

ORIENTED NUCLEATION IN THE REVERSE TRANSFORMATION
OF Fe-30 Ni

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Abstract: Reverse transformation of different martensite textures has been investigated. Starting with austenite the martensite was produced by rolling, deep cooling, or by a combined treatment. In most cases, during the reverse transformation the reconstitution of the original austenite texture occurred. This is explained by oriented nucleation of residual austenite during reverse transformation.

Reconstitution of the original austenite orientation in the transformation cycle austenite-martensite-austenite is a frequently observed process in alloys of iron with 30 wt.% Ni. On cooling to low temperatures, austenitic single crystals yield 12 different martensite orientations which can be derived from the orientation of the austenite with the aid of the Nishiyama orientation relationship.^{1,2} After the reverse transformation they again become single crystals with the same orientation as in the initial state.² Polycrystalline sheets with the cube texture also revert to the cube orientation after a transformation cycle^{3,4}; and the reversion to the original austenite orientation is observed in wires⁵⁻⁷ and sheets with orientations differing from the cube texture,⁸ even when plastic deformation had occurred between the forward and reverse transformations leading to individual deformation textures in each of the two phases.^{6,7} The reconstitution of the original austenite texture, the few exceptions to which are based on electron microscopic observations,⁹ has been explained in terms

of nucleation by retained austenite.^{6,7} Since this interpretation is disputed, the reverse transformation of various martensite textures has been reinvestigated.

PROCEDURE

The iron-nickel sheet under investigation (30.28 wt.% Ni, 0.02 wt.% C, 0.02 wt.% Cr, remainder Fe) was hot-rolled to 10 mm, and cold-rolled to 0.7 mm (as received). According to magnetic measurements of the retained austenite, and x-ray determination by Debye-Scherrer photographs, the as-received material contained almost 50% strain induced martensite. The cube texture was produced by a 3-hour vacuum anneal at 1000°C. All other anneals were carried out in salt baths for 5 min.

On deformation, the material is converted to about 60% deformed martensite; and it transforms almost completely to martensite when cooled in liquid nitrogen. Specimens in the as-received (deformed) and then cooled condition, contain somewhat more retained austenite than cooled cube-textured specimens. For microscopic examinations, the specimens were etched with a reagent¹⁰ which permits the differentiation of austenite from martensite based on their color contrast.

The determination of the pole figures in the back reflection and transmission regions was undertaken with an automatic texture-determination device.¹¹ The results are quantitative with the exception of the {111} pole figure of the cube texture. The intensity is expressed in the calculated units of random orientation.

EXPERIMENTAL RESULTS

Textures

Starting material. The austenite of the deformed sheet has the normal copper-type rolling texture. Figure 1 shows the texture of the strain induced martensite which develops during the rolling process. Whereas this texture shows the normal orientation spread of the rolling texture of b.c.c. iron, its maxima are not in accordance with the b.c.c. rolling texture, but correspond only to the orientation (112)[$\bar{1}10$].

Annealing the as-received sheets at 500°C leads to the disappearance of the martensite. It does not, however, change the austenite texture, and is therefore the first example of reversion to the austenite texture in an $\alpha \rightarrow \gamma$ reverse transformation. At temperatures above 600°C a very sharp cube texture is produced.⁴

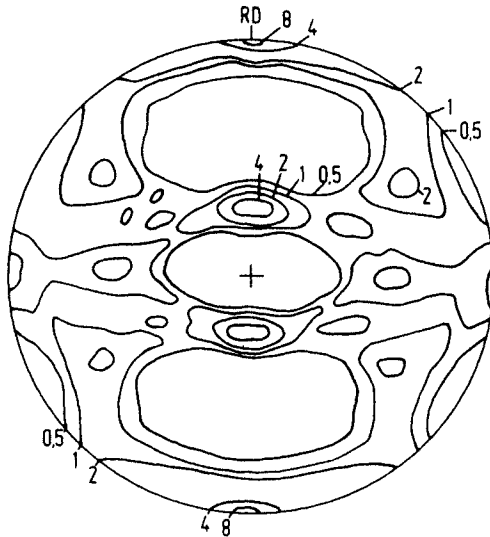


FIGURE 1. $\{110\}$ pole figure of strain induced martensite in the as-received material.

