

Effect of pre-strain and reheating on isothermal α' martensite formation in Fe–25.5Ni–4Cr austenitic steel

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Abstract

The effect of pre-strain on α' martensitic transformation was examined for Fe–25.5Ni–4Cr austenitic steel. α' martensitic transformation was gradually suppressed with pre-strain up to about 10%, but was contrarily enhanced with pre-strain over about 10%. Specimens pre-strained to 10% and 30% were reheated to clarify the mechanism of suppression and enhancement on α' martensite formation. The diffusion activation energy of component atoms in this steel was obtained from the relation between the amount of α' martensite and the reheating time. In specimens pre-strained to 10%, the apparent activation energy is fairly close to the diffusion activation energy of C in austenitic steels. In specimens pre-strained to 30%, the apparent activation energy is close to the diffusion activation energy of Ni and Cr in austenitic steels. The suppression and enhancement of α' martensitic transformation is attributed to diffusion of C, Ni and Cr in Fe–25.5Ni–4Cr.

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1. Introduction

Due to the superior non-magnetic property, austenitic stainless steels are expected to be used as various component materials of a fusion reactor system. However, the formation of α' martensite occurs isothermally in these steels at low temperature. The formation of α' martensite causes mechanical property degradation as well as non-magnetic property degradation. Therefore, it turns out to be very important to investigate the mechanism of isothermal α' martensite formation in detail. Shibata et al. [1–4] have investigated the effects of solution heat treatment temperature, cooling rate from this temperature, and pre-strain on the amount of α' martensite in 304L steel. The formation of α' martensite was enhanced by the solution treatment temperature

and as the subsequent cooling rate increased. As for the effect of pre-strain, the formation of α' martensite was enhanced with very small pre-strain and was suppressed with pre-strain over about 5% [1–4]. This is considered that stacking faults or ϵ martensite play an important role in the formation of α' martensite in 304L steel. 304L steel forms α' martensite mainly through ϵ martensite. Since stacking fault and martensite are similar to the crystal structure locally, it is appeared that stacking faults work as nucleation sites for α' martensite. However, the details of such a role have not been clarified. Therefore, we decided to investigate whether or not similar effects are observed in Fe–25.5Ni–4Cr steel whose the stacking faults energy is higher than that of 304L steel. In addition, this steel is thought to form isothermally α' martensite directly from the austenitic phase, whereas 304L steel forms α' martensite mainly through ϵ martensite. Shibata et al. [3,4] have also investigated the effects of solution heat treatment temperature and the subsequent cooling rate on the amount of α' martensite in Fe–25.5Ni–4Cr steel. It has turned out that the effect of solution heat treatment temperature and the subsequent cooling rate in Fe–25.5Ni–4Cr steel have little affect the formation of α' martensite. In

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this paper, the effect of pre-strain and reheating on isothermal α' martensitic transformation was examined using Fe–25.5Ni–4Cr steel.

2. Experimental procedure

2.1. Effect of pre-strain

The chemical composition of the steel used is shown in Table 1. Round bar blanks cut from the hot-rolled plates were machined to tensile test specimens. The dimensions of the gage region of the tensile test specimen are 60 mm in length and 8 mm in diameter. The tensile test specimens were sealed in silica tubes that were evacuated to 10^{-3} Pa, before filling with small amount of high-purity Ar gas. The specimens were heated to 1573 K for 18 ks and water-quenched. Pre-strain was introduced using tensile testing machine at room temperature. Small rectangular blocks of $2.5 \times 2.5 \times 4$ mm³ size were cut from the tensile test specimens. These blocks were held in liquid nitrogen, and then an external magnetic field was applied. The application of external magnetic field was carried out using a hybrid magnet and superconducting magnet at the National Institute for Material Science (NIMS). While holding in liquid nitrogen for 9.24 ks, an external magnetic field was applied to specimen for 3.6 ks. The saturation magnetization of each block was measured using a Vibrating Sample Magnetometer (VSM) at room temperature after electrolytic removal of each block. The volume fraction of α' martensite V_f (%) was calculated from Eq. (1):

$$V_f (\%) = \frac{4\pi I'_s}{10^4 I_s} \times 100, \quad (1)$$

where I'_s is the saturation magnetization of the specimen obtained with a VSM and I_s is saturation magnetization of the specimen when it transforms entirely to α' martensite. I_s was calculated using Eq. (2) obtained from the Slater–Pauling curve [5],

$$I_s(T) = \frac{\{2.2 \times (1 - x - y) + 0.6x\}7.0434}{a}, \quad (2)$$

where x and y are the molar fraction of Ni and Cr respectively, and a is the molar volume of material.

