

## Coordinated EBSD and TEM Analysis to Decipher Shock Deformation Effects in the Oldest Solids in the Solar System

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Calcium-Aluminum-rich Inclusions (CAIs), hosted in primitive meteorites, are the oldest solids in the solar system, e.g., [1]. The microtextures and crystallographic orientation of minerals in CAIs therefore have the potential to reveal the earliest events recorded in them. Optical microscopy, secondary electron and back-scattered electron imaging in the scanning electron microscope have provided a wealth of information on CAI textures. In comparison, electron backscatter diffraction (EBSD) coupled to an SEM can provide both textural and crystallographic information on CAI materials [2]. Here we report on the use of EBSD and transmission electron microscopy (TEM) to determine the composition and microstructure of CAIs in order to understand the effects of shock deformation.

The mineralogy of a compact type-A CAI (Hedgehog) from the Northwest Africa (NWA) 5028 CR2 carbonaceous chondrite was characterized using the CAMECA SX-100 electron microprobe at the University of Arizona (UA). Hedgehog is surrounded by a rim sequence made of a mono- to multi-mineralic layers. The interior of Hedgehog contains melilite  $[(Ca,Na)_2(Mg,Al)(Si,Al)_2O_7]$ , perovskite  $[CaTiO_3]$ , spinel  $[MgAl_2O_4]$ , and metal grains  $[Fe-Ni]$ . The rim sequence around Hedgehog contains spinel + hibonite  $[(Ca)(Al,Ti,Mg)_{12}O_{19}]$  + perovskite in the innermost layer, melilite in the intermediate layer and Ti-rich pyroxene  $[Ca(Mg,Ti,Al)(Al,Si)_2O_6]$  in the outermost layer.

The EBSD analysis was conducted using the FEI Helios NanoLAB 660 focused ion beam scanning electron microscope (FIB-SEM), equipped with EDAX EDS and EBSD detectors, located at the UA. The EBSD analysis shows that almost all melilite grains in the interior of Hedgehog are severely deformed as seen in the color variation in the inverse pole figure (IPF) maps (Figure 1). The misorientations in individual interior melilite grains are up to  $30^\circ$ . In comparison, the rim melilite and spinel grains are much less deformed, i.e., the fraction of melilite grains showing deformation is limited and the misorientations are  $<10^\circ$ . Spinel grains show a much lower degree of heterogeneous deformation.

Shock experiments conducted on spinel at room temperature show that it forms deformation twins [3]. We used methods described in [4] to identify spinel twins using the EBSD data, which suggest that both the interior and rims contain twinned spinel grains. However, in order to further confirm if the spinel twins were caused by shock deformation similar to the twin lamellae reported in experimentally shocked spinel [4], we selected a pair of spinel grains containing twins, as identified by EBSD, for further TEM investigation (Figure 2). We extracted and thinned the spinel grains to electron transparency with the FIB-SEM and analyzed it using the 200 keV spherical-aberration-corrected Hitachi HF5000 TEM located at UA. EDS elemental maps (Figure 3) and selected-area electron-diffraction (SAED) patterns (not shown) for the various mineral phases in the FIB section were acquired.

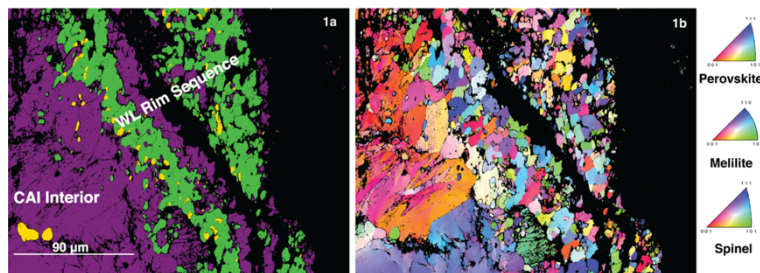
The FIB section contains an assemblage of spinel and melilite grains (Figure 3). There is a layer of unidentified Ca-rich material between spinel and melilite. SAED patterns show that spinel grains are

closely oriented with respect to each other. While the SAED patterns did show the minor orientation deviations, the twinning noted from the EBSD data was not confirmed as the algorithm for finding twins in EBSD covers a range of orientation to account for possible errors.

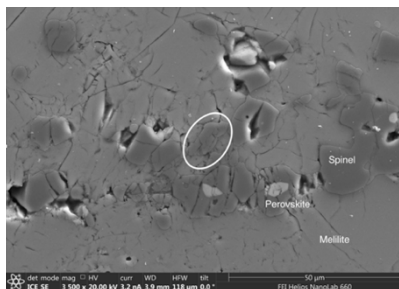
Deformation in CAI materials was previously observed using transmission electron microscopy (TEM) [5,6]. For example, melilite grains from several type-A and type-B CAIs show high dislocation densities, deformation lamellae, and low-angle grain boundaries indicating significant strain [5,6]. Further, in many of these CAIs, spinel grains do not show twinning, which also suggests that these minerals were already at high temperature when they were shocked (~1000 °C) [5,6]. Our results are consistent with the previous studies. The absence of deformation twinning in spinel from Hedgehog, but the presence of deformation lamellae in melilite and spinel suggests that this CAI experienced shock deformation in high ambient temperatures in a nebular environment [7].

### References:

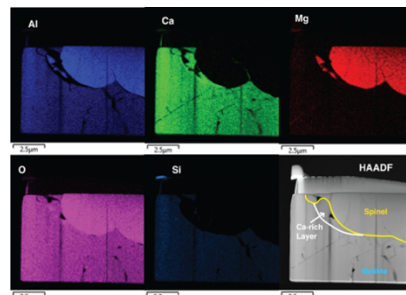
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**Figure 1:** a. False color composition map showing mineralogy of Hedgehog and its rim. Purple: Melilite; Green: Spinel; Yellow: Perovskite. b. Inverse pole figure (IPF) map obtained from EBSD dataset showing deformation in melilite.



**Figure 2:** SEM image showing the selected area containing spinel, before the extraction of the FIB section.



**Figure 3:** EDS Maps of the FIB section.