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ELECTRON-MICROSCOPE INVESTIGATION OF  $\alpha \rightarrow \gamma$   
TRANSFORMATIONS IN AN IRON-NICKEL ALLOY

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16. Abstract  This is a study to investigate $\alpha \rightarrow \gamma$ transformations in an iron-nickel alloy.  Procedures used are presented. Some results are discussed.  Seven electron-microscope pictures included.					
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# An Electron microscope Study of $\alpha \rightarrow \gamma$ - Conversion in an Iron-Nickel Alloy

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The question concerning the kinetics and the mechanism <sup>/113\*</sup> of  $\alpha \rightarrow \gamma$  conversion in iron-nickel alloys has been repeatedly investigated and discussed in the literature [1-4]. Recently it has acquired considerable significance due to the development of new alloys on this base and the application of hardening treatment by means of direct and reverse  $\gamma \rightarrow \alpha \rightarrow \gamma$  conversion.

It is well known that in a broad interval of concentrations  $\alpha \rightarrow \gamma$  conversion in these alloys is due to the martensite mechanism. The formation of a contour on the polished surface of a sample containing an  $\alpha$ -phase after heating above the temperature of the beginning of  $\alpha \rightarrow \gamma$  -conversion is evidence of this. Moreover, the contour is similar to that which appears during cooling as the result of  $\gamma \rightarrow \alpha$  conversion [2]. The martensite character of the reverse conversion is also confirmed by its distribution over a broad temperature interval.

In addition, it has been shown that along with the martensite mechanism of  $\alpha \rightarrow \gamma$  conversion, a certain part of the  $\gamma$ -phase is converted into the  $\alpha$ -phase by the diffusion mechanism. Thus in [5] it was established for a number of iron-nickel alloys, containing from 9.5 to 18% Ni, that  $\alpha \rightarrow \gamma$  conversion was accomplished by a quick acting martensite mechanism and then by a diffusion mechanism whose rate depends essentially on the temperature and the composition. In the same paper a similar conversion process is reported for an alloy containing 30.9% Ni.

During conversion by the diffusion mechanism, diffusion

\* Numbers in margins indicate foreign pagination.

of nickel from the  $\alpha$ -phase into the  $\gamma$ -phase occurs with subsequent fusion of the supersaturated  $\alpha$ -solid solution into two equilibrium  $\alpha'$ - and  $\beta$ -solid solutions (the first of which is poor in nickel, and the second, rich). After cooling to room temperature, the  $\gamma$ -phase, which is rich in nickel, was converted into the  $\alpha$ -phase in accordance with the metastable diagram in the corresponding temperature region. When heated to 450°C the  $\beta$ -phase which precipitated out was so rich in nickel that it did not convert to the  $\alpha$ -phase at room temperature [5].

/114

Using X-rays, we observed an increase in the amount of the  $\gamma$ -phase during isothermal holding of an alloy with 32% Ni in the 400-425°C temperature range. The samples were cooled in advance in liquid nitrogen. After heating briefly to 500°C no traces of the  $\alpha$ -phase lines could be detected on the X-rays. To detect small amounts of the  $\alpha$ -phase which might still remain unconverted, the electron microscope method of examining thin films for breaks was employed. An iron-nickel alloy with 32% Ni concentration and initial martensite conversion temperature of -60°C was selected for the study. The conclusion of the reverse conversion for this alloy lies below 450°C. Samples in the form of 0.5 mm thick sheets were quenched from 1000°C and cooled to -196°C. Then they were placed in an oven and kept there until completely warmed up (4 min) at different temperatures. After being subjected to heat treatment, the samples were thinned down to 0.1 mm by a grinder and polisher. Disks of diameter 3 mm were cut out of these sheets by the electric spark method and then by electropolishing, thin foils were obtained for scanning in the electron microscope UEMV-100.

On the electron diffraction patterns obtained from the hardened samples only ghosts of the face-centered cubic lattices were observed. After cooling to -196°C, martensite con-

version occurs, which was easily observed both on the X-rays, as well as by means of the electron microscope. The  $\alpha$ -phase yield is nearly 80%.

