Indium Nitride at High Pressures and High Temperatures

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Introduction

Indium nitride (InN) is a very important industrial material because of its semiconducting properties. Even though it is very widely studied at ambient conditions, the pressure-temperature phase diagram is still unknown. M. Ueno and co-workers[1] reported a phase transition from hexagonal to rocksalt structure at a pressure of ~12 GPa at room temperature in 1994. No further report of the phase transition is present in the literature. This report presents some new data on this transition at temperatures to 1000K and pressures to 20 GPa.

Methods and Materials

Using a resistively heated diamond anvil cell (DAC), we measured x-ray diffraction patterns of InN at high pressures and high temperatures. The heater of the DAC was made using a platinum wire (200 mm diameter) coiled around an alumina ring. Subsequently we covered it with a layer of high-temperature zirconia cement. In association with this fragile heater, we placed a larger band heater around the cylinder part of the cell. In order to estimate the temperature at the sample position, a pre-calibration of the temperature on the table of the diamond versus the temperature at the sample position is necessary. We did the calibration by placing a thermocouple between the two diamond anvils with the thermocouple insulated from the anvils with a thin layer of zirconia cement. A second thermocouple was placed in the table of the top diamond. During the experiment, only the thermocouple sitting on the table of the diamond measured the temperature.

We placed the InN sample in a 100-mm-diameter hole in a 30-mm-thick rhenium gasket. NaCl was used as the pressure medium and the pressure calibrant. The angle-dispersive x-ray diffraction experiments were conducted at the bending magnet beamline 13-BM with the monochromatic beam at an energy of 29.200 keV. The exposure time was from 5 min. to 15 min.

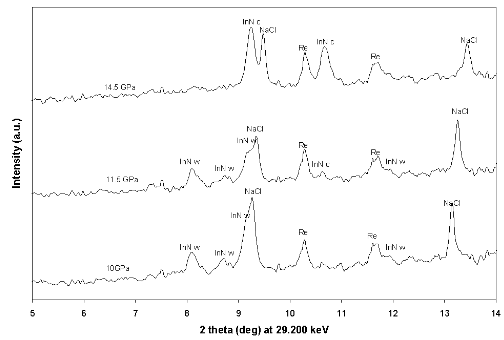

FIG 1. InN x-ray diffraction patterns at 600K showing the pure wurtzite phase (10 GPa), the appearance of the rocksalt phase (11.5 GPa) and the pure rocksalt phase (14.5 GPa). We denote InNw and InNc to indicate the peaks of the wurtzite phase and the cubic phase, respectively.

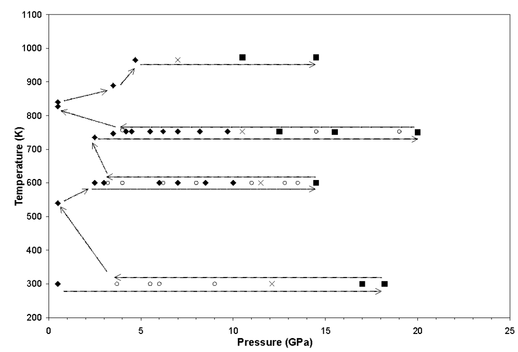

FIG. 2. The observed InN phases in the pressure and temperature field of this study. Black diamonds represent the wurtzite phase, black squares the rocksalt phase, crosses the appearance of the cubic phase, and open circles represent the cubic phase in decompression. Pressure-temperature paths are shown.

Results

Figure 1 presents three typical spectra observed at 600K and pressures between 10 and 15 GPa with the first showing the hexagonal wurtzite phase, the second showing the appearance of the cubic phase, and the third showing clearly the cubic phase and the complete absence of the hexagonal phase.

Our experiments covered a pressure and temperature range from 300K and 0.5 GPa up to 965K and 20 GPa. In this range, indium nitride transforms from a low-pressure hexagonal wurtzite phase to a high-pressure cubic rocksalt structure. We could notice that kinetics exist in the phase transition as both phases could be seen in some collected spectra especially in the processes of decreasing pressures. It can be seen from Fig. 2 that the rocksalt-to-wurtzite transition during unloading occurs at very low pressures, indicating a very large hysteresis in pressure of the phase transition.

Discussion

The main remark is the fact that, even if the temperature and pressure of the observed phase change are fairly well determined, a clear phase boundary is still difficult to determine because of the involved kinetics. Figure 2 shows that the observed stable phases depend on certain pressure-temperature paths. The data are too scattered to be able to define a slope for the phase transition. The metastability domain of the cubic phase is found to be large even at moderate temperatures to 750K where it persists at 4 GPa. Because we used screws to change pressure, a constant pressure path is difficult to achieve. In addition, there is always a drop (up to 20%) in pressure at high temperatures. We attempted one cycle at constant pressure with increasing temperature, but at around 925K, the pressure dropped from 10 GPa down to 7.5 GPa (pressure too low to observe the phase transition). At the following step, we increased pressure, which initiated the phase transition.

Constant temperature with increasing/decreasing pressure remains the best choice for phase transition studies with this technique.

In conclusion, we have studied the behaviour of InN at simultaneously high pressure and high temperature in the region of the phase transition from wurtzite to rocksalt structures. The phase transition shows large hysteresis in the pressure-temperature region of this study. The high-pressure cubic phase is found to be metastable at pressures down to 4 GPa even at moderate temperatures (< 750K).

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Reference

[1] M. Ueno, M. Yoshida, A. Onodera, O. Shimomura, and K. Takemura, Phys. Rev. B **49**, 14-21 (1994).