

Letters

The influence of prior deformation on the yield stress of a metastable Fe–Ni–C austenite

The effects of deformation on the martensite transformation have been the subject of many studies. In 1932, Scheill [1] proposed that slip and transformation could be competitive modes for the flow of austenite. Later, Patel and Cohen [2] showed that elastic straining could raise the temperature for the spontaneous transformation of austenite into martensite, the M_s temperature. Richman and Bolling [3] studied several aspects of the mechanical behaviour of metastable austenites and their results supported Scheill's ideas; two distinct regimes were identified. At lower temperatures the yield stress increased with temperature, while at the higher temperatures the usual decreasing yield stress with increasing temperature was observed. More recently, Olson and Cohen [4] have used the temperature-dependence of the yield stress to rationalize the deformation–transformation behaviour of metastable austenites. This communication describes some results of a study whose objective was to understand the roles of the stress and strain on deformation-induced martensitic transformation of austenite which develops at intermediate temperatures ('strain-induced' according to the Olson and Cohen criterion). The basic experiment was to determine the mechanical response of a predeformed austenite to a subsequent reloading at different levels of stability. Since the effects of elastic straining have been successfully treated by Patel and Cohen [2] the results should be readily interpreted. The predeformation which was introduced may be considered to simulate a 'thermo-mechanical' treatment similar to those used in processing the complex TRIP steels [5].

Tensile specimens with useful gauge dimensions of 27 mm × 4 mm × 1 mm were machined from flat 1 mm thick strips of an Fe–31% Ni–0.1% alloy* and given a final annealing at 1373 K for 2 hours while sealed inside fused quartz tubes. This procedure resulted in essentially equiaxed grained austenitic specimens with total internal surface

area per unit volume, $S_v = 14.2 \pm 0.5 \text{ mm}^{-1}$, determined by the method of the intercepts. Tensile tests were conducted in a screw-driven machine at a nominal strain rate of 10^{-4} sec^{-1} at temperatures ranging from 208 to 298 K. The M_s temperature (which coincided with the burst temperature, M_b , in this alloy) was always determined using unloaded specimens, the transformation being detected by observing the development of tilts on a pre-polished surface of the sample or by monitoring the sample temperature, while cooling it in a bath kept at a fixed temperature. Both methods yielded results in good agreement within the scatter typical of either M_s or M_b determinations.

In establishing the yield stress of the material the following criteria was used: the occurrence of a load drop within the elastic regime was taken to be indicative of flow; in the absence of such a development (attributed to transformation) the yield stress was taken as that at which departure from the initial linear stress–strain behaviour became obvious.

The data relevant to the forthcoming discussion are depicted in Fig. 1. Attention is called to the origin of the right hand plot which is displaced along the horizontal axis. It is used to allow a better display of the data. The filled circles show the data obtained with annealed specimens. The behaviour is typical of metastable austenites and may be rationalized by assuming that both transformation and slip are competitive modes of deformation. Below some critical temperature, M_s^0 , the yield stress decreases with temperature (because the transformation provides plasticity) and should become zero at M_s . This possibility is supported by a linear extrapolation of the data, as indicated by the dashed line in the figure.

The behaviour of the material given predeformation is not so simple. These data are represented by the filled triangles in Fig. 1. They were obtained by reloading, at the indicated temperatures, specimens given 25% deformation (600 MN m^{-2}) at room temperature. This initial deformation does not induce transformation (discernible by optical metallography). However it raises

*By weight.

