Control of Inhibitor Precipitation for Producing Grain-Oriented Silicon Steel

T. Obara, H. Takeuchi, T. Takamiya, and T. Kan

Keywords

magnetic properties, precipitation, silicon steel

1. Introduction

GRAIN-ORIENTED electrical steels have been widely used for various products, and hence, improving their properties is greatly desired to thus save electricity. Magnetic properties of grain-oriented 3% Si steels have been steadily improved since Goss invented this steel, as shown in Fig. 1. The authors are convinced that the possibility of further improvement still exists. Techniques for improvement include reducing the thickness of steel sheet and minimizing misorientation from (110)[001] texture.[1] Following this principle, Arai et al.[2] recently showed that superior magnetic properties could be obtained by using tertiary recrystallization for thinner gage (110)[001] textured 3% Si steel. This result reveals that further possibilities for improvement may exist even in conventional grain-oriented 3% Si steel.

T. Obara, H. Takeuchi, T. Takamiya, and **T. Kan,** Iron and Steel Research Laboratories, Kawasaki Steel Corporation, Kurashiki, Japan

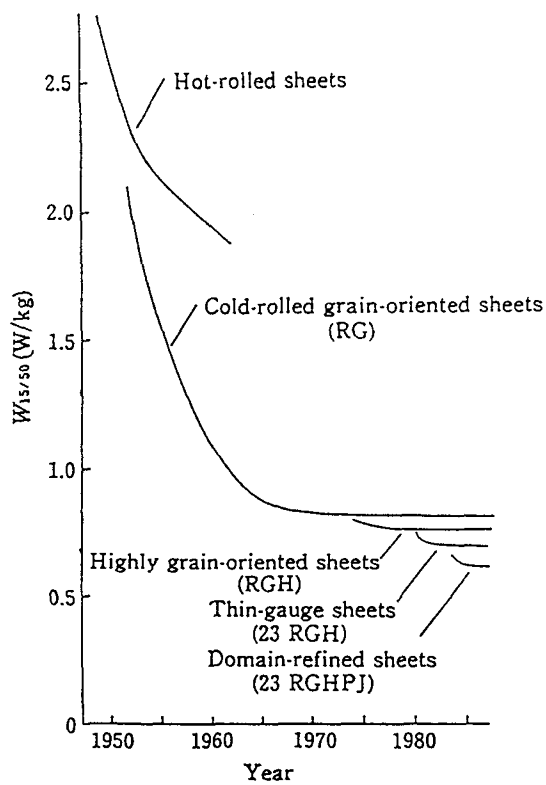

Fig. 1 History of iron loss improvement in 3% Si steel sheet.

An important metallurgical basis for producing grain-oriented Si steel is controlling the primary recrystallization texture and the dispersion of inhibitors. Mechanisms of secondary recrystallization and the influence of primary texture on it have been discussed by Harase et al.[3]

Conditions for obtaining the optimum dispersion of an inhibitor have not been described fully, although analysis is quite important to attain optimal secondary recrystallization. Many processing factors are already optimized empirically, but it is evident that a complete understanding of the precipitation phenomena will provide further possibilities for improvement. Many types of precipitates such as MnS, MnSe, AlN, and Cu_2S have been proposed as inhibitors for secondary recrystallization.[4-6] Precipitation processes of these inhibitors, however, have not been well clarified. In the present article, the details of precipitation behavior of MnS are studied to determine the optimum processing conditions.

It is well known that the inhibitor plays its role at the secondary recrystallization stage. Three important metallurgical factors related to the inhibitor exist in the process: dissolution, precipitation, and Ostwald ripening. The most important of these processes is precipitation, which is achieved primarily during hot rolling and comprises a cooling process and a deformation process. The purpose of this article is to clarify the metallurgical sequence of precipitation during hot rolling and then to improve the process by the application of this information to obtain improved magnetic properties.

2. Experimental Procedure

Vacuum-melted extra-low C steel ingot containing S with the chemical composition as shown in Table 1 was hot rolled to 40 mm thick. Specimens with 8-mm diameters and 12 mm high were machined from a hot rolled sheet bar. Thermomechanical treatment was performed by the facility with an induction heating and compression system. Solution treatment was done at 1623 K for 600 s. After solution treatment, specimens were rapidly cooled to various temperatures between 1573 and 1073 K, held for 10 to 60 s with or without 50% compression, and then cooled to room temperature. For comparison, specimens cooled to room temperature prior to aging were also studied.

