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To cite this article: Jongun Moon, Sun Ig Hong, Jae Bok Seol, Jae Wung Bae, Jeong Min Park & Hyoung Seop Kim (2019) Strain-rate sensitivity of high-entropy alloys and its significance in deformation, Materials Research Letters, 7:12, 503-509, DOI: [10.1080/21663831.2019.1668489](https://doi.org/10.1080/21663831.2019.1668489)

To link to this article: <https://doi.org/10.1080/21663831.2019.1668489>



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Published online: 25 Sep 2019.



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ORIGINAL REPORT



Strain-rate sensitivity of high-entropy alloys and its significance in deformation

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ABSTRACT

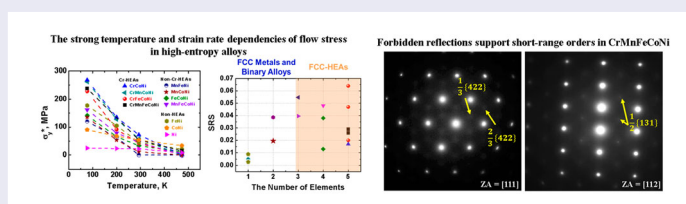
Strain-rate dependence in face centered cubic high entropy alloys still remains controversial despite extensive efforts on this topic. Strain-rate sensitivity reflects underlying thermally-activated deformation and the controversy boils down to the deformation mechanism. There has been disagreement even on the experimental values of the strain-rate sensitivity and activation volume. This study reviewed and analyzed the differences in experimental values and proposed mechanisms to resolve the controversy over the nature of thermal obstacles in CrMnFeCoNi alloy. TEM study on CrMnFeCoNi supports the presence of nanoscale heterogeneity elucidating the nature of rate-controlling mechanism in the alloy.

ARTICLE HISTORY

Received 27 July 2019

KEYWORDS

High-entropy alloy; strain-rate sensitivity; thermally-activated deformation; activation volume



IMPACT STATEMENT

Strong temperature dependence of flow stress, high strain-rate-sensitivity and small activation-volume are key features for deformation of CrMnFeCoNi. TEM analysis supports presence of nanoscale heterogeneity acting as the rate-controlling barrier.

1. Introduction

The understanding of the temperature and strain-rate dependence of compositionally and structurally complex alloys [1] is of importance in the study of the rate-controlling and strengthening mechanisms because the plastic flow occurs by overcoming the obstacles through thermal activation. One important advantage of the thermally-activated deformation analysis is that thermal barriers which control the dislocation motion and deformation kinetics can be reasonably determined out of various obstacles observed by nanostructural analysis tools. Over the past decade, high-entropy alloys (HEAs) have been investigated in a wide range of research fields such as irradiation resistance [2], corrosion resistance [3], and mechanical properties [4,5]. In particular, temperature and strain-rate dependence on the plastic flow [1,6] of HEAs has attracted attention from materials

science society. In recent years, there has been debate over the deformation and strengthening mechanisms of HEAs. There is no consensus among researchers regarding the thermally-activated deformation mechanism and even the strain-rate dependence of CrMnFeCoNi HEA. Recently, Laplanche et al. [7] reported that CrMnFeCoNi exhibits negligible strain-rate sensitivity (SRS) and deformation behavior close to those observed in the conventional face-centered cubic (FCC) alloys. On the other hand, some investigators reported strong temperature and strain-rate dependence in CrMnFeCoNi [1,8,9].

Another controversial issue in the research of solid-solution HEAs is the presence or absence of nanoscale heterogeneity [1,7,10,11]. Nanoscale heterogeneity such as short-range clusters (SRCs) and/or short-range orders (SROs) in highly concentrated solid-solution alloys has been a subject of extensive research [12,13]. The presence

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of nanoscale heterogeneity has been suggested to explain the more strain-rate sensitive behaviors compared to conventional alloys [12,13]. Accordingly, both theoretical and experimental aspects of nanoscale heterogeneity [10,14] have been endlessly raised on HEAs having multiple principle elements. Recently, Hong et al. [1] suggested that the possible presence of nanoscale heterogeneity induces high friction stress for dislocation motion in CrMnFeCoNi HEA based on thermally-activated deformation and SRS analyses. The thermally-activated process for CrMnFeCoNi alloy was in contrast with that of typical FCC metals, and the difference was attributed to the presence of SRCs and SROs acting as important obstacles to dislocations [1].

