MINIMALLY DESTRUCTIVE SAMPLE CHARACTERISATION: IMPLICATIONS FOR SAMPLE RETURN MISSIONS. S. S. Russell[1], E. Vaccaro[1], A. J. King[1], N. V. Almeida[1] and T. Salge[2], [1]Department of Earth Sciences, Natural History Museum, Cromwell Road, London, SW7 5BD, UK, [2]Core Research Laboratories, Natural History Museum, Cromwell Road, London, SW7 5BD, UK. (sarr@nhm.ac.uk).

Introduction: Studying precious and often rare meteorite specimens can be a good "dress rehearsal" for sample return missions. Over the next few years materials will be brought back from asteroid Ryugu via Hayabusa2 (in 2020) and from asteroid Bennu via OSIRIS-REX (in 2023). Preliminary examination will be required to make first pass assessments of the sample type and to make good matches for samples to investigators and analysis types.

In a European extraterrestrial sample curation study [1,2] we have road mapped protocols for the sample retrieval, sample holder opening and preliminary examination of samples returned from space. As part of the current study we have undertaken a specific project to investigate how samples, once removed from the sample return canister, can be best analysed non-destructively and to assess the effect on samples of such analyses. As analogues to primitive asteroids, we have characterized chips and grains of carbonaceous chondrites. Here we show examples of scanning electron microscopy (SEM), micro X-ray diffraction (XRD) and computed tomography (XCT) analyses of the Colony (CO3) meteorite. This work is part of the EC EURO-PLANET RI Joint Research Activity [3, 4].

Methods: We analysed uncoated, unprepared chips of carbonaceous chondrites (CC) (~1 mm across) using a Carl Zeiss Ultra Plus Field Emission SEM and a FEI Quanta 650 SEM. Thin sections of the same meteorites were analysed for comparison. Energy dispersive X-ray (EDX) element maps of the chips were acquired at 8 kV using a novel annular Bruker XFlash FlatQUAD silicon drift detector (inserted between the pole piece and sample) on the Quanta SEM in low vacuum mode [4, 5].

Micro-XRD analyses were directly collected from selected matrix areas on the thin sections using a Rigaku D max Rapid II diffractometer. A pin-hole of 30 µm was used to achieve an X-ray beam footprint on the sample of ~50 x 500 µm [4].

XCT analyses of the same CC meteorite chips were undertaken using Zeiss 520 Versa system. Several scans were carried out at a range of parameters from 50-80 kV, 80-90 µA, and exposure times of 6-20 seconds, using optical magnification of 0.4x, 4x or 20x.

Results and Discussion: *SEM* - Secondary electron images of uncoated chips (e.g. Fig. 1) can be used to observe the morphology of the exterior and help with sampling decisions. The images often show delicate features that may be lost during sectioning (e.g. Fig. 2 and [6]). Elemental maps of the uncoated chips can be obtained with a spatial resolution at the sub-micrometre scale (Figs. 1 & 3). This enables the identification and characterisation of chondrules, CAIs, metal/sulphides and matrix and typically unique identification of the meteorite group can be undertaken using these data.

XRD - The micro-XRD patterns of selected areas of Colony matrix indicate the presence of olivine, clinoenstatite, pyrrhotite, magnetite and kamacite [3]; compatible with observations of this meteorite from other techniques and consistent with bulk XRD studies of CO meteorites [7].

XCT - XCT analyses were acquired at pixel sizes of 0.6-1.2 µm. From these analyses, the abundance and size of chondrules,metal and sulphide grains can be ascertained (e.g. [8, 9]), the degree of porosity can be measured, and brecciation is often evident. Work is underway to combine SEM analyses of one side of the sample with 3D XCT analyses to allow the 3D elemental composition and therefore mineralogy to be determined. Such a correlated microscopy approach is commonly used in the medical sciences (e.g. [10]) and has the capability to provide important insights into extraterrestrial geology as well.

Conclusions: Preliminary examination as outlined above allows the capability of examining the 3D chemistry and mineralogy of a rock chip at a spatial resolution of ~1 µm. Larger rock chips can be analysed similarly, although the spatial resolution for XCT will be lowered.

Effects on sample - While XCT is often considered "non-destructive" there are concerns that the X-ray dose administered can affect samples and compromise their natural radiation record, which is used, for example, for thermoluminescence (TL) studies. A recent study showed that XCT analysis of an L6 chondrite produces a radiation dose comparable to the highest observed in natural samples [11]. X-ray diffraction analyses are unlikely to damage the sample, although further work is required to verify this.

For SEM, the electron beam penetrates only a micron or so into the surface and is not expected to damage the chip interior. Low voltage analyses such as those described (at 5 kV) here reduces the surficial damage still further. However, contamination of the sample can occur at any stage, including during transport to the SEM and inside the analysis chamber, and must be carefully reduced and monitored. A dedicated SEM would be required for precious sample return materials.

