BII-11 THE FRACTURE OF MARTENSITE

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ABSTRACT

The effect of grain size and prior austenite deformation on the fracture of martensite has been studied. Grain size has been varied by changing the prior austenitising time from a few seconds to several minutes. It has been shown that by this means a five-fold refinement of austenite grain size can be achieved but the effect of this refinement on fracture properties is not as marked as in equivalent polygonal ferrite structures. The reason for this is twofold. Firstly, the refined austenite does not produce an equivalent refined martensite due to the tendency of the martensite transformation to produce bundles of martensite needles whose width varies little with austenite grain size. Secondly, the rapid austenitising and quenching process necessary for the refining operation changes the dislocation substructure of the martensite in such a way as to increase frictional stress. This increase in frictional stress offsets to some extent the small degree of refinement achieved.

Prior austenite deformation also refines the martensite structure but in a different way to rapid austenitising. Dislocation substructure is also altered considerably.

The influence of the two processes on the fracture characteristics of martensite is described.

INTRODUCTION

The factors governing the fracture behaviour of ferrite in both its single crystal and polycrystalline form have been the subject of intensive study for many years. As a result a great deal is now known about the influence of grain size, prior deformation, chemical composition, temperature, etc. on the fracture characteristics of pure iron ferrite and its commercial derivatives, carbon-manganese steels.

Very little similar information exists for martensite despite the substantial increase in the need for ultra-high strength martensitic materials in the aerospace and defense industries and despite the fact that brittle fracture is probably the greatest single factor limiting the use of ultra-high strength materials.

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Attention has largely been concentrated on the evaluation of fracture toughness in these materials and many types of test have now been proposed and evaluated[1]. Some attempts have been made to employ these tests in the development of improved high strength materials[2,3]. In these cases and in earlier work on high strength steel development[4] the principal parameters considered have been chemical composition and heat treatment. No effort appears to have been devoted to studying the specific influence of grain size, friction stress and the like although a similar study has been so important in contributing to the understanding of fracture behaviour in ferrite[5] and has greatly assisted metallurgical development. Indeed, at one of the most recent major conferences on Fracture (see refs. 5 and 6) only one paper[6] made reference to martensitic structures and merely mentioned in passing that fracture properties may be related to martensite needle size.

More recently, increasing interest in thermomechanical techniques[7] for improving the properties of martensite has focussed attention on the need for understanding the morphological changes occurring as a result of mechanical deformation prior to transformation. Schmatz and his colleagues[8] concluded, however, that neither the austenitic nor martensitic grain size affected the properties of martensite.

This conclusion for ausformed steels has recently been contested by Hale and McLean[9]. Using other techniques Taranto[10], Grange[11], Phillips and Duckworth[12] have found that martensite grain size influences properties. In view of this conflicting evidence and in the belief that a knowledge of the way in which grain size influenced properties would stimulate the development of martensitic steels as it had done with ferritic steels a comprehensive examination was begun with the financial assistance of the U.S. Navy Bureau of Aeronautics under Contract No. N 62558-3718.

EXPERIMENTAL PROCEDURE

Two techniques have been used for studying the affect of grain refinement on the fracture characteristics of martensite:-rapid re-austenitising and ausforming.

RAPID RE-AUSTENITISING

In the rapid re-austenitising process bars of steel 9/16 in. square x 6 in. long are resistance heated by a direct electric current from a welding transformer rated at 45 kVA. The current is controlled by an electronic timer and phase shift unit in the primary winding. The timer provides for heating times from 1 to 10 seconds and the phase shift allows satisfactory control over the current flowing through the specimen.

A great deal of preliminary work[12,13] was carried out to establish the optimum conditions for rapidly re-austenitising the steel chosen. Too short a time at too low a voltage merely resulted in tempering of the prior martensitic structure, too long a time at too high a voltage resulted in inferior properties. The conditions finally selected were a 3.5 second treatment at the fastest heating rate possible. The potential drop across the bars was about 5 volts and the current about 18,000 amps. Quenching was by intense water sprays onto all faces of the specimen.

To ensure homogeneity of the material prior to rapid re-austenitising each bar was given a preliminary treatment at 850°C for half an hour followed by a water quench. This was done to ensure, as far as possible, that the distribution of alloying elements throughout the steel would be reasonably uniform and that all carbides were in solution prior to rapid heat treatment. This was to ensure that on subsequent rapid heating to the austenitising temperature all the precipitates would be finely dispersed throughout the matrix and would be readily soluble above the Ac_3 temperature. The necessity for this prior homogenisation was demonstrated in some trials in which the steel bars were tempered at 500°C for one hour prior to rapid re-austenitisinn. The response to the treatment was less than in the case of the prior quenched steels and the scatter in results was greater.

