SOLID-STATE PHYSICS AT MICROKELVIN TEMPERATURES: IS ANYTHING LEFT TO LEARN?

Research at these temperatures on superconductors, spin glasses, structural glasses and nuclear magnets is really only in its infancy.

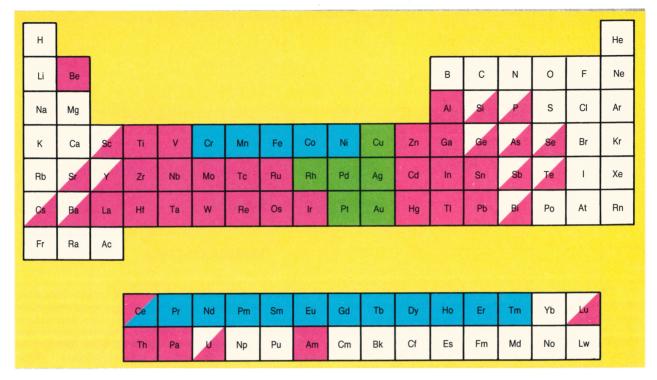
Frank Pobell

can vary in the laboratory to change the properties of matter so as to get a better understanding of its behavior. Heike Kamerlingh Onnes's liquefaction of helium-4 in 1908 made the low-kelvin-temperature range accessible. Subsequent discoveries in low-temperature physics included verification of essential predictions of quantum mechanics and statistical physics—for example, the temperature dependence of the specific heat of insulators and of metals. The introduction of the ³He-⁴He dilution refrigerator in the late 1960s extended condensed matter physics into the millikelvin temperature range. Once again the opening of a new temperature range allowed many fundamental discoveries, particularly in condensed matter physics—heavy-fermion superconductivity and the quantum Hall effect, for example.

Temperature is the most important parameter that one

Recent advances in refrigeration and thermometry have made even lower temperatures available to condensed matter physics. Microkelvin temperatures can be reached by adiabatic nuclear demagnetization.¹⁻³ This technique, first applied in 1956 by Nicholas Kurti and his coworkers at Oxford University, was substantially advanced in the 1970s by Olli Lounasmaa and his coworkers in Helsinki. They were the first to combine a ³He-⁴He dilution refrigerator with a superconducting magnet to obtain4 the required starting conditions for nuclear refrigeration of about 15 mK and 8 tesla. (See Lounasmaa's article in Physics today, December 1979, page 32.) About two dozen groups now have achieved temperatures below 1 mK, and seven groups have refrigerated condensed matter to 100 microkelvin or lower by adiabatic nuclear refrigeration. Liquid helium has been refrigerat $ed^{1,2}$ to 100 μ K; experiments with solid matter have been performed to 10 µK; gases have been optically pumped to 2 μK (see the article by Claude N. Cohen-Tannoudji and

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Periodic table of the elements. Blue indicates elements that undergo magnetic ordering; filled red boxes, superconducting elements; half-red boxes, elements that become superconducting at high pressure. For discussion of the elements marked in green, see the text. **Figure 1**

William D. Phillips in Physics Today, October 1990, page 33); and nuclear spin temperatures of slightly below 1 nanokelvin have been reached⁵ (see Lounasmaa's article in Physics Today, October 1989, page 26).

Experimenters reached the kelvin and the millikelvin temperature ranges only by overcoming substantial experimental difficulties. The problems presented by the microkelvin temperature range are at least as severe. It is therefore valid to ask whether we can really learn anything new about condensed matter at microkelvin temperatures, and if so, is that knowledge sufficiently valuable to justify investing the required manpower and financial resources?

The optimistic answer to these questions is that whenever we have entered a new temperature range, we have discovered new and important phenomena, widening our understanding of nature; there is no reason to suspect that this should be different for the microkelvin temperature range.

