An appraisal of the energy crack zero (ECo) test in the evaluation of tempered martensite embrittlement (TME)

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Abstract
Tempered martensite embrittlement (TME) was investigated in an electroslag remelted (ESR) aeronautical grade SAE 4340 steel. The Charpy - V impact, plane strain fracture toughness (K_{Ic}) and energy crack zero (ECo) tests were employed to evaluate the reduced toughness of 300 °C as compared with 200 °C-tempered specimens after fracture in the temperature range from − 70°C to + 150°C. The ECo and Charpy - V results were comparable. The K_{Ic} test, however, failed to detect TME, even though a fatigue precracked specimen was used as in the ECo test. This discrepancy between the two tests is attributed to the different influence of a microtough zone a few μm thick at the crack tip. Caution is therefore advised in the conversion of ECo values to their K_{Ic} equivalents.

Riassunto
La prova " Energy crack zero " (ECo) nello studio della fragilità di rinvenimento della martensite.
È stato esaminato il comportamento di un acciaio SAE 4340 " aeronautical grade " prodotto all'ESR nei confronti della fragilità di rinvenimento della martensite. Le riduzioni di tenacità che si verificano tra i rinvenuti a 200 e quelli a 300 °C sono state misurate con prove Charpy-V, Eco, e K_{Ic} in un campo di temperature da − 70 a + 150 °C. Le prove ECo e Charpy-V forniscono dei risultati confrontabili tra loro mentre le prove di K_{Ic} non sono in grado di individuare il fenomeno del TME nonostante che, come nella prova ECo, si adoperi un provino con cricca di fatica. Questo comportamento viene addebitato alla diversa influenza che una zona micro-tenace, dello spessore di pochi μm all'apice della cricca, può esercitare sulla misura della tenacità al variare delle condizioni di prova.

The tempered martensite embrittlement (TME) of alloy hardening steels such as SAE 4340 is a bar to their employment at very high strength levels [1-4], since tempering at 300 ± 50°C after quenching results in a loss of toughness compared with material tempered at 200 °C, or, in some cases, merely quenched. TME has been investigated by many workers and attributed to one or more of the following factors [1-8]:

- precipitation of carbides and nitrides during tempering
- segregation of impurities (especially P and N) on the primary austenitic grains
- decomposition of the residual austenite
- inclusional status
- austenitization temperatures and times

Embrittlement becomes particularly evident when toughness is measured by resilience tests, and appears as a nadir of the fracture energy vs tempering temperature curve.
A previous work has shown[4] that this phenomenon can be closely followed by introducing the parameter JJ%, i.e. the % difference in energy between specimens tempered at 200 and 300 °C:

$$JJ\% = \frac{C_{v200} - C_{v300}}{C_{v200}} \times 100 \qquad (1)$$

where C_v is the fracture energy measured through resilience tests on Charpy-V notch specimens. If, on the other hand, the plane strain fracture toughness test (K_{Ic}) is used, embrittlement is not detected, toughness increases with tempering temperature, and the parameter equivalent to JJ% becomes negative.
Several characteristic features of TME have been described in the recent literature [1-7], especially in the papers presented at the " Peter G. Winchell Symposium on Tempering of Steel " [8]:

- the role of residual austenite, and the effects of both thermal decomposition and mechanical instability[5-7]. It is thought that the residual austenite to martensite transformation induced by strain in the plasticised zone at the crack tip is able to absorb energy during fracture, with the result that alloys tempered at 200 °C are tougher than those tempered at 300 °C because they contain martensite + austenite instead of martensite + carbides.
- the absence of TME minimum toughness in steels with a composition similar to that of SAE 4340, but without Mn and Si, and with very low P (0.003%) and S (0.005%).
- the presence of a ductile zone in the first 50 μm of fracture propagation at the fatigue crack tip. The all-important contribution this zone makes to triggering unstable propagation of the crack helps to explain why the K_{Ic} is not sensitive to TME.

A recent work [4] illustrated the influence of the SAE 4340 production process on TME. Electroslag remelted (ESR) and vacuum arc remelted (VAR) alloys performed better than those made with conventional methods. TME appeared over the entire test temperature range (from - 170 to + 150°C). It has also been shown that embrittlement is not necessarily associated with intergranular fracture along the primary austenitic grains.