In samples heated to 500°C we observed mainly the structure of the  $\gamma$ -phase which contains a large number of dislocations. However, regions with an  $\alpha$ -phase structure were also encountered comparatively often. Figure 1 contains a photomicrograph of such a region (x46000). On the photograph broad twins in the  $\alpha$ -phase (so-called deformational twins) and dislocations are visible. In the upper right corner an electron diffraction pattern is shown which was taken from this part of the sample. All spots of the electron diffraction pattern are contained in the body-centered cubic lattice. Spots belonging to the face-centered lattice were not detected.

Using the single-surface method of analysis, we determined the plane and direction of twinning. It turned out that deformational twinning occurs according to the system  $(121)_{\alpha}$   $[\bar{1}\bar{1}\bar{1}]_{\alpha}$ .

Besides the deformational twins, we also observed thin twins (the so-called conversion twins), traces of which are on the electron diffraction patterns. In Fig. 2 (x57000) the structure of a portion of a martensite crystal with thin twins is shown, and in the upper left [?] corner is an electron diffraction pattern of this portion. In it only ghosts of the  $\alpha$ -phase are observed. Besides the basic ghosts, the ghosts of the twins and traces are visible. The traces are located along  $[\bar{1}10]_{\alpha}$ ; d, perpendicular to the direction of the thin twins. Stereographic analysis has shown that thin twinning occurs due to the system  $[\bar{1}\bar{1}\bar{2}]_{\alpha}$   $[\bar{1}\bar{1}\bar{1}]_{\alpha}$ .

Sometimes a dislocational network was observed (Fig. 3, x40000) with two systems of almost parallel dislocations.



