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Measurement of de Haas-van Alphen Effect with Hybrid Magnet*

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Synopsis

An apparatus to measure the de Haas-van Alphen effect in steady high magnetic field has been constructed by using hybrid magnets at Tohoku University. Fermi surfaces were investigated on cuprate high-temperature superconductor and rare earth antimonide. The spectral analyses were performed by using the maximum entropy method as well as the Fourier transformation. In magnetically oriented $YBa_2Cu_3O_{7-\delta}$ and $Tl_2Ba_2CuO_{6+\delta}$ powder, the spectral density shows a peak at 456 T and 400 T, respectively. A slight change of extremal cross-section was found in PrSb above 15 T.

I. Introduction

The de Haas-van Alphen (dHvA) effect is concerned with periodic oscillation in magnetization against an inverse magnetic field. The oscillatory frequency, F, is related to the extremal cross-section area, A_{ext}, of the Fermi surface perpendicular to the magnetic field,

$$F = \hbar c A_{ext}/2\pi e. \qquad (1)$$

Followingly, the measurement of the dHvA effect is a direct way to study the Fermi surface. However, to observe the dHvA oscillation, two conditions of $\omega_c\tau>1$ and $\hbar\omega_c>k_BT$ have to be satisfied, where $\omega_c=eH/m^*c$ is the cyclotron frequency, τ is the collision time, m^* is the effective mass of carrier. High magnetic fields and low temperatures expand the adaptability of dHvA measurement on new materials.

Since the oxide superconductors were discovered, much attention has been paid to figure out the nature of the strange material properties. The presence of large Fermi surface has been suggested by the experimental results such as angle-resolved photoemission, two-dimensional angular correlation of positron annihilation radiation, etc. [1-2]. A precision dHvA measurement using steady high magnetic field had been expected to establish the existence of Fermi surface in cuprate high-temperature superconductors. We have measured the dHvA effect of ceramic super conductors and determined the cross-sectional area of the Fermi surface [3-5].

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On the other hand, interesting phenomena, such as dense Kondo behavior and heavy fermion state, are expected in rare earth compounds. We measured the dHvA effect in rare earth antimonide crystals and found a field dependence of Fermi surface at very high magnetic field range [6]. Since a clear oscillation can be seen at high field range in these compounds, it was quite convenient to check our dHvA equipment [7].

II. Experimental

Steady high magnetic fields were generated by hybrid magnets, HM1b (27T) and HM2 (23T), at Institute for Materials Research, Tohoku University. The oscillatory part of the magnetization corresponding to the dHvA effect was extracted by a modulation technique (see Fig. 1). An alternating field of up to 20 $mT_{p\text{-}p}$, which is monitored by the H-coil, is generated in the modulation coil using a high power amplifier. This modulation field is sufficiently larger than the ac-ripple of about 2 mT in the magnetic field. In the case where an altanating field is produced in a large magnetic field, an electromagnetic force shakes the modulation coil and the pick-up coil may induce a large spurious signal. Cu rings are attached at both sides of the bobbin to prevent the vibration of the modulation coil with the reluctant force due to the eddy current in the rings. The M-coil for detecting oscillatory magnetization is composed of a 1000-turn inner coil and an approximately 600-turn compensation coil. Four samples can be measured simultaneously in one sweep of the magnetic field by employing four M-coils and four lock-in amplifiers. Since the modulation is small, we used fundermental frequency as the reference of the lock-in amplifiers. The intensity of the magnetic field is derived from the currents in the resistive and the superconducting parts of the hybrid magnet.

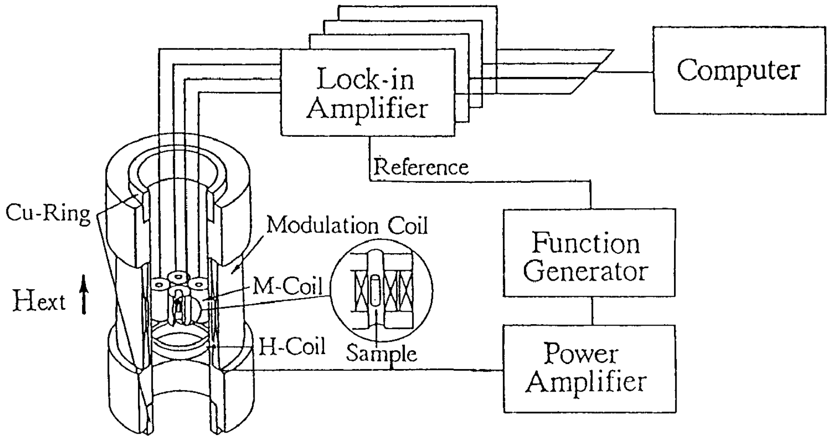

Fig. 1. Block diagram of the dHvA measuring system. The hybrid magnet and cryostat are omitted in the figure.