The present paper is a further examination of the influence of the test method in the assessment of TME. Reference is made to data published for the resilience and K_{Ic} test on similar ESR SAE 4340 steels tempered in the critical temperature range [4-9] in an evaluation of the energy crack zero (ECo)

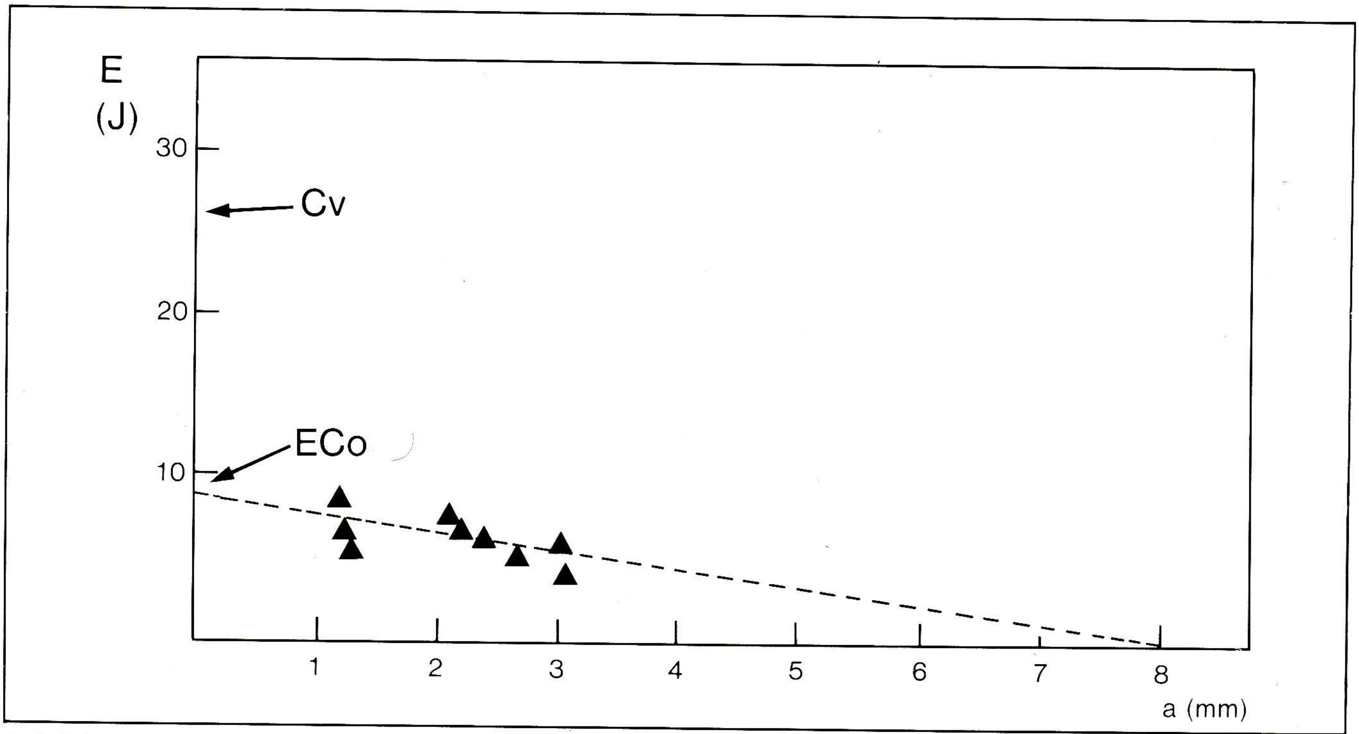


Fig. 1 - Estimation of ECo from the experimental data for nine fatigue precracked specimens. a = depth of fatigue crack.

test [10-12] as a sensitive indicator of TME.
This test is based on the fracture of fatigue precracked Charpy-V specimens with a resilience pendulum. Specimens measuring 10 × 10 × 55 m have a 2 mm, 45° V notch, and a 1-3 mm deep fatigue crack from the notch tip [12]. The parameter ECo is the fracture energy per fatigue crack length tending to zero. Energies vs crack lengths are plotted for at least three tests. The broken line in Fig. 1 is defined by the points:
a) Energy = 0, Depth = 8 mm
b) The mean value line of the experimental points

The Eco test lies between the resilience and K_{Ic} tests. It combines the dynamic loads and absorbed fracture energy measurements of the former with the fatigue crack propagation of the latter. The influence of the crack is apparent when the C_v and ECo values for the same material (i.e. the experimental values and those deduced from crack zero) are compared (Fig. 1).

