## Preparation of Ryugu Aggregate Samples for SIMS Oxygen Isotope Analysis

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During the initial analysis of Ryugu samples, high precision oxygen 3-isotope analyses of dolomite, breunnerite, and magnetite were conducted using a secondary ion mass spectrometer (SIMS) IMS 1280 at the WiscSIMS laboratory [1]. The results showed resolvable variations in  $\Delta^{17}O$  among dolomite and breunnerite in samples A0058 and C0002 (0.0-0.8‰) and they differ from those of magnetite in the same sections (typically +2‰). These results indicate that the oxygen isotope ratios of aqueous fluids in the Ryugu parent asteroid were isotopically heterogeneous, either spatially, or temporary. Exception was coarse-grained pyrrhotite- magnetite- dolomite assemblages in A0058, which show consistent  $\Delta^{17}O$  between dolomite and magnetite and have been used to estimate oxygen isotope thermometry [1-2]. Including literature data,  $\Delta^{17}O$  values of magnetite and carbonate might have recorded changes of fluid compositions from higher values in early stages to lower vlaues in later stages. In order to further examine the isotope systematics among carbonates and magnetite in Ryugu using IMS 1280 at WiscSIMS laboratory, two aggregate samples each from chambers A and C, A0475 and C0371, have been allocated. They were mounted in 25.4 mm epoxy resin and polished for high quality SIMS analyses. We report our progress of sample preparation and SEM examination of these Ryugu fragments.

Sample preparations: We prepared polished sections of randomly picked fragments from Ryugu aggregate samples A0475 and C0371, partly followed the method in [3]. Total 57 fragments, 20 from A0475 and 37 from C0371, have been mounted in six 25.4 mm epoxy disks. They were typically 300  $\mu$ m to 800  $\mu$ m in sizes. At first, 2-3 fragments from each sample were embedded in 5.6 mm diameter tube mold (Fig. 1a-b) and were lightly surfaced. Six of them (3 each for A0475 and C0371, respectively) were mounted to 25.4 mm disk along with SIMS standards (olivine and calcite) at the center (Fig. 1c). We found that it is more convenient to mount a single 5.6 mm disk that include 7-10 fragments along with the SIMS standards (Fig. 1d), because small bubbles appeared in resin during gliding, which should be filled with epoxy resin and cured before continuing to polish the section. The sections were grinded using 30  $\mu$ m and 9  $\mu$ m lapping films under dry condition, 9  $\mu$ m and 2  $\mu$ m lapping films with baby oil, and 1  $\mu$ m diamond paste with baby oil, followed by hexane wash (Fig. 1e).



Figure 1. Production of SIMS mounts. (a) placing the fragments on Kapton tape, (b) add the mixture of Epofix and ethanol and cure. (c-d) Schematic view of sections with multiple unknown samples (7-13) and SIMS standard at the center. All samples should be within 8 mm from the center of the mount for high precision stable isotope analyses [4]. (e) The surface of the samples was polished using lapping film with mineral oil as the lubricant, followed by hexane wash.

<u>Preliminary sample examination</u>: We examined the textures of polished surfaces by using FE-FIB-SEM (TESCAN S8000) and obtained BSE and EDS X-ray maps. Combined elemental maps (Ca, Si, and Mg) in Fig. 2 distinguish dolomite and breunnerite from other phases, such as clay minerals, calcite, phosphates, and mafic minerals (olivine and pyroxene). Previous studies indicated that samples from chamber A are mainly with altered lithologies, while those from chamber C occasionally contain less-altered lithologies that include anhydrous minerals [e.g. 5]. Fragments from A0475 and C0371 show a similar tendency in that coarse-grained dolomite (>10  $\mu$ m) grains are commonly observed from A0475 and several fragments from C0371 contain multiple mafic minerals. Among fragments from A0475, sizes and abundance of dolomite are highly variable; one fragment includes ~200  $\mu$ m large dolomite grain containing small magnetite inclusions (Fig. 2a), a few are dominated by coarse dolomite (Fig. 2b), others hardly contain dolomite (Fig. 1d). This is consistent with heterogeneous distribution of dolomite in Ryugu [6], which is also indicated from Mn-Cr isotope study of mm-sized Ryugu fragments [7]. In addition, 100  $\mu$ m long breunnerite and

a few small mafic minerals were observed from A0475 (Fig. 2c-d), which are uncommon in chamber A samples. Subset of fragments from C0371 contain coarse-grained dolomite (>10  $\mu$ m; Fig. 2e) suitable for high precision oxygen 3-isotope analyses. Some contain relatively coarse dolomite (>50  $\mu$ m) with magnetite inclusions, which could be useful for oxygen thermometry [1-2]. Several fragments from C0371 contain fragments of mafic minerals similar to those reported in [8] (Fig. 2f). There is a single ~200  $\mu$ m breunnerite grain (Fig. 2g). Bruennerite is less abundant than dolomite in Ryugu, though they often appear as large grains (>100  $\mu$ m [1]).



Figure 2. Pseudocolor x-ray elemental maps (CRY: Ca-Si-Mg) show a variety of lithologies among aggregates in Ryugu A0475 (a-d) and C0371 (e-g). Scale bars are 200 µm. Mineral abbreviations: dol-dolomite, bre- breunnerite, ol- olivine, px-pyroxene. Dolomite and breunnerite are shown as green and olive color. Bright orange indicates Mg and Si-rich phase, likely olivine or pyroxene. Magnetite and sulfide are shown as black. Dark blue thin rim in (a) is an artifact. Marked area in (f) indicates a fine-grained less altered lithology containing multiple tiny mafic minerals.

<u>Summary</u>: Our strategy to use Ryugu aggregate samples is successful in obtaining a wide range of sample suites similar to those reported previous studies and useful in comprehensive SIMS high precision oxygen isotope investigations.

## References

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