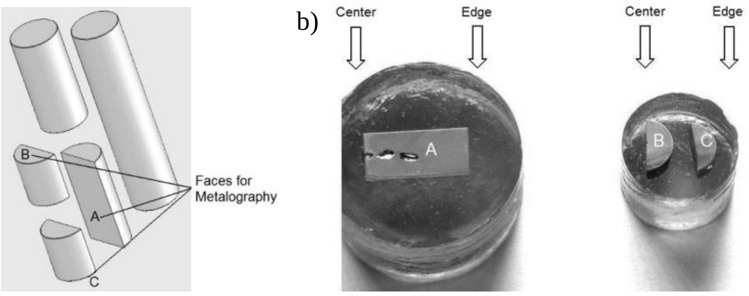

Fig. 2 (a) parts for OM evaluation showing the faces analyzed nominated as A1, B1 and C1 for the billet 1 and A2, B2 and C2 for the billet 2; (b) Left: sample longitudinal to the billet axis; right: sample transversal to the billet axis.

It is possible to see the beginning of the melting process revealing, therefore, that the temperature inside the part achieves the melting point of the Ti alloy (1670 ^oC) while the temperature of the billet surface is 1630 ^oC, measured by the laser pistol.

a) b)

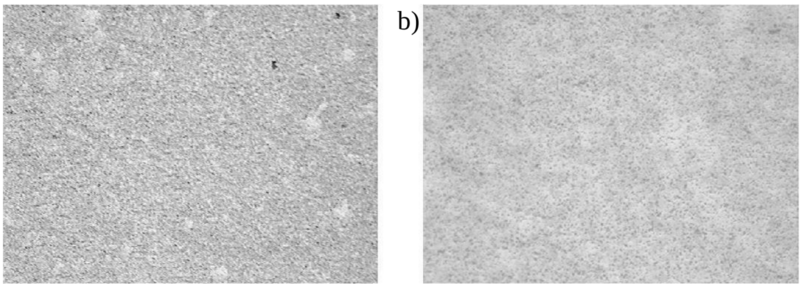

Fig. 3 OM images from the billets as supplied. (a) image took from field B (Figure 2a) close to the center of the sample,100x and (b) 400x.

One can see that the microstructure is relatively homogeneous in 400x magnification. Fig. 4 presents the OM images from the marked fields. From the microstructure shown in Fig. 4 it is possible to suggest that with the heat treatment employed, a different microstructure from the as supplied one is formed, indicating that freezing of the metastable microstructure has been performed. The cooling rate is, proximately, 54 ^{o}C/s. The darker area inside the globular formations is characteristic for the β phase of the Ti6Al4V alloy [10]. Fig. 5 shows the microstructure of the first billet, cut longitudinal to its axis.