Materials and methods

An aeronautical grade, ESR SAE 4340 steel hot-rolled into Ø 60 mm bars was used. Its composition is shown in Table I. Longitudinal specimens measuring 11 × 11 × 56 mm were austenitized in salt at 850 °C for 1 h, quenched in stirred oil, and tempered at 200° or 300 °C for 2 h. Various characteristics of this steel are illustrated in Table II.
Standard Charpy-V specimens with a 2 mm deep 45° notch were pendulum fractured at −170 °C to + 150 °C. Three sets of three Charpy-V specimens were fatigue precracked to a nominal 1, 2 and 3 mm for the ECo test. A single Eco value was taken for each group of nine specimens, using the method illustrated in Fig. 1. The lower temperature is dictated by the lower fracture energy absorption values of the specimens, while the higher temperature must not influence the results of the heat treatment. A smaller test run was also performed on specimens tempered at 400 °C (Fig. 3). Ravez's equivalence for poorly plasticised fractures [11] was used to obtain the corresponding K_{Ic} values.

$$K_{Ic} = 10^{(\frac{1}{2} \log ECo + 1.5)} \quad (2)$$

Even though fatigue precracked Charpy-V specimens cannot be compared with those standardised for the K_{Ic} test, a check was made of the compatibility of the thickness with the plane strain state by using these

TABLE I - Chemical composition of ESR SAE 4340 steel

Element	%/wt	Element	ppm
C	0.42	P	80
Ni	1.70	S	20
Cr	0.85	Sb	16
Mn	0.80	Sn	80
Mo	0.22	N	80
Si	0.33	O	64
Cu	0.05		
Al	0.04		

