



ESRF

Experiment title:

Structural studies of fullerenes and heterofullerenes

Experiment

number:

CH262

Beamline:

BM16

Date of experiment:

from: 27 March 97 to: 31 March 97

Date of report:

29/8/97

Shifts:

9

Local contact(s):

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Received at ESRF:

1 SEP. 1997

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Report:

Fullerene superconductivity has been mostly dominated by the alkali intercalation compounds with stoichiometry A_3C_{60} . Much less is known about other fulleride superconductors, mainly because of the difficulties associated with the preparation of phase-pure bulk samples. Typical among these are the alkaline-earth superconductors, AE_xC_{60} ($AE = Ba, Sr$) which show superconductivity in the 4- 7 K range and in which the (LUMO+1) t_{1g}-derived band is now populated. The true stoichiometry of these superconducting phases has been controversial. Our recent work¹ has been successful in isolating a number of these phases in pure form and thus unambiguously showing that both Ba_4C_{60} and Sr_4C_{60} are superconducting, while Ba_6C_{60} and Sr_6C_{60} , though metallic, are not. As part of our present beam allocation on BM16, we have now performed X-ray diffraction measurements on an almost phase-pure Ba_4C_{60} sample. Rietveld refinements show that Ba_4C_{60} adopts at room temperature an orthorhombic structure with lattice constants, $a = 11.6105(3) \text{ \AA}$, $b = 11.2354(3) \text{ \AA}$, $c = 10.8834(2) \text{ \AA}$. Extensive searches of minima in R_{wp} as a function of C_{60} orientation have produced two candidates for the correct structure, related by a 90° rotation about the c axis of the unit cell, with space groups *Immm* and *Pnmm*. Fig. 1 shows the results of the full *Immm* refinement and Fig. 2 shows the derived Ba_4C_{60} in this case.

References

[1] Gogia, B. et al., in preparation; Brown, C. M. et al., in preparation.

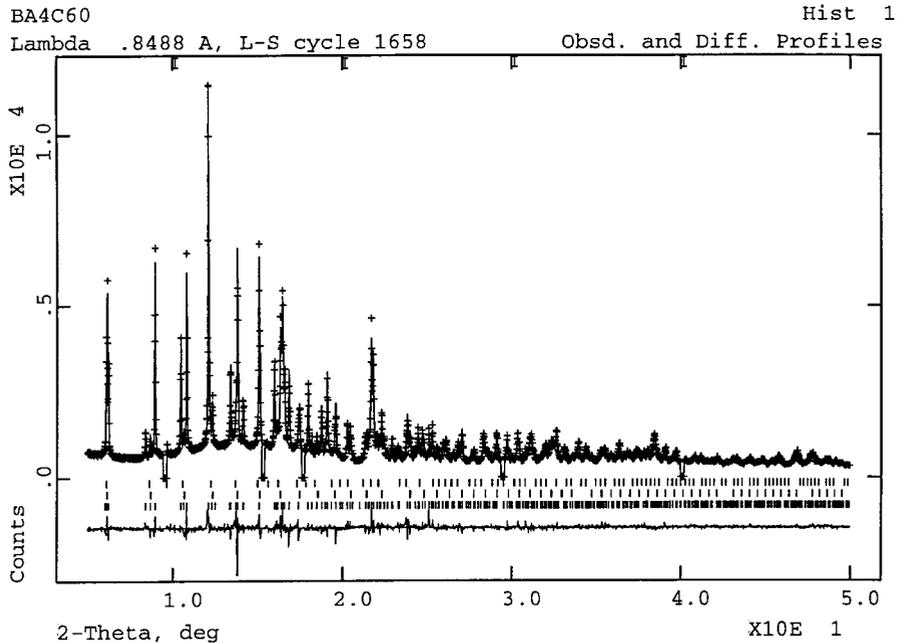


Fig. 1 Rietveld refinement of the synchrotron X-ray diffraction profile of Ba_4C_{60} at room temperature in space group $Immm$ ($\lambda = 0.84884 \text{ \AA}$). Small fractions of Ba_6C_{60} (11.5(2)%) and Ba_3C_{60} (4.7(3)%) phases have been encountered.

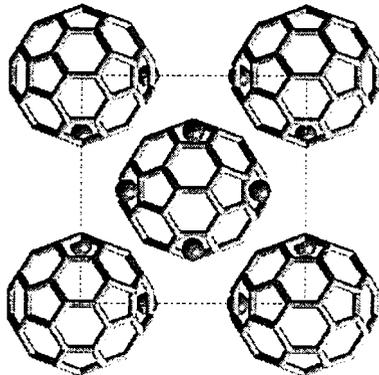


Fig. 2 Projection of the Ba_4C_{60} structure on the (ab) plane.