Shubnikov de Haas Study in the High Magnetic Field State of η-Mo_4O_{11}

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The η-Mo_4O_{11} compound undergoes two distinct charge density wave (CDW) transitions,[1] at T_{CDWI} = 109 K and T_{CDWII} = 30 K respectively, as the temperature is decreased. The CDWII phase is quite sensitive to the application of a magnetic field along the least conducting **a*** axis. The magnetoresistance shows a series of peaks between quantum Hall plateaus[2] until a certain field B_{QL} ~ 20 T is reached, above which a high resistivity phase is observed. Assuming that the ground state is an incommensurate CDW phase the effect of a magnetic field can be explained through a mechanism analogous[2,3] to the so-called standard model for the magnetic-field-induced spin-density wave phases.

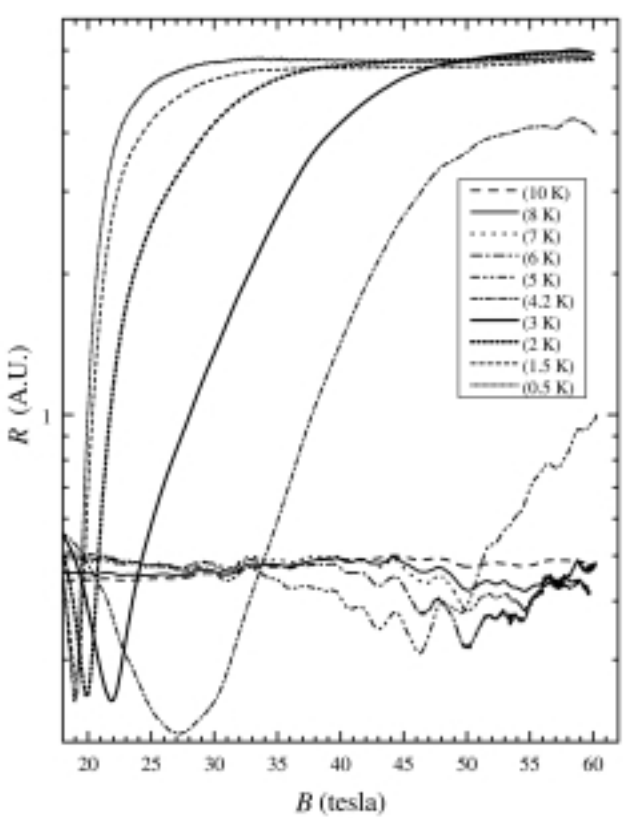

Figure 1. Magnetoresistance of a η-Mo_4O_{11} single crystal as a function of field B above 20 T at several temperatures.

In this picture, the high field phase appears when the Fermi level is no longer placed in the gap between adjacent Landau levels but in the gap between the conduction and valence bands. Nevertheless, this picture is difficult to reconcile with the observation of Shubnikov de Haas (SdH) type oscillations with frequencies of 170 and 235 T [2] respectively, in the high field region. These oscillations are not detected in magnetization experiments.

In Figure 1 we show the magnetoresistance taken in pulsed magnetic fields up to 60 T at different temperatures ranging from 0.5 K up to 10 K. At

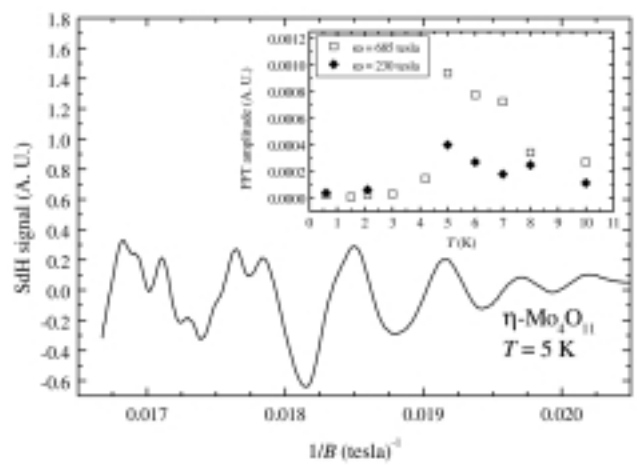

Figure 2. The oscillatory behavior of the magnetoresistance (SdH signal) as a function of inverse field for a η-Mo_4O_{11} single crystal at T = 5 K. Inset: Temperature dependence of the FFT amplitude of the SdH signal observed in the high field phase.

low temperatures, the high field phase is identified by a sharp increase in the resistance above 20 T. Characteristic oscillations periodic in 1/*B* are detected as seen in Figure 2.

