

Effect of Thermo-mechanical Treatment on Reverse Transformation Behaviour and Micro-duplex Structure Formation in Fe-Ni alloys

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Introduction

In the reverse transformation of martensite (α') to austenite (γ), a diffusion controlled process has been observed as well as a martensitic reversion, depending upon heating rate and temperature (1-5). The diffusional reversion is of interest as a method of ultra-refining of the microstructure (6,7). However, some inconsistent results were revealed in previous work on diffusional reversion behaviour with regard to temperature dependence and the sequence of transformations (1-4). Although the effects of prior deformation on the reversion behaviour have been studied (8,9), less attention has been focussed so far on the effect of ausforming.

In this study thermo-mechanical treatments (TMTs), including ausforming and marforming (deformation of martensite), were employed to determine their effects on the reverse transformation behaviour in lath and twinned α' and to obtain an ($\alpha'+\gamma$) micro-duplex structure.

Experimental Procedure

Fe-19.7wt%Ni(0.003C-0.076Mn-0.0035P-0.0007S-0.0010N) and Fe-31.1wt%Ni(0.003C-0.070Mn-0.0031P-0.0006S-0.0008N) alloys were employed in this study. These alloys were melted in a vacuum furnace and cast into 50kg ingots. 50x50x10mm samples were prepared after hot rolling. Three types of treatments, (i) conventional quenching (ii) ausforming and (iii) marforming, were performed as shown in Fig.1. Subsequently, samples were reheated at temperatures between 300 and 650°C for times of 360 to 3.6x10⁶s to study the reverse transformations. The average rate of both reheating and cooling was about 10°C/s.

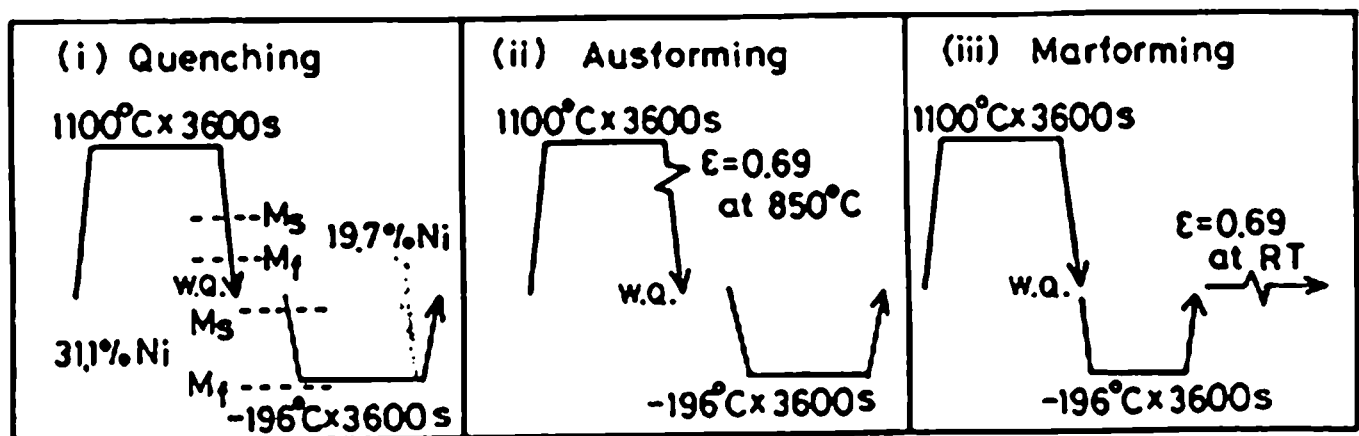


Fig.1: Thermo-mechanical treatments to produce martensitic structures.

After these treatments the volume fractions of austenite were measured using an X-ray diffractometer. Before the measurements, a layer of about 100 μ m thickness was removed by electro-polishing to avoid deformation induced martensite possibly formed during mechanical polishing. Optical and transmission electron microscope observations were carried out on electro-etched samples or jet

electro-polished thin foils. In both cases a solution of 5 or 10% perchloric acid and acetic acid was used.

