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In situ observation of pressure-induced phase transition using Raman spectroscopy

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It is known that ruthenium dioxide is an isostructual with stishovite, a high-pressure polymorph of silica, and can therefore be a model for the post-stishovite phase of SiO2. Silica undergoes a pressure-induced rutile-type to CaCl2-type second-order phase transition. In the case of ruthenium dioxide, the second-ordered phase transition from rutile-type to CaCl2-type structure has been observed. However, a significant discrepancy in the transition pressure has been reported in previous studies. Therefore, we investigated the transition pressure of second-ordered phase transition in ruthenium dioxide.
The starting material used in this study was polycrystalline RuO2. High-pressure Raman spectroscopy experiments were carried out in an external heated diamond anvil cell. The small sample sandwiched between pellets of NaCl powder was loaded into a hole that had predrilled into a rhenium gasket. Raman spectra were obtained by the Raman microscope system (HORIBA) using 532 nm laser.
After loading the starting material to the desired pressure, it was slowly heated to release the differential stress in the sample chamber. After the temperature increased to the desired value, we made in situ Raman spectroscopy measurements. The duration of each measurement was typically 3 to 5 min. The determination of the stable phase at each condition was carried out by observing the change in Eg mode of the rutile-type structure. In the first experiment, the change in the Raman spectra of rutile-type phase was observed at pressures of around 5 GPa. Our results were in good agreement with those reported in previous study. In the second experiment, the boundary was determined at high temperatures (400 - 1000 K). We also performed the first-principles calculations to investigate the send-ordered phase transition in ruthenium dioxide. The results from our calculations were in general agreement with those observed in our experiments. Our new data indicate that the difference in the stress conditions of the sample led to the discrepancy of the transition pressure in previous studies.

Keywords: Raman spectroscopy, high pressure experiments, phase transition, ruthenium oxide