Recovery and Recrystallization Behaviors of Ni–30 Mass Pct Fe Alloy During Uniaxial Cold and Hot Compression

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The recovery and recrystallization behaviors of the high-temperature γ -phase of carbon steel during deformation strongly affect the mechanical properties of steel. However, it is difficult to evaluate such behaviors at a high temperature. This study proposes the deformation behavior of the high-temperature γ -phase of low-carbon steel based on the quantitative observation of dislocation density and vacancies in the Ni–30 mass pct Fe alloy. This alloy was used because its stacking fault energy (60 to 70 mJ m⁻²) is similar to that of low-carbon steel. Uniaxial compression tests were conducted at a strain rate of 10⁻³ s⁻¹ and 1473 K (1200 °C) for dynamic recrystallization and at 293 K (20 °C) for work hardening. The compression process was interrupted at different strain values to systematically investigate microstructural changes. The changes in work hardening, recovery, and recrystallization behaviors were obtained from the true stress–true strain curves of the uniaxial compression tests. Further, the microstructure changes during cold and hot uniaxial compression were investigated from the viewpoint of lattice defects by X-ray diffraction, positron annihilation analysis, transmission electron microscopy, and electron backscatter diffraction to comprehend the work hardening, dynamic recovery (DRV), and dynamic recrystallization (DRX). This study helps understand the DRV, DRX, and work hardening behaviors in the γ -phase of the Ni–30 mass pct Fe alloy during cold and hot compression.

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I. INTRODUCTION

STEEL is typically produced by the thermomechanical control process with precise control of the reduction rate and heat treatment temperature to form microstructures that improve its mechanical properties. In particular, significant research has been done on grain refinement by static recovery and recrystallization during heat treatment to improve the strength and elongation in low-carbon steel.^[1–3] Conversely, the coarsening of grains is used for controlling the distribution of crystal orientation (*i.e.*, the crystallographic texture) for electromagnetic steel. In addition to the static recovery and recrystallization by cold working and heat treatment, dynamic recovery (DRV) and dynamic recrystallization (DRX) have been observed in steel materials during large-strain deformation processing.^[4–6] Currently, DRX is an important phenomenon for grain refinement. In DRX, the recrystallized grain size and

deformation stress are linked by the Zener–Hollomon (Z) factor, $Z = \dot{\epsilon} \cdot \exp(Q/RT)$, where $\dot{\epsilon}$ is the strain rate, T is temperature,^[7] Q is the material activation energy, and R is the gas constant. DRX occurs during high-temperature deformation because of the hardening caused by the multiplication of dislocations, and the softening caused by the formation of subgrains. Most of the energy added to a system dissipates as heat, while a small amount is accommodated by plastic deformation, which introduces dislocations and vacancies.^[8] Moreover, when heated above 0.5Tm (*i.e.*, melting point in absolute temperature), grains with large-strain stored energy and internal defects recrystallize to lower the overall energy of the system.

Significant knowledge about DRV and DRX has been already obtained. During recovery, the stored energy of the material is mainly lowered by annihilation and rearrangement of dislocations into lower-energy configurations, both of which are achieved by glide, climb, and cross-slip of dislocations. For materials with high stacking fault energy, such as Al alloys,^[9] α -iron,^[10] and nickel,^[11] dissociation of the perfect dislocation into two partials is more difficult, and the perfect dislocation glide, climb, and cross-slip readily occur.^[10] The driving force required to promote discontinuous DRX (DDRX), which depends on the total dislocation energy,

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is insufficient owing to the dislocation annihilation. In materials with a low SFE, such as SUS304 (21 mJ m^{-2}), dislocations are decomposed into partial dislocations to form stacking faults.^[12] Therefore, because of the dislocation pile-up, DDRX easily occurs instead of the dislocation climb and cross-slip. Regarding the nucleation of DDRX, Miura *et al.*^[13] reported that the difference between the dislocation densities of the grains causes grain boundary migration, resulting in bulging. Furthermore, Momeni and Dehghani^[14] developed DRX kinetic models for the 410 martensitic stainless steel during hot deformation. Ebrahimi *et al.*^[15] studied the DRX behavior of superaustenitic stainless steel containing 16 mass pct Cr and 25 mass pct Ni.

In carbon steel, DRX occurs in the γ -phase, the crystal lattice of which is the face-centered cubic (FCC) at high temperatures. However, the γ -phase transforms into the pearlite, ferrite, or martensite phase during cooling. This transformation behavior is strongly influenced by the grain boundary area per unit volume before the transformation, and by lattice defects such as dislocations and vacancies. Therefore, it is important to clarify the effect of rolling conditions on the grain size and dislocation density in the γ -phase at high temperatures. Steel is the most widely thermomechanically processed metal. However, the transformation of austenite during cooling has made it difficult to study the evolution of microstructures during hot deformation. Recently, model alloys (e.g., Ni–30 mass pct Fe) have been used to study the austenite behavior at high temperatures.^[16] The model alloy retains a stable austenitic structure after cooling to room temperature, allowing the high-temperature deformation microstructures to be readily characterized. In addition, the stacking fault energy (SFE) of this alloy is similar to that of pure iron and austenite in low-carbon steels.^[17] Furthermore, the behaviors of work hardening, DRV, and DRX in the γ -phase during hot rolling based on the measurement of mechanical properties, such as hardness, have been quantitatively investigated.^[18] Instead of the high-temperature γ -phase of low-carbon steel, this study used the Ni–30 mass pct Fe alloy with a low SFE of 60 to 70 mJ m^{-2} , which was characterized by transmission electron microscopy (TEM)^[19] to quantitatively investigate the lattice defect behavior during deformation. The typical changes in dislocations and vacancies caused by work hardening, DRV, or DRX in the γ -phase during cold and hot compression were systematically investigated in terms of the microstructure to understand the role of lattice defects. Deformed structures were assessed using TEM and electron backscatter diffraction (EBSD). In addition, the lattice defect behavior with respect to work hardening, recovery, and recrystallization was investigated through X-ray diffraction and positron annihilation. Then, the differences between the behaviors of dislocations and vacancies during cold and hot compression, and the distribution of the dislocation density on a uniaxial compressed plane were examined and compared with the rolling process.