All surfaces of the bars were ground to remove scale prior to rapid heat treatment. One face of each bar was surface ground to a flat smooth finish to improve electrical contact during rapid heating. This was found to enhance the reproducibility of results. The steel chosen for the investigation was purchased to B.S. 970 specification En.30b and had the actual composition:-

C	Si	Mn	S	P	Ni	Cr	Mo	Cu
0.27%	0.20%	0.53%	0.009%	0.012%	3.93%	1.11%	0.14%	0.29%

After treatment a surface layer was removed by grinding, a copious supply of liquid coolant being maintained in order to avoid tempering the martensite. Grinding marks were then eliminated by polishing with emery paper and the prepared surface was electro-polished at three points along the bar. The electro-polished areas were etched in 2% nital, examined optically at magnifications up to 400 and hardness tested. Only those bars having a hardness of 650 ± 10 VPN and a very fine martensitic microstructure were considered as successfully treated.

AUSFORMING

The ausforming treatment was carried out on 1⅛ in. diameter bar

from the same stock as that used for rapid re-austenitising. The bars were solution treated for 1 hour at 950°C and then quenched into a lead bath at 500°C. The material was deformed at this temperature by drawing in 1/16 in. passes from its original diameter to $\frac{5}{8}$ in., the total amount of reduction being 70%. During the ausforming operation the bars were returned to the lead bath for two minutes between each pass and the material was air cooled after the last pass. Air cooling was employed and not water quenching as with the rapidly re-austenitised bars because of the length of bar and the difficulty of ensuring a uniform quench.

The ausforming treatment was also applied to a similar steel En.30a, with a lower molybdenum content. This was also examined in the normal hot rolled and air cooled condition, as was another steel to the En.30b specification but with a higher molybdenum content than the original En.30b steel. The compositions are given below:

Steel	C%	Si%	Mn%	S%	P%	Ni%	Cr%	Mo%
En.30a	0.29	0.26	0.57	0.22	0.030	3.85	1.37	0.06
En.30b	0.32	0.29	0.47	0.20	0.022	4.13	1.21	0.30

TENSILE RESULTS

Conventional unnotched tensile tests were carried out on both En.30b steels both in the as-quenched and air cooled conditions where appropriate and also after tempering for 1 hour at 200°C. The results are shown in Table I.

The effect of both the rapid re-austenitising and ausforming treatments was to raise the proof and tensile strengths with little sacrifice of ductility. This is in accordance with previous experience[8,12].

FRACTURE CHARACTERISTICS

The initial investigation into fracture characteristics was carried out on the rapidly re-austenitised En.30b steels using Charpy V-notch impact tests to British Standard 131 Part 2 1959. The results obtained at 20°C for both rapidly re-austenitised and conventionally treated En.30b steel in the as-quenched condition and after tempering at 200°C for 1 hour are given in Table II.

The higher impact absorbed energies recorded for the conventionally treated steel were a surprise in view of the considerable degree of grain refinement obtained by rapid re-austenitising as shown in Fig. 1.

The difference in energy absorbed was greater for the tempered steels and so a full transition curve was obtained for each. The results are shown in Fig. 2. It will be seen that below -50°C the rapidly re-austenitised steel had a higher resistance to fracture than the conventionally treated material but that above this temperature the reverse is true. The difference in the room temperature V-notch values is therefore due to the different shape of transition curve arising from the two treatments.

In order to assess whether this difference in room temperature impact behaviour persisted at all tempering temperatures samples of En.30b steel treated by both processes were tempered for 1 hour at temperatures up to 600°C and then impact tested. The results are shown in Fig. 3. It will be seen that the superiority of the conventionally heat treated specimens, when tested at 20°C, persisted up to a tempering temperature of 270°C but that at higher tempering temperatures the fracture resistance of the rapidly re-austenitised steel was superior even when tested at 20°C.

It was considered at first that both the tempering and testing temperatures may have had some effect on the relative mode of crack propagation after each type of treatment and so modified the grain refining influence of the rapid heat treatment. Fractographic studies showed, however, that at all testing temperatures and after each tempering treatment the cleavage facets, in the case of brittle fracture, and the ductile cusps, in the case of ductile fracture were always smaller with the rapidly re-austenitised specimen than with the conventionally heat treated ones. Some examples are shown in Figs. 4, 5 and 6. It thus appeared that the refinement of structure observed under all conditions did not always result in the rapidly re-austenitised steels having a higher fracture resistance than the conventionally treated ones.

MICROSTRUCTURAL EXAMINATION

A more rigorous examination of the microstructure was carried out by means of thin film election microscopy. Several parameters were measured, the prior austenite grain size, the martensite unit length and width and the length and width of "bundles" of martensite units.

Austenite grain sizes were measured by tempering for one hour at 500°C, polishing and etching in 4% picral and 1% teepol[14]. The maximum and minimum diameters of several grains from different areas of the same sample were measured and the mean diameter calculated.

A martensite unit is defined as the smallest unit clearly defined by sharp boundaries in a thin foil transmission electron

micrograph. It is probably the entity called a needle or plate in the terminology of Kelly and Nutting[15]. The misorientation at unit boundaries is usually very small but groups of units of similar orientation are contained within high angle boundaries. The groups within the nearest high angle boundary are termed "bundles". These are called sheaves of needles by Kelly and Nutting[15].

