Transactions of the Korean Nuclear Society Autumn Meeting
Gyeongju, Korea, October 29-30, 2015

Mechanical Properties of Refractory High Entropy Alloys Fabricated by Powder Processing

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1. Introduction

Global research activities on high entropy alloys are increasing rapidly as these new alloys provide possibilities to the development of advanced engineering materials in addition to novel functional materials [1-7]. Generally, high entropy alloys are defined as multicomponent alloys with more than five principal elements with a significant atomic fraction of each element, ranging from 5 at.% to 35 at.%. Among many materials properties, very high specific strengths, high temperature compressive strengths, cryogenic toughnesses are most remarkable highlights for some representative high entropy alloys including CoCrFeMnNi [2]. The effects of high configurational entropy, lattice distortion and sluggish diffusion are attributed to the distinguishable behavior of high entropy alloys. The structural applications of high entropy alloys are also promising in advanced nuclear energy systems for nuclear fission and fusion applications. Because of the randomly occupied lattice points by atoms with different atomic radius, lattice distortions and local atomic level strain were developed. The local lattice distortions influence the mechanical properties of high entropy alloys. The strengthening of high entropy alloys is attributed to the lattice distortions and local atomic level strain that increase the resistance to the dislocation motion. Some high entropy alloys exhibit remarkable irradiation resistance. Nagase *et al.* reported that the CoCrCuFeNi alloy was irradiation resistant up to 40 dpa [8]. Egami proposed that the irradiation defects can be self-healed because the recrystallization happens more easily in high entropy alloys [9].

However, fundamental understandings of the deformation mechanisms and irradiation stabilities are deficient seriously, considering the worldwide active research on structural high entropy alloys.

Among many high entropy alloy systems, refractory alloy systems are strong candidates for high temperature structural applications for GEN-IV nuclear energy systems or thermonuclear fusion reactors [9]. Metallic elements with a high melting point high than 2000°C including W, Nb, Mo, Ta, and Hf are considered major alloying element for the refractory high entropy alloys [10 - 12]. Recently, Senkov *et al.* have reported that WNbMoTaV provides extraordinary high temperature strengths when compared with Ni-base superalloys [4, 5]. However, the arc-casted WNbMoTaV alloys were very brittle and the ductility was only approximately 2%. When they cast TaHfNbZrTi, the ductility was increased significantly up to 50% at room temperature, while the high temperature strength has been decreased [12]. Kuk *et al.* reported powder metallurgy processed WNbMoTaV by mechanical alloying and spark plasma sintering [13]. The Vickers hardness of the Powder Metallurgy (P/M) processed samples was much higher than that of the cast WNbMoTaV high entropy alloy with the equiatomic compositions.

In this study, the mechanical properties of the P/M refractory high entropy alloys were characterized by the instrumented nanoindentation technique.

2. Experimental Procedures

A quinary alloy of W, Nb, Mo, Ta, and V and a senary alloy of W, Nb, Mo, Ta, V and Ti were fabricated by mixing of elemental powder and mechanical alloying. Mixed powders were mechanically alloyed for 30 hours by planetary ball milling in an Ar atmosphere. Spark plasma sintering was used to consolidate the mechanically alloyed powder into disk type bulk samples. The sintering of high entropy alloy samples was conducted at temperatures up to 1700°C under vacuum. The microstructures were observed by Scanning Electron Microscopy (SEM) and the crystalline structures were identified by X-ray diffraction (XRD), and the compositions of each phase were characterized by energy dispersive spectroscopy (EDS). The average hardness of each sample were measured by Vickers microhardness with 500 g load.

Instrumented nanoindentation was conducted on sintered samples at room temperature to characterize the mechanical properties of each phase in the high entropy alloys. Indentations were made using MTS-XP nanoindenter with a Berkovich diamond tip with a tip radius of 50 nm, loading time of 15 s, loading rate of 2 nm/sec and the indentation depth of 500 nm. The nanoindentation hardnesses and Young's moduli of refractory high entropy alloy samples were measured with the indentation depth and compared with their Vickers hardness data.

3. Results

Fig. 1 is a typical load-displacement (P-*h*) curve of a equiatomic WNbMoTaV high entropy alloy meausured by nanoindentation. The mean hardness of the quinary high entropy alloy from unload was 14.3 ± 3.8 GPa and the mean elastic modulus from unload was 314.2 ± 28.1 GPa (see Fig. 2). The hardness of the quinary equiatomic WNbMoTaV high entropy alloy via vacuum arc melting has been reported as 5.25 GPa [4]. The huge deviation indicated that the mechanical properties of P/M high entropy alloys might be different from those of cast high entropy alloys. Kuk *et al.* reported the average grain size of the equiatomic WNbMoTaV high entropy alloy fabricated by P/M processes ranged approximately 1 μm, which is much finer than that of the cast high entropy alloy with the same composition. The increase fine microstructure was attributed to its finer microstructure, in addition to basic solid solution strengthening effects. The hardness of the quinary equiatomic high entropy alloy is not only higher than that of literature fabricated via vacuum arc melting (5.25 GPa), but also the highest value of the present metallic high entropy alloys to the best of authors' knowledge.