Cooling of copper-type rolling texture to low temperatures. If an annealed (500°C) sample of the starting material is cooled to low temperatures, the alpha texture in Figure 2 results. The remarkable feature of this texture is that the $\{110\}$ poles in the plane ND-RD are at the same position as two $\{111\}$ poles of the austenite texture. On reverse transformation at 500°C the initial texture is reconstituted. Furthermore, at temperatures above 600°C a cube texture is produced, which is as sharp as that produced by annealing the starting material (Figure 3).

Rolling and cooling of cube texture. If a sheet with the cube texture is rolled, which was possible to a degree of 87%, the austenite texture illustrated in Figure 4 is formed, whilst the martensite produced by the deformation assumes the texture shown in Figure 5. Reverse transformation leads to an austenite texture scarcely different from the initial texture (deformed cube texture) as shown in Figure 6. Perfect coincidence of the two textures cannot be expected inasmuch as different textures could not be investigated on the same specimen. If cooling to low temperatures is interposed between the deformation and the reverse transformation (Figure 7), reverse transformation again yields a texture similar to Figure 6. The texture of the strain induced martensite is, however, slightly altered by the cooling and the associated transformation of the retained austenite (Figure 7).

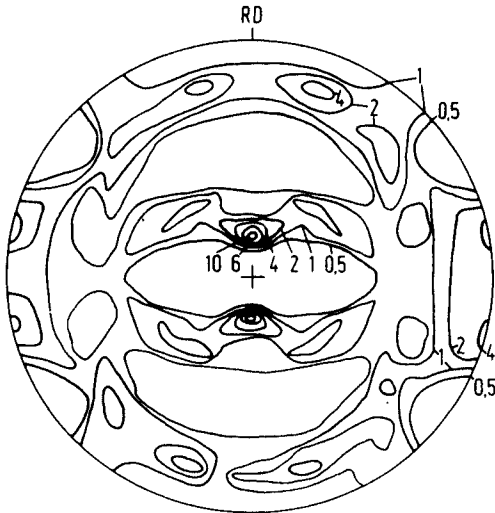


FIGURE 2. $\{110\}$ pole figure of the martensite formed by deep cooling the annealed (at 500°C) austenite with the copper-type rolling texture.

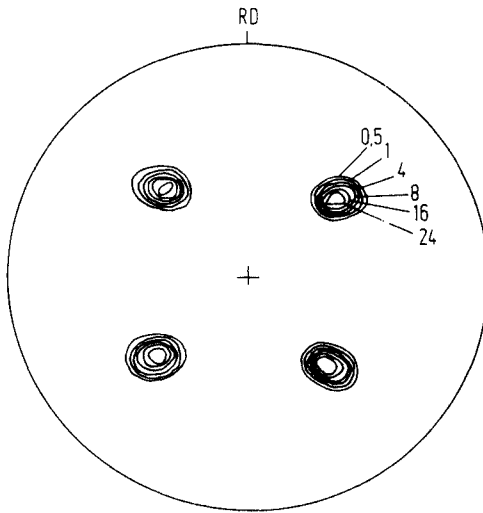


FIGURE 3. $\{111\}$ pole figure showing the cube texture developed at 700°C in a specimen deep-cooled and reverse-transformed at 500°C .

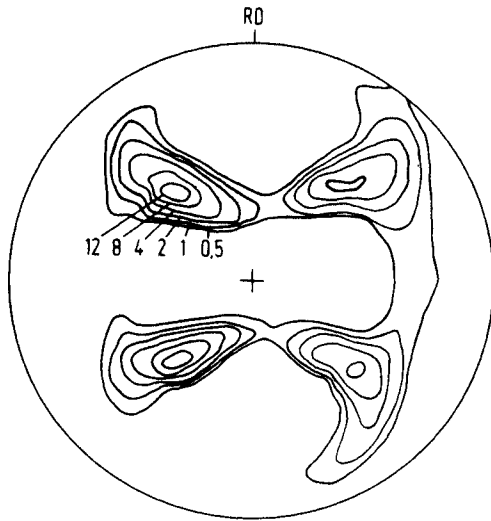


FIGURE 4. $\{111\}$ pole figure showing cube-textured austenite after rolling 87%.