Results and Discussion

Austenite to Martensite Forward Transformation - Dislocated 1st martensite without detectable retained austenite was produced in Fe-19.7%Ni for every thermomechanical treatment. Ausforming gave an elongated microstructure due to the direct quench from deformed austenite and resulted in an increase in hardness ($\Delta H_v=16$). This could arise from the dislocation structure inherited from the deformed austenite. Marforming also caused an elongated microstructure and increased hardness ($\Delta H_v=38$). In Fe-31.1%Ni the sub-zero treatment into liquid nitrogen produced twinned lenticular martensite with 12.1% retained austenite. Ausforming increased the amount of austenite to 14.7%, showing it had an austenite stabilizing effect. Despite this, hardness still increased ($\Delta H_v=22$). Marforming decreased the amount of retained austenite to 5.1%, as a result of deformation induced transformation, and increased hardness ($\Delta H_v=30$).

Martensite to Austenite Reverse Transformation - The changes in austenite volume fraction (V_γ) after the reverse transformation treatments are shown as a function of holding temperature in Fig.2. In Fe-31.1%Ni, V_γ monotonically increases with increasing temperature. In Fe-19.7%Ni, however, V_γ increases

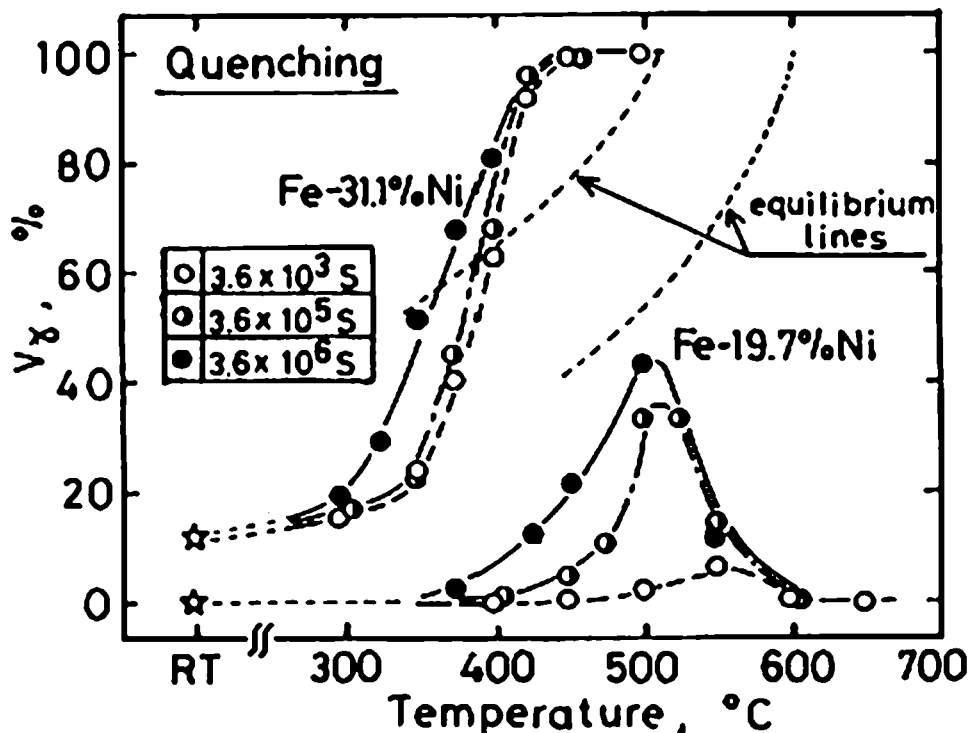


Fig.2: Change in austenite volume fraction with reheating treatment for different times.

to a maximum with increasing temperature to about 500-525°C. Austenite formed above 525°C, contains at the most 29wt%Ni even in the equilibrium condition, and so transforms to α' on cooling after treatment. Broken lines in the figure give the equilibrium volume fraction derived from the equilibrium diagram for Fe-Ni. It is of interest that more austenite than the equilibrium amount is apparently formed at 350<T<450°C in Fe-31.1%Ni. Figure 2 also indicates that the reverse transformation is time dependent because V_γ increases with increasing holding time.

