# CREEP PROPERTY OF AUSTENITIC STAINLESS STEEL WITH LATH STRUCTURE FORMED BY DISPLACIVE REVERSION[1]

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## Abstract

The effectiveness of microstructure control using displacive reversion on creep property was verified in a metastable austenitic stainless steel with a chemical composition of Fe-14%Cr-11%Ni. The steel plates were cold-rolled until the austenite phase fully transforms to deformation-induce α' martensite, and then rapidly heated to 923K to reverse the martensite to austenite. By the thermomechanical treatment, fine lath austenitic structure containing high-density dislocation was formed with a mechanism of displacive reversion from deformation-induced α' martensite to austenite. The thermomechanical-treated specimen with lath structure had approximately two times longer creep fracture lifetime compared to the specimen with equiax-grained structure. This results means that the microstructure control to lath structure effectively enhances creep property of austenitic heat-resistant steel.

**Key words:** Martensitic reversion; Austenite; Lath structure; Creep.

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## 1 INTRODUCTION

Since the power generation efficiency in thermal power plant has to be increased to reduce emission of $CO_2$ in terms of global environmental protection, heat-resistant steels are strongly required to have a good mechanical property in a tougher environment at higher temperature and pressure.(1) Therefore, it is important to optimize the alloy design and microstructure of the heat-resistant steels. As for a practical ferritic heat-resistant steel with lath martensitic structure (α' ferritic heat-resistant steel), an excellent creep property is realized by combination of various strengthening mechanisms; the creep strength is enhanced by fine microstructure composed of laths formed though displacive shear transformation from austenite to α' martensite,(2-7) solid-solution strengthening by Mo and W(8) and particle dispersion strengthening du e to fine carbonitrides.(9) The service temperature of α' ferritic heat-resistant steel can be increased up to 923 K by these strengthening mechanisms. When the service temperature is required to be increased above 923 K, it is necessary to use austenitic heat-resistant steel with fcc crystal structure possessing a lower atomic diffusivity than that of bcc. However, stable austenitic stainless steel never undergoes phase transformation, and thus, it is impossible to apply fine lath structure to this kind of steels for improving mechanical properties.

On the other hand, authors reported that a cold-worked metastable austenitic steel with a controlled chemical composition with an optimum balance between Cr and Ni undergoes displacive shear transformation from deformation-induced α' martensite to austenite (martensitic reversion) during rapid heating. The austenitic steel with this thermomechanical treatment exhibits fine lath structure containing high-density dislocations, which is similar to α' lath martensitic structure formed by conventional austenitizing followed by quenching. Authors called that structure "lath austenite".(10)

In this study, the alloy-designed metastable austenitic stainless steel was subjected to thermomechanical treatment to obtain lath austenitic structure, and then the creep property of the steel obtained was evaluated in comparison with that of an austenitic steel with equiax-grained structure. In addition, a stable austenitic steel containing high-density dislocations introduced by cold rolling was also prepared to clarify the difference between lath and deformed austenitic structures in thermal stability and creep deformation behavior.

## 2 EXPERIMENTAL PROCEDURE

### 2.1 Alloy Design

Some metallurgical conditions are required for the microstructural control of metastable austenitic stainless steel using phase transformation from deformation-induced α' martensite to austenite (reversion). When the reversion treatment is performed at 873 K for 10 s, the chemical compositions of Ni and Cr must satisfy the following conditions:(11)

(1) More than 90 vol.% of austenite transforms to α' martensite by cold rolling of 90 % reduction in thickness.

$\mathrm{Ni} + 0.35\mathrm{Cr} < 16$ mass%

(2) Deformation-induced α' martensite retained after reversion is less than 10 vol.%.

$\mathrm{Cr} - 1.2\mathrm{Ni} < 4.0$ mass%

(3) Reversed austenite has an Ms temperature below ambient temperature to avoid α’ martensitic transformation on cooling.

Ni + 0.65Cr > 19.7 mass%

These conditions are essential to obtain a single structure of reversed austenite. In addition to them, another condition is required for formation of lath austenitic structure by the martensitic reversion mechanism. Authors reported that martensitic reversion could be occurred when the driving force for reversion reaches to approximately 500 J/mol.(12) Therefore, the additional condition is given as follows:

(4) Gap of chemical free energy between martensite and austenite is over 500 J/mol at 873 $K^{\dagger}$.

