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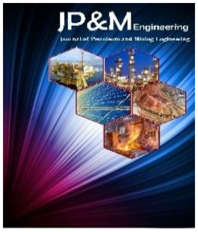

Heat Treatment of Low Cost Beta Titanium Alloy Produced by Investment Casting

Reham Reda[1*] and Abdellattief Adel[2]
[1] Mechanical Engineering Department, Faculty of Engineering, Suez University, Suez, Egypt
[2] Mechanical Engineering Department, Faculty of Engineering, Helwan University, Helwan, Egypt
*Corresponding Author Email: Reham.Reda@suezuni.edu.eg

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Abstract

Beta (β) Ti-alloys are promising class of Ti-alloys for high performance structural applications, thanks to their high strength to weight ratios and outstanding corrosion resistance. In the current work, Ti-4.5Fe-7Cr low cost beta Ti-alloy was produced by investment casting. DSC test was carried out to determine the transformation temperatures. The influence of solution treatment and aging temperatures on microstructure and mechanical properties were studied. Six heat treatment cycles were carried out, two solution treatment cycles in β- and α+β-range at 900 °C and 750 °C, respectively, and four precipitation hardening cycles, i.e. α+β-aging at 450 °C and 650 °C after solution treating at both 900 °C and 750 °C. Microstructure examination, XRD analysis and compression test had been conducted to investigate the influence of the different heat treatment regimes on the proposed alloy. Solution treatment at 900 °C results in coarser β-grain size than at 750 °C; hence lower strength. Strengthening by α-phase precipitation and grain refinement was achieved during aging. Aging at 650 °C results in higher strength than at 450 °C although coarser grain size, this is attributed to stronger effect of precipitation hardening of α-phase at 650 °C as a result of its higher amount than at 450 °C. The designed alloy shows high strength and excellent malleability, i.e. the alloy reveals superior plastic deformability, where specimens did not reveal any fracture; and the compression tests were interrupted at a compressive strain of 33%; therefore, this alloy may find applications as a healthcare, automotive or aerospace material.

Introduction

In spite of the outstanding properties of titanium alloys, its high cost limits the widespread utilization in industry [1-4]. This high cost of titanium alloys includes the high price of the raw materials plus the cost of production. In order to reduce the total cost, low cost production technique should be undertaken. Commonly, titanium requires vacuum casting followed by deformation using any metal forming technique aiming at enhancing the alloy properties as possible to promote its performance in the application. Investment casting is a distinctive technique. The products of the investment casting usually have near-net shape and comparable properties to the deformed products. Hence, the high cost of deformation can be economized. Investment casting may be followed by heat treatment for more enhancement of the properties. Short holding time heat treatment can be conducted to reduce the total production cost.

β Ti-alloys possess high strength-to-weight ratio besides the outstanding corrosion resistance [5,6]. The outstanding properties of β-alloys are greatly manifested after solution and aging treatments as their properties are influenced by the morphology, size, volume fraction and distribution of α-precipitates, these characteristics depend on heat treatment parameters, such as: ageing temperature and time [6,5,7]. Maintaining high ductility with high strength can be achieved by controlling the β-grain size. Finer β-grains enhances both strength and ductility, this structure is attained by utilizing lower aging temperatures [6].

β Ti-alloys find applications in healthcare, biomedical, aerospace, automotive, etc. [1-3,5,8-11]. Despite their advantages, β Ti-alloys have a limited spread in applications, this is attributed to the relatively high costs [7]. In order to promote the extensive application of these materials, the cost reduction while maintaining high properties should be the basic trend in designing the new alloys. Taking cost in consideration, low cost β Ti-alloys had been previously developed [1-3,8,9,12]. This trend utilizes low cost ferro-alloys (ferro-chrome, ferro-molybdenum, etc.) to be added to pure titanium. Therefore, reducing both the cost of production and materials with keeping acceptable properties were taken in consideration in the current study.

