

used,  $\sigma$  the angular range in  $2\theta$  over which the intensity distribution is appreciable and  $n$  is the order of a reflection), for two orders of the same reflection, would give a straight line whose slope and intercept would be proportional respectively to the mean square strain and the reciprocal of the apparent particle size in the corresponding direction. Sometimes one can take different sets of ( $W$ ,  $\sigma$ ) values, estimate the particle size and strain for each set and obtain an average value for these quantities through a least square fit.

The variance method was applied to the case of a copper-silicon-manganese alloy (containing about 6.7 at. % silicon, 1.3 at. % manganese, rest copper) possessing a face-centred-cubic structure. Copper  $K\alpha$  radiation and a Siemens diffractometer with a scanning speed of  $\frac{1}{8}^\circ \text{ min}^{-1}$  in  $2\theta$  were employed to record the diffraction patterns of the cold-worked and annealed filings. Variance measurements were carried out on the  $\{111\}$  and  $\{222\}$  profiles for one set of ( $W$ ,  $\sigma$ ) values. (The very weak cold-worked  $\{400\}$  reflection precluded any accurate studies along the  $\langle 100 \rangle$  direction.) The variance due only to deformation was obtained by subtracting the variance of the annealed sample from that of the cold-worked one [6].

The value of the apparent particle size and strain obtained from the linear plot were 98 Å and 0.0018 respectively. This can be compared with the values obtained for the effective domain

size and strain by Fourier analysis [7] and integral breadth [8] measurements on the same alloy for the same direction (viz.  $\langle 111 \rangle$ ) which were 97 Å, 0.0025 and 163 Å, 0.0041 respectively. It can be seen that the values obtained by variance analysis are closer to those given by Fourier analysis than by integral breadth measurements.

### Acknowledgement

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### References

1. M. TOURNARIE, *C.R. Acad. Sci. Paris* **242** (1956) 2016; 2061.
2. A. J. C. WILSON, *Proc. Phys. Soc.* **80** (1962) 286.
3. *Idem, ibid* **81** (1963) 41.
4. *Idem*, in "Advanced Methods of Crystallography", edited by G. N. Ramachandran (Academic Press, London and New York, 1964) p. 221.
5. G. B. MITRA, *Acta Cryst.* **17** (1964) 765.
6. N. C. HALDER and G. B. MITRA, *Proc. Phys. Soc.* **82** (1963) 557.
7. R. VASUDEVAN, to be published, (1969).
8. *Idem, J. Mater. Sci.* **4** (1969) 1116.

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### Etching of Fracture Surfaces

In the course of a fractographic investigation of fatigue in a 0.1% plain carbon steel it became necessary to devise a technique that would enable the principal features of the fracture surface to be related to the underlying microstructure.

In addition to taper-sectioning, an etching technique was adopted. The fracture was immersed in a solution of 2% nitric acid in ethyl alcohol for progressively increasing times and was then examined on a Cambridge Stereoscan scanning electron microscope. Before etching, a composite photograph of an area of the fracture was produced at a magnification of 2400 times and this same area was re-examined in detail after each etching step.

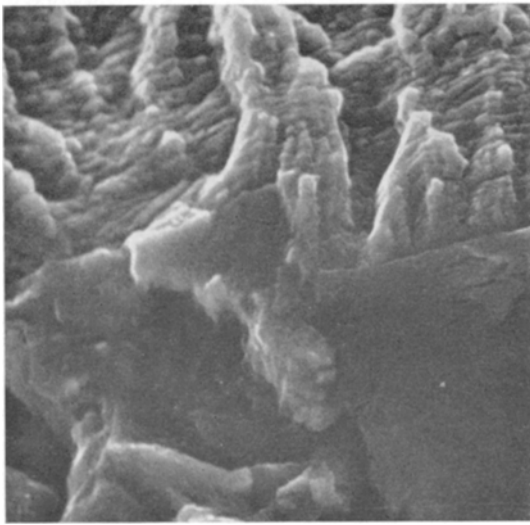
Pearlite colonies were revealed after 20 sec and their numbers increased with longer etching times as indicated in table I. Up to 100 sec, little

damage occurred to the fractographic features, but after 300 sec general attack was obvious, although isolated areas remained unetched.

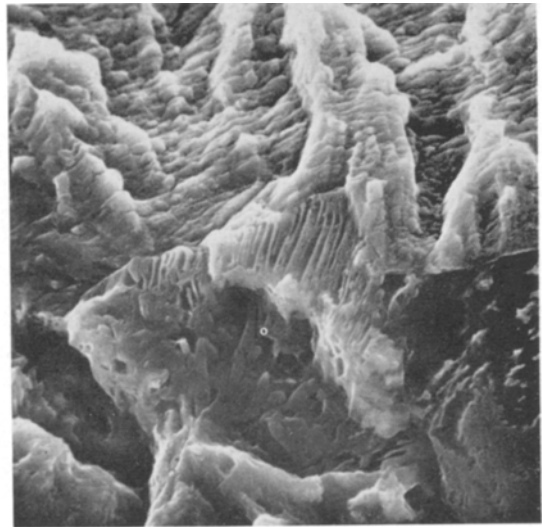
Fig. 1a shows an enlarged area of the composite photograph before etching, and figs. 1b and c show the same area after etching times of 30 and 300 sec respectively. The pearlite colony in figs. 1b and c clearly lies in the surface of the