Table 1 Chemical composition of test samples

Composition, wt%					
C	Si	Mn	S	P	Al
0.002	3.06	0.068	0.018	0.001	0.002

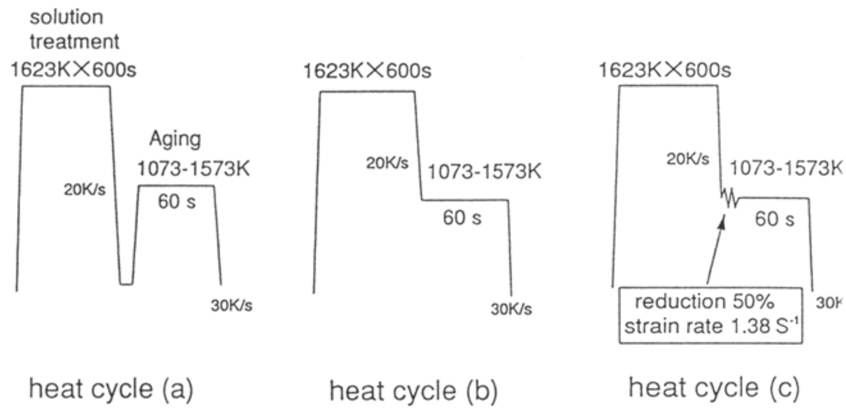

Fig. 2 Heat cycles studied in the present experiment.

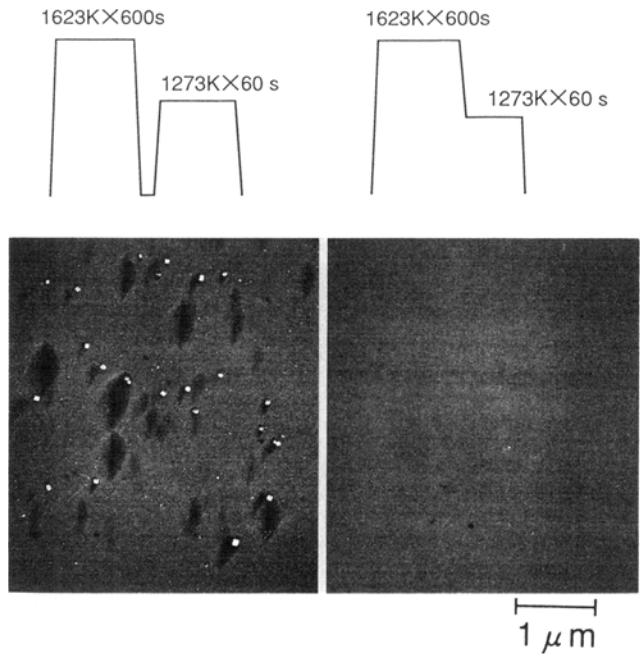

Fig. 3 SEM photos showing the effect of thermal history prior to aging.

The heat cycles studied are shown in Fig. 2. Microstructures and precipitates were observed by optical microscopy and also transmission electron microscopy (TEM).

3. Results

3.1 *Effect of Heat Cycle*

As shown in Fig. 3, dense precipitation of MnS was observed in the specimen cooled to room temperature prior to the aging treatment at 1273 K for 60 s, as shown in Fig. 2 heat cycle (a), but no precipitates were observed in the specimen held at 1273 K for 60 s without supercooling after the solution treatment, as shown in Fig. 2 heat cycle (b). Fine precipitates were observed in the specimen deformed prior to aging at 1273 K for 60 s after solution treatment. Precipitation was strongly accelerated by supercooling prior to aging or deformation even at high temperatures. In the actual hot rolling process, supercooling does not occur, and the results from heat cycle (a) cannot be applied to the analysis. Deformation during processing is considered another important factor. Therefore, the experiment in the present study was based on the heat cycle (c) in Fig. 2.