II. EXPERIMENTAL PROCEDURE

A. Materials

A typical Ni–30 mass pct Fe alloy with an initial grain size of approximately 170 μm , an SFE of 60 to 70 mJ m^{-2} ,^[19] and the following impurities: 0.074 mass pct C, 0.061 mass pct Al, 0.05 mass pct Mn, and 0.005 mass pct Nb was used in this study. First, a melted and forged 100-mm-thick ingot of the Ni–30 mass pct Fe alloy was heated at 1523 K (1250 °C) for 60 minutes to obtain a homogeneous solid solution, which had a melting point of 1711 K (1438 °C). Then, the ingot was hot rolled to a 42-mm-thick rough plate. A cylindrical test piece with a diameter of 8 mm and height of 12 mm was cut from the hot-rolled plate *via* machining. Subsequently, uniaxial compression tests were performed under cold and hot conditions at a strain rate ($\dot{\epsilon}$) of 10^{-3} s^{-1} using Thermecmaster-Z (Fuji electronic industrial Co., Ltd., Japan) and an LRX-10-type load cell with an accuracy of 0.1 kN.

The cold compression test was interrupted at true strains of 0.06, 0.10, 0.15, 0.34, 0.49, and 0.77. For the hot compression test, the rough rolled plate was heated to 1473 K (1200 °C) at a rate of 5 K s^{-1} and maintained at this temperature for 60 seconds. Then, to clearly observe DRX, the hot compression test was performed at 1473 K (1200 °C), which was interrupted at true strains of 0.05, 0.09, 0.18, 0.38, 0.78, and 1.30. The cold and hot compression tests were interrupted at different true strains to observe the microstructural changes based on the characteristics of the true strain–true stress (SS) curves. In this study, true strain was defined as $\epsilon = \ln(h_1/h_2)$, where h_1 and h_2 are the specimen heights before and after compression, respectively. After hot compression, the specimen was quenched with helium gas at a rate of 90 K s^{-1} to freeze the microstructure. It required approximately 5.6 seconds to reach 0.5Tm (approximately 973 K). The microstructure and lattice defects of the cold- and hot-compressed specimens were investigated at the center of the plane perpendicular to the compression axis. We compared the typical changes that occur during compression at 293 K and 1473 K (20 °C and 1200 °C), where dynamic recrystallization is clearly observed.

B. Electron Backscatter Diffraction Observations

EBSD patterns were obtained to observe the crystal orientation distribution and strain distribution in the compression process. The samples used for EBSD were first analyzed by XRD. EBSD was performed using a crystal orientation measurement device (OIM, TSL Solution Co., Ltd.) attached to a scanning electron microscope (MERISS, Carl Zeiss Co., Ltd., Germany) in an area of 2 mm $\times$ 2.5 mm; the scanning step was 5 μm . Generally, grain boundaries provide preferential information for the hot forming processes of metals or alloys. The coincident site lattice (CSL) model is an important method for describing grain boundary characteristics. According to the CSL misorientation angle (θ) between adjacent grains, low-angle grain boundaries

(LAGBs or $\Sigma 1$ boundaries) and high-angle grain boundaries are defined as $2 \text{ deg} \leq \theta < 15 \text{ deg}$ and $\theta \geq 15 \text{ deg}$, respectively.^[20]

Therefore, grains were defined with a misorientation angle of 15 deg or less and an equivalent grain size of 15 μm or less, and measured assuming a hexagonal lattice. The strain distribution was estimated based on the grain orientation spread (GOS) and kernel average misorientation (KAM) values, which is the first neighbors only type. The crystal orientation was confirmed using inverse pole figure (IPF) maps. The GOS is the average deviation in orientation between each point in a grain and the average orientation of the grain. The KAM measures the average misorientation between a central point and its nearest neighbors. It has been shown to be dependent on the geometrically necessary dislocation (GND) density and lattice curvature.^[21] The GOS, KAM,^[22] and average KAM (KMAve)^[23] were calculated for an orientation difference of 5 deg or less using the following equations:

$$\begin{aligned} \text{GOS} &= \frac{\sum_{i=1}^n \alpha_{i,\text{Ave}}}{n}, \quad \text{KAM} = \frac{\sum_{i=1}^n \alpha_i}{6n}, \\ \text{KMAve} &= \frac{\sum_{j=1}^m (\text{KAM})_j}{m}, \end{aligned} \quad [1]$$

where n represents the number of measurement points within each grain, $\alpha_{i,\text{Ave}}$ denotes the difference between the average orientation within a grain and the orientation at the measurement points, α_i denotes the difference between the orientations of a reference point and its adjacent measurement point, and m is the number of measurement points with an orientation difference of less than 5 deg.

C. Transmission Electron Microscopy Observations

The microstructures of the cold- and hot-compressed specimens were assessed *via* TEM (JEOL2010, Japan; accelerating voltage of 200 kV) to visualize dislocation substructures. The compressed specimens used for substructure analysis were prepared using standard electropolishing techniques. Specifically, thin slices (approximately 1-mm-thick) were cut parallel to the cold-rolled surface and ground to a thickness of approximately 0.1 mm. Next, disks with a diameter of 3 mm were cut from these slices. Then, double-jet electropolishing was performed using a 10:1 solution of acetic acid and perchloric acid, with an applied potential of 40 V from 281 K to 283 K (8 °C to 10 °C).