Alloys based on Ti-Fe-Cr ternary system had been early proposed [1,2,4,8,11-13]. Gunawarman et al. [12,13] had previously developed two low cost beta Ti-alloys, using Fe-Cr ferro-alloy with Al-addition, for healthcare and biomedical applications. In their work, the effect of β-solution treatment and thermo-mechanical treatment on the microstructure, tensile and fatigue properties were investigated. Ikeda et al. [4] studied the influence of cooling rate from solution treatment temperature on the microstructure and tensile properties of Ti-Fe-Cr-Al alloy. They stated that cooling rate from solution treatment temperature has a significant effect on the microstructure and tensile properties of this alloy as Fe and Cr have higher diffusion coefficients in β-phase than other β-stabilizers. Hence, if β-phase matrix composes of high diffusion coefficient β-stabilizers, brittle ω-phase precipitation becomes fast and a high cooling rate is required to hinder this ω precipitation. Similar observation of the effect of Cr and Fe on ω precipitation was reported by Markovs'kyi [2]. Guo et al. [1] also developed another two low-cost α+β Ti-Cr-Fe titanium alloys used for automobile springs. They examined the effect of cooling rate during heat treatment on microstructure and tensile properties. Wang et al. [11] designed and prepared a novel α+β type Ti-Al-Cr-Fe titanium alloy based on aluminum and molybdenum equivalent, the quaternary system of the alloy. They investigated the microstructure and mechanical properties of the designed alloy after thermo-mechanical treatment.

The current work aims at developing a new commercial alloy and studying the effect of solution treatment and aging temperatures of Ti-4.5Fe-7Cr low cost beta alloy produced by investment casting on the microstructure and mechanical properties in compression. Shortening the time of heat treatment to reduce the total production cost was adopted.

Materials and Methods

Alloy Design

Based on a molecular orbital method, Bo-Md map is utilized to define the Ti-alloy type. Bo is the average bond order which gauges the covalent bond strength between Ti and alloying elements. Md is the average d-orbital energy level of Ti with alloying elements, it relates to the electronegativity and the radius of the metallic elements. The average values of Bo and Md for an alloy are determined by calculating the compositional averages of Bo and Md values, as reported by Matsugi et al. [9] and Gunawarman et al. [13], using the following equations:

$$\mathrm{Bo} = \Sigma\, X_i\, (\mathrm{Bo})_i$$

$$\mathrm{Md} = \Sigma\, X_i\, (\mathrm{Md})_i$$

Here, X_i is the atomic fraction of component i in the alloy, $(\mathrm{Md})_i$ and $(\mathrm{Bo})_i$ are the Md and Bo values for component i, respectively. The values of $(\mathrm{Md})_i$ and $(\mathrm{Bo})_i$ are listed in Table (1).

Table (1) List of Bo and Md values in bcc Ti.

Elements	Bo	Md
Ti	2.79	2.447
Fe	2.651	0.969
Cr	2.779	1.478

The design of low cost beta Ti-alloy depends on selection of low cost β-stabilizers that stabilize β-phase at room temperature. Cr and Fe, as β-stabilizers, are β-eutectoid forming elements. These two elements are available materials and can substitute the expensive elements (such as V, Mo, Nb, etc.) in order to reduce the cost of the raw materials [2,8,11,13]. Fe and Cr have relatively low density and strong β-stabilizing effect; therefore, they are used as the major alloying elements for designing high-strength β-type titanium alloys [10]. Fe-Cr ferro-alloy had been used in the current study instead of using high cost pure elements, the proposed alloy is Ti-4.5Fe-7Cr.

The average Bo and Md parameters for the proposed alloy are 2.784 and 2.326, respectively. By locating these values on the Bo-Md map, i.e. the phase stability map of Ti-alloys (Fig.1) [9,13], the resultant is a Ti-alloy of β-phase structure. The position of the proposed Ti-4.5Fe-7Cr alloy is identified in the map.