In this study, the recent progress in thermally-activated deformation analyses and SRS of FCC-HEAs will be addressed. In addition, the analyses on thermally-activated deformation of CrMnFeCoNi HEA in the previous studies will be discussed to resolve the controversy.

2. Materials and methods

The detailed preparation of CrMnFeCoNi alloy and the dimensions of tensile specimens were identical to Ref. [1]. Tensile deformed specimens for transmission electron microscopy (TEM) analysis was prepared by deforming at a strain-rate of 10^{-3} s^{-1} in an Instron 1361 testing machine at room temperature. TEM samples deformed by 5% and 25% of true tensile strains (ε_T) were prepared using dual-beam focused ion beam (FIB, FEI Helios 650). TEM lamellae on Cu grids were milled from $1 \mu\text{m}$ to a final thickness of 50 nm at 5 kV and 3° of beam incidence. TEM characterization was carried out using a JEOL JEM-2100F operating at an acceleration voltage of 200 kV.

3. Results and discussion

3.1. Temperature and strain-rate dependencies of flow stress in FCC-HEAs

In the thermally-activated deformation, the temperature and strain-rate dependencies of flow stress have the following relationship [15]:

$$\left(\frac{\partial \ln \dot{\varepsilon}}{\partial \sigma} \right)_T \left(\frac{\partial \sigma}{\partial T} \right)_{\dot{\varepsilon}} = \frac{\Delta H}{kT^2}, \quad (1)$$

where $\dot{\varepsilon}$ is the strain-rate, σ is the flow stress, k is the Boltzmann's constant, T is the temperature, and ΔH is the activation enthalpy.

Since the activation volume $V^* = MkT \frac{\partial \ln \dot{\varepsilon}}{\partial \sigma}$ [1], equation (1) can be modified as follows:

$$V^* \left(\frac{\Delta \sigma}{\Delta T} \right)_{\dot{\varepsilon}} = \frac{M \Delta H}{T}, \quad (2)$$

where M is the Taylor factor.

It is apparent that the V^* is inversely proportional to the temperature dependencies of flow stress $((\Delta \sigma / \Delta T)_{\dot{\varepsilon}})$ in Equation (2). Since the V^* is also inversely proportional to the SRS ($m = MkT/V^*$) [7], the following equation can be obtained:

$$\left(\frac{1}{\sigma} \right) \left(\frac{\Delta \sigma}{\Delta T} \right)_{\dot{\varepsilon}} = \frac{m \Delta H}{kT^2} \quad (3)$$

Temperature dependence of flow stress $((\Delta \sigma / \Delta T)_{\dot{\varepsilon}})$ and SRS (m) are roughly proportional unless the thermal barriers responsible for the deformation vary with temperature or strain-rate in the temperature range of analysis [16]. Therefore, the comparison of temperature dependence of yield stress in FCC-HEAs with other FCC metals and alloys would rationally predict the difference in SRS between them. The ratio of yield stress (RYS) at 77 K (σ_{77K}) to that at room temperature (σ_{RT}) can be used as a measure of temperature dependence of stress excluding the effects of recovery, dynamic strain aging, and deformation twinning at 77 K in FCC-HEAs [17,18]. Figure 1 shows the strong temperature dependence of stress in the reported FCC-HEAs [8,9,17–22] compared to conventional FCC metals and alloys [23,24]. The average RYS (σ_{77K}/σ_{RT}) of FCC-HEAs is 1.71, while that of other FCC metals and alloys is 1.31.

Figure 2(a) shows the temperature dependence of yield stress (σ_y) for equiatomic HEAs, binary alloys, and pure Ni with wide ranges of temperatures from 77 to 673 K. The σ_y of equiatomic HEAs is more temperature-sensitive compared to that of binary alloys and pure Ni. The stronger temperature dependences of HEAs suggest high SRS and low V^* according to Equations (2) and (3). The flow stress consist of the athermal stress (σ_G) and thermal stress (σ^*) components [15]. The σ_G is known to be proportional to the shear modulus of the materials [15]. In general, the stress plateau in metals and alloys is observed in the temperature range between one-third and two-thirds of the melting temperature [11], thus, it can be estimated that the stress plateau of CrMnFeCoNi appears in the temperature range of 538–673 K. Indeed, the temperature dependence of the flow stress data reported by Otto et al. [20] suggests that the stress plateau in CrMnFeCoNi occurs at temperatures of 473–673 K. The σ^* of alloys can be obtained by subtracting the σ_G modified using the temperature dependence of shear modulus [9,25,26] from the total

