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Stable nanocrystalline NbMoTaW high entropy alloy thin films with excellent mechanical and electrical properties

Xiaobin Feng, Jinyu Zhang *, Ziren Xia, Wei Fu, Kai Wu, Gang Liu *, Jun Sun *

State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an 710049, PR China

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ABSTRACT

In this work, we systematically investigated the hardness, electrical resistance and thermal stability of nanocrystalline NbMoTaW high entropy alloy films with thickness varying from about 100 to 2000 nm. The nanocrystalline high entropy alloy film exhibited the peak hardness (~16.0 GPa), strong size-independent electrical resistance (~170 μΩ·cm) as well as stabilized single phase body-centered cubic (BCC) structure even after exposed to vacuum annealing at 800 °C for 2 h. In the meantime, we provide reasonable insights into understanding behaviors above. With superior mechanical and electrical properties as well as good thermal stability, this kind of refractory nanocrystalline HEAFs will find extensive potential applications.

1. Introduction

High entropy alloys (HEAs), the newly surged concept, have gained much attention and set the trend in recent years. Compared with conventional alloys based on one primary element, each element of HEAs has the same possibility to occupy the site, resulting in severe lattice distortion for their different atom radius [1]. To some degree, the severe lattice distortion may render increased strength as well as a change in electrical resistance [2]. Due to the unique high-entropy effect and the sluggish diffusion effect, high entropy alloys present a distinguished thermal stability [1]. These excellent core effects benefit HEAs as promising candidates for high-strength and high-temperature sustaining alloys and diffusion barriers materials.

Limited studies to date indicate that the severely distorted lattice and high thermal stability associated with sluggish diffusion rates make the HEAs, in particular the refractory HEAs in the form of thin films/lines, appealing candidates for diffusion barrier or electrical resistance materials in micro/nanodevice [3–5]. For instance, the AlMoNbSiTaTiVZr high entropy alloy film (HEAF) is determined to prevent copper-silicide formation up to 700 °C for 30 min [3]. However, the contribution to electrical resistance of the HEAF itself to the whole Cu/HEA/Si sandwich structure and the underlying reasons remain unclear. Therefore, it is critical to know the electrical properties as well as thermal stability of refractory HEAFs and to what extent the characteristic sizes of HEAFs could affect their electrical resistance so as to prepare a prominent nanocrystalline (NC) NbMoTaW HEAF.

2. Experimental

A series of the nominally equiatomic $Nb_{25}Mo_{25}Ta_{25}W_{25}$ HEAFs with various thicknesses (t) from about 100 to 2000 nm were deposited on (1 0 0)-Si substrates by using the direct current magnetron sputtering. Nanoindentation experiments were conducted using a TI950 TriboIndenter (Hysitron, Minneapolis, MN) with a standard Berkovich tip. Details about the nanoindentation measurements refer to previous work [6]. The sheet resistance of the HEAFs with different thicknesses was measured by a standard four-probe technique at room temperature (300 K). Each sample was measured at least 9 times at different locations, and the measurement error was less than 3%. The as-deposited 250 nm-thick NbMoTaW HEAF was vacuum-annealed at 800 °C for 2 h. The pressure was below 5×10^{-4} Pa to avoid oxidation and the heating rate was 5 °C/min. X-ray photoelectron spectrometer (XPS) measurement was performed to detect the in-depth composition of annealed HEAF with Ar ion etching for 5, 15, 20, 25, 30 min. The etching rate was estimated to be 0.13 nm/s of the guide sample SiO_2. In order to work out the phase structure and crystallographic orientations of both as-deposited and post-annealed HEAFs, X-ray

* Corresponding authors.
E-mail addresses: jinyuzhang1002@mail.xjtu.edu.cn (J. Zhang), lgsammer@mail.xjtu.edu.cn (G. Liu), junsun@mail.xjtu.edu.cn (J. Sun).

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diffraction (XRD) experiments were carried out on a Bruker D8 Discover powder X-ray diffractometer with Cu K_α radiation at room temperature. JEM2100 high-resolution transmission electron microscopy (HRTEM) with 200 kV accelerating voltages was used to observe the inner structure of both the as-deposited and post-annealed HEAF. Oxford instrument energy-dispersive X-ray (EDX) detector was employed with a spatial resolution of ~1 nm for chemical analysis. A minimum of 100 grains were measured to determine the average grain sizes (*d*) of the HEAFs.

