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Published version

ROURKE, K., EBRAHIM, M., LUO, Quanshun and WILSON, E A (2016). Hump on upper shelf of ductile-brittle transition temperature curve of a plain carbon steel. *Materials Science and Technology*, 27 (3), 693-695.

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Hump on upper shelf of ductile–brittle transition temperature curve of a plain carbon steel

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Abstract: The ductile–brittle transition temperature (DBTT) in a plain carbon steel has been studied in a series of Charpy V notch samples. Both non-standard and standard samples were used in the as drawn, normalised and annealed conditions. In all cases, a hump was recorded on the upper shelf of the DBTT. Examination of fractures in the SEM in the vicinity of the hump showed typical microvoid coalescence. Low temperature fractures showed classical cleavage fractures with river markings. In the normalised and annealed conditions, both yield stress σ_y and impact transition temperature T_c varied inversely with square root of the ferrite grain size, in agreement with previous workers. However, the points for ‘as drawn’ samples lay off the linear plot. The reasons for this are discussed. The hump on the upper shelf of the DBTT is thought to be associated with dynamic strain aging.

Key Words: Ductile–brittle transition, Plain carbon steel, Hall–Petch relationship, Hump on upper toughness shelf

Background

In Fig. 4 of Ref. 1, there was suspicion of a hump on the upper shelf, in the vicinity of 100°C, of the ductile–brittle transition temperature (DBTT) of a Fe–8Mn alloy. Half-size Charpy V notch specimens 5x10x55 mm were used to determine this DBTT. There were two points at 141°C (68 J) and 159°C (69 J) and one at 120°C (76 J) significantly above the other two. The present paper describes work to confirm this suspicion on a plain carbon steel. It was also planned to develop a practical for undergraduates using a 9 or 10 mm square bar and a minimum of machining.

Experimental procedure and results

The composition of the plain carbon steel consisted of 0.20%C, 0.38%Mn, 0.035%Si and 0.203% total residuals. The main study was carried out on 9 mm square bar, as it was more economical than using 10 mm square samples machined from bulk material.² However, towards the end of the investigation, 10 mm square bars also became available and were also used.³ The 9 mm square samples were tested in three conditions: as drawn, normalised and annealed. The purpose of this was to establish the difference that grain size had on the position of the DBTT.

Bars were cut to 55 mm in length. After coating in Berkatekt (a preparatory coating for reducing oxidation and decarburisation), the following heat treatments were applied to the 9 mm bars: annealing for 1 h at 1150°C followed by furnace cooling and normalising for 1 h at 920°C followed by air cooling. The 10 mm square bars were coated in Berkatekt and homogenised for 50 h at 1200°C in clay sealed cast iron boxes, followed by air cooling. One set was then annealed by coating in Berkatekt and heat treated for 1 h at 1100°C followed by furnace cooling. The other set was then also coated in Berkatekt, given 1 h at 920°C followed by air cooling, i.e. normalised. After heat treatment, all the bars were V notched in the middle to an angle of 45° and a depth of 2 mm with a 0.25 mm radius of curvature at the base of the notch.

Microscopic samples from each set of samples were prepared in the longitudinal and transverse directions. Specimens were etched in nital. The ferrite grain sizes were measured using the mean linear intercept to an error or 95% confidence limit of $\pm 2\%$.⁴ Results are given in Table 1, together with the impact transition temperature T_c evaluated at 26 J, hardness and mechanical properties to be described below. Vickers hardness was measured using a 20 kg load and an average of 10 impressions on two samples. The results are presented in Table 1. Errors are 95% confidence limits.

