AlN Precipitation in Dual-phase 3% Si Electrical Steels

Jae-Hoon OH, Sang-Hyun CHO¹⁾ and John J. JONAS¹⁾

Si Steel Technical Team, Cold Rolling Department, Pohang Iron & Steel Co., Ltd., Pohang, Korea.

1) Department of Metallurgical Engineering, McGill University, 3610 University Street, Montreal, H3A 2B2 Canada.

(Received on October 2, 2000; accepted in final form on February 14, 2001)

The existing approaches to the detection of precipitation are largely based on room temperature measurements on quenched samples. However, the direct measurement of precipitation kinetics at high temperatures is also possible through analysis of the mechanical properties of the specimens. In this work, a creep method was developed and applied to the detection of aluminum nitride precipitation in a dual phase 3% silicon steel containing 0.038% C. Prior to loading, the specimens were solution treated for 20 min and then cooled to the test temperature. A constant stress was applied to each sample by means of a computerized MTS machine and the strain was recorded continuously during testing. Microstructural examination revealed that the austenite fraction and morphology and the microstructure of the ferrite matrix are quite different depending on whether samples are directly heated or heated and then cooled to the test temperature. The resulting creep rate is sensitive both to the occurrence of precipitation as well as to phase transformation; when the microstructure remains fixed, the slope of the true strain-log(time) curve decreases immediately after the initiation of precipitation. The precipitation-time-temperature diagrams determined in this way are of classical C shape.

KEY WORDS: electrical steel; dual-phase steel; precipitation; aluminum nitride; phase transformation; creep; hot deformation; grain oriented silicon steel.

1. Introduction

Electrical steels, or silicon steels, are used in electric power applications, typically as magnetic core materials for transformers, electric motors and generators.^{1–4)} Broadly speaking, electrical steels can be classified into two categories, one being grain-oriented and the other non-oriented. In the grain-oriented steels, the highly aligned texture is developed through a process called secondary recrystallization. In the manufacture of grain-oriented 3% Si steels containing the (110)[001] Goss texture, two essential requirements must be met to foster the process of secondary grain growth: i) a grain growth inhibitor, MnS, MnS+AlN *etc.*, must be added; ii) a suitable prior structure of completely recrystallized fine grains must be present, some of which contains the cube-on-edge Goss component.^{5–7)}

These two requirements are in turn affected by the slab reheating and hot rolling processes. The slabs or ingots are usually reheated at a high temperature prior to roughing and finishing. By such a treatment, the grain growth inhibitors are dissolved and subsequently reprecipitated as a fine dispersion of particles during hot rolling. After hot rolling is completed, the basic distribution of the inhibiting particles has been established, although it can still be modified by subsequent heat treatment. A more profound understanding of the nucleation behavior of precipitates during hot deformation is therefore desirable. During slab reheating, because of the very high temperatures involved (1250°C), some of slab surface is melted and accumulates on the floor of the furnace. Thus the furnace must be repaired after each

production campaign. Some researchers have therefore studied the use of lower slab reheat temperatures; in these cases, they employ AlN as the inhibitor instead of MnS or MnS+AlN.^{8,9)}

Although aluminum nitride (AlN) with manganese sulfide (MnS) has been extensively used as an elementary “inhibitor” in commercial silicon steels, the precipitation of AlN without MnS has not been as widely explored. Since precipitates of AlN and MnS are of considerable importance in improving the final texture and, therefore, the magnetic properties of these steels, it is also of interest to study the precipitation of AlN in the absence of MnS precipitation. Because it has been found that large particles only exhibit a very small pinning effect on grain boundaries, it is important to know how to produce a fine dispersion of AlN precipitates during hot rolling. Thus the monitoring of AlN precipitation at high temperatures is of considerable interest and requires detailed investigation.^{10–12)}

As is well known, a large number of studies have dealt with the precipitation of sulfides or nitrides. By contrast, there has been little research related to the precipitation of aluminum nitride in electrical steels.^{13,14)} Furthermore, metallurgists often concentrate on the hot working of steels in the single-phase austenite or single phase ferrite range. However, the hot deformation and heat treatment of 3% silicon steels in industrial practice are carried out within the ferrite range (from 80 to 90% volume fraction) of dual-phase steels; thus the events occurring during these processes are particularly interesting and less familiar. Although the precipitation of Ti(C,N) in such dual-phase

Table 1. Chemical composition in weight percent of the steel tested.