TABLE I

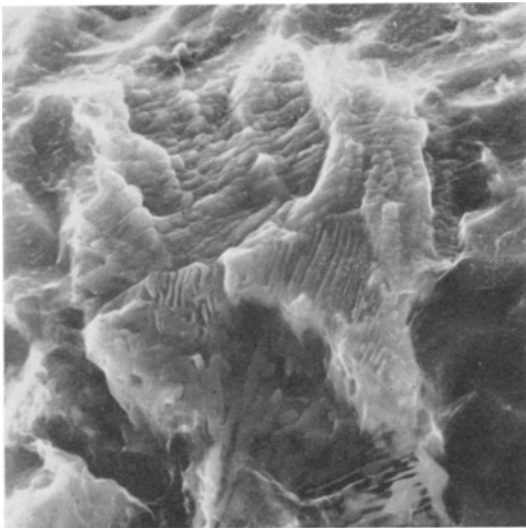
Etching time, sec	Number of pearlite colonies observed	Estimated % pearlite
As-fractured	0	0
10	0	0
20	2	less than 0.5
30	5	less than 0.5
50	18	1.5
70	34	3.0
100	48	4.0
300	166	12.0



(a)



(b)



(c)

Figure 1 Scanning electron micrographs of an area of fatigue fracture: (a) before etching ( $\times 4125$ ), (b) after 30 sec etch ( $\times 4125$ ), (c) after 300 sec etch ( $\times 4125$ ).

discrepancy may be due to pearlite colonies of certain orientations remaining unetched. Alternatively, crack propagation may not occur through the pearlite. Sections taken through the fracture will elucidate this point.

The technique described above, used in conjunction with taper-sectioning of fracture surfaces, can clearly provide useful information on the relationship between the fracture path and microstructure.

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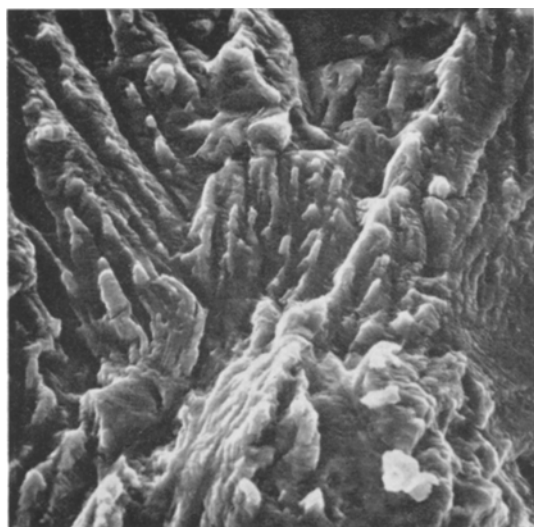
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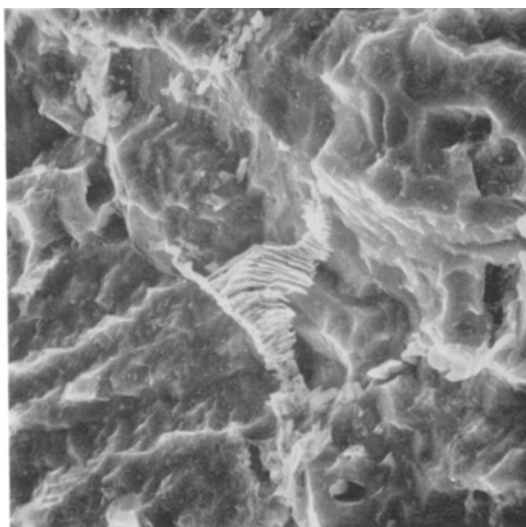
fracture and it is interesting to note the colony is not well-defined in the original fracture surface (see fig. 1a). Fig. 2a shows an area of fracture before etching and fig. 2b shows the same area after a 300 sec etch. In this case most of the original fractographic detail had been removed before the colony became apparent.

The expected pearlite content for this material as established by quantitative metallography on polished and etched microsections is about 12%. Estimates of the quantity of pearlite observed after 100 sec etching time (i.e. before removal of fractographic detail) suggest a figure of 4%. This

See over for Fig. 2

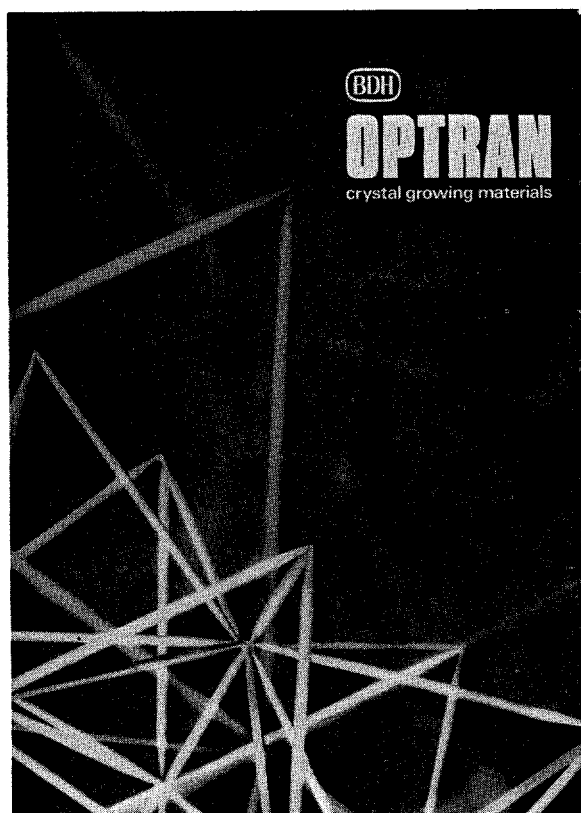


(a)



(b)

Figure 2 Scanning electron micrographs of an area of fatigue fracture: (a) before etching ( $\times 1650$ ), (b) after 300 sec etch ( $\times 1650$ ).



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