Microstructural Assessment of Biocompatible TNZT Alloy Cast by Counter-Gravity in Yttria Doped Titania Investment Shell Moulds

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Abstract: Alpha + beta Ti-6Al-4V alloy has limitation for load-bearing orthopaedic implants due to the presence of vanadium which is considered as toxic element. Beta titanium alloys are promising materials for load-bearing orthopaedic implants due to their excellent corrosion resistance and biocompatibility, low elastic modulus and moderate strength. The gravity pouring and counter-gravity pouring methods were employed to cast TNTZ alloy in the investment shell moulds. The counter-gravity pouring gave fine grain structure and better mechanical properties than those of the castings fabricated by gravity pouring method. The variation in the oxygen concentration (porosity) did not cause changes in the microstructure. Larger quantities of ceramic adhered to TNZT alloy surface indicating the adherence of TNZT alloy with ceramic coatings. The tensile curves of TNZT alloys exhibit double yielding phenomenon.

Keywords: Investment casting, biocompatible TNZT alloy, colloidal silica binder, Yttria doped Titania, counter-gravity pouring, gravity pouring.

1. INTRODUCTION

Beta-titanium alloys are capable materials for load-bearing orthopaedic implants due to their outstanding corrosion resistance and biocompatibility, low elastic modulus and moderate strength. The most commonly used is Ti-6Al-4V alloy that belongs to α+β Ti-alloys. The main drawback of this alloy is toxic nature of vanadium. In addition, a high amount of aluminum interferes with cell viability, besides having a long-term cytotoxic effect ***in-vitro*** and ***in-vivo*** [1]. ***In vitro*** refers to the technique of performing a given procedure in a controlled environment outside of a living organism. In microbiology ***in vivo*** is often used to refer to experimentation done in live isolated cells rather than in a whole organism, for example, cultured cells derived from biopsies. The α+β Ti-6Al-7Nb alloy has been developed to avoid the bad effect of vanadium [2]. Another adverse property is too high elastic modulus (115 GPa of Ti-6Al-4V and Ti-6Al-7Nb alloys) that is higher than that of cortical bone (20 GPa). Too high elastic modulus causes stress shielding and consequent osteoporosis that results in decreased life-time of orthopaedis implant [1-2]. In spite of reducing the elasticity modulus of titanium alloys without causing cytotoxic effects, new promising alloys that add niobium, tantalum, zirconium and molybdenum to titanium have been developed. These alloys represent a new class of titanium-based alloys, which are free of aluminum and vanadium, while exhibiting low values of the Young's modulus. Ti-35Nb-7Zr-5Ta (TNZT) alloy was developed in 1990s in USA and patented in 1999 [3]. The biggest current interest is focused on metastable β-titanium alloys with increased biocompatibility and decreased elastic modulus to avoid stress-shielding and increase of life-time of an implant [4]. The elastic modulus of TNZT alloy is 55 GPa. The mechanical properties of titanium-based alloys are directly related to their microstructure [5]. In fact, the strength and toughness of many β alloys are improved by the presence of the ω phase. The reason was attributed to the fact that α forms in a compositionally invariant way, having no long-range effect on the β matrix.

Investment casting differs from all other casting processes in the use of a disposable pattern to form the cavity into which the metal is poured. The complexity, detail and surface finish of the casting is directly dependent upon the integrity and dimensional stability of the original pattern [3-10]. Refractoriness is one of the desirable properties for investment casting moulds. The refractoriness of a material is the ability of the substance to withstand high temperatures without fusion or decomposition. The thermal expansion of a refractory is very important as this can influence mould dimensional stability and thermal shock resistance [11-18]. Crystalline silica has very undesirable thermal expansion characteristics due to the fact that it exists in a number of distinct allotropic forms. During heating or cooling, transformations between these crystalline forms take place. These changes involve expansion or contraction due to molecular rearrangements. No one single refractory has all the desired characteristics for producing an investment casting, but experience gained from using a variety of materials has led to the wide acceptance of mullite, zircon and alumina based refractories [19-27].

The purpose of this investigation was microstructural assessment of TNZT alloy cast by counter-gravity in Yttria-doped Titania investment shell moulds. The mechanical properties with loading and unloading conditions, porosity levels and metal-mould reactions were also investigated.

