ESRF	**Experiment title:In** situ diffraction studies of C60 polymerisation under high pressure high temperature using the LV cell coupled with the new fastscan 2D.. .	**Experiment number:** **HS505**
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Report:

As other non-saturated molecules, C60 polymerises under high pressure and high temperature (HPHT). It is quite surprising that the C60 polymers formed in this way present crystalline order. The new polymerised phases can be recovered at room temperature and pressure and thus they can be studied at ambient conditions. Up to now, and depending on pressure and temperature synthesis conditions, one-dimensional (1D) and two-dimensional (2D) C60 polymers have been obtained. The new polymer phases present lower lattice symmetry than the C60 monomer, which has, at room temperature, a face centred cubic structure. Hence the C60 solid state polymerisation could be followed by x-ray diffraction, as it leads to important changes in the diffraction pattern. The low dimension character of these polymerised phases implies that the polymerisation process takes place along some preferred directions.

In situ diffraction measurements were used to follow the C60 polymerisation process in order to draw the fullerite phase diagram and to clarify how the polymerisation process takes place. We have used the large volume pressure cell (Paris-Edinburgh cell) which allows working at high temperatures. The diffraction patterns were collected in the angle

dispersive mode using a fastscan 2D detector.

Several temperature-pressure cycles were done up to 70kBar and 700°C. We have observed the expected (quenched) 1D and 2D polymer phases but only when the pressure is increased before temperature, i.e., the polymerisation starts in the non-rotational (orientationally ordered) C60 state. On the other hand, when the C60 is polymerised from the orientationally disordered state, i.e., the temperature is increased before pressure, we do not obtain the 1D and 2D C60 polymers but instead a compressed fcc polymer is formed [l]. In this fcc polymer the directionality of polymerisation is lost as it takes place in random directions in accordance with the rotational (orientationnaly disordered) C60 state from which the polymer is formed. Moreover, the fcc polymer state presents 2D diffraction patterns displaying Debye-Sherrer ellipses instead of circles, as we have observed previously on quenched 3D C60 polymers synthesized at very high pressures, 13Gpa (fig.) [2]. The ellipsoidal X-ray diffraction patterns show that the deviatoric stress introduced by the non-hydrostatic compression has been frozen in the quenched samples. The multiple, almost degenerate, bonding possibilities permitted by the highly symmetrical C60 molecule in the polymerized 3-D structure allow the fixing down to ambient pressure of this feature.

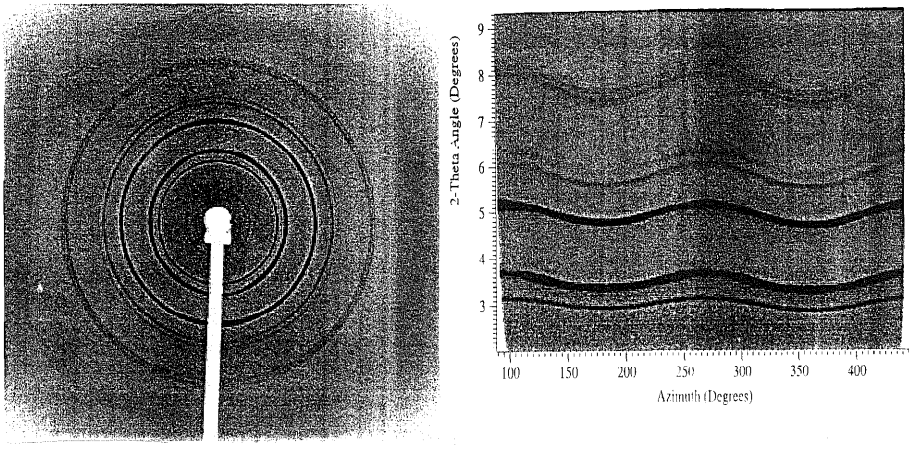

Fig. Debye-Sherrer ellipses from C60 polymer

[l] L.Marques, J-L. Hodeau, M. Mézouar, M.Núñez-Regueiro (to be published).

[2] L.Marques, M. Mézouar, J-L. Hodeau, M.Núñez-Regueiro, N. Serebryanaya, V. Ivdenko, V. Blank, G. Dubitsky (submitted for publication).