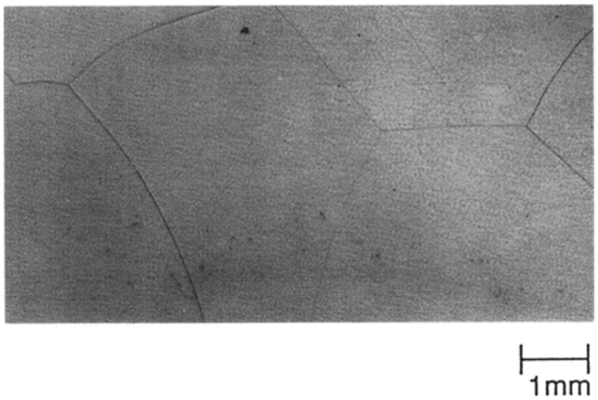

Fig. 4 Optical microstructure of specimen after solution treatment at 1623 K for 600 s.

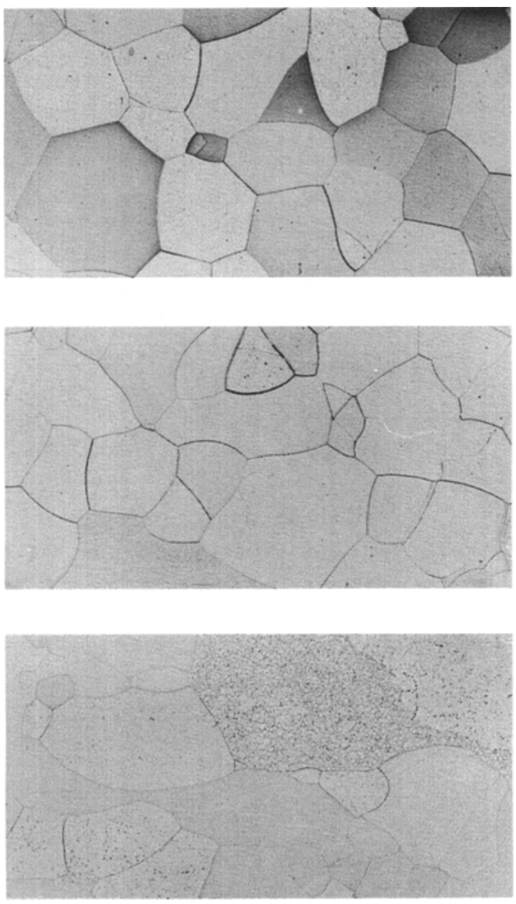

Fig. 5(a) Optical microstructures of specimens deformed 50% and subsequently aged at various temperatures for 60 s.

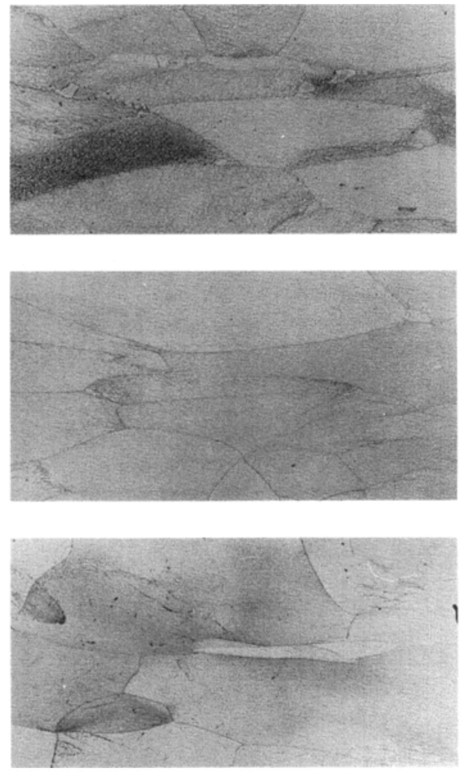

Fig. 5(b) Optical microstructures of specimens deformed 50% and subsequently aged at various temperatures for 60 s.