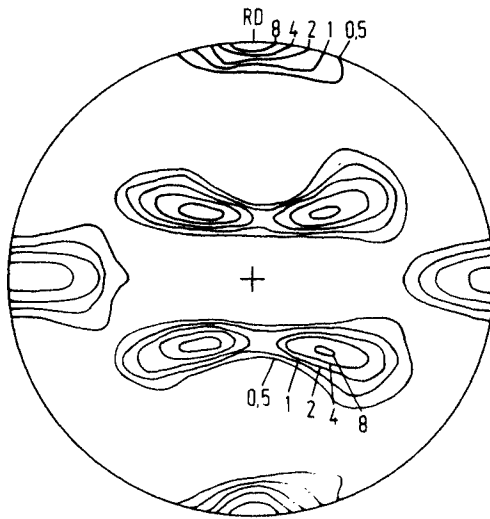


FIGURE 5. $\{110\}$ pole figure of martensite developed from austenite in cube texture (Figure 4) by rolling.

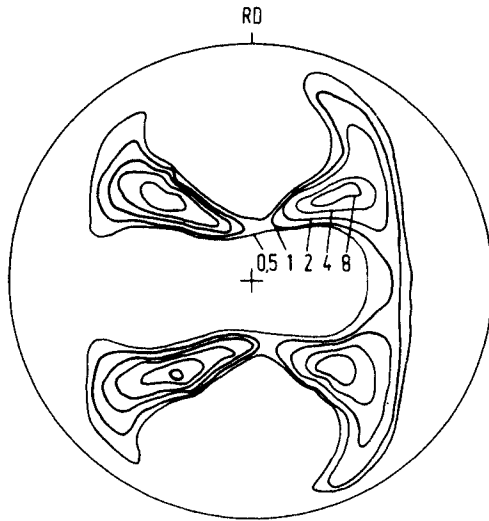


FIGURE 6. $\{111\}$ pole figure of austenite after reverse transformation at 500°C . The texture of the strain induced martensite is shown in Figure 5, and that of the residual austenite is shown in Figure 4.

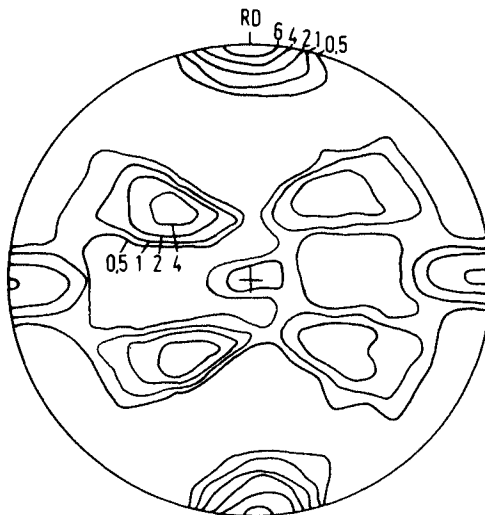


FIGURE 7. $\{110\}$ pole figure of martensite developed by deep cooling a sample with deformed cube texture (Figure 4) and strain induced martensite (Figure 5).

Rolling of previously cooled starting texture and cube texture. If the as-received material having the copper-type rolling texture and a martensite texture, as in Figure 1, is first cooled to low temperatures and then deformed, reverse transformation does not lead to the copper-type rolling texture but to a completely new texture. The same applies if the cube texture is first cooled, then deformed, and transformed.¹² If, however, these newly formed austenite textures are cooled to low temperatures and then transformed by annealing, the material always reverts to the austenite texture existing before the cooling.

