An aspect of the nucleation of burst martensite

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In steels where the martensite transformation occurs in a well-behaved and progressive manner, it has been experimentally established that the number of pre-existing nuclei that develop into new plates of martensite (per unit volume of austenite) on undercooling below the martensite transformation temperature, M_s , is directly related to the corresponding increase in transformation driving force. Such a relationship cannot, however, be studied in steels which transform by a "burst" mechanism, since the role of the initial distribution of nuclei is swamped by the creation of numerous new (autocatalytic) nuclei. In the present work, an experiment designed to overcome this difficulty by suppressing the growth of autocatalytically generated nuclei is conducted, thus enabling the verification of the above relationship for a classic Fe–Ni–C burst-transformation alloy.

1. Introduction

The progress of the martensite transformation as a function of undercooling below the martensitestart temperature, M_s , depends critically on the potency distribution of nucleation sites, and can be approximately expressed [1] by a relationship of the form

$$(1-f) = \exp\left\{\bar{v}\Phi \frac{\mathrm{d}(\Delta G)}{\mathrm{d}T}(M_{\mathrm{s}}-T_{\mathrm{q}})\right\},$$
 (1)

where f is the volume-fraction of martensite, T_q is the quench temperature, \bar{v} is the average volume per new plate of martensite (considered [1] to be independent of f), Φ is the proportionality constant, ΔG is the volume free-energy change accompanying transformation and T is the temperature.

The origin of the constant Φ lies in the assumption that the increase in the number of new plates of martensite, dN, per unit volume of austenite, accompanying a drop in T_q is directly related to the corresponding change in ΔG , so that

$$\mathrm{d}N = -\Phi\mathrm{d}(\Delta G). \tag{2}$$

We note that the corresponding increase in the number of martensite plates when referred to a unit volume of sample is $dN_v = (1 - f)dN$.

Martensitic transformations in several steels have been observed [1-3] to empirically obey a relationship of the form of Equation 1, so that the assumptions mentioned above seem reasonable. However, it is usually only possible to study the variation of f against T_q for well-behaved martensite reactions; obvious difficulties arise when the transformation occurs in a series of "bursts", so that large volumes of austenite suddenly change into martensite. Bursts of this kind are a result of autocatalytic nucleation [1, 4] in which the role of the initial distribution of nuclei is swamped by the creation of numerous new nuclei, whose origins can probably be attributed to the plastic deformation effects that accompany the growth of martensite. These new nuclei grow such that they form into plates which accomodate the strains resulting from the formation of the first plate (which gave rise to the autocatalytic nuclei), and a repetition of this basic process triggers the sudden zig-zag propagation of groups of mutuallyaccommodating plates (i.e., the burst occurs).

2. Experimental procedure

The present work is concerned with an attempt to verify Equation 2 for an alloy of Fe-28.0 wt%

Ni-0.41 wt % C, which normally undergoes the classic burst transformation; an experiment was designed to suppress any contribution to the transformation resulting from autocatalytically generated nuclei.

The alloy was prepared from high-purity constituents as a 65 g melt in an argon-arc furnace, and was homogenized at 1300° C for a period of two days. All high-temperature heat-treatments were carried out with the specimen sealed in a quartz tube containing a partial pressure of pure argon. After air-cooling to room temperature, the ingot was plunged into liquid nitrogen and then re-austenitized at 1000° C for 30 min, after which the specimen was machined into a form suitable for tensile loading (discussed later). After machining was complete, the specimen was given another anneal at 1000° C for 30 min, and naturally cooled to ambient temperature to give a fully austenitic specimen with an M_s of about $- 80^\circ$ C.

The design of the experiment is based on the following principles:

(a) The driving force for transformation can be provided either by changing the temperature or by altering the state of stress of the austenite. It is well known that the application of a stress, σ , raises the temperature at which martensite first forms to a maximum temperature $M_{\rm D}$. More significantly, the formation of those crystallographic variants of martensite which best accommodate the applied stress is always favoured. It was therefore considered that transformation under stress at a temperature well above $M_{\rm s}$ could suppress the onset of martensite bursts, whose propagation requires the growth of at least two mutually-accommodating variants, which cannot both be expected to comply favourably with the applied stress. Hence, even if the formation of a particular favoured variant results in the generation of autocatalytic nuclei, the latter would not be expected to grow because of the constraint provided by the applied stress. It should then be possible to study the nature of pre-existing nucleation sites, even in burst transformation alloys.