C	Si	Mn	P	S	Sol-Al	N
0.038	3.0	0.2	0.01	0.01	0.010	0.0095

Fig. 1. Fe-Si phase diagram, showing the effect of two C levels.¹⁶⁾

materials has been studied by using advanced analytical methods such as electron microscopy, diffraction and spectroscopy, there has been little research related to analysis of the mechanical properties exhibited during creep, stress relaxation *etc.*

In this work, a creep method was developed and applied to the detection of aluminum nitride precipitation in a dual-phase 3% silicon electrical steel.

2. Experimental Procedure

2.1. Materials

An electrical steel was used in the present investigation of AlN precipitation during hot deformation. This steel, in the form of 39 mm thick hot rolled plate, was provided by POSCO, Pohang Iron & Steel Co. Ltd., Korea. The chemical composition is listed in **Table 1**. The soluble aluminum was 0.010% and the total nitrogen was 0.0095%. The silicon level was 3% and the carbon content was 0.038%; the latter causes this steel to have an austenite volume fraction of 10 to 20% within the hot rolling temperature range,¹⁵⁾ see **Fig. 1**.¹⁶⁾

Compression samples 11.9 mm in height and 7.9 mm in diameter were machined from the as-received plates, with their longitudinal axes parallel to the rolling direction. These dimensions were chosen based on studies that indicated that an aspect ratio of 1.5 promoted homogeneous deformation during compression testing.¹⁷⁾

2.2. Thermomechanical Treatment Schedule

The level of the preset stress must be carefully selected if precipitation events are to be investigated by means of the present creep method. In the current work, various prior thermomechanical treatment schedules were executed at constant strain rate in order to estimate this stress level. These schedules are represented schematically in **Fig. 2**. The first step in the heat treatment schedule was to vary the soaking temperature. Each specimen was soaked for 20 min at a test temperature in the range from 1 000 to 1 280°C. A constant strain rate (10^{-4} s^{-1}) was then applied to the sam-

Fig. 2. Thermomechanical treatment schedules for determination of the applied stress. **Fig. 3.** Thermomechanical treatment schedule for a creep test.

ples after cooling to the test temperature (*e.g.* 1 000°C) and the stress was recorded continuously. In this way, it was found that the microstructure present prior to a test has a significant effect on the results. This is because it affects the flow curves determined during testing.

2.3. Hot Compression and Creep Testing

The creep experiments were performed on the same computerized materials testing machine set up for constant strain rate testing. This machine is basically made up of an automated MTS testing system of 25 kN capacity and an induction furnace. The compression test output was connected to a computer using the OS/2 operating system for data acquisition; this also provided a graphical user interface for the compression machine. To produce the constant true strain rate needed for the first set of experiments, the actuator displacement speed had to be reduced in proportion to the decreasing specimen height. A computer program was generated for this purpose using the MTS TestStar software; it enabled the crosshead displacement speed to be controlled in steps.

The thermomechanical treatment schedule used for the present creep tests is presented in **Fig. 3**. Each specimen was solution treated prior to application of the load for 20 min at the solution temperature. As soon as the solution treatment was completed, the specimen was cooled to the test temperature. The test temperatures ranged from 900 to 1 100°C for the present steel. On attaining the test temperature, a holding interval of 1 min was employed to permit the specimen temperature to become approximately uniform. A constant stress was then applied to the specimen for up to 1 hr and the strain was recorded continuously. During each creep test, the temperature was held constant to within $\pm 1^\circ\text{C}$.¹⁸⁾

Fig. 4. Typical flow curve obtained at a strain rate of 10^{-4} s^{-1} at 1000°C .

