ESRF	**Experiment title:** Asteroidal aqueous alteration processes: a nano-focused XRD-CT study	**Experiment number**: ES-535
Beamline: ID11	**Date of experiment**: from: 21 June 2017 to: 26 June 2017	**Date of report**: 02/03/2020
Shifts: 15	**Local contact(s)**: Jonathan P. Wright	*Received at ESRF:*

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Report:

Hydrogen- and carbon-rich carbonaceous chondrites are primitive meteorites presenting evidence for early water-rock interactions that occurred on their asteroidal parent bodies. Understanding how these interactions proceeded requires the characterisation of alteration phases (mainly serpentines in the case of CM chondrites) and their relationships to primary minerals, which can be quite challenging given the small scale heterogeneity of these objects and the complexity (variable compositions, complex crystal-chemistry, sub-µm size, order-disorder) of hydrous phases. Here we performed nano-XRD-CT measurements of 5 samples of carbonaceous chondrites, which were mostly ≈5x3x30µm samples cut by Focused Ion Beam and mounted on W fibers.

Two samples were extracted from mm-sized samples previously studied by XRD-CT at ID11 with a ≈10 µm resolution (ES-296), after analysis and localisation of the areas of interest. This was followed by TEM and STXM-XANES investigations (HERMES beamline, SOLEIL), allowing us to perform a study of the distribution (ES-296 data) and mineralogy of serpentines (ES-535 and later experiments) in two areas of the Paris chondrite, presenting various degrees of alteration, thus bringing insight into the processes, and scale, of water-rock interactions in the type of small bodies that may have significantly contributed to the accretion of water to Earth. An article based on these multi-scale data is now in preparation.

Two other samples were cut in an object that was previously studied by TEM and STXM-XANES. We showed first that using nano-XRD-CT, it was possible to extract a single-crystal diffraction dataset for well crystallized olivine, and to proceed to structural refinement. Second, we extracted single-crystal diffraction data of Fe-rich serpentine crystals. By combining these data to the chemical data we obtained previously at the nanometre scale, we propose a new methodology for characterising the mineralogy of highly substituted serpentines. In the following, we present the abstract of an article to be submitted to *Geochemical Perspective Letters*, as well as the first two figures.

Title: Mineralogy of chondritic serpentine at the sub-micron scale using nanofocus XRD-CT

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Abstract: Understanding the reactions of serpentinisation in Fe-enriched environments and the way iron partitions among oxides and serpentines is essential for understanding how H_2 is produced due to the oxidation of ferrous iron by water. The mixing properties of serpentine solid solutions and the structure-chemistry relationships in this system are, however, poorly understood. The task is complicated by the sub-micron size of most substituted serpentines. Here we propose a new approach coupling nanofocus X-ray Diffraction Computed Tomography and crystal-chemistry at the nanoscale. We focus on a carbonaceous chondrite, in which assemblages of serpentines covering a large range of compositions are found within a few μm^3. Our results yield robust structural parameters of sub-micron substituted serpentine crystals. The new data combined with conventional crystallography data support a partial solid solution between cronstedtite and lizardite, offering means to better understand serpentinisation processes in chemical systems that are relevant to Earth and to other bodies of the Solar System.

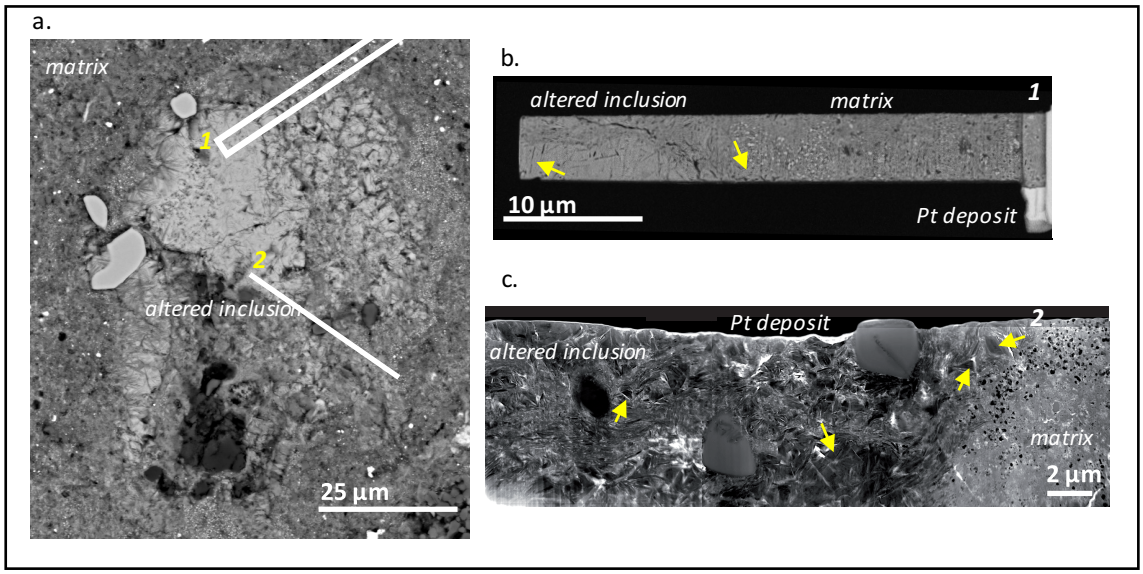

Figure 1: **a.** Scanning electron microscopy image (SEM, backscattered mode) of the aqueously altered refractory inclusion studied, surrounded by a fine grained matrix (Murray chondrite). The location of the thick prismatic FIB slab (labelled 1) studied by XRD-CT (≈3 µm thick, mounted on a W fibre) and of the ultrathin (≈100 nm thick) FIB section (labelled 2) studied previously is shown. **b.** SEM secondary electrons image of the thick slab; right, **c.** STEM image of the ultrathin section (from Elmaleh et al. 2015, *Geochim. Cosmochim. Acta* 158, 162-178.). The arrows point to > 100 nm crystals of serpentine with a flat structure. A second slab, not shown here, was extracted 20 µm deeper than the one labelled 1. The forsterite grain was found in this deeper slab, in the rim surrounding the inclusion.