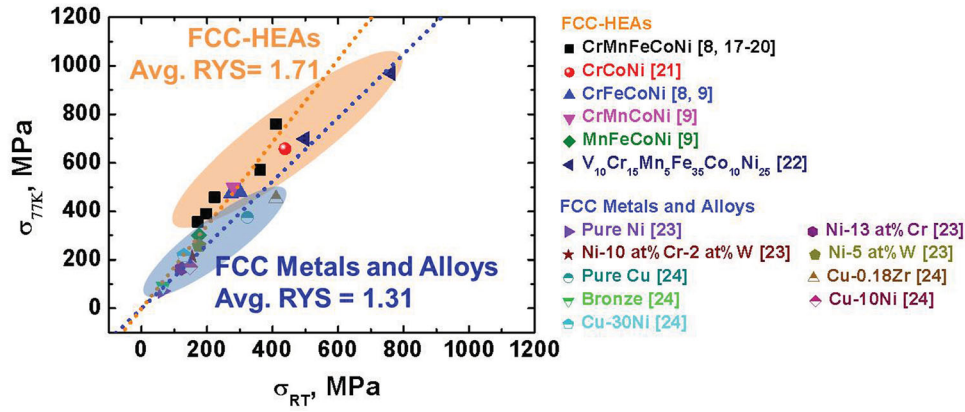


Figure 1. Temperature dependence of yield stress plotting σ_{77K} versus σ_{RT} of FCC metals, alloys, and HEAs. The dotted lines represent the average RYS of FCC-HEAs and FCC metals and alloys, respectively. The chemical composition of the alloys is expressed in at%.

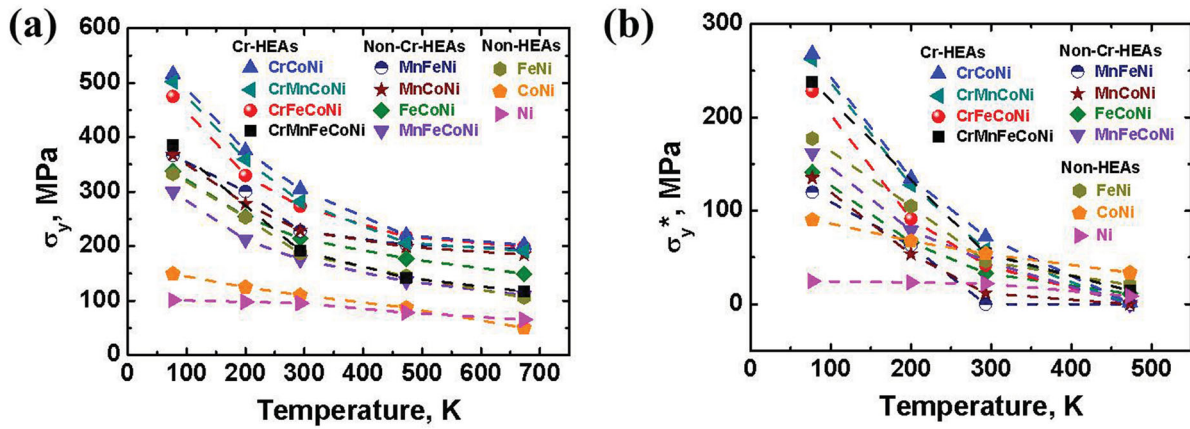


Figure 2. Temperature dependence of: (a) σ_y ; (b) σ_y^* for Cr-HEAs, Non-Cr-HEAs, and Non-HEAs. Data are taken from Ref. [9].

flow stress and are plotted as a function of temperature in Figure 2(b). It is interesting to note that Cr-containing HEAs (Cr-HEAs) have the stronger temperature dependence of σ_y^* than binary alloys and pure Ni (Non-HEAs) as well as non-Cr-containing HEAs (Non-Cr-HEAs) in Figure 2(b).

