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Strain-rate effect upon the tensile behavior of CoCrFeNi high-entropy alloys

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#### ABSTRACT

High-entropy alloys could replace conventional materials in critical load-bearing applications, owing to their excellent mechanical properties. Supported by X-ray diffraction, microstructure analysis and tensile testing, the mechanical response of CoCrFeNi HEAs to varying strain rates was investigated. The alloys were found to be a single-phase face-centered cubic solid solution. Their tensile strength and ductility increased with rising strain rate. The dominant deformation mechanism of the high-entropy alloy was observed to shift from the dislocation motion at the lower strain rates to stacking faults at the higher strain rates. The CoCrFeNi high-entropy alloys exhibited greater hardness following tensile deformation with higher strain rates.

#### 1. Introduction

High entropy alloys (HEAs) consist of multi-principal elements [1,2], representing a significant departure from traditional alloy design in quest of high performance metallic materials. Among HEAs Al<sub>v</sub>CoCrCuFeNi allovs have been studied widely [3-7]. Single facecentered cubic (FCC) solid solution exists in these alloys with Al content below 0.5 [3]. With increasing Al content, two phases could be detected; one is FCC solid solution, and the other is body-centered cubic (BCC) solid solution. It is worth noting that the FCC solid solution showed mediocre strength but good ductility. With the increase of BCC solid solution, the HEAs showed high strength but poor ductility [3]. Alloys with poor ductility are not suitable for industrial applications. Strength-ductility trade-off has hindered high performance alloy development for many years [8,9]. Incorporating foreign elements into FCC structured HEA systems have the potential for forming second phases [10]. The FCC structured CoCrFeNi HEA is widely used as a base alloy of multiphase HEA for its high toughness. The CoCrFeNi HEA has FCC crystal structure and high toughness for wide range temperature applications [11-15]. A great number of CoCrFeNi based HEAs shows a great potential for application. The microstructures and mechanical properties of CoCrFeNi based HEAs are studied by many researchers [16-21]. CoCrFeNi HEA is strengthened through solid solution by the mixing of different elements. The deformation behavior of CoCrFeNi FCC solid solution is important for the coordinated deformation between FCC solid solution and second phases.

The strain-rate effect of metallic materials is an interesting and important property, and has been studied extensively [22–24]. The strength of some materials usually increases with increasing strain rate, and that of some of the others are the exact opposite. The same holds true for the discussion of ductility. Changes in the dominant deformation mechanisms or microstructures will affect the strain-rate effect. In any case, it is desirable that the metallic materials show positive and preferably strong strain rate sensitivity, which means that the faster the loading is applied, the more the material will resist deformation before fracture.

In this work, CoCrFeNi HEAs were prepared by arc melting technique. Uniaxial tensile testing was applied in the room temperature and strain rate ranges  $1 \times 10^{-2} - 1 \times 10^{-4} \text{ s}^{-1}$ . Tensile behavior of the HEAs was characterized at different strain rates. The strain-rate effect of CoCrFeNi HEA was discussed. Subsequently, the difference in deformation mechanism activated at different strain rates is proposed to account for the effect of strain rate on tensile strength and ductility.

#### 2. Experimental procedure

The CoCrFeNi HEAs were produced using high-purity elements (Co  $\geq$  99.95 wt%, Cr, Fe and Ni  $\geq$  99.99 wt%) by arc melting in a Ti-gettered high-purity argon atmosphere. The ingot was melted five times to ensure the homogeneity. The crystal structure was identified by an X-ray diffractometer (XRD, Bruker D8) with Cu K $\alpha$  radiation, operating at a voltage of 40 kV and a current of 30 mA. The microstructures and chemical compositions of the alloys were examined using a field

