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Superb strength and high plasticity in laves phase rich eutectic medium-entropy-alloy nanocomposites

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ABSTRACT

Laves phase and laves-phase based conventional composites usually show extreme brittleness at room temperature due to poor fracture toughness. However, in this work, we design a $FeCoNiNb_{0.5}$ medium-entropy-alloy nanocomposite which possesses a high volume fraction (> 50%) of a cubic laves phase but shows superb strength and excellent malleability at room temperature. This high mechanical performance results from the formation of an in-situ nano-scale lamellar structure that joins the hard cubic laves phase and soft medium entropy face-centered cubic (FCC) phase through a semi-coherent interface. When the size of the lamellar structures is tuned below a critical value, this nanocomposite exhibits strong and sustainable strain hardening, leading to a fracture strain over 20% and fracture strength over 3.5 GPa in conventional compression. The mechanism for the unusual strain hardening in the laves-phase rich nanocomposite is explored afterwards with micromechanical experiments and theoretical modeling, which unveils a size-controlled transition in the plasticity mechanism from dislocation slip to twinning in the nano-scale laves phase. Our current work demonstrates that, through mixing a set of carefully selected elements, one can obtain high performance dual-phase eutectic nanostructures which are promising for structural applications.

1. Introduction

Laves phases are an important type of intermetallic compounds which have a great potential in applications as high temperature alloys (Livingston, 1992), superconducting wires (Olzi et al., 1988), hydrogen storage materials (Kandavel et al., 2008) and wear resistant alloys (Halstead and Rawlings, 1985). They are generally perceived to have the stoichiometric composition of LS_2, where L and S stand for the large and small atoms respectively, which constitute the biggest group of intermetallic compounds with the total number exceeding 1400 as reported so far (Villars and Calvert, 1985). In principle, laves phases can be classified into three categories (Stein et al., 2004), namely, hexagonal C14, cubic C15 and di-hexagonal C36. Unlike the simple crystalline structure in a cubic system, such as face centered cubic (FCC), atomic stacking in laves phases are topologically close packed, which comprises alternating four-layer structures that consist of a small atom layer stacked with a tri-layer (L-S-L) (Chisholm et al., 2005). As a result, dislocation movement in laves phases becomes difficult due to a high Peierls stress and the coordinated atomic movements (Takasugi et al., 1996). Consequently, laves phases containing refractory alloys can retain a 550 MPa yielding strength even at the elevated temperature of 1300 °C (Kazantzis et al., 1997). However, laves phases are generally perceived to be extremely brittle. According to

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Taub et al. (Taub and Fleischer, 1989), accommodating plastic deformation under multi-axial loading conditions requires the activation of five independent slip systems. However, the number of independent slip systems in laves phases is less than five (Taub and Fleischer, 1989) which could cause brittlement of laves phase. Moreover, the fracture toughness of the laves phase alloy was found to be very low, merely around 1 MPa $m^{1/2}$ at room temperature (Zhu et al., 1999) and less than 10 MPa $m^{1/2}$ even at 600 °C (Ravichandran et al., 2011).

To enhance plasticity, laves phases are usually alloyed with a third element, such as V or Mo (Takasugi et al., 1996). The rationale is to introduce anti-site defects or vacancies through the alloying element, which could in turn facilitate synchroshear (Chen et al., 2011; Chu and Pope, 1993b) by "opening up" the close packed lattice and/or "weakening" atomic bond strengths. Alternatively, one could also form ductile secondary metal matrix composites to constrain the crack propagation and imparting strain non-localization (Wu et al., 2017). This scenario can be achieved by in-situ eutectic composites which usually exhibit lamellar structures. It have been reported that dual phase alloys with lamellar structures can achieve a balance between the strength and ductility which are usually mutually exclusive in materials science (Cao et al., 2017; Okumura et al., 2011; Zhou and Chen, 2016). For instance, (Takeyama and Liu, 1991) synthesized hypoeutectic Cr-Cr_2Nb dual phase composite, which contained laves phase lamellars ~1 μm wide dispersed in a plastic solid solution matrix with a total volume fraction less than 30%. As a result, an improved compressive ductility of 5%–11% was obtained. By further alloying the Cr-Cr_2Nb dual phase alloy with minor Re, Al, Fe, Ni(Liu et al., 1996), the failure strain could increase to 15% with a moderate decrease in the fracture strength. Meanwhile, Liu et al. (2000) fabricated a series of hypo- and hyper-eutectic dual phase eutectic Cr-Cr_2Ta with the alloying elements Mo, Ti, Ce, La and Si, which showed fairly good fracture toughness around 15 MPa $m^{1/2}$ at ambient temperature. Recently, He et al. (2016a) demonstrated that laves-phase containing eutectic high entropy alloys (EHEAs) (Lu et al., 2014) could reach a compressive plasticity over 20% for a small volume fraction (25%) of the laves phase, which exhibit a lamellar structure with a typical width of ~300 nm. These previous studies are promising, which suggests that laves phase based alloys could be toughened through the design of high entropy alloys (He et al., 2017; Ye et al., 2016; Yeh et al., 2004) and the formation of dual phase eutectic composites. However, the volume fraction of the laves phase reported thus far was low (< 30%), leading to the low composite strength.

