JOURNAL OF MATERIALS SCIENCE 14 (1979) 693–698

The structure of burst and isothermal martensites in an Fe–24 wt % Ni–0.5 wt % C alloy

D. S. SARMA
Department of Metallurgical Engineering, Banaras Hindu University, Varanasi-221005, India

J. A. WHITEMAN, S. R. KEOWN
Department of Metallurgy, The University of Sheffield, UK

It has been found that an Fe–24 wt % Ni–0.5 wt % C alloy transforms to martensite on cooling to −80° C by two separate processes to give burst and isothermal plates. Previous work has only indicated burst transformation at −196° C. The burst plates have a characteristic lenticular shape with midribs and internal twinning whereas the isothermal plates are chevron-shaped, each having two arms meeting at an obtuse angle on a common crystallographic plane. The isothermal plates have dislocation as their substructure with no evidence of twinning or midribs. Trace analysis shows that the isothermal martensite has a $\{2\,5\,9\}_\gamma$ habit plane which is the same as that of burst martnesite as found by earlier workers. This suggests that the habit plane is controlled by composition rather than by the substructure of martensite or kinetics of the process.

1. Introduction

It has now been well established that martensites formed in iron-base alloys can be of different morphologies depending on composition [1]. In low alloy steels the usual form is of lath martensite containing dislocations and as the alloy content is increased the laths are replaced by plates with various morphologies, internal structures and habit planes depending on the particular alloy additions.

The structure of an iron-base alloy containing 24 wt % Ni and 0.5 wt % C has previously been studied by transmission electron microscopy [2] to show that on transformation at −196° C virtually all the austenite transforms to fully twinned lenticular plates of burst martensite with a non-uniform twin distibution. This particular alloy has an M_s temperature of about −30° C and it has been found in the present work that the transformation behaviour of cooling to a temperature of −80° C is different from the previously reported [2] transformation to burst martensite at −196° C. On cooling to −80° C, partial transformation to burst martensite with audible clicking occurs followed by further transformation of the austenite to give isothermal martensite on holding the alloy at −80° C for periods of up to 240 h. Optical and electron microscopy show that the morphology and internal structure of the two types of martensite are quite different and the purpose of this paper is to describe these differences.

2. Experimental procedure

A 11 kg ingot of an Fe 23.7% Ni–0.53% C alloy was prepared by vacuum-melting high-purity raw materials. The ingot was hot-extruded and rolled to 12 mm bar, homogenized at 1200° C for 50 h before cold swaging to a 5 mm diameter bar and finally machined to give 3 mm diameter rods suitable for thin foil preparation from disc specimens. The rod material was austenitized at 950° C for 1 h in evacuated silica tubes before rapid quenching into iced brine at 0° C. The

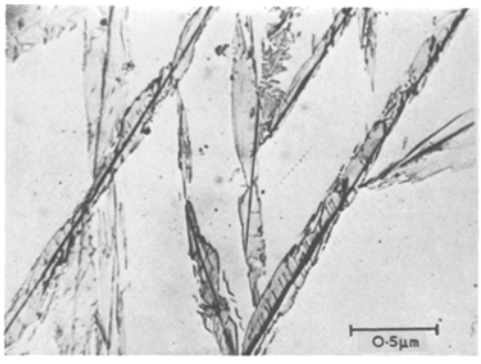

Figure 1 Optical micrograph of the Fe–24% Ni–0.5% C alloy, obtained soon after hearing the first click, showing burst martensite plates.

Figure 3 Optical micrograph of the alloy after holding at $-80°$ C for 10 days, showing large number of isothermal chevron plates in addition to burst plates.

specimens were still fully austenitic and were cooled to $-80°$ C and held at this temperature for various periods of time up to 240 h to give partial transformation to martensite.

Transverse sections of the heat-treated rods were mechanically polished and etched in 4% nital for examination by optical microscopy. Thin foils were prepared by the jet technique of sectioning the 3 mm rod to give 125 μm thick discs, rapidly profiling both sides by jet electropolishing in 20% perchloric acid in methanol and finally electropolishing in 10% perchloric acid in methanol to give performation. The foils were examined in an AEI EM6G electron microscope at 100 kV.

