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Mechanical property and microstructure evolution of nitrogen-modified Ti-6Al-4V alloy with core-shell structure by hot compression

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ABSTRACT

Core-shell (CS) structured Ti-6Al-4 V (Ti64) alloy with single α shells and duplex $\alpha + \beta$ cores was fabricated by powder nitriding and spark plasma sintering. The mechanical property and microstructure evolution of CS-Ti64 alloy was investigated by compression at temperatures of 950–1100 °C and strain rates of 0.001–1 s^{-1}. The nitrogen-induced solid solution strengthening and the nitrogen-stabilized CS structure significantly enhanced the compressive strength. Microstructure evolution of CS-Ti64, involved inhomogeneous deformation of the core and shell, which was quite different from that of the commercial Ti64. Regions adjacent to shells and central locations of cores suffered large strain and therefore preferentially underwent dynamic recrystallization (DRX). The deformation mechanism of the novel CS-Ti64 alloy was discussed in terms of the α shells, α laths and DRX.

1. Introduction

Since titanium alloys exhibit specific strength-to-weight ratio and excellent corrosion resistance, they have successfully been applied in aerospace, ship, petrochemical and biomedical industries [1]. Ti-6Al-4 V (Ti64) alloy, a typical titanium alloy with $\alpha + \beta$ structure, is one of the most widely utilized titanium alloys for the benefit of the balanced presence of both α- and β-phase [2]. By means of various thermo-mechanical treatments and/or heat treatments, Ti64 with different grain sizes and $\alpha + \beta$ morphologies, such as lamellar/basket-weave, equiaxed, bimodal and Widmanstätten structure, can be acquired [3–6]. These easily obtained diverse microstructures enhance and extend the application of Ti64 in different fields.

In addition to microstructure manipulation, composite technology by reinforcements, including ceramic particles, whiskers and fibers, is commonly adopted to solve the problem of the shortage in properties of titanium alloys [7–10]. In past years, alloys prepared with innovative structural design have been put in place and successfully attracted interests for excellent properties and specific performances [11–16]. For instance, biomedical Ti-24Nb-4Zr-8Sn porous structure with designed porosity can be carefully produced with expectant low modulus, high biocompatibility, strength and corrosion resistance [11]. Equiaxed Ti-based composite with high strength and plasticity is prepared through mechanical alloying and spark plasma sintering (SPS) [13]. By TiBw reinforcements, Ti64 and Ti60 composite with network structure are obtained with enhanced superplasticity and strength in comparison to commercial counterparts [14,15].

Recently, novel core-shell (CS) structured titanium alloy with single α-phase, incorporation of α-phase shells into soft matrix, has been reported to possess high strength, excellent thermal stability and good plasticity [17,18]. Similar CS structured Ti64 alloy (CS-Ti64) can be synthesized with enhanced strength without sacrificing its plasticity [19]. However, the high-temperature deformation behavior of this CS titanium alloy, especially the deformation mechanism of CS structure, has not yet been well investigated. The aim of present work is to evaluate the hot deformation characteristics of CS-Ti64 by the flow stress and microstructure obtained through compression testing at duplex $\alpha + \beta$ region. The deformation mechanism of CS structure is also discussed.

2. Experimental Procedures

Commercial Ti64 powder with a particle size ranging from 150 to 300 μm was chosen as feedstock. The powder was first encapsulated in a porous stainless steel pot which could rotate and make a homogeneous nitriding. Ti64 powder together with the steel pot were then placed in a tube furnace and nitrided at 1000 °C for 15 min in a nitrogen atmosphere, followed by air cooling to room temperature. Subsequently, the nitrided powder was sintered by SPS in vacuum at 1200 °C for 5 min under a pressure of 45 MPa, to obtain full density compact. High-strength graphite die with 60 mm in inner diameter was employed in the sintering process. After sintering, the load was released and the

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sintered compact was furnace cooled to room temperature.

Cylindrical specimens with 8 mm in diameter and 12 mm in height were machined from the sintered compact. Hot compression tests were carried out by using a Gleeble-3800 thermal/mechanical simulator at temperatures of 950, 1000, 1050 and 1100 °C respectively and strain rates of 0.001, 0.01, 0.1 and 1 s^{-1}, respectively. Prior to compression tests, all specimens were fast heated to 1100 °C with a heating rate of 10 °C/s and soaked for 3 min, and then cooled to testing temperature at a rate of 5 °C/s. All specimens were compressed to 60% of the original height and subsequently cooled to room temperature within 10 s. In addition, heat treatment at 1100 °C for 3 min was carried out by Gleeble-3800 to acquire the microstructure before compression, adopting same heating and cooling procedure with compression testing.

For metallographic observation, the specimens were polished to 0.5 μm diamond finish and etched by the solution of 20 ml hydrofluoric acid +20 ml nitric acid +60 ml water. Specimens for electron backscatter diffraction (EBSD) were prepared by standard mechanical polishing with 0.5 μm diamond finish, followed by electrochemical polishing in a solution of 10 ml perchloric acid +70 ml ethyl alcohol +20 ml glycerine at room temperature. The step sizes used for EBSD collection of undeformed specimen and of deformed specimen were 1.35 μm and 0.5 μm, respectively. All of microstructures were collected from the center of deformed/heat treated samples by using optical microscope (OM), scanning electron microscope (SEM), electro probe (EP) and EBSD.

