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ENHANCING MICROSTRUCTURAL CHARACTERISTICS AND MECHANICAL PROPERTIES OF Ti-Al-Dy ALLOY THROUGH BALL MILLING AND SPS CONSOLIDATION

In this study, pure Ti, Al, Dy powders were ball-milled with zirconia balls in argon atmosphere for 3 h at 800 rpm, producing a Ti- 6 wt.% Al- 4 wt.% Dy alloy powder. The alloy powder was consolidated by SPS technique at 1373 K for 15 min under 50 MPa pressure in vacuum. The sintered body had approximately 99% in density and 6 μm in grain size. XRD and TEM revealed the presence of the second phases such as Ti_3Al, Al_3Dy and $Ti_4Al_{20}Dy$ phases, and their sizes were approximately 50 nm. Micro-hardness was approximately 960 Hv at room temperature, which decreased as temperature increased. However, there remained the micro-hardness significantly higher compared to the commercial Ti-6Al-4V alloy. After hardness test at 1173 K, XRD analysis did not show any difference in peaks, while the grain size and second phases size increased by ~4 μm and ~2 nm, respectively.

Keywords: High energy ball milling; SPS; Ti-Al-Dy; High temperature hardness; Microstructure

1. Introduction

Titanium-based materials have been widely used in aerospace, automobile, and biomedical industries due to their unique properties, such as high specific strength, excellent creep and fatigue properties, low modulus of elasticity, high temperature strength, and good thermal stability [1-3]. Adding rare earth elements on TiAl alloy can increase its ductility, oxidation resistance, and hardness at high temperature [4-6]. Recently, a method was developed to successfully extract Dy from discarded (Nd,Dy)FeB magnets [7,8]. This led to investigation on the microstructure and micro-hardness of titanium alloys with Dy added as an alloying element [9]. However, not much has been known about the effects of Dy on mechanical properties of Ti alloys.

Ti alloys have been generally manufactured by arc melting and powder metallurgy processing methods. Among powder metallurgy processing, high energy ball milling is an economic, simple, and effective method to obtain nanocrystalline structure and amorphous materials [9-13]. Additionally, spark plasma sintering (SPS) method has been used to process new materials such as nano phase materials [14] and intermetallic compounds [15] due to rapid heating rate, short holding time and low temperature. These unique features lead to high relative density, mechanical characteristics, and fine microstructure [9,16,17].

In this study, therefore, pure Ti, Al and Dy powders were high energy ball milled and subsequently SPSed to investigate the micro-hardness at high temperature and the change of microstructure after the high temperature hardness test.

2. Experimental

Ti, 6 wt.% Al and 4 wt.% Dy powders were high energy ball milled with zirconia ball at 800 rpm for 3 h in argon atmosphere [9]. The obtained alloy powders were consolidated by SPS at 1373 K for 15 min under 50 MPa pressure [9]. The microstructures of the powders and sintered sample were observed by using scanning electron microscope (SEM). Image analysis was used to measure the mean size and shape factor of the powders. The relative density of sintered sample was obtained by Archimedes' principle. The micro-hardness was carried out under a load of 1 kg for 10 sec. The high temperature hardness test was measured from 573 K to 1173 K at argon atmosphere. X-ray diffractometer (XRD) and transmission electron microscope (TEM) were utilized to analyze the phases of the sintered sample. The size of second phases and particles was measured by TEM and calculated by measuring full width at half maximum (FWHM) ($=0.9\lambda/\beta\cos\theta$, where λ is x-ray wavelength, θ is Bragg angle, β is line broadening in radians) in XRD peaks.

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3. Results and discussion

The mean sizes of initial Ti, Al, and Dy powders used in this study were approximately 20, 40, and 200 μm, respectively [9]. Fig. 1 presents the SEM and TEM images of as-milled powders. These powders showed a mean size of 8 μm and a shape factor of 0.8, consisting of fine particles ranging from 10 to 20 nm in size. The EDS image shows that the components of the initial powder were well distributed after high energy ball milling.