3. Experimental results

3.1. Optical microscopy

The microstructures obtained on transforming the alloy at $-80°$ C are shown in Figs. 1 to 4.

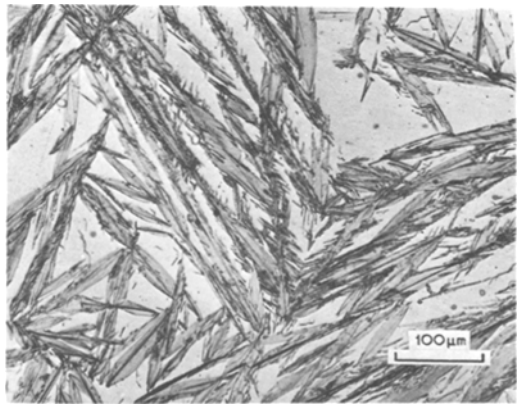

Figure 2 Optical micrograph, taken soon after the burst transformation is complete, showing burst plates and a few isothermal chevron plates.

Although controlled temperatures within the range between ambient temperature and $-80°$ C were not obtained, some specimens were removed from the refrigerant bath before the specimen had time to reach $-80°$ C and these showed the progress of transformation on cooling to $-80°$ C. Fig. 1 shows the microstructure of a specimen taken out of the refrigerant bath soon after hearing the first click or burst so that the temperature of this specimen was below $-30°$ C, the M_s temperature, but had not had sufficient time to reach $-80°$ C. The structure shows big lenticular plates with a few chevron plates adjacent to the large burst plates. By allowing a specimen to completely attain a temperature of $-80°$ C the structure revealed an increased number of burst and chevron plates (Fig. 2). In addition, the interfaces of some of the large burst plates appeared to have 'ragged' edges due to the nucleation of chevron plates on the austenite/lenticular plate interfaces.

Transformation for 240 h at $-80°$ C produced a further increase in the numbers of chevron plates

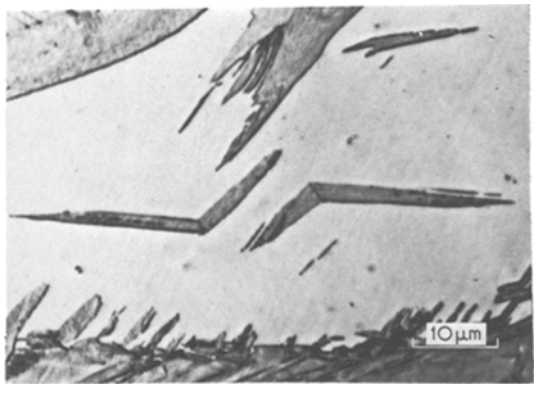

Figure 4 Optical micrograph showing two chevrons.

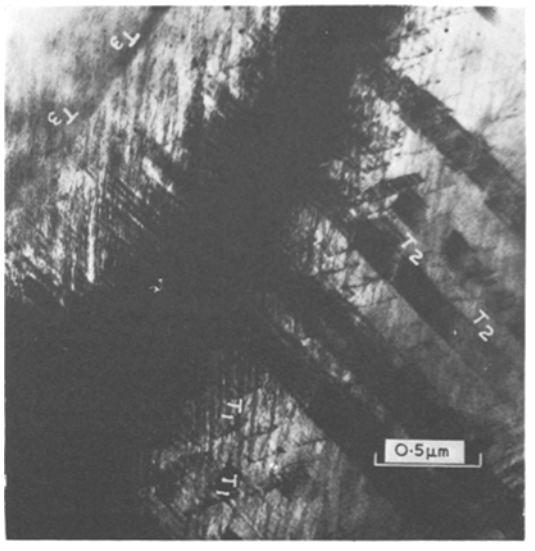

Figure 5 TEM of burst martensite showing fine twins (T_1T_1) coarse twins (T_2T_2) and deformation twin (T_3T_3).

nucleated in the austenite in between the burst plates (Fig. 3). At higher optical magnifications it could be seen that each of the chevron-shaped isothermal martensite plates consisted of two arms with a distinct boundary separating them (Fig. 4).