Figure 1

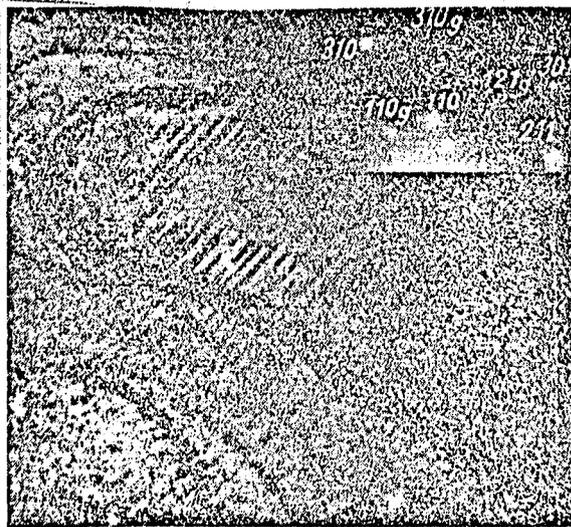


Figure 2

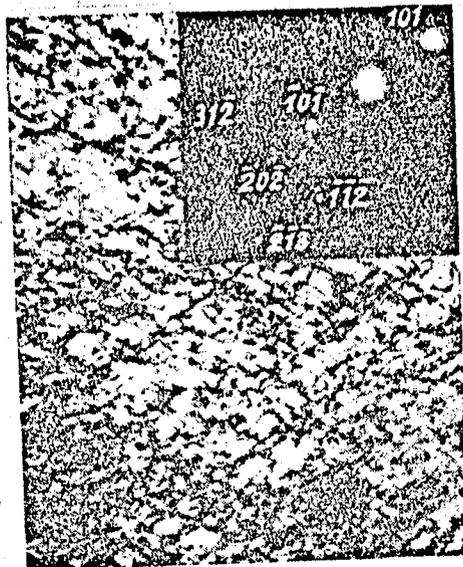


Figure 3

/116

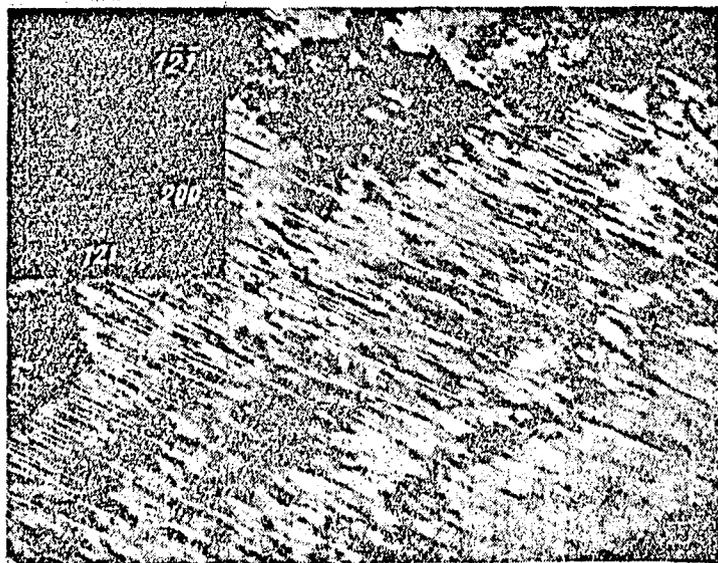


Figure 4

In the upper right corner is located a micro electron diffraction pattern of this portion. In it ghosts of the  $\alpha$ -phase are visible. One system of dislocations lies in  $(11\bar{1})_{\alpha} [101]_{\alpha}$ , and the other in  $(11\bar{1})_{\alpha} [\bar{1}10]_{\alpha}$ .

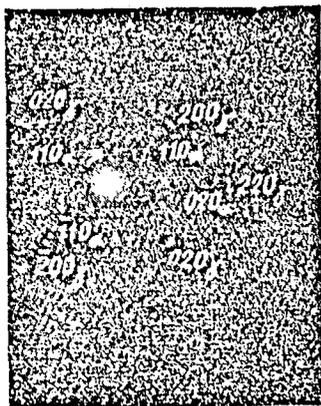


Figure 5

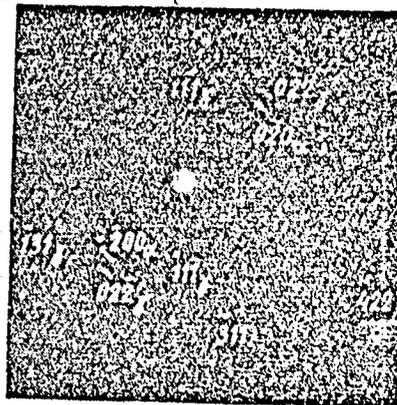


Figure 6

After heating the alloy to 600°C the number and magnitude of the portions with an  $\alpha$ -phase structure were substantially reduced. However, we were able to obtain micro electron diffraction patterns on which only ghosts belonging to the D-phase were nevertheless visible. On Fig. 4 (x29000) is a photomicrograph and the corresponding micro diffraction pattern. On the picture thin twins with high dislocation density are visible which have formed as the result of  $\gamma \rightarrow \alpha$  - conversion. As the temperature increases further, the number of portions with an  $\alpha$ -phase structure decreases more and more; however, at 700 and 800°C, along with  $\gamma$ -phase spots on certain micro electron diffraction patterns, ghosts are detected which belong to the body-centered cubic lattice.

On Figs. 5 and 6 there are photomicrographs taken of samples after heating to 700 and 800°C, respectively.

Taking into account the fact that after heating the alloy above 700°C we are unable to obtain electron photographs with

ghosts which belong to the  $\alpha$ -phase only, we can hence conclude that the  $\alpha$ -phase regions at these temperatures are smaller than the dimensions of the portion from which the microdiffraction was taken (5-7  $\mu$ c).

The presence of  $\alpha$ - and  $\gamma$ -phase ghosts on the same electron photograph makes it possible to determine their mutual crystallographic orientation. In this case there are the following orientational relations between the  $\alpha$ - and  $\gamma$ - phases.

$$\begin{aligned} [100]_{\gamma} &\parallel [\bar{1}\bar{1}0]_{\alpha}, \\ [010]_{\gamma} &\parallel [110]_{\alpha}, \\ [001]_{\gamma} &\parallel [001]_{\alpha}. \end{aligned}$$

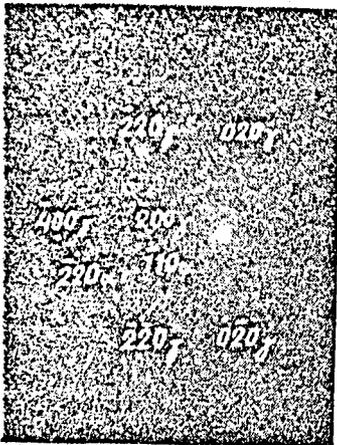


Figure 7.

