CONF-960292--2

LA-UR-96-2033

Title: de Haas-van Alphen Experiments in Pulsed Magnetic Fields

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Submitted to: International Workshop on High Magnetic Fields: Industry, Materials and Technology
Tallahassee, FL
February 27-March 1, 1996

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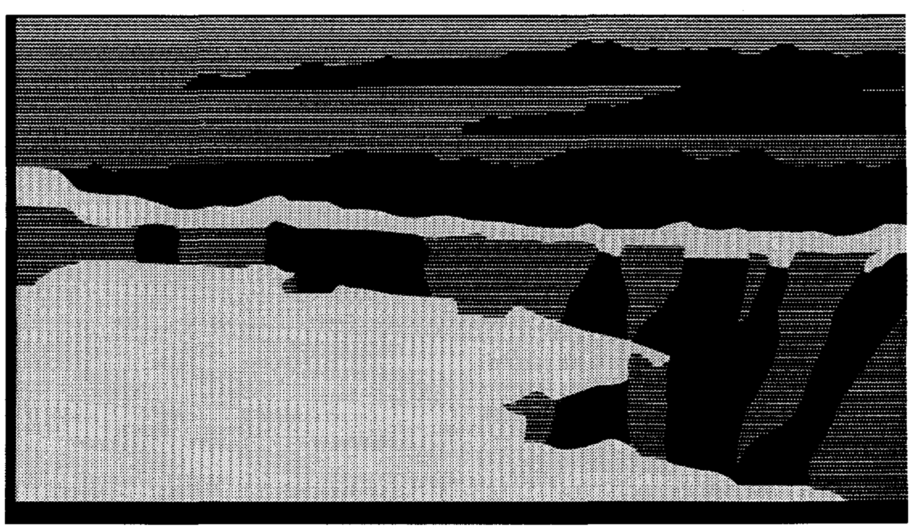

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de Haas-van Alphen Experiments in Pulsed Magnetic Fields

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The powerful combination of de Haas-van Alphen effect and pulsed magnetic fields enables the study of the Fermi surface and the electronic properties of various materials of current interest. In this contribution I discuss experiments on CeB_6, a heavy fermion, on $YBa_2Cu_3O_{7-\delta}$, a high-T_C superconductor, and on $Al_{70}Pd_{21.5}Mn_{8.5}$, a quasicrystal. For each of these experiments I will point out the essential contribution of the pulsed magnetic field.

1. Introduction

The importance of high magnetic fields for materials research and fabrication cannot be questioned. Magnetic fields have been used as an experimental tool to examine the electronic and magnetic structures of materials. When the energy associated with the magnetic field (*e.g.* cyclotron energy or spin-splitting energy) is small compared to the relevant energy scales of the material under study, the experiment probes the zero-field electronic or magnetic structure of the material. In the opposite limit the magnetic field can induce transitions which are interesting to study in their own right. Besides as an experimental tool, high magnetic fields have been put to use for the fabrication of high-quality single crystals, because a magnetic field can provide a preferential direction for crystal growth or reduce convective flow in the melt. Examples of this application of magnetic fields can be found elsewhere in the proceedings of this Workshop. Lastly, a magnetic field can be used to shift the equilibrium of chemical reactions, exploiting the small differences in magnetic properties of the reactions components.

This article deals with one experimental tool for the study of the electronic structure of metals: the de Haas-van Alphen (dHvA) effect. As we will see later, this is the oscillatory component of the magnetization of a metal, which can be observed at low temperatures and high magnetic fields. The dHvA effect is arguably the most precise tool for the study of the Fermi surface of a metal. After an introductory discussion about the theoretical basis of the dHvA effect and the experimental information that it contains, I will describe experiments using the dHvA effect in pulsed magnetic fields up to 52 T. The experiments, which benefited from the pulsed fields in an essential way, involved three different materials of current interest: CeB_6, a heavy fermion, $YBa_2Cu_3O_{7-\delta}$, a high-T_c superconductor, and $Al_{70}Pd_{21.5}Mn_{8.5}$, a quasicrystal. The experiments have been carried out at the Pulsed Field Facility in Los Alamos, and at the Service National des Champs Magnétiques Pulsés in Toulouse, France.