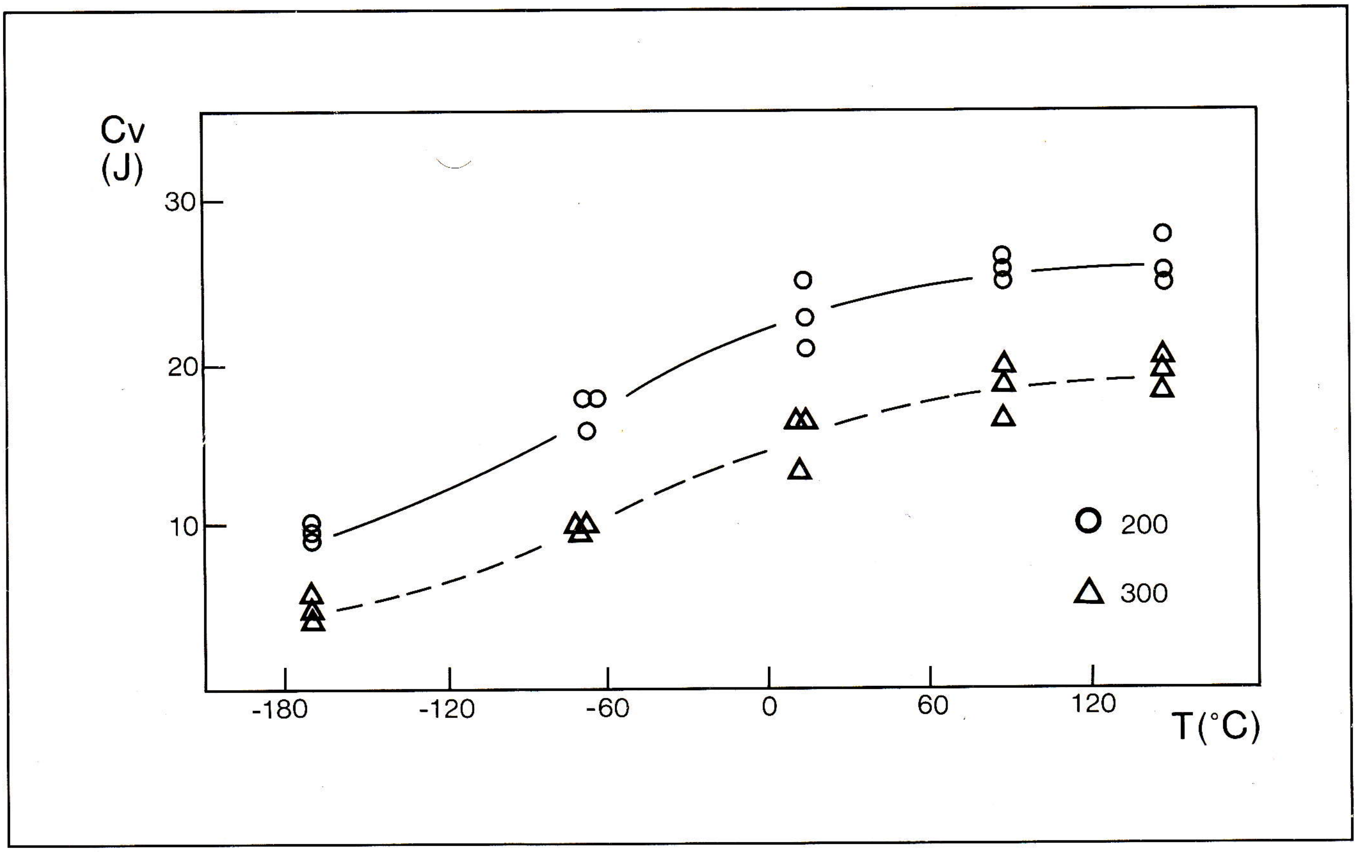


Fig. 2 - Resilience vs test temperature for specimens tempered at 200 °C and 300 °C.

TABLE II - Mechanical and metallurgical properties of ESR SAE 4340 steel

Primary austenitic grain: 6-8 ASTM

Tempering at	HRC	Yield point (σy)
200 °C	51	1550 MPa
300 °C	47	1480 MPa
400 °C	45	1400 MPa

Inclusion rating (ASTM E 45-76, method D)

		Type A	B	C	D
0.5	thin	8	12	8	6
	heavy	8	1	1	—
1.0	thin	1	7	1	—
	heavy	1	—	—	—

values in the well-known equivalence:

$$B = 2.5(K_{Ic}/\sigma_y)^2 \qquad (3)$$

The results are set out in Table III.

Mössbauer spectra were also made on specimens tempered at 200 °C and 300 °C. 0.2 mm slices from already fractured specimens were chemically thinned to about 50 µm for transmission analysis in an Elscint constant-acceleration spectrometer [13,14].

Results

The Cv results (fracture energy vs temperature) [4] for steels tempered at 200° and 300 °C are shown in Fig. 2. Those tempered at 200 °C were in all cases tougher and harder (Table II), showing that TME occurred over the whole temperature range.

The ECo test results (Fig. 3) show that energy values increased for all three tempering temperatures from very low, poorly reliable levels at −70 °C to a maximum at +150 °C. A transition effect between +20 and +90 °C was observed for the 400 °C tempered specimen only. Except at room temperature, where the 200 °C and 400 °C virtually coincide (see also Table III), ECo values rise in the order 300 → 200 → 400. The test is less selective than the resilience test, but does not give the 200 → 300 order suggested by the K_{Ic} test (Table III).

No evidence of intergranular fracture [4] even at the lowest temperature was observed in precracked or not precracked specimens from steels tempered at 200, 300 and 400 °C. Scanning electron microscopy (SEM) showed that embrittlement was greatest in specimens tempered at 300 °C and fractured at the lowest temperatures, whose surfaces were primarily characterised by quasi-cleavage with secondary fractures. Fig. 4 is a fractograph of fatigue crack tip fracture initiation on a 300 °C tempered specimen fractured at −70 °C. It was taken (like those in Figs 5 and 6) in the central zone, where fracture occurs in a state of plane strain and proceeds from left to right. The transition between the fatigue crack and the terminal failure (AA) is quite distinct. There is no transition zone, but a clear change from fatigue to brittle fracture with dominant quasi-cleavage and small microtough areas. In the Charpy-V specimens, the corresponding low-temperature fracture surface has exactly the same appearance, part from the gross plastic strain zone near the notch.

Fig. 3 - ECo vs test temperature for specimens tempered at 200 °C, 300 °C and 400 °C. Each point represents the fracture of nine fatigue precracked specimens.