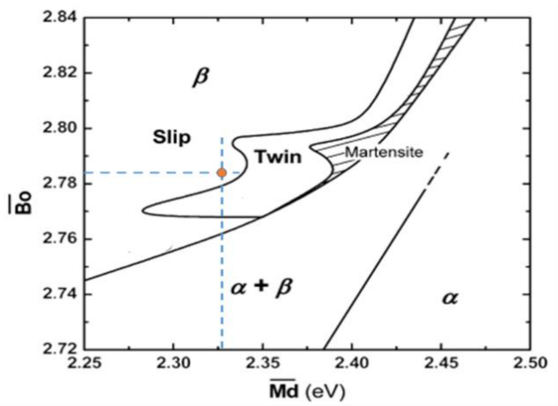

Figure 1 Phase Stability Map of Ti-alloys based on Bo and Md parameters [9,13]. The position of the proposed alloy is identified.

The effect of the different β-stabilizing elements that required to stabilize β-phase and prevent the martensite transformation on quenching can be described using molybdenum equivalent. It was reported that a minimum value of molybdenum equivalent needed to make the β-phase stabilized during quenching is about 10-11% [10]. Molybdenum equivalent of the proposed Ti-4.5Fe-7Cr alloy was calculated to be about 24%, using a previously reported equation [10,11]. This high value of the molybdenum equivalent was proposed to ensure high stability of β-phase matrix structure in cooling during heat treatment, therefore, avoid martensite transformation and omega precipitation on cooling, these phases decrease the ductility, in addition to reach maximum strength after quenching and aging [11].

Investment Casting

Fig. 2 shows photos of the investment casting process performed in the current work. A metallic wax injection die with a cavity of 15mm diameters and 200mm length (Fig. 2-a) was utilized to produce wax bars (Fig. 2-b), these bars were then used to make the wax tree (Fig. 2-c).

After wax tree preparation, a shell mold was formed through alternate dipping of a wax tree into a zirconia colloidal suspension and then covering with ZrO2 powder of about 210 µm particle size. This primary coating consists of three layers. After that, eight layers of the secondary coating were prepared to enhance the shell mold strength by pouring mullite ($3Al_2O_3.2SiO_2$ compound) with particle size of about 1200 µm.

Figure 2 Photos of investment casting process: (a) a metallic wax injection die, (b) the produced wax samples, (c) wax tree and (d) the metallic tree after melt pouring and solidification.

The mold was then dried, de-waxed and strengthen in a muffle furnace at 900°C for 1 hour to acquire suitable strength for subsequent handling and casting. The final thickness of the ceramic shell mold is about 7 mm.

Skull induction melting furnace (SIM) was used to melt the proposed alloy, 1Pa vacuum and 200 rpm mold rotation speed were undertaken during casting. In SIM, ceramic crucible was used in melting. The desired composition of the charge was adjusted by appropriate additions of Fe-70%Cr ferro-alloy and pure Fe to pure Ti. Molten charge was heated to 1520-1560°C. The temperature was monitored by mean of a calibrated W-Re thermocouple during melting. After melting, argon was released into the melting oven. Melting process of the ingot was repeated three times to ensure the alloy homogenization as possible. Finally, the melt was poured at 1450 °C into the preheated prepared ceramic shell mold at 600 °C to provide specimens in the shape of rods. The produced metallic tree is shown in Fig.2-d. The ceramic mold was broken using airborne-particle abrasion with 75 µm Al2O3 and 3 bar pressure. The as-cast rods then machined to remove the reaction layer at the rod surface using a CNC machine. The final chemical compositions of the alloy was tested using XRF portable device to be 7.11%Cr, 4.59%Fe, 88.2%Ti and 0.1% traces.

Differential Scanning Calorimetry (DSC)

The transformation temperatures of the designed alloy were determined using heat-flux differential scanning calorimetry (DSC). A sample was heated to 1000 °C at heating rate of 10 °C/min, held for 3 minutes, followed by cooling with rate of 50 °C/min. the endothermic and exothermic flow was recorded with temperature during heating/ cooling. β-transus transformation temperature was determined on heating and cooling using the resulted heat flow-temperature curves.

