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35. Roughly 4000 additional acidic lakes in the eastern United States are smaller than the NSWS 4-ha nominal lower size limit (*8*). In the Adirondacks, lakes less than 4 ha are more likely to be acidic than are larger lakes, but they are also more strongly influenced by organic acids [T. J. Sullivan *et al.*, *Water Resour. Bull.* **26**, 1 (1989)]. There are four to nine times as many streams as are represented on the 1:250,000-scale maps from which the NSWS stream target population was drawn, although many of these unmapped streams are small ephemeral or intermittent streams unsuitable for fish. More importantly, we estimate that 1700 acidic streams in the Northeast were not sampled in the NSWS (*8*).

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Alkali-Fulleride Superconductors: Synthesis, Composition, and Diamagnetic Shielding

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The recent report of a superconductivity onset near the critical temperature $T_c = 18$ K in potassium-doped C_{60} raises questions concerning the composition and stability of the superconducting phase. The effects of mixing and heat treatment of $K_x C_{60}$ samples prepared over a wide range of initial compositions on the superconducting transition was determined from shielding diamagnetism measurements. A single superconducting phase ($T_c = 19.3$ K) occurs for which the composition is K_3C_{60}. The shielding reaches a maximum of greater than 40 percent of the perfect diamagnetism, a high value for a powder sample, in samples prepared from 3:1 mixtures. A Rb_xC_{60} sample prepared and analyzed in an analogous way exhibited evidence for superconductivity with $T_c = 30$ K and a diamagnetic shielding of 7 percent could be obtained.

THE RECENT DISCOVERY (*1*) AND separation (*2, 3*) of molecular forms of solid carbon has made possible the formation of new semiconducting (*4*) and conducting (*5*) ^{2}anionic charge-transfer compounds of C_{60} (and C_{70}) with molecular and alkali counterions, respectively. From the latter studies, Hebard *et al.* (*6*) have provided unambiguous evidence for superconductivity in solids composed of icosahedral C_{60} molecules "doped" with potassium (K_xC_{60}) with an onset near $T_c = 18$ K. They combined three experiments on two different morphologies (microwave absorption and magnetic susceptibility χ of powders, and dc resistivity of films) to assert superconductivity under unusually difficult, poorly controlled chemical conditions. As the powder is prepared by a solid-state reaction, only the initial composition is known, so no claim could be made about the homogeneity of the end product. Therefore, the actual composition of the superconducting phase was not directly determined, and the lower bound of 1% superconducting phase, established by shielding diamagnetism measurements, leaves open questions about the compositional stability of this phase within the K-C_{60} phase diagram.

We report measurements of the shielding diamagnetism curves (χ versus T) conducted over a range of compositions and treatments with the aim of separating and identifying the superconducting phase. In an attempt to narrow the composition range of the superconducting material, the initial composition was systematically varied to locate the maximum fraction of shielding diamagnetism, which is a measure of the actual quantity of superconducting material. This maximum, found at a composition K_3C_{60}, is >40%, a high value for a powder sample. This phase appears to be stable, that is, it is present after indefinitely long heating and mixing, for all nominal sample compositions, x <6. When this same procedure

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was applied to Rb-doped C_{60}, a 7% shielding diamagnetism was measured on a sample, starting at an apparent $T_c = 30$ K.

The molecular carbon sample was prepared following the resistive-heating procedure of Krätschmer *et al.* (*1*) as described by Ajie *et al.* (*2*) followed by solvent extraction, chromatographic separation of higher fullerenes, and several hours drying of solvent under vacuum. The potassium (Alfa, 99.95%) and rubidium (Mackay, high purity) were used as obtained. All manipulations were performed in a dry box under He atmosphere, with an O_2 background level <1 ppm. Our procedure for reacting the solids and for obtaining homogeneous samples differs from the previous report in that it consists of three heat treatment stages, as follows:

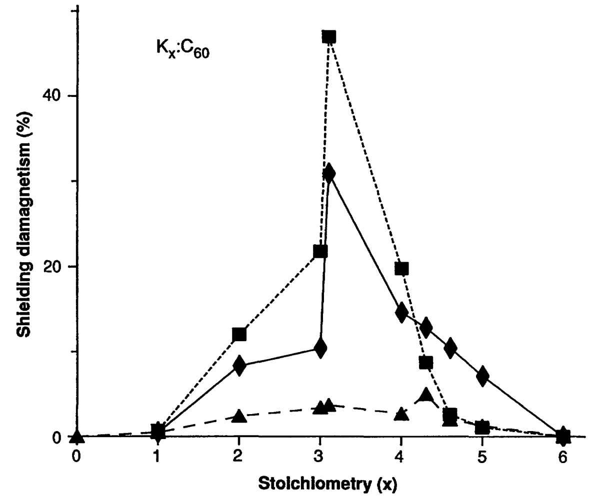

Fig. 2. The shielding diamagnetism, $\chi_{dia}(T)$, of a series of samples plotted versus nominal sample composition x at the three stages of treatment.

1) In the mixing stage, a given quantity of K was sealed under vacuum with C_{60} powder (typically 6 to 25 mg) in a 5-mm or 8-mm diameter Pyrex tube. This tube was placed in a furnace at 200°C for 20 to 24 hours, during which time the K appears to be completely absorbed by the C_{60} powder, which has negligible vapor pressure at this temperature. Residual unreacted K was evident only for mixtures of 6:1 composition and greater. The material was collected, put in a capillary tube (1.5 mm diameter), and sealed under (1 bar) He for initial shielding diamagnetism measurements.

2) In the diffusion stage, the samples from stage 1, still in the capillary under 1 bar He, were again heated to 200°C for ~22 hours, after which time the shielding diamagnetism measurement was repeated.

3) In the relaxation stage, we checked that an equilibrium had been achieved by heating the samples from stage 2 again but at 250°C for six or more hours, and additional changes in the shielding diamagnetism were measured. Further heat treatment on a limited number of samples revealed no further changes. Samples appear to be completely stable for at least 2 weeks.

Shielding diamagnetism curves, the primary quantity used here to assess the amount (volume fraction) of the superconducting phase and its homogeneity, were obtained with a standard magnetic inductance coil bridge, operated at 100 kHz with phase-sensitive detection (*7*). The amplitude of the ac susceptibility was typically measured over the range from 4 to 100 K. The diamagnetic contribution, defined here as $\chi_{dia} = \chi_{normal} - \chi(4.2\ K)$, is expressed as a fraction of a Nb standard. No correction has been applied to take into account the degree of compactness of the powder nor the magnetic field distribution inside the capillary. These effects both tend to reduce the observed superconducting fraction, so that the estimates serve as conservative lower bounds.

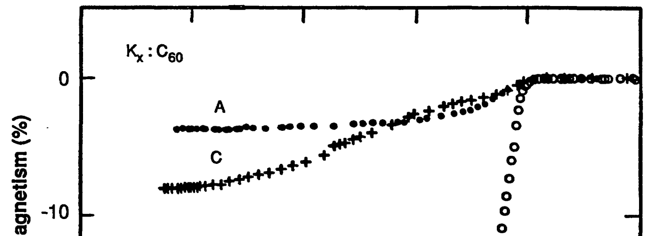

Fig. 1. Magnetic susceptibility curves $\chi(T)$ for three samples of K_xC_{60} powder exhibiting shielding diamagnetism expressed as a percentage of a Nb standard measured at 4.2 K. Curves A and B represent the same sample (nominal composition 3:1) after the mixing and final stages, respectively. Curve C is a composition 3.5:1 sample after the mixing stage.

Three characteristic diamagnetic shielding curves representative of those obtained on 15 samples of ten different nominal compositions at the varying stages (1, 2, and 3) are shown in Fig. 1. Curves A and B are of the shape expected for a single phase transition occurring at 19.3 K. The transition is sharp; the 10 to 50% full width of the diamagnetism transition is <1 K. These represent samples of x:1 nominal composition measured at stages 2 and 3, respectively. Curve C is typical of the measurement at the mixing stage on samples of nominal composition ≥ 3:1. These curves exhibit a more complicated structure. Their unusual form, somewhat similar to that of Hebbard *et al.* (*6*), might indicate the presence of more than one phase or a continuum of phases, or even grains with superconducting surface and normal interiors. After subsequent treatment (diffusion and relaxation stages), these samples also show curves of the same form as A and B, indicative of a single superconducting phase.