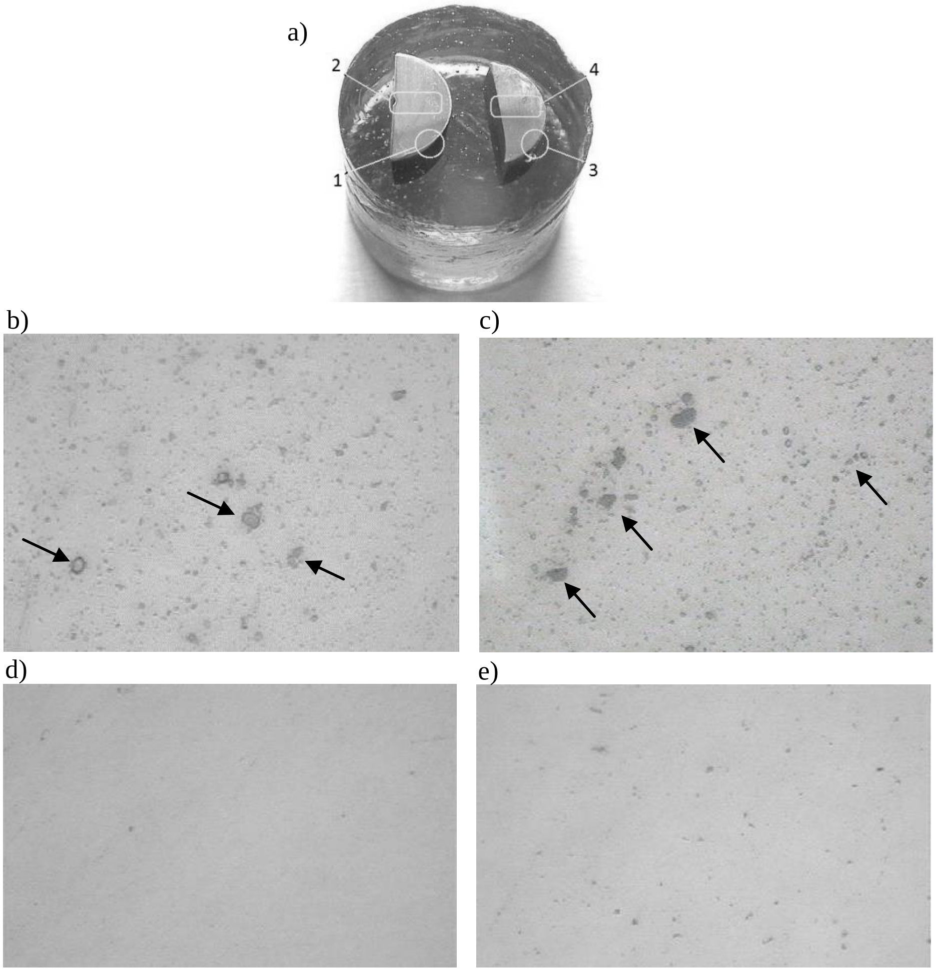

Fig. 4 (a) Samples B1 and C1 from the billet 1 showing the fields analyzed, 400x; (b) field 1, detail of globular formations; (c) field 2, detail of globular formations; (d) field 3 and (e) field 4

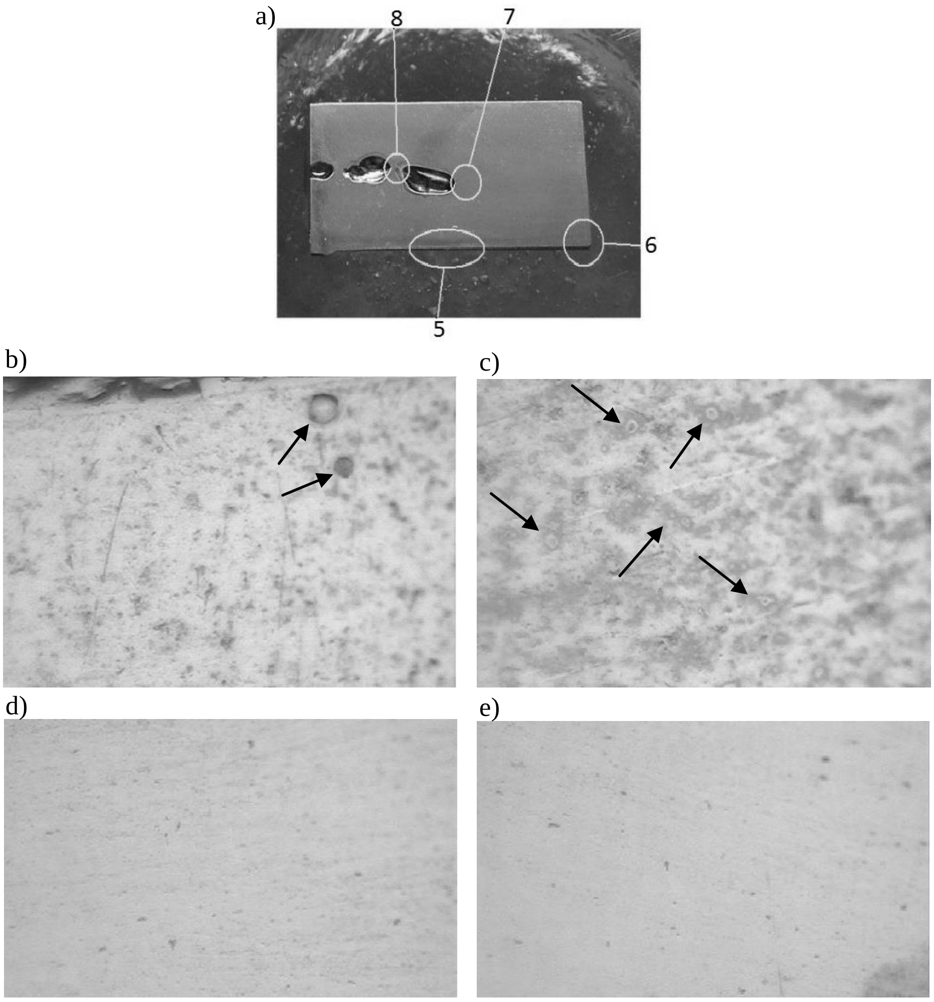

Fig. 5 (a) Sample A1 from billet 1 showing the fields analyzed; (b) field 5, detail of globular formations, 400x; (c) field 6, detail of globular formations, 400x; (d) and (e) field 7 and 8, respectively with no apparent globular formation, 400x

