A microstructural study of a melt-spun ultra high-strength alloy steel

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Melt spinning in a controlled atmosphere has been used to produce ribbons of an ultra high-strength alloy steel. The microstructure of these ribbons has been investigated using both optical and transmission electron microscopy. Comparison of the microstructure with that produced by conventional solid-state quenching shows that the martensitic structure is refined in the melt-spun ribbon and the formation of alloy carbides suppressed. These factors lead to a considerable increase in hardness of the melt-spun ribbon with respect to that of the solid-state quenched material. The occurrence of interlath austenite is discussed in terms of the crystallography of the martensite transformation.

1. Introduction

In recent years, a large number of investigations has been reported concerning the structure and properties of rapidly quenched crystalline metals and alloys, since improvements in strength, toughness and wear resistance are often achieved. These improvements arise from several phenomena including refinement of the microstructure, increased terminal solid solubilities and the production of metastable phases [1]. In the present investigation, an ultra high-strength steel has been rapidly quenched by melt spinning to examine the effects of rapid quenching on microstructure which is compared with the microstructure produced by conventional solid-state quenching. In particular, this work studies the refinement of the microstructure produced in the rapidly quenched material. The martensitic lattice parameters have been used as an indication of the supersaturation of the matrix, and the precipitation of carbides has also been studied. The micro-hardness of both solid-state quenched and rapidly quenched steels is discussed in relation to the super-saturation of the matrix and the refinement of the microstructure. Observations of the retention of austenite in both solid-state quenched and rapidly quenched

material are discussed in terms of the crystallography of the martensitic transformation.

2. Experimental details

The alloy used in this investigation had the following composition (wt %):

0.38 C, 0.55 Mn, 2.9 Ni, 0.7 Cr, 0.57 Mo, 0.23 Si, 0.2 V, balance Fe.

Rapid quenching was achieved by melt-spinning [2, 3] in a helium gas atmosphere which minimized gas pick-up in the liquid alloy and reduced subsequent internal porosity. The alloy was melted in a radio frequency induction coil and ejected by argon gas pressure onto a rotating copper wheel (circumferential speed ~ 13.5 m sec⁻¹). The resultant ribbons were approximately 2.5 mm wide and $55\,\mu\text{m}$ thick. Using the heat-transfer data of Ruhl [4] it was estimated that the average cooling rate during solidification was 10^5 to 10^6 K sec⁻¹. Solid-state quenched material was produced from 1 mm thick strip which was austenitized at 1473 K for 1 h and quenched into iced brine.

Specimens for light microscopy were prepared from both materials by mechanically polishing to $0.25\,\mu\text{m}$ using diamond lapping compounds



Figure 1 Optical micrographs of rapidly quenched steel (a) and solid-state quenched steel (b).

prior to etching in 2% nital. Thin foil specimens for transmission electron microscopy were ionbeamed thinned and examined in Philips EM300 and EM400 electron microscopes operating at 100 and 120 kV, respectively.

Lattice parameter measurements were obtained by X-ray diffractometry using $CoK\alpha$ radiation. Hardness values were measured using a Leitz micro-hardness tester with a 0.1 kg load.

3. Results

3.1. The melt-spun ribbon

Fig. 1a is a light optical micrograph of the meltspun specimen material. At this magnification, it is difficult to determine the exact nature of the martensitic reaction product although it appears to consist of plates ~ 1 to $3 \,\mu$ m wide together with a fine unresolved structure. However, the austenite grain size is much reduced compared with the solid-state quenched material (vid. Fig. 1b).

Transmission electron microscopy confirmed the tentative conclusions based on the light optical micrographs that the melt-spun microstructure was found to consist of both internally twinned martensite plates and highly dislocated martensite laths. Fig. 2 is a centred dark-field micrograph of internal twins in a large martensite plate and is typical of many such observations. The twins are seen to be very fine ($\sim 10 \text{ nm width}$) and are closely spaced. Figs 3a and b are bright-field micrographs of the lath martensite structures observed in the melt-spun ribbon. The laths are highly dislocated and are 20-150 nm wide. Figs 4a and b are a bright-field/dark-field pair of the lath martensite and illustrate the fact that alternate laths were often of the same orientation. Fig. 5 is the selected-area diffraction pattern from the area shown in Fig. 4 confirming the existence of two martensite orientations. Crystallographic analysis of this diffraction pattern showed that adjacent laths were twin related. This was frequently observed, a point which is returned to in Section 4.

Retained austenite was detected as an interlath film and Fig. 6a shows a 220 austenite centred dark-field micrograph with the corresponding diffraction pattern (Fig. 6b). The austenite was found to be fragmentary and was present to a much smaller degree than that found in pure laboratory made steels, e.g. Fe-Cr-C alloys [5] and Fe-V-C alloys [6]. A further example of the discontinuous nature of the retained austenite films is given in Fig. 7a which shows that austenite is not uniformly retained between the laths. Where sufficient diffracted intensity from the austenite was detected, it was possible to define



Figure 2 A centred dark-field electron micrograph of internal twins in a martensite plate, in the melt-spun steel.



