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The kinetics of deformation induced martensite reaction was studied in conditions of stress-assisted and strain-induced transformation defined on the basis of the temperature dependence of the flow stress of the alloy. The system was perturbed by either changing grain size or subjecting it to compressive shockwave (8 GPa). The results indicate that, at a given temperature, a prior shock-loading causes an enhancement of the initial rate of martensite formation while grain refining has the opposite effect. The total amount transformed up to necking is affected in the same way.

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I. Introduction

It is well known that the stability of austenite and consequently its transformation into martensite can be affected by changing its microstructure or substructure⁽¹⁾⁽²⁾. Recently⁽³⁾ it has been shown that it is possible to alter the deformation-transformation behavior of Fe-31 % Ni-0.1 %C either by changing the austenite grain size or by introducing in the matrix a high density of defects by a prior shock-loading treatment. However, that work was limited in scope because it did not consider the kinetics of deformation-induced martensite. The results of such a complementary study are the subject matter of this communication. Both the so-called "stress-assisted" and "straininduced" regimes of transformation, defined on the basis of the temperature dependence of the yield stress of the alloy(4), were investigated. Also, the data were analyzed by means of the equation

$$\left(\frac{1}{1+R}\right)\ln\left(\frac{R+f}{R(1-f)}\right) = Z\varepsilon \tag{1}$$

earlier advanced to describe the overall kinetics of this type of martensite transformation⁽⁵⁾. In eq. (1), f is the volume fraction of martensite, at plastic strain ε while R is a parameter related to the density nucleation sites. The parameter Z depends both upon the energetics of nucleation and upon the product pv, wherein p is the autocatalytic factor, and v the average volume of the martensite features.

II. Experimental Methods

The routine followed in the preparation of the Fe-31%Ni-0.1%C specimens from the original stock is described elsewhere(3) and will not be repeated here for the sake of brevity. Shock-loading was accomplished by parallel flyer-plate (copper) impact upon the system, yielding a pressure of 8 GPa and a peak duration of 2 µs. Grain refining was obtained by suitable annealing. Deformationinduced martensite was produced by tensile deforming flat specimens with reduced gage dimensions $(27 \times 4 \times 1 \text{ mm})$ in an Instron TT-DM machine at a nominal strain rate of 10⁻⁴ s⁻¹. A special fixture was used to allow the specimens to be deformed while being soaked into refrigerated alcohol baths. A copper-constantan thermocouple, kept as close as possible to the specimens during deformation, indicated that their temperature would have remained within 2 K of the nominal test temperatures. Mechanical data were ob-

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tained from the Instron chart. The volume fraction of martensite in the different specimens was determined by the method of point-counting; three mutually perpendicular sections of each specimen were observed. An iterative computer program was used to determine the values of R and Z which would result in the "best fit" of the data with eq. (1). The "best fit" criterion adopted herein was to minimize the area between the theoretical line and the polygonon determined by the data points.

The smaller grain size specimens had a total surface area per unit volume (S_v) of 29.6 mm⁻¹ measured by the method of the intercepts. The larger grain size set was characterized by $S_v = 14.2 \text{ mm}^{-1}$.

Transmission electron microscopy observations were performed in a JEOL 100-B microscope operating at 100 kV. Thin foils were obtained by electrolytic polishing small discs in a alcohol/perchloric acid solution.

III. Experimental Results and Discussion

Figure 1 shows the variation in yield strength of the alloy with temperature for the three different initial conditions. Below M_s^{σ} the yielding was initiated by "stress-assisted" martensite transformation. Above M_s^{σ} , slip was the initial plastic deformation mode; the martensite only formed after yielding and is termed "strain-induced" as suggested in ref. (4). At 248 K both the coarse and the

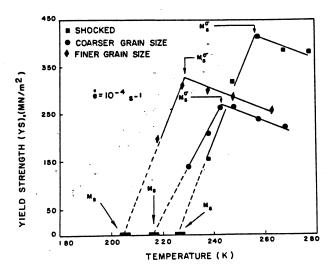


Fig. 1 Variation in yield strength of the material with temperature (from ref. 3).

fine grain alloys exibit "strain-induced" transformation. Thus, the effects of grain refining and shock-loading might be compared. The temperatures of 238 K and 220 K were selected in order to obtain "stress-assisted" transformation in the two recrystallized materials at the same initial flow stress (200 MN/m²).

The kinetics data obtained at the different temperatures and initial conditions are depicted in Fig. 2. Attention is called to the origins of some of the plots which were shifted along the the horizontal axis for a better display of the data. The most obvious observation which follows from Fig. 2 is that both the initial rate and the over-all transformation is less in finer grained material under "strain-induced" conditions (at 248 K). This could be a consequence of the martensite plates being naturally smaller in the finer grained alloy.

The reaction kinetics under "stress-assisted" conditions is different. The reaction starts out much faster and the matrix grain size does not appear to be very critical. However, this may be only apparently so because the specimens were at different temperatures. A higher driving force could overcome the opposing effects of a smaller austenite grain size (or larger S_v). On the other hand, a high rate, $df/d\varepsilon$ very early in the process, should be expected since it is the transformation which

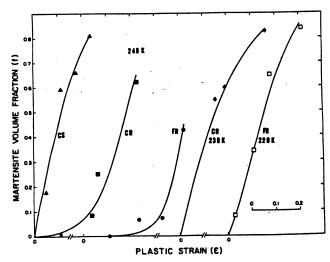


Fig. 2 Kinetics Data. The plots show the volume fraction of martensite as function of strain. The data obtained with finer and coarser grained material are marked RF and CR respectively. The set of points characteristic of the coarse grained and shock-loaded specimens are signaled by CS.

determines the initial flow of the material.