2. MATERIALS METHODS

The chemical composition of TNZT alloy is chemical composition 52Ti- 35Nb-7.3Zr-5.7Ta (wt. %). In the present work, the colloidal silica binder was used to fabricate the investment shell moulds from Yttria doped Titania as reinforced filler materials. The silica content in the colloidal silica binder was 30%. Two grades (primary and backup sands) of stuccoing sand were employed in the present investigation. Finer grade fused silica sand having AFS grain fineness number 140 was employed for primary coats. This is synthetic sand. This sand was used for first two coats, called prime coats to get good surface finish and every detail of the wax pattern. Coarser grade sand having AFS grain fineness number 60 was employed for back up coats. The backup sand was employed to develop more thickness to the shell walls with minimum coats [25-30]. The thickness of shell moulds were 10 mm. After all coats, the shells were air dried for 24 hours. Two shells of each treatment were made as shown in figure 1. The TNZT alloy was melted in an induction furnace under vacuum.

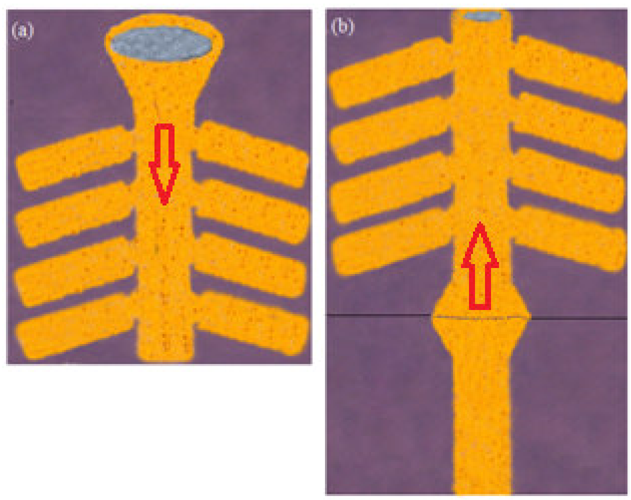

Figure 2: Investment shell moulds: (a) gravity poured and (b) counter-gravity poured.

The liquid alloy was gravity poured under vacuum into the pre-heated investment shell moulds. Also, the investment shell moulds were counter-gravity poured. In the counter-gravity casting process, as soon as the molten alloy is ready to be cast, a preheated investment shell mould was placed on the bottom opening of a mould chamber. A preheated investment shell mould was transferred into the chamber and placed on top of the ceramic tube. Support media is packed around the preheated shell mould and the chamber containing the shell mould is transferred to the melting furnace. The shell mould at the bottom of the chamber is then inserted into the argon atmosphere above the molten alloy. Argon was drawn into to the shell mould chamber by creating a vacuum in the shell mould. This action essentially displaced air in the shell mold cavity through the semiporous mould with argon. The mould was then inserted deep into the molten alloy and the vacuum in the shell mould cavity was increased at a controlled rate, enabling the mould filling. The level of vacuum in the mould cavity was 1/3 of an atmosphere to fill the mold in 6 seconds. The shell moulds were knocked off by hand hammer after solidification of the molten. The castings were cleaned with soft brush and visually inspected for pins and projections.

As-cast samples were sealed into quartz tube and beta solution treated at 1150°C/2h followed by water quenching. Such a high temperature was chosen to ensure full recrystallization. Samples for microstructure observations were carefully polished using SiC abrasive papers, Al_2O_3 (0.2 μm and 0.05 μm) and colloidal silica on vibratory polisher to obtain as clean surface. Grain structure was after etching in the solution consisting of 2 ml of 40% fluoric acid, 5 ml of 30% hydrogen peroxide and 50 ml of water for 2 mins. Tensile properties were obtained from the testing UTM (universal testing machine of 5 ton capacity) as per ASTM standard. Micro-hardness was measured on Vickers hardness tester.

3. RESULTS AND DISCUSSION

With gravity pouring, the microstructure of TNZT alloy reveals coarse gain structure; while it is fine grain structure through counter-gravity technique as seen from figure 2. The gravity poured TNZT alloy contains coarse grains of the size in the range of 75 – 100 µm. But, the counter-gravity poured TNZT alloy contains fine grains of the size in the range of 50 – 75 µm. The EDS analysis confirms the presence of various elements of TNZT alloy (figure 3). Figure 4 shows the porosity levels developed in the gravity and counter-gravity poured TNZT samples. It is observed that the porosity was high in the gravity poured TNZY samples. The densities of gravity and counter-gravity poured TNZT alloys were, respectively, 5.61 and 5.74 g/cm^3. The oxygen concentration in the pores was analyzed by EDS. Figure 5 shows the EDS pattern of the samples with1.142% O of gravity poured TNZT alloy and 0.516% O of counter-gravity poured TNZT alloy. Only the peaks from the β phase were detected. The variation in the oxygen concentration (porosity) did not cause changes in the microstructure. Fine particles of α phase are over the β grains. The stability of β-phase of TNZT alloy is greater because of the presence of β stabilizing elements (Ta and Zr).

