EUROPEAN **S**YNCHROTRON **R**ADIATION **F**ACILITY

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ESRF

ESRF	**Experiment title:** Study of the low temperature phase of KC_{60} by anomalous x-ray diffraction	**Experiment number**: hs1704
Beamline: ID01	**Date of experiment**: from: 19/06/02 to: 24/06/02	**Date of report**: 27/08/02
Shifts: 18	**Local contact(s)**: Myles Hamilton	*Received at ESRF:*

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Report:

The intriguing physical properties of the alkali fullerides AC_{60} (A = K, Rb, Cs) are intensively studied. RbC_{60} and CsC_{60} are usually described as quasi-one dimensional (1D) materials with a change of electronic ground state, still debated, below 40K. On the other hand, KC_{60} is considered as a three dimensional (3D) metal although a broad upturn of the resistivity has been reported around 50 K [1].

In these materials, the C_{60} molecules form parallel polymer chains aligned in orthorhombic structures. The chain orientations – i.e. both the orientation angle around the chain axis and the relative orientation angles of neighbouring chains – are important structural characteristics that appear to be related to the electronic properties of these compounds. While the individual chain orientation angles are similar in the three compounds, different relative chain orientations have been found in KC_{60} (described in Pmnn space group) with respect to RbC_{60} or CsC_{60} (I2/m space group) [2,3].

Recent ESR spectroscopy and x-ray diffraction experiments at low temperature on KC_{60} single crystals have identified a new phase transition around 50K [4]. This transition leads to an insulating ground state and to the stabilization of a superstructure which doubles the volume of the unit cell. The structural changes responsible for the formation of the superstructure are crucial for understanding the driving mechanism of the transition. Several models have been suggested : displacements of the K atoms and/or a modulation of the C_{60} charge. Previous results [2] showing anomalous Debye-Waller parameters would support the first model (not excluding the others) but the overall

intensity distribution of the superstructure reflections is difficult to reconcile with atomic displacement effects [4]. The presently available data do not allow one to go any further.

The aim of this proposal was to gather new information on the nature of the superstructure from the study of anomalous diffraction effects at the K absorption edge. Even if it is too early to do any conclusion about the role of the potassium in this transition, we present here very briefly some results and difficulties encounters.

Difficulties and experiment:

The main difficulty was the use *of low energies (E<4keV), at low temperature (T<50K).* For this, the helix system provided by the support group has been used successfully (see figure 1). In addition, a particular attention has been given in limiting the air path. In particular, the Argon k-edge (E_k=2.98 keV) may give a strong inelastic contribution at energies close to 4keV if the air path is too long.

The air scattering has been limited to less than 10cm before and after the sample using flight tubes (see figure).

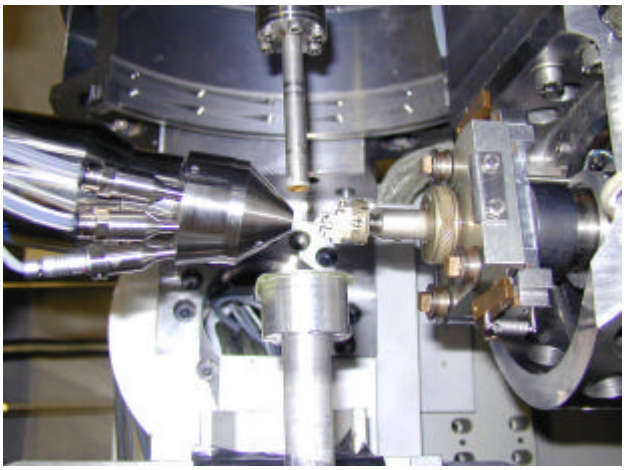

Figure 1: setting (He gaz flow and limited air path)

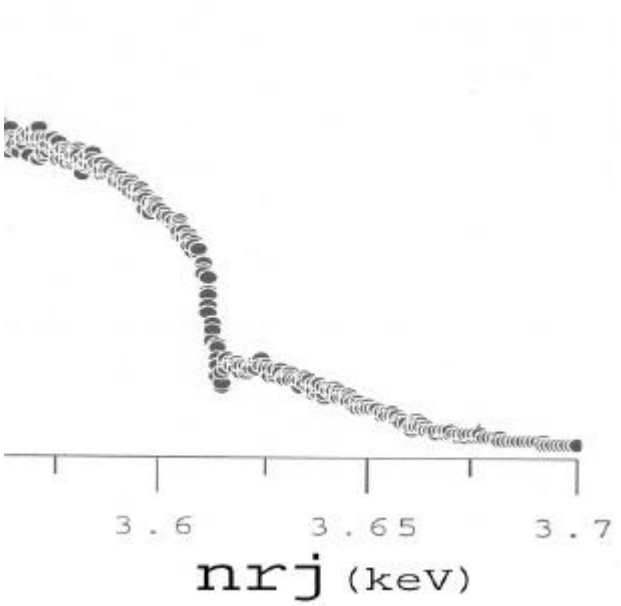

Figure 2: fluorescence of KC60 at the Potassium K edge (E=3.6keV)

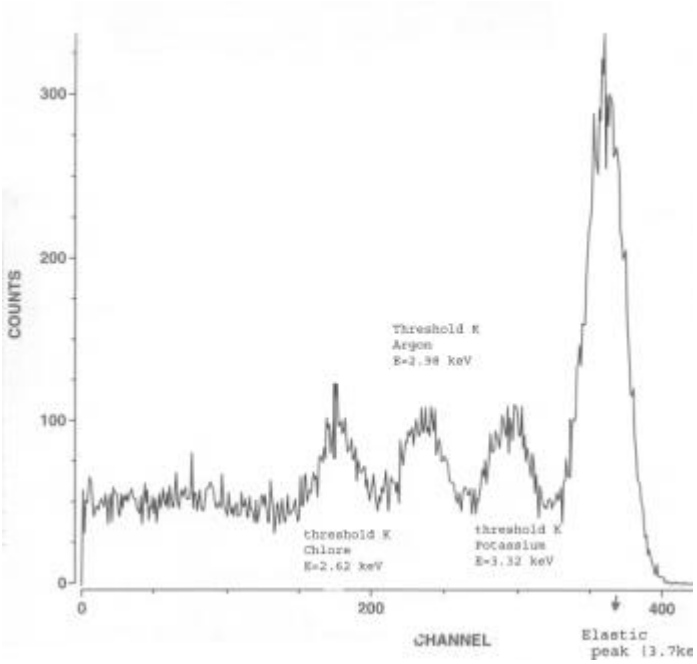

Figure 3: energy spectrum above the K edge of potassium(MCA channels). The K edge of chlorium comes from the MCA window. The argon contribution comes from the air. Compton contribution not visible.

The (11-1) Bragg reflection has been studied below and above the edge. The superstructure reflection (0.5;0;0.5) has been studied below the transition. Thanks to *in house* characterisation on rotating anode, the orientation has been done despite the presence of 12 orientation variants. In both cases, the intensity of the (11-1) and (0.5;0;0.5) superstructure has been measured as a function of energy across the K absorption edge (3.6 keV). Because of a lack of beamtime, only those two reflections have been observed. However, the DAFS pattern of the (11-1) Bragg reflection shows a clear different behavior before and after the transition. This preliminary observation tends to prove that the potassium plays a important role in the transition.