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To cite this article: Jongun Moon, Sun Ig Hong, Jae Wung Bae, Min Ji Jang, Dami Yim & Hyung Seop Kim (2017) On the strain rate-dependent deformation mechanism of CoCrFeMnNi high-entropy alloy at liquid nitrogen temperature, Materials Research Letters, 5:7, 472-477, DOI: 10.1080/21663831.2017.1323807

To link to this article: <https://doi.org/10.1080/21663831.2017.1323807>



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Published online: 15 May 2017.



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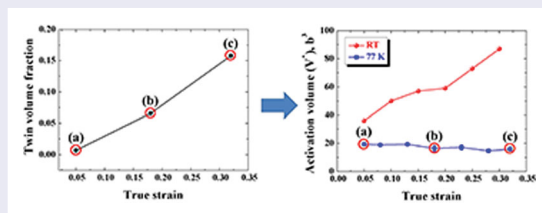
On the strain rate-dependent deformation mechanism of CoCrFeMnNi high-entropy alloy at liquid nitrogen temperature

Jongun Moon^a, Sun Ig Hong^b, Jae Wung Bae^a, Min Ji Jang^a, Dami Yim^a and Hyoung Seop Kim^{ib a,c}

^aDepartment of Materials Science and Engineering, Pohang University of Science and Technology, Pohang, Republic of Korea; ^bDepartment of Materials Science and Engineering, Chungnam National University, Daejeon, Republic of Korea; ^cCenter for High Entropy Alloys, Pohang University of Science and Technology, Pohang, Republic of Korea

ABSTRACT

In the present work, the deformation mechanisms of the CoCrFeMnNi high-entropy alloy at 77 K were investigated using thermal activation analyses. The strain rate jump test was performed to estimate strain rate sensitivity and the activation volume of the alloy. Transmission electron microscopy analyses were performed to identify the evolution of twins at low temperatures. The insensitivity of activation volume to strain observed in the CoCrFeMnNi alloy at 77 K was different from the observed increase in the activation volume with strain at room temperature, which occurred due to the shearing of nanoscale inhomogeneities, such as co-clusters and short-range orders.



IMPACT STATEMENT

The insensitivity of the activation volume to plastic strain in the CoCrFeMnNi alloy at 77 K can be attributed to the increasing fraction of mechanical twinning with strain.

ARTICLE HISTORY

Received 20 February 2017

KEYWORDS

High-entropy alloy; strain rate sensitivity; thermally activated process; transmission electron microscopy; twinning

Introduction

A novel type of alloy, the so-called high-entropy alloys (HEAs), was proposed in 2004 by Yeh et al. [1], who described them in this way: 'HEAs are composed of five or more principal elements, with each of the elemental concentrations between 5 and 35 at% forming single phase face-centered cubic (FCC) or body-centered cubic (BCC) structure due to their high mixing entropy'. The four core effects of the HEAs, that is, high configuration entropy, sluggish diffusion, cocktail effect, and large lattice distortion [2], lead to their promising properties, such as high strength and ductility, good corrosion resistance, and superior fracture toughness [3–5].

In particular, simultaneous enhancements in strength and tensile ductility at cryogenic temperatures have been observed in the CoCrFeMnNi HEA, which is attributable to high twinning activity, due to low stacking fault energy at low temperatures [5,6]. Otto et al. [7] provided

microstructural evidence for the formation of deformation twins in CoCrFeMnNi HEA after a tensile strain of 20.2% at 77 K. In addition, Laplanche et al. [8] reported that the formation of nanoscale twins in the CoCrFeMnNi HEA began at a lower true strain of ~6% during tensile tests at 77 K. In spite of several efforts, there remains a diversity of opinion about the role of twins in FCC HEAs at low temperatures.