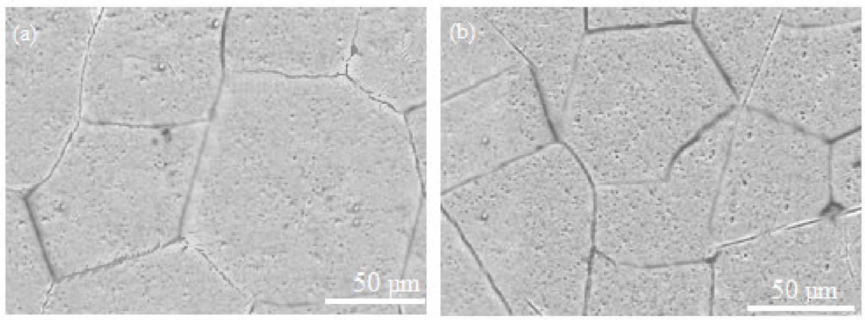

Figure 2: Optical microstructures of TNZT alloy: gravity poured and (b) counter-gravity poured.

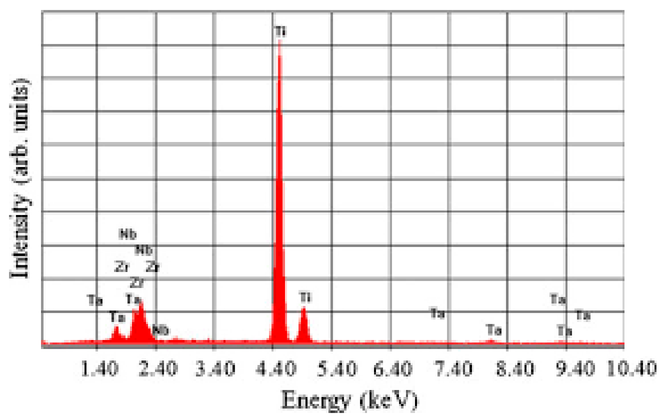

Figure 3: EDX analysis of counter-gravity poured TNZT alloy.

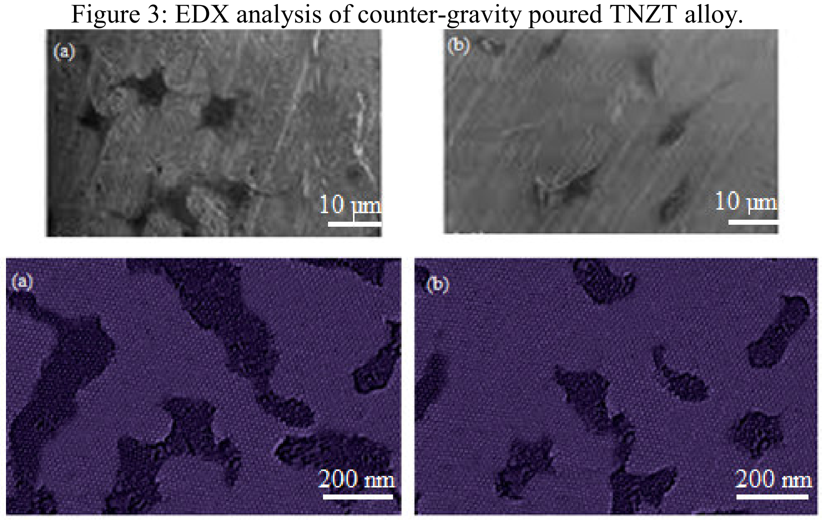

Figure 4: Porosity in as-cast TNZT samples: 9a) gravity poured and (b) counter-gravity poured.

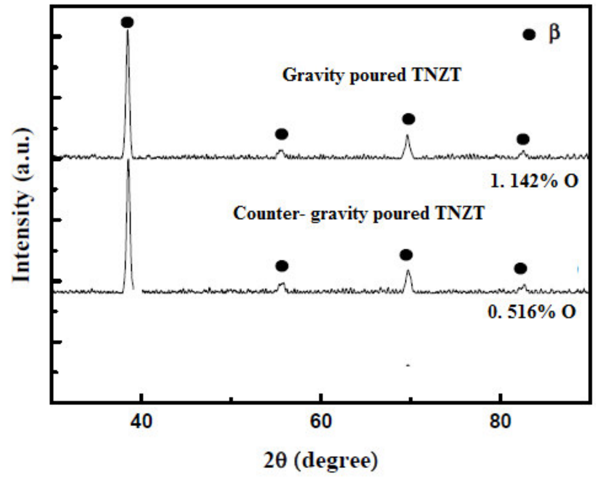

Figure 5: EDS analysis for porosity in as-cast TNZT alloy.