3.2 *Effect of Holding Temperature*

Effect of deformation temperature on microstructure was first observed. As shown in Fig. 4, a large grain size was observed in the specimen after solution treatment at 1623 K for 600 s without deformation. Recrystallization occurred in the specimen deformed 50% at relatively high temperatures above 1373 K, as shown in Fig. 5(a), but recrystallization occurred minimally below 1273 K because of rapid recovery[5] prior to recrystallization. Large subgrains 100 μm diameter or more were observed in the specimen deformed at 1273 K, as shown in Fig. 6. The scanning electron microscopy (SEM) and TEM studies were performed in these specimens. No precipitation was observed in the specimen deformed and aged above 1473 K. Figure 7 shows the SEM results. Relatively large and inhomogeneously dispersed MnS precipitates were observed in the specimen deformed and aged at 1173 K. Fine and homogeneously dispersed MnS precipitates were observed in the specimen deformed and aged at 1073 K. As shown in Fig. 8, preferential precipitation at subgrain boundaries and dislocations were observed in the specimen deformed at 1173 K. Preferential nucleation on these defects is the cause of the inhomogeneous precipitation. Fine and homogeneous dispersion of precipitates was observed in the specimen deformed at relatively low temperatures, typically at 1073 K.

4. Discussion

From a metallurgical understanding, it is suggested that supercooling or deformation just before aging has a significant effect on the precipitation process. The present experiment confirmed that pre-aging condition influenced precipitation of

MnS. Heat cycle (c) in Fig. 1 comprises the necessary factors for the actual process, e.g., rapid cooling, deformation at high temperature, and holding, and hence studying this type of heat cycle provides adequate information.

The result is summarized in Fig. 9. It was found that the homogeneity of the precipitation depended strongly on the microstructure, and the changes in the dispersion of the precipitates were clearly defined by the deformation temperature. It is expected that, at high temperatures, recovery of the deformed microstructure proceeds quite rapidly before nucleation.[7] Figure 10 illustrates how rapidly recovery can occur. Dislocation density was promptly reduced in a few seconds at this temperature range, and thus, a relatively stable dislocation network remained as subboundaries. At lower deformation temperatures, dislocation and other defects must remain for a period of time after deformation, and hence, the possibility of nucleation may increase. This causes dense and homogeneous precipitation. A schematic illustration of this relationship between the recovery rate of the deformed microstructure and the nucleation rate of MnS is shown in Fig. 11.

It is well known that finely and homogeneously dispersed precipitation is preferable as an inhibition for secondary recrystallization. Many efforts have been devoted to obtain the optimum dispersion of MnS. Practical conditions are studied in connection with Fig. 11. The present result suggests that deformation in the optimum temperature range (A) results in fine and homogeneous precipitation. In the temperature range B where inhomogeneous precipitation occurred, deformation and long time holding should be avoided. In the actual hot rolling process, the condition in this temperature range has to be controlled very carefully. At the high temperature range (A), precipitation barely occurs, and hence, a severe problem does not exist. Depending on the production facility, the thermomechanical process for MnS precipitation should be controlled via a time-temperature-precipitation (T-T-P) diagram. A T-T-P diagram depends on chemical compositions. MnSe is known as an inhibitor similar to MnS and induces better magnetic properties than MnS.[1] The dispersion of MnSe processed by the same heat cycle as the present experiment is finer and more homogeneous than MnS, as shown in Fig. 7. It was also confirmed that Ostwald ripening of MnSe is slower than MnS due to its small diffusion coefficient.[8] Thus, obtaining fine and homogeneous precipitation and the prevention of Ostwald ripening in the process before secondary recrystallization are important to better magnetic properties. Considering the dispersion of the inhibitor observed in the actual processing of steel sheet, there is