Ni + 0.12Cr > 12.6 mass%

The chemical composition satisfying the above four conditions are displayed in Figure 1. From this result, 14%Cr-11%Ni steel was selected as a steel used in this study. In addition, 15%Cr-21%Ni steel with stable austenitic structure was also prepared for comparison. The detail chemical compositions of these specimens are listed in Table 1.

†: Gap of chemical free energy was calculated by thermodynamic calculation software (*Thermo Calc.*).

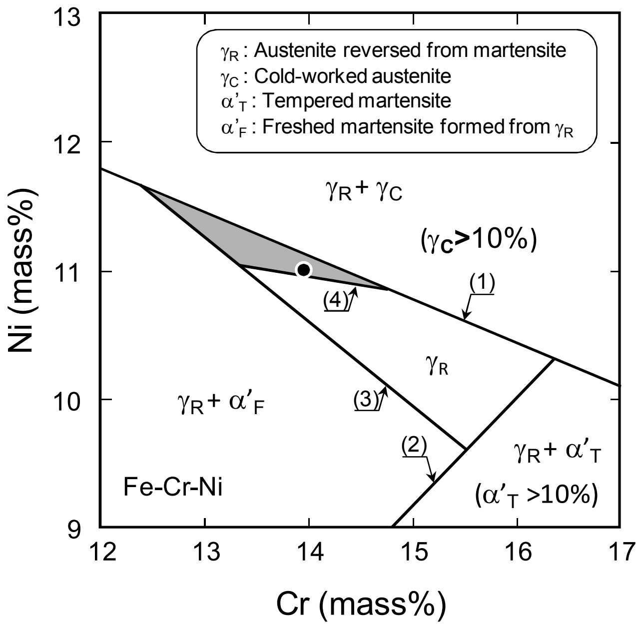

**Figure 1**. Effect of Ni and Cr contents on the structure of specimen annealed at 873K for 0.6ks after 90% cold rolling in Cr-Ni stainless steels.

**Table 1**. Chemical compositions of specimens used in this study

| | Cr | Ni | C | N | Mn | Si | P | S | Fe |
|---|---|---|---|---|---|---|---|---|---|
| Metastable Steel<br>Fe-14%Cr-11%Ni alloy | 14.1 | 11.1 | 0.004 | 0.0064 | 0.27 | 0.02 | 0.005 | 0.010 | bal. |
| Stable Steel<br>Fe-15%Cr-21%Ni alloy | 14.9 | 20.9 | 0.004 | 0.0012 | 0.17 | <0.001 | 0.006 | 0.0019 | bal. |

### 2.2 Thermomechanical Treatment

The ingot of 1.5 kg was produced by an induction melting in vacuum and then cast into a metallic mold of 100mm$^l$ × 50mm$^w$ × 28mm$^t$. The ingot was hot-rolled to a 15 mm thick plate at 1323 K after homogenizing at 1523K for 18ks. Figure 2 shows the thermomechanical treatment route for 14%Cr-11%Ni steel. The specimen cut from the hot-rolled plate was subjected to solution treatment (solution-treated material), and then cold-rolled up to 90% reduction in thickness to cause deformation-induced α' martensitic transformation. In order to obtain the lath austenitic structure, the cold-rolled specimen was heated to 673~973 K at heating rate of 300 K/s and held for 10 s at the temperature by using a salt bath, followed by water-cooling (lath austenite material). Microstructure was observed with an optical microscope (OM) and a transmission microscope (TEM). Measurement of volume fraction of austenite was performed by saturation-magnetization measurement.(13) Creep test was also attempted with a Lever-type creep testing machine for plate test pieces with the parallel part of 3 × 3 × 10mm. In the creep test, test piece was heated to 973 K at heating rate of 20 K/min and then soon tested at the initial stress of 114 MPa.