3. Results and discussion

XRD spectra of the as-deposited and post-annealed 250 nm-thick HEAFs are displayed in Fig. 1(a). Both spectra show a strong (1 1 0), weak (2 0 0) and (2 1 1) peaks, which infers an unchanged single phase BCC structure. To be noted, diffraction peaks of the post-annealed film shift to right implying that a stress relaxation may take place after the heat treatment. The XPS etching spectra of post-annealed sample in Fig. 1(b) show that relative contents

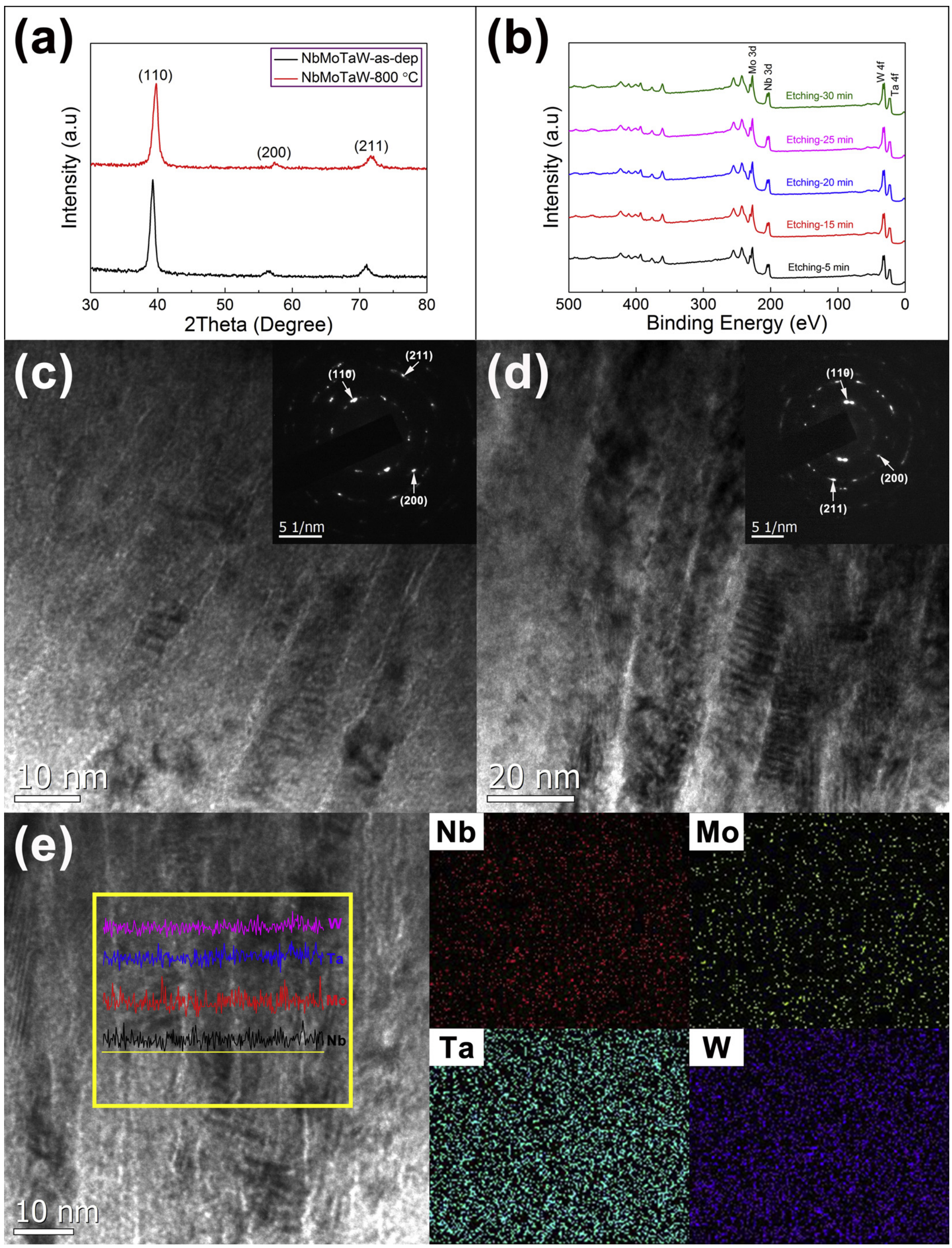

Fig. 1. (a) XRD patterns of as-deposited and post-annealed NbMoTaW HEAFs. (b) XPS spectra of annealed HEAF with Ar ion etching for 5, 15, 20, 25, 30 min. (c) and (d) are HRTEM images of as-deposited and post-annealed HEAFs respectively, inserted in corresponding SADPs. (e) The line/area scanning images of the selected yellow line/square of the post-annealed HEAF.