

Observation of elementary process in the formation of ultrahard phase from C₆₀ under high pressure

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Introduction

Many works have been done in recent years looking at fullerenes quenched from high pressure and high temperature and a fairly detailed reaction diagram now exists. However studies at high pressure above 10 GPa are a few. At high pressure above 10 GPa and at elevated temperature, very hard materials can be produced [1]. To know the structure of the fullerene-derived hard materials, in-situ x-ray diffraction measurements is required. We examined the behavior of C₆₀ up to 800°C at 12.5 GPa and up to 600°C at 14.3 GPa by energy-dispersive x-ray diffraction.

Experimental

The starting material was 99.9% purity C₆₀ purchased from MER Co. The sample after being dried in vacuo was packed into a tube made of semisintered MgO. The sample assembly was pressurized with a cubic anvil press (MAX80) installed at AR-NE5C beamline of KEK. The diffraction angle was chosen at 6°.

Results and Discussions

In order to know first the general trend in the behavior of C₆₀ at elevated temperature under constant pressure, x-ray diffraction measurements were carried out during stepwise heating to 800°C at 12.5 GPa and 600°C at 14.3 GPa. In this case, the temperature was increased by 100°C with a rate of 100°C/min and each diffraction pattern was recorded while the temperature was held constant for 200 sec. Evolution of x-ray diffraction patterns recorded at various temperatures at 12.5 GPa is shown in Fig. 1.

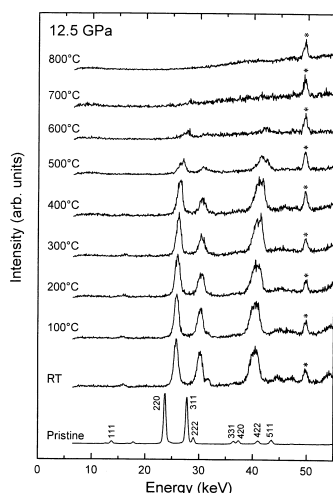


Fig.1. X-ray diffraction patterns taken at various temperatures at 12.5 GPa. The asterisked peak is from MgO used for the capsule.

With the pressurization to 12.5 GPa at room temperature, merging of the (331) and (420) peaks takes place. The pristine fcc structure persists to at least 600°C but a significant broadening occurs in each peak in a temperature range between 300 and 500°C. All the reflection from fcc phase lose their intensities in this temperature range and finally disappear at temperature higher than 700°C above which the sample becomes completely amorphized. We have got the similar results at various temperature at 14.3 GPa. The diffraction patterns are recorded by downward shift of about 100°C. Also, time-resolved x-ray diffraction study was undertaken at every 1-10 min interval while high temperature was held constants for longest 3 hrs at 12.5 GPa and 14.3 GPa. Changes of d-values at various temperatures at 14.3 GPa are shown in Fig.2. The detailed results and discussions are given elsewhere [2].

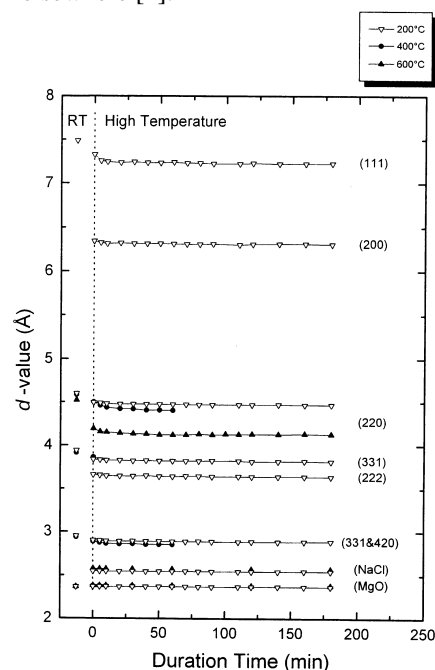


Fig.2. Change of the d-values of the residual fcc phase with duration time at various temperatures at 14.3 GPa.

Acknowledgments

We thank Shinji Kawasaki and Akifumi Yao for helping us with the experiment.

References

- [1] V.D.Blank et al. Phys. Lett. A188 (1994) 281
- [2] T.Horikawa et al. Phys. Lett. A (2001) in press.