The strengthening of CoCrFeMnNi HEA, which is controlled by thermally activated deformation, is more dependent on temperature than that of conventional FCC metals [9]. Nevertheless, there is a lack of systematic investigation on the thermally activated process of deformation in HEAs. It should be noted that deformation mechanisms vary with deformation conditions, such as strain, strain rate, and temperature. Therefore, in the present study, the thermally activated dislocation motion of CoCrFeMnNi HEA was analyzed by

CONTACT Sun Ig Hong ✉ sihong@cnu.ac.kr Department of Materials Science and Engineering, Chungnam National University, Daejeon 34134, Republic of Korea; Hyoung Seop Kim ✉ hskim@postech.ac.kr Department of Materials Science and Engineering, Pohang University of Science and Technology, Pohang 790-784, Republic of Korea; Center for High Entropy Alloys, Pohang University of Science and Technology, Pohang 790-784, Republic of Korea

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estimating activation volume, obtained from strain rate sensitivity at room temperature (RT) and liquid nitrogen temperature.

Experiment

Ingots of CoCrFeMnNi HEA were produced via the vacuum induction melting of pure elements (purity above 99.9%) in a graphite mold. The homogenization annealing of the ingot was carried out at 1100°C for 6 h in an argon atmosphere, followed by cold-rolling to reduce thickness from 7.8 to 1.5 mm. The cold-rolled plate was annealed at 800°C for 1 h in an argon atmosphere. The average grain size of the as-annealed specimen was 13 μm .

To conduct strain rate jump testing, dog bone-shaped tensile test specimens with a gauge length of 5 mm, a width of 2.5 mm, and a thickness of 1.5 mm were prepared from the as-annealed plate. The strain rate jump tests were conducted by changing the strain rate between 10^{-3} and 10^{-2} s^{-1} with a true strain, in an Instron 1361 testing machine at liquid nitrogen temperature (77 K). Strain rate jumps were carried out at the strain in the uniform elongation regime. An extensometer was used to measure the displacement of the gauge length at 77 K.

Transmission electron microscopy (TEM) analyses were performed using JEOL JEM-2100F with a Cs-corrector on a scanning transmission electron microscope operating at an acceleration voltage of 200 kV to examine the existence of nano-twins in the deformed samples at 77 K. Tensile tests were interrupted at true strains of 5%, 18%, and 32%; and the interrupted tensile specimens were sliced from their gauge sections for TEM analyses and then ground to a thickness of 90 μm using 600, 800, and 1200 SiC grit papers. TEM foils were prepared by jet-polishing at 23 V in a solution consisting of 70 vol.% methanol, 20 vol.% glycerine, and 10 vol.% perchloric acid.

Laplanche et al. [8] performed TEM analyses of the CoCrFeMnNi HEA using interrupted tensile specimens to determine the widths of the nanoscale mechanical twins, and spacings between twins. In this study, the widths and spacings of the mechanical twins were measured using their method. The mean spacing λ between twins and the volume fraction f of twins were calculated by the following equation [10]:

$$N = \frac{1}{\lambda} = \frac{1}{2t} \frac{f}{1-f}, \quad (1)$$

where N is the number of twins intersected per unit length of the grid lines and t is the width of the twins. At least 10 grains were examined to measure the widths and spacings of twins in each specimen for their reliability.

Results and discussion

Strain rate sensitivity and thermal activation volume

Figure 1 exhibits the results of the strain rate jump tests at RT and 77 K, with strain rate jumps between 10^{-3} and 10^{-2} s^{-1} . As reported in previous studies [5,7], the strength and ductility of the CoCrFeMnNi alloy were enhanced at 77 K due to twinning in addition to dislocation glide, compared to the RT tensile test results. Indeed, the twinning at cryogenic temperature enhances strain hardening, and uniform elongation generates an almost linear stress–strain curve with neither saturation of stress nor post-necking elongation. This stress–strain behavior at 77 K is qualitatively totally different from that at RT, which exhibited typical stress saturation and post-necking elongation behaviors.

Figure 2(a) shows the calculated strain rate sensitivity (m) of flow stress estimated from the strain rate jump tests using the following equation [12]:

$$m = \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}}, \quad (2)$$

where σ is the flow stress and $\dot{\epsilon}$ is the strain rate. For the precise calculation of strain rate sensitivity, data in the uniform elongation range were used. The strain rate sensitivity of the flow stress at RT was higher than that at 77 K, which is common in conventional FCC metals [13,14]. The measured strain rate sensitivity of flow stress at RT decreased with increasing strain. This tendency is also generally observed in steels, and is attributed to the exhaustion of work hardening potential by the accumulation of dislocations, leading to saturation during deformation [15,16].

