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Isothermal deformation and spheroidization mechanism of (TiB + La_2O_3)/Ti composites with different initial structures

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ABSTRACT

(TiB + La_2O_3)/Ti composites with different initial structures were obtained through designed heat treatment. The isothermal deformation and spheroidization mechanisms of (TiB + La_2O_3)/Ti composites with different initial structures were investigated. Initial structures were refined as the heat treatment temperature decreased. Simultaneously, the quenching and reheating resulted in finer α lamella colonies and α interlamellar spacing compared to the furnace cooling. The flow resistance of furnace cooled samples was lower than the quenched and reheated samples in terms of flow stress data. Following the deformation at 920 °C, the furnace cooled samples demonstrated lamellar α phase and partial spheroidized structure. Moreover, the spheroidization mechanism was similar to the boundary splitting model. Recrystallized grains of approximately 1 μm in diameter were obtained in the quenched and reheated sample following deformation at 920 °C. The spheroidization mechanism was attributed to the development of finer needles through quenching and reheating, as well as to subsequent dynamic recrystallization during the hot deformation. Additionally, the dislocation movement was blocked by the reinforcements on account of the composites with micron TiB and submicron La_2O_3 during the hot deformation. Furthermore, the reinforcements could facilitate the dynamic recrystallization (DRX) of the α phase.

1. Introduction

With the rapid development of aerospace technology, the demand for lightweight, high-strength and heat-resistant alloy structural materials has increased [1]. A series of near α high-temperature titanium alloys have been developed, such as the IMI834 (UK) [2], Ti-1100 (USA) [3] and Ti-60 (China) [4] alloys. Nevertheless, the service temperatures of these titanium alloys are lower than 600 °C. One major problem, which limits the development of near α titanium alloy with a higher service temperature, is insufficient strength and creep resistance beyond 600 °C [5–7]. It is urgent to develop materials with a higher service temperature.

Recently, titanium matrix composites (TMCs) have gained wide attention in the field of high-temperature structural applications, due to their excellent high temperature strength, higher specific strength, higher specific modulus and good corrosion resistance [8,9]. In particular, through the near-α titanium alloy selection as the matrix, the composites presented an abrupt increase in high temperature strength [10]. The reinforcements of the titanium matrix composites are the hard ceramic particles, such as TiB and TiC, which contribute to the corresponding high modulus and outstanding thermodynamic stability [11–13]. Adversely, the reinforcements reduce the plasticity, causing difficulties in material processing [14]. As a result, these reinforcements inhibit the application of TMCs. Furthermore, the matrix alloy, which is a near α titanium alloy, exhibits more sensitivity to processing parameters as well as a more narrow processing window [15]. Therefore, it is necessary to understand the suitable processing parameters of TMCs with a near-α titanium alloy as the matrix.

The isothermal forging can effectively reduce the deformation resistance of materials, homogenize the microstructure, eliminate the microtexture, induce the dynamic recrystallization, as well as improve the utilization and reduce the machining cost [16]. Therefore, isothermal forging is considered as a significantly effective technology for the processing of titanium matrix composites. The processing map obtained from the hot compression experiment could provide a basis for the isothermal forging technology [17]. Substantial research on the

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deformation behavior of TMCs reinforced with TiB whiskers and TiC particles were conducted. Wang et al. [7] analyzed the compressive behaviors and mechanisms of TiB whisker-reinforced Ti60 alloy matrix composites with network microstructure, in the temperature range of 900–1050 °C and at the strain rate range of 0.001–1 s^{-1}. It was suggested that the flow softening was caused by globularization of α phase in α + β field. Also, in the β field, dynamic recrystallization (DRX) of β phase occurred. Ma [18] investigated the hot-deformation behavior of Ti-1100 composites reinforced with 5 vol% of TiC particles. It was reported that the variations in activation energy, resulting in different deformation mechanisms of the composites, were affected by TiC particles along with the variation in the α/β phase volume ratio. Peak flow stresses of (TiB + TiC)/Ti composites are higher compared to the unreinforced alloy, according to Zheng [19]. Although many works were focused on TMCs with TiB and TiC, these reinforcements were both in the micron scale. In the authors' previous work, the thermal stability as well as the tensile properties of the near α Ti alloy with micron TiB and submicron La_2O_3 were studied [20–22]. Few researches were conducted on the compressive behaviors and mechanisms of titanium matrix composites with multi-scale reinforcements.

The present works were focused on the effects of different hot deformation parameters on the deformation behavior as well as microstructure evolutions among all titanium alloys and titanium matrix composites compression experiments [7,18]. As it is well known, titanium alloys with different structures can be obtained through cooling rate changes from the β solutionizing temperature [23]. B. Poorganji et al. [24] studied the microstructure evolution of Ti-1.5Fe alloys with different initial structures during hot deformation. The results demonstrated that the titanium alloy quenched in the β phase region can refine the lamellar spacing and colony size, also promoting the dynamic recrystallization. As a result of the reinforcement addition, the hot deformation behavior of titanium matrix composites with different initial structures has not been well investigated. This influence law disclosure has been proved to contribute to the development of a new processing technology for this composite.

