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NON-DESTRUCTIVE CHARACTERISATION OF FINE GRAINED SAMPLES: ASSESSMENT OF IN SITU MICRO-XRD OF PRIMITIVE METEORITE MATRIX.
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Introduction: The minute quantities or particle size of samples returned from future space missions makes it vital to develop non-invasive diagnostic research methods to study these precious materials. The mineralogy of such samples is a key parameter to determine, but presents many challenges when material is limited or high spatial resolution is required. In this study, we have carried out a non-destructive in situ micro-XRD analysis of selected matrix regions of the primitive CO3.0 carbonaceous chondrite ALHA77307. The results have been compared to TEM observations of the same regions to assess the validity of the in situ micro-XRD technique. Allan Hills (ALHA) 77307 is a CO3.0 chondrite [1] characterized by fine-grained matrix, which consists of abundant amorphous dust and unequilibrated assemblages of Fe and Si-rich material [2]. ALHA77307 has experienced minimal thermal metamorphism and aqueous alteration [1, 2, 3, 4].

Experimental methods: Ultra high-resolution image maps of regions of the matrix of ALHA77307 were acquired using a Carl Zeiss Ultra Plus Field Emission SEM [5]. Micro-XRD analyses were directly collected from the selected areas on the thin section using the GeniX Hi Flux source. A pin-hole of 30 µm was used to achieve an X-ray beam footprint on the sample of ~50 x 500 µm. TEM work was performed at University of Glasgow on FIB-etched foils, using the FEI Tecnai T20 operated at 200kV. Bright-field diffraction contrast images were acquired with the TEM and a 200 nm diameter aperture was used to acquire the electron diffraction pattern of selected areas.

Results: The micro-XRD patterns indicate the presence of olivine, clinoenstatite, pyrrhotite, magnetite and kamacite in the matrix of ALHA77307. This is similar to the TEM observations, which show fine-grained crystals of olivine, clinoenstatite, and pyrrhotite surrounded by an amorphous material. Crystals of poorly ordered phyllosilicate also occur embedded in the groundmass of amorphous material but these were not detected by the micro-XRD due to the collection angle limits.

Conclusions: The preliminary TEM observations of the matrix region investigated have confirmed the presence of some of the mineralogical phases identified by the in situ micro-XRD analysis, acquired before FIB-ing. This demonstrates that in situ micro-XRD has the potential to be a useful tool in the characterization of minute samples or local areas of interest. The presence of magnetite and kamacite, shown by the micro-XRD pattern, have not been identified during the preliminary TEM investigation, therefore an additional study of the foils is required to confirm the presence of these mineralogical phases.