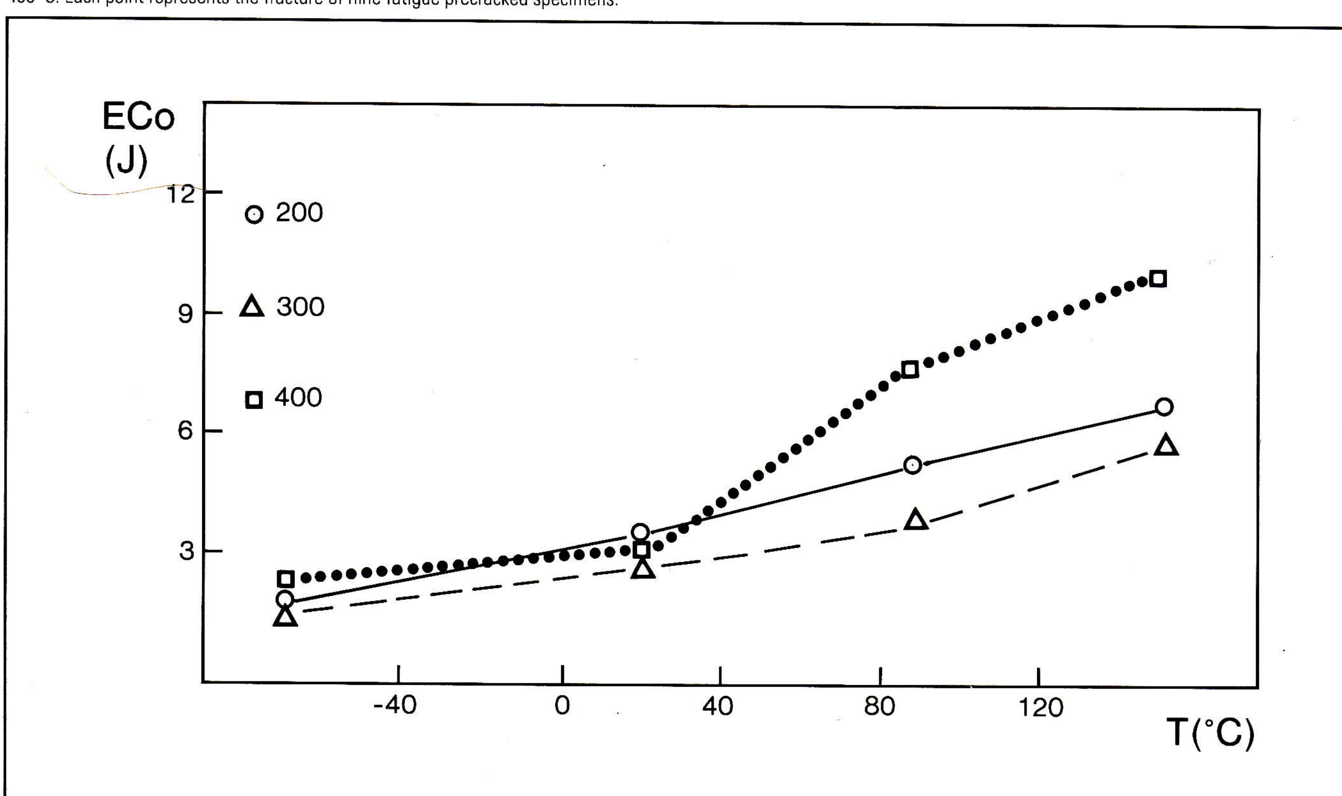


TABLE III - Room temperature toughness values (measured and calculated)

Tempering at	ECo (J)	K_{Ic} calculated (MPa $m^{1/2}$)	B (mm)	K_{Ic} measured[9] (MPa $m^{1/2}$)
200 °C	3.3	57	3.4	58
300 °C	2.7	52	3.1	80
400 °C	3.2	56	4.0	109

Fig. 4 - SEM fractograph of specimen tempered at 300 °C, fatigue precracked and fractured at −70 °C. Fracture from left to right. AA = transition between precracking and terminal failure (× 800).