3. Results and Discussion

3.1. Creep Method for Single-phase Steels

The present creep method was developed by Sun *et al.*¹⁹⁾ and is well suited for detecting precipitation in both single phase ferritic and single phase austenitic steels. Nevertheless, before each creep test, the level of the preset stress has to be carefully selected; thus preliminary tests were carried out to determine the flow curves of the steels at a strain rate of 10^{-4} s^{-1} directly at each temperature of interest without employing any prior solution heat treatment.

One of the true stress–true strain curves established in this way at 1000°C is shown in Fig. 4. The steady state stress under these conditions, in which the net rate of work hardening is approximately zero, was hard to find. Furthermore, this stress–strain curve differs somewhat from the flow curves that are typical of ferrite or austenite; ferritic steels follow the dynamic recovery shape of curve, while austenitic steels follow the dynamic recrystallization shape of curve. In Fig. 4, the flow curve initially displays a negative slope; then, in the region between strains of 0.2 and 0.4, it approaches a steady-state value. The stresses defined at the largest strains were employed as the applied stresses for the creep tests at each test temperature of interest.

Oscillations can be seen in Fig. 4 and there are two possible explanations for this phenomenon. They could be caused by DSA (dynamic strain aging), even though this phenomenon is not frequently found at high temperatures. In general, DSA, when caused by the interstitial elements carbon and nitrogen, occurs between 150 and 300°C , depending on the strain rate. The diffusivities of the substitutional elements are many orders of magnitude lower than those of the interstitial elements. Much higher temperatures are therefore required in order to develop the type of DSA caused by substitutional elements compared to when DSA is generated by the diffusion of interstitial elements. Recently, high temperature DSA phenomena have been reported by Cho *et al.* in 304 stainless steel.²⁰⁾ Furthermore, the diffusion coefficients of the Si and Mn contained in the present material at 1000°C are similar to those of carbon and nitrogen when DSA occurs at temperatures between 150 and 300°C .

Another possible explanation for the oscillations involves the Kurdjumov–Sachs relationship. When austenite forms in the present type of dual-phase steel on heating, it obeys the Kurdjumov–Sachs correspondence relationship. It is

Table 2. Austenite volume fractions after 60 min at 1000°C (vol%).

Soak Temperature ($^\circ\text{C}$)	Prior to test	After deformation
1000	12.1	2.8

Fig. 5. Typical true strain curve obtained by heating directly to 1000°C using creep testing. The data are plotted against (a) linear time and (b) log time.

therefore coherent and acts to inhibit deformation in the softer ferritic matrix. The work hardening rate is also increased. During deformation, the austenite loses its coherency and therefore its hardening effect. This, together with the decreasing volume fraction of the austenite, will lead to flow softening. This decrease is confirmed in Tables 2 and 4, from which the austenite fraction can be seen to decrease from 12.1% prior to testing to 2.8% after deformation.

From Fig. 5, it can be seen that steady state creep sets in after about 400 s, Fig. 5(a). When the data are plotted semi-logarithmically, Fig. 5(b), the true strain increases smoothly as $\log(\text{time})$ increases and no plateau is present on this curve. Here, the strain is increasing relatively rapidly because the applied stress is too “high”. Under these conditions, it is hard to detect the initiation or completion of AlN precipitation.

The dependence of the microstructure and austenite volume fraction on the deformation conditions is illustrated in Fig. 6 and summarized in Table 2. As shown in the table, when the load was applied to the sample, the transformation was accelerated by the strain, *i.e.* a strain-induced transformation took place. Under these conditions, the amount of austenite decreased from 9.8 to 2.8 vol%; this is one reason why it was difficult to define the steady-state stress.

Fig. 6. Microstructure of the present steel after deformation at 1000°C compared with that of the undeformed material: (a) total strain=0.36 and (b) undeformed (F=ferrite, A=austenite).

