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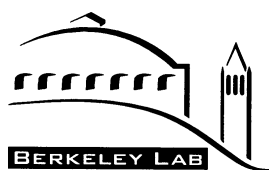
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ELECTRON MICROSCOPY IN OPTIMIZING MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HOT-PRESSED SILICON CARBIDE

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SiC, as one of the most promising candidate ceramics for high temperature structural applications, offers many intrinsic advantages, including high melting temperatures, low density, and high elastic modulus. However, the use of SiC to date has been severely limited by its poor fracture toughness ($\sim 2\text{-}3 \text{ MPa}\sqrt{\text{m}}$ for commercially available materials) and crack-growth resistance. Our study focuses on the development of silicon carbide as a potentially tough, high-temperature, and damage-tolerant material. The approaches include identifying roles of sintering additives in modifying grain morphology, effects of post-annealing on grain boundary phases, and possibility in introducing nanoscale precipitates in SiC grains. Central in these efforts is structural characterization using state-of-the-art electron microscopy.

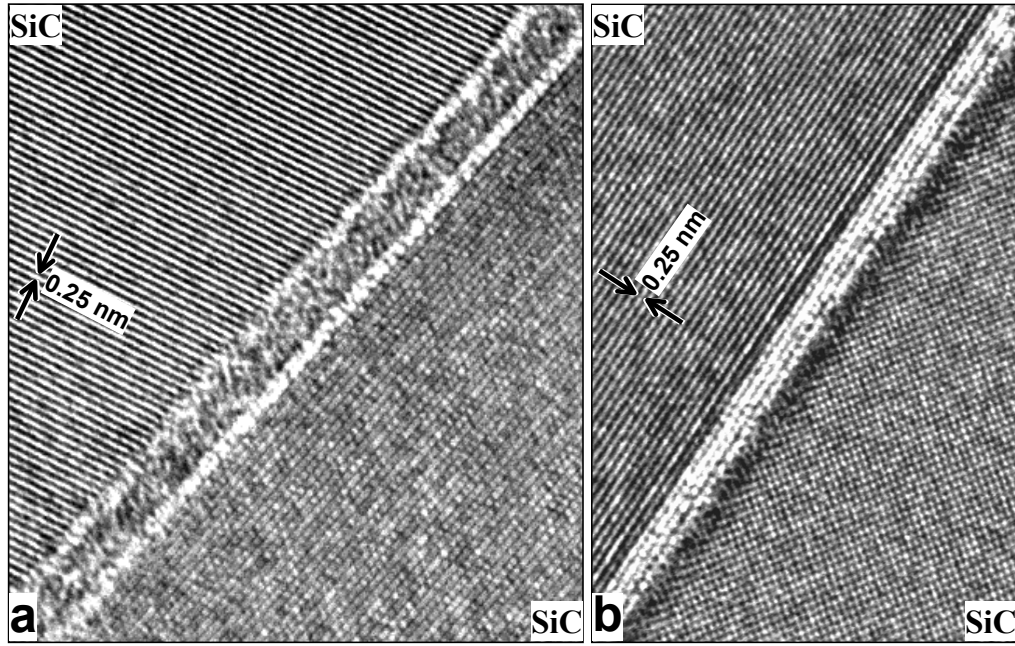
The first success was *in situ* toughening the SiC, by hot pressing in the presence of Al, B and C additions. Elongated and interlocked grains were developed surrounded by an Al-containing amorphous grain boundary film. Electron microscopy identified the different roles of Al, B and C additives in promoting grain elongation and the associated phase transformation. A fracture toughness of up to $9 \text{ MPa}\cdot\text{m}^{1/2}$ was achieved as a result of elastic bridging and frictional pullout of the interlocking grains in the process of intergranular fracture.¹

The grain boundary films in as-hot-pressed SiC were characterized using high-resolution electron microscopy (HREM) and quantitative X-ray energy-dispersive spectroscopy (EDS). Amorphous intergranular layers, typically 0.7 to 3 nm wide, were observed (Fig. 1a), with Al-O-Si-C constituents. Oxygen was from SiO₂ layers on the surface of the SiC starting powders. Quite remarkably, it was found that heat treatment at or above 1000 °C effectively crystallized the amorphous grain boundary films (Fig. 1b). Quantitative EDS analyses found Al site densities in the grain boundary films to be consistent with aluminosilicate or alumina-derived phases, which were further confirmed by detailed HREM study.² The microstructural changes in interfacial phases dramatically improved the performance in various mechanical tests at room, and at elevated temperatures.

In a different approach to strengthen SiC, uniformly dispersed, nanoscale precipitates were induced interior SiC grains through prescribed post-annealing. HREM and nano-probe EDS chemical analysis identified a crystalline structure with an Al-C-B-Si composition for the precipitates (Fig. 2).³ A positive effect of the nano-precipitates on wear resistance of the SiC was resulted.

References

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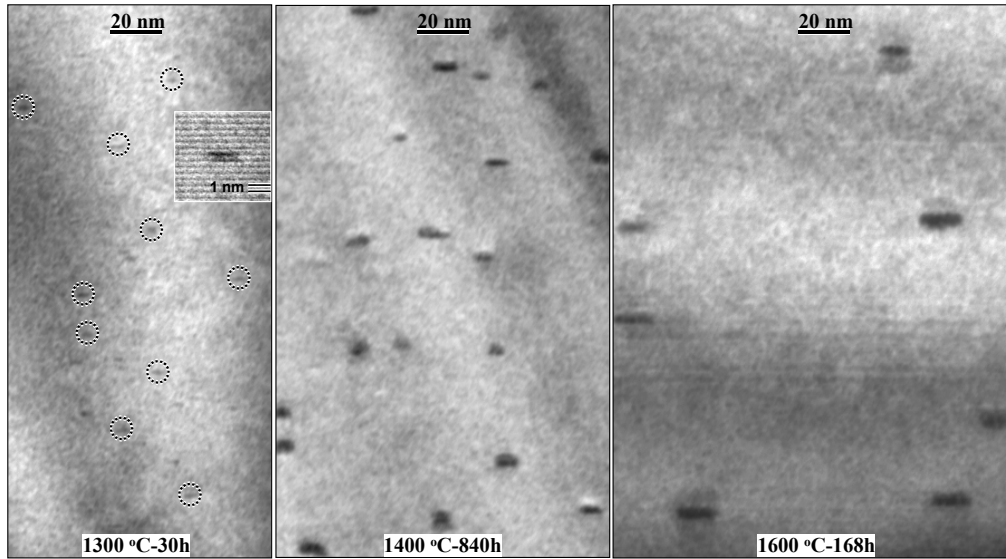


FIG. 1 (a) An amorphous grain boundary film in as-hot-pressed SiC. (b) A crystallized grain boundary film after post-annealing at 1000 °C for 5 hrs.

FIG 2 Micrographs showing nano-precipitates formed in SiC after annealing at 1300 °C, 1400 °C, and 1600 °C, respectively. Some precipitates are circled in image for 1300 °C-annealed SiC, and the inset shows an enlarged precipitate.