References: [1] Hutzler et al. (2016) *LPSC XXXXVII* abstract #1937 [2] Grady et al. (2017) *Planetary Science Vision 2050 Workshop* abstract #8075 [3] Vaccaro et al. (2014) *Hayabusa Workshop* [4] Vaccaro et al. (2014) *Meteoritical Society Meeting* abstract #5327 [5] [5] Terborg Ret al (2017). Microscopy Today 25, 30-35 [6] Russell and Howard (2013) *Geochim. Cosmochim. Acta* **116**, 52–62 [7] Bonato et al., (2016) *Meteoritical Society abstract* #6466 [8] Hezel et al. (2013) *Geochim. Cosmochim. Acta* **116**, 33–40 [9] Almeida et al. (2016) *Meteoritical Society Meeting* abstract #6230 [10] Caplan et al. (2011) *Curr. Opin. Struct. Biol.* **21(5)**, 686–693. [11] Sears et al. (2016) *Meteoritics Plan. Sci*. **51**, 833–838.

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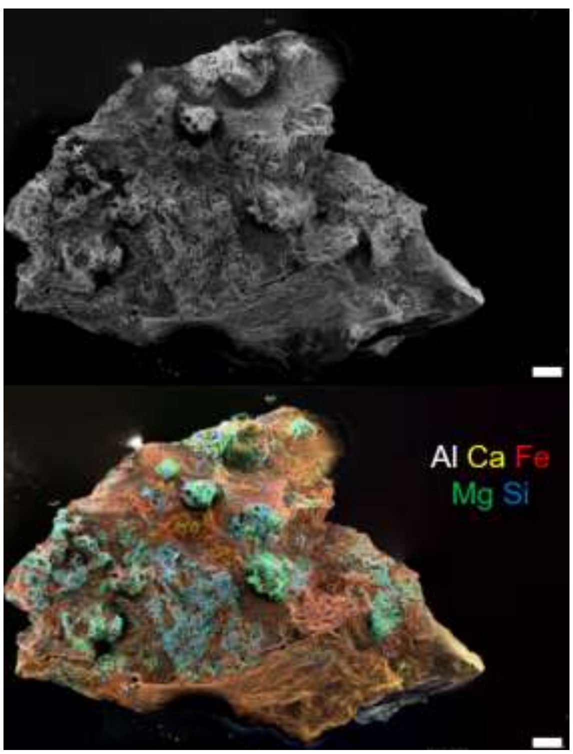

Figure 1. *Secondary electron image (top) and EDX element map (bottom) of an uncoated chip of the Colony (CO3) meteorite; data acquired at 8 kV. Element lines analysed are: Al K, Ca K, Mg K, Si K, Fe L. Chondrules (Mg-rich) and matrix (Fe-rich) are easily distinguishable. Scale bar is 40 microns.*

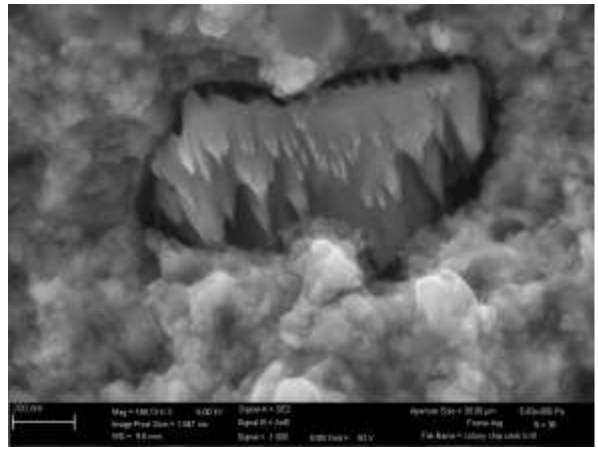

Figure 2. *Infilled vug in the Colony (CO3) meteorite.*

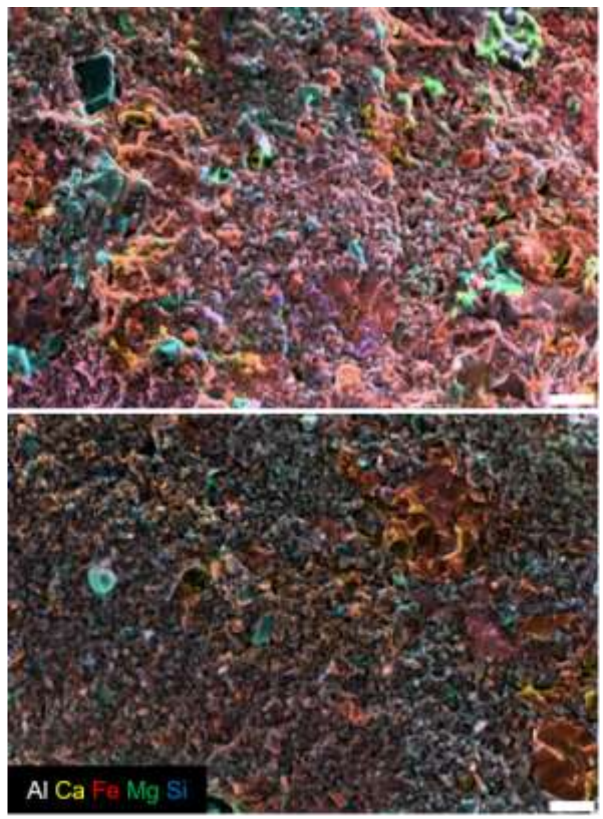

Figure 3. *High resolution EDX element maps of the matrix in a chip of the Colony (CO3) meteorite. Element lines analysed are: Al K, Ca K, Mg K, Si K, Fe L. Scale bars are 3 microns. Mg-rich silicate fragments are visible (green) embedded in a submicron Fe-rich groundmass.*