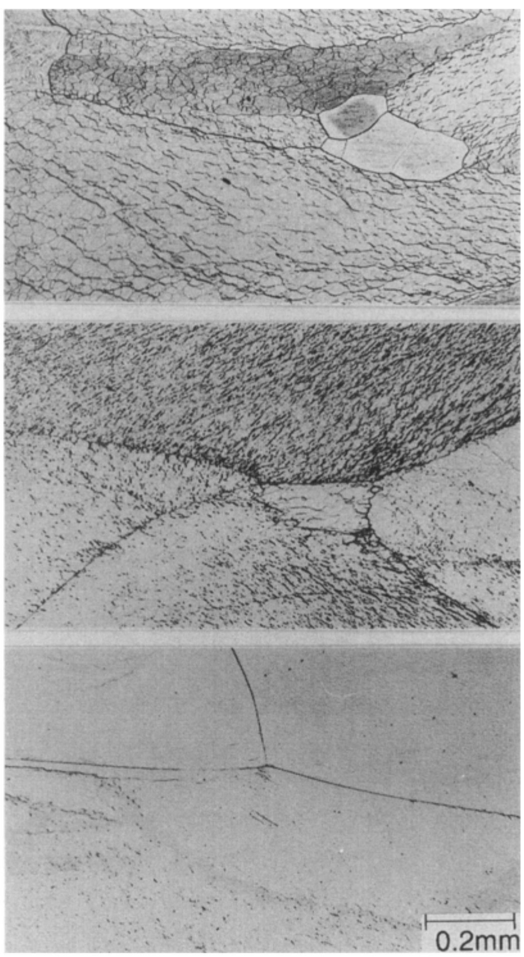

Fig. 6 Optical microstructures at high magnification of the same specimens shown in Fig. 5(b). Subgrains are visible.

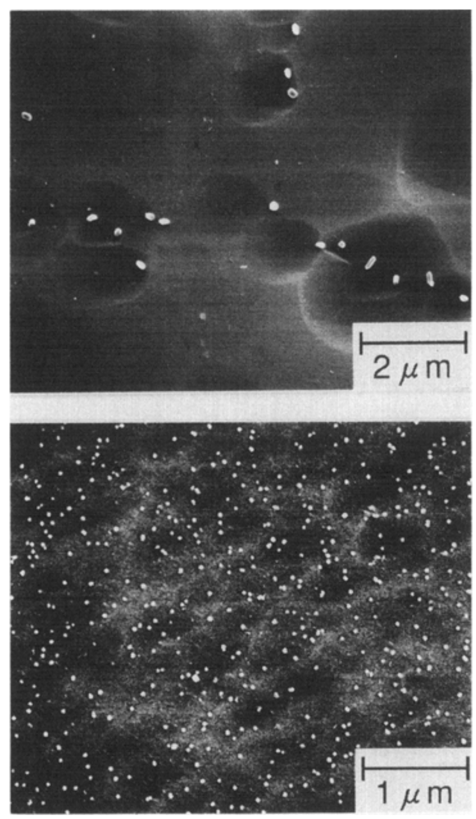

Fig. 7 MnS particles observed by SEM in the specimens deformed 50% at 1173 or 1073 K and aged for 60 s. No precipitation was observed above 1473 K.

still the possibility for improvement of the dispersion of the inhibitor.

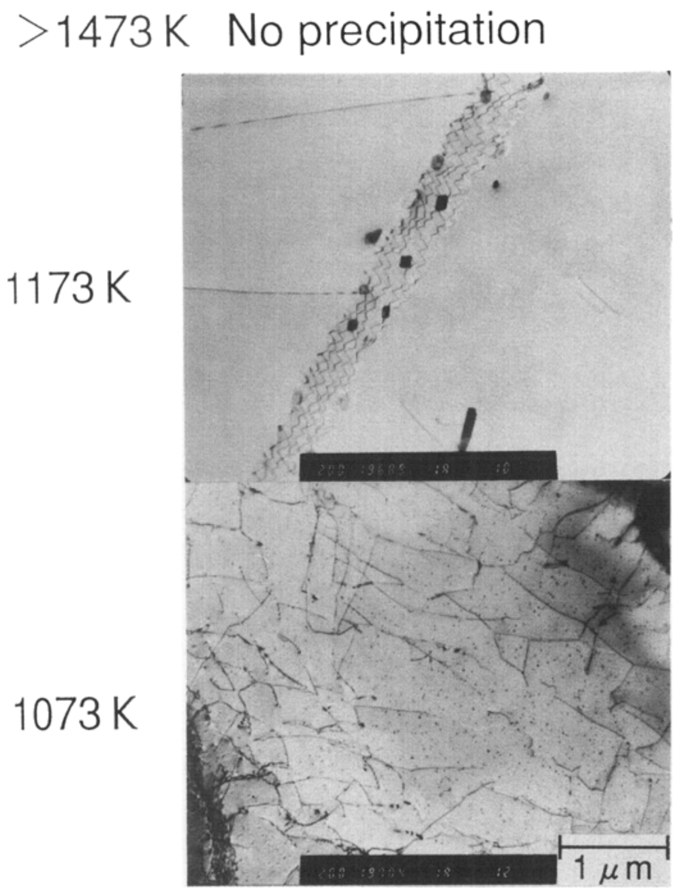

Fig. 8 TEM photographs of the specimen deformed 50% and aged at 1173 or 1073 K and aged for 60 s. No precipitation was observed above 1473 K.