A total of 370 units were measured in different areas of a rapidly re-austenitised and quenched sample of En.30b steel and 180 units were measured in a conventionally heat treated steel. The lengths and widths of the corresponding bundles were also measured, the length parallel to the length of the units contained within a bundle and the width normal to the length.

Since, in the majority of cases, martensite deforms by slip[16] and in brittle fracture fails across rather than along martensite units, the unit width was considered to be one of the major structural features. Figs. 7 and 8 show how the area fraction of different unit widths differed with the two types of treatment. It is seen that, although the mean prior austenite grain size was refined by a factor of 5 the spread of unit widths was so great in each steel that only the coarsest unit widths were missing from the rapidly re-austenitised specimens.

The bundle lengths, and the unit lengths, were refined by a factor of four, i.e. similar to the refinement in prior austenite grain size and the bundle widths by a factor of three.

The microstructural observations thus confirmed the fractographic studies that a substantial measure of refinement of at least some of the parameters measured was achieved by the rapid re-austenitising.

ANALYSIS OF STRESS/STRAIN CURVES

In an attempt to understand why this refinement did not improve the fracture behaviour of the En.30b steel at all temperatures an examination was made of the stress/strain curves obtained under various conditions.

Armstrong et al[17] have shown that in steels with equiaxed grains the flow stress σ_f can be represented by

$$\sigma_f = \sigma_\varepsilon + k_f d^{-\frac{1}{2}}$$

where σ_ε is the friction stress for a given strain
k_f is a constant for a given boundary in a given steel. The larger the boundary the greater is k_f.
d is the martensite unit width or the martensite bundle width.

Cohen [18] confirmed that this type of relationship held for martensitic steels and found that a decrease in prior austenite grain size (related to bundle width) from 1 mm to 10 μm could raise yield stress by about 30 ton/sq.in. This would imply a value of k_f of about 3.

In Table I it is seen that a refinement of prior austenite grain size from 34 μm to 7 μm raises the proof stress of the martensite by 15 tons for the untempered steel and $18\frac{1}{2}$ tons after tempering at 200°C. These increases in proof stress are at least twice those which would be expected with a k_f of 3. Since the refinement of austenite grain size is greater than the refinement in any of the other parameters measured it follows that if any of the other parameters were used instead the discrepancy between the expected and observed increase in proof stress would be even greater.

Further evidence was obtained from an analysis of true stress-true strain tensile curves plotted from the tensile test results for conventionally and rapidly heat treated En.30b after tempering at 200°C for 1 hour. By comparing observed stress strain curves with the generalised expression derived by Phillips and Chapman[19].

$$\sigma_f = \sigma_i + k_f d^{-\frac{1}{2}} + K\varepsilon_p^{\,n}$$

where σ_i is the yield stress at $d^{-\frac{1}{2}} = 0$ and $K\varepsilon_p^{\,n}$ is a power term relating flow stress to true plastic strain ε_p it was possible to determine that the $k_f d^{-\frac{1}{2}}$ contribution to the proof stress was of the order of 11 ton/sq.in. for the conventionally heat treated steel and 19.5 ton/sq.in. for the rapidly re-austenitised one. The difference is 8.5 ton/sq in. which, although only approximate is again about half of the observed difference.

It is clear therefore that the strength increase on rapid re-austenitising is greater than can be accounted for by grain refinement alone and that this additional strength increment may be responsible for the different fracture behaviour.

DEFECT CONCENTRATION

One possible explanation for the additional strength increment is an increase in internal defect concentration postulated by Sastri and West[20]. They showed from a kinetic study of the austenite/martensite transformation that the defect concentration in the martensite was increased by a rapid austenitising treatment. An increased defect concentration would be expected to raise the proof stress by increasing the frictional stress and so raise the impact transition temperature by analogy with ferritic steels[5].

Some further evidence for this possibility was obtained from a study of the effect of tempering upon the hardness of En.30b steel treated by the two techniques. Fig. 9 shows that the hardness of the rapidly heat treated steel drops more rapidly with tempering temperature than the conventionally treated steel but that the difference in hardness remains constant at tempering temperatures above 200°C. It is above this tempering temperature that the fracture behaviour of the rapidly re-austenitised steel is superior to that of the conventionally treated steel.

With this evidence therefore it seems certain that the effect of rapid re-austenitising is both to refine the martensite structure and increase the defect concentration. The latter is annealed out at tempering temperatures above 200°C and hence the fracture behaviour of the rapidly re-austenitised steel is then governed by grain size and is superior to that of conventionally heated steel. Below this tempering temperature the high defect concentration impairs the impact resistance of the rapidly heat treated steel in ductile failure.

AUSFORMED STEELS

Figs. 10 and 11 show the results of Charpy V-notch impact tests on En.30b and En.30a steels ausformed and conventionally treated. The impact behaviour of the two steels in the conventionally treated condition is very similar but that of the ausformed steel with a higher molybdenum content is superior. This may be further evidence for the effect of alloy carbide precipitation on the properties of ausformed steel[21].