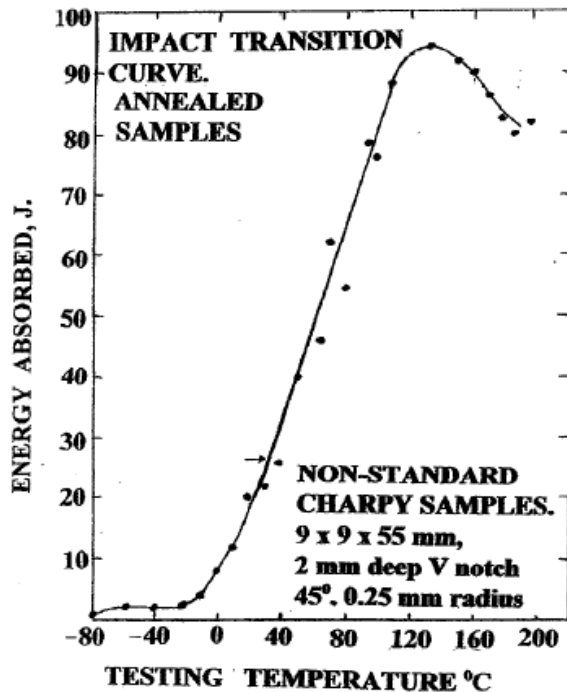
Hounsfield tensile specimens with a gauge length of 10 mm and diameter of 3.58 mm were machined from broken Charpy specimens. These were tested in a tensometer at a velocity of 20 mm min⁻¹, i.e. a strain rate of 2 min⁻¹. The yield stress σ_y and tensile stress σ_b were estimated from the stress–strain curves. Percentages of reduction of area ψ and total elongation δ_t were measured on the broken specimens. The results, with 95% confidence limits, are presented in Table 1.

Samples were tested on a Charpy impact machine with a maximum impact energy of 300 J. For testing above room temperature, samples were held for at least 30 min in an air circulating

Table 1 Grain size d , impact transition temperature T_c (26 J), hardness and mechanical properties of steel in various conditions

Condition	$d, \mu\text{m}$	$d^{-1/2}, \text{mm}^{-1/2}$	$T_c, ^\circ\text{C}$	Hardness, HV20	σ_y, MPa	σ_b, MPa	$\psi, \%$	$\delta_b, \%$
As drawn	8.7 ± 0.17	10.72 ± 0.11	32 ± 4	194 ± 6	432 ± 7	480 ± 7	50	12
Normalised	15 ± 0.3	8.16 ± 0.09	0 ± 4	137 ± 7	264 ± 13	440 ± 13	70	25
Annealed	37 ± 0.74	5.2 ± 0.053	34 ± 4	119 ± 8	203 ± 15	377 ± 15	55	25

oven, and for testing below room temperature, in liquid refrigerants for at least 10 min. They were then transferred to the Charpy impact machine and tested within 5 s.



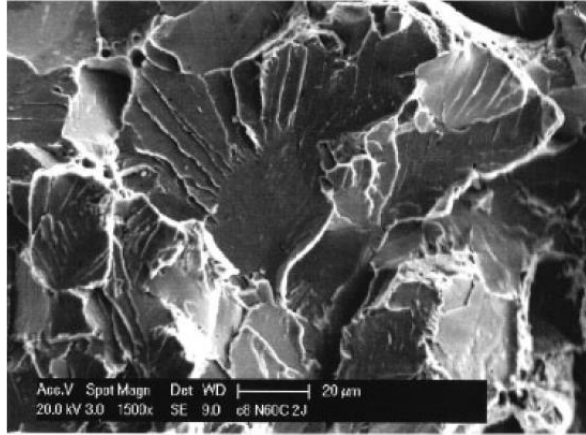
1 Impact transition curve of annealed material: non-standard Charpy specimens, 9610655 mm, V notch machined after heat treatment

A typical impact transition curve is shown in Fig. 1. Note the hump on the upper shelf. Similar results were observed on normalised and as drawn material. Impact transition temperatures T_c taken from these impact curves, are presented in Table 1. After impact testing, broken specimens were placed in alcohol and dried in an air dryer so the fracture surfaces could be examined in a scanning electron microscope (SEM). Fractures were examined in an FEI NanoSEM at acceleration voltages of 15 and 20 kV. A typical low temperature fracture is shown in Fig. 2. This shows classical cleavage facets with river markings. Fractures examined in the vicinity of peak energy showed a cellular structure due to microvoid coalescence.

Hall-Petch plot

The variation of yield stress^{5,6} and impact transition temperature⁷ with grain size is shown in Fig. 3. Although in the original paper,⁷ T_c varied linearly with $\ln(d^{-1/2})$, to a good approximation, $d^{1/2}$ can be used rather than $\ln(d^{-1/2})$.⁸

The yield stress σ_y is related to the grain size d by the Hall–Petch equation^{5,6}



2 Low temperature fractograph of normalised steel (specimen C8, 2 J at 260uC), showing cleavage facets with river markings

$$\sigma_y = \sigma_i + k_y d^{-1/2}$$

where σ_i is the friction stress, equivalent to the yield stress of a constrained single crystal, and k_y is the locking parameter, now thought to be related to the ease of generating dislocations from the grain boundary to move into the adjacent grain.⁹