5. Conclusion

Precipitation behavior of MnS was studied to improve the uniformity of the dispersion, and results are summarized as fol-

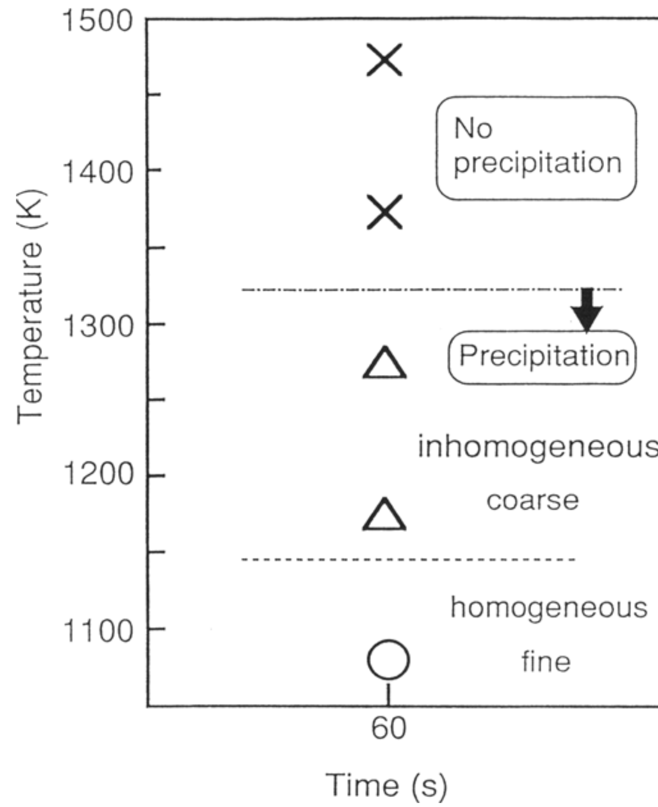

Fig. 9 Schematic showing the effect of deformation and aging temperature on the dispersion of MnS.