Metallographic Investigation

Figures 8a-d show a series of micrographs of a specimen cooled to low temperatures and annealed 30 seconds at successively higher temperatures. Just as the athermal martensite transformation the reverse transformation into austenite comes to a halt if the material is held at a constant temperature. The micrographs all show the same specimen which has been repolished and re-etched so that each time a lower level is exposed. This results in small changes in the proportion and shape of the crystals which have nothing to do with the anneal. The austenite phase is the structural component which shows up most brightly. Even in Figure 8a where parts of the martensite have very light contrast, the retained austenite can still be recognized clearly.

Before cooling, the sheet had the cube texture. Figure 8a shows a polished section of the cooled specimen with athermal martensite and residual austenite (white areas). An earlier investigation⁴ has already shown that some of the martensite crystals are very large and others very small. Small quantities of residual austenite could be detected metallographically, but were not indicated by the Debye-Scherrer photographs, or by the magnetic measurements of the residual austenite.

The austenite transformation began at about 370°C. Upon annealing at only 380°C, the proportion of austenite had increased, and this increase continued as the annealing temperature was raised (Figure 8b and c) until the reverse transformation was complete at about 482°C. The pictures show clearly that the growth of austenite starts in the residual austenite regions, i.e. in the region among fine martensite needles, after which the large martensite needles are also gradually consumed.

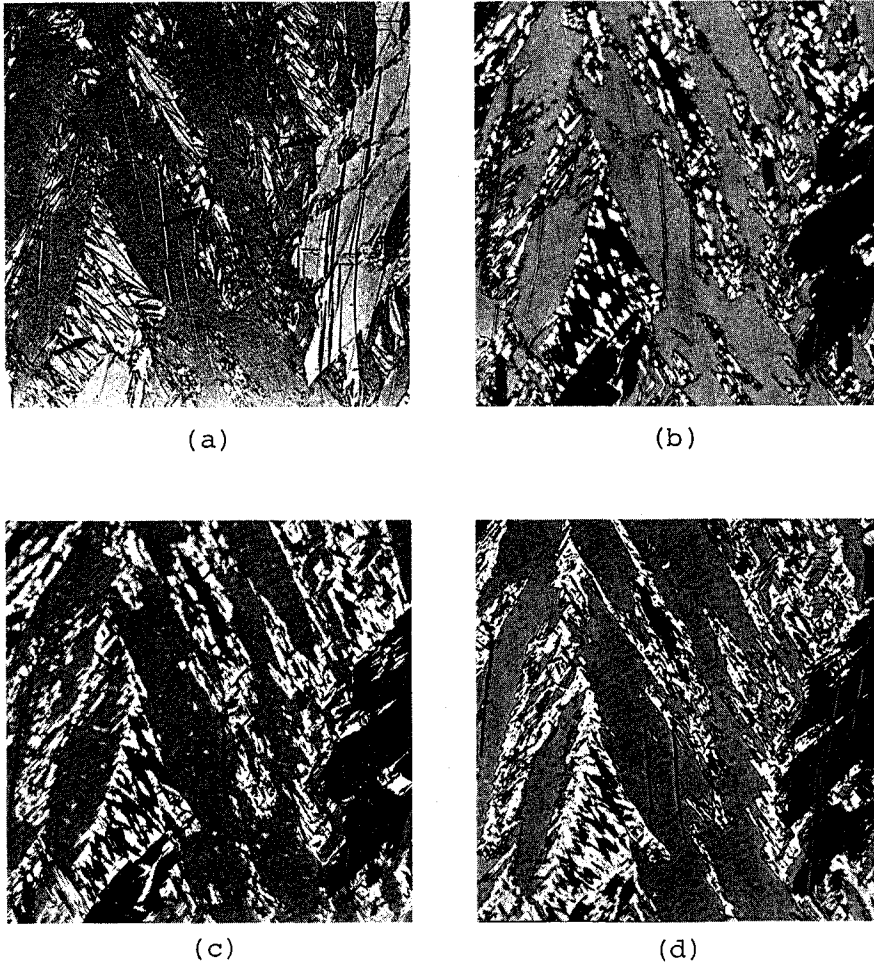


FIGURE 8a-d. Microstructures of a deep-cooled specimen with cube texture (a), after heating to 380°C (b), 380 + 386 + 391°C (c), and 380 + 386 + 391 + 400°C (d) (in every case 30 s), x 400. The arrows point to the retained austenite.