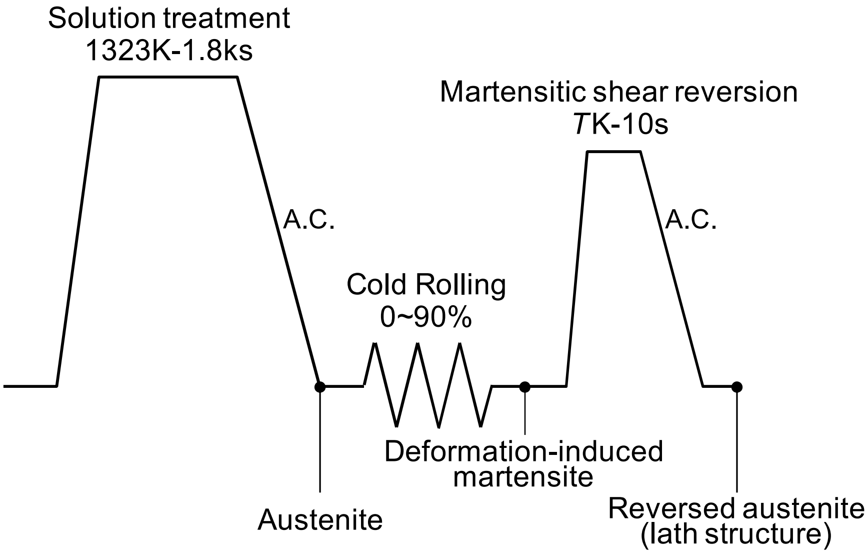

**Figure 2**. Thermomecanical process for obtaining martensitic shear reversed austenite in 14%Cr-11%Ni steel.

## 3 RESULTS AND DISCUSSION

### 3.1 Lath Austenitic Structure Formed by Martensitic Reversion from Deformation-induced Martensite

Figure 3 shows change in volume fraction of α' martensite formed by cold rolling as a function of equivalent strain in 14%Cr-11%Ni steel. Volume fraction of α' martensite is continuously increased with increasing equivalent strain and reaches more than 95 % by cold rolling at an equivalent strain of 1.0 (approximately 60 % reduction in thickness). However, it is known that the reversion behavior of deformation-induced α' martensite is changed depending on the cold rolling even when the reduction is more than the critical value of 60 %, because the lath structure in deformation-induced α' martensite is broken by further deformation after martensitic transformation.(14) Figure 4 shows change in volume fraction of α' martensite by heating after cold rolling as a function of the heating temperature in 14%Cr-11%Ni steel. The reversion from deformation-induced α' martensite to austenite started at 773 K and completely finished at 873 K regardless of the reduction of cold rolling, which suggests that further cold working after completion of deformation-induced α' transformation has no effect on the velocity of reversion. It should be noted here that the reversion has taken place in a narrow temperature range between 823 and 873 K. This is a typical feature of displacive transformation.(10) It is well understood that the reversion mechanism whether displacive or diffusive, depends on not only chemical composition but also heating rate. In order to confirm the effect of heating rate on the reversion mechanism, the relation between reversion temperature and heating rate in 14%Cr-11%Ni steel cold-rolled at 70% is shown in Figure 5. Both of start and finish temperatures for reversion are unchanged over a wide range of heating rate between 2 and 100 K/s, which means that diffusive reversion hardly occur in this material. Figure 6 represents TEM images showing deformation-induced α' martensitic structure formed by cold rolling at 70% (a) and lath austenitic structure formed by martensitic reversion at 923 K for 10 s (b) in 14%Cr-11%Ni steel. Although the reduction of 70 % is higher than the critical reduction for completing deformation-induced α' martensitic transformation (60 %), it is confirmed that a small amount of lath structure remains in the 70 % cold-rolled specimen (a). If the reduction in thickness is increased over 70 %, deformation-induced α' martensitic structure becomes heavily-deformed structure with equiaxed dislocation cells, resulting in the formation of an ultrafine grained austenitic structure instead of lath austenitic structure after reversion.(14) Therefore, 70 % was selected as an optimum cold rolling reduction so as not to break lath α' structure in the following experiments. The austenitic structure formed by reversion from this deformation structure (b) exhibits fine lath structure similar to lath α' martensitic structure, and it is also found that high-density dislocations are uniformly distributed in the laths.

