# Ultrahigh Pressure Generation in the Kawai-type Apparatus: Application to the Wurztite-rocksalt Transition in GaN

The Kawai-type high-pressure apparatus has been widely utilized in the mineral physics field because of its ability to keep a sample of at least several milligrams under controlled pressure and temperature conditions for a desired duration. Nevertheless, the maximum attainable pressure has been limited to *ca*. 28 GPa when tungsten carbide (WC) is used as the anvil material. Recently, by utilizing sintered diamond (SD) as the cubic anvil of the apparatus, the attainable pressure range has been remarkably extended. We [1] have carried out experiments to evaluate the maximum attainable pressure of the Kawai-type apparatus equipped with the SD anvil and to examine whether the wurzite (W)-rocksalt (R) transition in GaN can be usable as a pressure fixed point.

An assembly of eight SD cubes with an edge length of 14 mm and a truncated corner length of 1.5 mm compressed an octahedral pressure medium of MgO+5%$Cr_2O_3$ using the DIA type press SPEED-Mk.II installed at beamline **BL04B1**. Both the phase identification of the GaN sample and pressure determination were performed by *in situ* energy-dispersive X-ray diffraction analysis. Two experimental runs, M139 and M148, were performed at 300 K and at temperatures up to 850 K, employing the specimen assemblies schematically shown in Fig. 1. A white X-ray beam was introduced to the sample and the MgO capsule (pressure standard), independently. The diffracted beam was detected by a Ge solid-state detector with a diffraction angle 2θ of *ca*. 6.0°. In run M139, measurement of the electrical resistance of GaN was also carried out by inserting two Pt wires at opposite positions on the cylindrical outer surface of the sample.

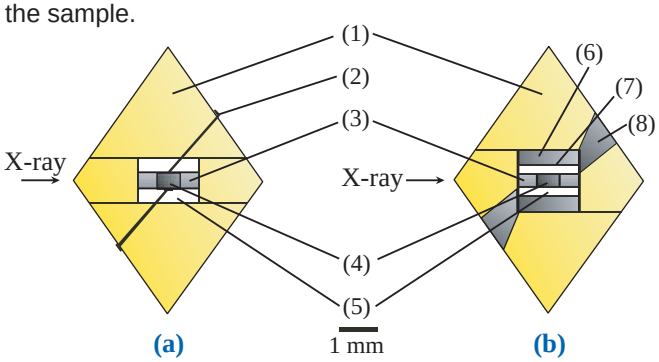


Fig. 1. Cross sections of the specimen assemblies employed in experiments at 300 K **(a)** and at high temperatures **(b)**. (1): Pressure medium; (2): Pt wire; (3): Diamond powder; (4): GaN sample; (5): MgO capsule; (6): $LaCrO_3$; (7): Nichrome heater; (8): TiC electrode.

*Pressure generation*: The pressures generated at 300 K are plotted against press load in Fig. 2 for runs M139 and M148 and independent runs M162 and M181 carried out using similar assemblies (Fig. 1). Pressure is generated with a repeatable accuracy of 2-3% up to *ca*. 60 GPa over the four runs. The maximum pressure of 63.3 ± 0.4 GPa was achieved at load of 8.6 mega Newtons (MNS). However, stagnation of generated pressure at higher loads may be a sign of onset of deformation or fracture of SD anvils. To achieve higher pressures higher-quality SD is urgently required.

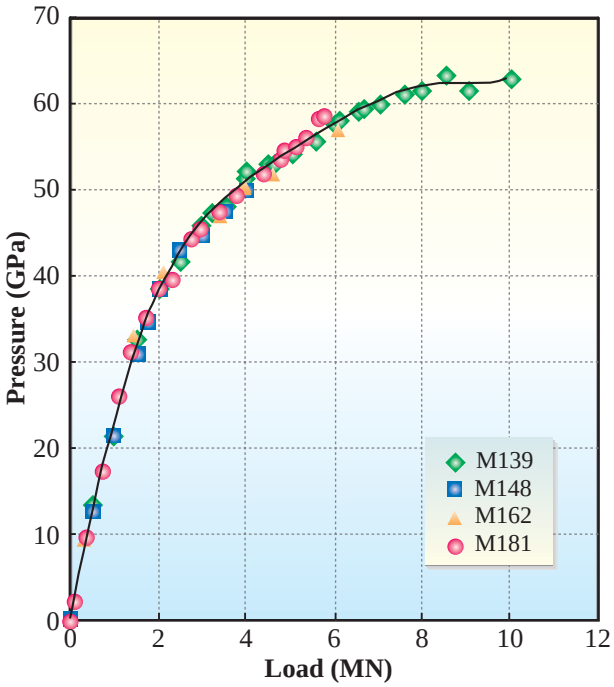


Fig. 2. Pressure *versus* press load at 300 K. Pressure values are based on the MgO scale.

