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Published in:
Nature
DOI:
10.1038/350600a0

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Document Version
Publisher's PDF, also known as Version of record

Publication date:
1991

Link to publication in University of Groningen/UMCG research database

Citation for published version (APA):

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Superconductivity at 18 K in potassium-doped C\textsubscript{60}

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The synthesis of macroscopic amounts of C\textsubscript{60} and C\textsubscript{70} (fullerenes)\textsuperscript{1} has stimulated a variety of studies on their chemical and physical properties\textsuperscript{2,3}. We recently demonstrated that C\textsubscript{60} and C\textsubscript{70} become conductive when doped with alkali metals\textsuperscript{4}. Here we describe low-temperature studies of potassium-doped C\textsubscript{60} both as films and bulk samples, and demonstrate that this material becomes superconducting. Superconductivity is demonstrated by microwave, resistivity and Meissner-effect measurements. Both polycrystalline powders and thin-film samples were studied. A thin film showed a resistance transition with an onset temperature of 16 K and essentially zero resistance near 5 K. Bulk samples showed a well-defined Meissner effect and magnetic-field-dependent microwave absorption beginning at 18 K. The onset of superconductivity at 18 K is the highest yet observed for a molecular superconductor.

The conductivity measurements were performed on potassium-doped films of C\textsubscript{60} that were prepared in a one-piece all-glass version of the apparatus described previously\textsuperscript{4}. This configuration allowed both in situ doping and low-temperature studies of thin films. All measurements were made in a four-terminal Van der Pauw configuration using a 3-\mu A a.c. current at 17 Hz. Figure 2 shows the temperature dependence of the resistivity of a 960-Å-thick K\textsubscript{x}C\textsubscript{60} film. The film was doped with potassium until the resistivity had fallen to 5 \times 10\textsuperscript{-7} \Omega cm. The resistivity increases by a factor of two on cooling the sample to near 20 K. Below 16 K, the resistivity starts to decrease; zero resistivity (\textless 10\textsuperscript{-4} of the normal state) is obtained below 5 K. The 10-90% width of the transition is 4.6 K. At 4 K we measured the lower bound to the critical current to be 40 A cm\textsuperscript{-2}.

A bulk polycrystalline sample of nominal composition K\textsubscript{x}C\textsubscript{60} was prepared by reaction of 29.5 mg of C\textsubscript{60} with 4.8 mg potassium. The amount of potassium was controlled volumetrically by using potassium-filled pyrex capillary tubing cut to size in a dry box. The reaction was run with the C\textsubscript{60} in a 5-mm fused silica tube joined to a larger tube in which the potassium-containing capillary was placed. The tube was sealed after being evacuated and refilled with 10\textsuperscript{-2} torr of helium to serve later as a thermal-exchange gas for low-temperature measurements. With the C\textsubscript{60}-containing end of the tube at room temperature,
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FIG. 2 Temperature dependence of the electrical resistivity of a 960-Å-thick film of K$_x$C$_{60}$.

the potassium was distilled from the capillary in a furnace at 200°C. Some reaction of the potassium with the quartz tube, visible as a dark brown discoloration, was observed at this temperature. Unreacted potassium was observed after this period. Following distillation of the potassium to the C$_{60}$ end, the tube was shortened by sealing to about 8 cm and heated to 200°C for 36 h. Finally, the tube was resealed to a length of about 4 cm for magnetic measurements.

The temperature dependence of the d.c. magnetization of the sample with nominal composition K$_3$C$_{60}$ was measured in a SQUID magnetometer (Fig. 3). On zero-field cooling the sample to 2 K, a magnetic field of 50 Oe was applied. On warming, this field is excluded by the sample to 18 K; this verifies the presence of a superconducting phase. The bulk nature of superconductivity in the sample is demonstrated unambiguously by cooling in a field of 50 Oe. A well defined Meissner effect (flux expulsion) develops below 18 K. The shape of the magnetization curve, in particular the temperature-independent signal at low temperature, indicates good superconducting properties for this sample. Also noteworthy is the relatively narrow transition width. The magnitude of the flux exclusion for the zero-field-cooled curve corresponds to 1% volume fraction. This small fraction is possibly due to non-optimal doping or the granular nature of the sample. The large value of the Meissner effect for the field-cooled curve relative to the total exclusion, however, indicates bulk superconductivity in the electrically connected regions.

The universally accepted tests for superconductivity, namely a transition to zero resistance and a Meissner effect showing the expulsion of magnetic field, demonstrate unequivocally the existence of superconductivity in K$_x$C$_{60}$. The 18-K transition temperature is the highest yet reported for a molecular superconductor. This may be compared with the previously reported occurrence of superconductivity at 0.55 K in potassium-intercalated graphite. We expect that optimization of composition and crystallinity will lead to further improvement in the superconducting properties.

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FIG. 3 Temperature dependence of the magnetization of a K$_x$C$_{60}$ crystalline sample. The direction of temperature sweep in the field-cooled (FC) and the zero-field-cooled (ZFC) curves is indicated by the arrows.