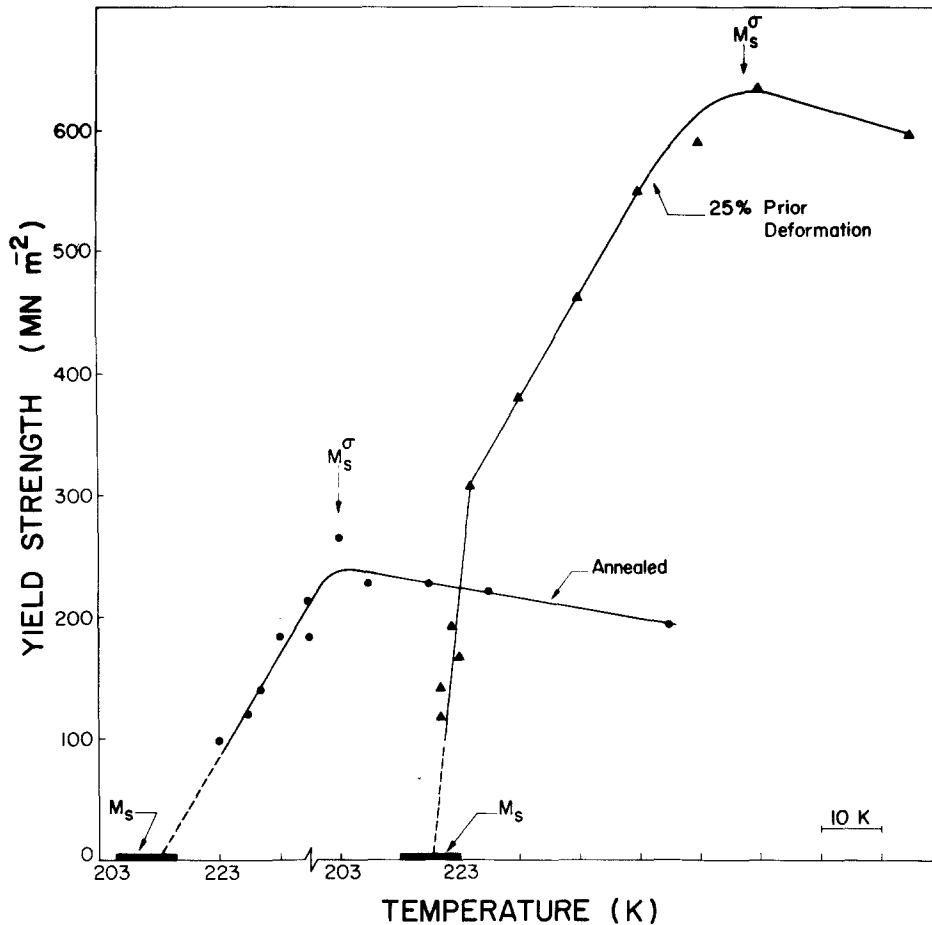


Figure 1 The variation of the yield strength of the alloy with the temperature of deformation. Notice the origin of the right hand plot displaced along the horizontal axis; the scale is not split for the curve on the left. Below M_s^σ , the yield stress values were determined from the bursts in the elastic regime, while those above M_s^σ correspond to the deviations of the stress-strain curve from linearity. Near M_s^σ the two results merge.

the M_s temperature of the alloy as well as makes it more reproducible [6]. Testing specimens 15% pre-deformed (475 MN m^{-2}) yielded a similar set of data. The data is not shown here for it would not add to the interpretation of the overall results but it is mentioned because it suggests that the behaviour depicted in Fig. 1 is typical.

The critical discovery from the study of pre-deformed specimens was the existence of a second regime of positive dependence of the yield stress. In the case of the alloy given 25% pre-deformation the yield stress below M_s^σ , begins to decrease smoothly and reaches a rate ($8.6 \text{ MN m}^{-2} \text{ K}$) about the same as that observed with annealed material ($8.1 \text{ MN m}^{-2} \text{ K}$). However, closer to the temperature for spontaneous transformation the stress falls toward zero at a much faster rate ($32 \text{ MN m}^{-2} \text{ K}$).

From Patel and Cohen's treatment one may write

$$\frac{d\sigma}{dM_s} = \frac{\Delta S}{\epsilon}, \quad (1)$$

where σ is the applied stress, ΔS the entropy change from the transformation and ϵ the shape strain of a martensite plate. Bolling and Richman [3] found that below M_s^σ (flow initiated by transformation) the yield stress of austenite displays a positive temperature dependence with a slope, $d\sigma/dT$, inversely proportional to ϵ , concluding from this that the Patel and Cohen [2] theory could be apparently extended to dynamic testing conditions by identifying σ with the yield stress and M_s with the testing temperature in Equation 1. This means that a steeper slope, $d\sigma/dT$, in such a plot could be

attributable to a reduction in ϵ by self-accommodating effects in martensite formation.

In order to recognize the feasibility of this proposition it is important to notice that Patel and Cohen considered the influence of an external stress on the formation of a single plate of martensite. Consequently, in order to apply their rationale to dynamic testing it must be assumed that the material will yield by the formation of one martensite plate. Then ϵ may be computed by considering it to be made up of two strains: a shear in the habit plane plus a distortion normal to it. However, if the yielding by transformation is caused by a large burst, the plates will form in a self-accommodating manner and the shear component of ϵ may average out. It amounts to a smaller value of ϵ . Thus, $d\sigma/dT$ should have larger values closer to M_b , as is shown by the data for the prior deformed condition at the lowest temperatures. The size of the burst also appears to be important, and this is so because the steeper slow only developed very close to M_b . However, sonic emission ('clicks') typical of martensite bursts always signalled the onset of yielding (a load drop) within the other regime of 'stress-assisted' transformation, immediately below M_s^0 . Consideration that the burst-size is related to the transformation driving force [7] leads to the conclusion that some critical temperature (or driving force magnitude) must be reached before a burst becomes large enough to average-out the shear portion of the transformation strain.