Fig. 2(a) depicts the appearance of as sintered sample which was cylindrical shape with a diameter of 10 mm and a height of 5 mm. It showed densified microstructure with a relative density of approximately 99%, achieved through a rapid densification process that effectively minimized grain growth and the presence of second phases by enhancing sinterability. The sample had a grain size of 6 μm, as observed through SEM (Fig. 2(b)) and contained round shaped second phases measuring approximately 50 nm in size, as revealed by TEM (Fig. 2(c)). In addition, from diffraction pattern (Fig. 2(d)), it was confirmed that there exists new ternary phase of $Ti_4Al_{20}Dy$. We previously reported that two secondary Ti_3Al and Al_3Dy phases were detected and two ternary Ti-Al-Dy phases could be formed after high energy ball milling and SPS [9]. In this study, accordingly, the existence of Ti_3Al, Al_3Dy and $Ti_4Al_{20}Dy$ phases is reasonably understood.

In Fig. 3, the change in micro-hardness of as sintered Ti-Al-Dy alloy with increasing temperature is presented and compared to that of the commercial Ti-6Al-4V alloy. The micro-hardness of Ti-Al-Dy alloy was approximately 960 Hv at room temperature, which was higher than 340 Hv of the commercial Ti-6Al-4V alloy [18]. This difference can be attributed to the presence of fine second phases such as Ti_3Al, Al_3Dy and $Ti_4Al_{20}Dy$ as depicted in Fig. 2 and supported by previous results [9]. As the temperature increased to 1173 K, the micro-hardness of commercial Ti-6Al-4V alloy decreased to approximately 10 Hv [18]. In contrast, the Ti-Al-Dy alloy maintained a macro-hardness hardness of approximately 310 Hv at 1173 K, which was similar to the hardness of commercial Ti-6Al-4V alloy at room temperature.

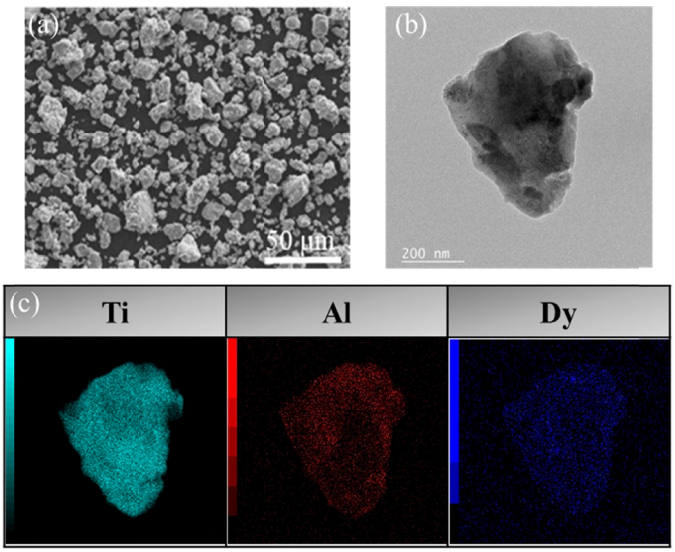

Fig. 1. Images of (a) SEM, (b) TEM and (c) EDS of as milled powders

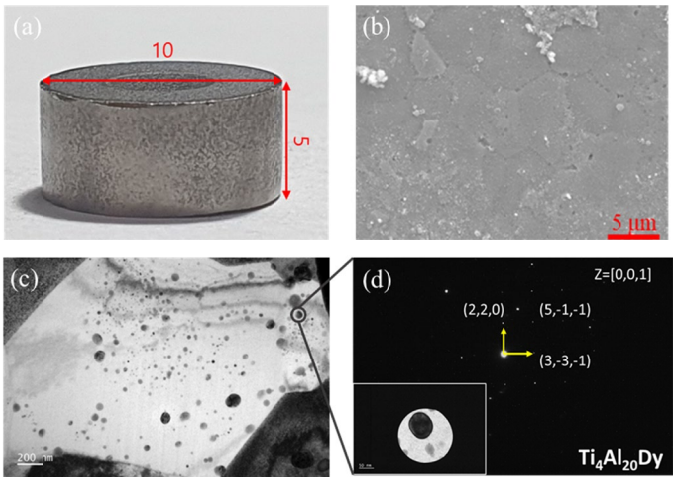

Fig. 2. (a) Appearance, (b) SEM image, (c) TEM image and (d) diffraction pattern of as sintered sample

