Consideration of the interlayer spacing of saponite in Ryugu samples observed under transmission electron microscopy

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Previous studies have revealed that the matrix of asteroid Ryugu samples collected by the JAXA Hayabusa2 spacecraft mainly consists of phyllosilicate minerals such as serpentine and saponite, indicating that the parent rock underwent aqueous alteration over an extended period [e.g., 1,2]. Saponite is a trioctahedral smectite, and it is well known that smectites, which have negatively charged 2:1 layers, can incorporate hydrated cations and polar organic molecules such as alkyl ammonium or ethylene glycol between the layers, causing the expansion of interlayer spacing. However, these water and organic molecules are generally lost due to the vacuum environment inside transmission electron microscopes (TEMs), leading to collapsed interlayer spacing of less than 1.0 nm. Recently, it has been reported that saponite in Ryugu samples showed an average interlayer spacing of about 1.2 nm even in TEMs, which implies that some of the interlayer spaces of saponite may contain organic matter [3]. However, our analysis suggests that this interlayer spacing might be due to the epoxy resin used in the sample preparation process.

We used a Ryugu particle (C0032) supplied by JAXA as the first AO study as well as a synthetic saponite (Reference Clay Sample JCSS-3501, manufactured by Kunimine Industries and distributed by Clay Science Society of Japan). The Ryugu particle and synthetic saponite pellets compacted by hand pressing, were embedded in epoxy resin (Petroepoxy). The samples were heated to 60 °C while vacuum-degassing to ensure thorough epoxy impregnation into the samples and were cured at 80 °C for one day on a hot plate. The samples were polished with limited ethanol, and TEM samples were prepared using a focused ion beam (FIB, Hitachi FB-200). TEM observations were conducted with a JEOL JEM-F200 (HRP) operated at 200 kV, and images were recorded using a Gatan OneView CMOS camera.

In the high-resolution TEM images of Ryugu samples impregnated with epoxy, the interlayer spacing within saponite particles which consist of several sapnite layers, was found to be 1.2-1.3 nm (Fig. 1). Similarly, synthetic saponite impregnated with epoxy showed an interlayer spacing of about 1.2 nm, while epoxy-free synthetic saponite exhibited an interlayer spacing of about 1.0 nm. These results suggest that discussion about the materials present in the interlayer space of saponite in Ryugu samples should carefully consider the sample preparation methods and observation conditions.

This presentation also discusses the layered structure of serpentine and other related topics.

References

[1] Yokoyama T. et al. (2022) Science, doi: 10.1126/science.abn7850. [2] Nakamura T. et al. (2022) Science, doi: 10.1126/science.abn8671. [3] Mouloud B. E. et al. (2024) Meteoritics & Planetary Science 59: 2002-2022.

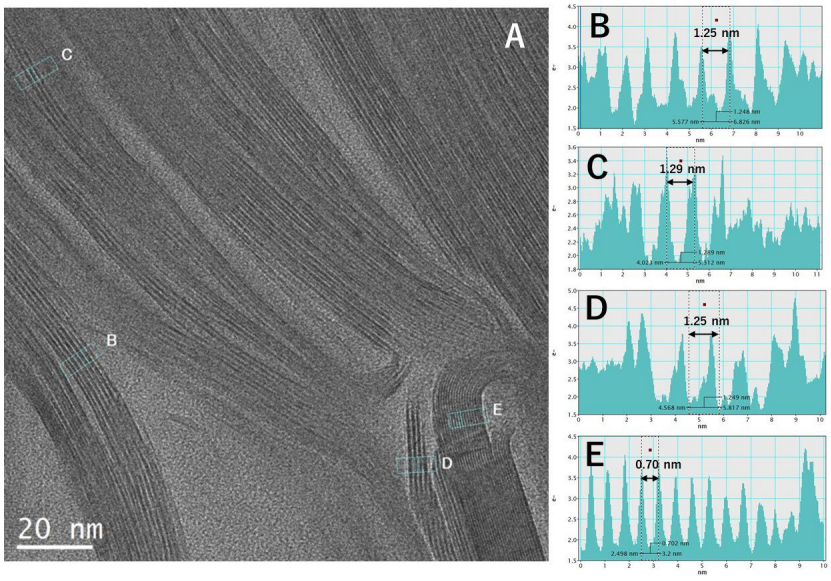

Fig. 1. (A) HRTEM image of the Ryugu sample prepared by impregnating in epoxy resin and thin-sectioned using FIB. The area consists of mainly serpentine-saponite interstratification, a serpentine crystal at the bottom-right, and saponite packets with several unit layers. (B-E) Line profiles across the phyllosilicate layers (B-D: saponite, E: serpentine) at the area B-E in the image A, measured using DigitalMicrograph.