Fig. 6 presents the OM images from the marked fields for the second billet. It shows that, in these fields of samples B2 and C2, the globularization of the alloy fails. In Fig. 6e it is possible to notice a fully lamellar coarsed microstructure, one of the three microstructures found in the annealed Ti6Al4V alloy (fully lamellar, fully equiaxed, and bimodal) [11]. Probably this field has reached a temperature above 995 ^{o}C (β transus) but below the semi-solid range. Therefore, an extensive grain growth occurs and large grains are formed which have transformed into lamellar $\alpha + \beta$ upon cooling [12].

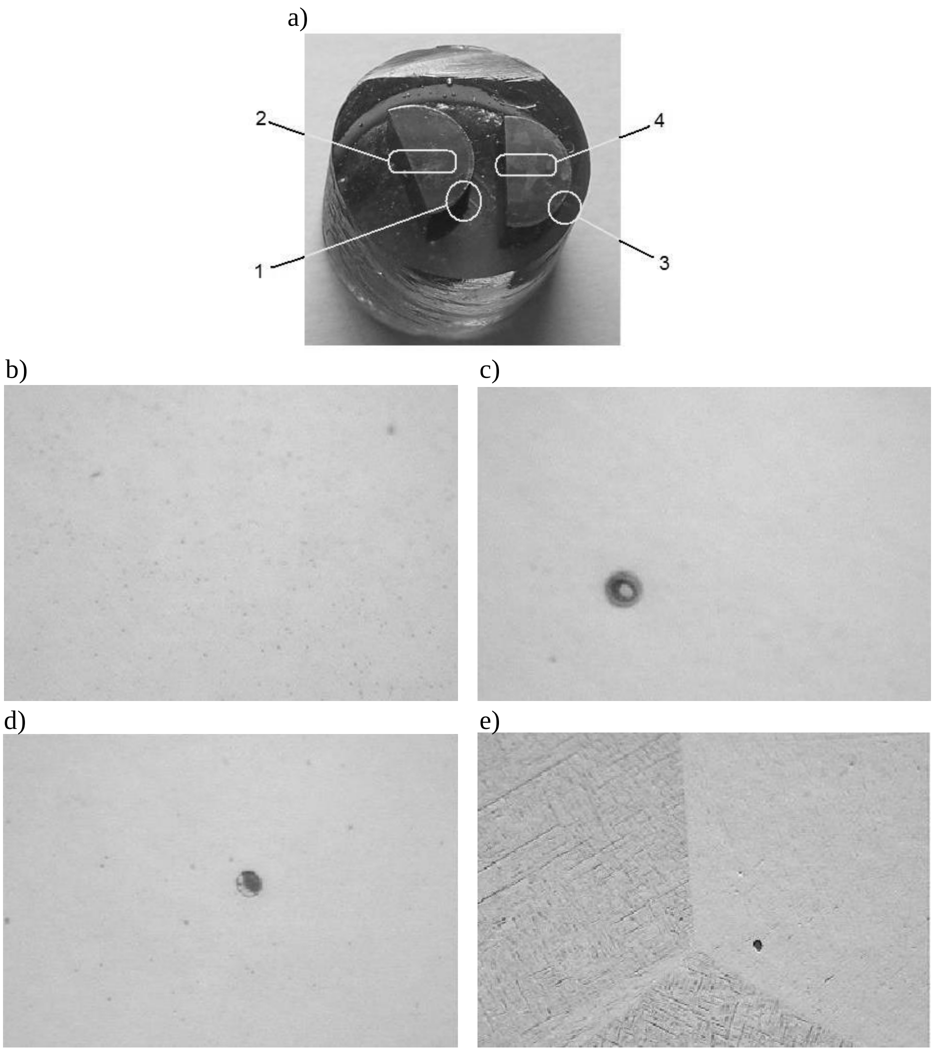

Fig. 6 (a) Samples B2 and C2 from the billet 2 showing the fields analyzed: on the left the area taken from the center of the billet and on the right took from its bottom; (b) field 1, apparently with no globular formations, 400x; (c) and (d) field 2 and 3, respectively, with one globular formation with a phase inside, 400x; (e) field 4, detail of grain boundaries, 400x

Fig. 7 displays the OM images from the marked fields for the second billet.