3.2. Electron microscopy

Transmission electron microscopy clearly revealed the internal structure of the two types of the martensite plates. Considerable amounts of twinning were observed in the burst plates. Fig. 5 shows fine twins close to the midrib (T_1T_1), coarse twins (T_2T_2) and a deformation twin (T_3T_3). A study of stereographic projection of the various trace normals revealed that the fine twins and the deformation twins are of $\{2\,1\,1\}_\alpha$ type whilst the coarse twins are on $\{1\,0\,1\}_\alpha$. Occasionally several lenticular plates formed adjacent to one another and such an area is shown in Fig. 6. Only fine twinning is found in these plates with no midrib.

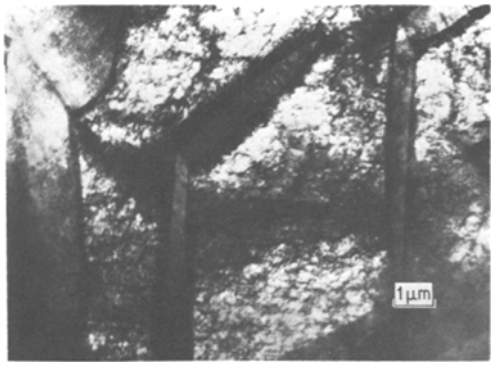

Figure 6 TEM of burst plates showing fine twins.

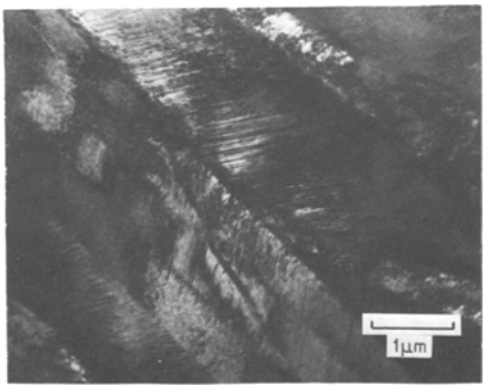

Figure 7 TEM showing three chevron plates in an austenite matrix.

The internal structure of the isothermal plates is shown in Figs. 7 to 9. Fig. 7 shows three martensite plates having chevron morphology, in the austenite matrix. The internal structure of these plates consists of a very high density of dislocations without twinning. Occasionally it was observed that one of the arms of a chevron-shaped plate would branch into several smaller arms (Fig. 8). Plates with a single arm were also observed occasionally as in Fig. 9. These plates were parallel to one of the arms of a larger chevron plate.

The orientation relationships between austenite and martensite for isothermal plates were determined from Fig. 10, an electron diffraction pattern containing $[1\,1\,2]_\gamma$ and $[0\,1\,2]_\alpha$ zones, and gave the following relationship:

$$[1\,1\,2]_\gamma \sim \| [0\,1\,2]_\alpha$$

$$(1\,\bar{1}\,0)_\gamma \sim 3^\circ\,(1\,0\,0)_\alpha$$

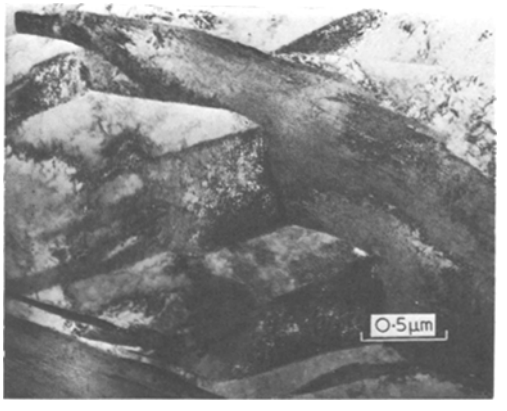

Figure 8 TEM of isothermal martensite showing branching of the chevrons.