Electron fractographs are shown in Figs. 12, 13 and 14. These show, that, as with the rapidly re-austenitised steel the fracture cusps are much smaller than with the conventionally treated steels. An interesting feature of the fractographs shown in Figs. 12 and 14 is that the cusps become smaller with increasing tempering temperature over the range 400°C - 600°C in the case of the ausformed steels and there is evidence of carbide precipitation. The fracture appearance of the conventionally treated steel is not greatly affected by tempering temperature.

The role of carbide precipitation in changing the fracture appearance and improving the impact behaviour of ausformed steel has been discussed at length by Irani[22]. There is some grain refinement of the steel obtained by ausforming and this is also influenced by carbide precipitation as can be seen from the fact that the as-quenched impact absorbed energy of the ausformed En.30b is appreciably higher than that of the ausformed En.30a because of the effect of molybdenum. Microstructural examination confirmed that the influence of ausforming on the width of martensite units was also marginal (Fig. 15).

In the absence of initial carbide precipitation with low molybdenum contents there is probably also an inheritance of a high concentration of defects which could explain the observation in Fig. 11 that at low tempering temperatures the conventionally treated steel is marginally superior to the ausformed one. The behaviour of ausformed steel on increasing the tempering temperature is, however, dominated by carbide precipitation in the manner described by Irani[22].

CONCLUSIONS

1. Rapid re-austenitising of a medium carbon low alloy steel refined the prior austenite grain size by a factor of five, the resulting martensite bundle lengths by a factor of four and the bundle widths by a factor of three. The degree of refinement of the width of individual martensite units within a bundle was very small. Ausforming produced a similar small degree of refinement.

2. Both processes resulted in a refinement of the fracture appearance after Charpy impact tests and in an improvement in impact properties after tempering above 300°C. Temper embrittleness was also reduced by both processes.

3. Both processes also appeared to cause an increase in the frictional stress of the martensite. This was removed by tempering above 300°C and also appeared to be diminished in the case of ausforming by the presence of a strong carbide former.

ACKNOWLEDGEMENTS

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TABLE I

MECHANICAL PROPERTIES OF RAPIDLY RE-AUSTENITISED AND AUSFORMED EN.30b

Treatment	Tempering Temperature °C	0.1% Proof Stress t.s.i.	Tensile Strength t.s.i.	Elongation on 2in.G.L. %	Reduction of Area %
Conventional	-	68.6	122.0	15.7	49.5
Rapid R-A	-	83.4	137.8	10.0	43.9
Ausformed	-	64.2	123.1	13.5	33.2
Conventional	200°C	78.2	106.7	19.4	61.2
Rapid R-A	200°C	96.8	118.4	11.4	59.7
Ausformed	200°C	80.0	121.4	13.5	42.0

TABLE II

CHARPY IMPACT TEST RESULTS ON En.30b STEEL

(a) As-quenched condition

	Conventionally Treated 850°C ½ hour Water Quench		Rapidly Re-austenitised Water Quenched	
	Hardness of Charpy Specimen HV/30	Impact Energy to Fracture Ft/lb	Hardness of Charpy Specimen HV/30	Impact Energy to Fracture Ft/lb
	561	13.4		
	560	14.5	541	12.5
	549	13.8	593	12.5
	566	14.0	583	12.8
	562	15.5	578	12.6
Mean	559	14.3	574	12.6

(b) Quenched and Tempered 200°C 1 hour

Conventionally Treated 850°C ½ hour, Water Quenched, Tempered		Rapidly Re-austenitised Water Quenched, Tempered	
Hardness of Charpy Specimen HV/30	Impact Energy to Fracture Ft/lb	Hardness of Charpy Specimen HV/30	Impact Energy to Fracture Ft/lb
487	33.0	543	20.0
496	33.5	537	23.0
487	33.5	517	24.5
476	35.0	496	24.0
478	33.0	490	26.5
		508	25.0
Mean 485	33.6	515	24.6

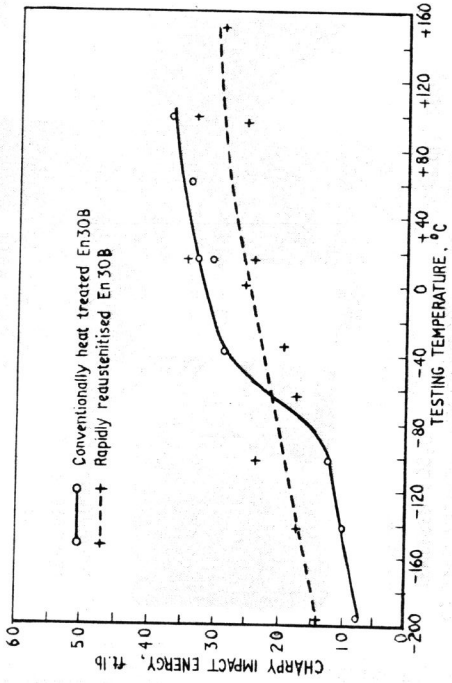
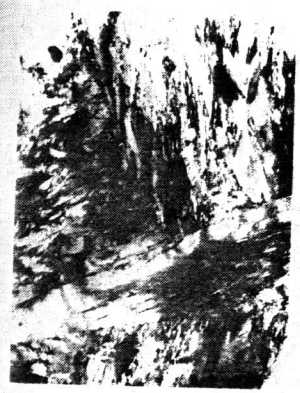