For this particular sample we did not detect a frequency of 175 T but only oscillations at the frequency of 230 T and an anomalous third harmonic at 685 T (not seen in other samples). The most interesting observation is the anomalous temperature (*T*) dependence of the amplitude of the Fourier transform of the SdH signal. The amplitude increases as the temperature is lowered reaching a maximum at T_{max} ~ 4.5 K. Below this value, the amplitude decreases almost exponentially upon cooling. This

behavior, as a function of T, is quite similar to that of the "rapid" quantum oscillations (RO) observed in the $(TMTSF)_2X$ compounds with a spin-density-wave ground state.[4] In this case, the temperature dependence above T_{max} is well fitted to the Lifshitz-Kosevich (LK) formalism while an activated prefactor to the LK formula is necessary to fit the data below T_{max}.[4] Similarly to the oscillations reported here, the RO's are not detected in the

Ergodic Behavior of Granular Matter in Low Gravity IHRP

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We report studies on the dynamics of macroscopic particles in a low gravity "magnetic levitation" environment. In a real sense, this allows the investigation of new states of granular matter. Particle ensembles (rods, spheres, or grains) are held in a weak confining potential due to diamagnetic forces in a high field resistive magnet. In such a case "k_BT" is not zero, and assemblies of particles undergo ergodic processes to find the lowest configurational ground state. The examples given in the four figures exploit the capabilities of the 20 T, 195 mm bore resistive magnet at the NHMFL. This magnet has an advantage since the very wide bore allows excellent visual or instrumentation access to materials in the levitation region.

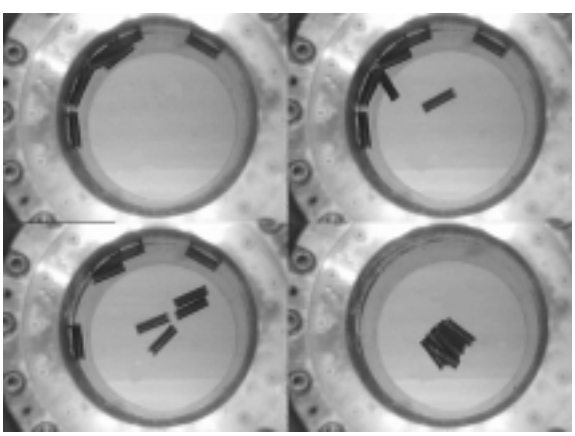

Figure 1. Macroscopic crystallization. We show a sequence of frames where a set of nearly identical rods (~3 cm long and 1 cm in diameter) is placed at the bore tube wall at high field. (Here the high field/gradient product pushes the particle high enough to enter the region where the radial field gradient reverses sign.) The field is then lowered to a pre-determined value where the rods levitate freely, and self-assemble into an ordered structure.

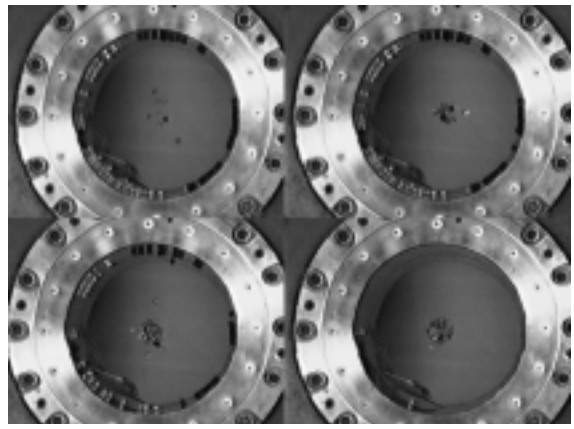

Figure 2. Granular structures. We show a sequence of frames where a number of multicolored beads are brought in from the bore tube wall (by decreasing the field from its maximum value) to form a "nucleus." (Note the paramagnetic dollar bill at the center).

