



	Experiment title: In Situ diffraction study of polymerization of superhard 3D C60 under high-pressure and high-temperature	Experiment number: HS-1679
Beamline: ID30	Date of experiment: from: 6 th June 2002 to: 11 th June 2002	Date of report: 1/03/03
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Report:

C60 molecules polymerize under high-pressure high-temperature (HP-HT) forming 1D, 2D and 3D crystalline polymers [1,2]. Below 8 GPa the C60 phase diagram is now well known: 1D (orthorhombic) and 2D (tetragonal and rhombohedral) C60 polymers, based on the 2+2 cycloaddition reaction, are formed [1,2]. For pressures above 8 GPa the situation is not so clear. Previous X-ray diffraction analyses conducted at the E.S.R.F. have shown that C60 samples quenched from 13Gpa and 550°C are 3D C60 polymers. Furthermore unusual Debye-Sherrer ellipses are observed indicating that the transformed sample imprints the applied stress field [2]. It must be emphasized that the detailed atomic structure of such 3D polymers is not yet known due to the poor crystallinity of the samples.

In order to study the upper part ($p > 8\text{GPa}$) of the pressure-temperature phase diagram of C60 we have extended the range of pressures provided by the Paris-Edinburgh large-volume press by using sintered diamond anvils. This has permitted to reach pressures up to 15Gpa far more than the maximum pressure (7Gpa) reached by the same press employing the more traditional WC anvils. Calibration curves applied-force versus pressure has been obtained for different temperatures using known standards. The two-dimensional angular dispersive diffraction patterns recorded with a 2D MAR detector have an excellent quality. Preliminary patterns of C60 have been obtained under such high-pressure conditions. More beam-time is needed to scan all the phase diagram of C60 searching possible new phases and determining their structures.

[1] M.Núñez –Regueiro et al. Phys. Rev. Lett. 74, 275 (1995).

[2] L.Marques et al Science 283, 1720(1999); M.Mezouar et al ESRF Newsletter 33, 7(1999).