DISCUSSION

Martensite Textures

As was shown in an earlier publication,⁴ all 12 orientations possible according to the Nishiyama mechanism $[(111)\gamma||(\bar{1}10)\alpha]$ and $[(\bar{1}10)\gamma||[001]\alpha]$ arise when the cube texture is cooled to a low temperature. Whether

or not this is also true for the copper-type rolling texture cannot be established because of the large number of different orientations shown by this texture in the scatter regions around its ideal orientations. At all events, attempts in calculating a martensite texture by utilizing various ideal copper-type orientations were unsuccessful. The earlier mentioned coincidence of the two $\{110\}_\alpha$ and the two $\{111\}_\gamma$ poles in the plane of the normal to the sheet and the rolling direction (ND-RD) is however striking.

Two different martensite textures were formed on deformation. In one case the cube texture, which, as a result of deformation, had developed a strong scatter best described by rotation about the rolling direction (RD), gave rise to martensite in the $(001)[\bar{1}10]$ orientation. In the other case, the copper-type rolling texture yields a texture corresponding to the overall rolling texture of b.c.c. metals but with maxima which could be described by only one ideal orientation, namely $(112)[\bar{1}10]$. This ideal orientation is normally used to describe the rolling texture of b.c.c. materials, but in that case it is only one among many orientations which can be derived from a fiber texture parallel to the rolling direction.¹³ Besides, a second fiber texture is also present.

In interpreting these textures, the superposition of transformation and change in orientation due to rolling must be taken into account. On rolling, the austenite, which, as in the case of the cube texture, can itself change its orientation, is transformed into martensite; and the transformed martensite also undergoes orientational changes on rolling. The transformation is accomplished continuously while traversing various austenite orientations during the rolling process. Assessment of these relationships is complicated by the fact that it is not known whether strain induced and athermal martensite have the same orientation relationship with the austenite. If, however, the pole figures of the deformed cube texture (Figure 4) and that of the martensite derived from it (Figure 5) are compared with the martensite orientations derived from the cube texture according to the Nishiyama mechanism in Figure 9, a good agreement is observed. From this it can be inferred that the orientation relationships must at least be similar. The absence of certain orientations, especially at the center of the pole figure, does not contradict this inasmuch as it is known that one or more of the possible orientations may fail to arise as a result of stresses.^{4, 14} Also to be taken into account is the deformation of the martensite which starts virtually as soon as it forms.

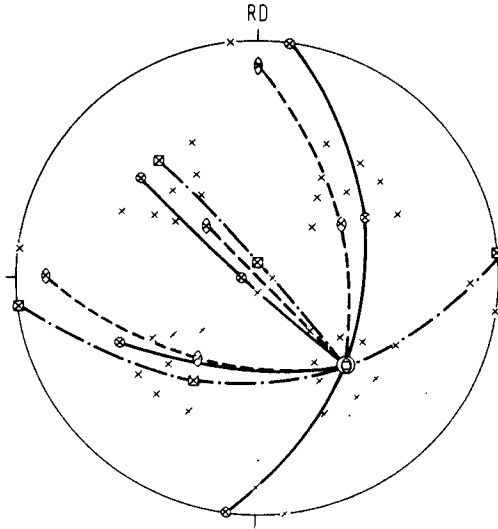


FIGURE 9. $\{110\}$ poles of martensite constructed by Nishiyama orientation relationship from cube texture $(001)[100]$. 3 of 12 possibilities are marked by different symbols.

If the specimen with the deformed cube texture is cooled to a low temperature after rolling, the orientations previously absent at the center of the $\{110\}$ martensite pole figure (Figure 7) appear (*cf.* Figures 5, 7, and 9). Also, the scatter towards ND around the RD in the pole figure shown in Figure 7 is now such that all the constructed $\{110\}$ poles (Figure 9) fall within the experimentally observed orientations.