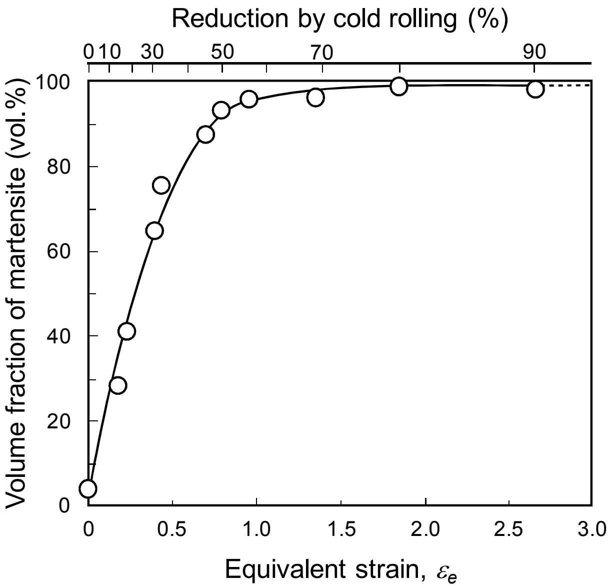

**Figure 3**. Thermomecanical process for obtaining martensitic shear reversed austenite in 14%Cr-11%Ni steel.

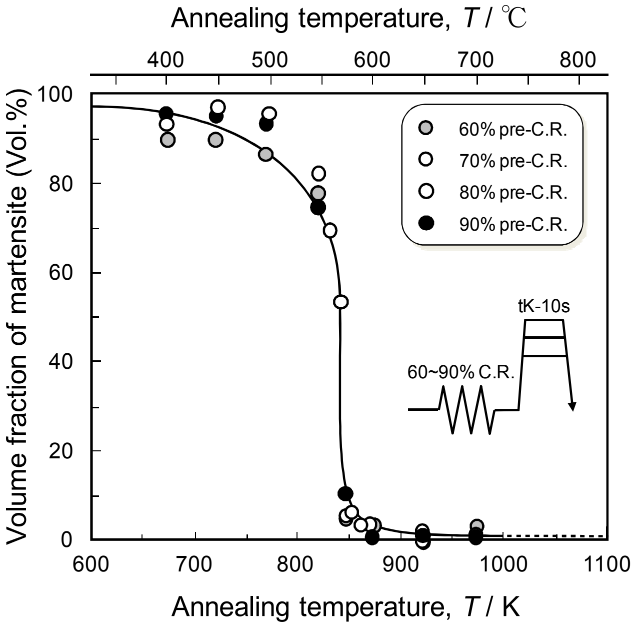

**Figure 4**. Change in volume fraction of deformation-induced martensite as a function of annealing temperature in cold-rolled 14%Cr-11%Ni steel.