Figure 3 shows the relationship between the amount of austenite and time in Fe-19.7%Ni. The results represent typical behaviour of a diffusion controlled process in which after long treatment, V_{γ} should approach the equilibrium values, i.e. 42% at 450°C and 53% at 500°C. Another point shown in the figure is an effect of TMTs on the reverse transformation behaviour. Both ausforming and marforming are effective in promoting the diffusion controlled reverse transformation of lath martensite.

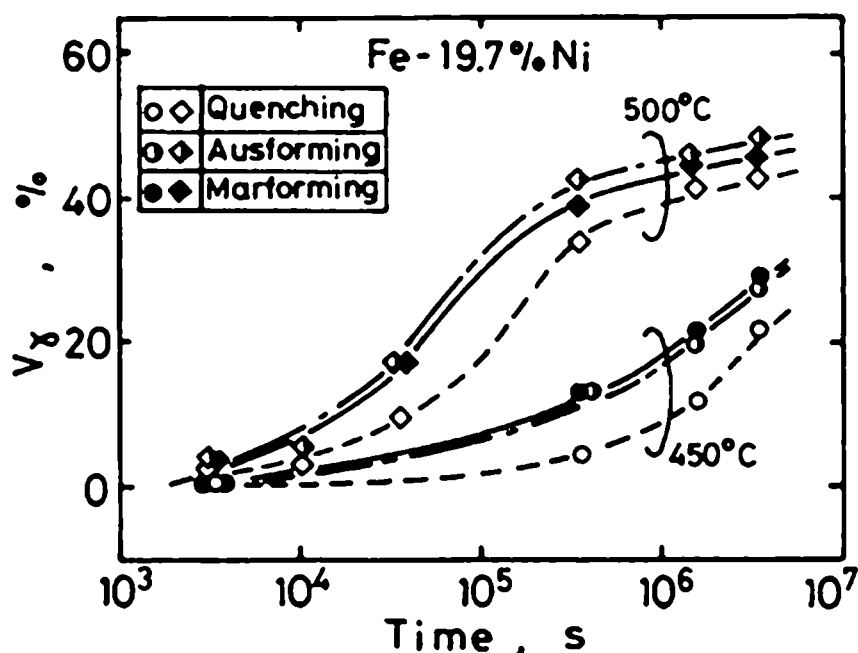


Fig.3: Change in austenite volume fraction with reheating time in Fe-19.7%Ni.

Figure 4 shows the equivalent results obtained for Fe-31.1%Ni. For clarity, the results of marforming are shown separately. In every condition drastic increases in V_{γ} occur immediately during the reverse transformation treatment at higher temperatures. This athermal behaviour shows that the transformation occurs as a diffusionless process. As temperature increases, the amount of the diffusionless reverse transformation increases. In most cases, diffusion controlled transformation occurs subsequent to the diffusionless transformation. After the diffusion controlled reversion, it seems that V_{γ} approaches a certain limit at each temperature. As shown in Fig.2, the limiting values are far beyond the true equilibrium amount. Pseudo-equilibrium is therefore achieved. V_{γ} approaches a pseudo-equilibrium in which the austenite produced on a relatively massive scale by the diffusionless reverse martensite transformation remains virtually unchanged in composition during the subsequent period of treatment, whereas the very fine austenite precipitation produced by diffusional transformation is of the equilibrium composition expected for the treatment temperature. This assumption gives

$$V_{\gamma}(\text{pseudo-equilibrium}) = V_{\gamma 0}(\text{retained austenite}) + V_{\gamma}(\text{d-less}) + V_{\gamma e}(\text{d-na}) \quad \text{Equ.1}$$

and from a mass balance for nickel

$$V_{\gamma e}(\text{d-na}) = \{100 - V_{\gamma 0} - V_{\gamma}(\text{d-less})\} (31.1 - \text{Ni}[\alpha]) / (\text{Ni}[\gamma] - \text{Ni}[\alpha]) \quad \text{Equ.2}$$

where $Ni[\gamma]$ and $Ni[\alpha]$ are the equilibrium nickel contents of austenite and ferrite.