However, in certain cases it was observed that the directions with low indices were not parallel. Thus in Fig. 7  $[100]_{\gamma}$  makes an angle of  $7^{\circ}$  with  $[\bar{1}\bar{1}0]_{\alpha}$ . The reasons for this will be stated below.

The interrelationship between the  $\alpha$ - and  $\gamma$ -phase lattices defined here is the typical mutual Bain's orientation [6]. This orientation was observed in a Fe-30.2% Ni alloy under compression along one of the cubic axes  $\langle 100 \rangle$ . The orientational relation between the  $\alpha$ - and  $\gamma$ - phases which was established differs from the familiar common orientation of these phases during martensite conversion [8].

Preservation of the  $\alpha$ -phase portions up to temperatures

significantly exceeding the temperature established previously for the conclusion of martensite conversion is in agreement with the results of dilatometric and microstructural studies in which it was shown that a certain part of the  $\alpha$ -phase is converted into the  $\gamma$ -phase by the diffusion mechanism [5,9,10]. Three portions can be isolated on the temperature differential curve, obtained for slowly heated samples of a Fe-32.5% Ni alloy [9]. Two of them are related to absorption, and one involves heat loss. The authors explain the course of the curve as follows. The absorption found on the first portion of the curve (up to a temperature of approximately 440°C) is connected with the conversion of approximately 60% of the martensite into austenite. Then the reverse conversion does not occur during heating to a temperature of 475°C. In this temperature range (440 - 475°C) the authors assume that diffusion of iron and nickel occurs across the interphase boundary between martensite and austenite. Here the martensite sheet combines with the nickel, the  $\alpha$ -phase is stabilized, and no structural changes are observed. Above 475°C (the third portion of the curve) a reverse conversion begins again which has, however, the characteristic features of a diffusion conversion: /119

- 1) a surface ghost is not formed;
- 2) the conversion continues for 30 hr at 500°C;
- 3) the structure which appeared when the austenite regions were heated does not differ from the structure of the portions due to the diffusion growth mechanism.

On the dilatometric curves obtained for a Fe-34% Ni alloy [10] a gradual decrease in the length of the sample was observed between 200 and 280°C, which is connected, the authors believe, with the controlled diffusion process. Above 280°C the conversion occurs in accordance with the martensite mechanism.

In the alloy studied by us, the basic part of the  $\alpha$ -phase

is converted into the  $\gamma$ -phase by the martensite mechanism. /119  
However, the conclusion of the reverse martensite conversion is in a temperature interval where the mobility of the atoms becomes somewhat greater so that conversion can proceed by the martensite mechanism. Subsequent conversion takes place by the slow-acting diffusion mechanism. The time during which the samples were kept in the oven in the experiments performed proved to be insufficient for completing the  $\alpha \rightarrow \gamma$  conversion by this mechanism even at 700 and 800°C temperatures. Moreover, in connection with the nickel deficiency the residues of the martensite phase are converted into the austenite phase at higher temperatures.

The presence of two reverse  $\alpha \rightarrow \gamma$  conversion mechanisms in iron-nickel alloys is apparently the reason why the ghost for reverse conversion does not always agree with the ghost for a direct martensite conversion [9, 11]. This is probably involved in the observation that the directions  $[100]_{\gamma}$  and  $[1\bar{1}0]_{\alpha}$  are sometimes not parallel (cf. Figure 7).

The possible occurrence of  $\alpha \rightarrow \gamma$  conversion due to the two mechanisms must be taken into account when subjecting iron-nickel alloys to heat treatment. The role of the diffusion mechanism is strengthened when the nickel content in alloys is lowered and the rate of heating is reduced. In this regard a substantial difference between the structure of the internal and surface layers may arise during the hardening of large test specimens.

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