It is now appropriate to consider the effects of a previous shock-loading on the reaction kinetics comparatively with that of grain refinement and temperature. The data (filled triangles) indicate that both the initial and the overall reaction rate are higher in the shocked material than in the other types of specimens deformed at the same temperature (248 K). The trend is typical of "stress-assisted" transformation however the annealed material can only undergo strain-induced transformation at 248 K. This is in agreement with the data of ref. (3) as well as it suggests that the substructure provided by the shock-wave indeed renders the matrix highly unstable toward martensite. Transmission electron microscopy of this material did not, unfortunately, provide any definitive evidence for the presence of peculiar defect arrangements which could be linked to martensite nucleation. The observed substructure, which can be described as a high density of dislocations wherein incipient cell formation is obvious, Photo. 1, is typical of high stacking fault materials subjected to a medium pressure shock-wave (8 GPa at 2 μs pulse duration, in the present case).

The ability of eq. (1) to describe the data may be evaluated by observing the lines drawn through the experimental points in Fig. 2. They represent the best-fit lines obtained in the form earlier described. The values attributed to the parameters R and Z are listed in Table 1.

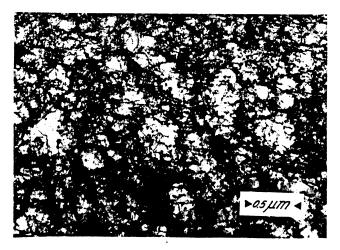


Photo. 1 Transmission Electron Micrograph. Typical substructure observed in the shocked material. The beam is along the shock wave direction.

Table 1 Values of R and Z.

Specimen type	Deformation temperature	R	Z
Coarser Grain (CR)	248 K	4×10 ⁻³	14.6
Finer Grain (FR)	248 K	9×10^{-5}	21.6
Shocked (CS)	248 K	9×10^{-1}	5.3
Coarser Grain (CR)	238 K	3.0	1.4
Finer Grain (FR)	220 K	4×10 ⁻¹	7.1

For the reasons which were discussed in ref. (5) only the variation of R will be considered. By definition, R is equal to ratio of the concentration of pre-existent preferred nucleation sites to the autocatalytic factor, R=n/p. The figures in Table 1 indicate that R increases with decreasing temperature (or austenite stability). This trend which has also been observed in the case of stainless-steels(5) suggests that either "p" becomes smaller or the number of pre-existent sites for nucleation increases with the driving force. On the other hand, "R" was found to decrease with grain size which is opposition to the behavior of stainless-steels⁽⁵⁾. Although this difference may be attributed to differences in the nucleation mechanism, it is duly recognized that it could result from some inadequacy of the kinetics model. This is so, because in the derivation of eq. (1) it was assumed that the variation of martensite nucleation sites with the fraction transformed would be a "smooth" function. However the transformation of Fe-31% Ni-0.1 %C during deformation is signalled by sonic emission and simultaneous load drops. These phenomena occur more obviously in the coarser grain material and at the lower temperatures. Thus it appears that the reaction in Fe-31% Ni-0.1%C would be occurring by a sequence of bursts, i.e., discontinuously. It is not clear, at present, if the usual description of autocatalysis by means of the parameter "p"(6) would properly account for such a situation. This is a valid issue since autocatalysis in a burst is believed to be, by and large, caused by a cooperative coupling of the shape strain of the plates rather than by some "seeding" of the matrix with "embryos". The latter may, however, be the dominant process in a small grained austenite

wherein burst activity is minimum.

IV. Conclusions

Experimental data are presented which support the contention that refining the austenite grain results in its stabilization. An initially sluggish deformation-induced transformation would be a direct consequence of it. The sensitization of the austenite by a prior shock loading treatment was also substantiated, i.e., shocked austenite was found to transform faster.

Equation (1) can be considered to describe reasonably well the reaction kinetics in Fe-31% Ni-0.1%C. Its use allowed the discovery of a difference between the behavior of the alloy studied and stainless-steels. Although this could be taken as a consequence of differences in the transformation mechanism, a critical consideration of the issue brought-in the possibility that the occurrence of bursts may have influenced the results of the analysis based upon eq. (1), particulary in the case of the coarser grain material. Further development is required to decide the adequacy of

available kinetics models in accounting for such a "discontinuous" transformation mode.

Acknowledgments

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REFERENCES

- (1) M. Umemoto and W. S. Owen: Met. Trans., 5 (1974), 2041.
- (2) J. R. C. Guimarães and J. C. Shyne: Scripta Met., 4 (1970), 1019.
- (3) J. R. C. Guimarães, J. C. Gomes and M. A. Meyers: Proceedings of the First Intl. Symposium of The Japan Institute of Metals, Kobe (1976), Suppl. to Trans. JIM, 17 (1976), p. 411.
- (4) G. B. Olson and M. Cohen: J. Less-Comm. Metals, 28 (1972), 107.
- (5) J. R. C. Guimarães: Scripta Met., 10 (1976), 223.
- (6) V. G. Raghavan and A. R. Entwisle: The Iron the Steel Institute Special Report, no. 93, London (1965), p. 30.