D. X-Ray Diffraction

In metallurgical microstructures, lattice strains are induced by plastic deformation around dislocations, and LAGBs and cell boundaries develop because of the arrangement of the dislocations. The change in the characteristic shape of X-ray diffraction profiles can be used to assess the microstructure of a domain and the dislocations. The evaluation of the shape of these profiles is referred to as X-ray line profile analysis (XLPA). In the 1950s, two XLPA methods were

developed by extending the Williamson–Hall^[24] and Warren–Averbach^[25] procedures. In the 1980s, Ungár and Borbely^[26] proposed a theory that analyzes X-ray line profiles by considering the influence of anisotropic lattice strain on the crystallographic orientation and plastic deformation around dislocations. This theory is the most accurate for determining the relationship between the dislocation density and X-ray line profile shapes. According to the theory, large lattice strains occur in specific crystallographic orientations because the Burgers vector depends on the crystal system characteristics. Thus, XLPA using the mean contrast factor based on elastic anisotropy provides quantitative substructure data, such as the dislocation density, fraction of edge/screw dislocations, and arrangement of the dislocations, for various materials. The dislocation density in the cold- and hot-compressed specimens was systematically estimated using XLPA. For this purpose, disks were cut from the center of the specimens after compression, and their thickness was reduced to 1 mm using emery paper. Then, they were mechanically polished using #2000 emery paper and polished with Al_2O_3 powder to a roughness of 0.3 μm . Finally, they were finished by buffing and electropolishing to prevent any additional induction of dislocations in their surface layer while being prepared for measurement. The diffraction profiles on the $\{111\}$, $\{220\}$, $\{200\}$, $\{311\}$, $\{222\}$, $\{400\}$, $\{331\}$, $\{420\}$, and $\{422\}$ planes were measured for Bragg reflections within the 2θ angle range of approximately 10 to 60 deg using a SmartLab-II diffractometer (Rigaku, Japan) with a Mo target operated at 45 kV and 200 mA. The centers of the specimens were irradiated with 0.3 mm ϕ collimated X-rays, and the scattered radiation was registered by a two-dimensional X-ray detector (Hypix-3000, Rigaku, Japan). The diffraction profiles were separated into $\text{K}\alpha_1$ and $\text{K}\alpha_2$ components using a pseudo-Voigt function.

E. Positron Annihilation Lifetime Analysis

Positron trapping in dislocations and vacancies was measured using positron annihilation lifetime (PAL) and coincidence Doppler broadening (CDB) analyses. Positrons are sensitive probes for detecting vacancy-related defects, such as vacancies, vacancy clusters, and dislocations, in materials that lack a repulsive positive-charged nucleus. Thermalized positrons diffuse several hundreds of nanometers into a sample before annihilation, and can be trapped in a defect within the diffusion range. When a positron is trapped in a defect and annihilated therein by surrounding electrons, the data on the local electronic environment around the defect are obtained using annihilation parameters. The positron annihilation lifetime is longer in vacancy-related defects owing to their low electron density.

In this study, the specimens used for PAL and CDB measurements were electrolytically etched after mechanical polishing to a mirror surface. The measurements were performed at 293 K (20 °C). A ^{22}Na positron source deposited on a thin Kapton film was sandwiched between two identical samples. The strength of ^{22}Na positron source was approximately 1 MBq. PAL was

measured using a time spectrometer (APV8702, Techno AP, Japan) and BaF₂ scintillators with a time resolution of approximately 190 ps at full width at half maximum (FWHM). CDB measurements were performed using two Ge detectors and a digital signal processing module (APV8002, Techno AP, Japan). In the experiments, the overall energy resolution was approximately 0.9 keV at FWHM, which corresponded to a momentum (P_L) resolution of approximately $3.5 \times 10^{-3} m_0 c$ at FWHM, where m_0 is the electron/positron rest mass and c is the speed of light. The S parameter was defined as the ratio of the counts in the low-momentum region ($|P_L| < 4.0 \times 10^{-3} m_0 c$) to the total counts in the CDB spectrum. The W parameter was defined as the ratio of the counts in the high-momentum region ($P_L = 20 \times 10^{-3}$ to $28 \times 10^{-3} m_0 c$) to the total counts in the CDB spectrum.

III. RESULTS

A. True Stress–True Strain Curve

Figure 1 shows the SS curves obtained during uniaxial compression, where the arrows indicate the strains at which the compression test was interrupted. Three compression tests were repeated for confirmation. Since the SS curves of the tests performed at different final strains overlap, the curves in Figure 1 represent a single continuous test of the maximum strain. For hot compression, the decrease in true stress after reaching maximum true stress is consistent with the decrease in the true stress of the specimens, which results after the interruption at each strain indicated by the arrows. Cold compression exhibits work hardening behavior, in which true stress increases with true strain (Figure 1(a)). A DRX feature appears in the SS curve for hot compression (Figure 1(b)). True stress reaches its maximum value (σ_p) at $\varepsilon = 0.065$ and then decreases. This behavior is presumed to indicate the initiation of DRX, *i.e.*, the specimen softens owing to the formation of

dislocation-free dynamically recrystallized grains. True stress is approximately constant above $\varepsilon = 0.3$, indicating that a DRX cycle is completed.

B. Microstructure and Crystal Orientation

The crystal orientation distribution was evaluated via EBSD. Since the measurement step of EBSD is 5 μm , the nucleation of DRX grains cannot be accurately detected. However, the DRX grains in the growth stage can be detected owing to their large size. Therefore, the changes in KAM and GOS values are considered to indicate DRX grain growth. Figure 2 shows the change in IPF, GOS, and KAM in the normal direction (ND) parallel to the compression surface. Here, the legend is different because different strain values were used for the cold and hot compression tests to elucidate their typical microstructures. For cold compression, the GOS and KAM values increase with true strain (Figure 2(a)), and the large KAM close to the grain boundary, at $\varepsilon = 0.10$, indicates the accumulation of strain. The IPF map shows a {101} preferred orientation at $\varepsilon = 0.15$. That is, the {101} crystallographic texture is formed by uniaxial compression, which is the typical rolling texture of FCC structures. The area of {101} plane further increases at $\varepsilon = 0.34$. Then, slip deformation may be introduced as true strain increases. As the KAM value at $\varepsilon = 0.34$ is large, the plastic strain energy is considered to be high owing to an increase in the dislocation density. The behavior observed during hot compression differs from that during cold compression (Figure 2(b)). In the IPF map during hot compression, it is unclear whether the crystal orientation changes as true strain increases. Moreover, at $\varepsilon = 0.05$, a coarse grain is formed by heating at 1473 K (1200 °C), and the GOS is uniform. The GOS represents the degree of deformation of an entire grain by comparing the orientation at each measurement point with the average orientation inside the grain. Consequently, the GOS is small at low dislocation densities. In DRX, as in the case of ambient temperature deformation, the average cell size of