Fig. 7. Flow curves obtained at a strain rate of 10^{-4} s^{-1} and 1000°C after preheating for 20 min at: (a) 1000°C, (b) 1150°C and (c) 1280°C.

The true stress–true strain curves established in this way at 1000°C are shown in **Fig. 7**. A steady state of flow is readily seen in the 1150°C (curve b) and 1280°C (curve c) soak temperature tests. By contrast, there is flow softening when the sample was simply heated to the test temperature, curve a. As stated previously, such softening is probably due to: i) the gradually decreasing volume fraction of austenite (Table 2); and ii) the loss of coherency of the austenite phase resulting from deformation.²¹⁾ Because of the significant flow softening taking place in the absence of solution heat treatment, the soak temperature for preheating was fixed at 1150°C for all the creep tests.

The true stress–true strain curves that resulted when the influence of transformation during deformation was reduced to a minimum are presented in **Fig. 8**. These samples were soaked at 1150°C for 20 min prior to cooling to the test temperature, *i.e.* 900, 1000 or 1100°C. Then a constant

Fig. 8. Typical flow curves obtained at a strain rate of 10^{-4} s^{-1} at: (a) 900°C, (b) 1000°C and (c) 1100°C.

Table 3. Applied stresses employed in the creep experiments.

Test temperature (°C)	900	1000	1100
Applied stress (MPa)	10–12	5–7	3–5

strain rate of 10^{-4} s^{-1} was applied to the samples. The values defined in this way were employed as applied stresses for each creep test temperature of interest (**Table 3**). In **Fig. 8**, the dynamic transformation of austenite-to-ferrite at 900 and 1000°C seems to lead to the appearance of oscillations on the flow curve, which gradually disappear. This can be attributed to the very low volume fractions of austenite present at large strains. The effect is missing at 1100°C because, although the volume fraction of austenite is higher, it is more stable.

To investigate the effect of soak temperature on the austenite volume fraction and morphology, samples were preheated to temperatures of 1000, 1150, 1280°C for 20 min prior to testing at 1000°C. The microstructures after heat treatment but prior to testing are displayed in **Fig. 9** and the austenite volume fractions are shown in **Table 4**. When the soak temperature of the sample prior to testing was increased to 1150 and 1280°C, the austenite volume fraction also changed. The austenite morphologies after heating to 1150 and 1280°C were similar and differed from that of the 1000°C sample.

3.2. Creep Testing of the Dual-phase Steels

Some of the true strain–log(time) curves acquired by the present creep technique on the dual-phase electrical steel are illustrated in **Fig. 10**. In these figures, the slopes of the creep curves first increase during loading and then decrease after some seconds of creep. The points on the curves identified here as P_s can be associated with the initiation of precipitation during creep. Unfortunately, the plateau on the strain curve is not level enough for the precipitation finish time to be identified. In the earlier work carried out by Sun and Jonas¹⁹⁾ on a MnS-containing electrical steel, the plateaus were nearly horizontal, making it relatively easy to estimate the P_f times. This may be because the MnS precipitates were finer than the present AlN particles. Some transmission electron microscopy must therefore be carried out to establish the particle size distribution in the present material and to verify this hypothesis. For the successful production of Goss-oriented material by secondary recrystallization, the AlN precipitates must be suitably fine. Thus the chemistries of AlN-based electrical steels and their pro-

(a) (b)

(c)

Fig. 9. Microstructures after preheating prior to testing at 1000°C at: (a) 1000°C, (b) 1150°C and (c) 1280°C (F=ferrite, A=austenite).

Table 4. Austenite volume fractions at 1000°C after preheating for 60 min at the temperatures shown.

Soak temperature (°C)	Prior to test	After deformation
1000	9.9	2.8
1150	15.6	7.8
1280	16.4	8.7

Fig. 10. True strain-log(time) curves obtained by creep testing at 900°C under the following loads: (a) 553 N (12.2 MPa), (b) 500 N (11 MPa) and (c) 462 N (10.2 MPa).

cessing conditions must be appropriately optimized if this approach is to be successful. As shown in Fig. 10, when the load is increased, the precipitation start time, P_s , is shortened. Precipitation is accelerated because the dislocation

(a) (b)

(c)

Fig. 11. Microstructures of the present electrical steel after reheating at 1280°C and then creep testing at: (a) 900°C, (b) 1000°C and (c) 1100°C (F=ferrite, A=austenite).