*Phase transition of GaN*: Some characteristic diffraction patterns are reproduced in Fig. 3. At pressures higher than 50 GPa, it was difficult to acquire the diffraction profile of the sample separately from that of the MgO capsule. Diffraction profiles in run M139 were further complicated by the coexistence of $Ga_2O_3$ (see Figs. 3(a) to 3(d)). Fortunately, the complexity due to the additional phases did not cause any difficulty in identifying the phases of GaN. The

onset of the W-R transition was observed at 54 GPa (Fig. 3(b)), consistent with the previous work [2]. A very quick transition from the W- to R-types, however, took place at 51.4 GPa and 750 K in the course of heating at a fixed press load (4.1 MNS). We monitored the sample by X-ray diffraction analysis during the heating at lower press loads, paying special attention if the reverse transition could be observable. Growth of the R-types was not observed under conditions up to 49 GPa and 850 K. However, the dashed line connecting points a and b in Fig. 4 would be close to the equilibrium phase boundary, because both denote the onset conditions of transition from the W- to R-types at 300 K and 750 K, respectively. The Clapeyron slope for the phase boundary is suggested to be negative at approximately dT/dP = –170 K/GPa.

The electrical resistance of the GaN sample showed no significant change upon the W-R transition. This observation and the very slow reaction rate prevent the W-R transition in GaN from being used as a fixed point.

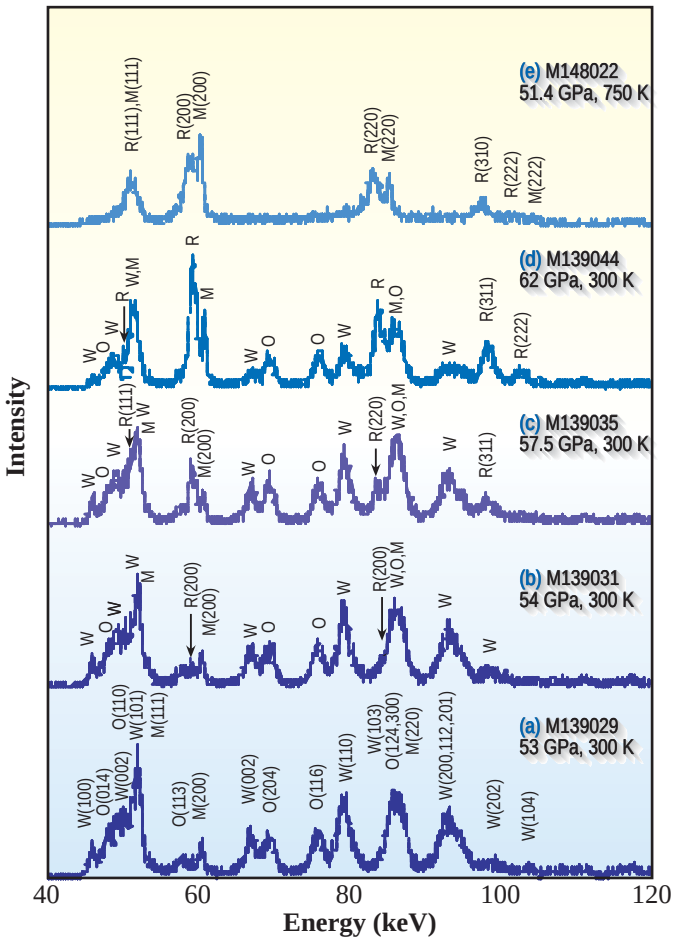


Fig. 3. Selected diffraction profiles of GaN sample with indices. W: wurzite-type GaN; R: rocksalt-type GaN; M: MgO; O: $Ga_2O_3$.

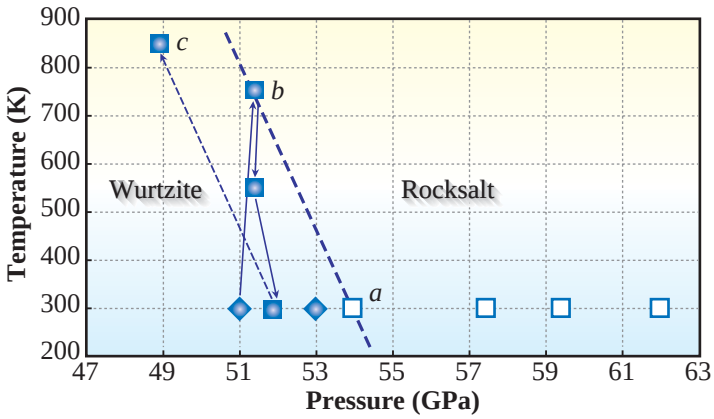


Fig. 4. Results of synthesis experiments on GaN. Solid diamond: wurzite-type; solid square: rocksalt-type; open square: composite of wurzite- and rocksalt-types.

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## References

[1] *E. Ito, T. Katsura, Y. Aizawa, K. Kawabe, S. Yokoshi, A. Kubo, A. Nozawa and K. Funakoshi: Frontiers in High Pressure Research, Geophysical Applications (2005) - in press.*
[2] S. Uehara *et al.*: J. Phys. Chem. **58** (1997) 2093.