2. The de Haas-van Alphen effect

The de Haas-van Alphen (dHvA) effect [1] is the oscillatory component of the magnetization of a metal, which can be observed at low temperatures and high magnetic fields in high-quality single crystals. The oscillations occur as a function of changing magnetic induction B and are periodic in $1/B$.

Electrons in a magnetic field describe orbits (in k-space) which are the intersection of a surface of constant energy and a plane perpendicular to the direction of the applied field. Only closed orbits contribute to the dHvA effect. Each closed orbit must enclose a quantized area proportional to B. On increasing B the orbits expand. Some orbits cross the Fermi surface and depopulate, states outside the Fermi surface being empty. The expansion and depopulation of successive orbits crossing the Fermi surface gives rise to oscillations in the free energy periodic in $1/B$, which show up as oscillations in, *e.g.*, the magnetization. Calculations show that only those orbits crossing the Fermi surface where it has an extremal area, minimum or maximum, contribute to the dHvA effect.

Being periodic in $1/B$, the oscillating magnetization $\tilde{M}$ can be written as a sum of oscillating terms with frequencies multiples of a fundamental frequency F:

$$\tilde{M} = \sum_{r=1}^{\infty} M_r \sin\left(2\pi r \frac{F}{B} + \varphi_r\right) \tag{1}$$

In this expression the fundamental frequency F is the dHvA frequency associated with a particular extremal orbit, M_r is the amplitude of the r-th harmonic and φ_r is a phase factor irrelevant for the present discussion. The amplitudes M_r rapidly decrease with increasing r, so that in practice only one or few harmonics are observed. If the Fermi surface allows several extremal orbits, each orbit gives a contribution of the form of Eq. 1, and the total oscillatory magnetization is the superposition of all these contributions.

The frequency F and the amplitudes M_r contain much information about the metal under study. The frequency is proportional to the extremal cross-sectional area in reciprocal space enclosed by a cyclotron orbit, A_{extr}:

$$F = \frac{\hbar}{2\pi e} A_{extr} \tag{2}$$

Measurement of the dHvA frequency thus provides a measure of the size of the Fermi surface of a metal. By changing the orientation of the magnetic field with respect to the crystallographic axes of the sample, and often guided by band structure calculations, it is possible to reconstruct the entire Fermi surface.

The amplitudes of the various frequency components contain much additional information about the electrons on the Fermi surface. Lifshitz and Kosevich [2] presented a detailed theory of the dHvA effect, valid for independent electrons moving in a crystal potential. The expression describing the dHvA effect bears their name: Lifshitz-Kosevich (LK) formula. It gives the following expression for M_r:

$$M_r = \frac{C\sqrt{B}}{r^{3/2}} \frac{\alpha r m^* T / B}{\sinh\left(\alpha r m^* T / B\right)} \exp\left(-\alpha r m^* T_D / B\right) \tag{3}$$

where $\alpha = 14.69$ T/K is a combination of physical constants, m^* (in units of m_e, the free electron mass) is the effective mass of the electrons on the cyclotron orbit, T is the absolute temperature and T_D the Dingle temperature, inversely proportional to the life time of the electrons. At constant magnetic field, the effective mass can be determined by measuring the variation of the amplitude with temperature. At constant temperature, the Dingle temperature, and thus the life time of the electrons, can be determined by measuring the variation of the amplitude with magnetic field. Precise measurement of the amplitude can furnish additional information on the g-factor of the electrons and on the curvature of the Fermi surface, both of which are contained in the prefactor C in Eq. 3.