Table 5. Austenite volume fractions after creep at different temperatures (reheating temperature: 1280°C).

Creep temperature (°C)	After preheat	Prior to test	After deformation
900	22.9	8.9	5.3
1000	22.9	16.4	7.6
1100	22.9	18.8	21.7

density increases with stress or strain rate.

The P_s time can be evaluated more precisely using the second derivative of the strain with respect to log(time). This derivative changes from positive to negative at P_s and becomes positive again at P_f . Thus P_s and P_f can be defined by employing the equations proposed by Sun and Jonas.¹⁹⁾ Nevertheless, when the creep test temperature was increased, it became hard to detect AlN precipitation. This can be explained quite simply in terms of the reduced volume fraction and larger mean diameter of the particles under these conditions.

Some typical microstructures after reheating at 1280°C and then creep testing are illustrated in Fig. 11 and the austenite volume fractions are shown in Table 5. The austenite volume fraction and microstructure morphology produced at 900 and 1000°C differ sharply from that resulting from 1100°C testing. When the creep experiments were carried out at 900 and at 1000°C, the austenite volume fraction decreased. By contrast, the austenite volume fraction of the sample creep tested at 1100°C changed lit-

Fig. 12. Austenite volume fractions: (a) on heating, (b) on cooling after soaking at 1280°C, (c) on cooling after soaking at 1150°C, and (d) deformed after soaking at 1280°C.

tle. This means that the austenite-to-ferrite transformation is accelerated by deformation, particularly when the sample temperature is decreased from the preheat temperature of 1280°C to test temperatures below 1100°C.

The formation of Widmanstätten austenite along the grain boundaries and within the grains can be seen quite clearly in Figs. 11(a) and 11(b). It has been reported²²⁾ that the austenite in dual-phase steels obeys the Kurdjumov–Sachs relationship with respect to the ferrite. For example, the $\{011\}_\alpha$ planes are parallel to the $\{111\}_\gamma$ planes, while the $\langle 111 \rangle_\alpha$ and $\langle 110 \rangle_\gamma$ directions are also parallel. Thus, when the austenite phase forms by solid state transformation of the ferrite, it is coherent with the latter. When these particles are coherent with the matrix, stress fields are present around the “precipitates”. This is because coherency increases the degree of interaction between the second phase and the glide dislocations. In Fig. 11(c), the 1100°C austenite is spheroidized and is therefore incoherent. Such spheroidization appears to be possible during creep at 1100°C (but not at 900 and 1000°C) because of the much higher diffusivities that apply to this temperature.

In the present study, the effects of soak temperature and deformation on the volume fraction of austenite were studied by subjecting a series of specimens to different thermo-mechanical processing schedules. Some samples were heated directly to respective test temperatures of 900, 1000, 1220 and 1280°C. Other samples were cooled to 900, 1000 and 1100°C after soaking at 1150 and 1280°C. Still other samples were deformed at 900, 1000 and 1100°C after soaking at 1280°C for 20 min. The associated austenite volume fractions are illustrated in Fig. 12.

When the soak temperature of a given sample was increased, the austenite volume fraction increased. The austenite volume fractions and morphologies after heating to 1150 (curve c) or 1280°C (curve b) were similar and differed from that of the 1000°C sample. They also differed from those of the directly heated samples (curve a). When a creep experiment was carried out at either 900 or 1000°C, the austenite volume fraction decreased and the grain size increased significantly (curve d). The austenite volume fraction of a sample creep tested at 1100°C changed little but the austenite was coarsened.