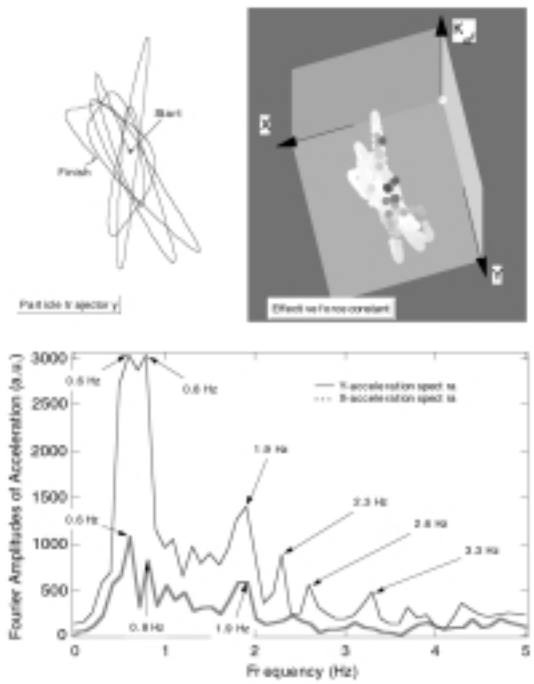

Figure 3. Single particle dynamics. In this experiment the initial state of the particle is prepared in a field well above the value needed for levitation, such that the particle is forced to the wall. The field is then lowered, either continuously, or in a single step, and the particle leaves the bore tube wall and begins a quasi-cyclic trajectory with a major period of order 1 second. To quantify this motion, we have digitized the trajectories [x(t), y(t)] for numerical analysis. We then compute the velocity $[v_x(t), v_y(t)]$, the acceleration $[a_x(t), a_y(t)]$, and the instantaneous spring constant $[k_x = -ma_x/x, k_y = -ma_y/y]$. Finally, we compute the instantaneous vector sum $k_{eff}=(k_x^2+ k_y^2)1/2$.

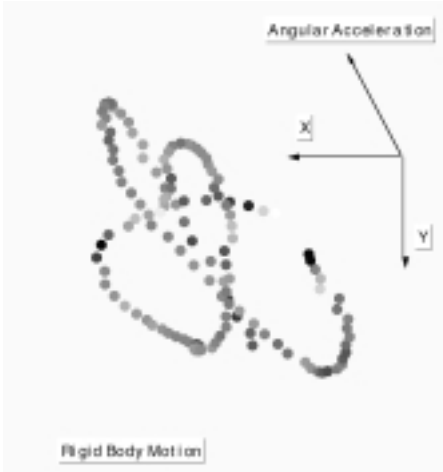

Figure 4. Rigid body motion. Finally, we turn to an analysis of a rigid body. Here we have digitized the motion of two opposite points on an irregular graphite block ("aquadag"). The center of mass motion and the angular acceleration is shown.

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NMR Investigations of Three Low-Dimensional Systems

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We have investigated several aspects of low-dimensional materials using NMR at very high magnetic fields. One project is to use the nuclear spin-lattice relaxation rate ($1/T_1$) of ^{85,87}Rb to probe the charge density wave (CDW) fluctuation spectrum and to test the hypothesis of a coherence peak from coherent pairs[1] just below the CDW transition in $Rb_{0.3}MoO_3$ ("blue bronze"). Although our initial efforts on this topic have shown that there is a significant frequency dependence to the phason fluctuation spectrum in this material, they are not yet complete enough to provide a strong test of the coherence peak hypothesis.

Another project in which we have applied high field NMR is to study the dynamics of electrical transport and ionic diffusion on a microscopic scale in the one-dimensional material KCu_7S_4,

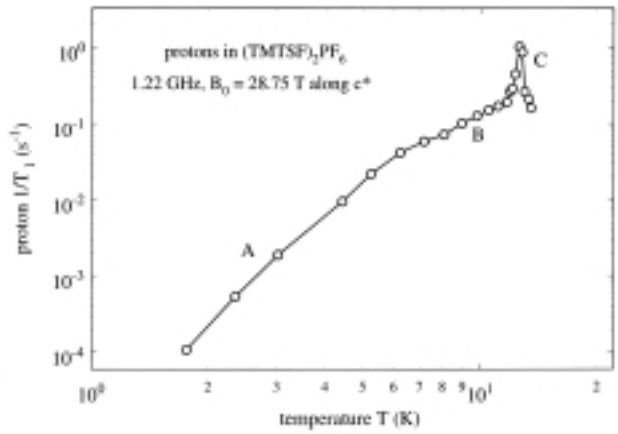

Figure 1. Proton spin-lattice relaxation rate in $(TMTSF)_2PF_6$.

which has several unusual properties.[2] On the basis of our ^{63,65}Cu ($1/T_1$) measurements over a wide range of frequencies, we find that on a microscopic scale below 80 K, this material mimics a low electrical conductivity metal, which is in apparent conflict with its macroscopic conductivity, which is relatively low and decreases with decreasing temperature. At higher temperatures, ionic diffusion becomes very rapid; our NMR measurements show that the activation energy for this diffusion is approximately 500 K.