Heat Treatment

Six cycles of short holding time heat treatments were carried out to the machined as-cast rods, i.e. two solution treatments and four precipitation hardening, i.e. solution treatment followed by aging. Solution treatments were selected to be one in β-range and theother in α+β-range, in order to study the difference in the resultant microstructure and mechanical properties. Therefore, solution treatments for one hour were executed at 900 °C (designated by ST900) and 750 °C (designated by ST750), followed by water quenching. Subsequent aging at 450°C and 650°C for two hours were performed for both solution treated temperatures, followed by air cooling, in order to examine the effect of low and high aging temperature on the resultant microstructure and mechanical properties. The aged specimens are designated by STA900-450, STA900-650, STA750-450 and STA750-650.

Materials Characterization

Specimens for characterization were machined from the heat treated rods. For metallographic investigation and X-ray diffraction analysis, specimens were ground and polished using a 9 µm diamond paste, then specimens were acid-etched with a Kroll's reagent (a mixture of 10 ml HF, 5 ml HNO3 and 85 ml H2O). Optical microscope equipped with digital camera and XRD analysis with CuKα irradiation were utilized. The grain size was estimated by the line-intercept method.

Cylindrical specimens with 7 mm diameter and 15 mm length were prepared for compression test. Tests were conducted on the heat treated specimens using uni-axial testing machine at room temperature and with a strain rate of 1.3x10-3 s-1. The average of three readings was reported for all properties.

Results and Discussions

Phase Transformation and Microstructure Investigation

Fig. 3 shows DSC results on heating and cooling of the as-cast alloy. On heating, α to β-transformation begins at 740 °C. At 785 °C, peak of α to β endothermic transformation reaction appears. The transformation ends on heating at 870 °C. These three temperatures are indicated on the heating curve (Fig. 3) by B_H, P_H and E_H for beginning, peak and end of α to β-transformation on heating, respectively. During cooling, β to α-transformation begins at 870 °C, represented by B_C. The peak of exothermic reaction of β to α-transformation on cooling appears at 760 °C (P_C) and the transformation ends at 670 °C (E_C).

As revealed from DSC curves (Fig. 3), the endothermic and exothermic reactions of α to β- and β to α-transformation on heating and cooling, respectively, appears as slight peaks. This refers to low amount of α-phase existing in the structure whether transformed to β-phase during heating or produced from β-phase on cooling. Hence, the proposed as-cast alloy has mainly β-phase structure at room temperature with the presence of α-phase traces that likely to form on slow cooling during casting.

In the current study, part of the as-cast specimens was solution treated in α+β-range at 750 °C while the other part was solution treated in β-range at 900 °C.

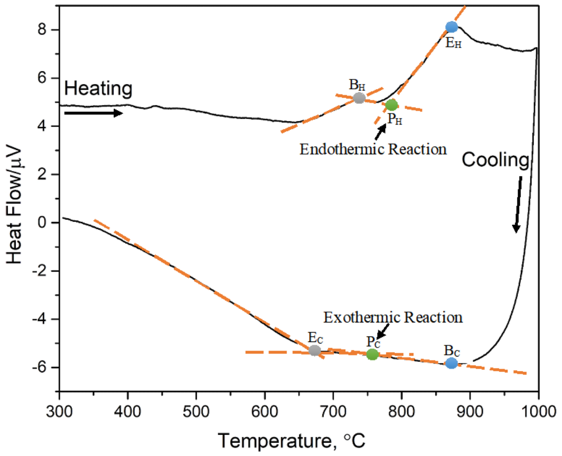

Figure 3 DSC curves during heating and cooling of the as-cast proposed alloy.