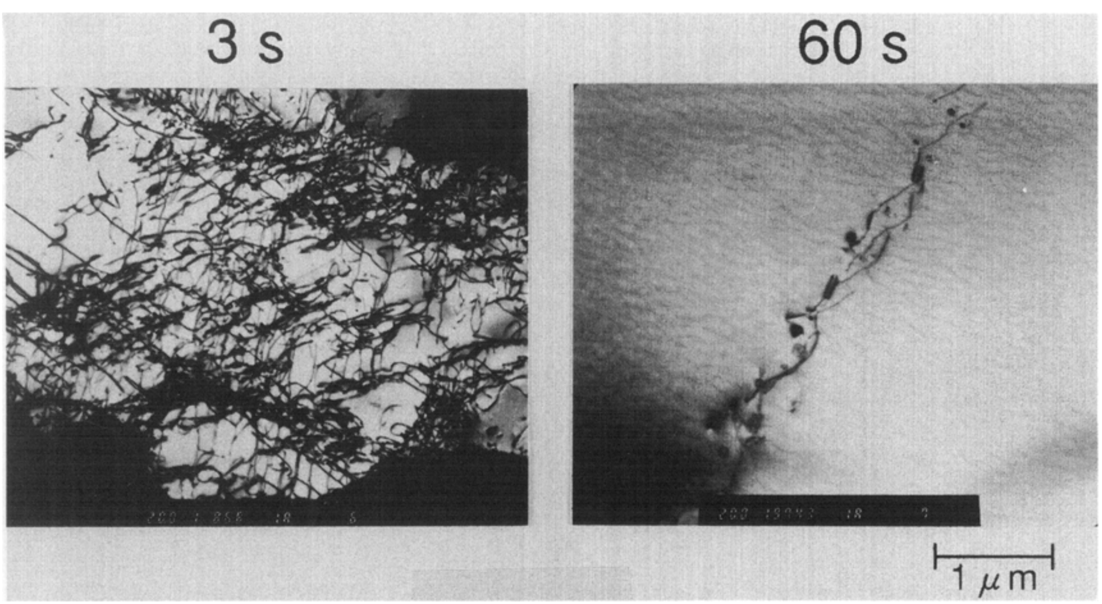

Fig. 10 Recovery of deformation structure of specimen deformed 50% at 1273 K and subsequently held for 3 or 60 s.

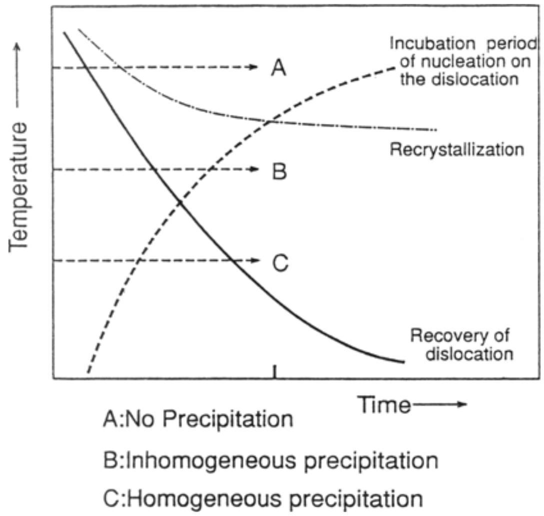

Fig. 11 Schematic showing the relationship between the recovery rate of the deformed structure and the incubation time for nucleation of MnS. A: No precipitation. B: Inhomogeneous precipitation. C: Homogeneous precipitation.

lows. The pre-aging condition was important to the precipitation behavior. MnS precipitation is accelerated by deformation. Deformation prior to aging was necessary. MnS precipitates primarily on the dislocations introduced by deformation. Minimal MnS precipitation occurred at high temperatures above 1373 K.

Dislocation density is relatively low in samples held between 1273 K and 1173 K due to the rapid recovery of the structure. As a result, MnS tends to precipitate inhomogeneously. Dislocation density is relatively high in samples held at lower temperatures due to the slow recovery of the structure, resulting in a homogeneous precipitation of the MnS. Hot rolling conditions and other processing variables have been optimized by taking into consideration the metallurgical information discussed above.

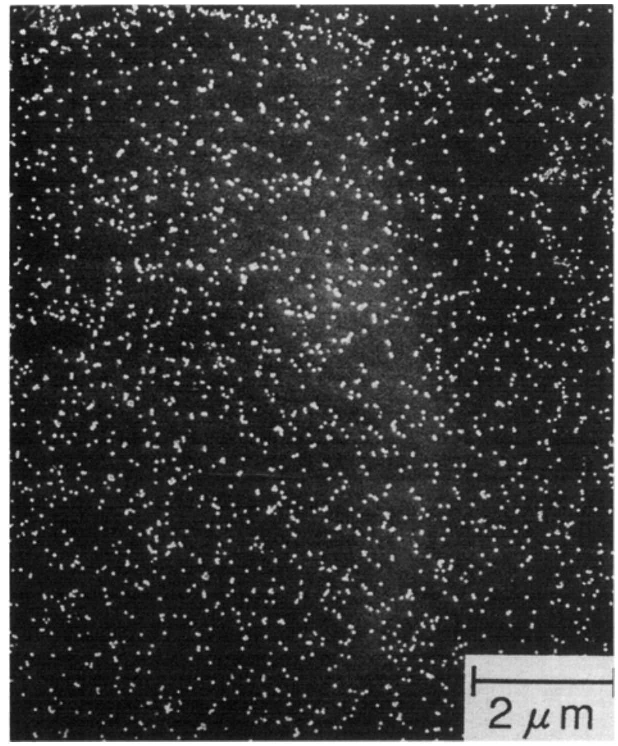

Fig. 12 SEM photograph of MnSe particles in specimen deformed 50% at 1173 K and held for 60 s.

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