(b) For transformation under the influence of an applied stress, it is useful to distinguish between stress-assisted and strain-induced nucleation [5]. The former refers to nucleation at the same sites which trigger spontaneous transformation on cooling, while the latter refers to the production of new nucleation sites by plastic deformation. These regimes may be broadly separated by the temperature M_s^{σ} (where $M_D > M_s^{\sigma} > M_s$) below which applied elastic stresses assist the spontaneous growth of pre-existing nuclei. For the present work it is clearly necessary to operate below M_s^{σ} .

Temperatures such as M_s^{σ} are difficult to establish, so that the maximum stress that should be used was not clear. Hence, a tapered (circular section) tensile specimen (see Fig. 1) was prepared so that a single test would generate all the necessary information on f against σ , since the stress level would vary along the length of the specimen. The angle of taper was deliberately kept small in order to keep the stress system as simple, and as near to a conventional tensile test, as possible. The specimen was pulled in an Instron tensile testing machine with a cross-head speed of 25 mm min⁻¹,



Figure 1 Graph illustrating the variation of volume-fraction of martensite with applied stress. The inset shows the sample geometry.

at a temperature of -44° C, until failure occurred in the thin parallel section. It should be noted that at the temperature of the test, the chemical contribution to the driving force for transformation amounts to $-920 \,\mathrm{J}\,\mathrm{mol}^{-1}$ (as estimated from the data in [6, 7]); this is sufficient to fully account for the stored energy (~ 700 $\mathrm{J}\,\mathrm{mol}^{-1}$, in [8]) of martensite, so that the applied stress truly serves to provide the additional push necessary to stimulate nuclei into growth.

After tensile testing, the specimen was sliced longitudinally along its centre-line and prepared for quantitative metallographic examination. The volume-fraction of martensite was determined at a magnification of 850, on a Quantimet image analysing computer, beginning at the point where the thin parallel section gave way to the tapered part of the specimen. Each determination was carried out for a region extending 0.5 mm along the length of the specimen, so that the stress calculated for each 0.5 mm section really refers to the centre of each of these areas. A special stage allowed automatic specimen movement of the required amount. The results of the analysis are plotted in Fig. 1, and optical micrographs of the tensile specimen are shown in Fig. 2.

Fig. 2 illustrates that the experiment has been largely successful in suppressing the burst effect, although some mutually-accommodating plate sequences can be seen in the high-stress regions. Furthermore, the martensite that has formed seems to comply with the applied stress; the traces of the martensite habit planes are inclined approximately symmetrically about the tensile axis, corresponding to the macroscopic planes of maximum shear stress at 45° to the tensile axis.

Actually, exact compliance with the latter cannot be expected, since both the shear and dilatational components of the martensite shape strain interact with the applied stress [9], although the former should dominate. In addition, the austenite grains are randomly oriented in space and it is remarkable that, despite all these limitations, Fig. 2 clearly depicts the influence of the applied stress. This probably reflects the fact that at least one of the 24 martensite variants which can form in any austenite grain is likely to be suitably oriented with respect to the stress.

Referring back to Fig. 1, it seems that σ and f are linearly related, over the whole range of measurements (f = 0 to 0.7). From Equation 2, it can be shown [1] that

$$df = -\phi \bar{v}(1-f) d(\Delta G)$$
(3)

and, for $f \ll 1$,

$$\mathrm{d}f \simeq -\phi \bar{v} \,\mathrm{d}(\Delta G). \tag{4}$$

Since σ and ΔG are directly related, f can be expected to be proportional to σ at low volumefractions of transformation. However, the fact that the proportionality extends to high values of f indicates that a greater than expected amount of transformation has been obtained in the high σ region. This is probably due to failure to completely suppress the burst phenomenon, as discussed earlier. A plot of df/(1-f) against σ was found to show the same effect again, and is therefore not included in this paper.

In summary, burst martensite does seem to follow the relationship indicated in Equation 2, and a simple method has been designed to facilitate the study of such transformations.



Figure 2 Optical micrographs illustrating stress-induced martensite transformation in regions of (a) low stress and (b) high stress. The small arrows mark the regions where some autocatalytic transformation has occurred.

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