Fig. 4 and Fig. 5 present the microstructure and XRD profiles of the designed alloy after heat treatment, respectively. After β-solution treatment at 900 °C (ST900), the microstructure reveals β-phase matrix with traces of α-phase while after α+β-solution treatment at 750 °C (ST750), a finer microstructure consists of β-phase matrix with higher amounts of α-phase as compared with ST900 condition.

Average grain size of β-phase matrix was measured to be 324.157±67µm and 279.792±65µm for ST900 (Fig 4-a) and ST750 (Fig 4-d), respectively, as presented in Table (2). Similar observation was reported by many authors [3,5,6,12] who stated that as the solution treatment temperature increases, the β-grain size becomes coarser and coarser structure formed upon cooling.

On aging, the decomposition of the β-phase formed in solution treatment to α-phase occur, as defined by XRD profiles (Fig. 5). According to the microstructures and XRD profiles, as the aging temperature raises from 450 °C to 650 °C, the amount of the precipitated α-phase increases at the expense of β-phase amount regardless the solution treatment temperature. This observation agrees with previously reported results [7].

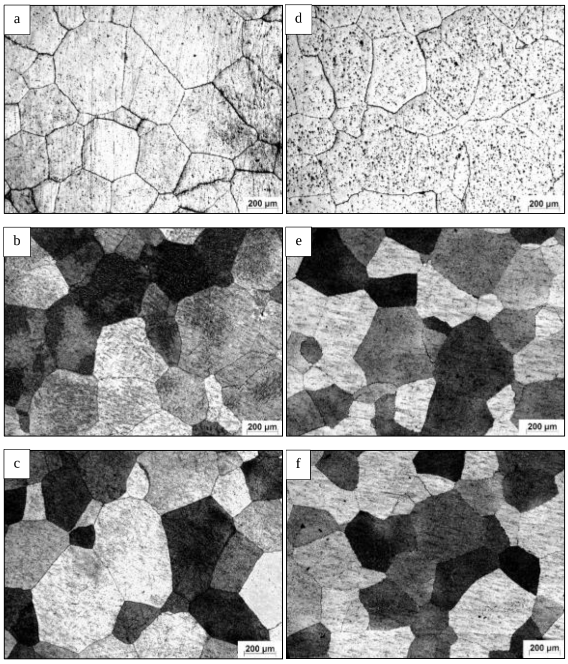

Figure 4 Optical micrographs of the heat-treated specimens: (a) ST900, (b) STA900-450, (c) STA900-650, (d) ST750, (e) STA750-450 and (f) STA750-650.

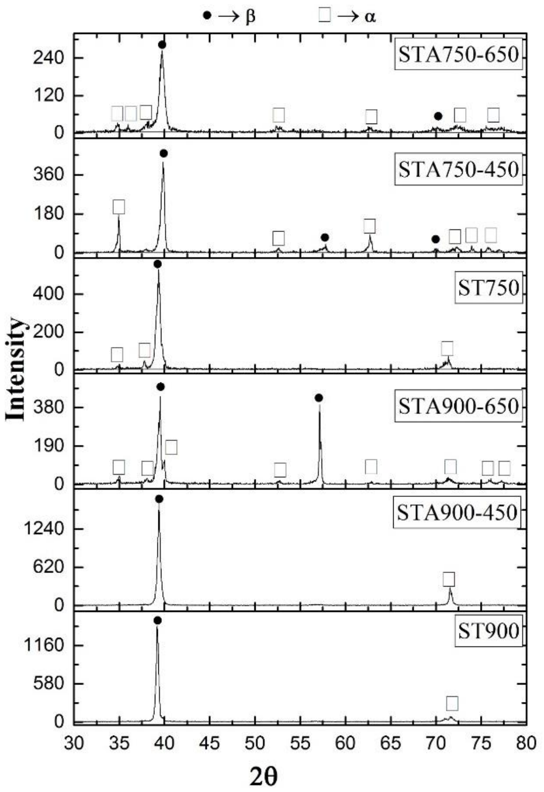

Figure 5 XRD profiles of the heat-treated specimens.