Often one would like to carry out dHvA experiments at as high a magnetic field as possible. In dirty metals or alloys, *e.g.*, very high fields are necessary to bring out the oscillations. In superconductors one would like to experiment at fields close to or above the critical field. Special effects may occur at very high fields, like field-induced phase transitions or a field-dependent effective mass. Superconducting magnets, able to generate fields up to 20 T, or water-cooled resistive magnets, able to generate fields up to 35 T, may not suffice. These magnets generate steady fields, having strengths constant in time. Pulsed magnets, by contrast, have the potential to generate considerably higher magnetic fields by allowing the field to exist for only a very short time.

In the experiments which I will discuss here, the magnetic field was generated by the discharge of a large capacitor bank over a magnet coil. The capacitor bank was previously charged to a high voltage, up to 10 kV, which corresponds to a maximum stored energy of the order of 1 MJ. The magnet coil is cooled in liquid nitrogen to take advantage of the reduced resistance and improved mechanical properties at low temperature of the copper wire used to wind the magnet. The profile of the field thus generated is sketched in Fig. 1. In Los Alamos the rise time to maximum field of the pulsed magnets is 6.5 ms and the total duration of the field about 50 ms. In Toulouse these values are 35 ms and 180 ms, respectively. The maximum field in the experiments described below was 52 T.

3. CeB_6

Heavy fermions are intermetallic compounds containing rare earth (mainly Ce) or actinide (mainly U) atoms [3]. At high temperatures, these materials show a Curie-Weiss-like magnetic susceptibility and a logarithmic divergence of the resistivity, both characteristic of the single-impurity Kondo effect. At low temperatures their behavior is completely different. There the specific heat and magnetic susceptibility behave like those of a Fermi liquid, but with values 100-1000 times the corresponding values for ordinary metals. Expressed in terms of the effective mass of the electrons (à la Sommerfeld), the electrons have effective masses of 100-1000 m_e, where m_e is the mass of the free electron, hence the name heavy fermion. It is believed that the typical behavior of heavy fermions results from the interaction between the conduction electrons and the *f*-electrons of the Ce or U-atoms.

Here I discuss dHvA experiments in magnetic fields up to 52 T on the heavy fermion compound CeB_6, a well-studied material [4]. CeB_6 has a high value of γ, the linear coefficient of the low temperature specific heat, $\gamma = 250$ mJ/mole·K^2. In comparison, LaB_6 has no *f*-electron and $\gamma = 2.6$ mJ/mole·K^2. Thus the electrons in CeB_6 have effective masses of order 100 m_e in zero magnetic field. Several workers have measured the effective mass of the electrons in CeB_6 by measurements of the low temperature specific heat and the dHvA effect [4]. In the course of these experiments Joss *et al.* [5] discovered that this effective mass decreases strongly with increasing magnetic field. Using pulsed magnets, we have extended these experiments to higher fields [6]. This enabled us to follow the mass decrease up to higher fields and to study the dHvA effect in this compound at temperatures in the vicinity of the Kondo temperature, which for CeB_6 equals about 2 K.

Oscillations obtained at a temperature of 1.7 K and at fields between 40 T and 50 T are shown in Fig. 2a. The magnetic field was applied parallel to the [001] direction of the cubic crystal structure. The signal contains several oscillatory components periodic in $1/B$. Fourier analysis of these data, shown in Fig. 2b, reveals that there are three main frequency components in the signal. Two of these with dHvA frequencies of 8.68 kT and 17.36 kT are harmonics and stem from an X-point centered belly orbit, referred to as the α_3-orbit. The third frequency of 11.0 kT was first observed in this work and stems from another X-point centered orbit, the $\alpha_{1,2}$-orbit. Similar spectra were observed by Harrison *et al.* [7].

