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Short communication

Effect of the addition of nano-Al_2O_3 on the microstructure and mechanical properties of twinned $Al_{0.4}FeCrCoNi_{1.2}Ti_{0.3}$ alloys

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ABSTRACT

This manuscript reports the synthesis of nanoscale Al_2O_3-reinforced $Al_{0.4}FeCrCoNi_{1.2}Ti_{0.3}$ high entropy alloy composites (HEACs) by mechanical alloying (MA) and spark plasma sintering (SPS). The effects of 12 wt% Al_2O_3 on the microstructure and mechanical properties were investigated. A major FCC phase along with BCC and Al_2O_3 were observed after SPS. Many deformation twinnings were found in HEACs from the SAED pattern. In the composite with 12 wt% Al_2O_3 quality fraction, the fracture strength, plastic strain and Vickers hardness of HEACs were as high as 2250 ± 10 MPa, 20.0 ± 0.50% and 743 ± 12Hv, respectively.

1. Introduction

The emergence of high-entropy alloys (HEAS) has changed traditional strategies for generating practical alloys. HEAs are termed “high entropy” because their mixing leads to high entropy ($11 \leq \Delta Smix \leq 19.5\ J\ K^{-1}\ mol^{-1}$) when more than five elements are mixed in nearly equi-molar proportion [1]. Such alloys have exhibited interesting properties such as high hardness, better oxidation resistance, and better corrosion resistance than traditional alloys. For HEAs, the properties of the alloys can be different from any of the constituent, individual elements [2,3].

Metal matrix composites (MMCs) have been developed in order to combine the advantageous properties of both ceramics and metals into a single material. Compared to high-entropy alloys, some of these with a single-phase face-centered cubic solid solution.

shows exceeding fracture toughness but a lower hardness [4], and therefore, there are few reports on high-entropy alloy composites (HEACs) [5]. Bulk materials that have good homogeneity can be easily fabricated with powder metallurgic processes and may be an ideal way to prepare HEACs. Although some HEACs have been developed, the fabrication of nanoscale Al_2O_3 reinforced non-equiatomic $Al_{0.4}FeCrCoNi_{1.2}Ti_{0.3}$ HEACs has not been reported. For this particular alloy, we hypothesized that the additions of nanoscale Al_2O_3 and more Ni would endow high compressive strength and high ductility [2]. In this manuscript, a systematic study was carried out to understand the effects of nanoscale Al_2O_3 on the microstructure and mechanical properties of HEACs synthesized by the powder metallurgic process.

2. Experiment details

Al_2O_3-reinforced $Al_{0.4}FeCrCoNi_{1.2}Ti_{0.3}$ was prepared dry milling the mixed elemental powders for 50 h. Nanoscale Al_2O_3 (20 nm, 12 wt%) and elemental powders Fe, Ni, Cr, Co, Ti and Al (>99.9 wt%, ≤45 μm) were processed by a high energy planetary ball milling machine (QM-3SP2) at 350 rpm under Ar atmosphere. The ball to powder weight ratio was 10:1. Milled powder was consolidated by SPS (Dr. Sinter 825) at 1000 °C for 10 min with a pressure of 30 MPa under vacuum.

The phases of HEAC were analyzed by a Bruker D8ADVANCE X-ray diffractometer (XRD) with a Cu Kα-radiation, and microstructural analysis was employed by a Zeiss Supra 40 (Carl Zeiss NTS GmbH) scanning electron microscope (SEM). For the

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microstructure observation, the samples were polished and then etched with aqua regia solution. Thin-foil specimens were prepared and observed under a transmission electron microscope (JEM-2100), during which selected area electron diffraction (SAED) was also performed. the room-temperature compressive properties of the cylindrical samples (Ø3mm × 4.5 mm in size) were evaluated by an Instron 5500 system. Hardness measurement was conducted with a Digital MicroHardness Tester (HVS-1000).

3. Results and discussion

3.1. Microstructure

SEM micrographs of bulk HEAC are shown in Fig. 1. The HEAC had a high relative density of up to 98.65% (measured by Archimedes method). Grain boundaries were not clearly observed in a low magnification image due to the fact HEACs were nanocrystalline (Fig. 1a). A higher magnification image of the alloy is shown in Fig. 1b. A few nano-sized spherical alloy phases were present marked by white arrows which detected by EDS. The size and shape of these phases can be mainly attributed to the short-time isostatic pressure and the complex pulsed electric current during the SPS process. The bulk HEAC may have possessed at least three types of phases. This was confirmed by the presence of Al_2O_3, FCC and BCC diffraction peaks in the XRD pattern (Fig. 2).