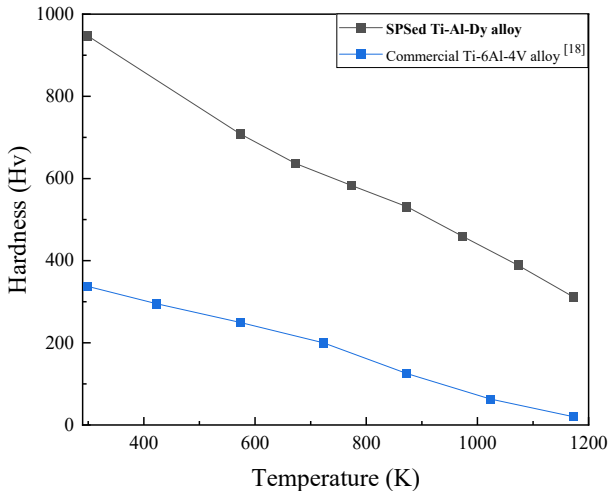

Fig. 3. Change in micro-hardness of as sintered Ti-Al-Dy alloy with increasing temperature

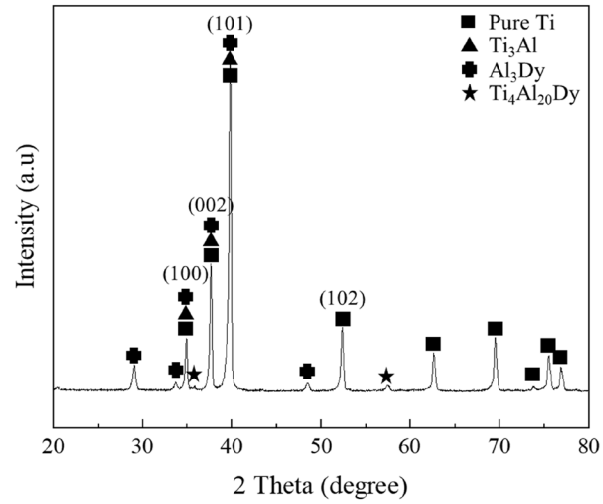

Fig. 4. XRD result of as sintered Ti-Al-Dy alloy after hardness test at 1173 K

On the other hand, Fig. 4 reveals the XRD pattern of Ti-Al-Dy alloy after micro-hardness test at 1173 K. There are no differences in the peaks before and after the high temperature hardness test. However, as shown in Fig. 5, the grain size increased from 6 μm to 10 μm and second phases size also become larger to 52 nm after high temperature hardness test.

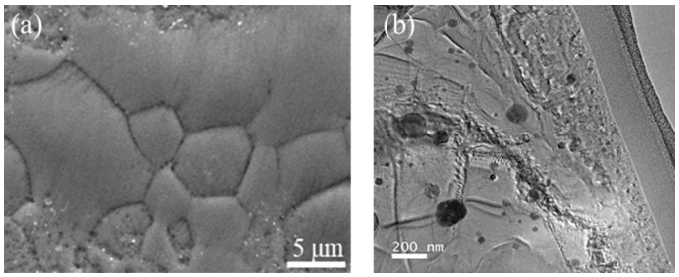

Fig. 5. (a) SEM and (b) TEM images of as sintered Ti-Al-Dy alloy after hardness test at 1173 K

Consequently, the decrease of micro-hardness with increasing temperature is considered to stem from the coarsening of grain and second phases. Nevertheless, the reason why the Ti-Al-Dy alloy showed micro-hardness of more than 300 Hv, compared to the commercial Ti-6Al-4V alloy at 1173 K, is due to the existence of second phases which is thermally stable.

4. Conclusions

Ti-6 wt.% Al-4 wt.% Dy powder was ball-milled with zirconia balls in argon atmosphere for 3 h at 800 rpm, resulting in powder size of ~8 μm, particle size of 10 ~ 20 nm. The ball-milled powder was consolidated by SPS under pressure of 50 MPa at 1373 K for 15 min to produce a sample of 99% in density and 960 Hv in micro-hardness. From the XRD and TEM analyses, it was confirmed that the binary Ti_3Al, Al_3Dy and ternary $Ti_4Al_{20}Dy$ phases were newly formed. The micro-hardness of as sintered Ti-Al-Dy alloy was approximately 960 Hv, which was three times higher than that of commercial Ti-6Al-4V alloy. Although, the micro-hardness decreased to about 310 Hv with increasing temperature to 1173 K, it remained similar to the micro-hardness of the commercial Ti-6Al-4V alloy at room temperature. After high temperature hardness test, XRD analysis did not show any difference in peaks. However, the sizes of grain and second phases both showed a slight increase – ~4 μm for grain ~2 nm for second phase, resulting in the decrease of micro-hardness with increasing temperature.

Acknowledgments

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