The third project is an extension of our previous work on spin density wave (SDW) order and fluctuations in the organic conductor $(TMTSF)_2PF_6$.[3] Recent measurements of ($1/T_1$) made at 1.22 GHz are shown in Figure 1. In these measurements, $1/T_1$ is proportional to the power spectrum of the magnetic fluctuations of the material, the origin of which we attribute to the power spectrum of the SDW fluctuations. Region C, which is dominated by critical fluctuations in the amplitude of the SDW, has the same rate as that observed for frequencies down to 100 times smaller.[3] This means that there is no critical slowing to time scales longer than 10^{-10} s in this transition. The region B, which is dominated by phase fluctuations of the SDW, has the interesting property that its power spectrum is starting to rise in comparison with lower frequency measurements,[3] which could mean that we are entering into the regime where the fluctuations have a short enough distance scale that their damping by screening currents is starting to disappear. Finally, in region C, there is a rapid drop in the power spectrum of the fluctuations, whose interpretation is too controversial to discuss in detail here.

Acknowledgements. This work was supported in part by NSF Grants DMR-9705364 (WGC) and DMR-9971530 (SB).

1 Matus, P., *et al.*, J. Phys. IV Proceedings, **9**, 267 (1999).
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Search for De Haas - Van Alphen Oscillations in Quasicrystals

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Quasicrystals were first discovered in rapidly solidified alloys of Al and Mn in 1984.[1] These materials are atomically well ordered but they do not possess the familiar translational symmetry of conventional crystalline systems. Indeed, to fully describe their diffraction patterns one needs more unit vectors than the dimensionality of the space in which they exist[2]—differing from incommensurate crystals in that they cannot be described as modulations of a crystalline system. While the first quasicrystalline alloys were metastable, there are now known to exist several families of thermodynamically stable quasicrystals, making possible the preparation of large single-grain quasicrystal samples by conventional crystal growth techniques.[3] These samples can be exceptionally well-ordered, in some cases having correlation lengths in excess of 1000 Å. With such well-ordered samples, it has become a realistic possibility to attempt Fermi surface investigations via quantum oscillation measurements, to which end this work is a preliminary study.

While the lack of translational symmetry in quasicrystals prevents Bloch electron states in a conventional sense, nevertheless a hierarchy of quasi-Brillouin Zones can be constructed from the most intense diffraction peaks.[4] Stationary states and pseudogaps are induced by the quasi-Brillouin zone boundaries, the largest of which is likely associated with a Hume-Rothery-like mechanism for the stabilization of the quasicrystalline structure itself. It is not obvious what topology the Fermi surface takes, if it exists at all, nor whether quantum oscillations will be readily observed in a system so dominated by weak localization. To our knowledge, only one previous report of dHvA oscillations in a quasicrystal has been published,[5] and these results remain unreproduced.

Here, we report initial high-field magnetization measurements of icosahedral $Y_9Mg_{34}Zn_{57}$ (Figure 1) and decagonal $Al_{71}Ni_{11}Co_{17}$ quasicrystals. The samples were prepared via a self-flux technique, as described elsewhere.[6] There is growing experimental evidence that the $Y_9Mg_{34}Zn_{57}$ system has the best atomic order of all of the known quasicrystals, and as such is surely the best choice for initial investigations of high-field behavior. In addition, the unusual symmetry of the decagonal system $Al_{71}Ni_{11}Co_{17}$, being made up of two-dimensional quasicrystalline sheets stacked periodically along the c-axis, lead us to also consider this material in this preliminary study.

The susceptibility for $Al_{71}Ni_{11}Co_{17}$ (H||c) and two orientations (field along 5-fold and 2-fold axis) of $Y_9Mg_{34}Zn_{57}$ was measured using a dHvA setup with a compensated arrangement of detection coils in pulsed magnetic fields up to 60 T. Temperatures between ~400 mK and 4 K were obtained by pumping on ^{3}He and ^{4}He. In all three cases the peaks observed in FFT of the susceptibility were not reproducible from experiment to experiment (even at the same base temperature of ~400 mK) and for increase and decay of the applied field in one measurement.

