AN X-RAY DIFFRACTION STUDY OF INCLUSIONS IN ALLENDE USING A FOCUSSED X-RAY MICROSOURCE. O. N. Menzies[1], P. A. Bland[2], G. Cressey[3] and F. J. Berry[4]. [1]Planetary and Space Sciences Research Institute, The Open University, Milton Keynes MK7 6AA, UK, o.n.menzies@open.ac.uk; [1]Department of Earth Science and Engineering, Exhibition Road, Imperial College London, South Kensington Campus, London SW7 2AZ, UK.; [3]Department of Mineralogy, Natural History Museum, London SW7 5BD, UK; [4]Department of Chemistry, The Open University, Milton Keynes MK7 6AA, UK

Introduction: We have analysed milligram samples of dark inclusion, CAI, and matrix from Allende using a novel X-ray powder diffraction (XRD) system that employs a high-brightness X-ray source. The MicroSource X-ray generator produces a finely focussed X-ray beam with an output intensity at least x30 greater than that from sealed-tube sources, allowing rapid data acquisition from small samples. Our XRD system utilises a position sensitive detector (PSD) - a fixed diffraction geometry means that mechanical movement is not necessary, so irradiated volume remains constant for each experiment. Consequently, diffraction patterns from standard phases and multiphase mixtures can be compared directly. This allows not only characterisation of phases, but also quantification of their abundance [1,2,3]. When our database of standards is complete it will be possible to quantify mineral abundance in milligram samples of extraterrestrial materials [3].

XRD-PSD is a well-established technique that has provided a rapid means of characterising and quantifying the mineral phases in bulk meteorite samples in a non-destructive manner [1-5]. The addition of a MicroSource to our XRD-PSD system significantly expands the range of analytical targets available to us. The MicroSource employs precision electron optics to focus electrons into a 10μm spot on the target. The X-rays emitted from this area are focussed by specular reflectance along a capillary with an internal mirror coating. This ensures that almost all X-rays generated are concentrated into a fine (almost parallel) beam. A portion of this beam can be selected to deliver a microbeam as small as 10μm at the sample. An initial trial study was carried out, utilising the MicroSource to analyse inclusions *in situ* [6]. This allowed identification of phases, but not quantification of abundance. The intention of the current study is to apply a methodology that will allow quantitative mineralogical analysis to be carried out on samples 10,000 times smaller than was possible using our earlier system.

Experimental Procedure: The samples were hand-picked from a specimen of Allende and, in the case of white inclusions, any obvious matrix contamination removed. We also analysed a sample of Orgueil that had previously been studied using a conventional source. Samples were crushed to a powder and placed in a well of 0.07mm^3 volume. The microwell was placed in a spinning goniometer stage where all angles can be adjusted so that a focussed 500μm beam is aligned with the centre of rotation of the spinning microwell. XRD patterns were collected over 30minutes and calibrated with an external silicon standard.

The samples were then fitted as described by [1-5]. In addition to Bragg scattering from crystalline phases, CuKα_1 radiation interacts with Fe in the sample causing Fe-fluorescence radiation to be emitted. This fluorescence is detected by the PSD and the resultant pattern is a superimposition of Bragg diffraction peaks on a fluorescence background signal. This characteristic fluorescence signal is of use in the fitting process and in determing the abundance of Fe in a sample.

Results: Analysis of the Orgueil sample revealed that a pattern recorded previously from a ground bulk sample in a 180mm^3 well, quantified with a suitable standard set, was essentially identical to the pattern recorded from a 0.07mm^3 well using the MicroSource.

XRD patterns were also obtained from the three white inclusions using the microwell (Figure 1). OM3

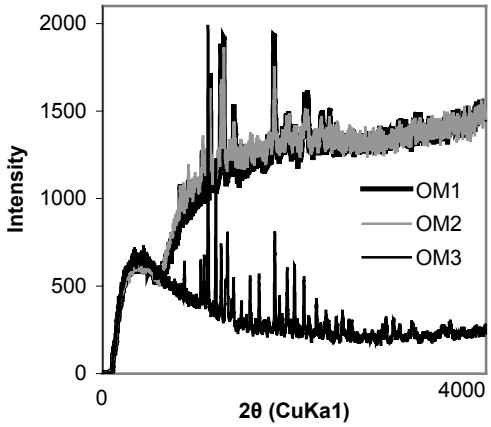

Figure 1. XRD patterns for three white inclusions from Allende. OM3 has a typical CAI mineral assemblage while OM1 and OM2 have elevated Fe fluorescence backgrounds indicative of a high proportion of Fe-containing minerals. Olivine compositions in these inclusions centre on Fo_{80}.

has a low Fe-fluorescence background and peaks indicating the presence of grossular garnet and gehlenitic melilite: a typical CAI assemblage. We are currently

searching for standards of suitable composition to allow phase abundance to be quantified. The inclusion is likely to be an altered type A CAI (e.g. [7]).