We have recorded dHvA oscillations in CeB_6 at nine different temperatures between 1.7 K and 2.25 K and at magnetic fields between 30 T and 52 T. The effective mass of the electrons was calculated from the temperature dependence of the amplitude A of the dHvA oscillations. Approximating the hyperbolic sine in the LK-formula by an exponential, one finds that a plot of $\ln(A/T)$ as a function of T should yield a straight line with a slope proportional to m^*. For the α_3 orbit (F=8.68 kT) such a plot is shown in Fig. 3. The amplitudes were determined from the Fourier transform of subsets of the data in field intervals centered around the values listed in Fig. 3. Below 2.2 K the straight-line behavior is indeed observed. The effective masses derived for this orbit, as well as for the two other frequencies, are listed in Table 1.

The effective masses calculated from all observed frequencies have been listed in Table 1 and shown as solid dots in Fig. 4. Also shown in Fig. 4 is a fit of all available effective masses determined by dHvA experiments to a theoretical expression derived by Wasserman *et al.* [8]. For the α_3-orbit the mass derived from the second harmonic is consistent with that calculated from the fundamental. The mass of the electrons in the $\alpha_{1,2}$ orbit is larger than the mass of those in the α_3-orbit and field dependent as well. These two facts may explain why the $\alpha_{1,2}$ orbit has not been observed at lower magnetic fields. Its existence may account for the discrepancy between the effective masses derived from specific heat measurements and those derived from dHvA experiments.

The dHvA signal vanishes rapidly for temperatures above 2.2 K, as can be seen in Fig. 3. This temperature is close to the λ-point of the coolant, ^4He. We do not believe, however, that this disappearance is related to the superfluid transition, or that the sample is insufficiently cooled above 2.2 K during the field pulse. Several high-purity metals like Ag, Au, and Pt have been studied using the same experimental setup and dHvA oscillations have been observed in all of those materials up to temperatures much higher than 2.2 K. Effective masses calculated from these experiments agreed with the established literature values for all temperatures. One expects problems of eddy current heating to be less important in CeB_6 than in the pure metals, in which such problems have not been observed.

Heavy fermions are characterized by a coherence temperature, which separates the low temperature Fermi liquid regime from the high temperature regime in which no dHvA effect is expected. As an alternative explanation for the vanishing of the dHvA signal above 2.2 K, we propose that we are witnessing the transition between these two regimes. This temperature is also close to the estimated Kondo temperature. However, Harrison *et al.* [7] do not agree with this scenario.

Note that this experiment was made possible due to the confluence of two factors: the availability of high magnetic fields and the simultaneous decrease of the effective mass of CeB_6.

4. $YBa_2Cu_3O_{7-\delta}$

Detailed knowledge of the electronic structure and the Fermi surface of the high-T_c superconductors is of great interest, as it may test the validity of theoretical models proposed to describe these materials and thus provide clues about the mechanism of superconductivity. The electronic structure of high-T_c superconductors has been studied extensively using angle-resolved photoemission spectroscopy, positron two-dimensional angular correlation of annihilation radiation, and the dHvA effect [9]. Three groups have reported observation of the dHvA effect in $YBa_2Cu_3O_{7-\delta}$. Fowler *et al.* [10] have used flux compression techniques to generate magnetic fields in excess of 100 T. They have reported the observation of three dHvA frequencies of (0.53 ± 0.02) kT, (0.78 ± 0.02) kT, and (3.51 ± 0.10) kT. Kido *et al.* [11] have used conventional field modulation techniques in a 27 T hybrid magnet and have reported a single dHvA frequency of (0.54 ± 0.03) kT.

We have conducted experiments on an oriented powder of $YBa_2Cu_3O_{7-\delta}$ in pulsed magnetic fields up to 52 T and at temperatures down to 1.7 K [12]. The grain size of the powder was smaller than 10 μm. The powder was mixed with a non-conducting epoxy and placed in a 10 T magnetic field while the epoxy cured. Due to the anisotropy of the magnetic susceptibility of $YBa_2Cu_3O_{7-\delta}$ the magnetic field exerts a torque on each grain, which aligns it with the *c*-axis parallel to the applied field. This was also the direction in which the field was applied during the experiments. In the *a,b*-plane the grains remained completely disordered. The high degree of orientation was verified using X-ray texture analysis [13].