The pessimistic answer is that we have already learned so much at kelvin and millikelvin temperatures that little or nothing is left to make the "microkelvin effort" worthwhile. This is partly true, because condensed matter has been so thoroughly investigated in the kelvin and millikelvin temperature ranges that one can state with reasonable certainty that for some branches of condensed matter physics nothing new will happen at lower temperatures. However, there are branches for which one cannot make such a statement. And there are even branches for which one can state with reasonable certainty that new discoveries will be made.

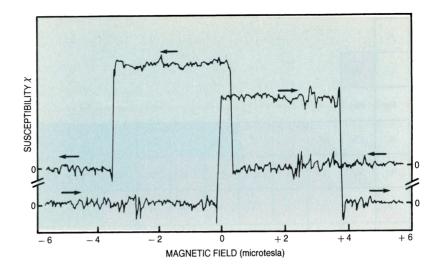
Solid and liquid helium-3

Liquid and solid helium-3 have already been studied extensively at temperatures below 1 millikelvin, giving

many spectacular results. Helium-3 has a nuclear spin of $^{1}\!\!/_{2}$ and a nuclear magnetic moment. In the solid bodycentered cubic state, the 3 He nuclei show a transition from a nuclear paramagnetic to a nuclear antiferromagnetic state. This transition occurs not at about 1 μ K, as originally predicted from the nuclear dipole interactions, but at a temperature a factor of a thousand higher. The antiferromagnetic transition at 0.93 mK was detected by William P. Halperin and his colleagues at Cornell University in 1974. (See the article by Michael C. Cross and Douglas D. Osheroff in Physics today, February 1987, page 35.)

This dramatic enhancement of the nuclear magnetic transition temperature is a result of the exchange interaction between ³He atoms in the solid state. Helium-3 is the only solid where the word "exchange" has to be taken literally: Due to the large zero-point motion of the light, weakly bound atoms, they exchange sites in the crystal even at absolute zero. The transition is to a state with a spin sequence of two up, two down, two up, two down and so on. This unusual magnetic structure occurs because the dominant exchange interaction involves the cyclic exchange of three or four atoms, resulting in a very strong pressure dependence of the parameters involved. Going from a molar volume of 24.2 cm³/mole (at melting) to a molar volume of 21 cm³/mole changes the transition temperature from 0.93 mK to about 0.05 mK. If a magnetic field is applied, a new magnetic phase occurs. If the molar volume is decreased further, solid ³He switches from the bcc structure to the hexagonal close-packed structure, and the ordering is expected to switch from an antiferromagnetic to a ferromagnetic state with a Curie temperature $T_{\rm n,c}$ less than 50 $\mu \rm K$. The real situation has turned out to be much more complicated, rich and interesting than originally anticipated.

Susceptibility of rhodium at 160 μK as a function of magnetic field. The data from two runs with the indicated directions of field change are shifted relative to each other on the ordinate. Due to supercooling, the sample does not become superconducting until the field drops to 0.2 μT even though its critical field at this temperature is 3.7 μT. (Adapted from C. Buchal, F. Pobell, R. M. Mueller, M. Kubota, J. R. Owers-Bradley, *Phys. Rev. Lett.* **50**, 64, 1983.) **Figure 2**

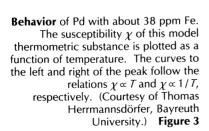


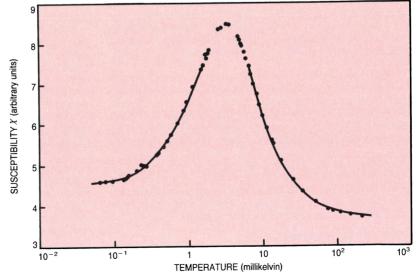
Even more exciting is the behavior of ³He in the liquid state when cooled to the low-millikelvin temperature range.^{2,7} Osheroff, Robert C. Richardson and David M. Lee at Cornell University found the superfluid transition of ³He in 1972. The Fermi liquid ³He shows three superfluid phases—in contrast to the Bose liquid ⁴He, which has only one superfluid phase, at temperatures below 2.2 K. ³He can become superfluid because the fermions "pair" in a similar manner to the electrons in superconducting metals. The ³He pairing interaction is so weak that pairing occurs only at temperatures below 2.5 mK. In conventional superconductors the electrons pair with zero total spin and zero total angular momentum. In ³He the nuclei pair with parallel nuclear spins and finite total angular momentum, giving the pairs a magnetic moment. This spectacular liquid shows properties of a superfluid (vanishing viscosity of the superfluid component and macroscopic quantum effects), of a superconductor containing Cooper pairs (pair-breaking effects), of a magnet (magnetization and a rich spectrum of nmr phenomena) and of a liquid crystal (directionality and textures).

