



	Experiment title: Investigation of the Carbon K near-edge structure in polymerized fullerenes by inelastic x-ray scattering	Experiment number: HE-579
Beamline: ID 16	Date of experiment: from: 23/06/99 to: 06/07/99	Date of report: 27/02/01
Shifts: 18	Local contact(s): J.P. Rueff	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): J.P. Rueff* , M. Krisch* , ESRF, BP 220 38043 Grenoble Cedex F. Bartolome* ICMA, CSIC-University of Zaragoza, Spain J.L. Hodeau Laboratoire de Cristallographie, BP 166 – 38042 Grenoble Cedex 9		

Report:

Fullerene molecules, like other non-saturated systems, have been shown to polymerize either photochemically or under high pressure and high temperature (HP-HT) [1]. In the 1-D polymer, the C₆₀ molecules are linked into polymeric chains while in the 2-D polymers they form hexagonal or square polymerized layers. In the 3-D polymer, the molecular bonding occurs in the twelve nearest neighbor directions. Valuable information could be gained about the effect of dimensionality on the electronic structure and the bonding character by near edge x-ray absorption spectroscopy (XAS). Conventional XAS measurements, however, carried out at the C K edge are extremely surface sensitive. This sensitivity may lead to a misinterpretation of the data, for the fullerene polymers are known to present structural properties at the surface with no comparison with the bulk, because of a large number of broken bounds. This difficulty can be overcome by a hard x-ray inelastic scattering experiment (IXS), in which the energy transfer is tuned to the C K-edge. With a typical incident photon energy of 10 keV, it is obvious that in this case true bulk properties are probed.

The experiment was carried out on the inelastic x-ray scattering beamline ID16. The incident photon energy was monochromatized by a Si(111) channelcut monochromator, yielding an energy resolution of 1.5 eV at 10 keV. The scattered photons were energy analyzed by a 1m Rowland circle crystal spectrometer, utilizing the Si(555) reflection order close to

backscattering, and detected by a Peltier cooled silicon diode. The IXS spectra were recorded at a scattering angle of 10 degrees, keeping the scattered photon energy fixed, while scanning the incident energy, thus varying the energy transfer from 270 eV to 320 eV through the C K-edge. The total energy resolution was about 2.0 eV. We also had the possibility to insert an additional channelcut between monochromator and the spectrometer, in order to increase the energy resolution. We chose to use an asymmetric Si(400) channel cut which yields a resolution of 0.4 eV. However, the improvement in terms of resolution was accompanied by a decrease in flux by a factor 5 (as expected from the reduction of the total energy bandwidth when switching from the low to high resolution setup). Such a drop in flux was too time-consuming, and we preferred, most of the time, to keep the low resolution in order to acquire as many as possible IXS spectra of the different C60 phases.

The C K edge absorption spectra were measured in a series of well characterized samples spanning the different C60 polymerized structures. The samples were synthesized under high T-high P, and compacted in pellets. Typical sample thickness was 0.5 – 1.5 mm. Pure C60 and graphite powder were used as reference samples. Typical absorption spectra are shown in figure 1. Large changes in the spectra can be noticed particularly in the 10 eV region above the edge, that can be traced back to the effect of dimensionality. Simulations of these spectra using a multiple scattering calculations within the finite difference approach are underway to further characterize these effects.

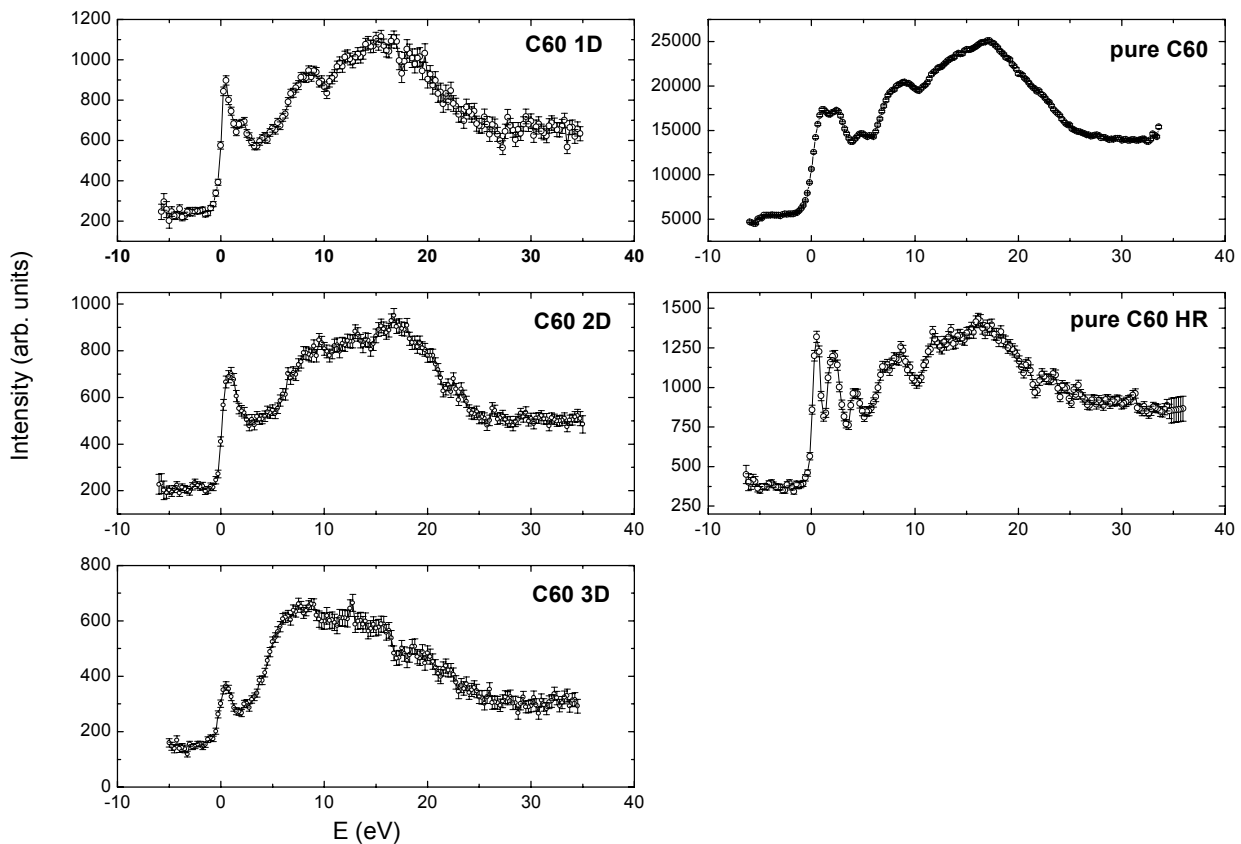


Figure 1: absorption spectra at K C edge in fullerene polymers. HR denotes spectra recorded with the 0.4 eV resolution setup.