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## The influence of Al elements on the structure and the creep behavior of Al<sub>x</sub>CoCrFeNi high entropy alloys



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#### ABSTRACT

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Keywords: High entropy alloy Crystal structure Thermal properties Creep Stress relaxation The microstructure, crystal structure, creep behavior and mechanism of  $Al_x$ CoCrFeNi (*x* is in molar ratio) high entropy alloys were examined. The low Al content of  $Al_{0.15}$ CoCrFeNi alloy had single FCC structure with columnar cell microstructure, while the high Al content of  $Al_{0.60}$ CoCrFeNi alloy contained FCC+BCC duplex crystal structure with columnar dendrite microstructure. The creep property, evaluated by stress relaxation test, showed that the  $Al_{0.15}$ CoCrFeNi alloy had a higher creep resistant property than that of the  $Al_{0.60}$ CoCrFeNi alloy. The creep constitutive equation for the  $Al_{0.15}$ CoCrFeNi alloy was established with an average stress exponent of 5.56 and an average activation energy of 385 kJ mol<sup>-1</sup>, while it was with a larger average stress exponent of 8.82 and a smaller average activation energy of 334 kJ mol<sup>-1</sup> for the  $Al_{0.60}$ CoCrFeNi alloy. The analysis of apparent activation volume reveals that both alloys were in a rate limiting mechanism of cross-slip. Then, the higher stacking fault energy of the  $Al_{0.60}$ CoCrFeNi alloy was considered to result in its higher creep resistant property of the  $Al_x$ CoCrFeNi alloys by increasing the stacking fault energy.

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#### 1. Introduction

High entropy alloy (HEA) is a new concept alloy, which is considered as a potential high-temperature material [1,2]. Several HEAs have been demonstrated excellent property at elevated temperature, such as good thermal stability [3], remarkable compressive strength [4], etc. The Al<sub>x</sub>CoCrFeNi (*x* is in molar ratio) alloys are typical HEA which have been widely studied [5–7] on microstructure, mechanical performance, electrical property, etc., but its creep resistant property was rarely reported. In the current paper, the creep properties of the Al<sub>x</sub>CoCrFeNi alloys are explored by using stress relaxation test (SRT) proposed by Woodford et al. [8] with the consideration of Al element. The stress exponent (*n*) and the activation energy (*Q*) were calculated to establish the creep constitutive equation, and the apparent activation volume (*V*\*) and stacking fault energy (SFE) were used for deformation mechanism analysis.

#### 2. Materials and methods

The studied Al<sub>x</sub>CoCrFeNi alloys with different Al content were designed by referring article [5]. It was prepared in vacuum

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http://dx.doi.org/10.1016/j.matlet.2015.11.016 0167-577X/© 2015 Elsevier B.V. All rights reserved. induction smelting furnace with 99.9% pure raw materials. The well mixed ingots with dimension of  $110 \times 110 \times 20 \text{ mm}^3$  were finally obtained after 5 more times re-melting process. The whole procedure was executed under pure the argon atmosphere. The final compositions of these ingots are Al<sub>0.15</sub>CoCrFeNi (denoted as Al<sub>0.15</sub>) and Al<sub>0.60</sub>CoCrFeNi (denoted as Al<sub>0.60</sub>).

The microstructure was observed by Zeiss supra 55 scanning electron microscope after etching in Aqua-regia. The crystal structure was analyzed by X-ray diffractometer (XRD, Cu target) under voltage of 30 kV and current of 40 mA, the diffraction angle  $(2\theta)$  ranged from 20° to 100° at a rate of 4 deg.min<sup>-1</sup>.

To ensure the uniformity of microstructure, the samples for SRT were cut from the center of the ingots with a diameter of 5 mm and a gauge length of 25 mm. SRT was executed at 0.20% strain for 4 h on electromechanical universal testing machine. The loading strain rate was  $8 \times 10^{-5} \, {\rm s}^{-1}$ . Generally, the common materials, such as SUPER304H and HR3C heat resistant steels, are often tested at temperature above 0.5 melting temperature (0.5Tm,  $\sim 600 \, {}^{\circ}\text{C}$ ) in their creep strength design test. Based on this, the test temperature was set at 580–700 ± 1  ${}^{\circ}\text{C}$  around 0.5Tm and above 0.3Tm.

#### 3. Results and discussion

Fig. 1 shows the microstructure and the XRD patterns of the  $Al_{0.15}$  and the  $Al_{0.60}$  alloys. The  $Al_{0.15}$  alloy in Fig. 1a shows the

columnar cell microstructure, whereas the Al<sub>0.60</sub> alloy in Fig. 1b displays a columnar dendrite microstructure. The XRD patterns in Fig. 1c reveal that the Al<sub>0.15</sub> alloy has single FCC structure and the Al<sub>0.60</sub> alloy has FCC+BCC mixed structure. It indicates that the increase of Al element inclines to form BCC structure and dendrite microstructure.

The SRT's results of the Al<sub>0.15</sub> and the Al<sub>0.60</sub> alloys are shown in Fig. 2a and b, respectively. It is seen that the Al<sub>0.15</sub> alloy has a lower relax rate than that of the Al<sub>0.60</sub> alloy. At high temperature, the total strain ( $\epsilon_t$ ) during SRT is divided into elastic deformation ( $\epsilon_e$ )

and creep deformation ( $\epsilon_c$ ) as  $\epsilon_t = \epsilon_e + \epsilon_c = 0.20\%$ . Therefore, the creep rate ( $\dot{\epsilon}_c$ ) can be deduced as follows:

$$\dot{e}_{\rm c} = -\dot{e}_{\rm e} = -\frac{1}{E}\frac{{\rm d}\sigma}{{\rm d}t} \tag{1}$$

where,  $\dot{e}_e$  is the elastic deformation rate,  $\sigma$  is stress, t is test time, and E represents elastic modulus

By Eq. (1), the response of  $\sigma - t$  was converted into relationship of  $\dot{\epsilon}_c - \sigma$  shown in Fig. 2c. The Al<sub>0.60</sub> alloy displays a higher creep rate than that of Al<sub>0.15</sub> alloy under the same stress. Normally, the



Fig. 1. SEM micrographs of (a)  $Al_{0.15}$  and (b)  $Al_{0.60}$  alloys. (c) XRD patterns.



Fig. 2. Stress relaxation curves of (a) Al<sub>0.15</sub> and (b) Al<sub>0.60</sub> alloys. (c) Stress versus creep rate curves. (d) Relationship between ln ec and 1/T.

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