It remains to discuss why the same behaviour was not observed with the annealed condition. We believe that it would be observed if the experiment were properly done close to the M_b temperature condition. Unfortunately this could not be accomplished due to experimental limitations. The burst-controlled regime occurs under too small a load for so that the local deformation in the gripped ends of the specimens may interfere with the outcome. Also the transformation temperature of annealed specimens is not as reproducible as in pre-deformed ones. On the other hand, it is clear from Patel and Cohen's results (determination of M_s under static loads) that a second stage could be assigned in the case of their alloy deformed in tension. This is shown by the dashed portion of the upper plot of their Fig. 1. Consequently, it is pro-

posed that the ϵ needed to interpret such data should be considered an 'effective strain'. Furthermore, it follows from this discussion that not only the nucleation of martensite specifically, but also the transformation mode (i.e. burst versus slow transformation), are relevant for determining the temperature dependence of the yield stress of metastable austenites.

Finally a word should be said about an apparent discrepancy between the results herein discussed and the data obtained with shocked specimens [8]. The latter did not depict the two-stage behaviour. This may possibly be due to differences in the substructure, or more probably, it results merely from a lack of enough data points to show a clear 'knee' in the curve. In fact, the observation that the slope of the line drawn through those experimental points is sensibly higher (about 2X) than obtained with annealed specimens supports this hypothesis.

These experimental observations suggest that the temperature-dependence of the flow stress of metastable austenites is affected both by martensite nucleation and the reaction mode. It is proposed that under conditions which lead to large bursts the transformation strain, in the formalism of Patel and Cohen, should be replaced by some effective strain related to the total transformation volume change for the burst, since the shear component of the shape strain of the individual plates are averaged out due to accommodation effects.

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Direct X-ray observation of lattice parameter changes due to magnetostriction in nickel single crystal

Till now the measurements of magnetostriction in single crystal or polycrystalline ferromagnetic materials have been performed by electrical (capacity or resistivity variations, strain gauges), mechanical or optical methods [1, 2], i.e. through an evaluation of the macroscopic effect of dimensional alteration produced in a suitably shaped specimen by the applied magnetic field.

Since a tested and reliable X-ray diffractometric technique has recently become available, which allows the measuring of lattice parameters in single crystals with a precision at least one order of magnitude better than that attainable by the previous most sophisticated powder procedures (up to ten years ago the best tool for the purpose), we thought of trying to analyse magnetostrictive effects on a microscopic scale (changes in the unit cell dimensions) in this way.

The instrument employed was the APEX automatic precision X-ray goniometer, designed and manufactured in the UK [3] which, through the Bond's method of symmetric equivalent reflections [4], allows one to obtain in standard working conditions a precision of about 2 ppm in the measurement of Bragg angles relative to single crystal reflections.

The examined material was a high-purity (99.995%) nickel single crystal of cylindrical shape (produced by Materials Research Corporation, Orangeburg, N.Y. 10962, USA), Czochralski-grown along the [110] direction. We selected $\text{CrK}\alpha_1$ X-radiation, with $\lambda = 2.28976 \pm 0.00002 \text{ \AA}$ (this value derived from Bearden [5] and modified according to Deslattes and Henins [6]), because

among the commonly used wavelengths it provides the highest Bragg angle pertaining to reflections from crystallographic planes parallel to the flat surface of the sample, namely about 66.78° for Ni (220), and also made allowance for the three fundamental cubic orientations.

The side to be studied was carefully polished by mechanical and chemical treatments, to remove any surface damage. Employing a cylindrical X-ray beam (generated by a fine-focus tube working at 30 kV and 30 mA) with a collimator diameter of 0.5 mm, becoming 0.8 mm on the crystal owing to the divergence, we performed several Bragg angle measurements for the (220) reflection in different points of the crystal surface fitted on a two-dimensional scanner (see Fig. 1), at first without a magnetic field and afterwards laying a suitable permanent magnet in contact with the opposite side of the crystal (local value of $H \approx 300 \text{ Oe}$), and averaging five readings in each position.

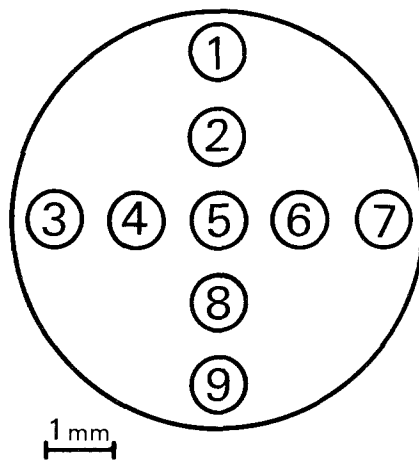


Figure 1 Schematic disposition on the Ni (110) crystal surface of different X-ray measurement points.