By regression analysis of the properties of plain carbon manganese steels, Gladman et al.¹⁰ obtained the expression

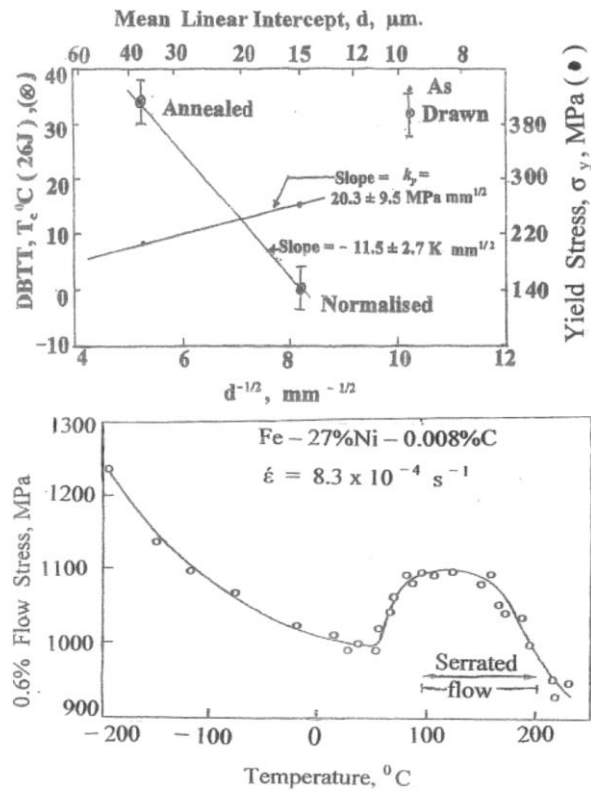
$$\sigma_y \pm 31 \text{ (MPa)} = 88 + 37 (\% \text{Mn}) + 88 (\% \text{Si}) + 29.18 (\% \text{N}_{\text{free}}) + 15.1 (d^{-1/2})$$

$$T_c \pm 30 \text{ (}^\circ \text{C)} = 19 + 44 (\% \text{Si}) + 700 (\% \text{N}_{\text{free}})^{1/2} + 2.2 (\% \text{ pearlite}) - 11.5 (d^{-1/2})$$

where d (mm) is the mean linear intercept in polygonal ferrite in millimetre. In the present steel, $\text{Mn} = 0.38\%$ and $\text{Si} = 0.035\%$; hence, $\sigma_i = 105 \pm 31$ MPa compared with 96 ± 62 MPa in Fig. 3 and $k_y = 15.1$ MPa $\text{mm}^{1/2}$ compared with 20.3 ± 9.5 MPa $\text{mm}^{1/2}$ in Fig. 3. The slope of T_c v. $d^{-1/2}$ in Fig. 3 is -11.5 ± 2.7 K $\text{mm}^{1/2}$ compared with Gladman et al.'s value of -11.5 K $\text{mm}^{-1/2}$.

The points for σ_y and T_c for as drawn material are off the plot in Fig. 3. This is because of three factors:¹¹

- (i) residual stress in the as drawn material
- (ii) large upward shifts in the DBTT due to prestrain¹²
- (iii) a very marked decrease in upper shelf ductility as a result of flow localisation.¹³



3 Yield stress σ_y and impact transition temperature T_c v. $d^{-1/2}$

4 Temperature dependence of flow stress of Fe–Ni–C martensite, showing dynamic strain aging in vicinity of 100°C: adapted from Owen and Roberts¹⁵

Hump on upper shelf

The hump on the upper shelf might be thought to be a result of using a substandard 9 mm square bar, but the 10 mm square bar also showed a hump on the upper shelf, both in the normalised and annealed conditions. The hump is thought to be associated with dynamic strain aging,^{14,15} as it occurs within the vicinity of 100°C (Fig. 4). However, this has yet to be fully proven. Impact tests are being carried out to see if the hump is absent in a fully stabilised interstitial free steel.

Acknowledgements

Sheffield Hallam University is thanked for provision of research and library facilities. Professor A. Howe is thanked for arranging full spectrographic analysis of the steel at Swinden Technology Centre, Rotherham, UK. Useful correspondence was held between Emeritus Professor N. J. Petch of Stathclyde University, Professor J. F. Knott of Birmingham University and Emeritus Professor F. B. Pickering, former Sheffield Hallam University.

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