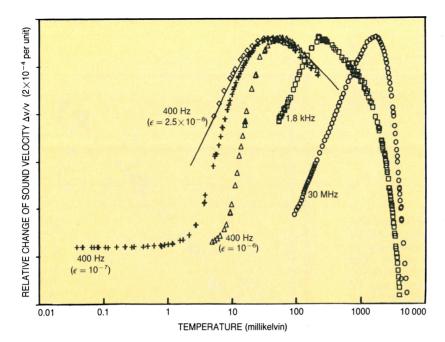
I will not discuss the fascination of superfluid ³He in detail, because various books and review articles have

already been written on it.2.7 However, I want to mention some of the results: The Moscow group of Andrei S. Borovik-Romanov and Yuri M. Bunkov has observed spin supercurrents, which are coherent magnetization transfers; they are analogous to superfluid mass transfers in liquid 4He and 3He and in superconductors. These experiments as well as the flow experiments of Olivier Avenel and Eric Varoquaux at Saclay exhibited the Josephson effect and a phase shift of the macroscopic wavefunction describing the coherent superfluid ground state. George R. Pickett and Tony M. Guénault and their coworkers at the University of Lancaster have investigated the ballistic flow of ³He quasiparticles over distances of 3 mm, a phenomenon that permitted the construction of a ³He quasiparticle spectrometer. Experiments by Matti Krusius and his coworkers at the Helsinki University of Technology have shown that superfluid ³He under rotation can form at least four different kinds of vortices with the superfluid circulating around either singly or doubly quantized cores.

Research on superfluid ³He is a frontier area, and shows the possibility of observing the unexpected at ultralow temperatures. The results of the investigations of ³He have been so spectacular that all groups that can at-







Noncrystalline material acoustics.

The relative change in the velocity of sound at three different frequencies in vitreous SiO_2 is plotted as a function of temperature. The solid line is from a theoretical description of glasses known as the tunneling model. The three runs at 400 Hz were taken with the indicated acoustic strains ϵ . (Adapted from ref. 11.) **Figure 4**

tain temperatures of 1 mK or lower do research on liquid or solid ³He. It is my belief that solid-state physics at microkelvin temperatures could be as rewarding as liquid helium research, and I wonder how much exciting new physics might emerge if a similar emphasis were directed to the properties of solids. The very few problems that have been tackled outside liquid helium research—problems I will discuss below—support this belief.

Superconductivity

Ever since the discovery of superconductivity, physicists have asked whether the superconducting state is the universal low-temperature state of nonmagnetic metals. If we look at the periodic table of the elements (figure 1), we see only two areas with metals that are neither magnetic nor superconducting: some alkali (Li, Na, K, Rb) and alkaline earth (Mg, Ca) metals, and then the noble metals (Cu, Ag, Au) and some platinum metals (such as Pd and Pt). In 1913, shortly after his discovery of superconductivity, Kamerlingh Onnes wrote, "Without any doubt, if one could get Au and Pt pure enough, they would enter the resistanceless, superconducting state at helium temperatures." This statement is not correct, at least with respect to the temperature range, because we know⁸ that the superconducting transition temperatures T_c of gold and platinum are not greater than $\hat{10}^{-4}$ K.

