

In-situ x-ray observation of graphite-diamond transition using catalysts under high pressures and high temperatures

*W.Utsumi⁺²⁶⁵, T.Mizutani⁺¹⁴⁶⁶, N.Nishiyama^{*3210}, and K.Funakoshi^{**1115}

[†]Japan Atomic Energy Research Institute, ^{*}Ehime Univ. and ^{**}JASRI

Many studies have been previously made on the diamond synthesis under high pressures. However, most of them are based on the information of the recovered specimen at ambient condition, and very few in situ experiments have been reported. The aim of this study is to make an in situ observation of graphite to diamond transition under high pressure, and to clarify the role of catalysts in the diamond formation process.

Experiments were carried out using a large volume high pressure apparatus, SPEED-1500, installed at BL04B1. Using tungsten carbide anvils with truncation size of 8.0mm, diamond synthesis was made under high pressures and high temperatures up to 13 GPa and 2100°C. In this study, $MgCO_3$ or $K_2Mg(CO_3)_2$ were used as catalysts. These catalysts were placed next to the graphite specimen in a sample chamber. Pressure was first increased at room temperature and then temperature was increased under constant applied load. Incident x-ray beam collimated by the slits irradiated the boundary area between the catalyst and graphite, and powder diffraction profile by the energy dispersive method was obtained under each P-T condition.

Fig.1 shows the variation of x-ray diffraction profiles of graphite- $K_2Mg(CO_3)_2$ system with increasing temperature at 9.3 GPa. In this experiment, diamond did not appear yet at the temperature where catalyst started to melt. This means that melting of the catalyst is not a sufficient condition for the diamond formation. In order to synthesize diamond using this catalyst, higher temperature than its melting point is needed. This result is contrast to the fact that when metal catalyst such as Ni or Co is used, diamond is always formed just at their melting temperatures.

On the other hand, when $MgCO_3$ was used as a catalyst, diffraction profiles showed a

different variation. In this case, the catalyst did not melt until 2100°C. But at around 1800 °C, which is far below the melting temperature, clear cubic diamond peaks appeared. At this temperature, $MgCO_3$ peaks were also very clear so that $MgCO_3$ was still in a solid crystal phase. Also, from the diffraction profile, there is no evidence of the breakdown or dissolution of $MgCO_3$. Therefore, this experimental data indicates that solid $MgCO_3$ plays a role of diamond formation catalyst.

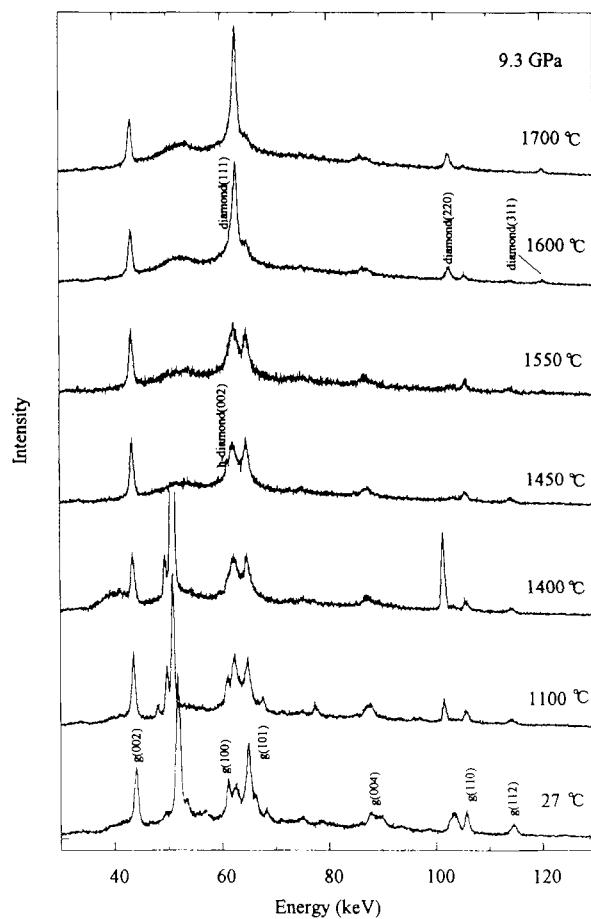


Fig.1 Variation of x-ray diffraction profiles of graphite- $K_2Mg(CO_3)_2$ system with increasing temperature at 9.3 GPa