FIG. 2 CHARPY IMPACT ENERGY TRANSITION CURVES
En30B TEMPERED 200°C ONE HOUR



MARTENSITE OBTAINED BY CONVENTIONAL HEAT TREATMENT
7,600X

REFINED MARTENSITE OBTAINED BY RAPID HEAT TREATMENT
19,000X

FIG.1

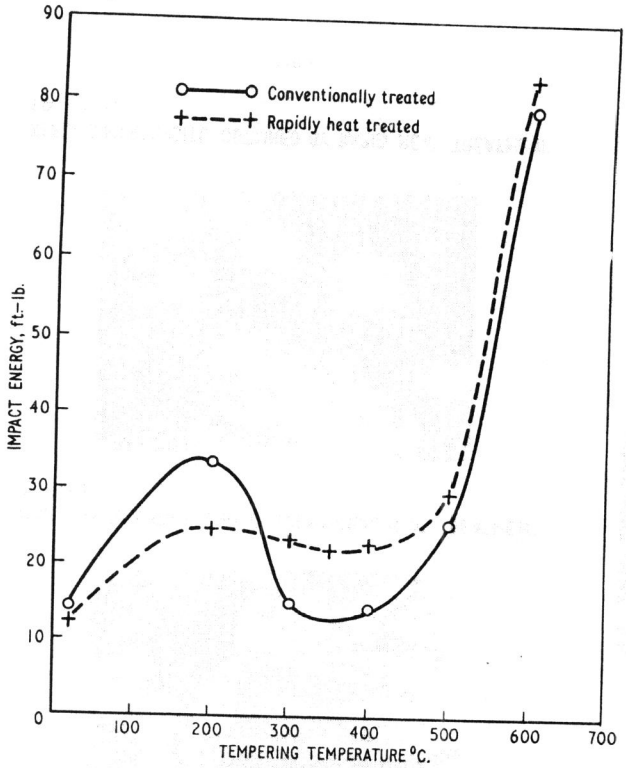
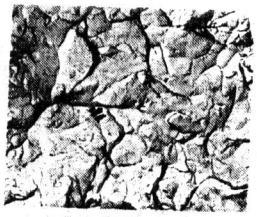
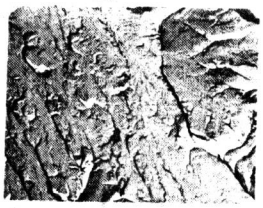


FIG.3 EFFECT OF TEMPERING ON V-NOTCH CHARPY IMPACT ENERGY

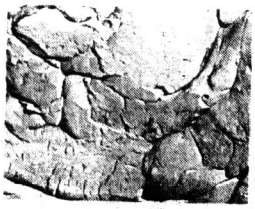
a) CONVENTIONALLY TREATED AND TEMPERED 200°C
1,300X



b) RAPIDLY TREATED AND TEMPERED 200°C
1,300X



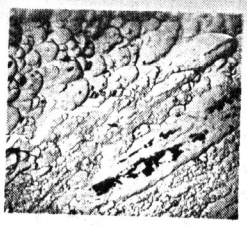
c) CONVENTIONALLY TREATED AND TEMPERED 200°C
2,500X



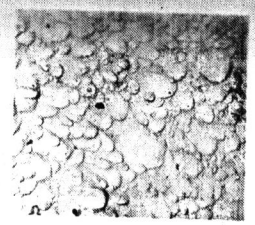
d) RAPIDLY TREATED AND TEMPERED 200°C
3,200X

ELECTRON MICROGRAPHS OF DIRECT CARBON REPLICAS FROM EN 30 B CHARPY SPECIMENS FRACTURED AT -196°C

FIG. 4

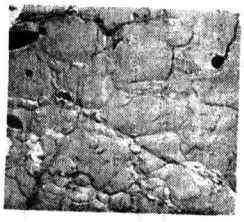


a) CONVENTIONALLY HEAT TREATED EN30B AS QUENCHED.
DIRECT CARBON REPLICA
1,300X

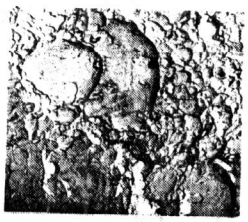


b) RAPIDLY REAUSTENITISED EN30B AS QUENCHED.
DIRECT CARBON REPLICA
1,300X

(CHARPY SPECIMEN FRACTURED AT R.T.)



c) CONVENTIONALLY HEAT TREATED AND TEMPERED EN30B.
DIRECT CARBON REPLICA
1,900X



d) RAPIDLY REAUSTENITISED AND TEMPERED EN30B.
DIRECT CARBON REPLICA
1,900X

(CHARPY SPECIMEN FRACTURED AT R.T.)