On the other hand, the strain rate sensitivity of the CoCrFeMnNi HEA at 77 K was almost unchanged with

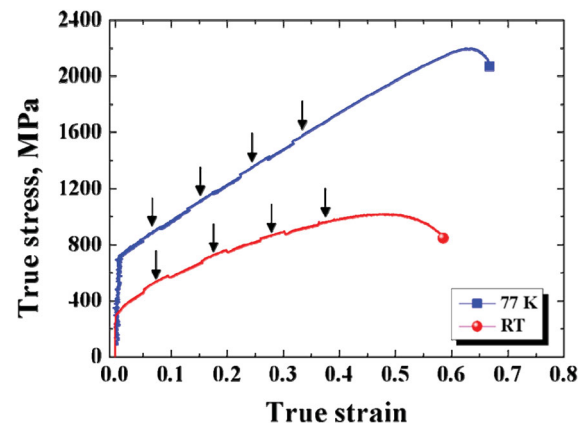


Figure 1. True stress–strain curves during strain rate jump tests at RT (data from [11]) and at 77 K. The vertical arrows indicate the points where rate jumps occurred from 10^{-3} to 10^{-2} .

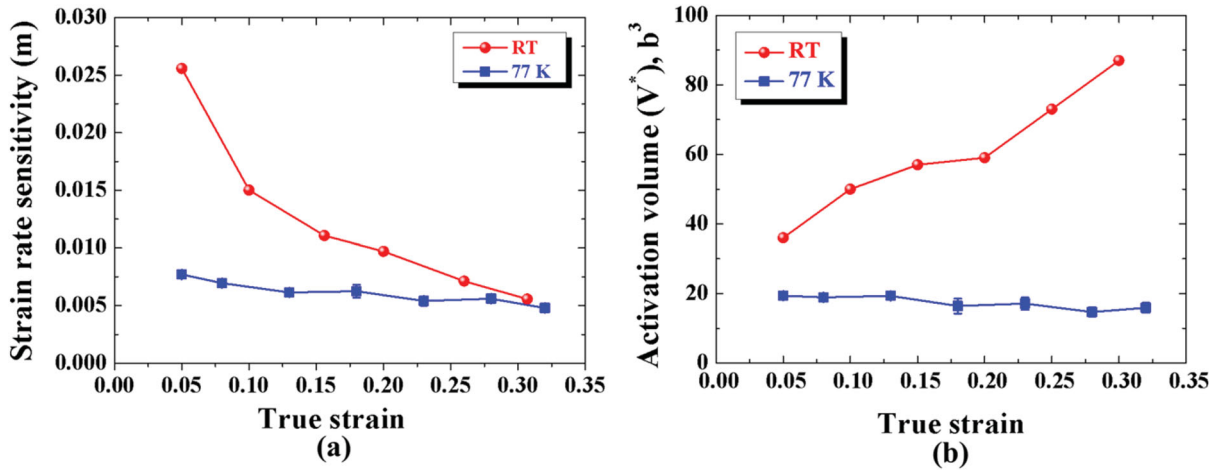


Figure 2. (a) Strain rate sensitivity and (b) activation volume at RT (from [11]) and 77 K measured from the strain rate jump tests.

strain. This is discussed below in relation to activation volume. Interestingly, the measured strain rate sensitivity of the CoCrFeMnNi HEA at RT was one order of magnitude higher than those of conventional coarse-grained FCC metals, for example, Cu ($m \sim 0.006$) and Ni ($m \sim 0.0028$) [17,18]. Komarasamy et al. [19] investigated the deformation mechanism of Al_{0.1}CoCrFeNi HEA and suggested that the high strain rate sensitivity in the Al_{0.1}CoCrFeNi HEA is due to a severely distorted lattice, which induces large friction forces and can obstruct the motion of dislocations.