The different forms of the diamagnetic shielding curve $\chi(T)$ found after the mixing stage give the impression that, despite the apparently complete macroscopic mixing, there exist many different compositions (or a continuum) of K_xC_{60}. None of these exhibit diamagnetism above 20 K. Among these, the curves like C resemble most the one shown in (*6*). Second, after stage 2, the various mixtures all have curves for which the amplitudes can be scaled to match one another, with an always sharp, identical transition temperature of $T_c = 19.3$ K, which implies that there is only one stable superconducting phase of K_xC_{60}. Thus the other phase (or phases) that account for the bulk of the material in most samples are *not* superconducting above 1.5 K or failed to segregate under the preparation conditions used.

In an attempt to determine the actual composition of this superconducting phase, we examined how the volume percent of diamagnetic shielding varied with composition at the mixing stage and at nominal equilibrium. In Fig. 2 the diamagnetic shielding, as defined above (χ_{dia}), is plotted against nominal composition (Table 1). At the first stage (mixing), the maximum in the χ_{dia} versus x curve is definitely at $x > 3$. At later stages, the χ_{dia} increases strongly for small $x \leq 3$, but decreases for most of the samples with $x > 4$. At the final stage, the $x = 3$ sample exhibits the largest shielding observed, 40% or more, a high value for a powder sample. As the typical grain size of the powder determined with an optical microscope is ~1 μm, this is necessarily a lower bound on the fraction of the sample that is in the superconducting phase; it could be that this sample attains a high degree of homogeneity. Despite their reputed air sensitivity (*1*) a powder sample exposed to air after 3 hours continued to exhibit a Meissner effect of one-half its original magnitude, and showed no diamagnetic shielding after 3 days.

We conclude that the single superconducting phase has a composition close to, or slightly greater than 3:1, and definitely less than 4:1. However, the surprising stability and reproducibility of T_c favors a stoichiometric composition, hence K_3C_{60}, which confirms the hypothesis of the AT&T group (*5, 6*). After completing this systematic study on relatively small amounts of material, we prepared several larger quantities (20 to 30 mg each) with the initial composition 3.1:1 and subjected them to extended heat treatments. These resulted in reproducible shielding diamagnetism fractions ranging from 40 to 67%. Keeping in mind that this measurement results in an underestimation of the actual superconducting fraction, it becomes clear that we have found the means to produce samples in which the K_3C_{60} phase is in the definite majority, with "impurity" phases as low as a few percent remaining to be removed, rather than the opposite situation in which the superconducting phase is a very minor component.

Table 1. Variation of fractional shielding diamagnetism as a function of composition x during different stages of preparation. The relaxation stage values were reproducible to ±5% (determined for two or more samples at several compositions).

x	Mixing	Diffusion	Relaxation
1.0	0.5	0.5	0.7
2.0	2.4	8.3	12.0
3.0	3.4	10.4	21.8
3.1	3.7	30.9	47.0
4.0	2.7	14.6	19.8
4.3	5.0	12.8	8.7
4.6	2.0	10.4	2.6
5.0	1.3	7.1	1.1
6.0	0.0	0.0	0.0

The mixing and heat treatment procedure developed on the K_xC_{60} system was used to explore other members of the alkali-doped fullerene class of materials, namely Rb and Cs. Although experiments on Cs_xC_{60} are so far inconclusive in showing signs of a phase transition, a Rb sample prepared from a 3:1 mixture exhibited a clear transition at T_c = 30 K with a diamagnetic shielding not less than 7% (Fig. 3). The sharpness (10 to 50% width of ~2 K) and distinctness (no other features) of this transition indicate that a well-defined composition is responsible (*8*).