Figure 3 Two bright-field electron micrographs of lath martensite structures, in the melt-spun steel. Numbers show different laths.

the austenite/martensite crystallography. Fig. 7b is the selected-area diffraction pattern from Fig. 7a from which can be shown that:

$$\begin{array}{l} (1\overline{1}1)_{\gamma} \parallel (110)_{\alpha'} \\ [\overline{1}01]_{\gamma} \parallel [\overline{1}11]_{\alpha'}, \end{array}$$

i.e. the austenite and martensite are related by the Kurdjumov-Sachs [7] orientation relationship*.

Autotempering was found to be virtually eliminated in the melt-spun ribbon, i.e. carbide

precipitation was rarely observed in the martensite structure.

3.2. The solid-state quenched material

In contrast to the melt-spun ribbon, the structure of the solid-state quenched material was found to be comprised almost entirely of lath martensite. Fig. 8 is a typical bright-field micrograph of a martenistic area and shows laths ~ 0.1 to 0.5 μ m in width together with a large autotempered lath ~ 5 μ m wide. It was found that many of



Figure 4 A bright-field (a)/dark-field (b) pair of the lath maretnesite illustrating that alternate laths were in the same orientation.

*This relationship is frequently found in solid-state quenched steels (e.g. [5, 6]).



Figure 5 The selected-area diffraction pattern of the area shown in Fig. 4, confirms the existence of two martensite orientations.

the laths contained a dispersion of cementite. Retained austenite was observed as an interlath film, but again was fragmentary in nature. No evidence for the formation of plate martensite was found although some martensite laths contained internal twins. In common with the meltspun material, adjacent laths were often found to be twin related as shown in Fig. 9. In this instance, the martensite centred dark-field micrograph from lath A has illuminated the isolated internal twins in lath B.

4. Discussion

The melt-spun material is characterized by a smaller austenite grain size (*vid.* Figs 1a and b) which results in a finer martensite lath structure (cf. Fig. 8 with Fig. 3) than that of the solid-state quenched material. Similarly, internally twinned plate martensite was found in the rapidly quenched material

TABLE I

	Average lattice parameter (nm)
Melt-spun	0.287 42 +/- 0.000 14
Solid-state quenched	0.286 90 +/- 0.000 12

and not in the solid-state quenched samples. At present, it is difficult to explain this result. However, the suppression of the Ms temperature attendant on rapid quenching [8] and the fine austenite grain size could promote the development of plate martensite.

The observation of a lack of autotempering in the melt-spun steel can be rationalized in terms of the rapid cooling rate inhibiting carbide nucleation. This inhibition leads to a greater supersaturation in the melt-spun material than that found in the solid-state material, as shown by measurements of the average lattice parameters^{*} of martensite in both materials, as shown in Table I.

The increase in supersaturation, together with the refined microstructure also lead to a large increase in the microhardness of the melt-spun ribbon (800 VPN) compared with 460 VPN for the solid-state quenched material.

Retained austenite was found in both materials but was observed to be fragmentary, and was present in a very low volume fraction. A similar result was found by Bhadeshia and Edmonds [9] in their work on an Fe-4Ni-0.4C alloy, where it was observed that adjacent laths were twin related (in contrast to the Fe-Cr-C and Fe-V-C alloys [5, 6] where adjacent laths tend to be low-angle related). It has, therefore, been suggested [9] that austenite, which can be mechanically stabilized between low-angle related laths, will



Figure 6 A 220 austenite centred dark-field electron micrograph (a) with its corresponding diffraction pattern (b).

*These values were produced from the X-ray diffractometer scans by calculating the interplanar spacings and the Nelson-Riley-Taylor-Sinclair function followed by a weighted least squares extrapolation to $\theta = 90^{\circ}$.



Figure 7 Another example of retained austenite found: (a) melt-spun steel, (b) with its corresponding selected-area diffraction pattern.

be unstable between twin related laths. This follows from the fact that twin units shear in opposite directions, so that they can form in a mutually self-accommodating manner. In contrast, the shear accompanying the formation of similarly oriented laths will be additive and promote the stress stabilization of the interlath austenite.

5. Conclusions

Comparison of rapid quenching of an ultra highstrength alloy with conventionally quenched steel leads to:

(1) a refinement in both the austenite grain size and average martensite lath width;

(2) an increase in the supersaturation of the martensite matrix and a consequent increase in the lattice parameters;

(3) a consequent increase in hardness from 460 VPN to 800 VPN.

For both specimen materials, the preponderance



Figure δ A bright-field electron micrograph of solid-state quenched steel, showing martensite laths together with a large autotempered lath.



Figure 9 The martensite centred dark-field micrograph of lath A, illuminating the internal twins in lath B.

of twin related laths suppressed the retention of interlath austenite.

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