The other two inclusions (OM1 and OM2) are intriguing. Apparently similar to CAIs under binocular microscope, their XRD patterns are dramatically different to OM3 (but closely related to each other). Peaks associated with typical CAI minerals are absent, and they show elevated Fe-fluorescence backgrounds indicating the presence of Fe-rich minerals. These inclusions were best fitted with a range of olivine compositions (Fo_{70-90}) with a mode at Fo_{80}.

In order to compare the unusual white inclusions with the surrounding matrix a small sample of Allende matrix was analysed. Figure 2 shows that matrix has an elevated Fe-fluorescence compared to the anomalous white inclusions. Both matrix and inclusions are dominated by the presence of olivine although matrix has a wider range of olivine compositions. In contrast, the white inclusion has more sharply defined, narrower peaks, centred on a composition at Fo_{80}.

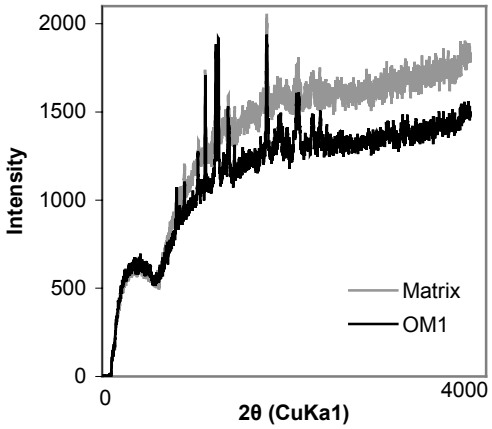

Figure 2. XRD patterns showing the anomalous white inclusion OM1 compared to Allende matrix.

In addition, we sub-sampled a dark inclusion in Allende to investigate its mineralogy and compare it to the mineralogy of adjacent matrix. The patterns obtained are shown in Figure 3, and are almost identical.

Discussion: The work described here is a preliminary study to explore the application of MicroSource XRD-PSD in characterising and quantifying the mineralogy of components in primitive chondrites, including fine-grained CAIs, matrix, and DIs. We have shown that this is an appropriate technique for such an investigation. One of the ultimate aims of this study is to characterise and quantify the mineralogy of fine-grained CAIs, work that may help to determine whether there is a sequence of mineralogical alteration that can be linked to oxygen isotopic variation. The next phase of the work will proceed in this direction.

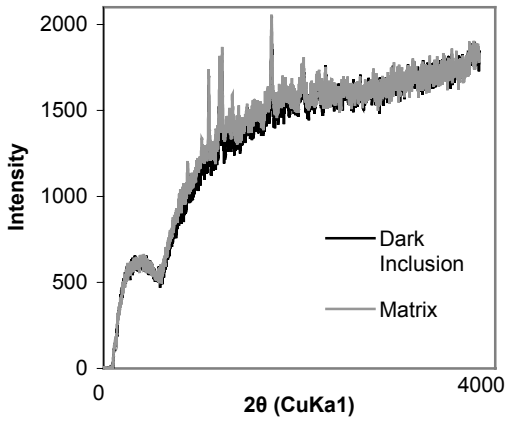

Figure 3. XRD patterns showing that matrix and DI in Allende are mineralogically almost identical.

The unusual white inclusions, OM1 and OM2, require further analysis with SEM to verify the XRD results. At this point it is clear that they are distinct from typical CAIs [7]. It is possible that they may be altered amoeboid olivine aggregates (AOAs), although AOAs are not known to contain high proportions of fayalitic olivine. Their mineralogical similarity to the matrix is interesting, although visually they are distinct entities and compositionally more uniform than matrix.

Several formation mechanisms have been proposed for dark inclusions ranging from size-sorted fragments of matrix to aqueously altered and metamorphosed CV fragments [8,9]. Our preliminary study shows that mineralogically the difference between the dark inclusion analysed and adjacent matrix is negligible.

Conclusions: This study emphasises the potential of the combined MicroSource XRD-PSD system, allowing for the mineralogical investigation of a wide variety of meteoritic components. Fine-grained, heterogeneous samples can be studied rapidly and their component mineral phases characterised and quantified. The results should help in elucidating genetic relationships between meteoritic components.

References: [1] Cressey G. and Schofield P. F. (1996) *Powder Diff., 11,* 35-39. [2] Batchelder M. and Cressey G. (1998) *Clay and Clay Min., 46*, 183-194. [3] Bland P. A., Cressey G. and Menzies O. N. (2003) *MAPS, 38, (accepted)*. [4] Bland P. A. and Cressey G. (2001) *LPSC XXXII,* #1853. [5] Menzies O. N. et al. (2002) *LPSC XXXIII*, #1500. [6] Bland P. A. et al. (2000) *MAPS, 35*, A27. [7] MacPherson et al. (1988) In *Mets. and the early Sol. Syst.* (ed. J. F. Kerridge and M. S. Matthews), pp. 746-807. [8] Fruland et al. (1978) *LPS, 9*, 1305-1329. [9] Krot al. (1997) *MAPS, 32*, 31-49.