



# High-temperature deformation behavior and microstructural characterization of high-Mn bearing titanium-based alloy

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## ABSTRACT

Ti-Mn alloys exhibit an excellent potential for biomedical applications as well as structural engineering applications, especially in the aerospace industry. In order to control and enhance grain structure during the manufacturing of Ti-Mn alloys and thereby help to enhance mechanical properties such as strength and toughness, we studied the hot-deformation behavior of βTi-10Mn alloys. Isothermal compression tests were conducted in the strain rate range of 0.01–10 s<sup>−1</sup> and temperatures in the range of 850–1000 °C using a Gleeble thermo-mechanical simulator. High-temperature flow stress curves exhibited discontinuous yielding and pronounced periodic serrations without any strain hardening during compression straining of these alloys. Such peculiar behavior of this alloy is due to active dynamic strain aging in its β-bcc structure. Metallographic observations by electron-backscattered diffraction (EBSD) analysis revealed that dynamic recovery (DRV) is more active than continuous dynamic recrystallization (CDRX) when the alloy is deformed at high strain rates, i.e. higher than 1 s<sup>−1</sup>. Furthermore, the constitutive behavior of the alloy was modeled and the apparent hot-deformation activation energy of the alloy was estimated to be 243 kJ/mol, which is ~60% higher than the self-diffusion energy in pure titanium.

## 1. Introduction

Titanium alloys have received extensive attention as structural materials in biomedical applications and aerospace industry owing to their high specific strength, high corrosion resistance, excellent formability, and high biocompatibility [1–5]. The phase structure of Ti alloys, such as α, α + β, or full β, depends mainly on the alloying elements. For instance, a Ti-6Al alloy has a phase structure of α + β at high alloying contents of 6% Al and 4% V. However, Al and V induce health problems due to dissolution when the alloy is implanted in the human body [6]. Another disadvantage of α + β Ti6Al alloy is that the alloy exhibits a large elastic modulus and high stiffness compared to the human bone. Consequently, stress shielding damage may be induced in bones [7–10]. To counter this problem, β-Ti alloys have been developed with non-toxic alloying elements, such as Mo, Nb, Zr, and Ta [11–16], to overcome the above-mentioned disadvantages.

The mechanical properties of β Ti alloys, particularly strength, are mainly governed by their high work-hardening capacity and elastic modulus, which in turn is related to the type of alloying elements and their contents [17,18]. For instance, β Ti-20 wt% Mo exhibits an elastic

modulus of 75 GPa and a significantly high yield strength of 428 MPa with a moderate elongation of 15% [19,20]. Moreover, a β-type Ti-15Mo-5Zr-3Al (wt%) alloy exhibits an elastic modulus of 80 GPa and an excellent combination of yield strength and ductility (838 MPa and 25%, respectively) [16]. Matsumoto et al. [21] studied the influence of cold-rolling deformation on the mechanical properties of β Ti-35Nb-4Sn alloys. They reported that the yield strength of the alloy increased significantly to 620 MPa after cold deformation, which led to a reduction in the elastic modulus to 43 GPa.

A review of the available literature indicates that research on Ti-based alloys has been continuous over the past several years with a particular focus on increasing yield strength using alloying elements, such as niobium, vanadium, zirconium, and molybdenum via substitutional strengthening [22,23]. Economically, this approach is not desirable to the industry because it may increase production costs as a result of using expensive alloying elements.

Ti-Mn alloys exhibit potential as a low-cost structural material for biomedical applications. Because manganese acts as a strong stabilizer for the β phase in Ti-alloys, a Considerable number of efforts have been directed towards enhancing the mechanical properties of Ti-Mn alloys.

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In this context, Niinomi's research group at Tohoku university studied the influence of Mn content (in the range of 8–19 wt%) on the mechanical properties of Ti alloys and reported that a Mn content of 9 wt% led to the best combination of strength and ductility. However, metallic ion release from Ti-Mn alloys depends on the Mn content, which is a crucial factor in biomedical applications [24,25]. In a following study, the same authors adopted cold-rolling deformation to increase the strength of Ti-13Mn alloys at ambient temperature. Severe plastic deformation (90% reduction in thickness) increased the strength of the alloy by 208% owing to microstructural refinement [26]. The effect of cold rolling on the mechanical properties of Ti-Mn systems was further studied by Gouda et al. [27]. They reported that low Mn-content alloys exhibited low cold workability and high Mn-content alloys showed high cold workability up to a certain Mn content, after which it decreased drastically. Park et al. [28] studied the cytotoxicity of Mn and V as alloying elements in Ti alloys and found that Mn exhibits much lower toxicity than V.

It is noteworthy that thermomechanical processing is an often-used metallurgical process for the microstructural control of metallic alloys [29,30]. Hence, mechanical properties can be governed by controlling the microstructure of alloys by optimizing hot-deformation parameters, such as temperature, strain, and strain rate [31–35].

In recent years, materials researchers have focused on the hot-deformation mechanisms of  $\beta$  Ti-alloys to optimize the deformation process and monitor microstructural evolution. For instance, Zhao et al. [36] studied the hot deformation mechanisms and activation energy during the hot working of Ti-10V-2Fe-3Al alloy in the temperature range of 820–900 °C. Chen et al. [37] studied the high-temperature deformation behavior of  $\beta$  Ti-2Al-9.2Mo-2Fe alloy. They analyzed the microstructural evolution and softening mechanisms during the hot deformation of that alloy and found that dynamic recrystallization (DRX) is the dominant restoration mechanism at low strain rates, while dynamic recovery (DRV) dominates at high strain rates ( $10 \text{ s}^{-1}$ ) and low temperatures.