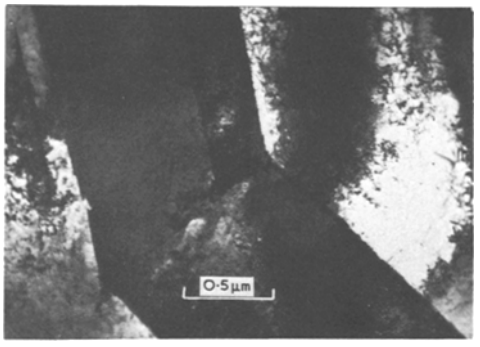

Figure 9 TEM of a chevron plate and a thin single arm martensite plate (on the left).

This is consistent with the Kurdjumov–Sachs orientation relationship (see, for example, [3]).

The habit plane of the isothermal plates was determined by standard trace analysis methods using transmission electron microscopy techniques by making measurements on six plates and was found to be close to $\{2\,5\,9\}_\alpha$. The accuracy of the measurement is about $\pm 2°$. The result is shown in Fig. 11 along with the observed scatter for the $\{2\,5\,9\}_\gamma$ martensites. The habit plane of the burst martensite plates in the Fe–24% Ni–0.5% C alloy, as measured by Brook and Entwisle [9] and Brook [18] is also shown in Fig. 11.

4. Discussion

The present work established that isothermal martensite forms in an Fe–124% Ni–0.5% C alloy in addition to the burst transformation previously reported [2] if quenched and held at $-80°$ C. The isothermal martensite has a morphology different

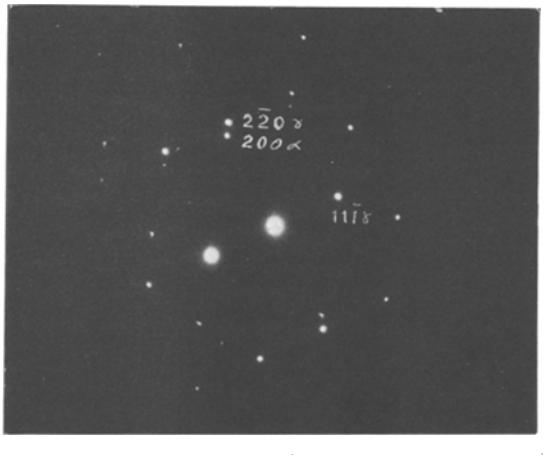

Figure 10 Selected-area diffraction taken from both austenite and martensite.

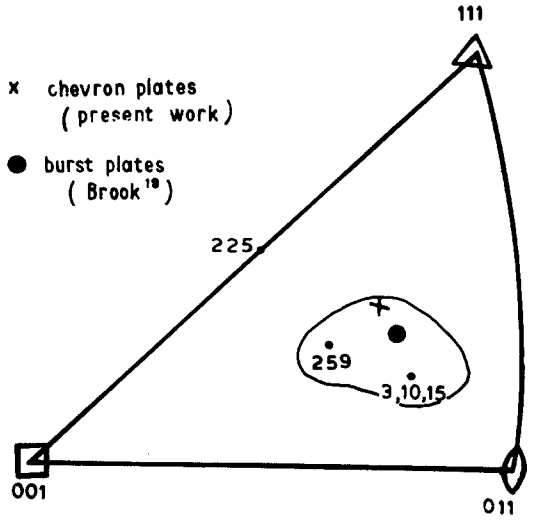

Figure 11 Habit plane of the isothermal martensite plates in the Fe–24% Ni–0.5% C alloy. The habit plane results for burst martensite are of Brook [9].

from the burst martensite and it is, therefore, possible to identify the two types unambiguously. The isothermal martensite can form in the present alloy only under favourable conditions, as quenching to very low temperatures (such as $-196°$ C) probably transforms all the austenite into burst martensite. Even if some isothermal martensite is formed at $-196°$ C it may be difficult to identify the same amongst a very large percentage of burst martensite. This investigation, therefore, records for the first time the co-existence of isothermal and burst martensites in the same alloy and points out the importance of adjusting the experimental conditions for obtaining isothermal martensite in an alloy that would have otherwise predominantly transformed to burst martensite.