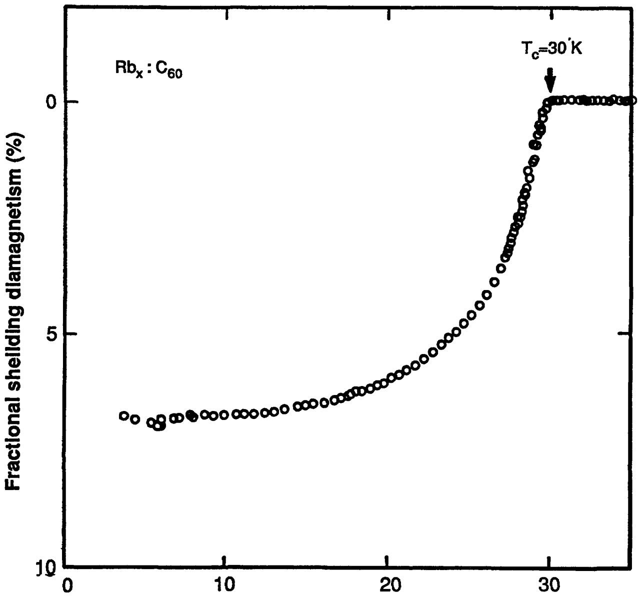

Fig. 3. Magnetic susceptibility curves $\chi(T)$, as in Fig. 1, for a Rb_xC_{60} sample. A single transition is observed at an apparent T_c = 30 K.

We now consider the kinetics of doping, both of the solid-gas reaction and diffusion in the solid. Besides the maximum at $x = 3$ (Fig. 2), there are two surprising aspects. For nominal compositions $x < 3$, and even at $x = 1$, observable shielding diamagnetism is present and increases monotonically with x toward the maximum, suggesting that there are macroscopic precipitates of the superconducting phase to provide a continuous pathway for shielding-current loops. For $x > 3$, the diamagnetic shielding increases during the diffusion stage, but then decreases as the final homogenization is assumed to occur, so that it achieves a maximum at an intermediate stage of the diffusion. All of these observations are consistent with the following simple picture of the doping process:

1) Mixing stage. In vacuum, the entire quantity of alkali metal is vaporized and adsorbed (irreversibly under the conditions used, $T < 300°C$) onto the C_{60} powder granules, which we suppose produces a uniform surface concentration (coverage) of all granules. Accompanying the adsorption, a limited amount of inward diffusion occurs, leading to a steep K concentration gradient toward the grain interiors. Even if at some radial distance the right composition is reached, no shielding would be observed unless the thickness of this shell of superconducting material is comparable to the penetration depth of the superconductor. Separate measurements (*9*) of the critical-field curves have evaluated this quantity to be λ = 2500 Å, or about one-fourth of the mean grain size. In fact, the criterion that the grain as a whole is shielded is that it has a closed shell of superconducting material of thickness greater than λ located somewhere between the surface and the grain center.

2) Diffusion and equilibration processes. The effective irreversibility of the adsorption process implies that the quantity of K adsorbed onto a C_{60} grain is proportional to its surface area, rather than to its volume (or quantity of C_{60}). Therefore the smaller than average grains take up more K per volume than average, and the larger grains have less K. In the second stage, under the He atmosphere, diffusion of dopant within a particle takes place. We believe that the small shielding diamagnetism observed at one-third of the optimum composition, that is, nominally 1:1 in Fig. 2, is produced in this way, by generating a 3:1 composition in small grains, which have a surface-to-volume ratio

such that after the first stage they have the right quantity to yield a sufficiently homogeneous particle of 3:1 composition. This process provides the continuous pathway for shielding currents. At the other extreme of nominal composition, the smaller grains become superconducting during the diffusion stage, but upon homogenization this superconductivity is destroyed by the excess K, much as the normal state conductivity is destroyed by extended doping (*5*), as supported by photoemission results (*10*) and orbital-filling arguments (*5*). On the other hand, the larger than average grains, at equilibrium, can turn out to have the right concentration to be superconducting. This accounts for the nearly symmetrical curve of shielding diamagnetism curve versus nominal composition (Fig. 2) and also for the rise and fall of shielding diamagnetism as a function of heat treatment in the high-x samples. The sample with the observed maximum shielding diamagnetism corresponds to transforming to the right concentration the average grain size and hence the majority of grains. We believe this conclusion reflects the inherent limitations of the gas-solid reaction procedure. Different techniques, such as precipitation from organic solution, could result in higher yields. This is why we view the >40% value as a very high yield from these powder samples.