FIG. 5

a) CONVENTIONALLY TREATED AND TEMPERED 200°C
1,260X

b) RAPIDLY TREATED AND TEMPERED 200°C
1,260X

FIG.6 ELECTRON MICROGRAPHS OF DIRECT CARBON REPLICAS FROM EN 30B CHARPY SPECIMENS FRACTURED AT +100°C

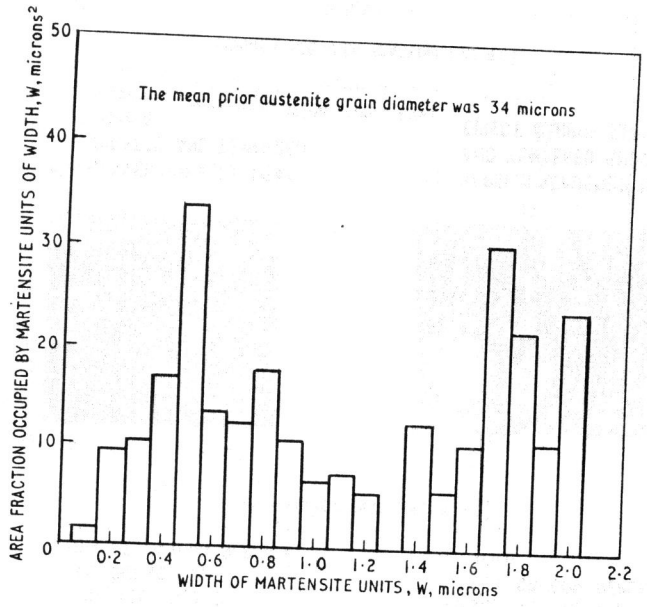


FIG.7 DISTRIBUTION OF AREA OCCUPIED BY MARTENSITE UNITS OF A GIVEN WIDTH FOR CONVENTIONALLY HEAT TREATED En 30 B STEEL

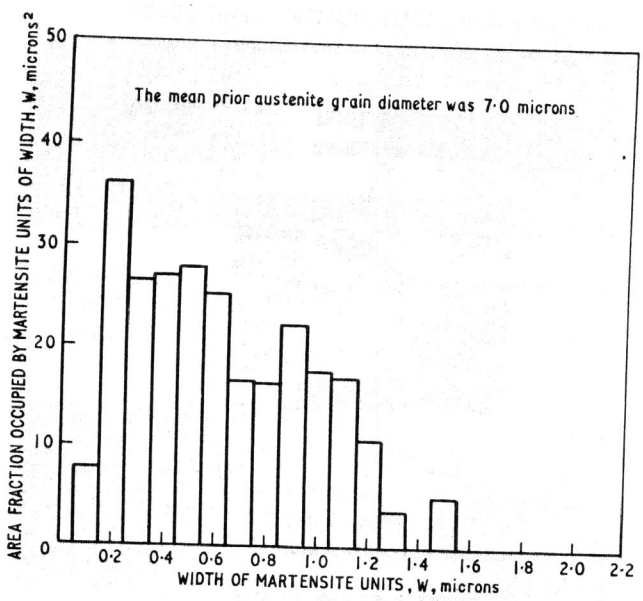


FIG.8 DISTRIBUTION OF AREA OCCUPIED BY MARTENSITE UNITS OF A GIVEN WIDTH FOR RAPIDLY REAUSTENITISED En 30 B STEEL

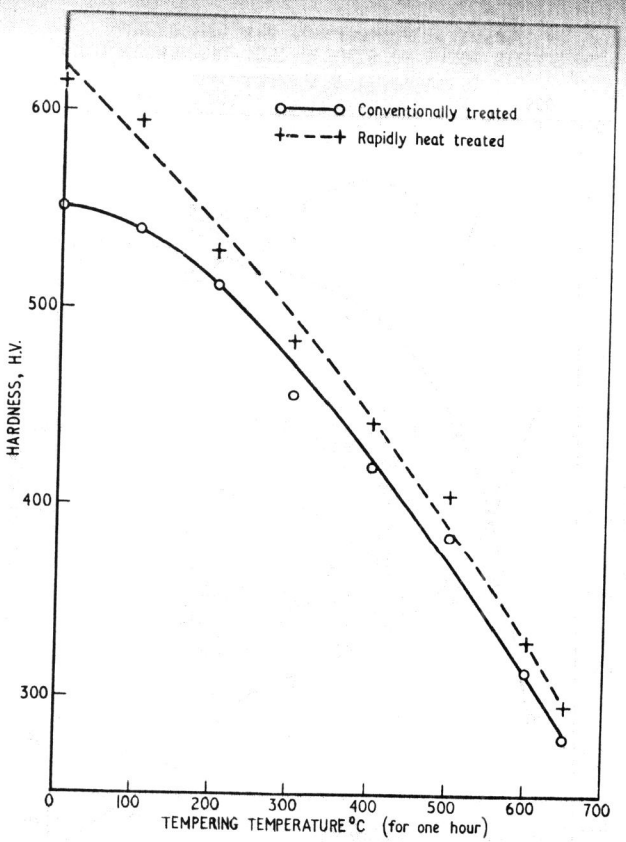