Temperature of the sample was varied by controlling vapor pressure of helium as shown in Fig. 2. Liquid helium is continuously introduced in a sample chamber through a thin capillary tube. The samples are soaked in liquid helium directly. In our system, the temperature can be changed from 1.6 to 4.3 K.

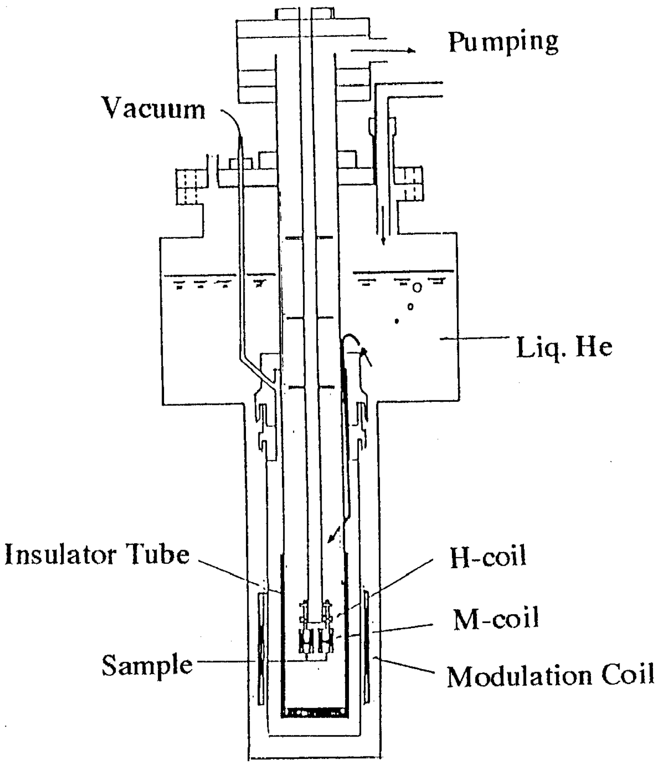

Fig. 2. Cross-section of the cryostat.

$YBa_2Cu_3O_7$ samples were made by conventional sinter technique at Osaka University. The powder was ground into fine crystallites with diameters smaller than 20 μm. The c-axes of the crystallites were aligned by a 5 T field during the hardening process of a mixture of the crystallites and epoxy resin. Several samples were prepared with different sintering process. $Tl_2Ba_2CuO_{6+\delta}$ powder with Tc=20 K was prepared by the high-Tc-group of NEC Corporation. Samples were fabricated by sintering raw materials in oxygen atmosphere and by annealing in argon for 3-5 hours [8]. The magnetic orientation of the c-axis was carried out using a 15T water-cooled magnet.

Single crystals of rare-earth monopnictide, SmSb, PrSb, TmSb, were grown by a Bridgman method. Pieces of rare-earth and antimony were sealed in a tungsten crucibles using an electron beam gun. The crucible was heated up to 2000°C by a high-frequency induction furnace and then was lowered slowly in the induction coil [9]. Samples for the measurements were made by cleaving the crystals.

III. Examples of dHvA Measurements

III. a Cuprate oxides

Figure 3 shows the best FFT spectra of the $YBa_2Cu_3O_7$ samples at different temperatures. A broad band appears around 500T in the ac susceptibility even at 3.1K. We have evaluated the effective mass ratio to be (2.1±0.5) by analyzing the dependence of spectral amplitude on the temperature [3].

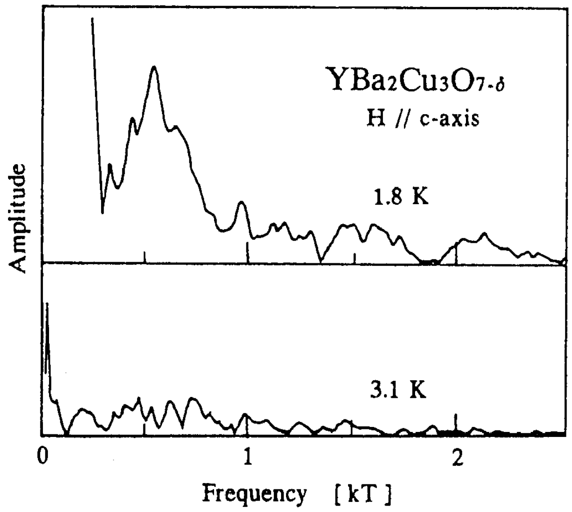

Fig. 3. FFT spectra of $YBa_2Cu_3O_7$ at 1.8 and 3.1 K.