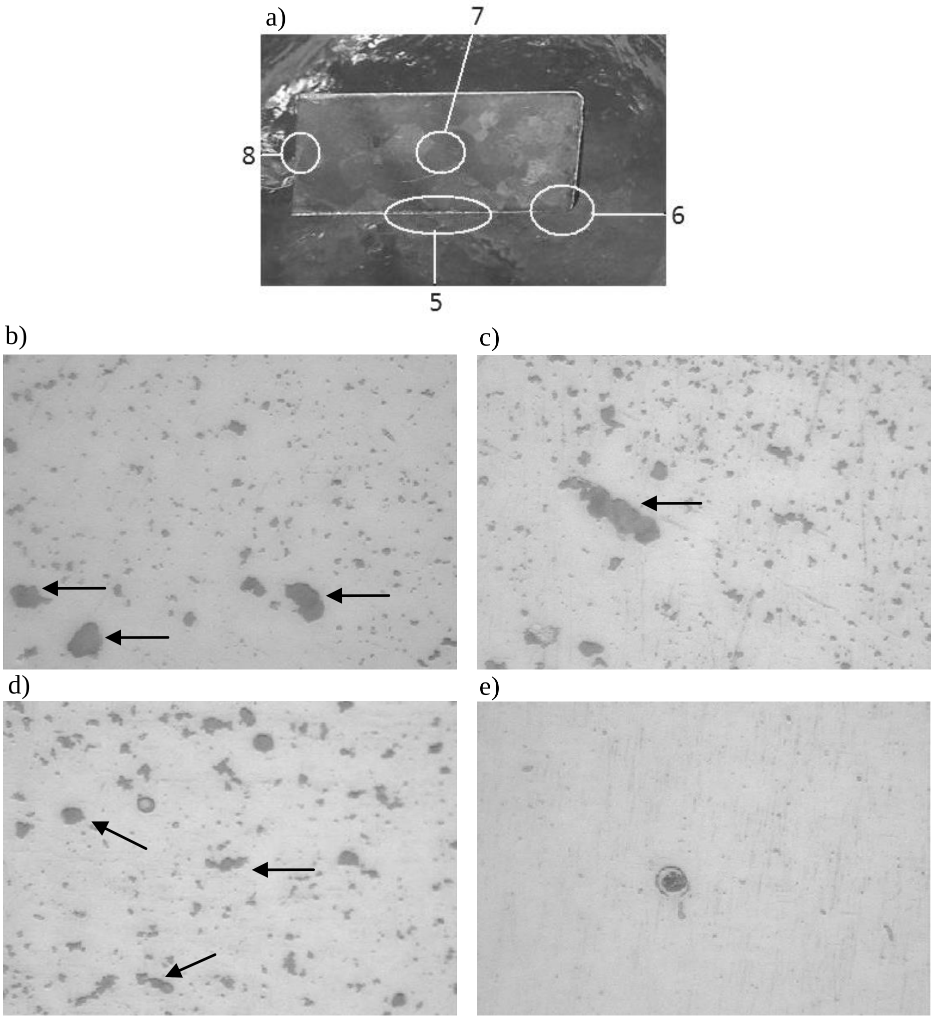

Fig. 7 (a) Sample A2 from billet 2 showing the fields analyzed: on the left side the center of the billet and the right side its bottom; (b), (c) and (d) field 5, 6 and 7, respectively, showing globular formations, some in a visible coalescence phase (arrows), 400x; (e) field 8 showing one globular formation with a phase inside, 400x