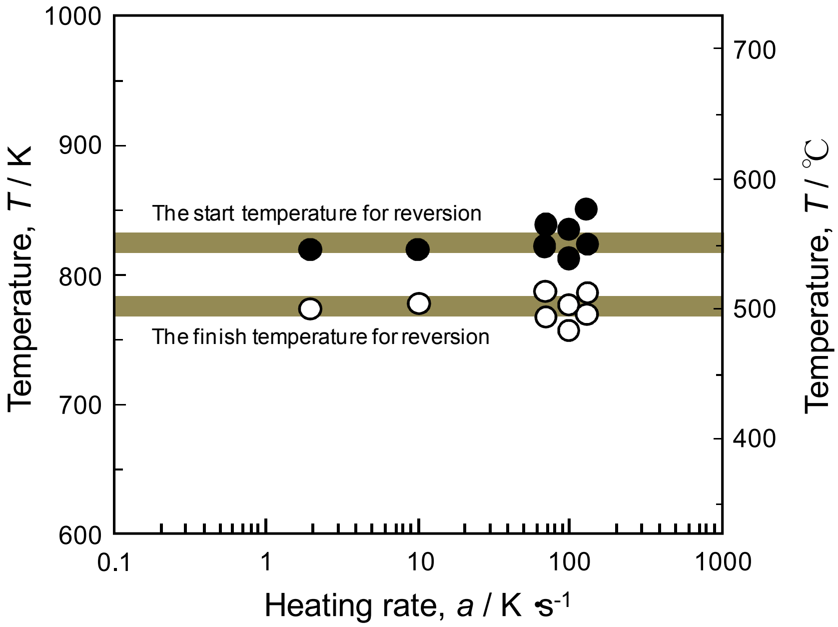

**Figure 5**. Effect of heating rate on reversion temperature in 14%Cr-11%Ni steel cold-rolled by 70% in thickness.

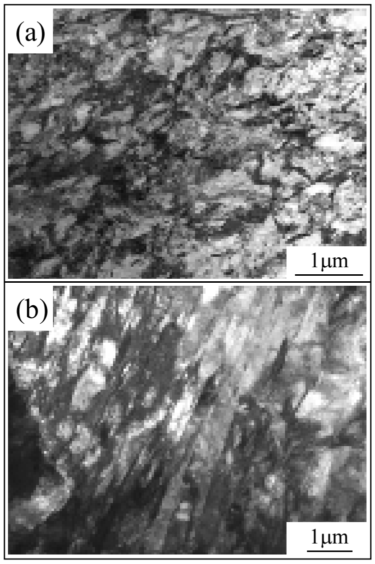

**Figure 6**. TEM images of 14%Cr-11%Ni steel before (a) and after (b) annealing at 923K for 10s.

### 3.2 Creep Property of Austenitic Steel with Lath Structure

In addition to the 14%Cr-11%Ni steel with lath austenitic structure, a solution-treated material with the same chemical composition and a stable 15%Cr-21%Ni austenitic steel cold-rolled at 70 % (deformed austenite material) were prepared to evaluate the effect of lath austenitic structure on the creep property. OM and TEM images for each material are represented in Figure 7. Solution-treated material exhibits a typical equiax-grained austenitic structure with average grain size of approximately 60 μm, while deformed austenite material has highly dislocated substructure constructed of dislocation cells and micro shear bands.

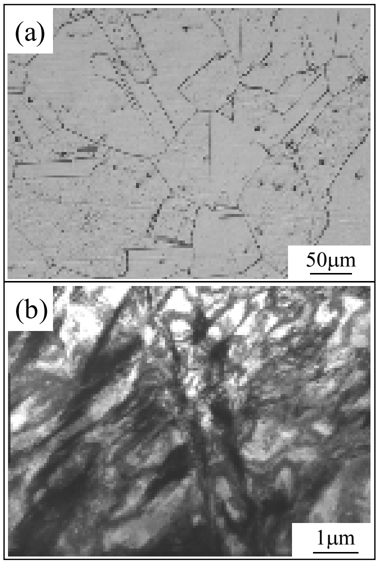

**Figure 7**. Microstructure of solution-treated 14%Cr-11%Ni steel (a) and 15%Cr-20%Ni steel cold-rolled by 70% (b).

Figure 8 shows the results of creep tests in the lath austenite, solution-treated and deformed austenite materials. Comparing the creep deformation behavior of the lath austenite material with that of the solution-treated material reveals that the lath austenite material has approximately two times longer creep fracture lifetime than the deformed austenite material. This result clearly indicates that lath structure with a high dislocation density works as an effective microstructural factor increasing the creep strength of austenitic stainless steel. However, the creep fracture lifetime of deformed austenite material was just 6 ks in spite of the high dislocation density, indicating the dislocations effective for strengthening is not introduced by cold rolling but by transformation. The difference in creep deformation behavior between lath austenite material and deformed austenite material should be reflected in the microstructural change during creep testing. Figure 9 represents TEM images of creep tested materials in which the testing was interrupted at different testing time. It is found that the lath austenite material maintains the fine lath structure for a long time up to 10.8 ks ($\varepsilon$=0.25). Straight lath boundaries are certainly observed in the Fig.9 (b). After the creep deformation for 21.6 ks ($\varepsilon$=0.41) (c), the lath structure seems to have recrystallized, leading to the rapid increase in strain rate of creep deformation. On the other hand, the deformed austenite material exhibits equiax-grained structure containing dislocations from the early stage of creep deformation (d). Such a dislocation substructure is known to be formed through dynamic recrystallization during creep deformation.