2.2. Effect of reheating

The tensile test specimens sealed in silica tube were solution treated at 1573 K for 18 ks and water-quenched. Pre-strain was introduced using tensile testing machine at room temperature. Small rectangular blocks of $2.5 \times 2.5 \times 4$ mm³ size were cut from the tensile specimens. These blocks were reheated at temperatures up to 828 K for various times and water-quenched. The external magnetic field was applied at 7.5 T. The application of external magnetic field and the calculation of the amount of α' martensite were performed by the same methods as for the effect of pre-strain.

3. Results

3.1. Effect of pre-strain

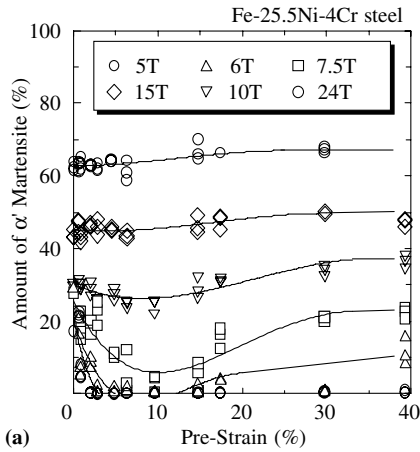
Fig. 1(a) and (b) show the relationship between pre-strain and the amount of α' martensite in Fe–25.5Ni–4Cr steel and 304L steel, respectively. In the case of Fe–25.5Ni–4Cr steel, the formation of α' martensite in zero magnet field is not formed in all pre-strained specimens. The formation of α' martensite is suppressed with pre-strain up to about 10%. However the formation of α' martensite is enhanced again with pre-strain over about 10%. Such change of the amount of α' martensite with pre-strain is reduced as the magnetic field is increased. In the case of 304L steel, pre-strain suppresses the formation of α' martensite, although very small pre-strain increase the amount of α' martensite [3]. These are completely different results. Specimens pre-strained to about 10% and about 30% in Fe–25.5Ni–4Cr steel were reheated to clarify the behavior of α' martensite formation by estimation of the diffusion activation energy of component atoms.

3.2. Effect of reheating

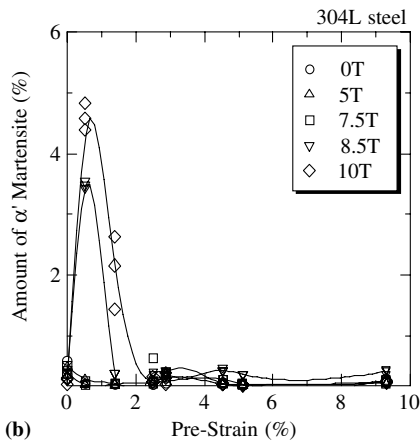
Figs. 2 and 3 show the relationship between reheating time and the amount of α' martensite formation at pre-strained to about 10% and about 30% in a magnetic field of 7.5 T. The formation of α' martensite in specimens pre-strained to about 10% is enhanced with increasing reheating time. However, the formation of α' martensite in specimens pre-strained to about 30% is suppressed with increasing reheating time. On the basis of these

Table 1
Chemical composition of the steel used (mass%)

	Ni	Cr	C	N	Mn	Si	Mo	Fe
Fe–25.5Ni–4Cr	25.44	3.89	0.0035	0.005	–	–	0.18	bal.
SU S304L	9.67	18.36	0.02	0.049	0.82	0.46	–	bal.



(a)



(b)

Fig. 1. Correlation between pre-strain and the amount of α' martensite in (a) Fe-25.5Ni-4Cr steel and (b) 304L steel.

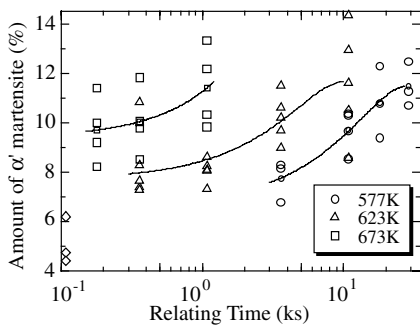


Fig. 2. Correlation between reheating temperature and amount of α' martensite at pre-strain about 10% in magnetic field 7.5 T.

results, the diffusion activation energy of component atoms in this steel is estimated from the slope of the Arrhenius plots, which are shown Figs. 4 and 5. In specimens pre-strained to about 10%, the apparent

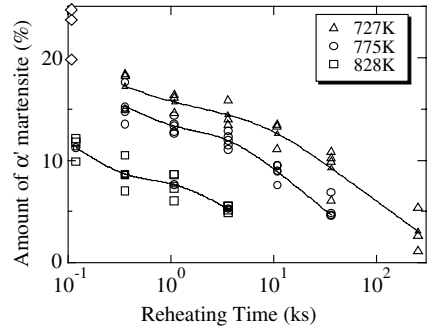


Fig. 3. Correlation between reheating temperature and amount of α' martensite at pre-strain about 30% in magnetic field 7.5 T.