4. Discussion

The semi-solid metal is obtained, usually, by heat treatment of the alloy in the muffle furnace with or without stirring or by induction heating. Among these processes, induction heating has the advantages of a short period of warm up, versatility in the workpiece sizes and energy efficiency. Some alloys such as Ti6Al4V, however, form a dense and protective layer of oxides at the semi-solid temperature range requiring atmosphere free of oxygen during the process. In the present work a device is designed and set up to evaluate the possibility of obtaining a semi-solid Ti6Al4V alloy within a chamber with argon rich atmosphere. During the process the chamber has shown efficiency in protecting the billet against excessive oxidation. The equipment used in this work is a medium frequency induction heater that has the characteristic of heating the workpiece positioned in the center of the coil, from its surface to the center. Fig. 5a displays cavities in the center of the billet that seem to have been formed by core melting. Therefore, although the temperature read at the top of the billet is 1630 ^{o}C, its center may have reached at least 1660 ^{o}C [9] the melting point of the alloy. Although the intermittent pulses 1s on/2s off during 6 s shown in Table 1 guarantee that the temperature at the surface of the billet remains in the range of semi-solid formation it does not happen in its center. This occurrence can be explained by the argon flow at room temperature constantly cooling the surface where the temperature is taken. The homogenization of the temperature in the center could be achieved by maintaining the intermittent pulses for a period longer than 6 s and at lower power also allowing homogenization of the microstructure and maintaining the argon atmosphere with no flow. Sample A1, taken from the first billet, form some globular phase characteristic of the semi-solid alloy better seen in Figs. 4 (b and c). Comparing the microstructure of Fig. 3 to Fig. 4 to Fig. 7, it is possible to suppose that the heat treatment provides different microstructures that are not stable as those of the supplied material, suggesting that the freezing of the high temperature microstructure, the metastable energy state, is achieved by the cooling rate of 54 ^{o}C/s. At the room temperature the low concentration β phase is present in the Ti6Al4V alloy in the lamellar formation of about 200 μm in thickness alternated with the lower concentration α phase. At temperatures above 900 °C the amount of the β phase increases [6, 11]. Thus, in the range of temperature for semi-solid, it is expected that spheroid microstructures should appear[13] and almost all α phase has been transformed into β phase. Although in a low amount, it is possible to see the β phase globular formation in Figs. 4-7. The lowest quantity of the β phase (darker) compared to the α phase (lighter) in the Figs. 4e and 5 (d and e) can be attributed to an insufficient residence time at the temperatures above 900 °C. Some characteristic semi-solid formation, the globular β phase (darker), can be seen in Figs. 4 (b and c), 5 (b and c) and 7 (b, c and d). In the center of sample A2, field 7, shown in Fig. 7d, it is possible to notice the β globular phase in formation. As discussed earlier, the central region does not receive the same thermal treatment as the surface regarding heating as well as the cooling process when the billet is dropped in cold water. Also in sample A2, fields 5, 6 and 7, Fig. 7 (b, c and d) a significant amount of the β phase is observed. The longer the permanence in the semi-solid range of temperature the greater the globular formation [6]. Fields 5 and 6 are located near to the sample surface, Fig. 7 (b and c) and the field 7, Fig. 7d, in the center of the sample but near to the bottom of the billet where the heat dissipation is faster than in the center of the billet, Fig. 7e, justifying the coalescenting of the β phase. Besides, the coalescent globules in Fig. 7 (b, c and d) are an important indicator that the residence time in the semi-solid temperature range is sufficient for the initial formation of a semi-solid alloy, because during the semi-solid formation small globules tend to join each other to form larger ones [14]. The microstructural differences between the samples from billet 1 and billet 2 can only be justified by further tests under more controlled conditions than those used in this work. The main difficulty of reaching a semi-solid microstructure of the Ti6Al4V alloy is its narrow temperature range between the *solidus* and the *liquidus* lines that is the range where the semi-solid globular structure appears [15]. However, from the results of the present work, despite the narrow range of semi-solid temperature of the Ti6Al4V alloy, it seems possible to get it in a semi-solid state.

5. CONCLUSION

Although heat treatments of the Ti6Al4V alloy have been extensively investigated and some studies have been performed on its semi-solid formation from the melting to room temperature, until now, no investigation has been done regarding the semi-solid structure achievement by heating the alloy from the room temperature to the semi-solid range one. This work has explored this possibility by using heating induction and the equipment that requires low investment and is applicable to many alloys. The authors have realized that induction heating of the cylindrical workpieces creates a heating profile more complex than the plane ones; hence it will be taken into account for further works. Despite the limitations of this study, it is new in the metallurgical field and there are no approaches to support our findings. Therefore, based on the results, the next steps would be to build a better controlled environment in order to get stronger evidence for the semi-solid acquisition.

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