Roger F. Hoyt and Ana C. Mota showed in 1976 that the $T_{\rm c}$'s of ${\rm Au_{1-x}\,In_x}$ alloys decrease from about 63 mK for 90% Au to 11 mK for 94% Au. My coworker Christoph Buchal confirmed Hoyt and Mota's data and extended them to 98% Au with $T_{\rm c}=0.50$ mK. Buchal extrapolated $T_{\rm c}$ down to approximately 100 μ K for pure Au. However, he did not observe a superconducting state in gold samples down to 38 μ K. The reason is probably that an impurity concentration of only 10^{-2} ppm Cr or Mn in Au may completely suppress superconductivity, because AuCr and AuMn are Kondo systems with rather low Kondo temperatures. Ag and Cu alloys have transition temperatures lower than those of the corresponding Au alloys, so the possible superconducting states of Ag and Cu would be even more difficult to observe.

A noble metal is not superconducting at temperatures above $0.1~\mathrm{mK}$ because of its low electronic density of states

at the Fermi energy and weak electron-phonon interaction. In a platinum metal, by contrast, both of these properties are quite substantial. However, no superconducting transition was found in Pt, Pd or Rh to 2 mK until 1978. By careful field shielding to less than 0.1 microtesla and refrigeration to 60 μ K, we were able eventually to observe8 superconductivity in Rh, with a critical field $B_{\rm c}=4.9\,\mu{
m T}$ and $T_{\rm c}=325\,\mu{
m K}$. (See figure 2.) The observed severe supercooling indicates the importance of field shielding for such experiments. With this discovery, Rh has become the lowest- $T_{\rm c}$ superconductor. Its transition temperature is about three orders of magnitude lower than those of its neighboring elements Ru and Ir. This suppression of T_c probably is a result of spin fluctuations in this exchange-enhanced paramagnet. As we discuss below, no superconducting transition to about 0.1 mK was found in Pd and Pt, where spin fluctuations are even

Superconductivity was found in Rh and probably exists in pure Au. However, the question of whether the ground state of all nonmagnetic metals is superconducting—whose solution would also answer the question of whether in all metals the effective electron—electron interaction is attractive—still awaits a final answer.

One of the fundamental discoveries of low-temperature physics during the last several decades has been "heavy-fermion superconductivity"—superconductivity of conduction electrons that have a strong tendency toward magnetism and effective masses of up to several hundred electron masses. Many properties of the exotic materials that exhibit this phenomenon indicate an interplay of magnetism and superconductivity but are not yet understood. Some of the experiments needed to settle the outstanding problems demand ultralow temperatures—for example, to enable identification of the ground states of these metals. This new branch of ultralow-temperature physics has just been started at the University of Florida and at Cornell University.

Spin glasses

As an impurity in palladium, iron polarizes the conduction electrons of this strongly paramagnetic host. The polarization decreases and has oscillating positive and negative

Polycrystalline material acoustics.

The relative change of the velocity of sound in cold-rolled polycrystalline Ag at 250 Hz is plotted as a function of temperature. Note the similarity to the behavior of amorphous dielectric SiO₂ in figure 4. (Adapted from ref. 11.) **Figure 5**

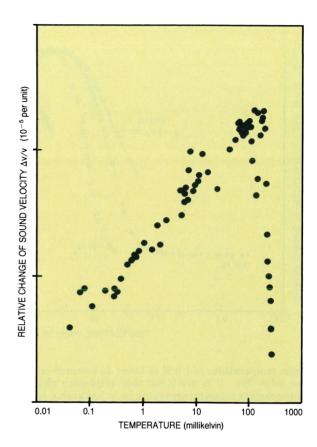
amplitudes as a function of distance from the Fe impurity. At low iron concentrations the statistically distributed Fe neighbors of an Fe atom can sit in a positively or negatively polarized conduction electron region. The irons are "frustrated," and a spin glass state should occur.