Fig. 1—True stress–true strain curves of the Ni–30 mass pct Fe alloy specimens subjected to (a) cold compression (293 K, $\dot{\varepsilon} = 10^{-3} \text{ s}^{-1}$) and (b) hot compression (1473 K, $\dot{\varepsilon} = 10^{-3} \text{ s}^{-1}$) at maximum strain. The arrows indicate the strains at which compression was interrupted for analysis.

Fig. 2—Inverse pole figure, kernel average misorientation, and grain orientation spread results in the normal direction parallel to the compression surface: (a) cold compression and (b) hot compression.

high-temperature dislocation substructures decreases during straining and the subgrain boundaries are sharpened. Finally, the nucleation of DDRX grains occurs and quickly reaches the maximum rate. At large strains, where DDRX has been propagated through the entire volume, the substructure density varies from grain to grain,^[27] i.e., a grain with a small GOS can be considered a recrystallized grain.^[28]

For hot compression, the grain size at $\epsilon = 0.09$ is smaller than at $\epsilon = 0.05$. Further, grains with a small GOS are formed, which may indicate DRX. At $\epsilon = 0.18$, numerous grains with the GOS smaller than that at $\epsilon = 0.09$ are formed. In addition, the average KAM

value slightly decreases, indicating the progress of DRX (Figure 3). Moreover, a few grains with a large GOS are formed immediately before DRX. As there is no significant change in the average KAM between $\epsilon = 0.18$ and 0.78 , DRX and recovery are considered to have repeatedly occurred. The average KAM significantly increases between $\epsilon = 0.78$ and 1.30 , indicating the multiplication of dislocations and high elastic strain energy.

The nucleation of DRX cannot be observed *via* EBSD. Therefore, the substructures of the dislocations were directly observed using TEM to monitor the nucleation of DRX (Figure 4). The magnification is

varied at each strain to observe the changes in the microstructures. In addition, different strains were selected for cold and hot compression to show their typical microstructures corresponding to the EBSD orientation map (Figure 2). The dislocation density in the as-rolled specimens ($\epsilon = 0$) is low. In cold compression, at $\epsilon = 0.10$, dislocations increase and form clusters. They further arrange at $\epsilon = 0.15$, and a cell structure is formed at $\epsilon = 0.34$ to 0.77. It is well known that the deformed microstructure of cold-rolled Ni is the Cu-type microstructure, which is mainly

formed by slip deformation;^[29–31] the deformation is due to the dislocation gliding of the $\{111\}\langle 110 \rangle$ slip system. In this study, the dislocation substructure is similar to the Cu-type microstructure. In hot compression, DRX has already started at $\epsilon = 0.05$, and recrystallized grains with a diameter of approximately $2\ \mu\text{m}$ are observed at the grain boundaries. This may have been caused by a bulging phenomenon, as widely observed in high-temperature creep deformation.^[32] Thus, dynamically recrystallized grains and a large number of subgrains are formed at $\epsilon = 0.09$. Numerous slip planes are observed at $\epsilon = 0.18$, and a large number of subgrains are formed at $\epsilon = 1.30$.

C. Dislocation Density Analysis

The dislocation density distribution on the compressed plane was evaluated by measuring it along the direction parallel to the compression surface. As shown in Figure 5(a), the dislocation density distribution significantly changes with the spread of the compressed plane owing to an increase in the true strain. The dislocation density reaches the maximum at approximately 3 mm from the center of the compressed plane at $\epsilon = 1.3$. Further, the dislocation density distribution is almost centrally symmetrical. This implies that an increase in true strain produces a large standard deviation of the mean dislocation density in conventional XRD with a large irradiated area. Fujimatsu *et al.* reported that equivalent strain is largest near the center of the specimen and smaller at the outside in cold compression by computer-aided engineering (CAE) analysis.^[33] Under our experimental conditions, the dislocation density is smallest at the center of the

Fig. 3—The average kernel average misorientation for cold and hot compression.

Fig. 4—Typical deformed microstructures observed in the compressed Ni–30 mass pct Fe alloy specimens at different strains: (a) cold compression and (b) hot compression.

Fig. 5—(a) Dislocation density distribution after uniaxial hot compression. (b) Relationship between dislocation density and true strain after hot and cold uniaxial compression and cold rolling; the cold-rolling results are from Yonemura *et al.*^[35]

compressed surface because significant dynamic recovery and recrystallization occur at the center where the strain is largest. Furthermore, Wang *et al.* used inverse finite element modeling to simulate the compression test and correlated it to the experimental SS curve.^[34] Consequently, the frictionless situation occurs where the specimen is uniformly deformed, showing ideal uniaxial strain distribution. On the contrary, for the friction condition, barreling is visible and a triaxial strain state is observed. This is corroborated by our results, where a distribution of a plastic strain in the compressed surface was generated. Therefore, XRD analysis was performed at the center of the compressed surface to analyze the dislocation density.