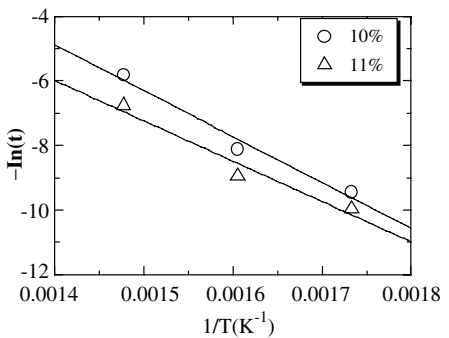


Fig. 4. Arrhenius plot resulted from 10.0%, 11.0% α' martensite using 9.8% strained sample.

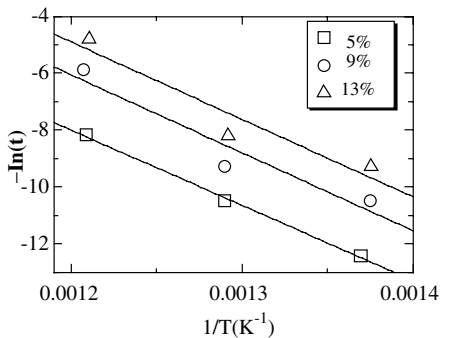


Fig. 5. Arrhenius plot resulted from 5.0%, 9.0%, and 13.0% α' martensite using 29.7% strained samples.

activation energy is estimated to be about 120 kJ/mol. This is fairly close to the diffusion activation energy of C in austenitic steels [6]. In specimen pre-strained to about 30%, the apparent activation energy was estimated to be about 240 kJ/mol. This is close to the diffusion activation energy of Ni and Cr in austenitic steels [6].

4. Discussion

In the case of zero pre-strain, it appears that existing lattice defects, such as grain boundaries, work as nucleation sites. It has already been shown that the effects of solution heat treatment temperature and the subsequent cooling rate have little effect on the formation of α' martensite. Therefore it appears that an existing lattice defect which do not depend on the solution heat treatment and cooling rate must provide the nucleation sites.

The formation of α' martensite is initially suppressed with pre-strain up to about 10%. The results of the reheating experiment indicate that the apparent activation energy is fairly close to the diffusion activation energy of C in austenitic steels. This indicates that the suppression of the transformation is attributed to C. C is an austenite stabilization and causes dislocation stabilization. So, it is considered that the formation of α' martensite is enhanced by C diffusion out of the martensite nucleation site during reheating. Moreover, it has already been shown that the effect of reheating without introducing pre-strain does not affect the formation of α' martensite. This indicates that a lattice defect which is caused by pre-strain contributes to suppression of α' martensitic transformation. Therefore, in specimens pre-strained to about 10%, it appears that the activity of nucleation sites was reduced by dislocation stabilization and lattice defects produced during plastic strain.

The formation of α' martensite is enhanced with pre-strain over about 10%. The results of the reheating experiment indicate that the apparent activation energy for this process is close to the diffusion activation energy of Ni and Cr in austenitic steels. This indicates that the enhancement of the transformation is attributed to a substitutional atom, such as Ni and Cr. It appears that

the residual stress affects this mechanism of enhanced formation of α' martensite. The residual stress is active to in the enhancement of α' martensite formation locally. It is considered that formation of α' martensite is suppressed with stress relief by Ni and Cr diffusion to martensite nucleation sites during reheating.

In addition, clear differences are observed in the effect of pre-strain on the formation of α' martensite between the Fe–25.5Ni–4Cr steel and the 304L steel. Such differences are attributed to definite differences in the nature of α' martensite nucleation sites and it is suggested that stacking fault energy has a large effect in determining the nature of the nucleation sites.

5. Conclusion

The causes of suppression and enhancement of α' martensitic transformation are attributed to diffusion of C, Ni and Cr in Fe–25.5Ni–4Cr steel.

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