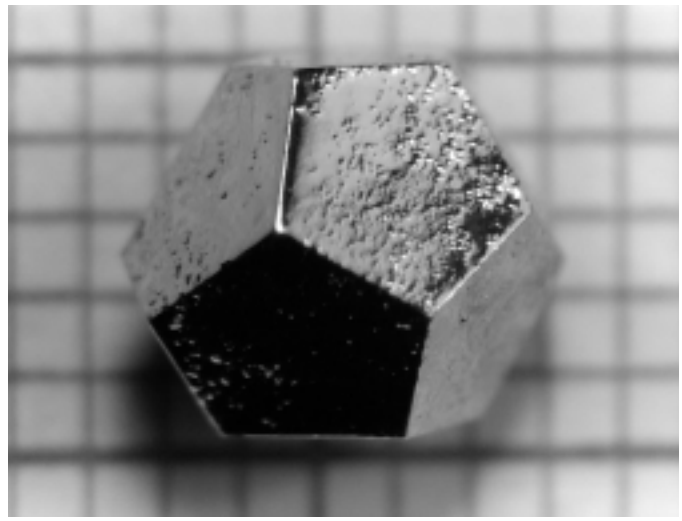

Figure 1. Photograph of an icosahedral $Ho_9Mg_{34}Zn_{57}$ quasicrystal over a mm scale.[7]

The failure to observe quantum oscillations is not necessarily indicative of the absence of a Fermi surface, but may be a consequence of rather short mean free path. We cannot exclude the possibility of observation of quantum oscillations in quasicrystals in general, but these measurements put limits on the size of such an effect if it is to be seen.

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High Field Shubnikov-de Haas Oscillations in Electrodeposited Bi Single Crystal Films

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Bismuth is the archetypal semi-metallic system in which quantum magnetotransport effects were first observed. Single crystals of the material exhibit large, positive magnetoresistance (MR) because of the long mean free paths of the multiband carriers. If high quality thin films of the material can be fabricated, it may be possible to use them as high field, magnetoresistive sensors. Normal thin film growth methods, however, have resulted in granular, polycrystalline films that inhibit the carrier mean free paths.

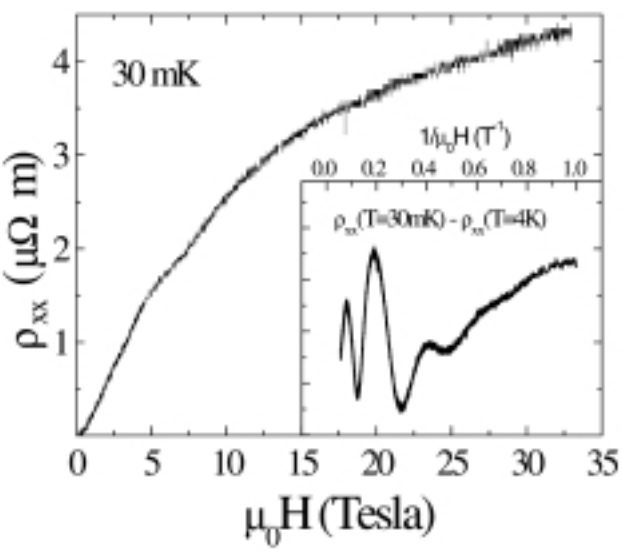

Figure 1. Magnetoresistance (MR) at 30 mK of a single crystal thin film of Bi produced by electrodeposition. The inset shows the Shubnikov-de Haas oscillations obtained by subtracting the 4 K MR data from the 30 mK data, which are periodic in inverse field.

Recently, single crystal thin films have been fabricated by electrodeposition.[1] We have performed MR and Hall measurements on Bi films grown by this method onto Si with an Au buffer layer, in both the superconducting magnet (18 T) and a resistive magnet (33 T), at a temperature down to 30 mK. The MR was measured in three different orientations, taking advantage of the in-situ sample rotator. The perpendicular field orientation, with H along (111), is shown in Figure 1. Shubnikov-de Haas (SdH) oscillations are a low temperature phenomenon, while the positive MR is insensitive to temperature in the residual resistivity regime. Therefore, the oscillations may be isolated by subtracting MR data obtained at 4.2 K from the 30 mK data; this is shown in the inset to Figure 1, where the data is plotted vs. inverse field since SdH is periodic in inverse field. Clear oscillations were observable in both the MR and the Hall effect data, attesting to the high quality of these films.

1 Yang, F.Y., *et al.*, Science, **284**, 1335 (1999).

Peculiarities of the Electron-Dislocation Scattering in Magnetotransport of Transition Compensated Metals at High Magnetic Field

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The transverse magnetoresistivity of pure tungsten and molybdenum single crystals with resistivity ratios of up to 80,000 were measured from 2 to 80 K and in magnetic fields up to 30 T. The samples have different defect structures and dislocation densities, such as dislocation walls formed during crystal growth, and isolated dislocations produced by compression and strain under deformation. We studied the peculiarities of the electron-dislocation scattering at high magnetic fields and showed that this scattering mechanism strongly affects the magnetotransport of these transition compensated metals. The dislocation breakdown, consisting of a change in the electron orbit types due to electron scattering by isolated dislocations, was observed. We also propose that an internal static skin effect involving a concentration of a dc-current near dislocation walls, may be observed in these crystals.