The actual quality of the material obtained here has provided the capability to measure critical field curves $H_{c1}(T)$ and H_{c2} (T) (*9*). The zero-temperature extrapolated values are $H_{c1}=132$ G and $H_{c2}=49$ T. These values allow one to evaluate the penetration depth $\lambda = 2400$ Å and the coherence length $\xi = 26$ Å, and hence a Ginzburg-Landau parameter $\kappa = \lambda/\xi$ around 100, an extremely high value approaching that of the high-T_c copper oxides.

We find that it is the K_3C_{60} composition that is the superconducting phase, and neither under- nor overdoped phases (relative to the correct one) are superconducting. The discovery of the $T_c = 30$ K superconducting phase of Rb_xC_{60} should stringently test the different theoretical possibilities, as it offers the possibility of comparing the dependence of T_c on slight chemical modifications. Prerequisite to this is a completion of the systematic study of the phase diagram of Rb and other alkali-C_{60} mixtures.

Note added in proof: During these studies we learned that Rosseinsky *et al.* (*11*) found a superconducting transition at 28 K in a Rb-doped C_{60} sample.

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New Mechanism of Cavitation Damage

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Enormous impact pressures can develop when a vapor cavity collapses in a liquid, and it is generally held that these pressures are the underlying cause of cavitation damage of surfaces. The rapid growth and disappearance of vapor cavities have been viewed with a surface forces apparatus in liquids, and their effects on nearby surfaces at the submicroscopic level have been simultaneously monitored. The inception of cavities is intimately connected with simultaneous relaxations of high local strain energies on nearby surfaces, and in many practical situations, damage is more likely to occur during the formation, rather than the collapse, of cavities.

CAVITATION DAMAGE OF SURFACES in liquids is an important industrial and biological problem, for example, in the damage or erosion of propeller blades, high-speed lubricated bearings, and metal surfaces subjected to ultrasonic vibrations, in the wear of knee joints, and in decompression sickness (the bends). On the basis of Rayleigh's classic 1917 paper on collapsing bubbles (*1*), cavitation damage was thought to be due solely to the extremely large implosive pressure generated at the moment when a vacuum cavity or bubble collapses. During the 1960s, it was shown that bubbles deform during collapse and that damage is also caused by the impacts of high-speed liquid jets that strike surfaces during the collapse phase, producing tiny pits or craters on the surfaces (*2*). The evidence that most directly links surface damage to bubble collapse has come from experiments with hydrofoils in cavitation tunnels, which generally show that maximum erosion along a hydrofoil surface usually correlates well with the location of collapsing bubbles (*3*).

Less direct but more detailed information has been gained from laboratory experiments in which high-voltage electric sparks or pulsed laser beams are used to artificially nucleate bubbles at specific locations within a liquid near a surface. Their subsequent time evolution is then recorded with a high-speed camera (*4, 5*). Such experiments have so far not been able to determine the exact stage of the inception-growth-collapse cycle of bubbles at which damage occurs (*6*). The whole process of cavity growth and collapse usually occurs very rapidly (10^{-6} to 10^{-3} s), and all of the interesting features have submicroscopic dimensions. For these reasons, it has been difficult to study the rapid growth and collapse of cavities and especially the relation of these processes to the (elastohydrodynamic) surface deformations and, ultimately, damage. However, it is generally believed that damage occurs only during the collapse (*4–7*).

The subject has received much theoretical attention, but it has proved too difficult to arrive at a general theory that satisfactorily accounts, even qualitatively, for many of the observed phenomena (*6, 8*). Most theoretical treatments start with cavities or gas bubbles already present in the liquid and then attempt to determine the course and consequence of their collapse. Curiously, little theoretical or experimental attention has been given to bubble formation under natural conditions and the effects of the first shock wave produced by rapidly growing bubbles (*6*).

Using the surface forces apparatus (SFA) technique (*9*) in studies of elastohydrodynamic deformations of two curved surfaces moving toward or away from each other in liquids, we noticed that vapor cavities developed when the surfaces were separated faster than some critical velocity. Because the optical technique used in these measurements (*10*) enables one to follow surface deformations in real time (*11, 12*) and to observe vapor cavities form (*13*) at the nanometer level, we decided to study the phenomenon of cavity formation and collapse in detail.

The SFA, with its molecularly smooth

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