Calculated values are shown in Fig.4, which indicates good agreement with the experimental results.

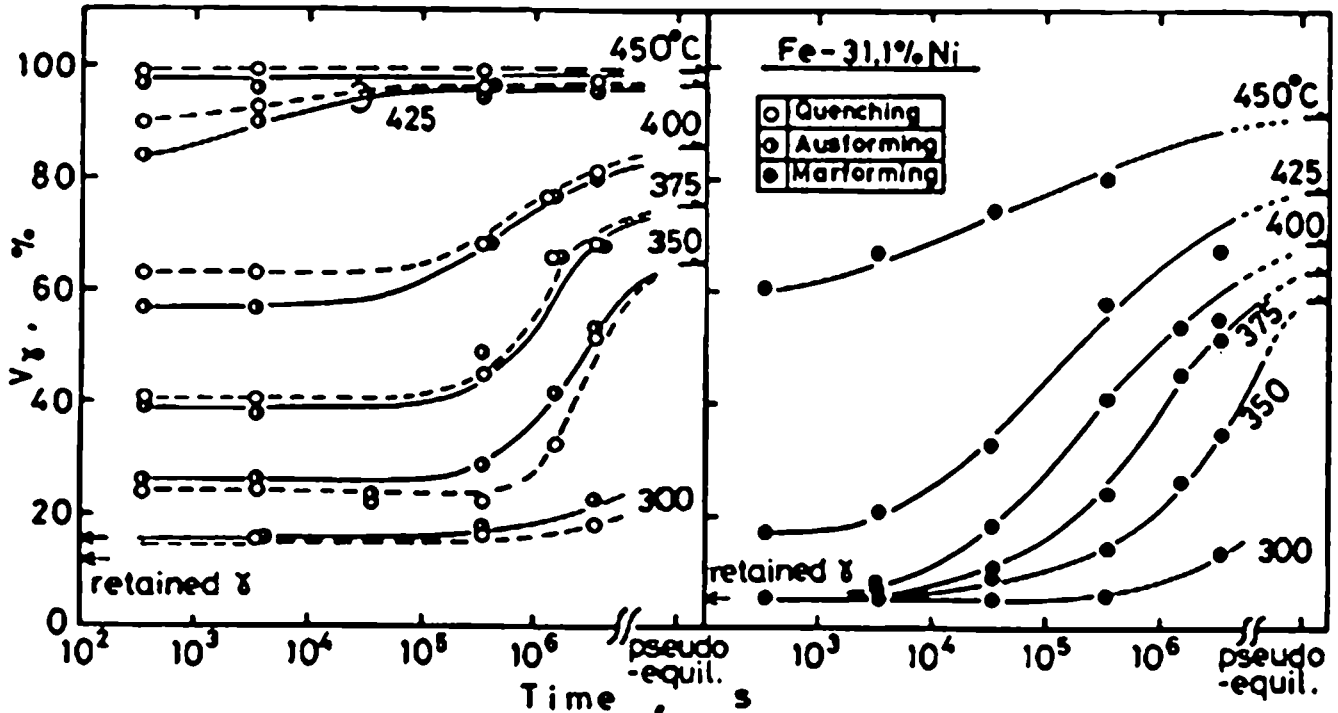


Fig.4: Change in austenite volume fraction with reheating time in Fe-31.1%Ni.

Change in the amount of diffusional and diffusionless reverse transformation in Fe-31.1%Ni, which can be derived from Fig.4, is shown as a function of temperature in Fig.5. Diffusional transformation occurs at lower temperatures, and diffusionless transformation takes place predominantly at higher temperatures. It can be seen in Fig.5 that marforming has a pronounced effect both on suppression of the diffusionless transformation and on promotion of the diffusional transformation. Ausforming has similar effects, but to a much smaller extent.

