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High-Precision Crystal Structure Determination of Fine-Grained Minerals Using Integrated Synchrotron HRPD and Total Scattering PDF Analysis

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INTRODUCTION & AIM

The determination of high-quality crystal structures is essential for modern science, but many materials such as metastable phases, clay minerals, and nanocrystals cannot be analyzed using single-crystal X-ray diffraction (XRD). Powder XRD combined with Rietveld refinement has long been used to determine average structures and phase compositions, and recent synchrotron developments have improved resolution for microstructural studies. However, this approach has limitations in probing local disorder and short-range structures. Pair distribution function (PDF) analysis, based on total scattering, has emerged as a powerful complementary method capable of revealing both local and long-range atomic structures, especially in nanocrystalline and disordered systems. In this study, synchrotron XRD and PDF are applied to low-temperature cristobalite and kaolinite to obtain precise structural parameters and displacement factors that cannot be resolved by single-crystal methods.

METHOD

High-resolution synchrotron X-ray powder diffraction and PDF experiments were performed at APS (beamlines 11-BM and 17-BM), and analyzed using TOPAS, GSASII, and PDFGui software. Neutron total scattering experiments were conducted at the NOMAD BL-1B beamline (SNS, Oak Ridge).

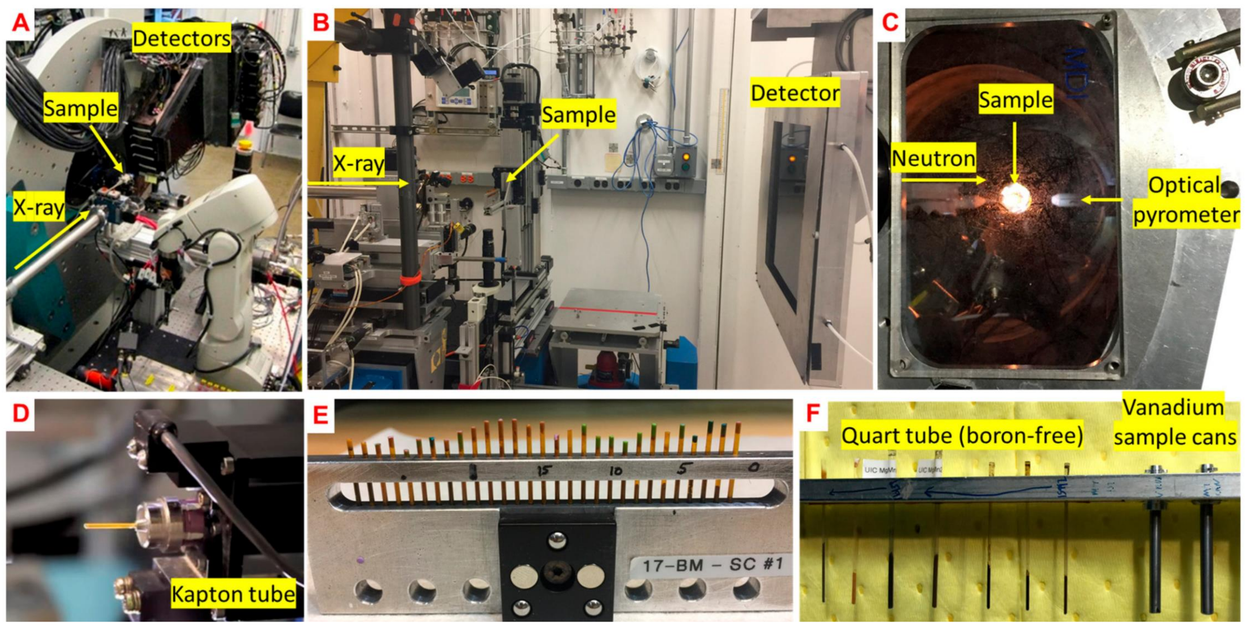

Fig. 1 High-resolution powder diffraction and rapid acquisition powder for XRD/PDF measurements in Advanced Photon Source(APS), Argonne National Laboratory

RESULTS & DISCUSSION

• Low-cristobalite

Determination of the structures of low-temperature metastable phases (e.g., low-cristobalite, and low-tridymite) has been challenging because they commonly occur as fine-grained crystals in geological environments. Therefore, applying powder diffraction and scattering techniques is important to characterize this kind of low-temperature crystal system. Synchrotron radiation XRD and the X-ray PDF method were used to investigate the crystal structure of low-cristobalite, including unit cell parameters, atomic coordinates, and ADPs. To confirm the validity of the refined structure, the results were compared with the previous single-crystal XRD results (Fig. 2)