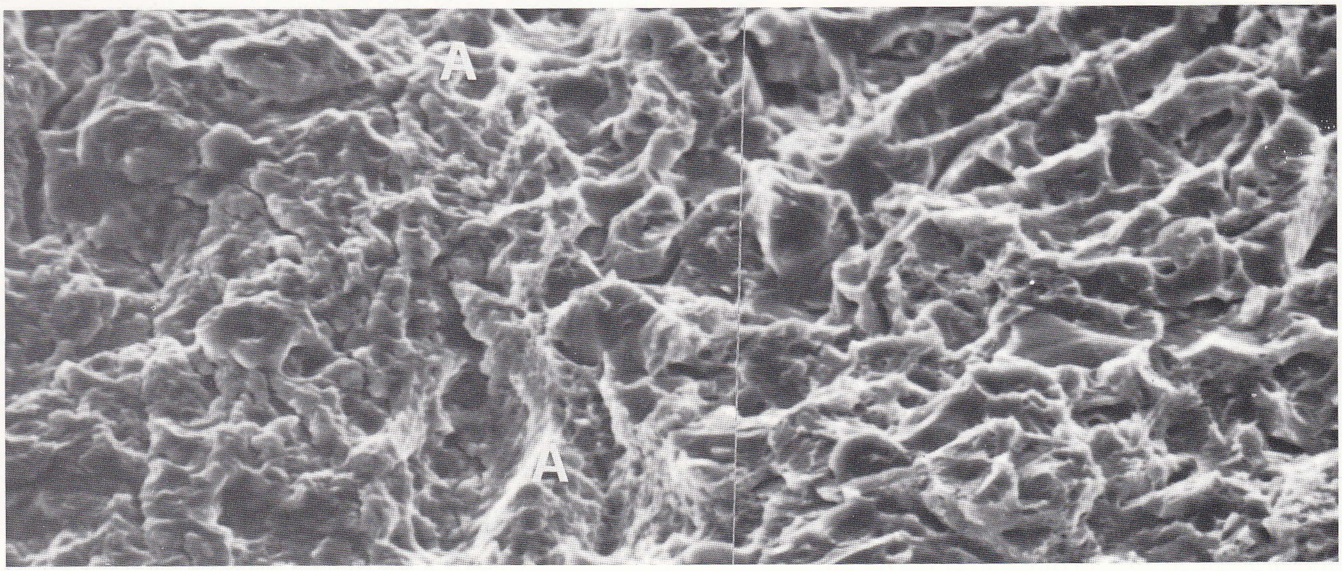
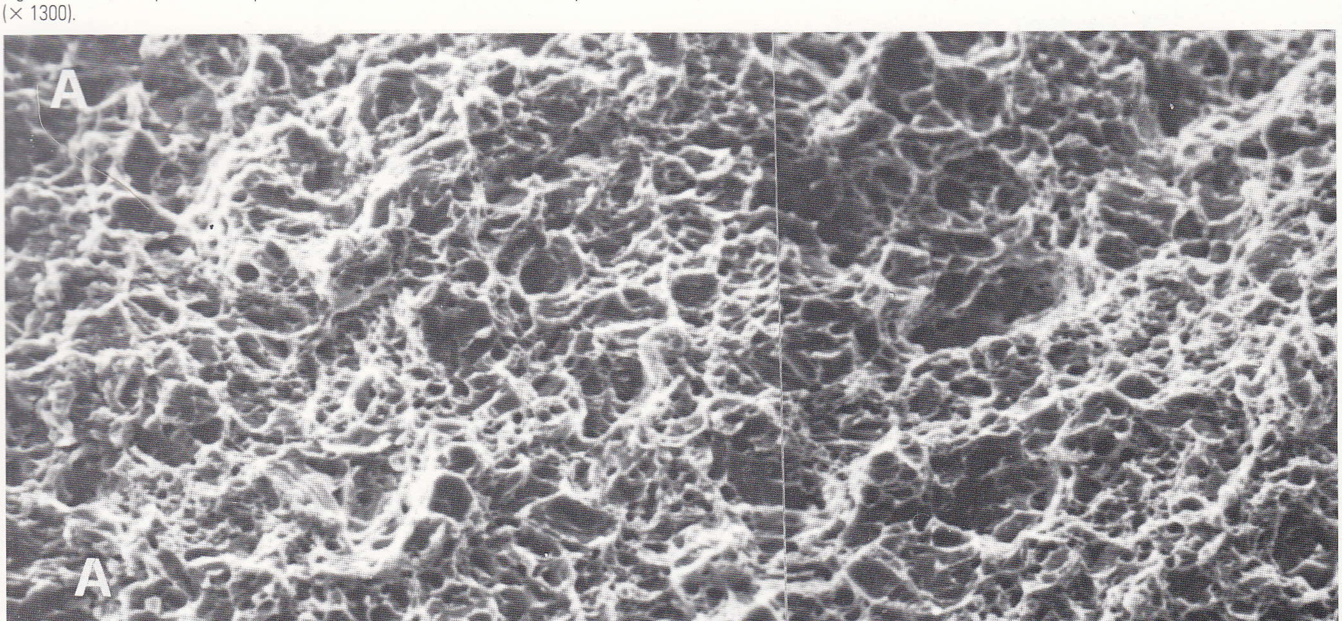


Fig. 5 - As last, for specimen tempered at 200 °C and fractured at room temperature (× 1300).



