Very High Electrical Conductivity in AsF_5–Graphite Intercalation Compounds

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Summary Arsenic pentafluoride–graphite intercalation compounds of uniform stages 1—3 have been prepared by direct synthesis; very high *a*-axis electrical conductivities, similar to that of copper, were determined by r.f. (100 kHz) and d.c. techniques.

WE report the synthesis of AsF_5–graphite compounds which show very high electrical conductivities comparable to that of copper. Lamellar intercalation compounds of uniform stages 1—3 have been prepared at ambient temperature by direct interaction of AsF_5 vapour with highly oriented pyrolytic graphite (HOPG). Compounds more dilute than stage 3 were observed to be inhomogeneous mixtures of stages. HOPG has good *c*-axis alignment (*ca.* 1°) which is essential to the observation of representative *c*-axis thickness change and *a*-axis conductivity measurements. Gravimetric analyses clearly demonstrated maximum AsF_5 concentrations with compositions corresponding to C_{8n}-AsF_5 (where *n* is the stage). This is in contrast to previous work[1] which indicated a compound with composition $C_{10\cdot0}AsF_5$ resulting from reaction of AsF_5 with graphite powder or grafoil.

Reactions were terminated at the desired stage by monitoring *c*-axis thickness with a microscope equipped with a calibrated reticule, layer spacings having been determined from *X*-ray analysis. Obvious 'staging,' observed as plateaus in *c*-axis thickness as a function of time, facilitated this technique. Maximum AsF_5 concentration for a particular stage was obtained by terminating reactions at the end of a plateau.

These compounds show an impressive lack of exfoliation or edge fraying, expanding as perfectly uniform platelets. This property, contrasting strongly with compounds with SbF_5 and other intercalates, contributes substantially to ease of stage selection and measurement of electrical conductivity. Stages 1—3 have highly specular surfaces (*c*-face) and are characterized by metallic blue, silver with a faint blue tint, and silver colourations respectively. The materials are stable in an environment of dry nitrogen sealed in Pyrex tubes but decompose under dynamic vacuum. Lower stages fume in moist air and are generally less stable than the more dilute compounds.

The room temperature *a*-axis electrical conductivity has been measured by two techniques. R.f. measurements have been made at 100 kHz using an eddy current technique[2] which has two advantages. First, making good electrical contacts which will withstand both chemical attack and mechanical stress on *c*-axis expansion is not straightforward. The r.f. technique is contactless. Second, the eddy currents are planar and the method is therefore insensitive to the *c*-axis conductivity, a parameter of only secondary interest, and a source of difficulty in making conventional four point d.c. bridge measurements. The system has been calibrated using a range of materials of known conductivity (including copper). Measurements of pristine graphite[3] and alkali metal intercalated HOPG gave conductivities in good agreement with previously published values determined by more conventional techniques. D.c. measurements by Montgomery's method,[4] a technique specifically developed for measurement of highly anisotropic materials, permitted measurement of both the *a*-axis and *c*-axis conductivities, σ_a and σ_c, respectively.

Results for both methods indicate an *a*-axis conductivity with a maximum in the vicinity of stage 3. A maximum *a*-axis conductivity of $6{\cdot}2 \times 10^5\ \Omega^{-1}\,cm^{-1}$ is observed (25 times pristine graphite) compared to $5{\cdot}8 \times 10^5\ \Omega^{-1}\,cm^{-1}$ for OFHC copper. The nature of the r.f. measurement and a thorough analysis of our Montgomery measurement tend to suggest that this conductivity may be conservative. The Montgomery measurements indicate a peak anisotropy ratio $\sigma_a/\sigma_c > 10^6$ compared to *ca.* 2×10^3 for pristine graphite. These sample parameters indicate the impractical nature of a simple four point bridge measurement and indicate a probable source of the substantial discrepancy between previously reported values for SbF_5 graphite.[5]

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