The formation of strain induced martensite from copper-type rolling texture is even more complicated than that of athermal martensite from the same starting texture. Here, again, differences exist between the textures of athermal martensite (Figure 2) and strain induced martensite (Figure 1), but there is also a certain similarity. Again, in the case of strain induced martensite it is noticeable that there is a pole in the ND-RD plane occupying almost the same position in the $\{111\}$ as in the $\{110\}$ pole figure, even though the agreement is not as good as in athermal martensite. Moreover, maxima occur around the RD in both pole figures of the strain induced martensite. These intensity maxima are absent in those pole figures of athermal martensite.

These investigations on the formation of martensite by deformation and by cooling to low temperatures do not permit a definite conclusion as to whether the two types

of martensite are formed according to the same orientation relationship. However, there are many indications that this is the case. The texture of strain induced martensite differs from that of athermal martensite in that not all orientations possible according to the orientation relationship are in fact formed because of the stresses. Moreover, the martensite orientations are changed in the direction of the stable final texture of b.c.c. metals by the deformation subsequent to the transformation.

Reverse Transformation

With the exception of the two cases mentioned earlier of a transformation by cooling to low temperatures before deformation,^{1,2} reverse transformation always results in the austenite texture existing prior to the first transformation. The metallographic investigations show very clearly that the reverse transformation begins at the retained austenite. On this basis, it is concluded that the retained austenite has a nucleating effect, and that this is a case of oriented nucleation in a phase transformation. In the case of specimens which showed no recovery of texture on reverse transformation, only extremely small amounts of residual austenite could be detected metallographically. Clearly, these were insufficient to control the formation of the austenite texture. So far one explanation for the reconstitution of the austenite texture was that in martensite transformation and in reverse transformation the same paths were followed. These would be destroyed if a deformation is interposed between the martensite and the reverse transformation. This could explain why a new martensite texture developed in the case which was mentioned last. Despite this, few examples do exist in which the austenite texture is reconstituted even though a deformation has been imposed. For example, during the rolling of a cube-textured specimen strain induced martensite is formed, which transforms again into cube texture upon annealing. It appears, therefore, that the reconstitution of the austenite texture can only take place by oriented nucleation of the residual austenite.

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REFERENCES

1. Z. Nishiyama, *Sci. Rep. Tohoku Univ.*, I, 23, 637 (1934).
2. G. Wassermann, *Mitt.K.-Wilh.-Inst. Eisenforsch.*, 17, 149 (1935).
3. G. Wassermann, *Arch. Eisenhüttenw.*, 10, 321 (1936/37); 11, 89 (1937/38).
4. D. Sattler and G. Wassermann, *J. Less Common Metals*, 28, 119 (1972).
5. G. Wassermann, *Arch. Eisenhüttenw.*, 6, 347 (1932/33).
6. J. Grewen and G. Wassermann, *Arch. Eisenhüttenw.*, 32, 863 (1961).
7. G. Wassermann and J. Grewen, *Texturen metallischer Werkstoffe*, Springer-Verlag, Berlin, 2nd Ed., 1962, Chap. 3.5, p. 400-407.
8. H. Abe, K. Ito and R. Fukumoto, *Nippon Kinzoku Gakkaishi*, 31, 37 (1967).
9. H. Kessler and W. Pitsch, *Arch. Eisenhüttenw.*, 38, 321, 461 (1967).
10. A. O. Bencoter, J. R. Kilpatrick, and A. R. Marder, *Prakt. Metallogr.*, 5, 694 (1968).
11. J. Grewen, D. Sauer, and P. Wahl, *Z. Metallkde.*, 61, 430 (1970).
12. About these results another paper will be published.
13. See for example: H.-J. Bunge, *Mathematische Methoden der Texturanalyse*, Akademie-Verlag, Berlin, 1969, Chap. 7.1.2, p. 118-127.
14. P. Sattler, *Z. Metallkde.*, 65, 479 (1974).