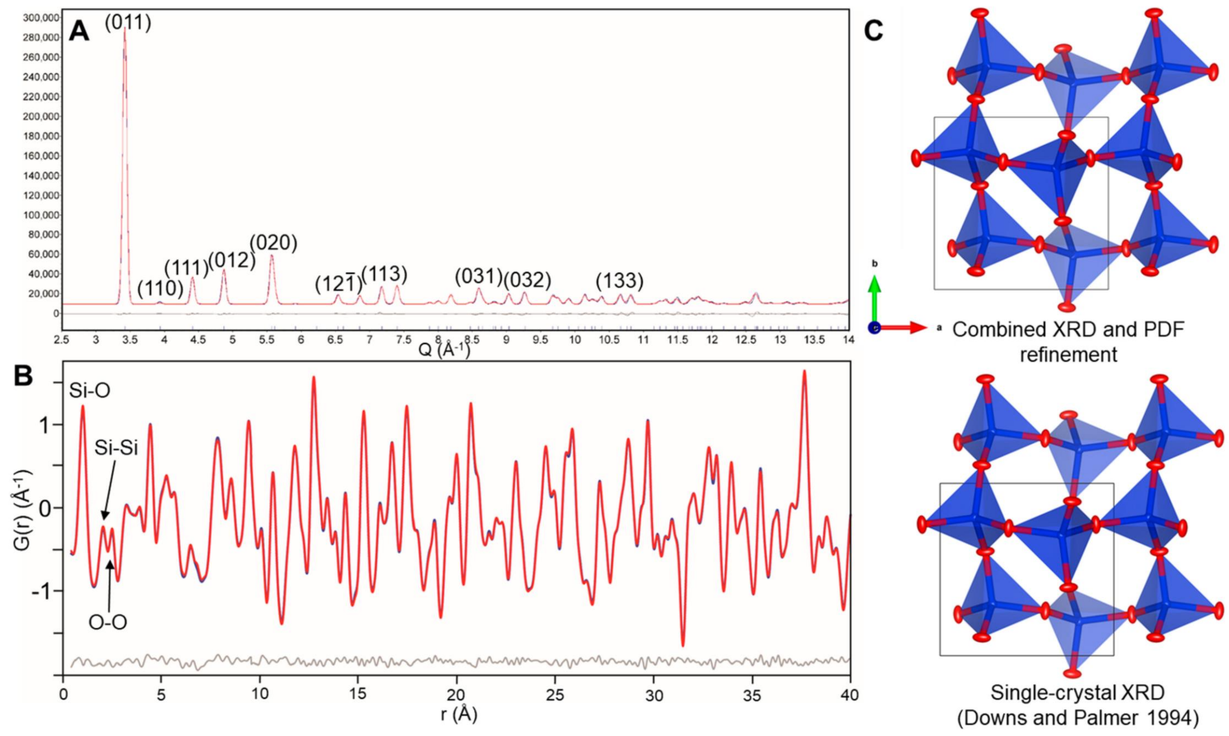

Fig. 2 Rietveld method results of synchrotron radiation XRD patterns of tetragonal cristobalite

• Kaolinite

Kaolinite, $Al_2Si_2O_5(OH)_4$, is the most common clay mineral which forms fromchemical weathering of aluminum silicate minerals (e.g., feldspar). The kaolinite plays significant roles in the geological process (e.g., weathering and adsorption of trace elements) and industrial applications (papers, ceramics, and medicines). Due to the characteristics of fine-grained crystals of the kaolinite phase, applying the single-crystal XRD technique has been challenged to kaolinite clay mineral. We used synchrotron high-resolution XRD and PDF methods to provide a detailed structure of kaolinite including average and local structure information.

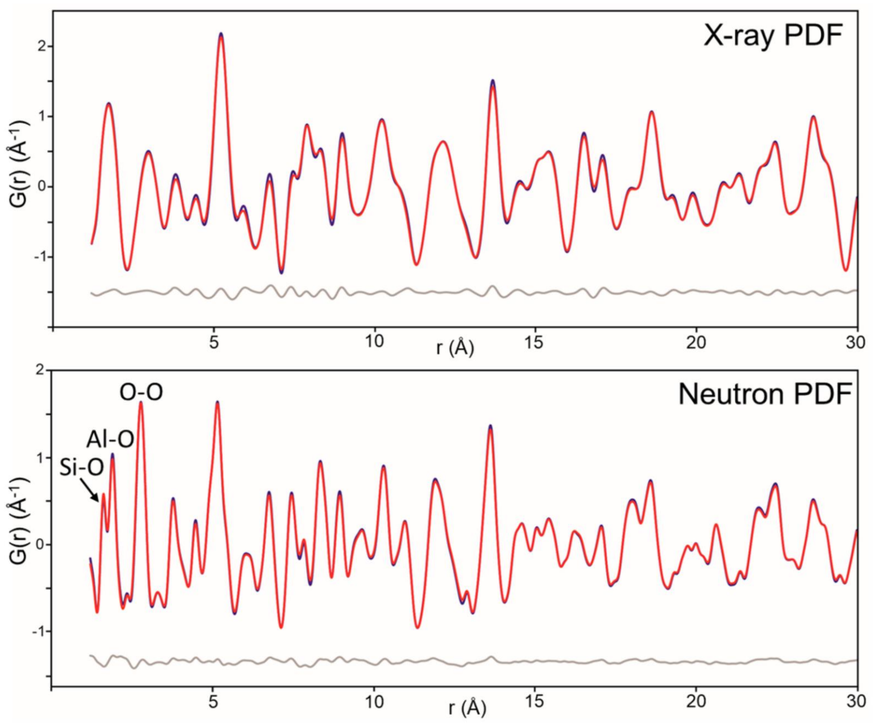

Fig. 3 PDF refinement of kaolinite from X-ray and neutron data.

CONCLUSION / REFERENCES

Thanks to the high-energy synchrotron sources, the powder diffraction and scattering techniques have now become a significant analysis toolkit for the crystallographic approach. This paper shows that combined methods of synchrotron radiation XRD/PDF are powerful complementary tools for fine-grained minerals such as metastable low-temperature mineral, clay mineral, and nanomineral if the material is not suitable for single-crystal XRD.

Lee, S., & Xu, H. (2020). Minerals, 10(2), 124.