Based on the foregoing observations, it is clear that the austenite-to-ferrite transformation in the present dual-phase steel is accelerated by deformation (curve d, 900 and

Fig. 13. Microstructures of the present steel after different hold times at 1000°C after solution treatment at 1280°C: (a) 1 min, (b) 60 min and (c) 60 min during deformation under a constant load of 265 N (5.82 MPa) (F=ferrite, A=austenite).

1000°C). In the cases of the 900 and 1000°C tests, when samples are cooled after soaking at temperatures higher than the test temperature, the austenite volume fraction decreased. Some of the austenite phase present prior to deformation transforms into ferrite. When deformation is applied to such samples, the austenite-to-ferrite transformation is accelerated by the presence of the dislocations.

The effect of deformation on the microstructure of the ferrite formed dynamically is illustrated in Fig. 13. In order to compare the microstructures, samples were quenched after 1 min (a) and 60 min (b) without deformation, and 60 min after deformation (c) at a constant load of 265 N (*i.e.* a stress of 5.4 MPa).

From metallographic examination of the quenched 1000°C samples, it is evident that the slightly deformed ferrite grains increased sharply in size but that the undeformed sample grain size increased only slightly. By contrast, when large deformations were applied to two ferritic steels, the grains decreased in size. Belyakov *et al.*²³⁾ and Baczynski and Jonas²⁴⁾ both described the effect of large deformations on grain size, the former on stainless steel and the latter on ferritic steels. In both cases, the ferrite grains decreased in size with the formation of new grains by the growth of subgrains and a gradual increase in the sub-boundary misorientation. These discrepancies seem to be associated with the magnitudes of the strains and strain rates. Under creep conditions (low strains and strain rates), the sub-boundary misorientation is unable to increase and strain-induced boundary migration (SIBM) probably takes place instead.

Table 6. Precipitation start times and strain rates as determined by the present creep technique.

Creep temperature (°C)	Applied stress (MPa)	P _s time (s)	Strain rate (10 ⁻⁶ s ⁻¹)
900	10	255	53
	11	239	79
	12.2	167	95
1000	4	246	34
	5.9	95	108
1100	3.1	355	38

Fig. 14. PTT curve for the dual-phase 3% Si steel acquired by the present creep technique. The points represent data obtained at strain rates of: (a) $3.4 \times 10^{-5} \text{ s}^{-1}$ and (b) $1.1 \times 10^{-4} \text{ s}^{-1}$.

3.3. Precipitation–Time–Temperature Diagram

In order to collect the experimental results and represent them in a single diagram, the P_s and strain rate data gathered on the electrical steel are listed in **Table 6**. These results are also presented in **Fig. 14** in the form of a precipitation–time–temperature (PTT) diagram. It is apparent from this figure that the curve for AlN precipitation in the present dual-phase steel is consistent with the generally observed classical C-shape, with the nose located at a minimum time of about 95 s at 1000°C. It is of interest that the nose is located at or near the temperature at which the volume fraction of austenite is at its maximum; *i.e.* between 1000 and 1100°C. Iwayama and Haratani¹⁴⁾ determined an AlN PTT curve in a somewhat similar steel using the electrochemical method; in their case, the nose of the curve was located at a minimum time of about 15 s at 1150°C. This difference probably arises because the carbon content of the present material, *i.e.* about 0.04% C, is significantly lower than that of the Iwayama and Haratani steel, 0.05% C.

Another matter of note involves the possible indirect effect of C level on the driving force for precipitation. For example, Kononov and Mogutnov²⁵⁾ suggested that the solubility of MnS in 3% silicon steel depends on the carbon content. An increase in carbon concentration raises the solution temperature sharply; this is the temperature below which the solution becomes supersaturated, leading to an increase in the driving force for MnS precipitation at a fixed temperature. Here, the change in the precipitation kinetics is more likely to be related to the state of deformation of the specimens. For example, the rate of AlN precipitation in

the steel studied by Iwayama and Haratani¹⁴⁾ was more rapid than in the present material. This difference can be justified in that their strains (and therefore their dislocation densities) were higher than ours. Such differences are also likely to affect the particle size distribution.