My group at the Jülich Research Center had Pd samples available with Fe concentrations ranging from 2 to 100 atomic parts per million. Of course, we refrigerated these samples to microkelvin temperatures.⁸ Figure 3 shows the magnetic susceptibility as a function of temperature for one of them. As temperature decreases, the susceptibility increases according to a Curie law, $\gamma \propto 1/T_c$ shows a peak at the spin glass freezing temperature and eventually decreases as $\gamma \propto T$. In the Pd sample with the smallest Fe concentration, the magnetic centers interact over the tremendous distance of 20 nm to form the spin glass state. This large distance between the magnetic moments undergoing a collective transition makes PdFe a model magnetic system for the study of single-impurity interactions and the spin glass state. The simple $\gamma - T$ relations over several decades in temperature make this spin glass also a very useful thermometric substance, which we use routinely in our experiments.

Acoustic properties

Noncrystalline materials have low-temperature properties that are distinctly different from those of their crystalline counterparts. Their specific heats have an almost linear temperature dependence and their thermal conductivities show a T^2 behavior. Their sound velocities v, at temperatures above the temperature for which v is a maximum, exhibit a logarithmic decrease with a slope that is very accurately $-\frac{1}{2}$ the slope of the logarithmic increase of v below its maximum (see figure 4). These as well as other low-temperature properties of glasses at temperatures between 10 mK and 1 K have been successfully explained by the phenomenological "tunneling model."

To shed more light on what, really, is a glass and how well glasses obey the tunneling model, my colleagues Pablo Esquinazi and Reinhard König and I have studied in Bayreuth the low-frequency acoustic properties of noncrystalline materials, as well as of polycrystals, at temperatures now accessible by nuclear refrigeration.¹¹ For vitreous silica (SiO₂), we observed clear deviations from the logarithmic decrease of v at temperatures below 10 mK; saturation of the sound velocity at temperatures below 1 mK; and strain dependence of the onset of saturation, of the slope of the sound velocity and of the position of its maximum (figure 4). Furthermore, the sound attenuation between 0.1 and 30 mK follows an unexpected linear temperature dependence and is essentially independent of the acoustic strain. These results, as well as the acoustic properties of the metallic glass PdSiCu at very



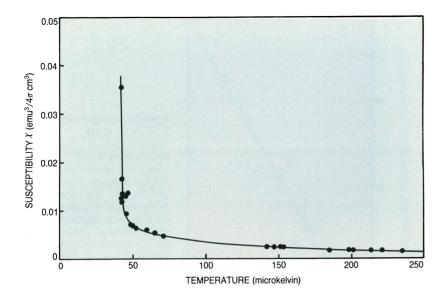
low temperatures,¹¹ cannot be understood within the "standard" tunneling model. It remains to be shown whether a modification of the model is sufficient to explain the results or whether a new theoretical approach is required.

We investigated also the acoustic properties of Ag, Cu and Pt as well as of superconducting Al, Ta, Nb and NbTi to tackle the question of whether polycrystals show glasslike behavior at very low temperatures. 11 Polycrystalline Ag and Cu, for example, show a maximum of the velocity of sound at approximately 0.1 K and then a decrease as $\Delta v/v \propto \ln T$ over three decades in temperature (see figure 5), as predicted by the tunneling model for glasses; they show the best "glassy behavior" ever observed! The acoustic properties of Ag, Cu, Pt, Al, Ta, Nb and NbTi indicate that these polycrystals contain low-energy excitations with properties very similar to those of the excitations in amorphous materials. Obviously, "glass-like anomalies" are far more universal than expected, and we have to understand better what "disorder" means and whether the difference between a glass and a polycrystal is of a qualitative or only a quantitative nature.

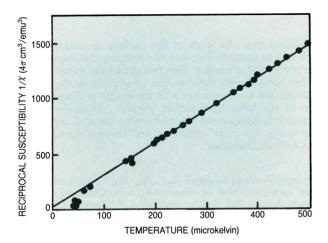
Nuclear magnetism as a probe

Because of the small size of nuclear magnetic moments, critical temperatures for nuclear magnetic ordering are expected to be only on the order of microkelvins or even lower. Ordering due to nuclear dipole interactions was first observed in the *insulators* CaF₂, LiF and LiH, by Anatole Abragam, Maurice Goldman and their coworkers at Saclay¹² in the 1970s. Solid ³He is another nuclear magnetically ordered insulator.