3. Results

3.1. Microstructure Before Compression

Fig. 1 illustrates the SEM and EBSD microstructure of the studied CS-Ti64 alloy before compression. It should be noted that β-Ti is ignored during EBSD collection and analysis, since the step size is much larger than the width of β lamella. As shown in Fig. 1a, heat treated specimen maintains a CS structure which is composed of net shells and equiaxed cores. Net distributed shells are about 16 μm in thick. The EBSD results (Fig. 1c–e) indicate that these shells are hexagonal close packed (hcp) α-Ti phase (a = b = 2.95 Å, c = 4.73 Å). That is, shells are with single α-phase. It also can be detected from Fig. 1d and e that shells are mainly composed of two-column granular α-phase. Similar to common titanium nitriding, nitrides and nitrogen-rich solution layers are formed on the surface during powder nitriding. Subsequent sintering at 1200 °C, dissolved nitrides and nitrogen diffusion result in the formation of α shells. More detailed formation mechanism of core-shell structure refers to our previous work [18,19].

The CS structure and α-phase formed shells indicate that the β transus temperature of the shells of the CS-Ti64 alloy is higher than 1200 °C, which is higher than the reported value (about 975 °C) of commercial Ti64 [4]. Therefore, all compression tests in the present work were performed at two-phase α + β field. Interestingly, the grain size is measured to be ~300 μm, which approaches to the powder particle size. Our previous work has concluded that the matrix grain growth is inhibited by the α shell [19], where the α-phase is stabilized by nitrogen in the synthesis process [18]. Understandably, the grain size is therefore associated with the powder size used.

As illustrated in Fig. 1, compared with the net shell composed of single α-phase, the equiaxed core resembles a lamellar structure with two-phase α + β. Cores are mainly composed of α + β colonies and encompassed by α shells. The volume fraction of α-phase is significantly higher than that of β-phase. Ultrafine-grained β lamellae with a thickness of ~0.5 μm are only found in the core and are embed between α laths. In addition to the granular α-phase in the shell, two types of α

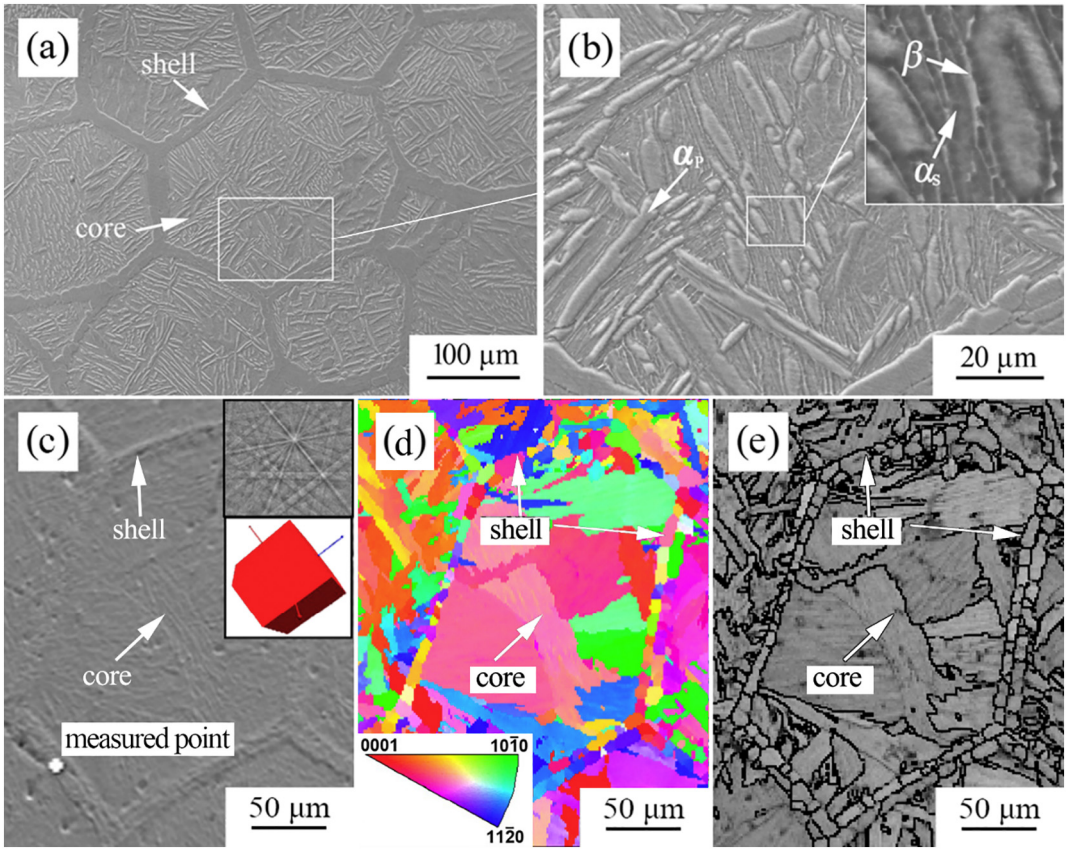

Fig. 1. Microstructure of heat treated CS-Ti64: (a) CS structure, (b) α- and β-phase in the core, (c) EBSD measured area and corresponding (d) orientation map and (e) image quality map. α_p is the primary α-phase and α_s is the secondary α-phase. In Fig. 1c, the Kikuchi lines and hexagonal structure sketch are detected from the location marked by white point.