FIG.9 CHANGE OF HARDNESS ON TEMPERING CONVENTIONALLY AND RAPIDLY HEAT TREATED En30 B

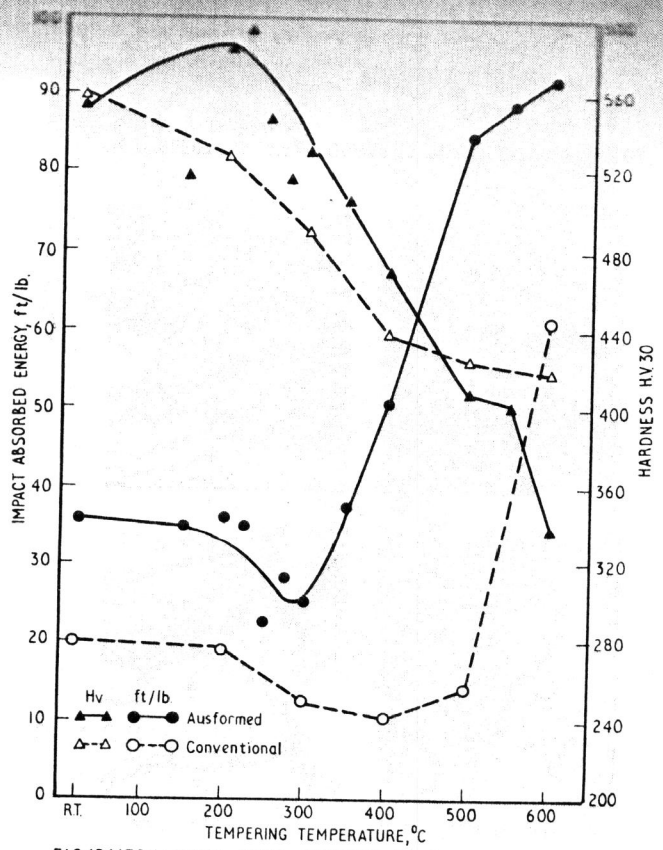


FIG.10 MECHANICAL TEST RESULTS ON En 30B STEEL AUSFORMED AND CONVENTIONALLY TREATED

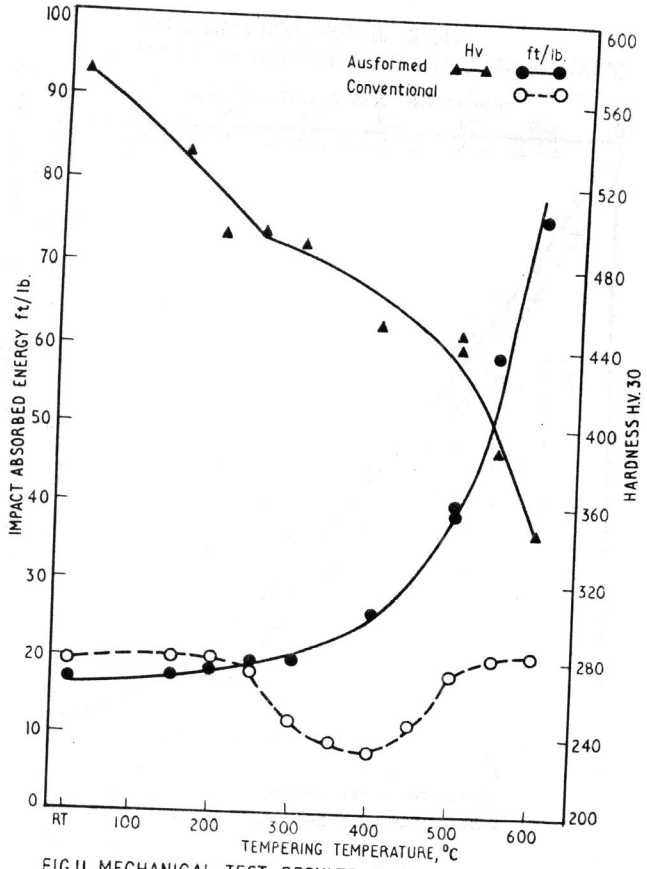


FIG.11 MECHANICAL TEST RESULTS ON En30A STEELS, AUSFORMED AND CONVENTIONALLY TREATED

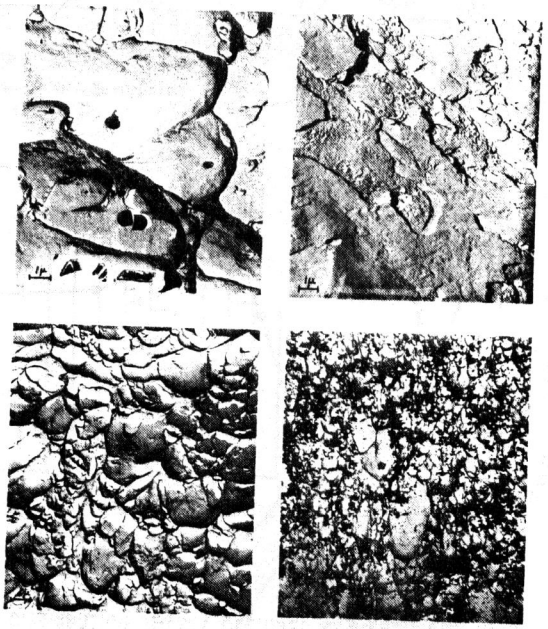


FIG.12 ELECTRON FRACTOGRAPHS OF AUSFORMED En.30B

a. Untempered 3,200X
b Tempered for 1hour at 300°C. 3,200X
c. Tempered for 1hour at 400°C. 3,200X
d. Tempered for 1hour at 600°C. 3,200X

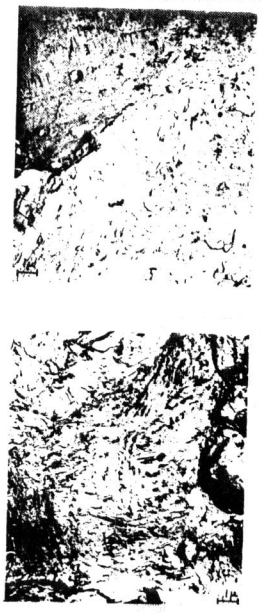