Nuclear magnetic moments in *metals* experience not only direct dipole interactions but also indirect exchange



Spontaneous nuclear transition at about 40 μ K is evident in plot of nuclear susceptibility χ of In in AuIn₂ at 2 millitesla as a function of temperature (top). Plot of $1/\chi$ as a function of temperature (bottom) shows no positive temperature intercept for the data at temperatures above 60 μ K. (Courtesy of Herrmannsdörfer.) **Figure 6**



where nuclear magnetic interactions become important. The best candidates for nuclear magnetic ordering among this group may be Tl, which has the smallest known Korringa constant of any metallic element ($\kappa=4$ mK sec), and the intermetallic compound AuIn₂.

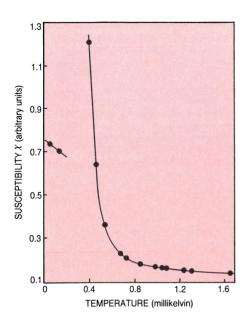
Nuclear magnetic ordering was first observed in a metallic element by a group in Helsinki,5 which found a transition in Cu at a nuclear spin temperature $T_{\rm n.c.}$ of 58 nK. Monitoring the susceptibility as a function of time while the nuclear spin system was warming (through its coupling to the thousand-times-hotter conduction electrons), they found an initial increase in the susceptibility, followed by a plateau and eventually a decrease. This behavior is typical for a transition from an antiferromagnetic state to the paramagnetic state. Subsequent susceptibility and neutron scattering experiments on a single crystal surprisingly showed three phases with different spin structures. Because at that time only one ordered phase was expected, as was a $T_{\rm n,c}$ for copper of 230 nK, these observations inspired substantial theoretical work. It is now understood that the reduction of $T_{
m n,c}$ and the ordered phases are the result of nuclear spin fluctuations and of competition between the "ferromagnetic" dipolar and "antiferromagnetic" exchange interactions of Cu

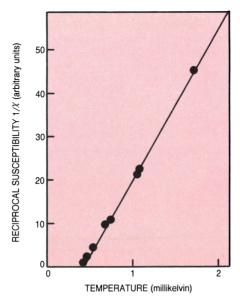
The nuclear magnetic moments of Ag are about 20 times smaller than those of Cu, resulting in a much lower transition temperature. The susceptibility of Ag again showed saturation during warm-up before it decreased.5 This observation, as well as changes in the nmr line shapes with temperature, is associated with spontaneous, antiferromagnetic ordering of silver nuclei at a nuclear spin temperature $T_{\rm n,c}$ of 0.56 nK. Recently the Helsinki group has studied the nuclear magnetic behavior of Ag at negative spin temperatures, where the higher energy levels are more populated than the lower ones.⁵ At T < 0an isolated spin system tries to maximize its free energy instead of minimizing it. The dominant antiferromagnetic exchange interaction in Ag, which leads to the antiferromagnetic state at T>0, gives, then, a ferromagnetic ordering of spins corresponding to the energy maximum at T < 0.

The two simple metals with strong nuclear—electron couplings that have been studied are Tl and In in the cubic compound AuIn₂. (The nuclear moment of Au is 40 times smaller than that of In.) Because of the strong nucleus—

interactions mediated by the conduction electrons, which are polarized by the nuclei. A great advantage of nuclear magnetism in metals in comparison to electronic magnetism is that nuclear magnetic moments are well localized, allowing a thorough comparison of theoretical and experimental results. When investigations of nuclear magnetism in metals began in the early 1970s, there was a debate over whether or not we might learn something beyond what we knew already from detailed investigations of electronic magnetism. The very few investigations of nuclear magnetism in metals performed up to now have given some rather unexpected results that show that we can indeed learn new aspects of magnetism.