^{109}Ag NMR Studies of Mobile Ions in the Glassy Fast Ionic Conductor 0.525 Ag_2S + 0.475(0.5B_2S_3+0.5SiS_2)

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Interest has risen recently in glassy ionic conductors due to the mobile ion's ability to decouple from the structural relaxation of the glass. Compositional optimization has led to conductivities of ~10^{-2} (cm)$^{-1}$ at room temperature for AgI-doped silver borate glasses. More recent work has shown, Figure 1, that the conductivity for the AgI-doped silver thioborate glasses (glasses made with B_2S_3 instead of B_2O_3) saturates at approximately 10^{-2} to 10^{-1} (cm)$^{-1}$ at room temperature. This saturation shows a non-Arrhenius behavior leading to conductivities well below that of values for room temperature from extrapolated Arrhenius behavior values at low temperature. The facilities at Iowa State University (ISU) have been used to probe the structural and dynamical reasons for this non-Arrhenius behavior present in the AgI-doped silver thioborate glasses.

Due to the nature of Ag being a "poor" NMR nucleus having a low gamma and low sensitivity, measurements at the NHMFL were performed to determine the feasibility of performing future experiments at NHMFL to help understand the origins of the non-Arrhenius conductivity behavior in these glasses. Chemical shift spectra, T_1, and T_2 measurements were performed using both the 20 T and 25 T stations at the NHMFL on a fast ion conducting glass of nominal composition 0.525 Ag_2S+0.475(0.5B_2S_3+0.5SiS_2).

The chemical shift measurements at ISU at 8.4 T, in Figure 2, the top spectra at * T revealed three distinct silver environments exist in these glasses. Chemical shift measurements at the NHMFL at 20 T, bottom spectra of Figure 2, showed only two

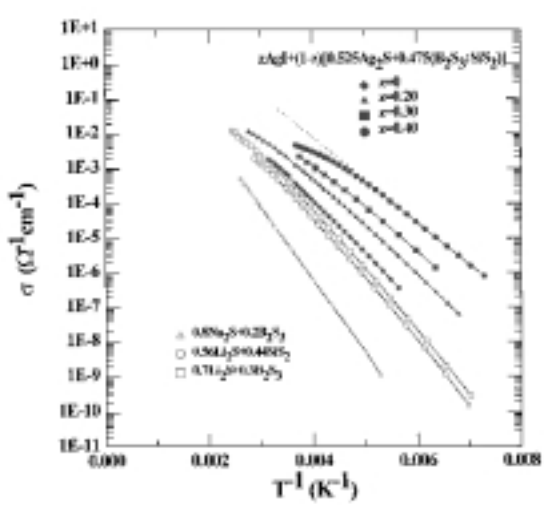

Figure 1. Conductivities for various fast ion conducting glasses.

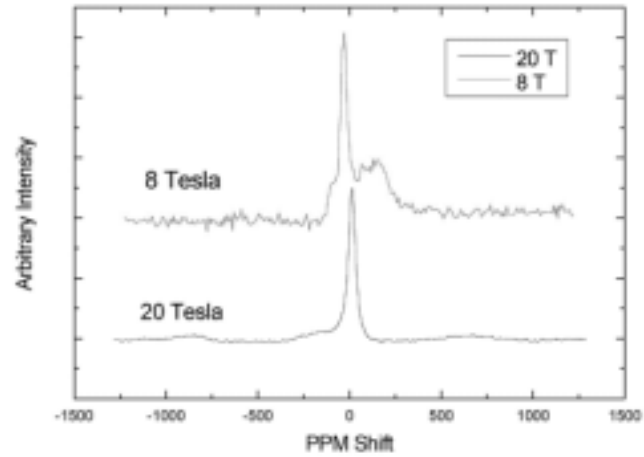

Figure 2. Chemical shift spectra for two separately prepared glass samples with composition 0.525 Ag_2S+0.475(0.5 B_2S_3+0.5 SiS_2).

of these sites. Both high field peaks could be in the envelope of the one main peak centered at approximately the resonance frequency due to dipolar broadening. The chemical shift of the third site, however, is too large for the possibility of it being encapsulated by the peak shown as a result of dipolar broadening. The two samples measured, while of the same nominal composition, were prepared at two separate times and by different researchers. This discrepancy in the chemical shift spectra could be due to a chemical and/or structural difference arising from the possible slightly different preparation techniques.