Figure 5(b) shows the change in the dislocation density with true strain. The dislocation density of the cold-compressed specimen is higher than that of the hot-compressed specimen. As true strain increases in cold compression, the dislocation density rapidly increases from $1.5 \times 10^{13} \text{ m}^{-2}$ ($\varepsilon = 0$) to approximately $2 \times 10^{14} \text{ m}^{-2}$ and then plateaus. The dislocation density increases by an order of magnitude owing to cold compression, which is considered to be a sufficient increase in dislocations during compression. The dislocation density may be smaller compared to conventional XRD because of selectively measuring at the center of the compressed plane (Figure 5(b)). In contrast, as true strain increases in hot compression, the dislocation density rapidly increases to approximately $6 \times 10^{13} \text{ m}^{-2}$ ($\varepsilon = 0.38$) and then plateaus. The dislocation density in cold compression is approximately three times that of hot compression. In Figure 5(b), the results of cold compression obtained in this study are compared with the cold-rolling results for the Ni–20 mass pct Cr alloy obtained by Yonemura *et al.*^[35] The dislocations introduced during the cold compression of the Ni–30 mass pct Fe alloy are equivalent to those introduced during

the cold rolling of the Ni–20 mass pct Cr alloy. As described above, the slight difference between the dislocation density during cold compression and cold rolling may be due to its uniformity because the dislocation density in the uniaxial compression was intentionally measured at the center of the compressed plane. Yonemura *et al.* reported that dislocations easily migrate up to $\varepsilon = 0.10$, which is the easy glide region. In this study, the dislocation substructure is sparse and uniform at $\varepsilon = 0.15$ (Figure 4(a)). This indicates an easy glide region at $\varepsilon = 0.15$, which is in agreement with the cold-rolling results.

D. Positron Annihilation Lifetime and Coincidence Doppler Broadening

The change in lattice defects was evaluated through PAL analysis. Figures 6 shows the changes in the mean positron lifetimes with true strain obtained *via* PAL analysis and the S – W plot obtained *via* CDB. As shown in Figure 6(a), the mean positron lifetime for cold compression is larger than that for hot compression; the former rapidly increases from 140 ps to approximately 170 ps with true strain. The mean positron lifetimes at true strains above 0.34 are larger than 157 ps, which represent the lifetimes at the dislocations,^[35] *i.e.*, the lifetime at the annihilation sites is longer than that in the dislocations. The mean positron lifetime for hot compression is lower than that for cold compression owing to the recovery of lattice defects at 1473 K (1200 °C). As the mean positron lifetime for hot compression indicates the lifetime between dislocations and the bulk (approximately 100 ps),^[36] positrons are mainly annihilated in dislocations and the bulk. Moreover, as shown in Figure 6(b), the S parameter increases and the W parameter decreases with an increase in true strain for cold and hot compression. The S and W parameters are

Fig. 6—(a) Changes in mean positron lifetime with true strain measured *via* positron annihilation lifetime analysis. (b) Relationship between the *S* and *W* parameters.

defined as $S = (1 - f)S_B + fS_D$ and $W = (1 - f)W_B + fW_D$, respectively. S_D and W_D are the *S* and *W* parameters of the positrons captured by defects, respectively, f is the fraction of the positrons captured by the defects, and S_B and W_B are the *S* and *W* parameters of the positrons captured by the bulk ($f = 0$), respectively. Furthermore, S_D and W_D are related to the defects ($f = 1$). The *S* and *W* parameters, which depend on the fraction of defects, are the interior division points of the line from (S_B, W_B) to (S_D, W_D) at $0 < f < 1$. The defect type is estimated based on the change in the slope of this line.^[37] Two solid linear correlation lines are presented in the plot. One line fits all the hot- and the cold-compression data points for $\epsilon = 0.06, 0.10$, and 0.15 , while the other line fits the cold-compression data points for $\epsilon = 0.34, 0.49$, and 0.77 . As the slope of the first line is identical to that of the dashed line from “pure iron” to “dislocations in pure iron,” this slope corresponds to the annihilation in the dislocations. The slope of the second line corresponds to the annihilation in vacancies.

IV. DISCUSSION

A. Behavior of Lattice Defects During Compression

The mean position annihilation lifetimes in Figure 6(a) include information regarding the bulk and defects, such as dislocations and vacancies. The information regarding vacancies was determined using the three-state model^[38]; the lifetime in dislocations was fixed at 157 ps. Figure 7(a) shows the change in the long lifetime component during cold compression, which is observed at $\epsilon = 0, 0.1, 0.34, 0.49$, and 0.77 . It increases up to $\epsilon = 0.1$ and then decreases. Mohamed *et al.*^[39] reported the formation of vacancy clusters during the cold rolling of pure iron based on PAL analysis, and their results corroborate with the change in cluster size

observed in this study. Consequently, the changes in dislocations and vacancies during cold compression are similar to those during cold rolling.

Compared with the lifetime for a vacancy and vacancy cluster in single Ni calculated by Ohkubo *et al.*,^[40] the lifetime obtained in this study is close to clusters V_2 – V_7 at $\epsilon = 0.34$ and clusters V_1 – V_4 at $\epsilon = 0.49$ and 0.77 (Figure 7(a)), *i.e.*, the cluster size decreases as true strain increases. Therefore, the positron intensity, which represents the density of defects, increases as the cluster size decreases. Furthermore, Yonemura *et al.*^[35] reported that edge dislocations absorb the vacancy clusters. Thus, a decrease in the long lifetime component corresponds to the annihilation of a vacancy cluster due to an increase in edge dislocations. In this study, the vacancy cluster size increases with true strain. Further, the behaviors of the dislocations and vacancies during cold compression are almost identical to those during cold rolling, as mentioned above. Therefore, it is considered that volume defect sweeping decreases the cluster size at $\epsilon > 0.34$.