The SRS of alloys is expected to increase with increase of the slope of the σ_y^* -temperature curves in Figure 2(b) as indicated in Equation (3). In FCC-HEAs with the strong temperature dependence of flow stress, high SRS has been reported in the previous studies [17,27–31]. The variation of SRS with increasing the number of alloying elements in FCC metals and alloys is shown in Figure 3. It is clearly shown that the reported SRS of FCC-HEAs [17,27–31] is greater than that of pure metals and binary alloys [27,32,33]. The Peierls-barrier-dominated lattice friction [30], Labusch-type solution strengthening mechanism [27], and cutting through forest dislocations [7] have been suggested to be responsible for the temperature and strain-rate dependencies of flow stress

in FCC-HEAs. In the following section, two apparently different perspectives [1,7] on the SRS and the thermally-activated deformation of CrMnFeCoNi that led to the controversy are analyzed.

3.2. Thermally-activated deformation analysis on CrMnFeCoNi HEA

The thermal obstacles that interrupt motion of dislocations can be determined by activation volume ($V^* = \lambda \lambda b$) which is related with the spacing (λ), width (λ) of thermal barriers, and Burger's vector (b). Two representative methods, strain-rate jump (SRJ) [1,27] and repeated stress relaxation (RSR) [7] tests, have been used to measure V^* of CrMnFeCoNi HEA. Regardless of which method is being used, the important point to measure reliable thermal activation parameters is that the parameters should be measured carefully to minimize sudden dislocation immobilization or possible changes of dislocation substructure during the tests [34]. The failure

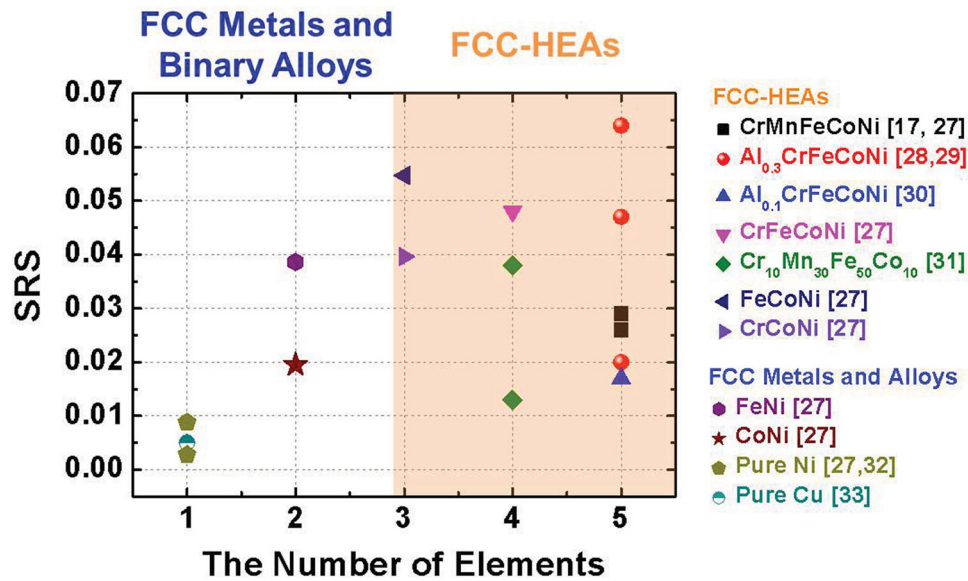


Figure 3. Variation in SRS as the number of alloying elements increases. The alloys which have three or more alloying elements in equiatomic or near-equiatomic ratio are clarified as HEAs. The chemical composition of the alloys is expressed in at%. The measurements of SRS were done by strain-rate jump test in Refs. [17,27,30,31], while two samples were tested at different strain-rates to measure SRS in Refs. [28,29].

to maintain the dislocation substructure and mobile dislocation density (ρ_m) during the tests adds to the inaccuracy and uncertainty in measuring thermal activation parameters because such changes affect the thermally-activated motion of dislocations and accordingly the values of thermal parameters.

Laplanche et al. [7] performed the RSR test [35] to obtain V^* of the alloy CrMnFeCoNi. The RSR test has been conducted to determine thermal activation parameters and the test is based on one assumption that the substructural changes are negligible during the test [35]. However, the decrease in ρ_m during the RSR test has been reported [36]. Okazaki et al. [36] pointed out that the gradual decay of ρ_m took place as plastic deformation and relaxation cycle proceed. It directly affects the accuracy of V^* , casting doubt on the V^* measured by the RSR test [7]. Laplanche et al. [7] concluded the SRS of CrMnFeCoNi alloy is low by comparing the stress-strain curves (which appeared to be close) obtained by two different specimens (Figure 1 in Ref. [7]). However, the earlier studies of thermally-activated process recommended that the thermal activation parameters such as V^* and SRS be measured by a single specimen to avoid errors associated with the local variations in microstructure and strength [33,35]. In addition to this, they calculated the SRS using the Haasen plot which is estimated by the measured V^* [7]. This suggests that the possible inaccuracy in V^* obtained by RSR test can lead to low SRS of CrMnFeCoNi alloy [7].

