

**Experiment title:**

Structural studies of fullerenes at high pressures and low temperatures

Experiment number:

CH277

Beamline:

ID30

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12

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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 ID30, we performed angle dispersive X-ray diffraction measurements on a multiphase Ba_xC_{60} sample to pressures as high as 10 GPa. The data were of excellent quality and Rietveld refinements were routinely performed, even though the sample proved to comprise of three phases: Ba_6C_{60} , Ba_4C_{60} and Ba_3C_{60} . The refined fractions were 66.6%, 29.7% and 3.7%, respectively. While both Ba_6C_{60} and Ba_3C_{60} are cubic ($a = 11.2016(7)$ Å and $a = 11.363(4)$ Å, respectively, near

ambient pressure), Ba_4C_{60} adopts an orthorhombic structure (space group Immm, $a=11.603(2)$ Å, $b=11.288(2)$ Å, $c=10.897(2)$ Å near ambient pressure). Fig. 1 shows the evolution of the volume of the unit cell for the three phases as a function of pressure and Fig. 2 shows the corresponding pressure dependence of the lattice constants for Ba_4C_{60} , displaying a remarkably anisotropic compressibility with the short c-axis showing little change with increasing pressure.

References

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

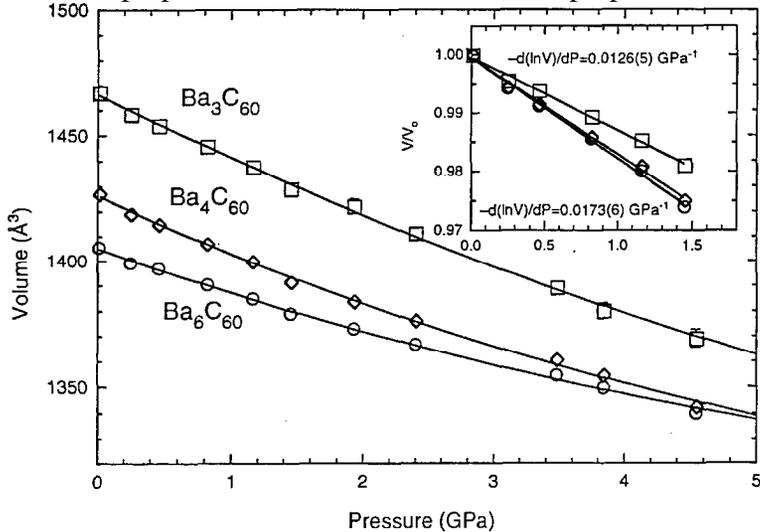


Fig. 1 Pressure evolution of the unit cell volume, V for Ba_6C_{60} , Ba_4C_{60} and Ba_3C_{60} .

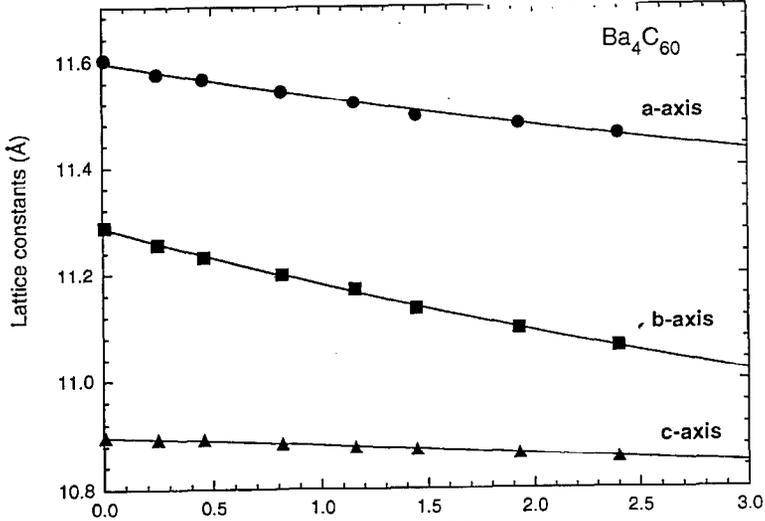


Fig. 2 Pressure evolution of the lattice constants of the orthorhombic unit cell of Ba_4C_{60} .