Furthermore, vacancies are thermodynamic equilibrium defects; hence, cold working vacancies are less likely to form as compared to high-temperature deformation. Although the FCC structure is close-packed, where the vacancy formation and migration energies are lower, Ni provides the recombination effect. That is, acceleration of the vacancy diffusion combined with deceleration of the interstitial atom diffusion in an Fe–Ni alloy should affect the swelling behavior due to (a) decreasing the conventional dislocation bias (*i.e.*, inducing vacancy supersaturation due to the preferential absorption of interstitial atoms on edge dislocations) and (b) increasing the recombination of vacancies and interstitial atoms in the bulk.^[41] Furthermore, Osetsyky *et al.* noted that Ni strongly interacts with the periphery of clusters, affecting their mobility.^[42] The total effect is defined by the number of Ni atoms interacting with the

Fig. 7—(a) Relationship between positron annihilation lifetime analysis (PAL) and true strain. Three positron lifetime spectrum component analyses were performed for the cold-compressed specimens. In these analyses, one of the components was fixed at 157 ps to represent the positron lifetime in a dislocation (d_1). The long lifetime component in PAL analysis includes information about the vacancies (d_2). (b) Pure Fe ratio curves of coincidence Doppler broadened annihilation for uniaxial compression, normalized to the momentum distribution of annealed (defect-free) pure iron.

cluster at the same time and can be significant, even in low-Ni alloys. Moreover, increasing cluster size and Ni content can enhance cluster immobilization. We speculate that this effect results in an increase in the vacancies during cold compression in our results.

As shown in the S – W plot (Figure 6(b)), the change in slope at $\varepsilon = 0.15$ during cold compression indicates a change in the main defect type. Therefore, in cold compression, positrons are trapped in the dislocations at $\varepsilon \leq 0.15$, mainly in vacancies at $0.15 < \varepsilon \leq 0.34$, and in vacancy clusters above $\varepsilon = 0.34$. Therefore, the interaction between the dislocations and vacancies affects work hardening during cold compression. In contrast, there are no significant changes in the slope of the S – W plot for hot compression. This indicates that the positrons are mainly annihilated in the dislocations, *i.e.*, vacancies are not introduced by the strain applied in DRV and DRX during hot compression. This further indicates that dislocations contribute to deformation. Figure 7(b) shows the normalized momentum distribution of annealed (defect-free) pure iron. Based on the tendency in the high-momentum region, the curve for the as-rolled material ($\varepsilon = 0$) is located between the curves for “dislocations in pure nickel” and “dislocations and vacancies in pure nickel.” According to the increase in strain during cold compression, the curve is close to that of “dislocations and vacancies in pure nickel.” This implies that dislocations and vacancies exist in the as-rolled material, and the vacancy density increases with the strain. In contrast, the curve is close to that for “dislocations in pure nickel” at $\varepsilon = 0.05$ for hot compression. Above $\varepsilon = 0.05$, as the strain increases during hot compression, the curve approaches the curve for “dislocations and vacancies in pure nickel.” For hot compression, the curve is close to that for “pure nickel”

because the alloy recovers when heated to 1473 K (1200 °C). At $\varepsilon > 0.38$, the curve for hot compression is located between the curves for “dislocations in pure nickel” and “dislocations and vacancies in pure nickel.” This suggests that vacancies are generated and their density increases. These results are in agreement with the previously discussed PAL results.

B. Fraction of Dynamic Recrystallization

Figure 8 shows the relationship between the work hardening rate ($\theta = d\sigma/d\varepsilon$) and true strain, obtained from the SS curve of uniaxial compression. The noise of the load cell contributed to the true stress. Mannan *et al.* approximately fitted the SS curve to a ninth-order function because this noise strongly affects θ .^[43] However, we used a sixth-order function because the fitting error was sufficiently small.

For cold compression (Figure 8(a)), it was impossible to clearly approximate the data from $\varepsilon = 0$ to 0.015 and $\varepsilon = 0.74$ to 0.77. Therefore, the range of $\varepsilon = 0.015$ to 0.74 was considered. Above $\varepsilon = 0.15$, θ rapidly decreases up to $\varepsilon = 0.55$ and negligibly above $\varepsilon = 0.55$. Figure 4(a) shows a sparse and uniform distribution of the dislocations at $\varepsilon = 0.1$, even after deformation. Therefore, the dislocations migrate easily in the main slip system after the easy glide region is enlarged. In particular, it is considered that θ is large because the KAM value is large in the vicinity of the grain boundary at $\varepsilon = 0.10$ and 0.15. From $\varepsilon = 0.15$ to 0.55, θ decreases and the dislocation density gradually increases. TEM observations indicate that the increase in the dislocation density inside the grain is small because the cell structure is formed above $\varepsilon = 0.15$.

Fig. 8—Work hardening rate curves for Ni-30 mass pct Fe: (a) cold compression and (b) hot compression.

Subsequently, the SS curve obtained for hot compression was divided into four regions ($\varepsilon = 0$ to 0.04 , 0.04 to 0.3 , 0.3 to 0.7 , and 0.7 to 1.30) and approximated using a sixth-order polynomial. As shown in Figure 8(b), the local dislocation density of the dynamically recrystallized grains increases owing to the nonuniform deformation and reaches a critical strain (ε_c). Then, the true stress decreases because of the softening during DRX. ε_c can be estimated from the θ - ε diagram. However, ε_c is the strain at which DRX occurs during 1 to 5 pct of the entire process, and not the strain immediately after nucleation.^[48] The inflection point of θ before softening, which is located at $\varepsilon = 0.025$, corresponds to ε_c . Moreover, θ is negative from $\varepsilon = 0.065$ (ε_p). It reaches its minimum value at $\varepsilon = 0.12$, which indicates the maximum softening point (ε_m).

Generally, work hardening occurs in the initial stage of deformation, reaches a peak (ε_p) at a certain strain, and then decreases and reaches a steady-state value. Typically, the dislocation density at ε_c , at which DRX starts, is the critical dislocation density (ρ_c). Sakai and Jonas^[44] reported that ε_c was lower than ε_p . Further, Dehghan-Manshadi *et al.*^[45] reported that ε_c and ε_p decreased with the Zener-Hollomon parameter. In addition, Poliak and Jonas,^[46] and Najafizadeh and Jonas^[47] proposed a method for estimating ε_c by analyzing the SS relationship.