However, to the best of the authors' knowledge, the hot-deformation behavior and deformation mechanisms under compression straining of  $\beta$  Ti-Mn alloys have not yet been reported. The present study focuses on the deformation mechanisms in the  $\beta$ -phase structure of Ti-10 wt% Mn during hot working. A thermomechanical Gleeble simulator was used to conduct a number of hot compression experiments, after which the alloy microstructure was analyzed comprehensively. We believe that this study would enable the manufacture of low-cost Ti-10Mn alloy via optimized processing routes and consequently, the mechanical properties of  $\beta$  Ti-alloys can be controlled by controlling the grain structure through activated deformation mechanisms.

## 2. Experimental Procedures

The Ti-10 wt% Mn alloy used in this study was prepared by melting high-purity elements in an electric arc furnace in an argon atmosphere (ARCAST 200, USA); the melt was subsequently cast as 200 g ingots. The ingots were homogenized at 1000 °C for 8 h in a protective argon atmosphere and subsequently hot-rolled at 850 °C to 8.5 mm-thick strips in a laboratory hot-rolling mill. The strips were solution heat-treated at 900 °C for 30 min in a vacuum tube furnace in an argon atmosphere, followed by quenching in ice water to induce a full  $\beta$ -phase structure at room temperature.

Bars, 7.5 mm in diameter, were cut and machined from the rolled strips by electrical discharge machining (EDM) wire-cutting. Subsequently, cylindrical specimens of  $\varnothing 6 \text{ mm} \times 9 \text{ mm}$  were machined for hot-compression testing with their axes parallel to the rolling direction.

To study the hot-deformation characteristics of the  $\beta$  Ti-10Mn alloy under consideration, isothermal constant true strain rate compression tests were conducted over the temperature range of 850–1000 °C and strain rate range of  $0.01\text{--}10 \text{ s}^{-1}$  using a Gleeble 3800 thermo-

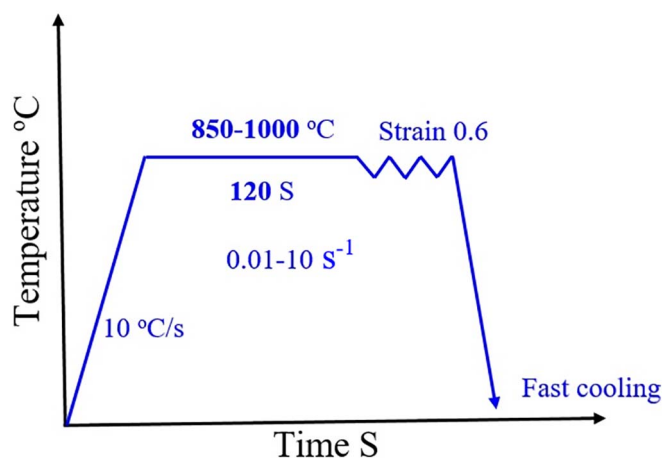


Fig. 1. A typical thermomechanical cycle used in the hot-deformation testing of  $\beta$  Ti-10Mn alloy.

mechanical simulator (Dynamic Systems Inc., Poestenkill, NY). A typical thermomechanical cycle adopted in this study is shown in Fig. 1. For this test, the specimens were reheated in vacuum at a rate of  $10 \text{ °C/s}$  to the deformation temperature (850, 900, 950, and 1000 °C) and held for 120 s in order to obtain a homogeneous temperature distribution throughout the specimen. Subsequently, the specimen was compressed in a single hit to a true strain of 0.6 at a constant strain rate of 0.01, 0.1, 1, or  $10 \text{ s}^{-1}$ .

The grain structure characteristics as well as the deformation mechanisms during the hot deformation of the alloy were studied using a field-emission gun scanning electron microscope (FEM-SEM) equipped with an electron-backscattered diffraction (EBSD) unit to examine the fine details of the hot-deformed structures. An acceleration voltage of 15 kV and a step size of  $1 \mu\text{m}$  were used during EBSD scanning. The hot-deformed specimens were also examined using an optical microscope after etching with a standard Kroll's reagent (3 mL HF + 6 mL  $\text{HNO}_3$  + 100 mL  $\text{H}_2\text{O}$ ).

## 3. Results and Discussion

### 3.1. Initial Microstructure

Microstructural observations of the studied Ti-10Mn alloy revealed a full  $\beta$ -bcc structure after solution treatment at 900 °C, as shown in Fig. 2(a). However, the microstructure showed a relatively non-homogeneous grain structure with large equiaxed and small grains with an average grain size of  $360 \mu\text{m}$ , as measured by the linear-intercept method (ASTM E112).

The XRD pattern of a solution-treated Ti-10Mn alloy is shown in Fig. 2(b). It can be seen that solution-treated Ti-10Mn exhibits a complete  $\beta$  phase with a bcc structure. It is reasonable to conclude that 10 wt% Mn is enough to retain a full  $\beta$ -phase structure at room temperature as it is a strong  $\beta$  stabilizer.

### 3.2. Flow Behavior

The typical true stress-strain curves of hot-deformed  $\beta$  Ti-10Mn alloy obtained at different temperatures and strain rates are presented in Fig. 3. It can be seen that the flow-stress curves of the alloy exhibit two distinct features. The first feature is a discontinuous yielding at early true strains, while the second is a pronounced periodic serration and fluctuation without strain hardening. A discontinuous yielding phenomenon was reported in several other  $\beta$  Ti-alloys, such as Ti-10V-4.5Fe-1.5Al, Ti-25V-15Cr-0.3Si, and Ti METAL LCB alloys [38–40]. It is apparent from the flow curves of the alloy at high temperatures that stress serrations became more obvious when the strain rate is  $1 \text{ s}^{-1}$  or

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