Average grain size of the aged microstructure are presented in Table (2). Average β-grain size, in α+β solution treated and aged conditions, is much finer as compared to β solution treated and aged conditions. In addition, aging at 450 °C results in finer microstructure than at 650 °C regardless solution treatment temperature, hence lowering aging temperature promotes the β-grain refinement. These observations agree with previous results [6,7]. The average grain size is 285.179±32µm and 289.342±44µm for STA900-450 (Fig. 4-b) and STA900-650 (Fig. 4-c), respectively, and 252.569±33µm and 261.419±30µm for STA750-450 (Fig. 4-e) and STA750-650 (Fig. 4-f), respectively.

Table (2) Average grain size of the heat treated specimens.

Conditions ***Properties***	***Average grain Size (µm)***
ST900	324.157±67
STA900-450	285.179±32
STA900-650	289.342±44
ST750	279.792±65
STA750-450	252.569±33
STA750-650	261.419±30

The current results reveal that average grain size after aging is finer than after solution treatment. Lin et al. [5] reported similar observation that the aging refines the microstructure but this depends on the undertaken aging time.

Mechanical Properties

Fig. 6 shows stress-stain curves of the heat treated specimens on compression. All specimens show high plasticity, i.e. no failure, so the test was interrupted at a selected value of 33% total strain and the compressive strength at this strain value was considered for comparison. Except for STA750-650 condition that failed at 19.43% total strain. Table (3) presents the compression properties of the specimens after heat treatment.

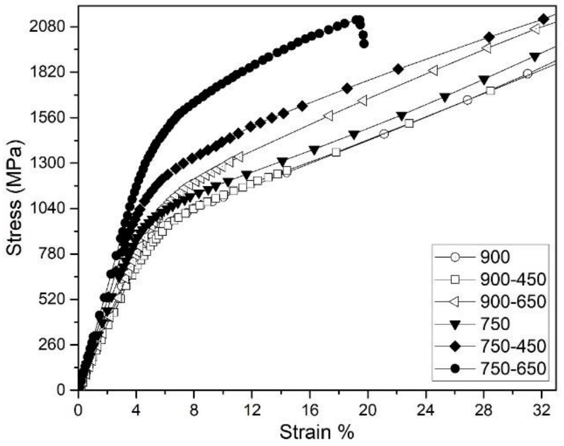

Figure 6 Compression stress-strain curves of the heat-treated specimens at room temperature.

The compressive strength increases from 1870.5 MPa to 1913 MPa as the solution treatment temperature decreases from 900 °C to 750°C. This is interpreted by the finer grain structure formed after solution treated at 750 °C as compared with that at 900 °C.

The strength of the specimens after aging is higher than after solution treatment regardless the solution treatment temperature. This is attributed to the precipitation hardening effect of the α-phase along with the grain refinement occur after aging as compared with solution treated conditions. These effects contribute in increasing the strength values. Lin et al. [5] stated that aging promoted the yield strength in compression as compared to the solid solution-treated only in at short aging time.

Table (3) Compression properties of the heat treated specimens.

Conditions **Properties**	**Compressive Strength (MPa)** **[At 33% total strain]**	**Yield Strength** **(0.2% offset) (MPa)**
ST900	1870.5	764
STA900-450	1877.6	793
STA900-650	2068.3	885
ST750	1913	870
STA750-450	2124	1082
STA750-650	2118.2*	1266

* The compressive strength was at 19.43% total strain before failure.

In spite of coarser grains formed on aging at 650 °C than 450 °C, STA900-650 specimen has higher yield strength than STA900-450. The same results for aged specimens after solution treatment at 750 °C were obtained, i.e. STA750-650 specimen has higher yield strength than STA750-450. This is attributed to the precipitation of the higher content of α-phase precipitated at 650 °C than at 450 °C that enhances the alloy strength, as reported previously by Li et al. [3] who stated that the fine α-phase precipitated during aging in the β-phase matrix are un-deformable particles; hence act as obstacles for dislocations movement and, therefore, enhance the strength of β-type Ti-alloys. Shekhar et al. [7] also stated that the precipitation of α-phase during aging of β Ti-alloy promotes the strength.