In this study, the microstructural evolutions and hot deformation behaviors of (TiB + La_2O_3)/Ti composites with different initial structures through isothermal deformation were researched. The microstructures of the composites obtained through different heat treatment processes were observed. Additionally, the microstructural effects on the recrystallization behavior of the composites were presented, while the spheroidization mechanism was analyzed.

2. Experimental Procedure

2.1. Initial Materials

The material used in this experiment was a Ti alloy reinforced with (TiB + La_2O_3). The nominal composition of the matrix alloy was Ti-6.6Al-4.6Sn-4.6Zr-0.9Nb-1.0Mo-0.32Si. The TiB and La_2O_3 were in situ synthesized according to the following reaction:

$$12Ti + 2LaB_6 + 3[O] = 12TiB + La_2O_3 \quad (1)$$

The cast ingot (Φ580 mm) of composites was melted three times in the vacuum consumable electrode furnace and consequently hot-forged into rods (Φ280 mm) in the β phase field. Following, the rods were hot-forged to Φ70 mm at the α + β phase region. The theoretical volume fractions of TiB and La_2O_3 were 1.82% and 0.58% respectively. Additionally, the β-transus temperature of the composites measured with the metallographic method was approximately 1040 °C.

The optical micrograph of an as-forged (TiB + La_2O_3)/Ti composites is presented in Fig. 1. The initial microstructure consisted of approximately 85% of elongated α phase within the transformed β matrix and the TiB was oriented along the forging direction. Besides, in the authors' previous work, submicron La_2O_3 particles of 200–700 nm in size were dispersed in the composites [17].

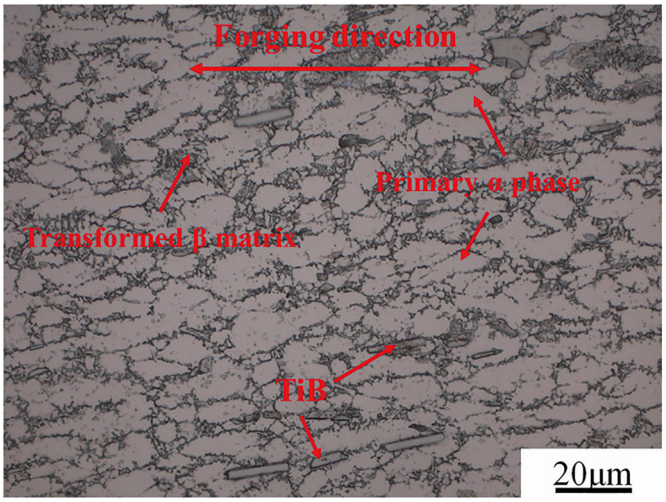

Fig. 1. Optical micrograph of as-forged (TiB + La_2O_3)/Ti composite.

2.2. Heat Treatments

Φ8 mm × 12 mm cylindrical specimens were obtained through machining from the forged bar with their axis parallel to the forging direction. In order to obtain different initial structures, two heat treatment (HT) processes were designed, which were called “furnace cooling” and the “quenching and reheating” processes (Fig. 2). Two groups of samples were processed through the heat treatment routes. Then those samples were directly quenched by water to obtain the initial structures of composites prior to deformation.

- HT 1: In the furnace cooling process, the samples were solution heat treated at 1060 °C in the β phase region for 30 min and consequently furnace cooled (FC) to the deformation temperatures (920 °C, 980 °C and 1020 °C) for 15 min.
- HT 2: In the quenching and reheating process, the samples were water quenched (WQ) following the same solution heat treatment. The quenched samples were reheated to the deformation temperatures (920 °C, 980 °C and 1020 °C) and isothermally retained at these temperatures for 15 min.

2.3. Hot Compression Tests

The hot compression tests were carried out with a Gleeble 3500 thermo-mechanical simulator with Ar as the protective gas, while the heating rate was 5 °C/s. Graphite foils were placed between the specimens and the anvils in order to minimize the friction effects during testing. In previous works [17,25–27], the high reductions of the samples were 60% or 70%. The dynamic recrystallization behaviors of materials could be well observed under this deformation. Therefore, a 60% reduction was designed for this study. When the samples were compressed to the height reduction of 60% (equal to the true strain of 0.92), they were immediately water quenched.

2.4. Microstructural Observations

Microstructural observations were performed with an optical microscope (OM, ZEISS AxioCam MRc5), a scanning electron microscope (SEM, Sirion 200), a JEM 2100 transmission electron microscope (TEM) and a VEGA 3 XMU electron backscatter diffractometer (EBSD). For OM and SEM observations, the compressed samples were sectioned parallel to the compressive direction and consequently embedded, mechanically polished and etched (HF:HNO_3:H_2O = 1:3:10). TEM samples were cut from the centers of the compressed specimens and mechanically ground to about 50 μm in thickness. The electron transparent regions were