Grain growth in soft magnetic ferritic stainless steels under compressive stresses at high temperatures

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Abstract. Precipitation-hardened ferritic stainless steels are used for soft magnetic actuators of engines, as these steels exhibit high strength and soft magnetic properties. These ferritic stainless steels are hardened by nanoscale precipitates formed during an aging process after solution treatment. Furthermore, texture control of these steels is required in order to improve the soft magnetic properties. Therefore, texture control of ferritic stainless steels with <100> fiber texture by deformation at high temperatures is attempted in this study, as it has been demonstrated that the texture of ferritic Fe-Si alloys is significantly changed by deformation conditions, including temperature and strain rate. The results demonstrate that grains with the <100> fiber texture in the present stainless steels are preferentially evolved by compressive deformation under specific strain rates at high temperatures.

1. Introduction

Soft magnetic stainless steels are widely used as magnetic core materials of solenoid valves in fuel injectors of automobiles because of their excellent soft magnetic and mechanical properties [1–8]. As their hardness (typical Vickers hardness of 85–200 HV) is lower than that required in automobile components (more than 300–350 HV), these materials are subjected to surface hardening treatments, such as hard chromium plating and/or nitriding, for the practical solenoid valves. Therefore, in order to improve the mechanical properties without the surface modification process, precipitation-hardened stainless steels have been developed with alloying elements, such as copper, nickel, titanium, aluminum, and molybdenum [1]. For example, the hardness of Fe–17Cr–4Ni–4Cu stainless steels is increased by nanometer-sized Cu precipitates [2]. In Fe–20Ni–23Co–0.07Al–0.17Ti austenitic steels, the hardening is primarily increased by precipitation of a nanometer-sized Ni₃Ti intermetallic phase in the austenite matrix [3]. A martensitic precipitation-hardened stainless steel containing nickel and aluminum is known to be hardened by fine β-NiAl precipitates [4–8]. However, studies on the application of precipitation-hardened stainless steels as a soft magnetic material have been reported, as exemplified by a precipitation-hardened soft magnetic stainless steel containing alloying elements Ni, Al, and Mo [9]. These steels exhibit high hardness (typical Vickers hardness of 370 HV) with excellent soft magnetic properties coupled with corrosion resistance. The properties are achieved by uniformly-dispersed nanoscale precipitates during adequate aging. Detailed microstructural characterization of the size and number density of the precipitates were performed using small-angle X-ray scattering (SAXS), to determine the factors controlling the mechanical properties of these steels [10].

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However, control of the microscopic texture of the steels is also required, in order to improve the soft magnetic properties of these ferritic steels. It is interesting to note that the preferential texture evolution has been controlled in ferritic steel by deformation at high temperatures [11, 12]. In this process, the low density of small angle grain boundaries and a high fraction of <100> oriented grains are achieved by deformation at low strain rates. Therefore, it is interesting to investigate the texture evolution of the <001> and <111> components in the present ferritic stainless steels under different conditions.

2. Experimental
The samples used in this study were a ferritic steel of Fe-14.5Cr-3Ni-2Mo-1Al-1Si (in mass%). The samples for the uniaxial compression deformation test were comprised of a cylinder of φ10 × H15 mm, which is prepared from bars produced by Tohoku Steel Co. Ltd. These samples were compressed under different strain rates at high temperatures between 973 and 1073 K. The microstructure and texture of the samples deformed up to 0.75 in true strain were characterized by analyzing the cross section of the cylindrical samples using electron backscatter diffraction (EBSD). The inverse pole figure (IPF) maps and the volume fractions of <100> and <111> texture components in the sample were primarily investigated in this work. The results of the texture components obtained by EBSD were compared with the process conditions at high temperatures.

3. Results and discussion
3.1. Mechanical properties of soft magnetic stainless steels at high temperatures
Figure 1 shows the true-stress-true-strain curves of the samples deformed under different strain rates at 1073 and 1173 K. These results demonstrate that flow stress is lower at higher deformation temperatures under a constant strain rate; flow stress is lower under lower strain rates at a constant temperature. Furthermore, the dislocation density in the sample appears to be unchanged during deformation, since the curves reveal little work hardening.