Suppression of the diffusionless transformation by marforming has been demonstrated in previous work (8,9). It was stated that cold working of martensite was effective in retarding the reverse shear transformation because of an increase in the yield strength of martensite, which resists shear deformation. An increase in hardness ($\Delta Hv=30$) due to marforming was observed in this study and therefore this explanation is accepted in general. However, the other result, that ausforming has little effect on suppression of the diffusionless transformation despite its substantial strengthening effect ($\Delta Hv=22$), suggests that not only a change in strength but also a change in microstructure such as the formation of deformation twins, also has an important role in determining the kinetics.

Micro-duplex Structure Formation - The diffusionless reverse transformation resulted in a coarse microstructure due to the massive transformation, whilst

the diffusional transformation brought about a fine micro-duplex structure.

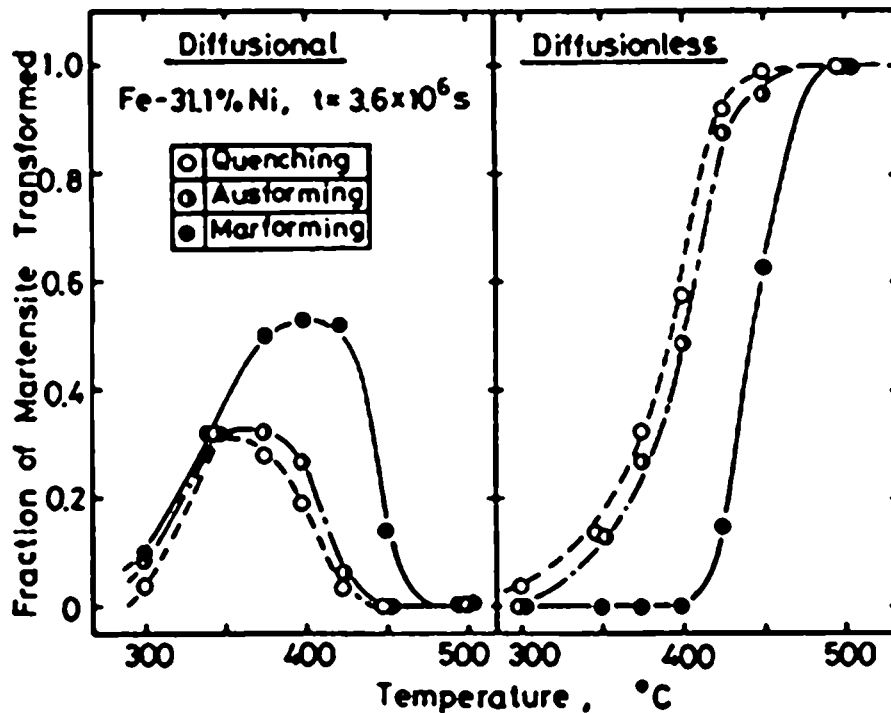


Fig.5: Diffusional and diffusionless reverse transformation behaviour after reheating treatment for 3.6×10^6 s in Fe-31.1%Ni.

Figure 6 shows the microstructures of Fe-19.7%Ni reheated at 450°C for 3.6×10^5 s. Dark and bright areas represent martensite and austenite respectively. In conventional quenching shown in Fig.6(a), relatively large austenite grains with $5\text{--}10\mu\text{m}$ grain size were observed. It is noteworthy that α' grain boundaries were not intersected by the growing austenite even after diffusional reverse transformation. Growth of nucleated γ was likely to be parallel to the lath direction. A fine ($\alpha'+\gamma$) micro-duplex structure of about $1\mu\text{m}$ in scale, was obtained in the ausformed condition, as shown in Fig.6(b). It was shown in previous work (6) that marforming has a beneficial effect on grain refinement. The results of this work show that ausforming has a similar effect on grain refinement as marforming.

TEM observation for conventionally quenched samples indicated that γ grains, which nucleated mainly on α' grain boundaries, grew in the lath direction and easily coalesced with neighbouring grains, which nucleated on different laths, as shown in Fig.7(a). In contrast, in the ausformed condition polygonal γ grains with $0.1\text{--}1\mu\text{m}$ grain size were observed inside a lath as shown in Fig.7(b). Nucleated grains have different orientations and are therefore hardly coalesced. The effect of ausforming on grain refining is mainly explained by the promotion of γ nucleation with different orientation inside laths as well as at lath boundaries.

