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HVEM Study of the Spinel-Type Phase in Kaolinite-Mullite Thermal Reactions

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The kaolinite-mullite reaction series by heating has been controversial since the new proposal of the reaction mechanism.[1] The crystal structure and chemistry of an intermediate phase, which is known to be a spinel-type phase, between metakaolinite and mullite is one of the outstanding issues. We have revealed the spinel-type phase coexisted topotactically with metakaolinite using energy-filtered electron diffraction data obtained from the specimen furnace-heated at 920℃.[2]

HVEM imaging of the spinel-type phase in the 920℃-heated kaolinite represents amorphous character while the FFT result shows pseudohexagonal array of spots indicating the existence of a spinel-type phase (Fig. 1). Randomly-oriented mullite crystals are induced by electron-beam irradiation on the 900℃-heated without forming the spinel-type phase (Fig. 2(a), (b)), while the spinel-type phase which is produced in advance of the mullite coexists with the mullite phase in the 940℃-heated kaolinite (Fig. 2(c), (d)).

It is suggested that low phase stability of metakaolinite phase against electron beam could not maintain the crystalline structure. As a result, the spinel-type phase was not formed due to the collapse of metakaolinite structure by knock-on effect of the electron beam. Crystallization study[3] of the amorphous phase in a metallic glass by electron-beam irradiation also supports this idea.

References

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[3] T. Nagase and Y. Umakoshi, *Sci. Technol. Adv. Mater*., 5 (2004) 57.
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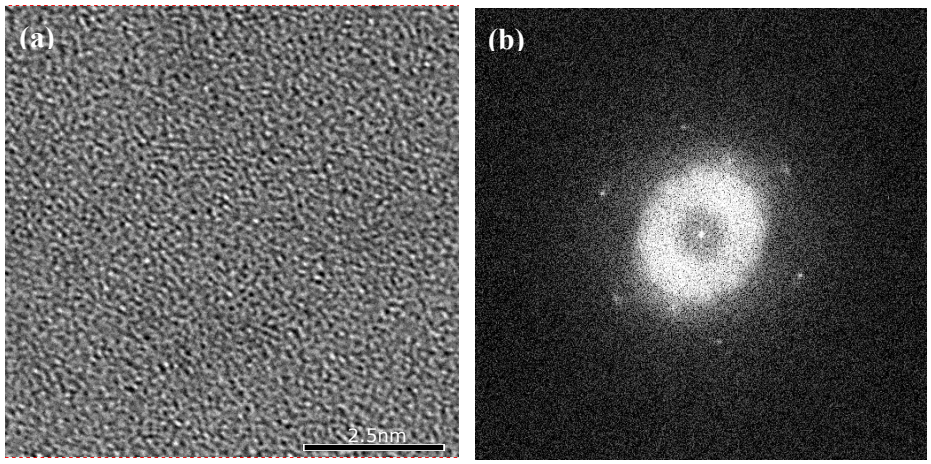

Fig. 1. (a) HVEM image of the spinel-type phase of the 920℃-heated kaolinite after 40min electron-beam irradiation and (b) the FFT result of (a).

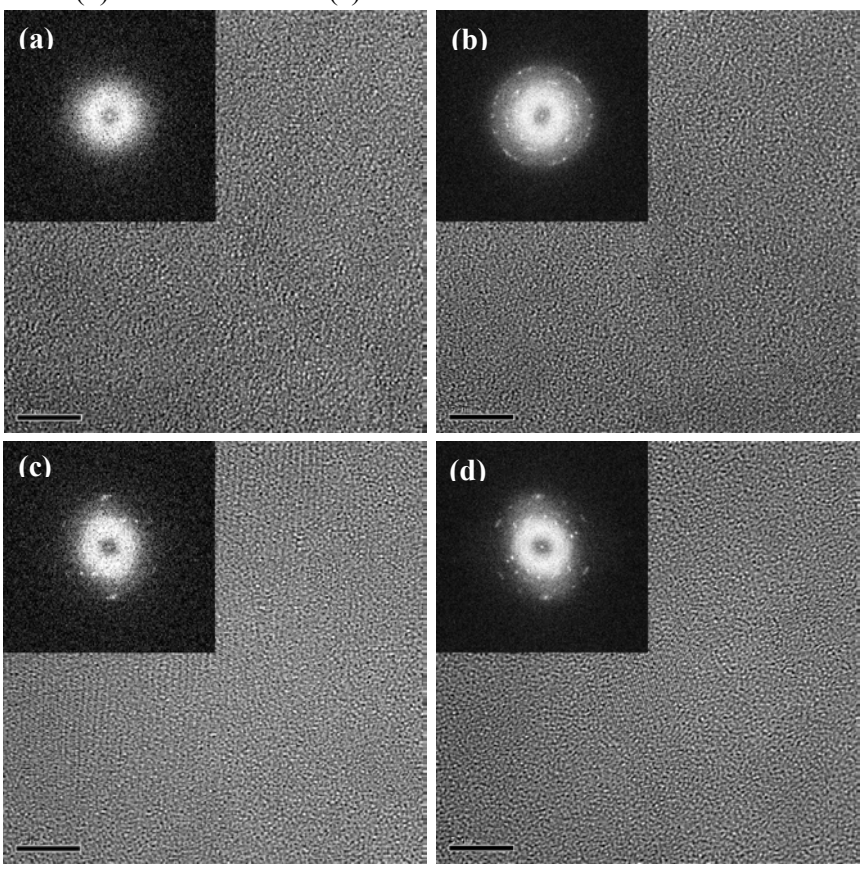

Fig. 2. HVEM images of the 900℃-heated kaolinite after (a) 3min and (b) 30min electron irradiation. Mullite crystals of randomly oriented are formed without the spinel-type phase.HVEM images of the 940℃-heated kaolinite after (c) 8min and (d) 30min electron irradiation. The spinel-type pahse which is produced in advance of the mullite phase persists with the mullite phase. Insets are the FFT results of each HVEM image.

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