The advantage of using oriented powders is that the magnetic field penetrates well in between and into the superconducting grains. This can be verified using, *e.g.*, the Bean model of the critical state. Eddy current heating is suppressed due to the small grain size. In extreme type-II superconductors like $YBa_2Cu_3O_{7-\delta}$, the magnetic field penetrates into the superconducting material in the form of vortices and due to the large value of κ, the Ginzburg-Landau parameter, the field variations in between vortices are small. The dHvA has been observed in the superconducting state of a variety of extreme type-II superconductors.

An example of a signal obtained on an oriented powder at a temperature of 1.7 K is shown in

Fig. 5. The large step-like feature around 45 T is an artifact of the measurement and should be disregarded. Between 20 T and 40 T oscillatory signals are observed. To examine the presence of components periodic in 1/B, we have Fourier analyzed this signal in the field interval between 20 T and 40 T. The resulting Fourier transform is shown in Fig. 6. The two largest peaks have frequencies of 0.52 kT and 0.74 kT. These values are in excellent agreement with the lowest two frequencies obtained by the Fowler *et al.* [10], and our lowest frequency is in good agreement with that observed by the Kido *et al.* [11].

The maximum amplitude of the dHvA oscillations occurs around 30 T. Maniv *et al.* [14] have published a theory of the dHvA effect in the superconducting state of two-dimensional extreme type-II superconductors. Their theory can account for a similar field dependence of the amplitude of the dHvA oscillations. For a specific choice of the parameters in their theory, reasonable in the case of $YBa_2Cu_3O_{7-\delta}$, a maximum amplitude can occur around 30 T. On the other hand, a recent theory by Tesanovic *et al.* [15], which successfully describes the field dependence of the dHvA amplitude in V_3Si, a three-dimensional superconductor, does not predict a maximum amplitude below B_{c2}.

5. $Al_{70}Pd_{21.5}Mn_{8.5}$

Quasicrystals [16] have rotational symmetries that prohibit real-space translational symmetry. Consequently, Bloch's theorem does not apply to these materials. How can the electronic structure of such materials be described? What is the meaning of such familiar concepts as the Fermi surface? We have addressed these questions by studying the dHvA effect in the face-centered icosahedral quasicrystal $Al_{70}Pd_{21.5}Mn_{8.5}$ [17].

The experiments have been carried out in magnetic fields up to 50 T and in a ^3He cryostat at temperatures down to 400 mK. We used two samples in the experiments. In one sample the magnetic field could be applied parallel to a five-fold axis, in the other one parallel to a two-fold axis. Both samples were cut from the quasicrystal in which Kycia *et al.* [18] have observed the Borrmann effect, the anomalous transmission of X-rays due to multiple scattering. In crystals, the observation of the Borrmann effect is a sign of the high structural perfection of the crystal.

In the Al-Pd-Mn system quasicrystals exist over a range of compositions. Small differences in composition result in differences in resistivity and magnetization. The quasicrystal we have used had a room-temperature resistivity of 1250 $\mu\Omega$ cm, decreasing by only 4 % between room temperature and 4 K. Although the resistivity is very large, larger values up to 9000 $\mu\Omega$ cm have been reported.

Fig. 7a and 7b show the power spectral densities of two separate measurements, both done under the same conditions. The temperature was 400 mK and the magnetic field was parallel to a five-fold quasicrystallographic axis. The spectra were obtained on subsets of the data between 50 T and 37 T. Both spectra show peaks at 0.96 kT and 3.55 kT. Not only the positions but also the heights of the peaks in both spectra are equal within 2 %. For fields below 37 T no oscillations were observed. On increasing the temperature to 470 mK the amplitude of the oscillations decreased much. Assuming that the LK formula applies to quasicrystals, this implies that the electrons in quasicrystals have large masses. In subsequent experiments we verified that without sample in the detection system no reproducible peaks were observed. The small peaks that were observed had heights lower by at least a factor 30 compared to those in Fig. 7.

