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Organic Molecular Soft Ferromagnetism in a Fullerene C_{60}

Pierre-Marc Allemand, Kishan C. Khemani, Andrew Koch, Fred Wudl, Karoly Holczer, Steven Donovan, George Grüner, Joe D. Thompson

The properties of an organic molecular ferromagnet [$C_{60}TDAE_{0.86}$; TDAE is tetrakis(dimethylamino)ethylene] with a Curie temperature T_c = 16.1 kelvin are described. The ferromagnetic state shows no remanence, and the temperature dependence of the magnetization below T_c does not follow the behavior expected of a conventional ferromagnet. These results are interpreted as a reflection of a three-dimensional system leading to a soft ferromagnet.

THE QUEST FOR A NONPOLYMERIC organic ferromagnet has intensified during the past 5 years with varying results (*1–10*). Curie temperatures (T_c's) on the order of 1 to 2 K have been observed (*7–12*). An organometallic molecular ferromagnet with T_c of ~4 K has been known for sometime (*13*), and similar systems with T_c of 6.2 K (*14*) and 8.8 K (*15*) have recently been reported. Polymeric ferromagnets with T_c's claimed to be greater than 300 K have also been described but their characterization remains incomplete (*2, 16*), and the possibility of a polaronic, polymeric ferromagnet has appeared (*17*). We report on the preparation and preliminary characterization of an organic molecular solid with a transition at 16 K to a soft ferromagnet state.

Our interest in the preparation of materials based on the reduction (*n*-doping) (*18, 19*) of fullerenes (*20–25*) prompted us to explore the use of strong organic reducing agents such as tetrakis(dimethylamino)ethylene (TDAE). Addition of a 20 M excess of the donor to a solution of C_{60} in toluene in a dry box afforded a black microcrystalline precipitate of $C_{60}(TDAE)_{0.86}$ [or $(C_{60})_{1.16}TDAE$] (*26*). The extremely air-sensitive (*27*) solid was washed with toluene and loaded into a capillary tube prepared from a Pasteur pipette, which had been previously sealed at the narrow end. The wide bore end of the pipette was then connected to a 5-mm vacuum stopcock, and the pipette was removed from the dry box to seal the sample under He in a vacuum line at ~2 cm above the powder fill line. The sample was then cooled in zero field in an ac susceptometer and then allowed to warm in an applied field of ≤0.1 Oe. Surprisingly, the transition observed was apparently to a ferromagnetic state.

The sample was studied in greater detail by dc magnetization (M) measurements taken on a Quantum Design superconducting quantum interference device (SQUID) magnetometer. Care was taken to ensure that the sample was not exposed to a field gradient greater than 0.03% of the applied field. In Fig. 1 we show M as a function of T for the sample cooled and warmed in an applied field H_a ~ 1 Oe. Two significant observations can be made from Fig. 1: (i) although M increases sharply below T_c ~ 16.1 K as expected for a ferromagnet, the temperature dependence of M does not follow that of conventional mean field theory; and (ii) within experimental error, there is no hysteresis between cooling and warming. The structure in $M(T)$ below 10 K is field-dependent; similar measurements in applied fields of 10, 100, and 1000 Oe show that with increasing field the minimum at 8 K

P.-M. Allemand, K. C. Khemani, A. Koch, F. Wudl, Departments of Chemistry and Physics and Institute for Polymers and Organic Solids, University of California, Santa Barbara, CA 93106.

K. Holczer, S. Donovan, G. Grüner, Department of Physics, University of California, Los Angeles, CA 90024.

J. D. Thompson, Los Alamos National Laboratory, Los Alamos, NM 87545.

seen in Fig. 1 evolves into only a change in slope for H_a = 1000 Oe. That the 16 K transition is one to a ferromagnetic state also is suggested by susceptibility $\chi \equiv M/H_a$ measurements as a function of temperature with H_a = 10 kOe. In Fig. 2, the product χT is plotted as a function of T. Above $\sim$30 K, χT increases approximately linearly with T, reflecting a weakly varying χ at these temperatures. However, below $\sim$30 K, χT increases sharply as expected from the onset of ferromagnetic correlations precursive to a ferromagnetic transition at T_c = 16.1 K. The maximum in χT at 10 K and the decrease at lower temperatures is a consequence of moment saturation below T_c. The absence of high-temperature Curie-Weiss susceptibility with a positive θ greater than T_c, though unusual for local moment ferromagnets, may be found in itinerant ferromagnets (see below) (*28*). Additional evidence supporting a ferromagnetic interpretation for $C_{60}(TDAE)_{0.86}$ comes from measurements of the field dependence of T_c, defined by the intersection of linear extrapolations of $M(T)$ from above and below the rapid rise in $M(T)$. The inset of Fig. 2 shows T_c increasing to 17.2 K for H_a = 1 kOe and then increasing approximately linearly with H_a to 24.3 K at 50 kOe. A field-induced enhancement of the Curie temperature is typical for ferromagnets.

