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Synchrotron analysis of toughness anomalies in nanostructured bainite

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ABSTRACT

High-resolution synchrotron X-ray diffraction has been used to characterise the notch root regions of Charpy impact test specimens of a superbainitic steel, both before and after loading. The changes in the volume fraction of austenite induced by the application of a three-point-bending load were quantified. Analysis of diffraction peak shifts revealed the extent of residual tensile and compressive strains present due to both machining and an applied load. The results lend support to the hypothesis that the comparatively low energies absorbed during Charpy impact testing of superbainitic steels, <7 J, are due to the formation of stress-induced martensite at the notch root, prior to crack initiation.

1. Introduction

Nanostructured bainitic steels, comprised of <10 nm-thick bainitic sheaves distributed within carbon-enriched austenite, have some exceptional properties with yield strengths of up to 1500 MPa and elongations in excess of 20% [1]. Critically, these properties can be achieved in thick sections following relatively low-temperature heat treatments, without the need for rapid cooling or severe deformation [2–4]. The benefits offered by these alloys are such that they are now produced commercially.

However, it has been asserted that the Charpy impact toughness of these nanostructured steels is unexpectedly small when compared with their fracture toughness, K_{1C} [1]. For alloys of almost identical chemical composition and heat treatment, the Charpy energy at room temperature is reported to be only 4–7 J whereas K_{1C} has been measured in the range 28–32 MPa $m^{1/2}$ [5,6]. However, it remains the case that most of the data published do not contain both measures of toughness on the same alloy [5,7–12]. In general, when comparisons are made across nanostructured bainite of different compositions, the range of Charpy energies remains within 4–7 J, whereas the fracture toughness ranges from 28 to 55 MPa $m^{1/2}$. Recently, a direct comparison has been made in a fast-transforming superbainitic steel, which again showed Charpy energies and fracture toughnesses in these ranges [13]. This apparent disparity may be a result of the different sample geometries required by the two tests; in a Charpy test a blunt notch is used, whereas a K_{1C} specimen has a sharp crack. During an impact test, the stress-affected volume at the Charpy notch root is more extensive, as a result of the larger root radius, compared with an equivalent K_{1C} sample. It has been hypothesised that this creates a large region containing brittle martensite prior to crack initiation [1]. This is supported by observations of martensite on the fracture surfaces of bainitic steels following Charpy impact testing using X-ray diffraction [14] and transmission electron microscopy [15]. As a result, when the sample fails, it does so in a brittle manner. Since a K_{1C} sample contains a sharp crack, the corresponding stress-affected region is smaller than the austenitic regions, such that any martensite formed will remain surrounded by retained austenite. Understanding such behaviour is important for assessing the relevance of the Charpy toughness results from these alloys, and to work towards controlling the toughness on the basis of phase-transformation theory.

During a Charpy impact test, the specimen is subject to rapid three-point-bend loading. By applying a three-point-bend at low strain-rates, it is possible to recreate the loading conditions that exist at the notch prior to failure, but not the consequences of

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strain-rate. High-resolution synchrotron X-ray diffraction provides a means of accurately measuring the lattice distortion and phase fraction distribution in the vicinity of the notch, before and after loading. It was therefore the aim of the present study to investigate, using synchrotron techniques, the response of the material at the notch root to an applied stress.

2. Experimental methods

The chemical composition of the alloy used is shown in Table 1. Compared to other nanostructured bainitic steels, this alloy has a relatively low silicon concentration for better continuous casting quality and a low manganese concentration to accelerate the bainitic transformation [16]. The samples were austenitised at 930 °C for 1 h in a tube furnace under an argon atmosphere and subsequently isothermally transformed at 200 °C for 96 h, followed by water quenching.

Microstructural characterisation of the heat treated material was performed by transmission electron microscopy (TEM) using a JEOL 200CX operated at 200 keV. Electron transparent samples were produced by twin-jet polishing with a solution of 15% perchloric acid in ethanol at 7.5 °C and a voltage of 26 V.

Sub-size Charpy specimens with dimensions of 10 mm × 7.5 mm × 55 mm were prepared from 8 mm-thick plate according to the EN 10045-1 standard. The specimens were heat treated as blanks before being sent for final machining. The Charpy specimens were oriented with the notch in the rolling direction, due to the presence of strong banding in the as-received plate.

Bend tests were carried out using an 100 kN servo hydraulic machine, with a support span of 40 mm and a displacement rate of 0.5 mm min^{-1}. Under these conditions, the failure load was found to be ~10 kN. Subsequent samples were loaded to 8 kN and then unloaded, allowing the intact notch root region to be examined. These samples were sliced lengthwise into 2 mm-thick sections using electro-discharge machining. Similar sections were taken from unstressed Charpy samples. The surface of each slice was ground and polished using SiC paper (800-grade to 4000-grade).