The power spectral densities shown in Fig. 8a and 8b were obtained at two different temperatures, 450 mK and 500 mK, and with the magnetic field parallel to a twofold axis. Several peaks are observed whose positions are unchanged on varying the temperature. The two most important peaks have frequencies of 0.36 kT and 1.21 kT. The height of the peaks decreases substantially on increasing the temperature. From the amplitude reduction we estimate the effective masses of the electrons to be 61 m_e and 25 m_e, for the 0.36 kT and 1.21 kT orbits, respectively.

In summary, we have observed oscillatory signals periodic in $1/B$, whose frequency is unchanged between different measurements, whose amplitude is constant for constant temperature and decreases either when the temperature is increased or when the magnetic field is lowered. It is fair to conclude that we have observed the dHvA effect in this quasicrystal.

To explain the existence of the dHvA effect in a quasicrystal, we propose that the Fermi surface of a quasicrystal consists of a multitude of small pieces. Certainly the dense presence of Bragg planes in a quasicrystal should fracture the Fermi surface in a multitude of tiny pieces, in principle infinitesimally small. Magnetic breakdown, electron tunneling through the small energy gap separating the different sheets of the Fermi surface, can reconnect the different pieces and give rise to the orbits of the size we observe. The tunneling probability depends exponentially on the the strength of magnetic field. Therefore we should observe a characteristic field dependence of the amplitude of the dHvA oscillations. Verifying this prediction will be the subject of future experiments.

It should be pointed out that the pulsed field was essential to observe the dHvA effect in the quasicrystal, because the effect was only observed in fields exceeding 37 T, in combination with very low temperatures.

6. Conclusions

In this paper I have discussed dHvA experiments in pulsed magnetic fields on three materials. In the heavy fermion CeB_6 we have observed the decrease of the effective mass up to 52 T. We have observed a new cyclotron orbit, which could not be observed at lower magnetic fields and which could account for a previously unexplained discrepancy. Because of the high magnetic field and the associated mass decrease, we were able to perform experiments at temperatures in the vicinity of the coherence temperature. This enabled us to possibly observe the breakdown of the heavy fermion state.

In the high-Tc superconductor we have observed the dHvA effect in fields between 20 T and 40 T. The two dHvA frequencies that we have been able to identify are in good agreement with those observed by two other groups. We have observed an unusual field dependence of the amplitude of the oscillations, which can be accounted for by a theory of Maniv *et al.*

In the quasicrystal $Al_{70}Pd_{21.5}Mn_{8.5}$ we have observed the dHvA effect at fields above 37 T and at temperatures below 500 mK. The rapid decrease of the amplitude led us to believe that the effective masses in $Al_{70}Pd_{21.5}Mn_{8.5}$ are large, 61 m_e and 25 m_e.

Measurements of the dHvA effect in pulsed fields up to 50 T have

7. Acknowledgments

I am indebted to S. Askenazy for allowing ample access to the Pulsed Field Facility in Toulouse. I am grateful to F. M. Mueller for many fruitful discussions and helpful suggestions, and to W. Joss and P. Wyder for support during several phases of this work.

Part of this work was supported through the NSF Cooperative Agreement #DMR-9016241, the State of Florida, and under the auspices of the U.S. Department of Energy.