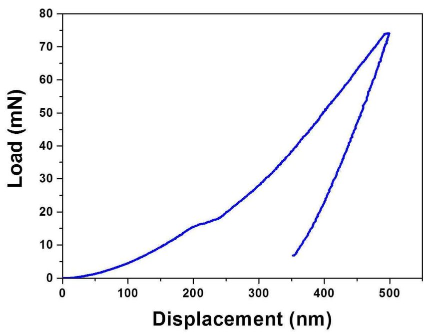

Fig. 1. A load-displacement curve of a quinary equiatomic WNbMoTaV high entropy alloy meausured by nanoindentation.

The mean hardness of the senary high entropy alloy from unload was 8.0 ± 0.7 GPa and the mean elastic modulus from unload was 212.7 ± 14.2 GPA (see Fig. 3). The hardness and elastic modulus of the senary high entropy alloy were decreased when compared with the quinary high entropy alloy because of the addition of Ti.

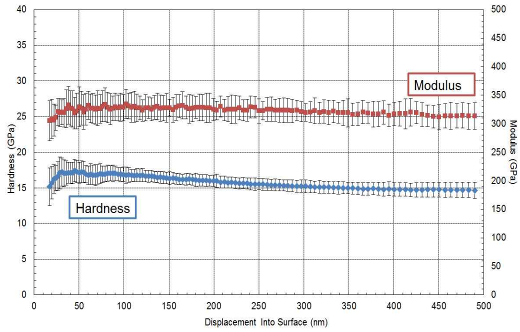

Fig. 2. The average hardness and average modulus of a quinary equiatomic WNbMoTaV high entropy alloy meausured by nanoindentation.

The hardness value of nanoindenter tends to decrease from surface and then saturated at approximately 500 nm. This is attributed to the nano-scale strengthening effect. The hardness values obtained by nanoindentation are compared with those by Vickers hardness tests as presented in Fig. 4. The Vickers hardness of the WNbMoTaV high entropy alloy was 10.9 ± 0.22 GPa and that of the WNbMoTaTiV high entropy alloy was 5.2 ± 0.09 GPa, respectively. The hardness value of Vickers was lower than that of nanoindenter due to the nano-scale strengthening effects.

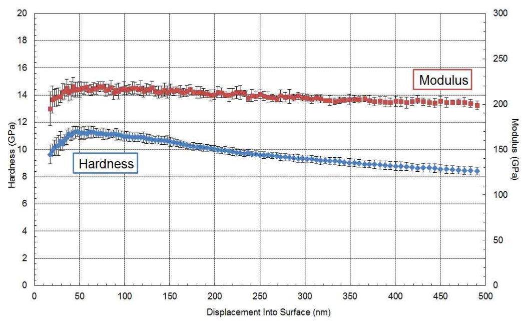

Fig. 3. The hardness and modulus of a senary equiatomic WNbMoTaVTi high entropy alloy meausured by nanoindentation.

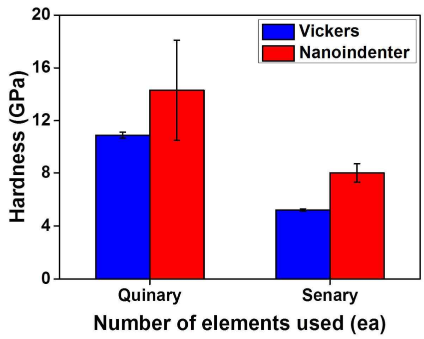

Fig. 4. The comparison of the Vickers hardesses and nanoindentation hardnesses of WNbMoTaV and WNbMoTaVTi high entropy alloys.

4. Summary

A quinary WNbMoTaV and senary WNbMoTaViTi high entropy alloys were fabricated by powder metallurgical processing. The mechanically alloyed and sintered samples have a much smaller grain size than that in cast high entropy alloys. When the mechanical properties of the refractory high entropy alloys were investigated by instrumented nanoindentation, very high hardness values of quinary WNbMoTaV were obtained when compared with arc-cast WNbMoTaV alloys. When Ti is added to the quinary WNbMoTaV high entropy alloys, the hardness of the senary WNbMoTaVTi high entropy alloy was decreased.

Acknowledgment

This study was supported by the National Research Foundation of Korea (NRF) grant funded by the Ministry of Science, ICT & Future Planning (NRF-2015R1A2A2A01002436).

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