FIG. 13 ELECTRON FRACTOGRAPHY OF CONVENTIONALLY TREATED En30B

a. Tempered for 1hour at 400°C. Electron Fractograph 3,200X
b. Tempered for 1hour at 600°C. Electron Fractograph 3,200X

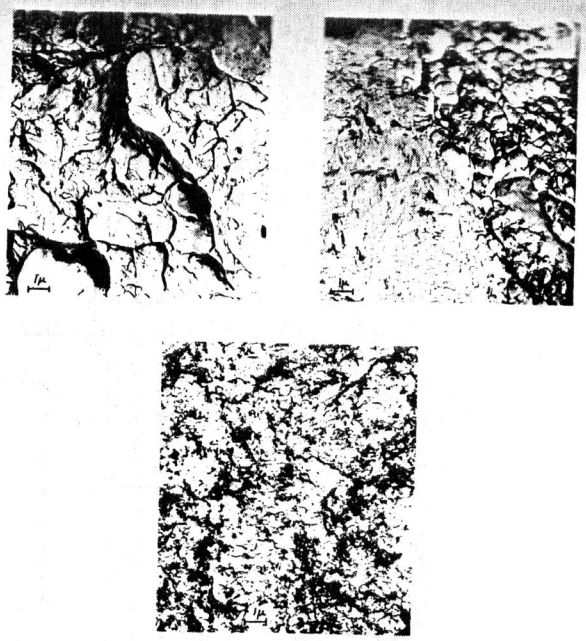


FIG. 14 ELECTRON FRACTOGRAPHY OF AUSFORMED En30A

a. Untempered Electron Fractograph 3,200X
b. Tempered for 1hour at 400°C. Electron Fractograph 3,200X
c. Tempered for 1hour at 500°C. Electron Fractograph 3,200X

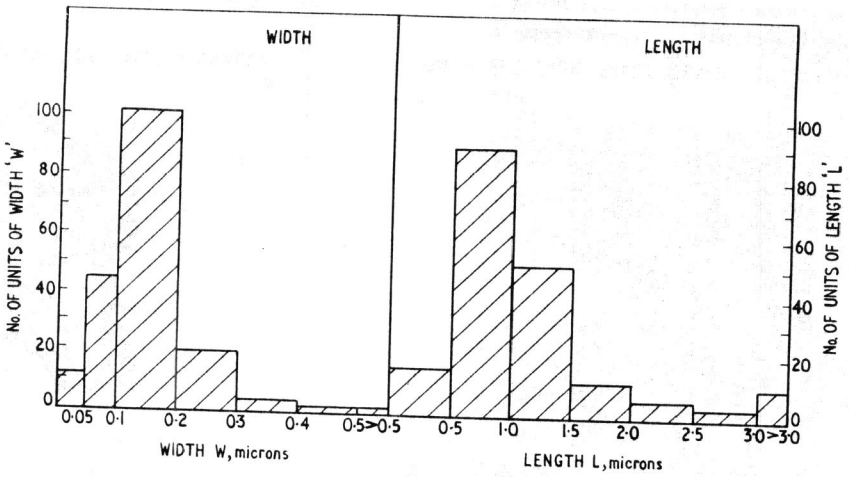


FIG. 15 HISTOGRAM PLOTTING THE DISTRIBUTION OF MARTENSITE UNIT WIDTHS AND LENGTHS IN AUSFORMED En 30 B STEEL