As shown in Equation (3), the SRS is proportional to the temperature dependence of flow stress in metals and alloys. Earlier, Otto et al. [20] and Gali and George [8] observed the strong temperature dependence of yield stress in CrMnFeCoNi alloy, suggesting high SRS. Laplanche et al. [18] also investigated the temperature dependence of yield stress and stated that their theory underpredicts the strength at low temperatures but otherwise is in generally good agreement with experiments. Their statement however suggests that the temperature dependence of flow stress in CrMnFeCoNi is greater than their theoretical estimate up to room temperature, suggesting that the SRS is larger and the V^* is smaller than their values obtained by RSR tests in CrMnFeCoNi [7]. Extensive data and reports [1,8,9,17,18–22,27–31] revealing the strong temperature dependence of flow stress in CrMnFeCoNi than conventional FCC alloys strongly suggest that the SRS is larger and the V^* is smaller in CrMnFeCoNi compared to those of conventional FCC alloys [1,15,17]. The strong temperature and strain-rate dependencies of flow stress observed by the previous results [1,8,9,17,18–22,27–31] do not support the suggestion of low SRS by RSR test [7].

It has been reported that the V^* s of CrMnFeCoNi were $40\text{--}118b^3$ at room temperature, much smaller than those of conventional FCC metals ($100\text{--}1000b^3$) by Hong et al. [1]. The SRS of CrMnFeCoNi was approximately ten times greater than conventional FCC alloys [17]. The low V^* and their increasing trend with strain were