8. References

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Figures and Tables

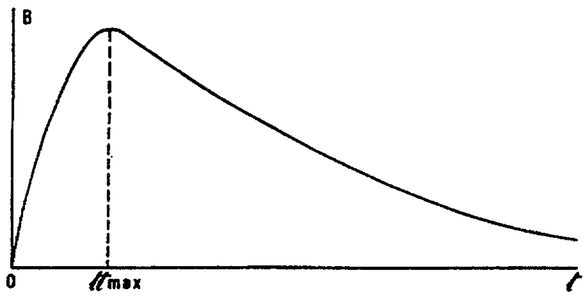

Figure 1. Typical field profile of a pulsed magnet

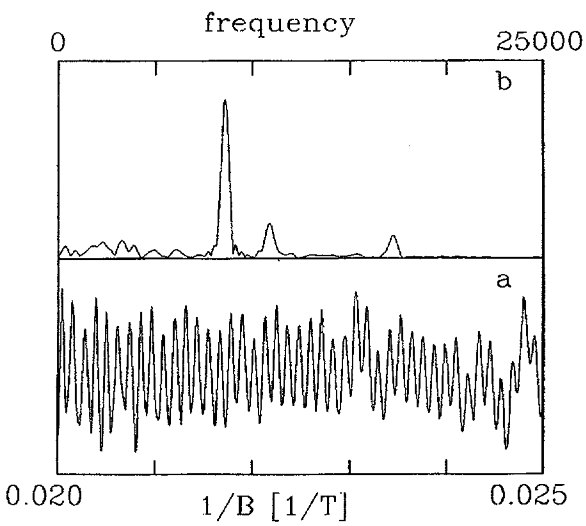

Figure 2. (a) De Haas-van Alphen oscillations observed in CeB_6 at a temperature of 1.7 K and at fields between 40 T and 50 T. (b) Fourier transform of this signal showing three principal frequency components at 8.68 kT, 11.0 kT, and 17.36 kT.

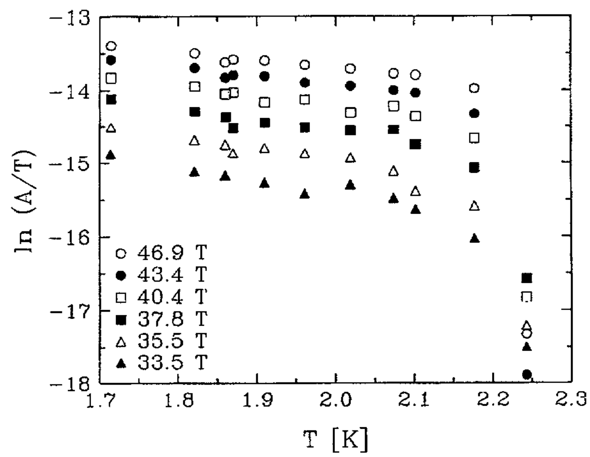

Figure 3. Temperature dependence of the amplitude of the dHvA component with F=8.68 kT for several field values.

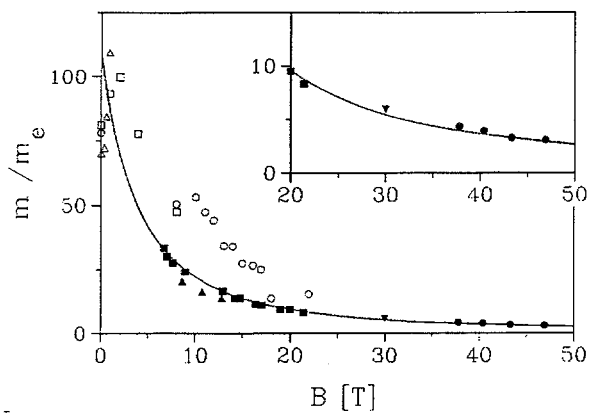

Figure 4. Field dependence of the effective mass of CeB_6. The open symbols were obtained from the low temperature specific heat. The closed symbols are from dHvA experiments for the α_3 orbit. The solid dots are the values listed in Table 1. The solid line is a fit to a theory by Wasserman *et al.* [8].