In theory, laves phase may exhibit plasticity by synchroshear (Chisholm et al., 2005; Hazzledine and Pirouz, 1993) via the slip of perfect dislocations (Korte and Clegg, 2012; Takata et al., 2013) or by twinning (Kazantzis et al., 1997; Livingston and Hall, 2011; Luzzi and Pope, 2003) via the formation and coordinated movements of Shockley partials, depending on the testing temperature (Kazantzis et al., 1997; Kimura et al., 2002) and/or the stacking fault energy of the laves phase (Chu and Pope, 1993a). Meanwhile, in metallic nano- and micro-composites, microstructural length scales also play an important role (Ardeljan et al., 2015, 2017; Okumura et al., 2011; Zhang et al., 2010), which could affect the dislocation behaviors (Abdolrahim et al., 2014; Beyerlein et al., 2011; Mayeur and McDowell, 2007) and thereby alter the overall mechanical properties (Wu et al., 2017). In this work, we demonstrate that, by following the recent EHEA design strategy discussed in Ref.(Ding et al., 2018), we can obtain a eutectic nanocomposite by mixing a set of carefully selected metallic elements, in which the volume fraction of the laves phase can reach ~50%. In this nanocomposite, the size of the laves phase lamellae can be controlled to below ~100 nm. As a result, brittleness can be evaded even at room temperature without sacrificing the overall strength of the composite.

2. Experiments

To design a concentrate multicomponent eutectic alloy, we followed the strategy as previously discussed in Ref. (Ding et al., 2018). In order to obtain a eutectic multicomponent alloy, Nb was chosen as the eutectic forming element to alloy with Fe, Co and Ni. Since Nb forms eutectic binaries with Fe, Co and Ni in the following composition, i.e., $FeNb_{0.14}$ (in atomic ratio), $CoNb_{0.15}$ and $NiNb_{0.19}$, the nominal composition of $FeCoNiNb_{0.5}$ was finally chosen. According to the entropy design criterion proposed by He et al. (2016b), we designed a medium-entropy-alloy (MEA) $FeCoNiNb_{0.5}$ which was expected to exhibit a multi-phase structure upon casting.

To validate the above thinking, ingots with the nominal chemical composition of $FeCoNiNb_{0.5}$ were prepared in a laboratory-scale arc melting furnace. All the raw materials had the purity level higher than 99.95% and the alloy ingots were fully re-melted at least 6 times to ensure chemical homogeneity with a Ti-getter to absorb residual oxygen in the furnace. Afterwards, samples were casted into water cooled Cu molds for the following dimensions: Φ5 × 50 mm, Φ3 × 50 mm, Φ2 × 50 mm, and Φ1 × 50 mm. After the subsequent cutting, grinding and etching, the microstructure of the sample was examined with a scanning electron microscope (SEM, Quanta FEG450). The lamellar sizes were measured from the SEM images using the conventional mean-linear-intercept method. To obtain the distribution of lamellar sizes, more than 200 measurements were made at different regions. After that, the rod samples were cut into specimens with an aspect ratio of 2:1 for the subsequent compression tests. Note that the top and bottom surfaces of the compression specimens were both grinded to ensure planarity during the mechanical testing. Compression tests were subsequently performed on the MTS Alliance RT/30 Electro-Mechanical Materials Testing System at the nominal strain rate of $10^{-3}s^{-1}$. To understand the deformation mechanisms, microcompression were also carried out on the Hysitron nanoindentation system.

3. Results

3.1. Structural characterization

Fig. 1 shows the X-ray diffraction (XRD) result of our $FeCoNiNb_{0.5}$ alloy, from which two phases could be identified, including a C15 laves phase with a lattice constant $a = 0.68nm$ and a face centered cubic (FCC) phase with $a = 0.36nm$. Furthermore, the sample

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