This hypothesis is being investigated by sending the sample studied at ISU to the NHMFL for study at high magnetic field and the sample studied at the NHMFL will be returned to ISU for study at lower magnetic fields.

The T_1 value measured at NHMFL revealed T_1 times of ~0.25 seconds at room temperature for the peak shown at 297 K. These values are consistent with those measured at ISU. The T_2 relaxation time was approximately 0.0004 seconds. No T_2 data from ISU exists to make a comparison of the T_2 measurement.

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Thermometry at Low Temperatures and High Magnetic Fields

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There exists no commercially available thermometer for mK temperatures that does not have significant magnetoresistance.

The resistive thermometers most often used in this temperature range for research in high magnetic fields are commercial RuO_2 chip resistors. These thermometers are also available packaged and calibrated from a number of vendors. At temperatures below 50 mK, these thermometers exhibit an anomalous spike in the resistivity at magnetic fields below 0.1 T. This anomalous behavior grows larger as the temperature is decreased and is asymmetric with magnetic field. We have performed detailed measurements using a torque magnetometer and an ac susceptometer that have revealed a magnetic moment that develops at temperatures below 50 mK. Measurements of a variety of chips with different types of contacts and measurements of bare substrate have proven that the moment is within the resistor and not external to the conductor. The development of the moment correlates with the anomalous resistive behavior and is thought to be due to an interaction between the weakly localized carriers and the local moments.

To address this shortcoming capacitance thermometers have been developed using multiple layers of kapton and copper in a unique design. This design has allowed the capacitance of the devices to be increased over conventional low temperature capacitance thermometers by a factor of roughly 10,000 while still maintaining a small size and low thermal mass. In addition, we have demonstrated that their sensitivity to temperature can be increased by a factor between 2 and 4 and the position of the minimum in capacitance can be moved by varying the construction parameters. Most importantly they exhibit no magnetocapacitance within the resolution of our measurement system. The capacitance vs. temperature measurements have been extended to room temperature and measurements on drift have also been performed.

Preliminary measurements on Coulomb blockade thermometers have been continued. These thermometers are expected to be a primary thermometer independent of temperature to very high magnetic fields. Precision experiments at 0.58 K have shown that these thermometers are independent of field to 33 T with a variance of about 1%. Our measurements were limited, however, by noise problems and did not provide a reliable absolute temperature as the devices had aged. These experiments will be continued with new CBTs that work at lower temperatures, are encapsulated to prevent aging, and are not superconducting thus allowing zero field measurements. In addition a custom designed bridge will be tested that will allow us to extend these measurements down to 50 mK and below.

Magnetic Alignment of Carbon Nanotubes

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Single-wall carbon nanotubes[1] have a broad range of potential applications due to their exceptional mechanical, thermal, electronic, and optical properties. To date, these properties have been studied primarily using mats of randomly oriented nanotubes, obscuring the true anisotropic behavior of the nanotubes. Theory[2] has predicted that nanotubes will be more diamagnetic when oriented perpendicular to a magnetic field than when parallel to it. Therefore, the magnetic energy $U=-\boldsymbol{m}\cdot\boldsymbol{B}$ of a freely rotating nanotube will be minimized when its axis is aligned with the field direction. To achieve alignment, the field must be strong enough that the alignment energy is many times the thermal energy k_BT.

Using the 19 T, 198 mm bore resistive magnet at the NHMFL, we measured the optical transmittance of a colloidal suspension of nanotubes as a function of polarization angle and field (Figure 1). The sinusoidal polarization dependence at fields >4 T is evidence for magnetic alignment of the nanotubes. The amplitude of the sinusoid saturates at the highest fields; our

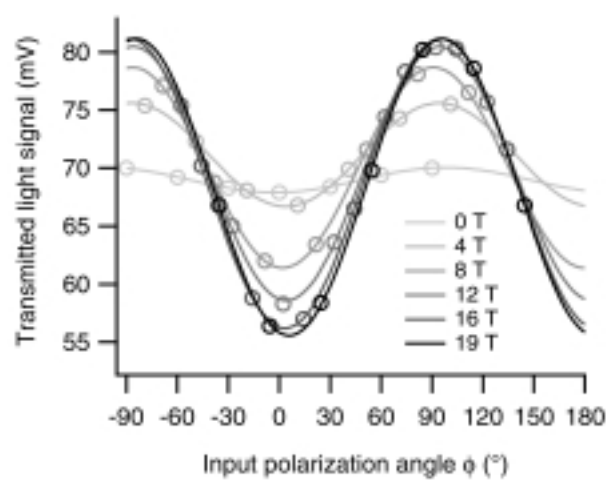

Figure 1. Optical transmittance at λ = 633 nm of a suspension of single-wall carbon nanotubes. The sinusoidal variation with ϕ indicates that nanotubes are aligned parallel to the magnetic field. The effect saturates at a peak-to-peak-modulation of almost 40%, due to the excellent alignment achieved in high fields. Note: the angle ϕ = 0° corresponds to incident polarization parallel to the field direction.