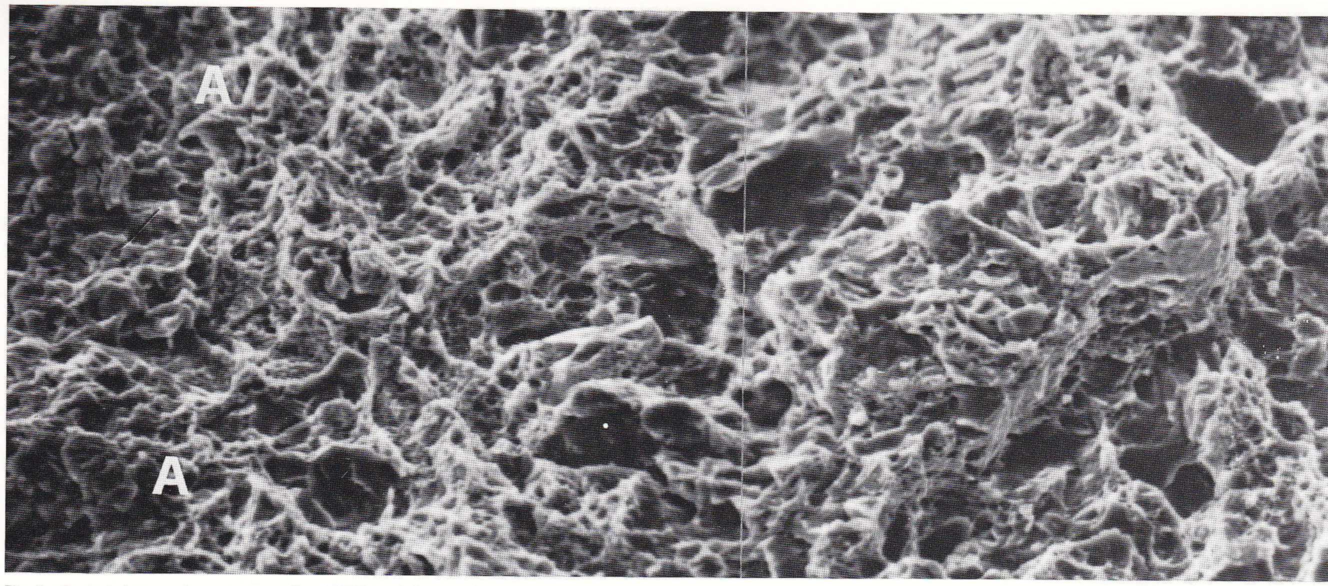


Fig. 6 - As last, for specimen tempered at 400 °C and fractured at room temperature (× 800).

The SEM fractograph of a precracked 200 °C-tempered specimen fractured at room temperature (Fig. 5) is marked by a prevalence of microvoids. Its ECo value and that of its 300 °C-tempered equivalent are very similar, though the 200 °C specimen appears to be tougher.

A fractograph of a precracked, 400 °C-tempered specimen fractured at room temperature is shown in Fig. 6. The distinctive feature of the initial propagation zone (about 20 μm thick) to the right of the fatigue zone is the large number of microvoids. As the fracture proceeds, there is a more even distribution of microvoids and quasi-cleavage surfaces, with an ECo value midway between those for the previous treatments. Toughness measured by the ECo test, therefore, increases in function of the number of microvoids, whereas the absolute values reflect levels typical of marked embrittlement. A fracture with a transition zone can also be obtained on specimens tempered at 300 °C, provided they are fractured at + 150 °C (ECo), or at room temperature (K_{Ic}). In this case, the transition zone is about 50 μm deep. Characteristic values calculated from equations 1 and 2 are compared with those of a previous work [9]. In Table III. Plane strain propagation conditions were observed for all three tempering temperatures. The results were only comparable with those in the K_{Ic} test at 200 °C, however.
The JJ% values (eq. 1) for the resilience (Cv), Eco and K_{Ic} tests are directly compared in Fig. 7