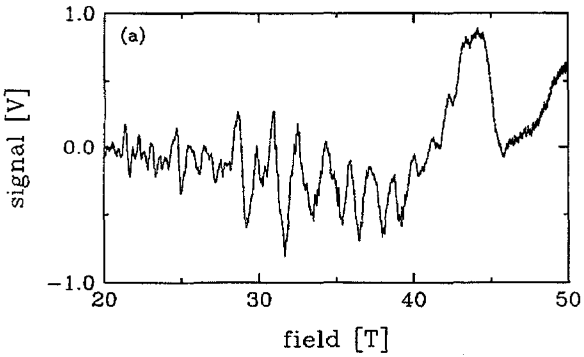

Figure 5. Oscillatory signals observed in a sample of oriented $YBa_2Cu_3O_{7-\delta}$ powder at 1.7 K.

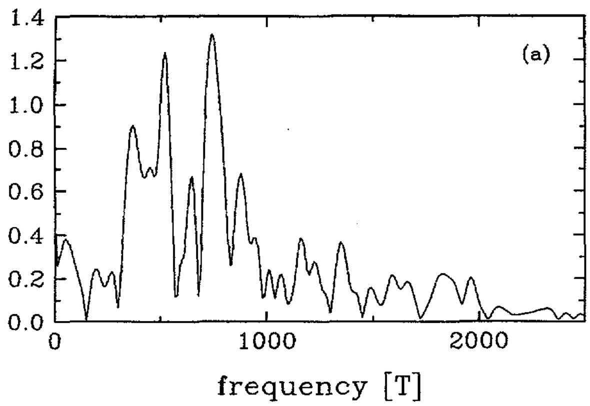

Figure 6. Fourier transform of the signal of Fig. 5 in the field range between 20 T and 40 T.

Figure 7. Power spectral densities of the dHvA oscillations in $Al_{70}Pd_{21.5}Mn_{8.5}$. In (a) and (b) results are shown from two separate measurements. In both measurements the magnetic field was parallel to a five-fold axis and the temperature was 400 mK.

Figure 8. Power spectral densities of the dHvA oscillations in $Al_{70}Pd_{21.5}Mn_{8.5}$ with the magnetic field parallel to a two-fold axis. In (a) the temperature was 450 mK and in (b) the temperature was 500 mK.

B[T]	m^*/m_e		
	F=8.68 kT	17.36 kT	11.0 kT
46.9	3.06±0.13	3.5±1.1	5.0±1.1
43.4	3.26±0.13	3.7±1.1	6.5±2.2
40.4	3.93±.037		
37.8	4.34±0.13		

H. Monkhorst (University of Florida): What do you mean when you talk about small pieces of Fermi surface?

E. G. Haanappel: Imagine not a quasicrystal but an approximant to a quasicrystal. An approximant is a crystal with a very large unit cell containing hundreds of atoms. The corresponding Brillouin zone is small. When the Fermi surface of such a material is mapped into the first Brillouin zone, the resulting sheets of Fermi surface are very small as well. Better approximants to a quasicrystal have larger units cells and smaller pieces of Fermi surface. The real quasicrystal

G. Kido (NRIM): dHvA oscillations have recently been observed by T. Terashima (*Solid State Commun.* **96**, 459 (1995)) in YNi_2B_2C, not only in the normal state but also in the mixed state down to fields of 0.2 B_{c2}.

E. G. Haanappel: dHvA experiments in the normal and superconducting state of YNi_2B_2C have also been reported by Heinecke and Winzer (Z. *Phys.* **B98**, 147 (1995)) and by Goll et al. (*Phys. Rev.* **B53**, R8871 (1996)). There are now a number of materials in which the dHvA effect has been observed in the mixed state: 2H-$NbSe_2$, V_3Si, $YBa_2Cu_3O_{7-\delta}$ Nb_3Sn, $Ba_{0.6}K_{0.4}BiO_3$, various organic conductors.