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emission scanning electron microscopy (FE-SEM, FEI Quanta 3D FEG), a transmission electron microscopy (TEM, FEI Tecani G2) and energy dispersive spectrometer (EDS). Thin foil TEM samples were prepared by a twin-jet electron polishing machine using a mixed solution of HNO<sub>3</sub>:CH<sub>4</sub>O=1:4. Tensile tests were carried out using dog bone-shaped tensile samples (CMT 5105) with different strain rates  $(1 \times 10^{-2}, 1 \times 10^{-3} \text{ and } 1 \times 10^{-4} \text{ s}^{-1})$  at room temperature. The tensile samples had a gauge length of 8 mm, a width of 1 mm and a thickness of 0.5 mm. Three samples were tested at each case to ensure the reliability of the results. Thin sections of 2.8×1×0.5 mm for TEM observations were cut from the samples after tensile deformation. The sections were firstly mechanically polished to a thickness of 50 um and then ion-beam thinned at a cold station of 100 K. TEM observations were performed in the TEM (FEI Tecani G2), operating at an accelerated voltage of 200 kV. Nano indentation experiments of the HEA after tensile deformation were conducted on MML NanoTest Vantage with a Berkovich indenter at a constant temperature of 20 °C. The loading rate was fixed to 5 mN/s. The tests were carried out until the thermal drift is reduced to below 0.05 nm/s. In addition, the drift correction calibrated at 10% of the maximum load during the unloading process was performed. The Poisson's ratio of HEAs was set as 0.3 in this work.

#### 3. Results and discussion

Fig. 1 shows XRD patterns of CoCrFeNi HEAs have three diffraction peaks. According to the JCPDS card (18-0646), these diffraction peaks resulted from a disordered FCC solid solution. Based on the XRD pattern, the (111) d-spacing is calculated as  $\sim$ 2.073 Å.

Fig. 2 shows SEM micrograph of CoCrFeNi HEA. The CoCrFeNi HEA is consisting of large grains. The size of grain is about 200– $300 \mu m$ .

Fig. 3 shows TEM image of the CoCrFeNi HEA with a corresponding SAED pattern. The SAED pattern was presented on the left bottom of the image. It indicates that CoCrFeNi HEA has an FCC structure. Based on measurements of interplanar spacing on the SAED pattern, the (111) d-spacing is calculated as ~2.073 Å. It is consistent with the results based on XRD pattern. The EDS result of 'A' was presented in Table 1.

The composition fulfills the phase formation criteria of HEAs, concerning  $\Delta H_{mix}$  (mixing enthalpy),  $\delta$  (atomic size difference) and  $\Delta S_{mix}$  (mixing entropy) [25,26]. The three controlling parameters are defined below:





Fig. 2. SEM micrograph of CoCrFeNi HEA.



Fig. 3. TEM micrograph and SAED pattern of CoCrFeNi HEA.

#### Table 1 EDS result of CoCrEeNi HEA

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Element	Co	Cr	Fe	Ni
Content, at%	26.6	25.4	24.4	23.6

$$\Delta H_{mix} = \sum_{\substack{i=1\\i\neq j}}^{n} \Omega_{ij}c_ic_j = 4 \sum_{\substack{i=1\\i\neq j}}^{n} \Delta H_{ij}^{mix}c_ic_j$$
(1)

$$\tilde{s} = \sqrt{\sum_{i=1}^{n} c_i (1 - \frac{r_i}{\bar{r}})^2}$$
(2)

$$\Delta S_{mix} = -R \sum_{i=1}^{n} (c_i lnc_i) \tag{3}$$

where  $c_i$  is the atomic percent of the *i*th element,  $\Delta H_{ij}^{mix}$  is the mixing enthalpy between *i*th and *j*th elements,  $r_i$  is the Goldschmidt atomic size of the *i*th element and R is the gas constant that equals to 8.314 J K<sup>-1</sup> mol<sup>-1</sup>.

According to Eqs. (1)–(3), calculated values of the parameters of CoCrFeNi HEA system are:  $\Delta H_{mix}$ =-3.75 kJ mol<sup>-1</sup>,  $\delta$ =2.87% and  $\Delta S_{mix}$ =11.53 J K<sup>-1</sup>·mol<sup>-1</sup>, respectively. In order to form simple solid solution, the aforementioned parameters have to be simultaneously

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