It is not, however, the first time that martensite plates with chevron morphology were observed. Maskimova and Nikonorova [4] observed the plates in a deformed and partially transformed austenite and termed them 'bracket martensite'. Similar plates were also noted by Kelly and Nutting [5] in an Fe–20% Ni–0.8% C alloy transformed by deformation at room temperature. Zackay *et al.* [6] and Reed [7] found a heavy concentration of "V-shaped satellite plates" along the sides of large lenticular plates in an Fe–24% Ni–0.4% C and Fe–31% Ni alloys, respectively, when the deformed austenites were partially transformed. Quite recently, Snell *et al.* [8] observed chevron plates (which they termed butterfly plates) in an Fe–21% Ni–0.6% C alloy

along with burst plates when it was transformed at -40° C, just 4%C below the M_s temperature. They have also reported that the structure is of predominantly butterfly martensite when the alloy was shock-loaded at room temperature. All these investigations suggest that the chevron plates could be formed either by transformation during the deformation of austenite or by transformation on lowering the temperature of a deformed alloy. It has been shown by some other investigators, however, that chevron plates become the predominant structure of martensite in an Fe–19% Ni–0.5%C alloy [9, 10]. Such plates are also formed alongside the lenticular plates in some other alloys like Fe–1.8%C [1], Fe–29% Ni [7], Fe–27% Ni–3% Cr [11] and Fe–25% Ni–6% Ti [12] when transformed from the undeformed austenite. In the present work also, the chevron plates were formed from undeformed austenite. The formation of chevron plates is, therefore, aided by the strains in austenite; these strains can result either by plastic deformation or by the prior formation of burst plates.

Apart from the morphological differences between the burst and isothermal plates, there is also a major change in internal structure. The burst plates are internally twinned over the entire width while the isothermal chevron plates always showed an internal structure of dislocations with no evidence of transformation twinning. The dislocation density was fairly high and it was not possible to resolve them into either screw or edge type as was possible for certain untwinned portions of burst martensites in Fe–Ni alloys [13]. Two distinct types of twins were noted in the burst plates, coarse twins on $\{0\,1\,1\}_\alpha$ and fine twins on $\{2\,1\,1\}_\alpha$. The fine $\{2\,1\,1\}_\alpha$ twinning near the midrib is consistent with previous work [13, 14] but the $\{0\,1\,1\}_\alpha$ coarse twinning was unexpected as the latter have been reported by Oka and Wayman [15] in $\{2\,5\,9\}_\gamma$ martensites having a very high carbon content such as in an Fe–1.82%C alloy. The $\{0\,1\,1\}_\gamma$ twins were, however, a common feature of several $\{2\,2\,5\}_\gamma$ martensites [16, 17].

The habit plane of the isothermal plates was found to be $\{2\,5\,9\}_\gamma$ although Brook and Entwisle [9] and Brook [18] previously found that the habit plane of similar plates in an Fe–19% Ni–0.5%C alloy was $\{2\,2\,5\}_\gamma$. This latter determination was by optical methods and the authors admit to considerable scatter due to difficulty in obtaining traces in the two surfaces due to small size of the plates. The trace analysis technique in the present work using electron micrographs and electron diffraction patterns is better suited because of the small size of the plates and has clearly shown a $\{2\,5\,9\}_\gamma$ habit (Fig. 11) and it is interesting to note that this is the same as the habit plane for burst martensite [9, 18]. It is perhaps surprising that martensite plates differing in kinetics, morphology and internal structure but having the same habit plane can form in the same alloy. This result suggests that the habit plane is dependent only on the austenite composition and not on other variables such as kinetics or morphology.

