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# Structural Phase Transition in Zr

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## 1 Introduction

Zr (zirconium) undergoes structural transitions under high pressure. The α phase, which is a stable structure at ambient condition, transforms to a ω phase. The ω phase transforms to a β phase with the cubic symmetry. The phase transition of Zr was first studied by Bridgman [1], and a resistance discontinuity was observed at 5.9 GPa, which corresponded to the first-order phase transition from the α to the ω phase. This phase boundary has a positive dP/dT slope. At higher pressures, another phase transition from the ω to the β phase was confirmed at ~30 GPa by the synchrotron X-ray powder diffraction technique. This study was consistent with that of previous shock-wave experiment. The dP/dT slope of the ω-β transition using the diamond anvil cell (DAC) had a negative slope. In contrast, a recent study using a large press apparatus showed that the transition boundary had a large negative slope, which is more than twice of the slope determined using the DAC. In this study, we present experimental results from *in situ* X-ray diffraction measurements on Zr at temperatures up to 800 K using a hydrothermal DAC high-pressure apparatus. The main objective of this work was to determine the accurate phase boundary between the ω and the β phase, and to elucidate the inconsistency in the transition pressure observed in previous studies.

## 2 Experiment

The high-pressure and high-temperature X-ray diffraction experiments were performed using a hydrothermal diamond anvil cell (HDAC) high-pressure apparatus. A culet size of the diamond anvil was 300 micron. The sample temperature was measured using a Type-K thermocouple, the junction of which was placed on the anvils. Both anvils were heated separately using molybdenum wire heaters. The temperature was controlled by adjusting the power supply. The Zr reagent was loaded into a 100 μm diameter hole drilled into a rhenium gasket, which was preindented to a thickness of 70 μm. The sample was sandwiched between pellets of NaCl powder, which were used as a primary pressure calibrant, to reduce any residual nonhydrostatic stresses. Small chips of ruby were loaded together with the sample for use as a secondary pressure calibrant. The sample was probed using angle-dispersive X-ray diffraction, employing the NE1A synchrotron beamline at KEK. The angle-dispersive X-ray diffraction patterns were obtained on an image plate system. The spectra were collected for about 10 min. The pressure was calculated from the NaCl unit cell volume using the equations of state for NaCl.

## 3 Results and Discussion

We performed approximately 4 experimental runs, and the boundary determined in this study is in general agreement with that reported in previous DAC experiments. However, the value of our dP/dT slope was inconsistent with that determined by the large press apparatus. It is likely that this discrepancy dues to the different range in temperatures. In previous large press experiments, the boundary was determined using data at temperatures above 750 K. It is known that a narrow temperature range often induces a significant uncertainty in the dP/dT slope. To avoid any influence of experimental temperature conditions, we investigated the phase boundary at temperatures from 300 to 800 K.

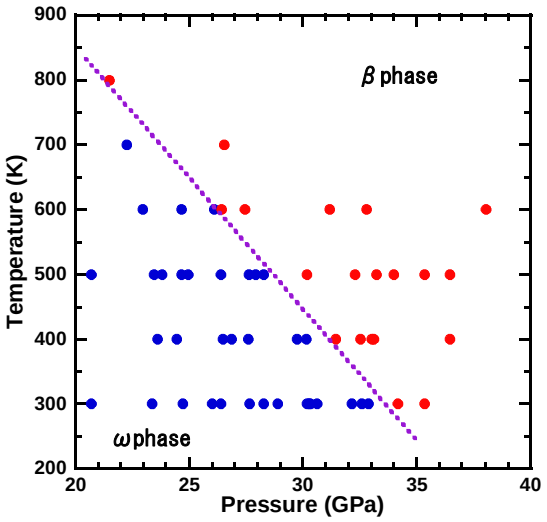


Fig. 1: Experimental results and phase boundary of the ω-β phase transition in Zr. The blue and red circles denote the conditions where the ω and the β phases were stable, respectively. The dashed line shows the inferred phase boundary between the ω and the β phases.

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## References

[1] P. W. Bridgman, Proc. Am. Acad. Arts Sci. **81**, 165 (1952).

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