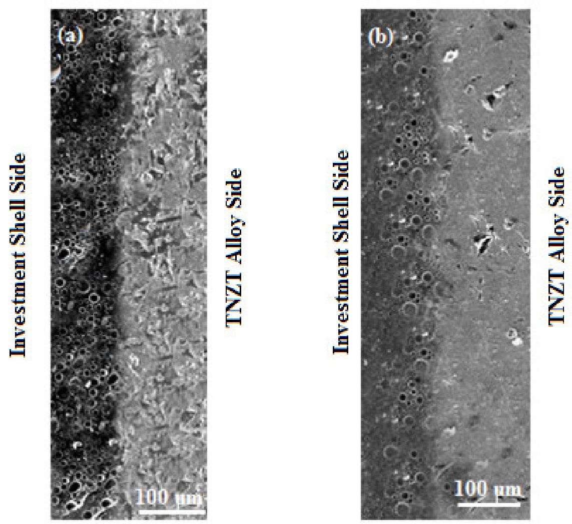

Figure 6: Metal-mould reactions: (gravity and (b) counter-gravity poured TNZT alloy.

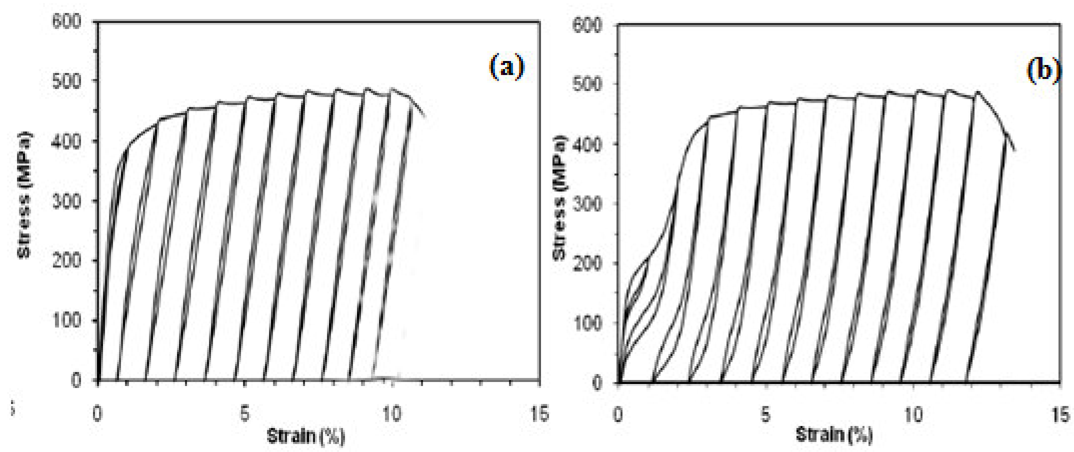

Figure 7: Loading and unloading curves of stress-strain.

Ceramic bits and pieces could be identified on metal as dark areas, in contrast, light regions represented TNZT alloy surface as shown in figure 6. The oxide layer is also seen. It was observed larger quantities of ceramic adhered to TNZT alloy surface indicating the adherence of TNZT alloy with ceramic coatings. Figure 7 shows tensile loading- unloading stress-strain curves of TNZT alloys. The gravity poured TNZT alloy has an elongation of 10 %; while the counter-gravity poured TNZT alloy has an elongation of 12 %. The tensile curves of TNZT alloys exhibit double yielding phenomenon. The first one corresponds to the critical stress for inducing the transformation of β to α upon loading and the second one the beginning of dislocation slip. The phase transformation due to loading for TNZT alloy improves the shape recovery and it presents a superelastic behavior. The gravity and counter-gravity poured TNZT alloy exhibited the modulus of elasticity 53.52 GPa and 55.96 GPa, which can be compared to the literature of 55 GPa. The micohardness of TNZT alloy was found to be 348-352 HV.

4. CONCLUSIONS

The counter-gravity pouring of TNZT alloy in investment shell moulds has yielded fine grain structure in the castings. Also, the mechanical properties of TNZT alloy are superior with counter-gravity pouring. The tensile curves of TNZT alloys exhibit double yielding phenomenon. Gravity and counter-gravity poured TNZT alloy exhibited the modulus of elasticity 53.52 GPa and 55.96 GPa, respectively.

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