In order to discuss the deformation mechanism of the samples at high temperatures, the relationship between the strain rate and true stress of the samples deformed under different conditions are plotted, as shown in Figure 2. The flow stress, σ, is related to the strain rates at high temperatures as follows:

\[
\sigma = A \left[ \varepsilon \exp \left( \frac{Q}{kT} \right) \right]^{1/n},
\]

where \( \varepsilon \) is the strain rate, \( Q \) is the activation energy for deformation, \( k \) is Boltzmann constant, \( T \) is temperature, and \( n \) is the stress index. The value of \( n \) (stress index) is often utilized in the discussion on the deformation mechanism and dislocation motion at high temperatures [13]. The present value of \( n \) is determined from the slope of the plot shown in Figure 2 to be about 3.9 at 1173 K and about 4.9 at 1073 K. It is expected that the deformation process is controlled by phenomena including diffusion of solute atoms or grain boundary sliding in materials, when the value of \( n \) approaches 1. However, the high \( n \) values in the present cases suggest that the dislocation motion in the matrix is dominant at high temperatures, where accumulating energy by dislocations increases and some deformation texture is formed in the present ferritic steels.
3.2. Texture evolution under different strain rates at high temperatures

Figures 3 (a), (b), and (c) show IPF maps of the samples compressively deformed up to 0.75 in true strain under $5 \times 10^{-4} \text{s}^{-1}$, which were deformed at 973 K, 1073 K, and 1173 K, respectively. The results demonstrate that the red region showing the $<100>$ texture component increases and the blue region showing the $<111>$ texture component decreases in the sample deformed at higher temperatures.

Pole figures of $\{001\}$ and $\{111\}$ in the samples compressively deformed under $5 \times 10^{-4} \text{s}^{-1}$ at 973, 1073, and 1173 K are shown in figures 4 (a), (b), and (c), respectively. These results indicate that $<100>$ and $<111>$ texture components in the samples deformed at different temperatures are changed under a constant strain rate. In particular, the volume fraction of the $<100>$ texture component increases and a texture component is formed in the direction perpendicular to compression with increasing deformation temperature. It is noted that the orientation distribution in the circular direction in the pole figures is inhomogeneous, which is thought to result from evolution of an initial texture component due to anisotropic elasticity of ferritic steels at high temperatures.
Figure 4. Pole figures of {001} and {111} in the samples compressively deformed under $5 \times 10^{-4}\text{ s}^{-1}$ at (a) 973, (b) 1073, and (c) 1173 K.

Figure 5 summarizes the area fraction of the $<100>$ and $<111>$ texture components in the samples deformed under $5 \times 10^{-4}\text{ s}^{-1}$ at 973, 1073, and 1173 K. This also indicates that dynamic grain growth occurs at high temperatures, and the texture evolution depends on the deformation conditions at high temperatures.

3.3. Optimum conditions to control the $\{100\}$ texture

Figures 6 (a), (b), and (c) show IPF maps of the samples compressively deformed under $5 \times 10^{-3}\text{ s}^{-1}$, $5 \times 10^{-4}\text{ s}^{-1}$, and $5 \times 10^{-5}\text{ s}^{-1}$ at 1173 K, respectively. Taylor factor maps of the sample area corresponding to these IPF maps of the samples compressively deformed under $5 \times 10^{-3}\text{ s}^{-1}$, $5 \times 10^{-4}\text{ s}^{-1}$, and $5 \times 10^{-5}\text{ s}^{-1}$ at and 1173 K are shown in figures 7 (a), (b), and (c), respectively. These results reveal that grains with the $<100>$ texture in IPF maps exhibit lower Taylor factors, and grains with the $<111>$ texture exhibit higher ones. Therefore, it is considered that the dislocation density is higher in $<111>$ grains than in $<100>$ grains, indicating that the accumulating energy is high in grains with the $<111>$ texture. As the flow stress is lower under low strain rates, as shown in the true-stress-true-strain curves, the dislocation density is considered to be low under such conditions, where the accumulating energy for grain growth is relatively low. Thus, the dislocation density is lowered by lowering the flow stress, and the accumulating energy for grain growth decreases, resulting in a decrease in the $<100>$ component.