High energy X-ray diffraction was performed on the I15 beamline at the Diamond Light Source in Oxford, UK, using a monochromatic X-ray beam with dimensions of 70 × 70 μm and a wavelength of 0.16557 Å. Each diffraction pattern was captured in transmission over 40 s using a flat, 2-D Perkin–Elmer detector mounted perpendicular to the incident beam. In the unstressed sample, a region roughly 1 × 1 mm in size was mapped by taking spectra over a fine grid (step size 70 μm) close to the notch root, and a coarser grid (step size 100 μm) further from the notch root. A step size of 70 μm was used over the full scan region (2 × 1 mm) underneath the stressed notch, and a step size of 100 μm was used to scan a 1 × 1 mm un-notched region. In the case of the unstressed notch, only half the notch region was scanned, due to time constraints. A lanthanum hexaboride (LaB_6) powder standard was used as a calibrant to refine the instrument parameters. A schematic illustration of the experimental setup is shown in Fig. 1.

Data were recorded as 2-D images and were integrated azimuthally to convert to 1-D intensity *versus* 2θ data using Fit2D [18–20]. This analysis was considered valid as a previous study of the present alloy has shown that it is not significantly textured [13].

Quantitative phase analysis was performed using Rietveld refinement with Bruker TOPAS v.4.2 software. The volume fractions of the austenite and ferrite were obtained by fitting to the first four reflections of each phase. By using multiple peaks, the influence of any textural effects upon the refinement results was minimised [21]. Although it is expected that martensite forms in the notch region, the quantities were too small to be characterised unambiguously and it was not possible to separate the ferrite peaks reliably into two distinct phases. Therefore, only austenite and ferrite were assumed to be present in the diffraction data analysis.

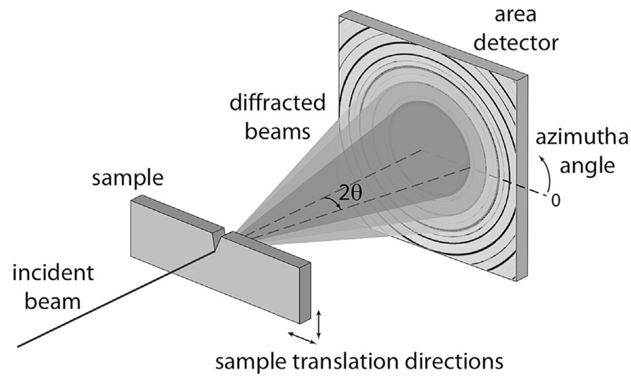

Fig. 1. Illustration of the experimental setup, adapted from Ref. [17].

The orientation dependence of the lattice distortion was evaluated through fitting of single (and dual, in the case of the $\{111\}_\gamma/\{110\}_\alpha$ reflections) peaks with a Voigt function from 10° azimuthal segments taken at the cardinal points (azimuthal angles of 0°, 90°, 180° and 270°) of the Debye-Scherrer rings using Fit2D, and peak fits were performed with Wavemetrics Igor Pro. The *hkl*-specific strain was calculated as $\varepsilon_{hkl} = \Delta d/d_i$, where d_i was taken as an average of the *d*-spacings measured in each sample away from the notch. Similarly, the average lattice strain, ε_{av} was obtained as $\varepsilon_{av} = \Delta a/a_i$, where a_i is the average lattice parameter obtained from the Rietveld refinement of multiple peaks. Obtaining the average lattice strain in this way has been shown previously to provide an effective measure of the macrostrain, avoiding the effects of type II stresses [22].

3. Results and discussion

3.1. Microstructure

A representative TEM micrograph of the alloy following isothermal transformation at 200 °C for 96 h and water quenching is shown in Fig. 2. The microstructure comprised nanoscale bainitic plates with a mean plate thickness of 57 ± 3 nm, as determined from over 800 individual measurements [13]. Whilst still in the nanoscale range, the observed plate thickness is coarser than that reported for other superbainitic steels. This may be a consequence of the lower concentrations of alloying additions, which provide less solid solution strengthening of the austenite and thereby allow coarser plates to form [23]. In addition, the lower silicon content of this alloy, compared with other nanostructured bainitic steels, does not fully suppress the formation of cementite within the microstructure. Detailed analysis identifying the presence of cementite in

Table 1
Chemical composition of alloy, wt%.

C	Si	Mn	Ni	Cr	Mo	Al	Cu	Sn	N	P	S
0.82	0.71	1.30	0.02	0.9	0.2	0.033	0.02	0.004	0.007	0.01	0.003