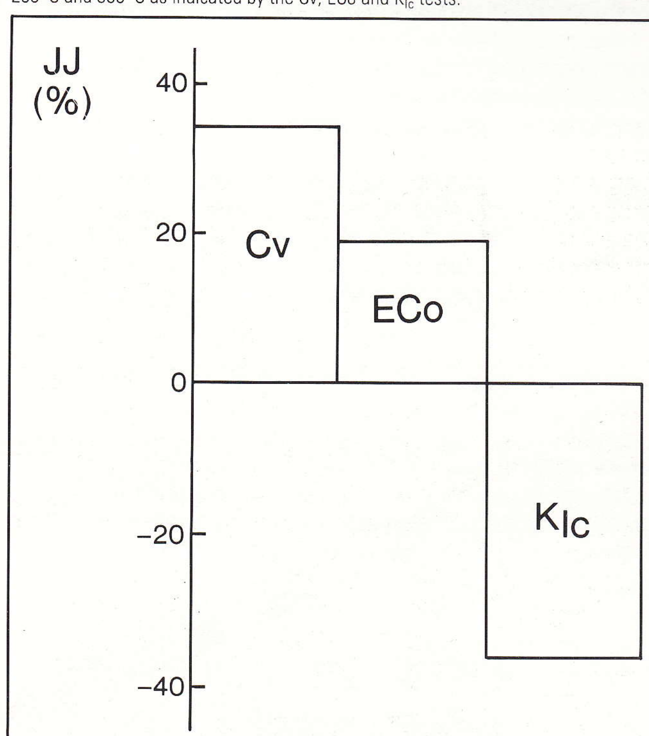
Mössbauer spectroscopy for the 200 °C-tempered specimens revealed a martensite spectrum with about 5.6% residual austenite by volume, whereas the 300 °C specimen displayed all martensite and about 2.6% carbides (as cementite) by volume [13,14].

Discussion

Materkowski's paper [1] on the TME of low-P 4340 has made it clear that a brittle fracture surface can go hand in hand with higher than expected K_{Ic} toughness values due to the presence of a microtough zone extending some tens of μm from the fatigue crack tip. This zone has a fundamental influence on toughness values, since it appears in the first extension of the fracture. During propagation, the fracture then takes on a more brittle appearance. This paper shows that a tough-brittle transition zone also exists on fatigue precracked specimens, and that its influence on toughness values depends on the method of measurement adopted.

The ECo and Cv tests were conducted on similar specimens under similar impact conditions. They are both sensitive to TME, though not to the same extent.

Fig. 7 - Comparison of % toughness differences between specimens tempered at 200 °C and 300 °C as indicated by the Cv, ECo and K_{Ic} tests.



Room temperature tests on precracked specimens differ more with regard to the appearance of the fracture surface than in their fracture energy values. The tough-brittle zone appears at this temperature on the 400°C specimens only. The ECo test is not so sensitive to this zone as the K_{Ic} test, since it measures the whole of the energy absorbed by the facture, whereas the latter is only influenced by the initial, unstable crack propagation. The difference in the temperatures at which the transition zone appears may be ascribable to the dynamic nature of the ECo test ($\dot{K} = 10^5 \div 10^6$ MPa $m^{1/2}s^{-1}$) whereas the K_{Ic} test is static ($\dot{K} \cong 3$ MPa $m^{1/2}s^{-1}$). Ripling et al. [15], for example, reported a shift in the transition appearance temperature when static and dynamic tests were carried out on 4140 and 4340 steels tempered at temperatures higher than those used in this paper.
The fractographs draw a sharp distinction between the specimens tempered at 200°C and the others. They have more abundant microvoids and display no changes between the crack tip and the propagation zone. The Mössbauer spectra indicated that tempering at 200°C for 2 hr is not able to transform the residual austenite. This is present in the forms of laths and does not appear capable of influencing the hardness of the material.
Strain-induced martensitic transformation during fracture is accompanied by the absorption of energy over all the surface and the rest of the strained area. It is suggested that the retained austenite also improves a fracture toughness by means of crack tip blunting and the scavenging of detrimental impurities. Here the K_{Ic} and ECo data can be validly compared, since transformation occurs both at the crack tip and in the rest of the fracture surface. The difference between them becomes apparent at higher tempering temperatures on account of the transition zone observed in the K_{Ic} specimens, but in only one ECo specimen. It is thus doubtful whether the K_{Ic} test is a sufficiently exhaustive indicator of how a steel behaves in TME.

ECo data, therefore, cannot be readily converted into K_{Ic} equivalents. Errors of the order of 100% are likely for values around 3 J at all events within the TME field. There is nevertheless considerable scope for this new test, especially for comparison of large sets of specimens, and when measuring products whose thickness is incompatible with the requirements of the K_{Ic} test [16].

Conclusions

The ECo test throws fresh light on the TME of SAE 4340 hardening steels. Fatigue precracked Charpy specimens display a reduction in toughness when tempering is carried out at 300°C, as in the classic resilience test. In spite of the similar crack geometry, the ECo results are by no means the same as those obained with the K_{Ic} test. This difference makes it clear that the influence of the first unstable crack propagation zone prevents the K_{Ic} test from indicating the overall behaviour of the steel during terminal failure. In the TME range, therefore, ECo data cannot be converted into their K_{Ic} equivalents.

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