The rate-controlling mechanism can be explained using the activation volume at a given temperature. The activation volume (V^*) for deformation can be calculated as follows [14]:

$$V^* = \sqrt{3}kT \frac{\partial \ln \dot{\epsilon}}{\partial \sigma}. \quad (3)$$

The measured activation volumes of the CoCrFeMnNi HEA at RT and 77 K are shown in Figure 2(b). When the dislocation barrier is modeled as a simple rectangular shape, the activation volumes V^* can be expressed by the following constitutive equation [20]:

$$V^* = l^* \lambda b, \quad (4)$$

where l^* is the barrier spacing, λ is the barrier width, and b is Burger's vector. Conventional coarse-grained FCC metals have V^* in the range of $\sim 1000b^3$ for a forest dislocation cutting mechanism at RT. The spacings between the forest dislocations will decrease as this forest dislocation density increases. Therefore, V^* decreases with plastic strain for conventional FCC metals [18,21]. However, as shown in Figure 2(b), the value of V^* at RT ranges from $\sim 36b^3$ to $87b^3$, while V^* at 77 K ranges from $\sim 15b^3$ to $19b^3$ which are slightly lower values than that at

RT. Hong et al. [11] suggested that the strong temperature dependence of yield stress and the small V^* in CoCrFeMnNi favor dislocation glide over the obstacles with high friction stress.

Interestingly, the V^* of the CoCrFeMnNi alloy in this study is close to those observed in BCC metals ($8\text{--}100b^3$) and HCP metals ($5\text{--}100b^3$) [11]. It was reported [11] that the increase in V^* with strain supports the deformation mechanism overcoming nanoscale inhomogeneities, such as co-clusters and/or short-range orderings (SROs), as the rate-controlling mechanism. The transition in dislocation structure from a planar array to cell structure at 20% strain in CoCrFeMnNi reported by Laplanche et al. [8] was attributed to the prevalent shearing of nanoscale inhomogeneities with strain.

In most metals and alloys in which slip is the dominant deformation process, the prevalent deformation mechanism, free from the effects of recovery and dynamic strain aging, is not known to change at low temperatures [11,22,23]. Therefore, we believe that the primary rate-controlling mechanism of the CoCrFeMnNi HEA at 77 K is not different from that at RT, that is, overcoming nanoscale inhomogeneities such as co-clusters and/or SROs. The planar slip observed at 77 K in the early part of plastic deformation [5,7] is indirect evidence of the SRO.

The V^* at 77 K was observed to be almost constant with increasing strain as shown in Figure 2(b), in contrast to the decrease in V^* with strain in most FCC metals and alloys [24]. The insensitivity of V^* to plastic strain in the CoCrFeMnNi HEA at 77 K is also different from the observation that the V^* increased with strain at RT. The insensitivity of V^* to plastic strain at 77 K can be attributed to the increasing fraction of mechanical twinning with strain, which is consistent with the insensitivity of strain rate sensitivity to plastic strain at 77 K.

Effect of mechanical twins to the activation volume at 77 K

According to previous studies [5,7,8,25], the deformation mechanism of the CoCrFeMnNi HEA is dislocation glide at RT, whereas it is attributed to both dislocation glide and nanoscale twinning at cryogenic temperatures. It is noted that twinning is a strain rate insensitive deformation mechanism; instead, the formation of twins is dependent on applied shear stress at a given strain rate and temperature [26]. In this study, the contribution of twinning to plastic deformation increases due to the high flow stress at low temperatures.