The DRX fraction (X_{DRX}) at each strain can be estimated using the following Avrami-type model equation^[49]:

$$X_{\text{DRX}} = 1 - \exp \left[-0.693 \left(\frac{\varepsilon - \varepsilon_c}{\varepsilon_m - \varepsilon_c} \right)^2 \right] \quad [2]$$

The X_{DRX} obtained from Eq. [2] corresponds with the experimental data of Ni-30 mass pct Fe-C by Mannan *et al.*^[43] (Figure 9(a)). The values are equal to the recrystallization rate of Ni-30 mass pct Fe-C processed at 1150°C with a strain rate of 0.01 s^{-1} . Although the processing temperature was 50°C higher and the strain rate was $1/10$ in the present study, the results were almost identical owing to the larger initial grain size of our specimens.

As the fraction of recrystallization at the maximum softening point is 50 pct, approximately 95 pct recrystallization is expected at $\varepsilon = \varepsilon_c + 2(\varepsilon_m - \varepsilon_c)$. ε is 0.215 at $\varepsilon_c = 0.025$ and $\varepsilon_m = 0.12$, which was estimated from the θ - ε diagram. By substituting $\varepsilon = 0.215$ into Eq. [2], X_{DRX} is obtained as approximately 0.94, which is in good agreement with the results obtained from the SS curve. Therefore, the DRX rate at $\varepsilon_m = 0.12$ is considered to be approximately 50 pct. Additionally, in the steady state, where θ approaches 0 from a negative value, strain (ε_{ss}) is 0.24; the X_{DRX} value of ε_{ss} is approximately 0.97. The values of each type of strain are provided in Table I. Mannan *et al.* investigated the DRX process in Ni-30 mass pct Fe-C through flow curve analysis and experimentally obtained the relationship between the Z value and critical strain.^[43] We applied this relationship to our data to obtain $\varepsilon_c = 0.021$, which approximately corresponds to $\varepsilon_c = 0.025$ obtained from the true strain and work hardening rate (θ). The X_{DRX} for ε_{ss} obtained by Stewart *et al.*^[49] using hypereutectoid steel ranged from 0.96 to 0.985, which corroborates with our results. From $\varepsilon = 0.79$ to 1.30 , softening and hardening are repeated and θ remains positive, which indicates work hardening.

Figure 9(b) shows the relationship between the average KAM (strain distribution) and dislocation density. From Figure 9(a), $\varepsilon = 0.25$ at the completion of recrystallization. After recrystallization is complete, the dislocations are expected to be uniformly distributed in the matrix. Below $\varepsilon = 0.25$, the change in the average KAM parameter is small compared to the increase in the dislocation density.

Here, the total dislocation consists of the density of the dislocations constituting the cell walls (ρ_w), the density of the dislocations randomly distributed within the cells (ρ_c), and the density of the dislocations present at the grain boundaries (ρ_{GB}). As the dislocations at the grain boundaries and those composing the cell walls involve misorientations, they can be grouped under the classical expression of GNDs,^[50,51] whereas the randomly distributed dislocations are called statistically stored dislocations (SSD). With TEM or SEM/EBSD, ρ_w or ρ_{GB} can be easily deduced from the local misorientation measurements. However, XRD is more

Fig. 9—(a) Dynamic recrystallization evaluation of Ni-30 mass pct Fe alloy during hot compression. (b) The relationship between the average kernel average misorientation and dislocation density.

Table I. Measured Critical Strain (ϵ_c), Peak Strain (ϵ_p), and Maximum Softening Strain (ϵ_m) and Steady-State Strain (ϵ_{ss}) Estimated From the Relationship Between the Work Hardening Rate and True Strain

ϵ_c	ϵ_p	ϵ_m	ϵ_{ss}
0.025	0.065	0.12	0.24

of a statistical method. Therefore, the analysis of the shape of a Bragg peak (especially the broadening of the peak) performed inside a grain or a texture component is used to assess one “average” dislocation density, usually estimated to be the sole density of the isolated dislocations ρ_c .^[52–55] The main arguments proposed to eliminate any influence of the dislocation boundaries observed in highly deformed materials on peak broadening are of different types. It is often claimed that statistical dislocations (SSD) can affect peak broadening. For example, this is an implicit assumption of the Wilkens model.^[56] More rarely, it is argued that the density ρ_c is by far the largest (by a factor of 10), and the other two (ρ_w and ρ_{GB}) can be considered negligible.^[52]

Recently, Wauthier-Monnin *et al.*,^[57] with the aid of additional EBSD observations and simple calculations proposed that the X-ray dislocation densities include the contribution of both cells and walls (SSD and GND). In the case that the X-ray dislocation density is dependent on the SSD and GND, pile-up dislocations near the grain boundary occurred as dynamic recrystallized grains of hot compression were observed *via* TEM. However, the pile-up dislocations are used for recrystallization, and the change in the KAM value is considered to be small compared to the increase in the X-ray dislocation density. In contrast, the slope of cold compression in Figure 9(b) is smaller than that of hot compression because the GNDs increase due to the formation of dislocation walls, which is caused by the multiplication of dislocations inside the grain.

Subsequently, we examined the relationship between the GOS and recrystallization rate. Hermant *et al.* investigated the hot deformation behavior and the associated microstructural evolution of a coarse-grained Nb-bearing austenitic stainless steel (316Nb), which is an FCC alloy.^[58] When 316Nb steel was water cooled immediately after hot working (dynamic recrystallization grain freezing), almost no dynamic recrystallization grains were observed, whereas post-dynamic recrystallization grains with DRX growth were observed after slow cooling (0.15 K s⁻¹). Then, the correlation between GOS < 2 deg and the fraction of post-dynamic recrystallization grains after cooling was obtained.