Generally, solution treatment in α+β range followed by aging gives better combination of strength and ductility as compared with solution treatment in β range followed by aging, this result agrees with previous work [7].

Fig. 7 presents photos of the specimens after compression test. All specimens solution treated at 900 °C (ST900 (Fig. 7-a), STA900-450 (Fig. 7-b) and STA900-650 (Fig. 7-c) specimens) exhibit obvious cracks in some tested specimens, this is due to the formation of coarse structure.

ST750 (Fig. 7-d) and STA750-450 (Fig. 7-e) specimens show high plasticity with very few tiny cracks during compression. This is due to the existence of fine high stability β-phase matrix with suitable amount of the α-phase formed at these conditions. STA750-650 specimens failed by brittle shear mode at 19.43% strain (Fig. 7-f). This is attributed to the precipitation of high amount of α-phase in STA750-650 structure which may subject to coarsening at 650 °C, as reported previously [7] and revealed by XRD profiles (Fig. 5).

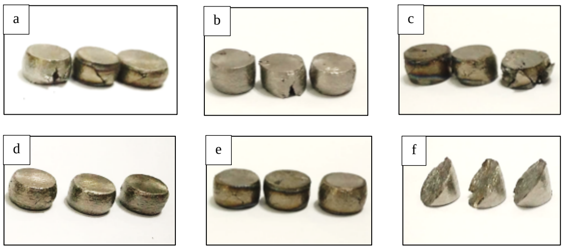

Figure 7 Photos of the specimens after compression test: (a) ST900, (b) STA900-450, (c) STA900-650, (d) ST750, (e) STA750-450 and (f) STA750-650.

Conclusions

Ti-4.5Fe-7Cr low cost beta Ti-alloy was designed with a molecular orbital method and Molybdenum equivalent approaches. The average Bo, Md and Moeq values for the proposed alloy are 2.784, 2.326 and 24%, respectively. The alloy was produced using investment casting process. DSC curves show that the peak of α to β endothermic transformation reaction occurs on heating at 785 °C while the peak of β to α exothermic transformation reaction occurs on cooling at 760 °C. The influence of solution treatment and aging temperatures on the microstructure and mechanical properties in compression were studied. The following conclusions were drawn:

(1) As solution treatment temperature increases from 750 °C to 900 °C, the average grain size of β-phase matrix increases from 279.792±65 μm to 324.157±67μm, respectively.

(2) The mechanical properties are improved after α+β-solution treatment at 750 °C by 13.87 % and 1.89 % for yield and interrupted compressive strength, respectively, as compared with β-solution treatment at 900 °C. ST900 specimen shows many cracks after compression up to 33% strain while few tiny cracks are appeared in ST750 specimen.

(3) The mechanical properties are promoted by aging as compared with solution treated conditions. This is attributed to the precipitation hardening effect of α-phase along with grain refinement on aging.

(4) Aging at 650 °C increases strength as compared with at 450 °C, in spite of coarser grain size. This can be interpreted by the precipitation of the higher amount of α-phase at 650 °C than at 450 °C that enhances the alloy strength.

(5) The optimum heat treated condition for the studied alloy is solution treatment in α+β-range followed by aging at low temperature (450°C) to avoid grain coarsening and enhances the precipitation hardening effect of fine α-phase.

(6) The proposed low cost beta Ti-alloy exhibits high strength and high malleability, i.e. an outstanding compressive strain where no fracture occurred and the compression tests were interrupted at a compressive strain of 33%, for most heat treated conditions; hence this alloy can be deformed under high pressure without failure and may find applications in healthcare, aerospace and automotive sectors.

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Conflicts of interest

There are no conflicts to declare.

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