Fabrication of W-Nb-Mo-Ta-V High Entropy Alloys by Mechanical Alloying and Spark Plasma Sintering

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1. Introduction

High entropy alloys (HEA) have been defined as multicomponent alloys composed of more than 5 constituent elements and the composition of each element is in the range of 5 to 35at.%. [1] Recently, high entropy alloys have been studied with increasing attentions because HEAs have unique characteristics such as high entropy of mixing, solid solution hardening, and lattice distortion. [1-3] Therefore, various alloy systems of HEAs are being investigated for various applications including high strength alloys, refractory alloys, cryogenic alloys, etc. [2, 3]

Among those HEAs, W-Nb-Mo-Ta and W-Nb-Mo-Ta-V systems have presented outstanding hardnesses and high-temperature strengths [4,5]. Therefore, those refractory alloys can be promising high temperature structural materials for future nuclear energy systems. Senkov *et al.* fabricated those systems by vacuum arc melting at a high temperature of 2700ºC [4]. However, HEAs fabricated by mechanical alloying (MA) can be an interesting study because lower temperature processing might be possible.

While the vacuum arc melting has been reported as a primary method for fabrication of single phase HEAs, powder metallurgical (PM) processing has not been reported for the W-Nb-Mo-Ta-V HEA system yet. Generally, it is reported that PM processed HEAs prepared by MA consist of multiple phases after sintering [6].

In this study, a W-Nb-Mo-Ta-V alloy was fabricated for HEAs by using Spark Plasma Sintering (SPS). The microstructure of sample was observed by Scanning Electron Microscopy (SEM) and the crystalline structures and chemical compositions of mechanically alloyed and sintered sample were measured by X-ray diffraction (XRD) and energy dispersive spectroscopy (EDS), respectively. The mechanical property of the sintered sample was measured by Vickers microhardness.

2. Experimental Procedure

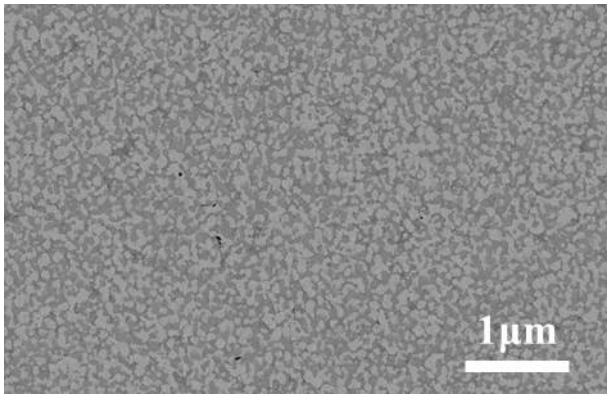
Five different elements of powders; W, Nb, Mo, Ta, and V, were mixed to prepare a HEA. Well mixed powders were ball milled for 30 hours by planetary ball milling in an Ar atmosphere to protect from oxidation. Mechanically alloyed powder was sintered by SPS at 1700ºC at a heating rate of 100ºC/min. Boron nitride was used as a lubricant and it also reduced carbon contamination. The sintering atmosphere was set at a vacuum level less than 1.5×10^{-2} torr. The diameter of cylindrical samples was 1.3cm. Sintered pellets were polished mechanically to remove surface areas which may have been contaminated by the graphite mold. The microstructure and XRD patterns were characterized to analyze the crystalline structures of HEAs after MA and SPS. Vickers microhardness was measured on polished surfaces with 500 g load in 10 different locations.

3. Results

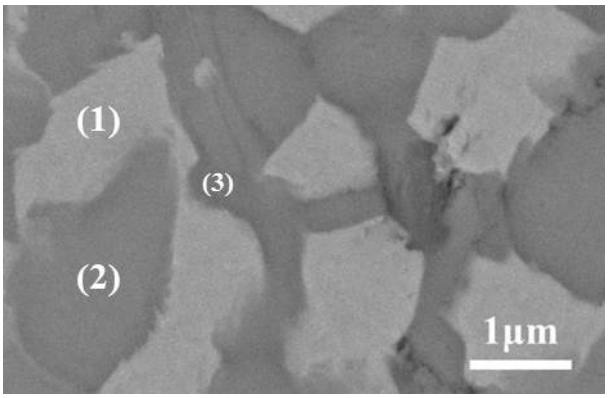
3.1 Microstructure

An SEM image of the sintered pellet was shown in Fig. 1(a) and (b). Three kinds of different phases were observed in the HEA sample. Bright (1), grey (2), and dark (3) phase were identified as W-rich, Ta-rich, and well mixed phases by EDS, respectively (Fig. 1(b)). While the initial elements primarily reacted for intermetallic compounds (IMC) during the vacuum arc melting [7], mechanically alloyed powder did not show diffraction peaks for IMC, but the BCC diffraction peaks were broaden due to the MA [6].

The grain size of the sintered alloy was approximately 1μm, which was 20 to 200 times smaller than that of reported vacuum arc melted HEAs [4, 5].



(a)



(b)

Fig. 1. An SEM image of spark plasma sintered HEA.

3.2 XRD analysis

Fig. 2 shows the XRD patterns of before and after SPS. Mechanical alloyed powder was identified as having a BCC structure. It means the powder might be already fabricated as a HEA by MA. The average mixing enthalpy of the W-Nb-Mo-Ta-V is -4.6 kJ/mole and atomic size difference parameter "δ" is 3.0 which are suitable to form HEAs. The enthalpy of mixing and the parameter δ are identified as ones of the most important factors to fabricate various HEAs. [2] X-ray peaks of either mechanical alloyed powder and the sintered pellet were matching BCC structures, respectively. However, two kinds of BCC peaks were overlaped in the XRD patterns of the sintered sample.

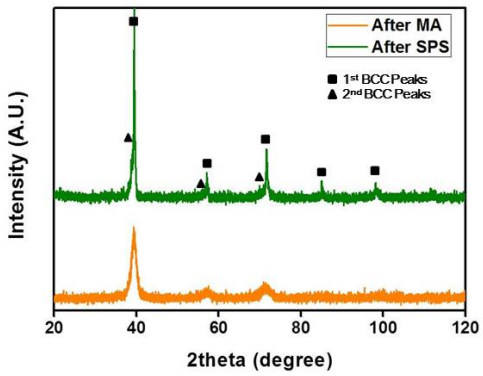


Fig. 2. XRD peaks of the HEA samples before and after sintering.

3.3 Vickers Hardness

The average value of hardness was measured 10.8 GPa, which is approximately 7 times larger than the hardness value calculated from the rule of mixtures, 1.6 GPa. This high hardness is attributed to solid solution hardening and grain size refinement.

4. Conclusions

A W-Nb-Mo-Ta-V HEA was fabricated by MA followed by SPS. The sintered HEA consisted of three different phases which are characterized as W-rich, Ta-rich, and well mixed phases. Two different kinds of BCC peaks were overlaped in the XRD patterns of the sintered sample. The average grain size of the sintered HEA was approximately 1μm, which are 20 ~ 200 times smaller than that of vacuum arc melted HEAs. The measured hardness value of the HEA pellet was 7 times larger than the hardness value from the rule of mixtures.

Acknowledgments

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