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2 December 1966

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Reply to the Discussion (J. Matls. Sci. 2 (1967) 196*) on the Letter "Grain Boundary 'Pest' in the Intermetallic Compound NiAl" (J. Matls. Sci.* 1 (1966) 113*)*

The purpose of our letter was to propose a mechanism for the occurrence of the pest, in NiAl containing 54 at. % Al, in terms of the observation of precipitates at the grain boundaries which underwent a morphological change on exposure to the atmosphere at room temperature. Grain-boundary hardening was mentioned because it and the pest appear to be related, since the pest is found in materials which also exhibit grain-boundary hardening. However, this does not mean that the pest is a natural consequence of grain-boundary hardening, and we do not know of any evidence in the literature which suggests that the NiAl composition used by Seybolt and Westbrook (51.3 at. % Al) is susceptible to the pest. Seybolt and Westbrook have not attempted, even in their discussion of our letter, to account for the pest phenomenon in terms of their proposed grain-boundary hardening mechanism.

We have in fact obtained microscopic evidence for progressive oxygen penetration down a grain boundary during pesting. Pested samples were examined after removal of successive layers from the surface exposed to air. We found that the longer the exposure time the greater was the depth of the layer which had to be removed before unoxidised particles were detected at the boundary. We consider that Seybolt and Westbrook's proposal, that the grain-boundary softening which occurs when the particles oxidise is due to a local impoverishment in dissolved oxygen, is unlikely; because softening which is detectable by the microhardness technique would require massive matrix diffusion in relatively short times at room temperature.

In the light of the experimental evidence available for NiAl containing 51.3 and 54 at. % Al, it is not unreasonable to conclude that the operative grain-boundary hardening mechanism depends upon composition (this may, for example, lead to an uptake of different concentrations of oxygen on heat treatment, which could be present as either oxide and/or solute), and, in the latter composition, that both the hardening and the pest result from the presence of grain-boundary precipitates. This view is supported by the fact that hardness traverses across grain boundaries, at points where no precipitates were observed, did not reveal hardening.

The important point to be clarified by further studies is whether pesting occurs only when precipitates, which undergo the type of transformation observed in NiAl, are present at grain boundaries; or, alternatively, whether alloys which exhibit grain-boundary hardening in the absence of grain-boundary precipitation are also susceptible to the pest.

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A Method for the Etching of Pyrolytic Silicon Carbide

Silicon carbide prepared by the pyrolytic decomposition of methyltrichlorosilane vapour upon uranium carbide spheres in a fluidised bed has variations in structure dependent upon deposition rate. To examine this in detail, a suitable etch was required. The following single-stage electrolytic etches were tried; some of which stemmed from previous authors: (i) 20% KOH, aqueous [1]; (ii) 10% "chromic acid" ($CrO_3/H_2SO_4/H_2O$) [2]; (iii) 1:3:6 $HClO_4/CH_3COOH/H_2O$ [3]; (iv) 10% K_2CO_3/10% $NaNO_3$, aqueous [4]; (v) 10% oxalic acid, aqueous [5]; (vi) 1:1:1 $CH_3COOH/HF/H_2O$. None of these was satisfactory because each produced gross surface scratching on the SiC – mainly by the opening up of hairline polishing scratches, and also from the running together of etch pits. Also, many coats cracked in half as a result of stress corrosion. A two-stage etch was therefore devised.

Stage 1 The ground and polished specimens, mounted in "Ceemar" resin, were attack polished for 15 to 20 min using a slurry of freshly prepared green chromium oxide (by igniting AR ammonium dichromate crystals) in 100 ml water and five drops of 0.002% aqueous HF. The slurry was flowed over a rotating "Selvyt" pad enclosed in a polythene bowl on a Beck polishing machine.

Stage 2 After washing, the specimens were electrolytically etched in one of the above solutions at 3 to 5 V and 1 A for 4 to 5 min using a stainless-steel gauze cathode. The anode was a stainless-steel gauze strip held in contact with the SiC by a pair of stainless-steel pincers, which was joined to the circuit wire with an alligator clip.

Two types of SiC coat were investigated.

Type A These coats had transparent, homogeneous, smooth-surfaced structures. Etching with stage 1 revealed the presence of radial grains in the section (fig. 1); stage 2 produced strong delineation of the grains and circumferential "tree-ring" twinning (fig. 2). This may be a form of deformation twinning (since the coats showed evidence of strain, as was revealed by their shattering upon mechanical crushing), or perhaps growth twinning associated with stacking faults.

Type B These coats, produced at higher methyltrichlorosilane pressures, had layered, partially opaque structures with blastular outer surfaces. (Embryol. Blastula: a single layer of cells arranged spherically around a central cavity [6].) They resembled a ball covered with tiny balls – as in a raspberry. These coats, when sectioned and polished, resembled certain layered, geological deposits under dark-field microscope examination. The first stage of the etch caused no definite visible changes, such as grain relief, but the second stage produced

Figure 1 Two-stage etch (stage 1). Smooth SiC coat. Nomarski interference contrast. Deposition direction – see arrow. (×610 – reduced from ×780 for reproduction)

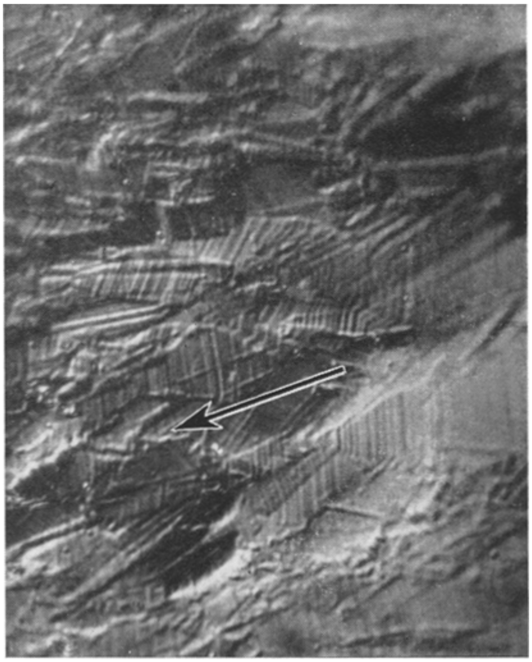

Figure 2 Two-stage etch (stage 2). Smooth SiC coat. Nomarski interference contrast. Deposition direction – see arrow. (×750 – reduced from ×1020 for reproduction)

innumerable circumferential striations – many more than in the unetched sample (fig. 3). An electron microscope study of these structures will be reported later.

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Figure 3 Two-stage etch (stage 2). Blastular SiC coat. Nomarski interference contrast. Deposition direction – see arrow. (×610 – enlarged from ×540 for reproduction)

Book Reviews

Strength of Materials

P. Black

Pp 454 (Pergamon Press, 1966) 45*s*

This is an excellent book, well-suited to the requirements of the students for whom it was written. A particularly valuable feature is the excellent coverage of first and second moments, a part of the syllabus which students often find difficult. The book may also prove useful to first-year undergraduates studying electricity or electronics as a main subject, who sometimes find "strength of materials" hard-going.

The chapter on beam deflections could be considerably shortened by introducing Macaulay's method almost at the beginning, since all the examples shown can be done by this means. There is no chapter on practical stress analysis; for the level at which the book is aimed, this is probably a wise omission.

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