The unusual morphology of the isothermal plates is that they consist of two arms meeting at a common plane to give a distinct chevron shape. The significance of this boundary plane is not understood at present but it seems possible that one arm is nucleated first of all, rather than the concurrent growth of both arms from the common boundary. This is supported by the observation of small single arms in some areas with the single arm parallel to one of the arms of a complete chevron as in Figs. 4 and 9. In addition, the fact that one arm of a plate could occasionally branch to give several other smaller arms, as shown in Fig. 8, indicates that the plates do not form by the two arms growing away from the common boundary plane. The present results thus suggest that the boundary plane is not the nucleus for an isothermal chevron plate.

5. Conclusions

(1) Isothermal martensite forms in an Fe–24% Ni–0.5%C alloy after the formation of burst martensite on cooling to -80° C. The isothermal martensite was found to form extensively on holding the alloy at -80° C for periods up to 240 h.

(2) The burst martensite was characteristically lenticular in shape with midribs and has fine internal twinning on $\{2\,1\,1\}_\alpha$ planes and coarse twinning on $\{0\,1\,1\}_\gamma$. Occasionally lenticular plates formed closely adjacent to one another. These plates showed no midrib and had only fine $\{2\,1\,1\}_\alpha$ twinning.

(3) The isothermal martensite plates were chevron-shaped, untwinned and contained a high density of dislocations. The habit plane was found to be $\{2\,5\,9\}_\gamma$ which is the same as the habit for the burst plates suggesting that the habit plane is

dependent on the composition of the austenite rather than on the kinetics or the morphology of martensite.

Acknowledgements

The authors would like to thank Professor G. W. Greenwood, Head of the Department of Metallurgy, University of Sheffield, for the provision of laboratory facilities. The continued encouragement of Mr J. H. Woodhead is acknowledged. One of the authors (D.S.S.) would like to thank the Association of Commonwealth Universities for financial support.

References

1. G. KRAUSS and A. R. MARDER, *Met. Trans.* **2** (1971) 2343.
2. M. G. H. WELLS, *Acta Met.* **12** (1964) 389.
3. K. W. ANDREWS, D. J. DYSON and S. R. KEOWN, "Interpretation of Electron Diffraction Patterns", 2nd Edn. (Adam Hilger, London, 1971) p. 52.
4. O. P. MASKIMOVA and A. I. NIKONOROVA, *Prob. Metalloved. Fiz. Metall.* **4** (1955) 123.
5. P. M. KELLY and J. NUTTING, *J.I.S.I.* **199** (1961) 1971.
6. V. F. ZACKAY, M. W. JUSTOSSON and D. J. SCHMATZ, "Strengthening Mechanisms in Solid" A.S.M., Metals Park, Ohio, 1962) p. 179.
7. R. P. REED, *Acta Met.* **15** (1967) 1287.
8. E. O. SNELL, J. C. SHYNE and A. GOLDBERG, *Metallogr.* **10** (1977) 243.
9. R. BROOK and A. R. ENTWISLE, *J.I.S.I.* **203** (1965) 905.
10. A. R. ENTWISLE and J. A. FEENY, "The Mechanism of Phase Transformations in crystalline Solids", Institute of Metals Monograph No. 33 (1969) p. 156.
11. J. F. BREEDIS, *Trans. Met. Soc. AIME* **230** (1964) 1583.
12. D. S. SARMA and J. A. WHITEMAN, *Met. Trans.* **5** (1974) 163.
13. R. L. PATTERSON and C. M. WAYMAN, *Acta Met.* **14** (1966) 347.
14. H. WARLIMONT, "Proceedings of the 5th International Conference on Electron Microscopy" Vol. 1, (Academic Press, London, 1962) HH-6.
15. M. OKA and C. M. WAYMAN, *Trans. ASM* **62** (1969) 370.
16. S. JANA and C. M. WAYMAN, *Met. Trans.* **1** (1970) 2825.
17. K. SHIMIZU, M. OKA and C. M. WAYMAN, *Acta Met.* **19** (1971) 1.
18. R. BROOK, Ph. D. Thesis, University of Sheffield (1962).

Received 9 June and accepted 21 July 1978.