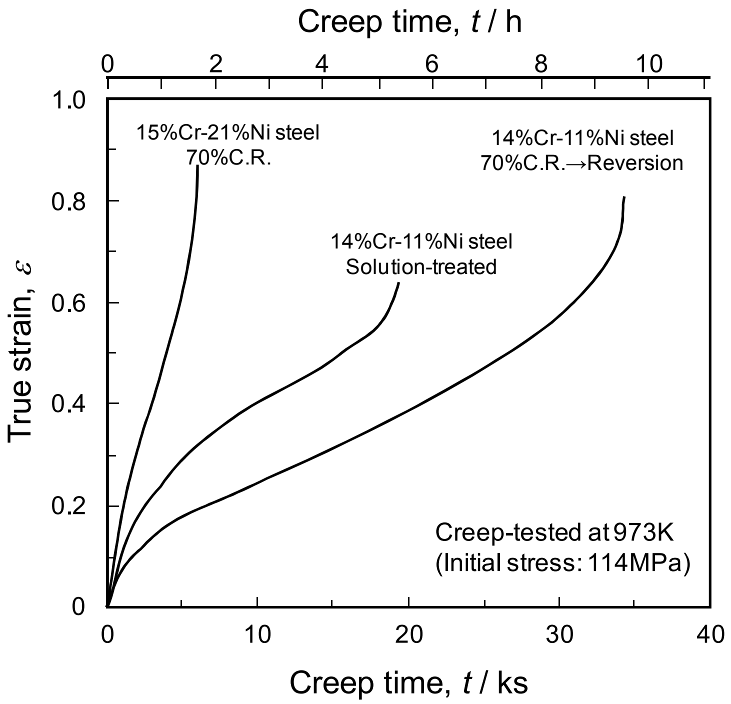

**Figure 8**. Creep curves of 14%Cr-11%Ni and 15%Cr-21%Ni austenitic steels.

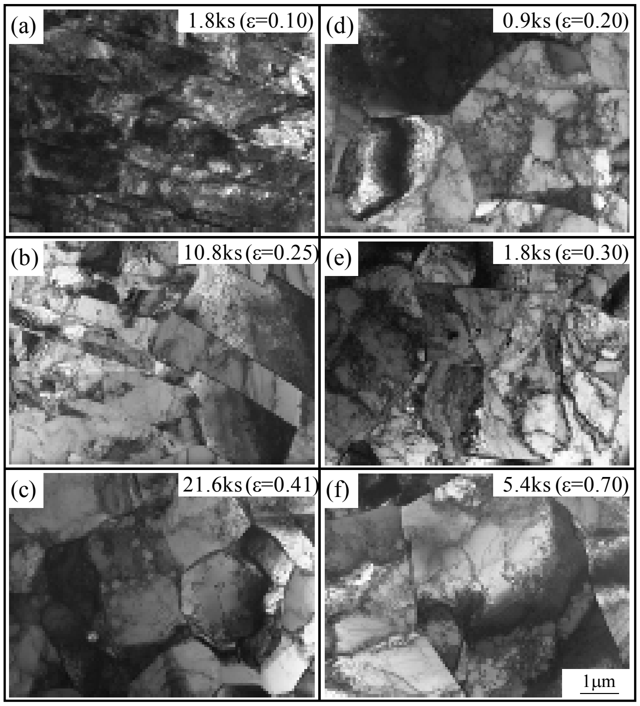

**Figure 9**. TEM images showing microstructural changes in14%Cr-11%Ni and 15%Cr-21%Ni steels. (a)~(c) : 14%Cr-11%Ni steel reversed at 923K for 10s after cold rolling by 70% in thickness (d)~(f) : 15%Cr-21%Ni steel cold-rolled by 70%.

From these results, it was concluded that lath austenitic structure formed by martensitic reversion is more thermally stable than deformed austenitic structure, and thus, the high-density dislocations can contribute to strengthening for a long time without causing recrystallization.

## 4 CONCLUSIONS

1. Fine lath austenitic structure was successfully obtained through displacive reversion of deformation-induced martensite in cold-rolled 14%Cr-11%Ni metastable austenitic stainless steel.
2. Thermally stable dislocation structure formed through displacive transformation is effective for improving creep property, while dislocation cell structure formed through cold-working is rather disadvantageous due to the enhancement of dynamic recrystallization during creep deformation.

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