

Novel Microstructures Produced by Flash Sintering LaPO₄/Al₂O₃ Composites

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The material system LaPO₄/Al₂O₃ has promise as a high fracture toughness material because it resists crack propagation by interfacial debonding, as demonstrated by Al₂O₃ fibers when coated by LaPO₄^[1]. Sintering Al₂O₃ and LaPO₄ typically requires a furnace temperature of over 1600°C for hours. A faster and more economical method to lower energy consumption and manufacturing cost is flash sintering^[2]. Flash sintering applies an electric field and controls current flowing through a freestanding pressed powder sample, lowering the temperature and shortening time to sinter to seconds or minutes, providing an economical route to fabricate ceramics. Moreover, rapid heating to high temperatures during the flash and fast cooling can provide unique non-equilibrium microstructures and compositions^[3].

Experiments were conducted with specific volume ratios of LaPO₄/Al₂O₃ to study the impact of flash sintering on the composite microstructure. Mixed powders were pressed into dog-bone shaped samples and placed in a tube furnace, with the sample connected to a power supply through Pt electrodes. At 1450°C, the applied voltage (250 kV/cm) on the sample was then increased up to 1000 kV/cm to induce the flash phenomenon. After the flash onset, the DC field was held for 10 to 30 seconds, with a current limit of 2-25 mA/mm² before it was turned off. Imaging was conducted on the top surface both before and after polishing, and a cross-section of the heat-affected zone was imaged with secondary electron (SE) and backscatter electron (BSE) imaging (FEI Magellan) and bright field transmission electron microscopy (TEM, Philips CM20). Phase identification and orientation relationships were determined by energy dispersive x-ray spectroscopy (EDS, FEI Magellan), electron backscatter diffraction (EBSD, Gaia Tescan), and transmission kikuchi diffraction (TKD, FEI Quanta).

Figures 1 - 6 show the range of unique microstructures found in these flash sintered composite samples. Temperatures in the core were high enough to achieve melting with eutectic-like microstructures formed (Fig. 1). Regions further away from this channeling effect had a more typical structure of polycrystalline grains of the two phases (Fig. 1(d)). Both lamellar (Fig. 1(c)) and anomalous (Fig. 1(b)) eutectic structures were observed. Due to the non-wetting properties of monazite, the monazite lamellae prefer to minimize interfacial area by pinching off and balling up into spherical grains. In other regions in the core, large grains of both phases are observed. LaPO₄ grains do not show any orientation preference, but the alumina tend to form hexagonal grains (Fig. 2) with (0001) surfaces and faceting along the prism planes, as found by EBSD. TKD showed that nearby eutectic lamellae of alumina have the same orientation as the large alumina single crystals. This study demonstrates that flash sintering can provide unique microstructures for improved mechanical properties.

References:

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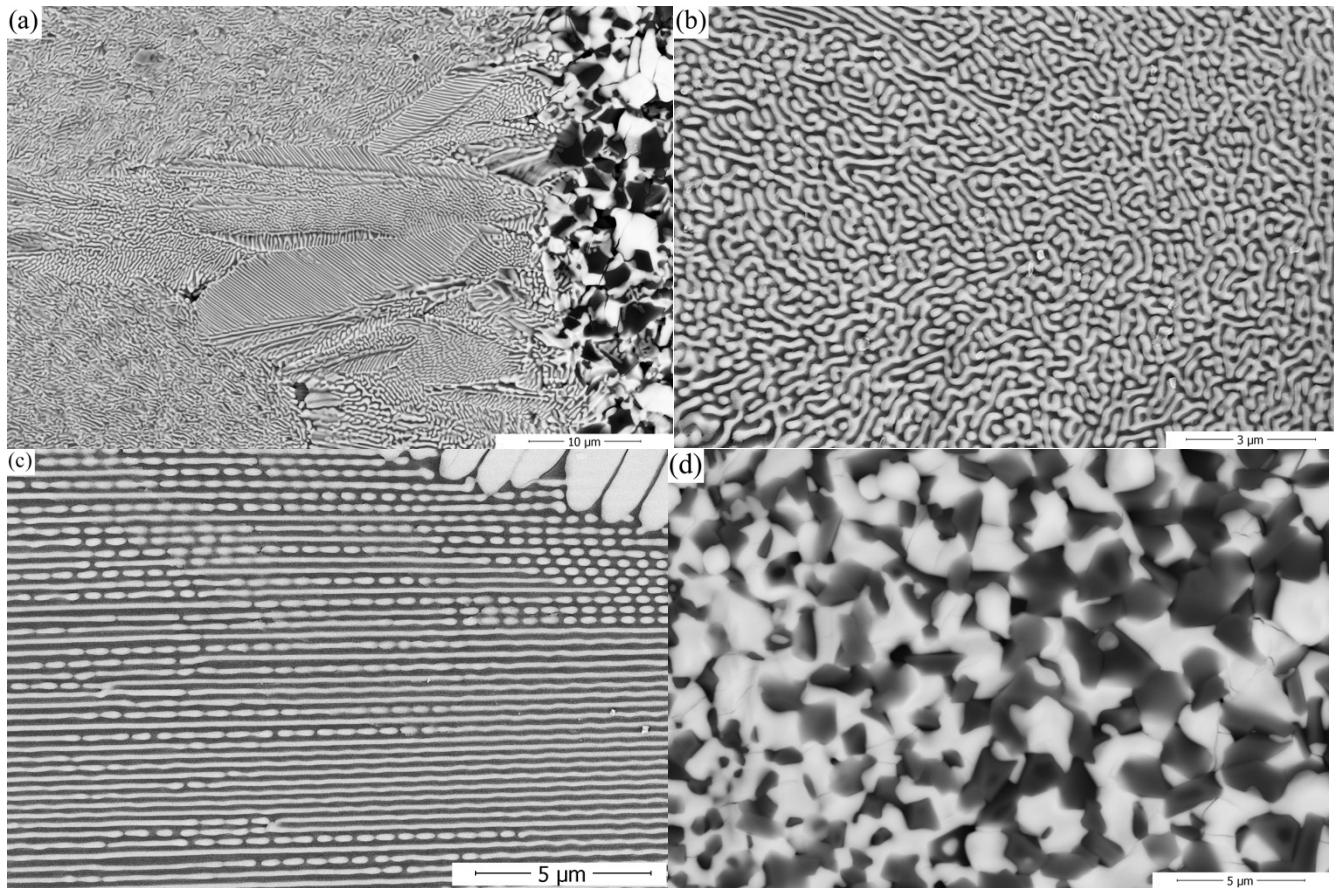


Figure 1. Unique microstructures on the surface of flash sintered LaPO_4 (light phase) and Al_2O_3 (dark phase) composite are shown by BSE images: (a) both lamellar and anomalous eutectic-like structures in the core, and conventional polycrystalline morphology outside of the core; (b) anomalous eutectic-like structures that lack prominent aligned orientations; (c) highly directional lamellae grains with pinched-off LaPO_4 subgrains; (d) conventional polycrystalline grains away from the heat-affected zone.



Figure 2. BSE image of an alumina hexagonal grain with monazite entrappedments, surrounded by large monazite grains and eutectic-like structures.

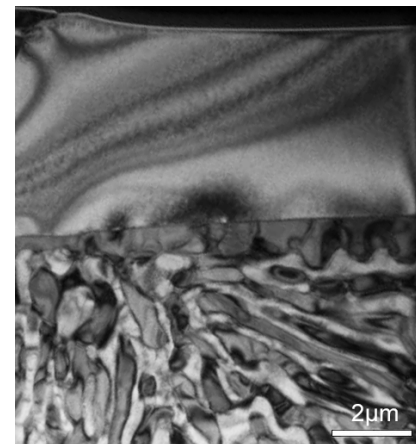


Figure 3. TEM bright field image of the microstructures. The TEM sample is milled perpendicular to one side of a hexagonal Al_2O_3 grain and normal to the sample surface.