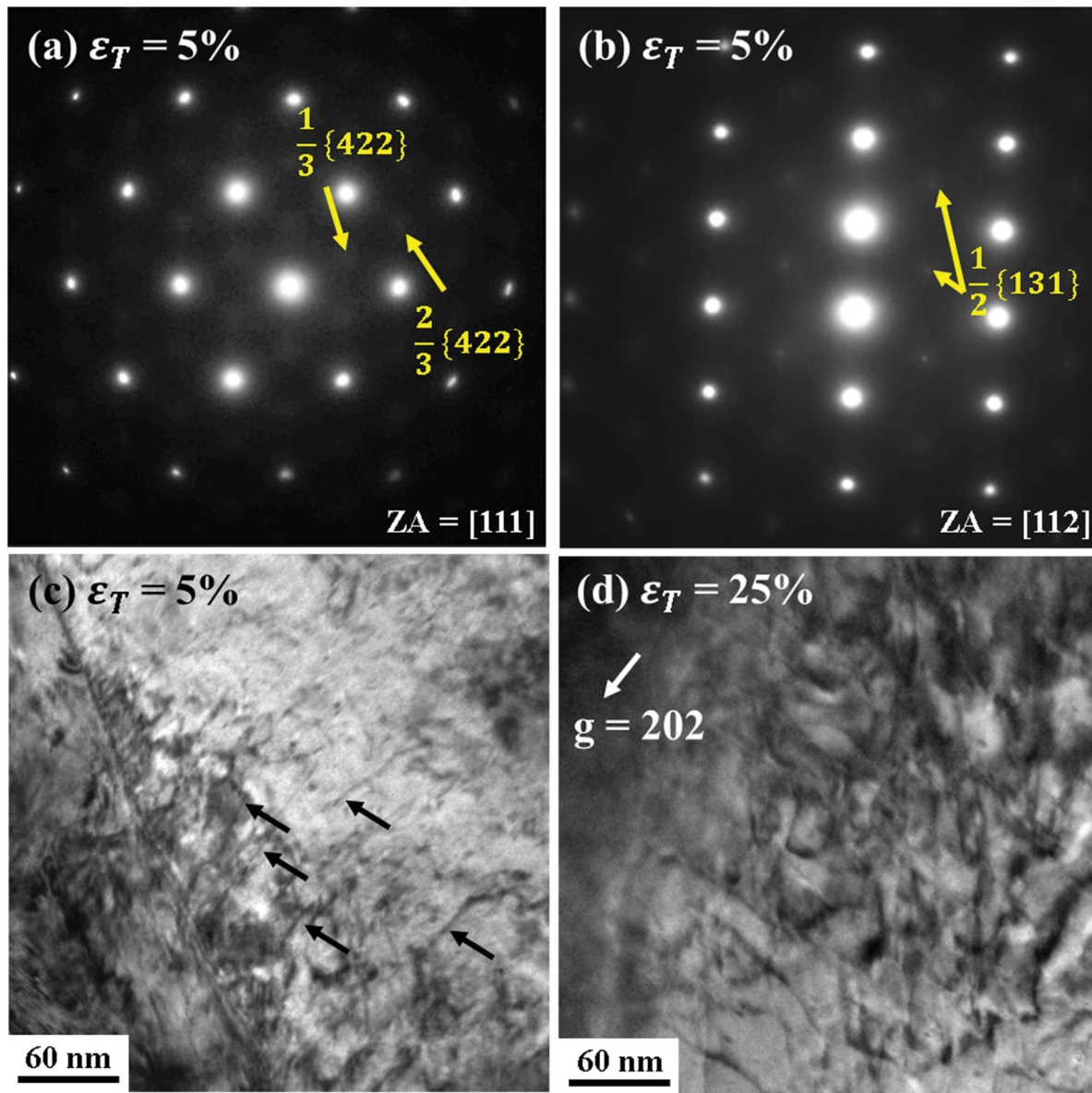


Figure 4. (a) Selected area diffraction patterns taken along the (a) [111] and (b) [112] zone axes of the CrMnFeCoNi alloy with 5% of ε_T . The diffuse forbidden reflections are marked by yellow arrows. TEM bright-field images of the alloy with (c) 5% and (d) 25% of ε_T . The dislocations are marked by black arrows in (b). Note: the alloy annealed at 1073 K for 1 h.

explained by shearing of SRCs and/or SROs in CrMnFeCoNi alloy [1]. Niu et al. [14]’s prediction of spin-driven Cr ordering by first-principles calculations along with the experimental measurements of the magnetic moments with Cr ordering supports the presence of co-clusters and SROs. Interestingly enough, the temperature dependence of flow stress and accordingly SRS in Cr-HEAs are greater than those of other alloys in Figure 2. Niu et al. [14]’s result suggests that the strong temperature dependence and high SRS of Cr-HEAs in Figure 2(b) could be attributed to the presence of co-clusters and SROs in these alloys.

Figure 4(a,b) show selected area diffraction patterns (SADPs) of the alloy deformed with 5% of ε_T . The SADPs were taken along [111] and [112] zone axes. The reflections which are forbidden by a FCC lattice, $1/3\{422\}$ and $1/2\{131\}$, were observed in the [111] and [112] zone axes, respectively. It is well known that local chemical ordering from the average atomic structure causes diffuse scattering resulting in forbidden reflections [37]. It should be noted that the forbidden reflections due to local chemical ordering were only observed in the [111] and [112] zone axes, not in the [110] zone, and are diffuse as in the previous studies [37]. We proposed that the forbidden

reflections in the present study are evidence of SROs in CrMnFeCoNi. The observation of planar array of dislocation with 5% of ε_T and the transition of dislocation arrangement from planar array to cell structure observed by Laplanche et al. [18] and also in Figure 4(c,d) can be explained in terms of presence of SROs and shearing of SROs as suggested in Ref. [1].

4. Conclusions

In this study, the published data regarding one of the most controversial issues on the fundamentals of deformation in HEAs were analyzed to resolve the controversy. There has been debate about whether the V^* and SRS of CrMnFeCoNi are different from those of conventional FCC metals and alloys or not. The stronger temperature and strain-rate dependences of flow stress have been observed in FCC-HEAs compared to conventional FCC alloys by some investigators. The most recent and detailed works on thermal activation analyses suggesting that CrMnFeCoNi deformed by the typical mechanism of conventional FCC alloys added fuel to the debate. We found that the low SRS measured by RSR test is incompatible with the strong temperature dependence of flow stress in CrMnFeCoNi. The TEM study supports that the strong strain-rate and temperature dependences in CrMnFeCoNi are associated with the presence of SROs.

Acknowledgement

This research was supported by Creative Materials Discovery Program through the National Research Foundation of Korea (NRF) funded by Ministry of Science and ICT (2016M3D1A1023384).

Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This research was supported by Creative Materials Discovery Program through the National Research Foundation of Korea (NRF) funded by Ministry of Science and ICT [grant number 2016M3D1A1023384].

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