Figure 5. Area fraction of the $<100>$ and $<111>$ texture components in samples deformed under $5 \times 10^{-4}\text{ s}^{-1}$ at 973, 1073, and 1173 K.
Figure 8 summarizes the area fraction of the <100> and <111> texture components in the samples compressively deformed under $5 \times 10^{-3}$ s$^{-1}$, $5 \times 10^{-4}$ s$^{-1}$, and $5 \times 10^{-5}$ s$^{-1}$ at 1173 K. The optimum strain rate to obtain the large <100> texture is not $5 \times 10^{-3}$ s$^{-1}$ but $5 \times 10^{-4}$ s$^{-1}$. One of the reasons for this is that the strain rate is related to the adequate deformation time, since the dynamic grain growth or preferential evolution of grains with the <100> texture occurs by creating a balance between adequate deformation temperature and time.

**Figure 6.** IPF maps of samples compressively deformed under $5 \times 10^{-3}$ s$^{-1}$ (a), $5 \times 10^{-4}$ s$^{-1}$ (b), and $5 \times 10^{-5}$ s$^{-1}$ (c) at 1173 K.

**Figure 7.** Taylor factor maps of the samples compressively deformed under $5 \times 10^{-3}$ s$^{-1}$ (a), $5 \times 10^{-4}$ s$^{-1}$ (b), and $5 \times 10^{-5}$ s$^{-1}$ (c) at 1173 K. The red region reveals a high Taylor factor, while the blue region reveals a low one.

**Figure 8.** Area fraction of the <100> and <111> texture components in samples compressively deformed under $5 \times 10^{-3}$ s$^{-1}$, $5 \times 10^{-4}$ s$^{-1}$, and $5 \times 10^{-5}$ s$^{-1}$ at 1173 K.
In addition, the sample after compressive deformation under $5 \times 10^{-4} \text{ s}^{-1}$ at 1173 K was annealed at 1173 K for 30 minutes without any static applied stress, to investigate the influence of static applied stress on the microstructure. However, the volume fractions of the $<100>$ and $<111>$ texture components are not significantly changed by annealing without applied stress. This suggests that the $<100>$ texture component is almost unchanged irrespective of static applied stress, and applied flow stress is an important part of the formation of the $<100>$ component. Thus, preferential dynamic grain growth (PDGG) fundamentally occurs in ferritic alloys at adequate strain rates and temperatures [11]. This phenomenon is achieved by the strain-induced grain boundary migration due to the difference in stored energy between grains with different crystal orientations. Since grains with the $<100>$ orientation have a lower Taylor factor than those with $<111>$, other deformation texture components during uniaxial deformation are expected to occur as a result of a lower dislocation density in $<001>$ grains than in $<111>$ grains. Therefore, it is considered that $<100>$ oriented grains expand by consuming $<111>$ oriented grains, causing the total stored energy in the bulk to be reduced. The present results also indicate that the PDGG occurs in practical ferritic stainless steels.

4. Concluding remarks

The present results indicate that grains with the $<100>$ texture component preferentially evolve during compressive deformation under low strain rates at higher temperatures. The optimum conditions for the $<100>$ texture evolution are a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ at 1173 K, although the soft magnetic properties should be further investigated. The dislocation density is considered to be lowered during deformation under low flow stress, where the evolution of the $<100>$ texture component is suppressed. When the samples are annealed without applied stress at high temperatures after loading, the $<100>$ and $<111>$ texture components were almost unchanged, indicating that dynamic flow stress is important in the texture formation. By decreasing flow stress, the dislocation density decreases and the $<100>$ texture component evolves, which implies that the $<100>$ texture formation during deformation is affected by the deformation conditions and the dislocation density variation due to flow stress.

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