In Fe-31.1%Ni optical microscopic observations could not show detailed features of the microstructure for the diffusional transformation because of the extremely fine duplex structure.

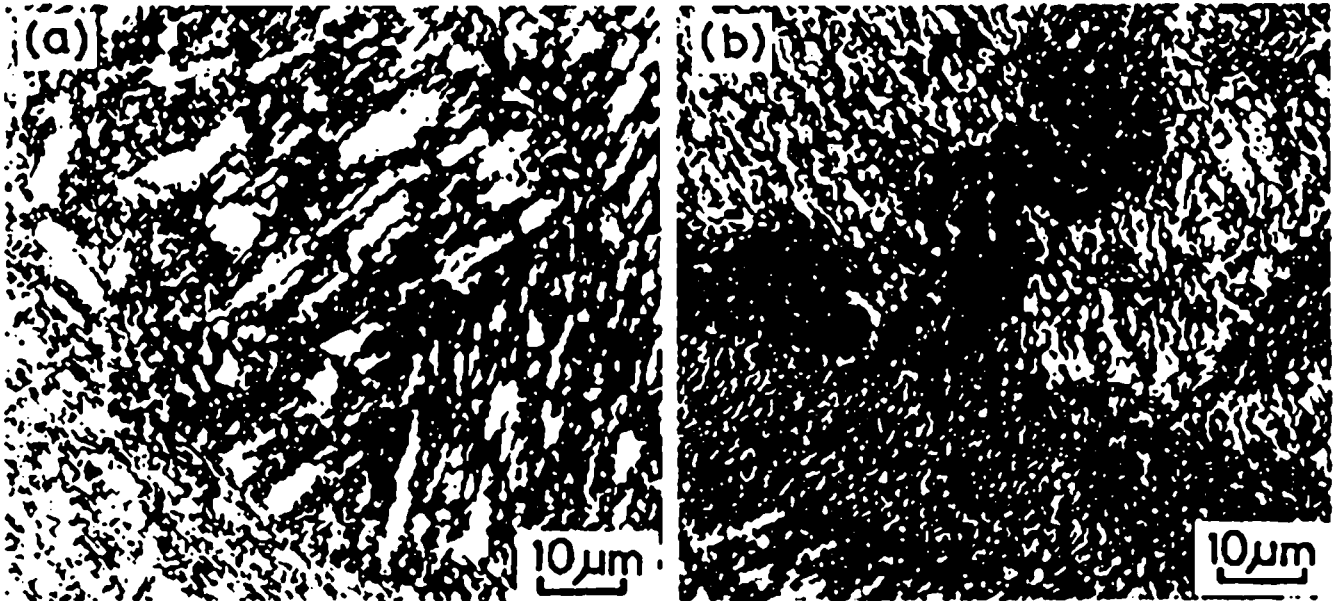


Fig.6: Fe-19.7%Ni cooled to -196°C and reheated at 450°C for 3.6×10^6 s, showing $(\alpha'+\gamma)$ duplex structures after conventional quenching (a) and ausforming (b).