Shown in Fig. 3 is M versus H_a measured at 5 K. The "S"-shaped curve is characteristic of ferromagnetism. However, within experimental uncertainty of ±2 Oe, there is no hysteresis and zero coercivity and remanence. Close inspection of $M(H)$ at various fixed temperatures below T_c shows that for small H_a, M is linear (nearly vertically) about the origin for positive and negative values of H_a. We define ΔM as the magnetization range over which M is linear in H, that is, ΔM is proportional to the spontaneous magnetization. The temperature variation of ΔM is plotted in the inset of Fig. 3. For reference we note that a value of $\Delta M = 2 \times 10^{-3}$ emu corresponds to a moment of 0.11 μ_B per mole of $C_{60}(TDAE)_{0.86}$. From measurements such as shown in Fig. 3, it is difficult to define unambiguously a saturated moment because of the paramagnetic contribution from the pyrex sample holder, which has not been subtracted from these data. (At the very small fields over which ΔM is defined, the pyrex contribution is negligible.)

An alternative explanation for the data of Fig. 3 is that it arises from superparamagnetism. The field variation of M is similar to that observed in superparamagnetic systems in which H_a is perpendicular to the easy magnetic axis (*29*). However, in our experiments the fine $C_{60}(TDAE)_{0.86}$ powder is constrained in the sample holder only by gravity, and it is unlikely that the powder satisfied this condition since it would tend to orient itself with the easy axis parallel to H_a. Further, for superparamagnetism, M should scale as H/T below T_c (*30*). Experimental $M(H)$ isotherms do not scale with H/T nor with H/T scaled by the saturated moment, which we assume is proportional to ΔM. Consequently, we conclude that $C_{60}(TDAE)_{0.86}$ is a very soft ferromagnet with a remanence of zero or nearly so. The lack of remanence may be attributed to a fully three-dimensional system devoid of domain pinning sites. The exact nature of the ferromagnetism remains an open question. The observation of relatively high ambient temperature conductivity in a compressed pellet ($\sim 10^{-2}$ S cm^{-1}) and preliminary electron spin resonance (ESR) results that show strong narrowing of the

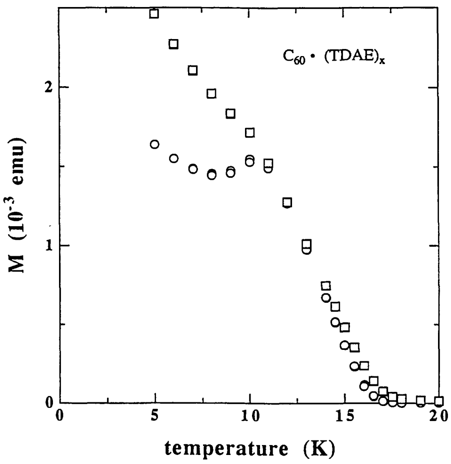

Fig. 1. Magnetization versus temperature for $C_{60}(TDAE)_x$ measured upon cooling and subsequent warming in an applied field of $\sim$1 Oe (○) and 100 Oe (□). The 1-Oe data were multiplied by 10 to give the same ordinate values as the 100-Oe data. Note disappearance of curvature below 10 K at high field. Data were not corrected for the sample holder or for demagnetizing effects.

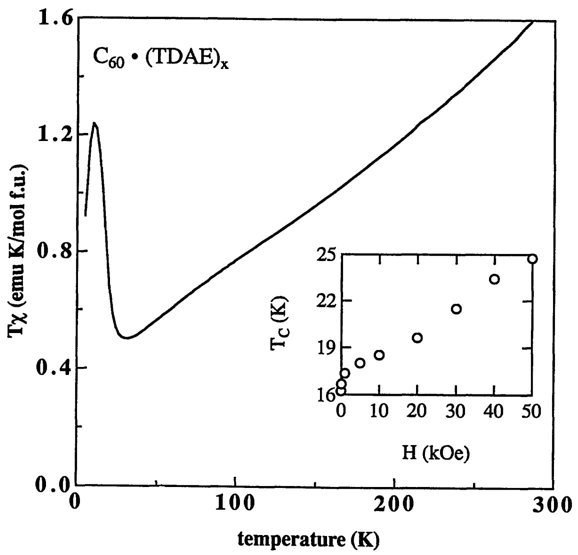

Fig. 2. Product of the molar susceptibility with temperature versus temperature for H_a = 10 kOe. The rapid rise in $T\chi$ below $\sim$30 K signals the development of ferromagnetic correlations prior to the ferromagnetic transition at T_c = 16.1 K. The inset shows the field dependence of T_c.