3.4. Effect of Strain Rate on Precipitation Start Time

In order to extend the present PTT results to mill conditions, the effect of strain rate should be taken into consideration. This is because mill strain rates fall in the range $1\text{--}200 \text{ s}^{-1}$, while the present experiments were carried out using a strain rate of $3.4 \times 10^{-5} \text{ s}^{-1}$. In general, increasing the strain and strain rate leads to an increase in the dislocation density, thus reducing the start time for precipitation.²⁶⁾

The Dutta and Sellars model²⁷⁾ for the precipitation start time is given below:

$$t_{ps} = A \varepsilon^{-1} Z^{-0.5} \exp \frac{Q_d}{RT} \exp \frac{B}{T^3 (\ln k_s)^2} \dots\dots\dots (1)$$

In this equation, A and B are constants, ε is the strain, and Z is the Zener–Hollomon parameter, given by $\dot{\varepsilon} \exp(Q/RT)$. Q_d is the diffusion activation energy of the precipitating species in austenite and k_s is the supersaturation ratio.

In the above equation, the precipitation start time (t_{ps}) and the strain rate are related (through the Z term in Eq. (1)) as follows:

$$t_{ps} \propto \dot{\varepsilon}^{-0.5} \dots\dots\dots (2)$$

The result of increasing the strain rate from $3.4 \cdot 10^{-5}$ to 200 s^{-1} is illustrated in **Fig. 15**. It can be seen that the precipitation start time is advanced from about 246 s (curve a) to about 0.1 s (curve c). However, the above relation is based on precipitation occurring in the austenite phase and the phases in the present sample consist of austenite and ferrite in a ratio of 1 : 4. The effect of strain-induced precipitation in the ferrite phase is expected to be less than in austenite because dislocations are harder to accumulate and easier to recover in ferrite. It can therefore be hypothesized that the precipitation start time (t_{ps}) and the strain rate may be alternatively related as follows:

$$t_{ps} \propto \dot{\varepsilon}^{-0.3} \dots\dots\dots (3)$$

Thus the exponent in the above power relationship has an uncertainty ranging from -0.3 to -0.5 . The two alternative “high strain rate” curves are illustrated in **Fig. 15**.

4. Summary and Conclusions

In this investigation, a modified mechanical method was employed to study the precipitation of AlN in a dual-phase 3% Si electrical steel. The AlN precipitation start times were determined by this method and a PTT diagram was established for strain-induced precipitation in this way. As described above, it was difficult to establish the correct applied stress for each creep test since a strain-induced transformation occurred during deformation. Oscillations on the flow curves and mechanical softening during hot deformation were also observed. The conclusions drawn in this work are summarized below.

(1) The volume fraction of austenite in the present

Fig. 15. Estimated effect on the precipitation kinetics of increasing the strain rate from 3.4×10^{-5} to 200 s^{-1} . Curve (a) represents the experimental data determined at $3.4 \times 10^{-5} \text{ s}^{-1}$. Curves (b) and (c) were calculated using a modified Dutta and Sellars equation.

dual-phase 3% Si electrical steel when cooled to a test temperature is higher than when it is directly heated to the same temperature. During the newly developed type of test, the creep rate is sensitive to both the occurrence of precipitation as well as to that of transformation. When the influence of the latter is reduced to a minimum, the slope of the creep strain-log(time) curve decreases after the initiation of precipitation.

(2) The PTT diagram determined by the present creep technique for AlN precipitation is generally C-shaped, with the nose located at a minimum time of about 95 s at 1000°C . Using a precipitation model, the laboratory results obtained at low strain rates were extrapolated to the industrial rolling range, which involves much higher strain rates, leading to start times in the 1 s range.

(3) During deformation, oscillations were observed on every flow curve. These could be caused by DSA (dynamic strain aging), as the diffusion coefficients of the Si and Mn contained in the present material between 900 and 1100°C are similar to those of carbon and nitrogen when DSA occurs at temperatures between 150 and 300°C .