Figure 3(a–c) shows TEM bright field (BF) micrographs of the HEA deformed by 5%, 18%, and 32% of the

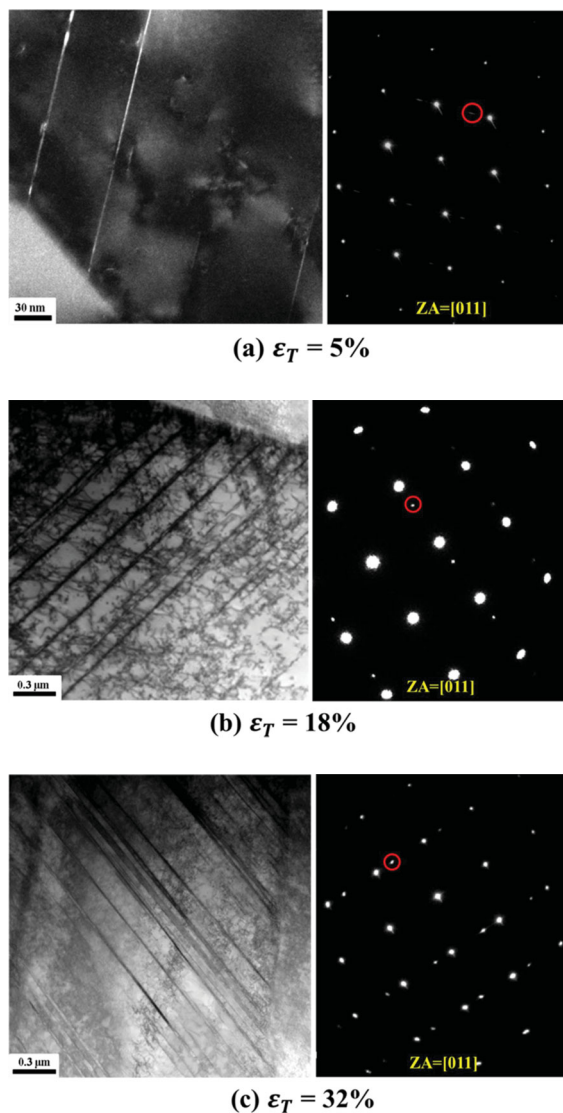


Figure 3. TEM micrographs of the alloy deformed by various strains at 77 K: true strains of (a) 5%, (b) 18%, and (c) 32%. Figures on the left are TEM BF images and their corresponding SAD patterns are on the right. The red circles show diffraction spots from the twin fault plane.

true strain at 77 K, respectively. The figures on the left are the BF images which contain mechanical twins at a given true strain. The grains were tilted to its [011] zone axis, and their corresponding selected area diffraction (SAD) patterns showing the diffraction spots of the FCC matrix and the deformation twin are indicated on the right. At a true strain of 5%, nano-twins were observed in only one grain among 20 grains. According to Laplanche et al. [8], the critical stress for twin initiation in the CoCrFeMnNi alloy is 720 ± 30 MPa. The tensile test at 77 K in the present study reveals that this critical twinning stress was already reached at the true strain of 1%. Therefore, it is evident that the twin evolution of the HEA started at an early stage of tensile deformation in this study. As the true strain increased, the volume fraction of twins increased, as seen in Figure 3(b,c). From these TEM results in Figure 3, the widths of the twins and the spacings between twins were measured.

Figure 4(a,b) shows the measured widths and spacings of twins and the calculated volume fraction of mechanical twins, respectively, plotted at the true strains at 77 K. As shown in Figure 4(a), the average twin width increases with an increase in true strain, whereas the mean twin spacing decreases. These tendencies were also observed in the work by Laplanche et al. [8]. The volume fraction of mechanical twins increases (up to 17% at true strain of 0.32) as the deformation progresses, as shown in Figure 4(b). Slip and twin can contribute to the plastic flow of the CoCrFeMnNi HEA at 77 K by both independent and interactive ways. Twin boundaries act as a barrier against dislocation movement, similar to a grain boundary, and they reduce the mean free path of dislocation glide, that is, the dynamic Hall–Petch effect occurs [27].

In general, polycrystalline FCC metals in the ultra-fine/nanocrystalline regime exhibit higher strain rate sensitivity than coarse-grained ones [28]. Lu et al. [29,30] reported that, when twin spacing decreases from the micrometer to the nanometer range, the V^* for nanotwinned Cu decreases significantly from $1000b^3$ to $10b^3$. Lu et al. [29,30] observed the appreciable formation of steps and jogs along twin boundaries, and the generation of high dislocation density around twin boundaries, especially in the vicinity of stress concentrations. They [29,30] suggested that these processes led to a significant decrease in thermal activation volume during plastic flow. In the present study, as shown in Figure 3, nanoscale twins were observed during plastic deformation at 77 K, and this can be attributed to negligible variation of V^* with true strain at 77 K.