In our study, since the cooling rate after processing is approximately 90 K s⁻¹, the formation of post-dynamic recrystallization grains is expected. Therefore, the dynamic recrystallization rate calculated by the θ - ϵ diagram is the real-time dynamic recrystallization rate. However, the dynamic recrystallization rate of GOS is the post-dynamic recrystallization grains where the dynamic recrystallization grains have grown. Therefore, there was no correlation between the GOS and recrystallization rate. The recrystallization rates of the θ - ϵ diagram and GOS at $\epsilon = 0.05$ were 5 and 67 pct, respectively. It is expected that the recrystallization rate determined by the GOS is large.

Finally, the microstructure formation cause by the increase in strain is discussed. The θ - ϵ curve significantly decreases, the KAM value slightly increases, and the X-ray dislocation density and positron annihilation lifetime also increase at $\epsilon = 0.05 \sim 0.09$. Further, the nucleation of the DRX grain was observed by TEM, which implies that the DRX grain formed and was propagated by the accumulation of dislocations. The TEM observations show dynamically recrystallized grains at $\epsilon = 0.05$; thus, ϵ_c is 0.025, which is likely to be the starting point of DRX. That is, the plastic strain energy accumulated up to at least $\epsilon = 0.05$ is the driving force for the DRX and causes bulging. The dislocation density at ϵ_c for DRX with bulging is given by Eq. [3],^[12] where $\dot{\epsilon}$ is the strain rate, γ_{GB} is the grain boundary energy per unit area, b is the Burgers vector, L is the free

path of dislocations, m is the boundary mobility, and τ is the dislocation line energy.

$$\rho_{\text{cr}}^{\text{DDR}} = \left(\frac{20\dot{\epsilon}\gamma_{\text{GB}}}{3bLm\tau^2} \right)^{\frac{1}{3}} \quad [3]$$

The dislocation density at $\epsilon = 0.05$ is approximately $3 \times 10^{13} \text{ m}^{-2}$ (Figure 5(b)), which is estimated to be larger than $\rho_{\text{cr}}^{\text{DDR}}$. The standard deviation of the dislocation density distribution is small at low true strains. Furthermore, as the standard deviation of the dislocation density distribution is particularly small at the center of the specimen (Figure 5(a)), the dislocation density at ϵ_c corresponds to that measured by XRD. Therefore, the first cycle of DRX is completed at $\epsilon = 0.24$, and work hardening and softening are repeated up to $\epsilon = 0.79$.

For $\epsilon = 0.09 \sim 0.18$, the θ - ϵ curve decreases and then rapidly increases, while the KAM value, and X-rays and positrons slightly increase. Further, slip lines begin to appear, as observed *via* TEM. This is the strain at which the slip system in FCC starts to move due to compression,^[59] and significant dynamic recrystallization occurs. For $\epsilon = 0.18 \sim 0.38$, the θ - ϵ curve and the KAM value are approximately constant, and the X-rays and positrons slightly increase. At $\epsilon = 0.18$, the dislocation density is relatively small owing to the absorption of dislocations by dynamic recrystallization. At $\epsilon = 0.38$, dynamic recrystallization is complete, and work hardening and dynamic recovery are considered to be balanced. For $\epsilon = 0.38 \sim 0.78$, the θ - ϵ curve slightly oscillates, but there is no significant change in either dislocation density or the KAM value, indicating that work hardening and dynamic recovery are balanced. Above $\epsilon = 0.79$, the dislocation substructure affects the microstructure rather than the DRX behavior. In Eq. [3], L is proportional to the average spacing between the dislocations ($1/\sqrt{\rho}$); therefore, $\rho_{\text{cr}}^{\text{DDR}}$ increases with the dislocation density. The high KAM value is due to the formation of the dislocation cell at $\epsilon = 1.30$, which is considered to be caused by the increase in the dislocation density to a value below $\rho_{\text{cr}}^{\text{DDR}}$. This result corresponds with the experimental result by Mannan *et al.*,^[43] where θ increased at $\epsilon = 0.01/\text{s}$, and 1348 K, 1273 K, and 1198 K (1075 °C, 1000 °C, and 925 °C) in Ni-30 mass pct Fe-(Nb, C).

V. CONCLUSION

The microstructure changes during cold and hot uniaxial compression were investigated from the viewpoint of lattice defects using XRD, PAL analysis, TEM, and EBSD to comprehend the work hardening, DRV, and DRX behavior. Our conclusions are as follows:

1. In the uniaxial hot-compressed specimen, the dislocation density at the outside of the plane parallel to the compressed surface significantly increases with applied strain. This corresponds to the simulations of the cold compression test in the previous works. In contrast to the highest dislocation density at the

center of the compressed surface in cold compression, the dislocation density is the smallest at the center of the compressed surface by DRV and DRX in hot compression.

2. Dislocations and vacancies mainly affect work hardening in cold compression, whereas they primarily affect DRX in hot compression. Thus, the recombination effect of Ni is suggested for cold compression since vacancies are thermodynamic equilibrium defects.
3. The behavior of dislocations and vacancies during uniaxial cold compression is almost identical to that during cold rolling, as indicated by our results of X-ray dislocation density and PAL. In contrast, the dislocation density is approximately three times smaller during hot compression because dislocations are consumed by dynamic recovery and recrystallization.
4. The information regarding DRV, DRX, and work hardening obtained from the SS curves is in good agreement with the crystal orientation and strain distributions obtained *via* EBSD, the dislocation substructure obtained *via* TEM, and the lattice defect behavior obtained *via* XRD and PAL analyses.

This study helps to better understand the behaviors of DRV, DRX, and work hardening in the γ -phase of the Ni-30 mass pct Fe alloy during cold and hot compression. However, to understand these behaviors in detail, the effects of temperature and the strain rate on hot rolling should be quantitatively investigated.

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