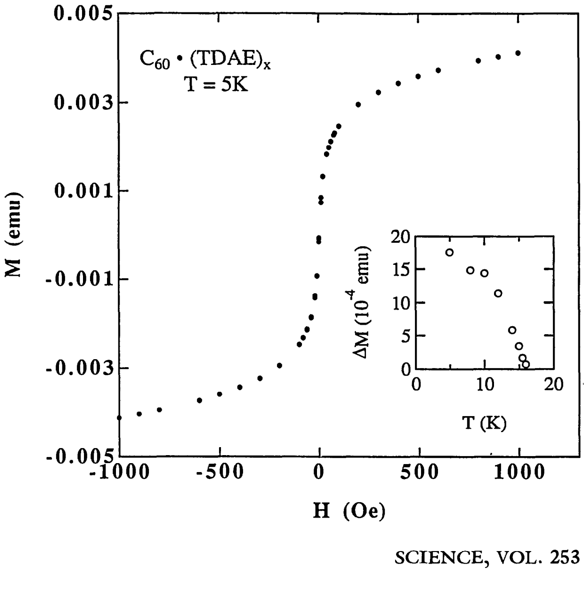

Fig. 3. Magnetization as a function of field at 5 K. Within experimental error, there is no hysteresis in these data. The inset shows ΔM, proportional to the spontaneous magnetization, as a function of temperature. See text for details.

ESR line with decreasing temperature imply that the material might be metallic; if so, it might be an itinerant ferromagnet. Examples of the latter (such as $ZrZn_2$ and Ni_3Ga) show, qualitatively, features that are similar to the ones reported here (*31*).

$C_{60}(TDAE)_{0.86}$ is a "metallic" organic ferromagnet with a Curie temperature higher than any reported for other molecular ferromagnets based strictly on first-row elements. We note that the same basic molecule, namely C_{60}, supports ferromagnetism, metallic conductivity, and superconductivity (in the forms of K_3C_{60} and Rb_3C_{60}), a rather interesting and unusual occurrence.

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Early Differentiation of the Earth and the Problem of Mantle Siderophile Elements: A New Approach

V. Rama Murthy

The long-standing problem of the excess abundances of siderophile elements in the mantle can be resolved by considering an equilibrium core-mantle differentiation in the earth at 3000 to 3500 kelvin. This high-temperature differentiation results in mantle siderophile element abundances that closely match the observed values. Some lithophile (light) elements could enter the core in this process as is necessary to account for its low density. The abundances of siderophile elements in the mantle are consistent with the conclusion derived from the recent physical models that the earth was molten during accretion.

Recent progress in the development of quantitative models of the accretion of planets has indicated that the earth was largely or totally molten during its accretion (*1*). For this initial state, the effect of pressure on melting in a planet of terrestrial size requires that a substantial amount of the planet would be at temperatures much higher than the low-pressure melting points of silicates and metallic iron. Differentiation to form an iron core and a silicate mantle would essentially be a high-temperature process, the temperature corresponding to the liquidus temperatures of iron and silicates at the prevailing pressures as the planet accretes. On the basis of a detailed investigation of the physical process of core separation in a largely molten earth, Stevenson (*2*) concluded that core segregation occurred rapidly under complete chemical equilibrium between the iron metal and mantle silicates. In such a case, the abundances of siderophile elements in the mantle would be controlled by distribution coefficients (K_d's) applicable to the temperatures and pressures at which the core-mantle equilibrium was established and not by the K_d's at the relatively low temperatures measured in the laboratory. This point has not been considered so far in the use of abundances of siderophile elements in the mantle as constraints to theories of the early chemical differentiation of the earth [for example (*3, 4*)].

In this report, I examine a model of core formation in which the siderophile element partitioning between the core and the mantle occurred at temperatures close to the liquidus temperatures in the accreting earth. Other than the consideration of the effect of temperature, all assumptions remain the same as in earlier discussions, namely, that the partitioning of trace components between two phases occurs at equilibrium and that the phases relevant to core-mantle differentiation in the earth are dominantly Fe-metal and liquid silicates.

The temperatures at which core-mantle equilibrium in the earth was established cannot be specified exactly. Most modern theories of accretion suggest that the earth would be molten by the time it had grown to about one-tenth of its present mass (*1, 5, 6*). Core formation will commence at this stage and continue through the stochastic accretion process by the infall of 10^{25}- to 10^{26}-gram planetary embryos (*1*). Thus we can expect that successive fractions of the core separate from the mantle silicates at their liquidus temperatures in this growing proto-earth. Because the interior pressure in the planet in the initial stages of accretion would be less than that in the present earth, the silicates and metal would have melted at temperatures lower than those at depth in the present earth. On the basis of data on melting of mantle silicates as a function of pressure, it can be inferred that core formation commenced at about 2500 K when the earth is about one-tenth of its present mass. As accretion continued, the sinking Fe-metal droplets would have equilibrated with mantle silicates at progressively higher temperatures because of the increase in the melting points of mantle silicates in response to the increased pressure in the interior. In the final stage of accretion when the earth

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Department of Terrestrial Magnetism, Carnegie Institution of Washington, and Department of Geology and Geophysics, University of Minnesota, Minneapolis, MN 55455.