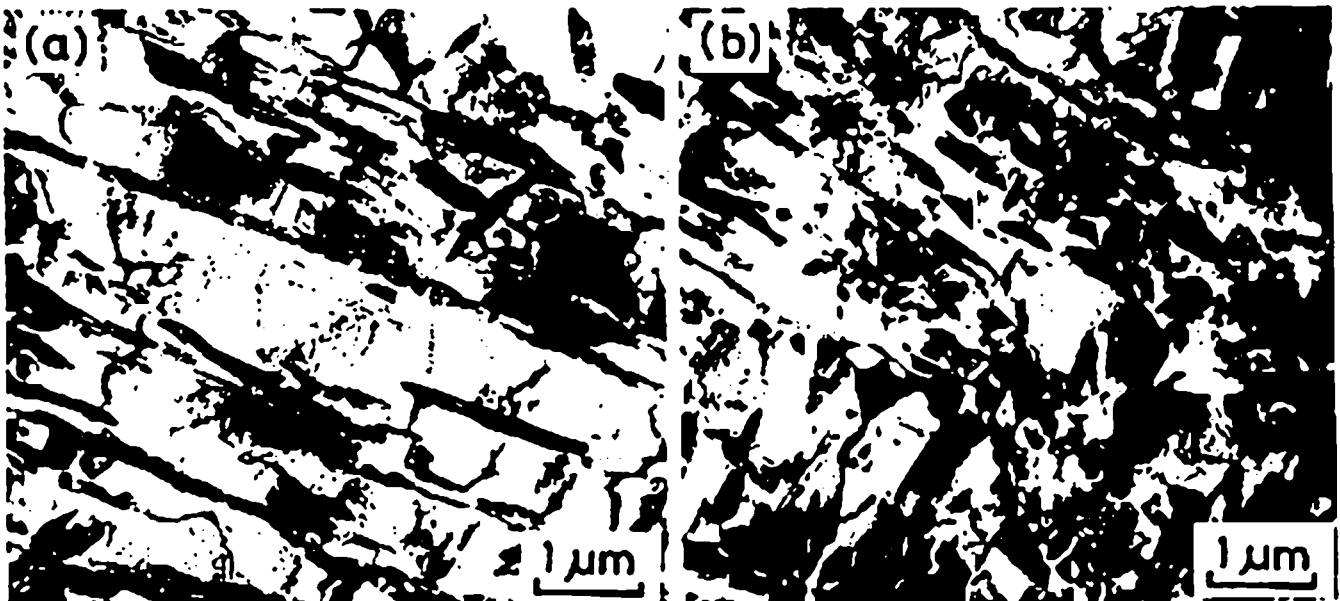


Fig.7: Electron micrographs, showing substructures of Fe-19.7%Ni cooled to -196°C and reheated at 500°C for 3.6×10^5 s, after conventional quenching (a) and ausforming (b).

Fig.8 shows an example of TEM observations for a diffusionally transformed sample. Ultra fine $(\alpha'+\gamma)$ duplex of scale about $0.1\mu\text{m}$, showing a homogeneous plate-like structure, was obtained. It is clear in the picture that there is a preferred orientation between α' and transformed γ . Diffraction analyses indicated that the Bain relationship $(001)_{\gamma} // (001)_{\alpha'}$, and $[010]_{\gamma} // [110]_{\alpha'}$, was at least satisfied.



Fig.8: Electron micrographs, showing the fine plate-like structure in Fe-31.1%Ni ausformed and reheated at 400°C for 3.6×10^5 s.

It can be noted from the above results that ausformed or marformed lath martensite and marformed twinned martensite are the most advantageous starting microstructures to obtain a homogeneous micro-duplex structure through the diffusional reverse transformation.

Conclusions

- 1) Reverse transformation proceeds in three patterns dependent upon temperature: (i) complete or predominant diffusional transformation at lower temperatures, (ii) diffusional transformation subsequent to diffusionless transformation at intermediate temperatures and (iii) complete diffusionless transformation at higher temperatures.
- 2) The amount of transformation depends only on temperature in diffusionless reversion, and depends on both time and temperature in diffusional reversion.
- 3) At the intermediate temperatures, diffusional transformation approaches a pseudo-equilibrium for which the retained austenite and austenite formed by diffusionless transformation retain the bulk composition, and the fine austenite precipitated in martensite attains the equilibrium composition.
- 4) In dislocated lath martensite, ausforming and marforming promote the diffusional transformation and bring about a polygonal micro-duplex structure with 0.1-0.15 μm grains. This is attributed to promotion of nucleation of γ inside laths.
- 5) In twinned lenticular martensite, marforming remarkably suppresses the diffusionless reverse transformation and leads to homogeneous plate-like micro-duplex structure of scale about 0.1 μm after the diffusional transformation.

References

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