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Names and affiliations of applicants (* indicates experimentalists):

L.Marques*, Dept. Fisica Universidade Aveiro (Portugal)
J.-L. Hodeau* Lab. Cristallographie CNRS, Grenoble (France)
M. Nunez-Regueiro* EPM-Matfromag CNRS, Grenoble (France)
M. Mezouar* **ESRF,** Grenoble (France).
N. Serebryanaya* Inst. of Spectroscopy, Troisk (Russia)
V. Ivdenko* "

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 presents 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 presents a 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 long range order presented by these new 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-Edimbourgh cell) which allows to work at high temperatures. Angle dispersive powder diffraction techniques using a fast readout 2D detector (Hanfland et al. ESRF Highlights97) was used. This kind of detector with high spatial resolution and high sensivity is very appropriate to study in situ

structural transitions, as it enables the peak splittings and the very weak peaks to be observed. The data analysis using the Rietveld refinement is rendered possible by using the angle dispersive diffraction technique and the preferred orientation effects on powders (usually present in the high pressures studies) are overcorned by integrating the Debye-Sherrer rings (Fit2D).

Although the weak scattering power of carbon, which is comparable to those of sample environnement (boron nitride and graphite furnace), the diffraction pattern displayed by C60 is very good even for small time exposures (3 minutes).

Several temperature-pressure cycles were carried out up to 70 kBar and 700C. Diffraction images collected with the fast readout detector permitted to follow the structural transitions on C60, as a result of changes in pressure and temperature. Several phase transitions have been observed confirming the richness of the C60 phase diagram. An increase in pressure leads to a strong displacement of the Bragg peaks to high diffraction angles, indicating that C60 is a highly compressible solid. When the temperature is ramped we start to observe structural distortions, corresponding to the formation of an ordered polymer phase. On quenching the HPHT phases do not revert to C60 monomer.

Quenched samples are highly textured as we may see in the figure. Moreover some unexpected features, as Debye-Sherrer elipses, were observed in the 2D diffraction patterns.

Further data analyses are under way.

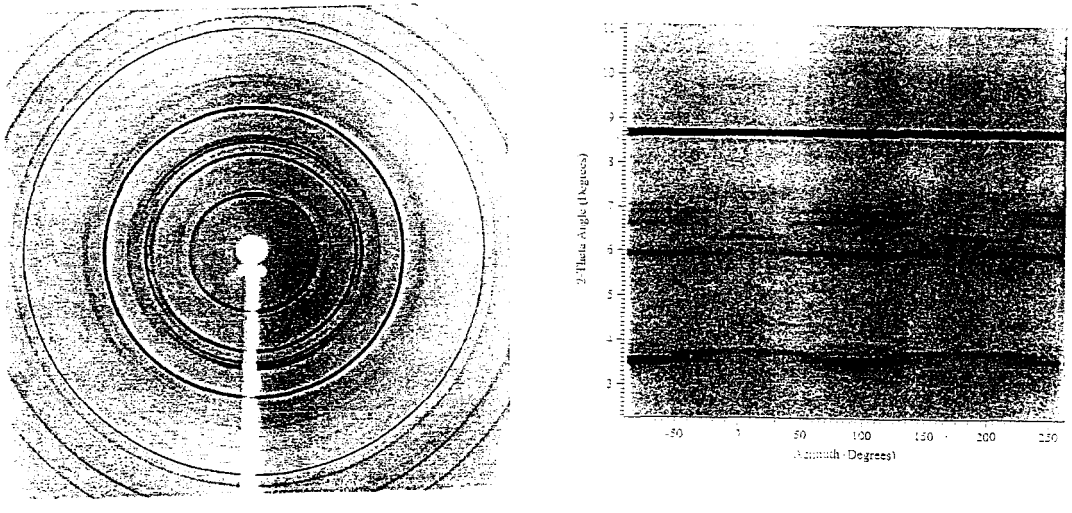

Fig. 2D diffraction of a C60 polymer.