In this case, slip and twinning can be considered to be parallel processes, and most of the volume of the specimen is deformed by slip, in which overcoming the nanoscale inhomogeneities is the rate-controlling

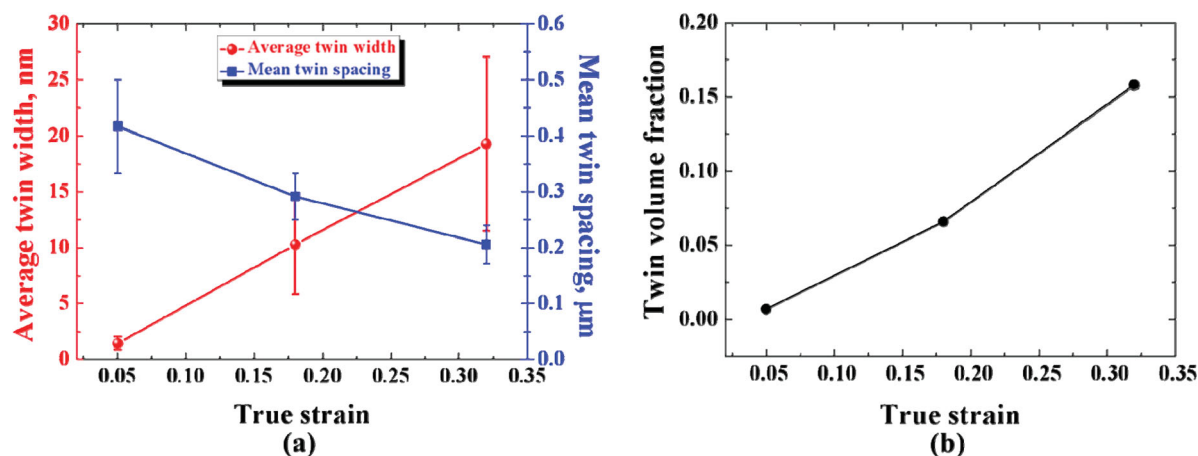


Figure 4. (a) Measured widths and spacings and (b) the calculated volume fraction of mechanical twins with various strains in the specimens deformed at 77 K.

process, as proposed by Hong et al. [11]. The volume fraction of mechanical twins increased with strain and its volume fraction increased up to 17% at the plastic strain of 0.32, which would contribute to the reduction of V^* with strain [29,30]. The insensitivity of V^* with strain at 77 K, in contrast to the gradual increase of V^* with strain at RT, is thought to be caused by the increased volume fraction of mechanical twins at 77 K. For further study, the modeling and quantification of the effect of twinning on V^* in the CoCrFeMnNi HEA at higher strains is underway.

Conclusions

To sum up, thermally activated deformation was investigated to explain the different deformation mechanisms at RT and 77 K. The strain rate sensitivity of flow stress at RT was higher than that at 77 K. The activation volume of CoCrFeMnNi HEA at 77 K was in the range of $15\text{--}19b^3$, which is much smaller than that at RT ($36\text{--}87b^3$). At low temperatures, the prevalent deformation mechanism is overcoming the nanoscale inhomogeneities, such as co-clusters and/or SROs. Slip and twinning can be considered to be parallel processes and most of the volume fraction of the specimen is deformed by slip at 77 K. The insensitivity of activation volume with strain at 77 K is caused by the combined effect of the increase in activation volume with strain by slip, and the decrease in activation volume with strain by twinning.

Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This research was supported by the Future Material Discovery Program of the National Research Foundation of Korea (NRF)

funded by the Ministry of Science, ICT and Future Planning (MSIP) of Korea [grant number 2016M3D1A1023532].

ORCID

Hyoung Seop Kim  <http://orcid.org/0000-0002-3155-583X>

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