Metals suitable for the study of nuclear magnetism can be divided into two groups. There are metals in which the interaction between nuclei and conduction electrons is so weak that a fast adiabatic demagnetization of polarized nuclei can pull them to ultralow temperatures while leaving the conduction electrons at the starting temperature; this technique has been applied in Helsinki⁵ to Ag and Cu. (See Lounasmaa's article in Physics Today, October 1989, page 26.) For the other group of metals, the interaction between the nuclei and the conduction electrons is so strong that one has to refrigerate the whole material in thermal equilibrium to ultralow temperatures,





Nuclear susceptibility γ of Pr in PrNi₅ at zero magnetic field as a function of temperature (left) indicates a spontaneous nuclear magnetic transition at 0.40 mK. The intercept of the plot of $1/\chi$ as a function of temperature (right) indicates a Weiss temperature θ of 0.40 mK. (Adapted from M. Kubota, C. Buchal, R. M. Mueller, F. Pobell, J. Magnetism Magnetic Mater. 31, 739, 1983.) Figure 7

electron interactions, we expect nuclear magnetic ordering in these two metals at the relatively high equilibrium temperatures (when the nuclear spin temperature $T_{\rm n}$ equals the electron temperature $T_{\rm e}$) of 1–10 $\mu{\rm K}$. Neither metal has yet been refrigerated to these temperatures. However, in both metals unexpected deviations from simple paramagnetic behavior have already been seen at substantially higher temperatures. In Tl nuclear magnetic resonance experiments by Georg Eska and Erwin Schuberth showed an increase of the susceptibility stronger than expected for a paramagnet, as well as a splitting of the nmr line. Birgit Schröder-Smeibidl's recent measurements of the nuclear specific heat of another Tl sample showed, in contrast, simple paramagnetic behavior.

Nuclear magnetic resonance experiments in Bayreuth by Kurt Gloos, Peter Smeibidl and Birgit Schröder-Smeibidl at temperatures down to 70 $\mu \rm K$ showed a tendency toward ferromagnetism of the In nuclei in AuIn_2. Below 1 mK they found an unexpectedly strong temperature dependence of the spin–spin relaxation time τ_2 , which is an indicator of nucleus–nucleus interactions. Thomas Herrmannsdörfer has measured the heat capacity and susceptibility of In nuclei in AuIn_2. At 2 mT, the lowest field in his experiments, he finds a dramatic increase in both properties, indicating that there is already a spontaneous ordering transition at about 40 $\mu \rm K$ (see figure 6).

Another group of metals suitable for the study of nuclear magnetic ordering at equilibrium are the so-called hyperfine enhanced van Vleck paramagnets. Walter Weyhmann at the University of Minnesota, Takeo Kodama at Osaka City University and their coworkers have studied the nuclear ferromagnetic state of PrCu_6 ($T_{\rm c}=2.6$ mK) and in Jülich we have done the same for PrNi_5 ($T_{\rm c}=0.4$ mK, as figure 7 indicates). Lois Pollack and her colleagues have recently applied the ultralow temperatures available for solid-state physics at Cornell University to study nuclear electric quadrupole resonance in Sc metal to 0.1 mK.

The future

The new efforts at Cornell and at the University of Florida have widened the "microkelvin solid-state physics club," and hopefully other groups will soon join as well. The field has momentum and an international flavor. I am convinced that the work of the various members of the club will be rewarded by new and exciting information on the properties of matter. The few experiments performed so far have shown the richness of solid-state physics at microkelvin temperatures and the magic of discovery at one of the true frontiers of physics. Experiments at ultralow temperatures are addressing general questions, and this is the most important and fascinating aspect of this difficult endeavor.

The experiments in Jülich on $Au_{1-x}In_x$, Rh, Pt, PdFe and $PrNi_5$ have been performed in cooperation with Christoph Buchal, Minoru Kubota, Ralf P. Peters and Robert M. Mueller. The results on Tl, $AuIn_2$ and PdFe as well as on the "glassy" acoustic properties were obtained in Bayreuth in collaboration with Georg Eska, Pablo Esquinazi, Kurt Gloos, Thomas Herrmannsdörfer, Reinhard König, Birgit Schröder-Smeibidl and Peter Smeibidl.

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