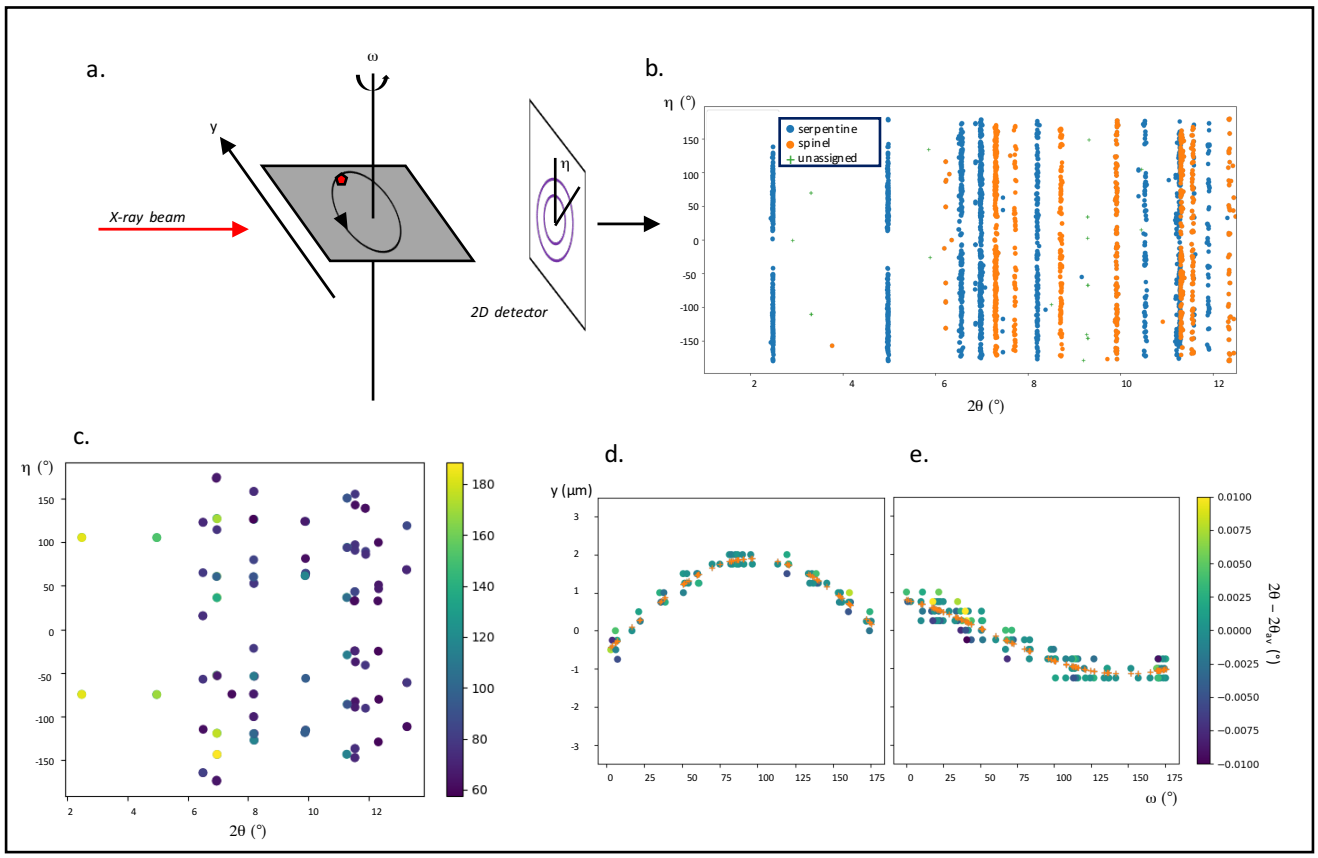

Figure 2: **a**. Acquisition setup for X-ray diffraction computed tomography measurements performed at ID11 beamline (ESRF). At a given vertical position, a layer of the sample (gray rectangle) containing crystals (red) is scanned with a nanofocus X-ray beam at various y and ω positions. Diffraction data (rings or spots) are collected at each step on a 2D detector. A peak search is then performed throughout the dataset. **b**. Example of the spotty diffraction pattern of a layer of the refractory inclusion (η value is plotted as a function of 2θ), which can be indexed mostly as a mixture of hexagonal serpentine and spinel crystals (*h*, *k*, and *l* tolerance = 0.05). Using geometrical information on the diffraction peaks, the X-ray diffraction pattern (example in **c.**) and the sinogram (examples in **d.** and **e.**, orange crosses: fitted centroid) of single crystals of serpentine are extracted. In **d**. and **e**. the size of the crystals is lesser than 750 nm and 1 µm, respectively.