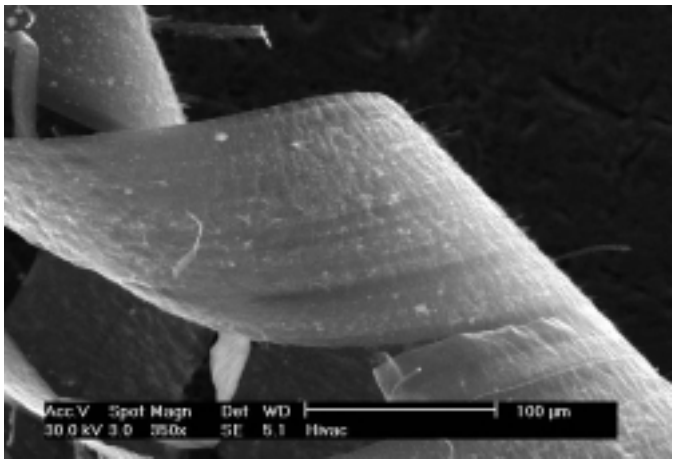

Figure 2. Ribbon of the novel aligned nanotube solid material produced upon filtering a colloidal suspension of nanotubes in a magnetic field of 25 T. Due to the high degree of organization, the material cleaves along lines parallel to the magnetic field. This material holds great promise for studying, in a bulk assembly, the anisotropic properties of nanotubes.

model reveals that the alignment energy of the nanotubes at 19 T is (28 ± 3) times thermal energy at room temperature.

To study further the properties of aligned nanotubes, we assembled them into a solid layer by filtration in a magnetic field parallel to the filter membrane surface. Upon drying, the resulting film could be peeled off the filter membrane. It cleaved readily into long ribbons with edges parallel to the magnetic field direction (Figure 2), demonstrating that it consists of long ropes of nanotubes that are well-oriented parallel to the field. Ongoing experiments are exploring the electronic and optical properties of this novel material. It may even be capable of acting as a seed crystal for the growth of continuous nanotube cables and fibers.

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Magnetic Resonance Techniques

Solid State NMR Studies in Polypropylenes

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Solid State NMR studies of polypropylenes have been performed within two ongoing research programs in our laboratory.

The first program focuses in understanding the partitioning of the defects of the polypropylene molecule among the different phases of the lamellar crystallite. Homo-polypropylenes and different types of random propylene copolymers have been studied and resonances associated with the defects in the crystallite regions have been identified directly by CP MAS ^{13}C NMR as well as theoretically by quantum chemical computations. This work has been carried out in collaboration with Dr. D. VanderHart of the National Institute of Standards and Technology (NIST). Two main papers have been submitted to Macromolecules and the work has been presented in different meetings of the American Chemical and Physical Societies.

A second program is directed to understand the conformational and dynamic properties that cause profound melting kinetics in the fusion of polypropylene crystals. This work is part of Ms. W.T. Huang's Ph.D. thesis. Spin lattice relaxation (T_{1H}) in the interlamellar amorphous region are measured aiming to understand relaxation and conformational properties of this region. These studies will be completed during the 2000 Spring semester. Polypropylenes with different defects contents will be crystallized for different lengths of time and the T_{1H} will be measured. The results will determine if melting kinetics is a consequence of entropic change in interlamellar constrained regions during crystallization. Ms. Huang is expected to defend her Ph.D. in the Fall of 2000.

Development of Large Volume High Frequency RF Coils

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Traditional design techniques used at lower field strengths begin to break down on high field instruments, requiring a new perspective at these higher frequencies. Recent techniques appearing in the literature involve the use of resonant cavities with Vaughans's TEM resonator the most widely applied.[1] The purpose of this work is to explore and simplify one specific type of cavity, the reentrant cavity, and as a proof of concept, construct and image with this simplified design. This work has been accepted by the next ISMRM meeting.[2]