(4) The austenite volume fraction of samples crept at 900 and 1000°C decreased significantly but that of specimens crept at 1100°C remained constant or increased a little. The decrease in volume fraction seems to be responsi-

ble for the flow softening observed at the two former temperature temperatures. Some contribution to the flow softening may also come from the loss of coherence during deformation of the Widmanstätten austenite phase.

Acknowledgment

The authors gratefully acknowledge the financial support of the Pohang Iron & Steel Company.

REFERENCES

- 1) R. A. Hadfield, W. F. Barrett and W. J. Brown: *Inst. Elect. Eng.*, **31**, (1902), 674.
- 2) *Nippon Steel Tech. Rep.*, **310** (1982), 150.
- 3) N. P. Goss: *Trans. Am. Soc. Met.*, **23** (1935), 511.
- 4) *Kawasaki Steel Tech. Rep.*, **21** (1989), No. 3, 93.
- 5) F. Assmus, K. Detert and G. Ibe: *Z. Metallkd.*, **48** (1957), 344.
- 6) A. Sakakura: *J. Phys. (France)*, **IV 5** (1995), No. 7, 219.
- 7) *Nippon Kokan Tech. Rep.*, **21** (1983), Special issue, 127.
- 8) X. Sun, Z. He, Y. Chen, H. Ma and Z. Liu: *Proc. Int. Magnetic Conf.*, '84, (1984), 26.
- 9) K. S. Choi, C. S. Lee, B. D. Hong and J. S. Woo: US Patent 5653821, (1997).
- 10) T. B. Massalski, J. L. Murray, L. H. Bennett and H. Baker: *Binary Alloy Phase Diagrams*, Vol. 2, ASM, Metals Park, OH, (1986) 1108.
- 11) K. I. Arai and K. Ohmori: *Metall. Trans. A*, **17A** (1986), 1295.
- 12) S. Nakashima, K. Takashima and J. Harase: *Metall. Mater. Trans. A*, **28A** (1997), No. 3, 681.
- 13) G. A. Jeffrey and V. Y. Wu: *Acta Crystallogr.*, **16** (1963), 559.
- 14) K. Iwayama and T. Haratani: *J. Magn. Magn. Mater.*, **19** (1980), 15.
- 15) E. Gunlich: *Wiss. Abhandl. Physik-techn. Reinsantadt.*, **4** (1918), 467.
- 16) W. C. Leslie, R. L. Rickett, C. P. Stroble and G. Konovat: *Trans. Am. Soc. Met.*, **53** (1961), 715.
- 17) R. A. Petkovic: Ph. D. Thesis, McGill University, Montreal, (1975).
- 18) W. P. Sun: Ph. D. Thesis, McGill University, Montreal, (1991), 72.
- 19) W. P. Sun, W. J. Liu and J. J. Jonas: *Metall. Trans. A*, **20A** (1989), 2707.
- 20) S. H. Cho, Y. C. Yoo and J. J. Jonas: *J. Mater. Sci. Lett.*, **19** (2000), No. 22, 2023.
- 21) O. Balancin, W. A. M. Hoffmann and J. J. Jonas: *Metall. Mater. Trans. A*, **31A** (2000), 1353.
- 22) J. M. Robinson and M. P. Shaw: *Int. Mater. Rev.*, **39** (1994), No. 3, 113.
- 23) A. Belyakov, R. Kaibyshev and R. Zaripova: *Mater. Sci. Forum*, **113-115** (1993), 385.
- 24) J. Baczynski and J. J. Jonas: *Metall. Trans. A*, **29A** (1998), 447.
- 25) A. A. Kononov and B. M. Mogutnov: *ISIJ Int.*, **39** (1999), No. 1, 64.
- 26) I. Weiss and J. J. Jonas: *Metall. Trans. A*, **11A** (1980), 403.
- 27) B. Dutta and C. M. Sellars: *Mater. Sci. Technol.*, **3** (1987), 197.