The TEM micrograph and the corresponding SAED pattern of the twinned phase along [011] the zone axis (region 5) are shown in Fig. 3. The EDS/TEM and SAED analyses confirmed that the twinned phase is FCC phase structure. Clearly, the lamella thickness of nanoscale twins (Fig. 3a) is approximately 30 nm. The chemical composition analyses of different regions are listed in Table 1 (detected by the EDS) and indicate that there were three different phases present. FCC was clearly enriched with Ni, slightly Cr–Ni–Fe–Co-rich and Al–Ti-depleted. The twined FCC phase was Ni–Cr–Co–Fe-rich and slightly Al–Ti-depleted. The BCC phase was enriched with Al and slightly Cr–Ni–Fe–Co-rich. Nanoscale Al_2O_3 phases displayed smooth nanoscale particles in the EDS analysis, and these phases connected with the alloy matrix by strong interfacial bonding at the grain boundaries. Similar situations have been reported: the BCC phase was promoted by Al, Ti et al. and the FCC phase was promoted by Co, Ni et al. The corresponding SAED pattern (the matrix axis was [011]M and the twin axis was$[0\overline{1}\overline{1}]_T$), which is shown in Fig. 3c, indicated that the nanoscale twins belonged to the FCC phase. The phase of the nanoscale twins was

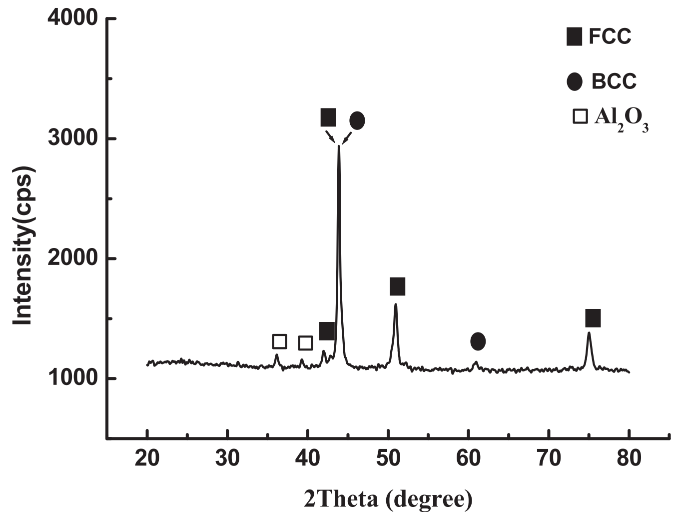

Fig. 2. XRD pattern of Al_2O_3–$Al_{0.4}FeCrCoNi_{1.2}Ti_{0.3}$ alloy.

Co–Ni–Fe–Cr-rich and the lattice parameter was 3.120 Å (FCC) according to SAED. The addition of Al_2O_3 might have affected deformation twinning because Al_2O_3 showed a hardness texture, and it could be assumed that the partial FCC phase between Al_2O_3 could not be readily deformed and consolidated. Consequently, twinning in the FCC phase may have occurred during the phase evolution and densification with the aim to be more stable and to attain complete densification. The same phenomena were also reported in $Al_{0.5}CrFeNiCo_{0.3}C_{0.2}$ and $CrCoNiFeAl_{0.6}Ti_{0.4}$ systems [6,7].

3.2. Compression tests

The stress-strain curve of bulk HEAC at room temperature is shown in Fig. 4. The compressive strength (σ_{max}) and compression strain (ε_p) were 2250 ± 10 MPa and 20 ± 0.50%, respectively. The average Vickers hardness of HEAC after SPS was measured to be 743 ± 12 Hv. Comparing these values to those of typical HEAs, which are listed in Table 2, these HEACs had excellent mechanical properties, high strength and good plasticity. The high strength and high hardness of HEAC was possibly due to the formation of the BCC phase, the nanoscale Al_2O_3 reinforcements, and the solid solution strengthening mechanism of Al atoms. It is worth noting that

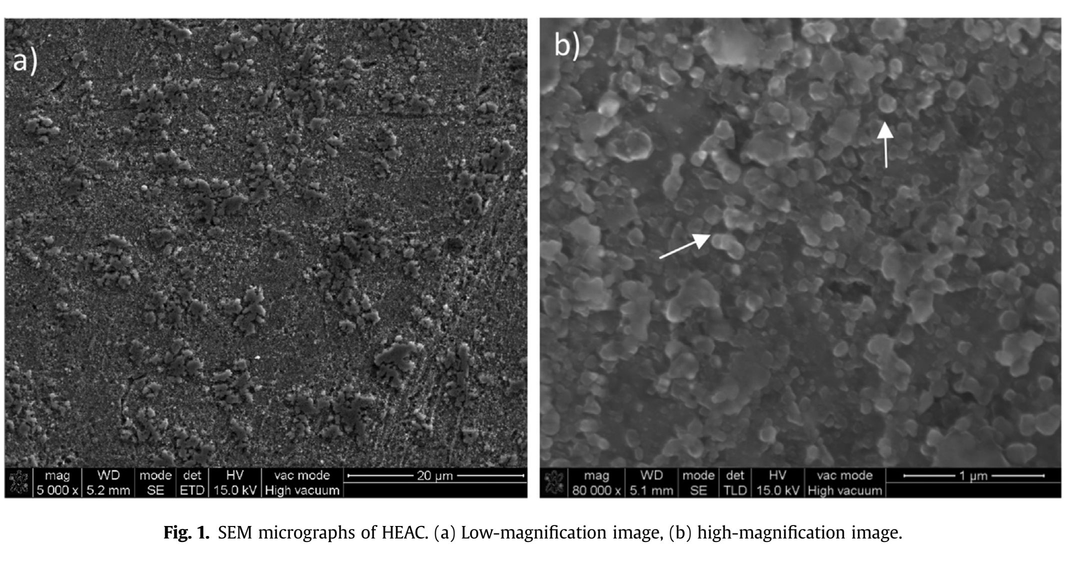

Fig. 1. SEM micrographs of HEAC. (a) Low-magnification image, (b) high-magnification image.