In the FFT analysis, the most smooth curve was obtained by using the data between 10 and 23T. Approximately 30 periods are included in this field range. In general, FFT spectral analysis requires several tens of oscillations. However, the oscillatory amplitude strongly depends on the magnetic field intensity. A spectral analysis based on the data in the narrow magnetic field range is appropriate to determine the oscillatory frequency. We have carried out the waveform analysis by means of the maximum entropy method (MEM). Figure 4 shows MEM spectra transformed from the data in the range of 18 to 21T, where a sharp peak is seen around 460T. Since the experiments were carried out in superconducting state, the spurious signal due to the movement of pinned flux is much larger than signal from dHvA effect. Therefore, observed oscillation frequency somewhat scattered in the different experiments. The average frequency of 4 different samples at various conditions was determined to be 456±35 T [5]. This frequency corresponds to the extremal cross section of 0.044±0.003 A^{-2}, which agrees quite well with the theoretical value (0.042 A^{-2}) of the columnar Fermi surface around the S-point containing CuO chain and CuO_2 layer characters [10, 11]. The dHvA effect of $YBa_2Cu_3O_{6.97}$ powder was also measured by a Los Alamos group [12, 13]. They employed an explosive driven flux compression technique to generate a megagauss field in a very short duration. In their Fourier transformation spectra, the spectral density has the maximum at 530T which is essentially same to our result.

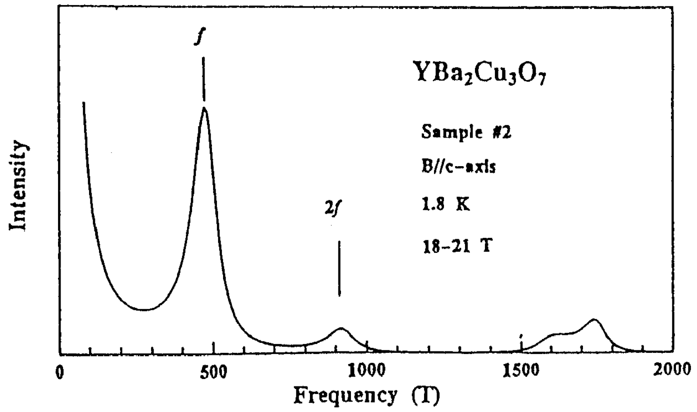

Fig. 4. Spectral density of MEM analysis in $YBa_2Cu_3O_7$.

The magnetic ac-susceptibility and the MEM spectrum in $Tl_2Ba_2CuO_{6+\delta}$ are shown in Fig. 5. The MEM analysis was made based on the data between 20 and 23 T where the dHvA oscillation is clearly seen in the susceptibility trace. The spectral density peaks at 400 T. In the energy-band diagram of $Tl_2Ba_2CuO_{6+\delta}$, a small electron ellipsoid is realized at the gamma point [14]. What we observed may correspond to this surface. In addition, the critical field of present sample was estimated to be approximately 20 T by the magnetization measurement.

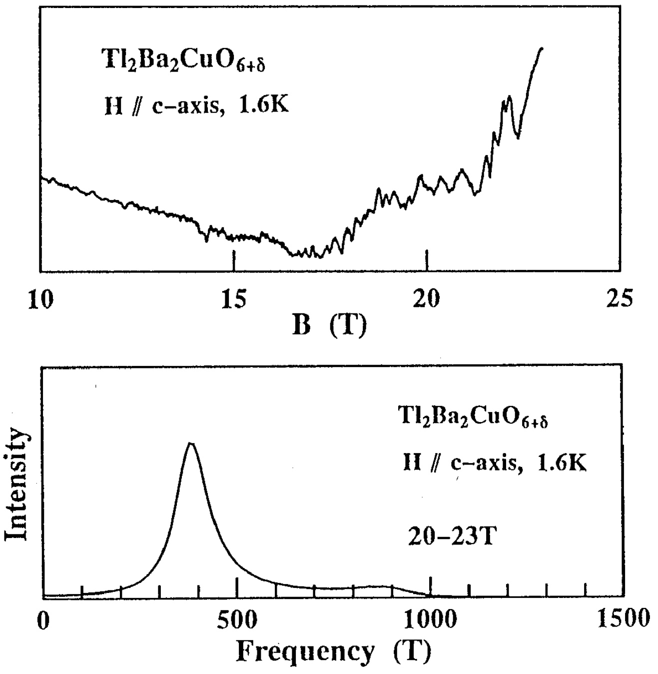

Fig. 5. Magnetic ac-susceptibility and the MEM spectrum in $Tl_2Ba_2CuO_{6+\delta}$.

III. b Rare-earth monopnictide

SmSb orders magnetically at 2.1 K and shows antiferromagnetism. Figure 6 shows the oscillatory ac-susceptibility and its Fourier transform spectrum. The peak α is related to the electron sheet at the X-point and β to the hole orbit at the Γ-point. The angular dependence of the extremal cross-sections has been studied precisely using the 10T magnet [15]. In SmSb, a similar shape of the Fermi surface of LaSb is realized with size 30% larger. We measured the dHvA effect at temperatures below and above T_N. No shift was found in the peaks α and β. So that, the Fermi surface does not change at the ordering temperature.

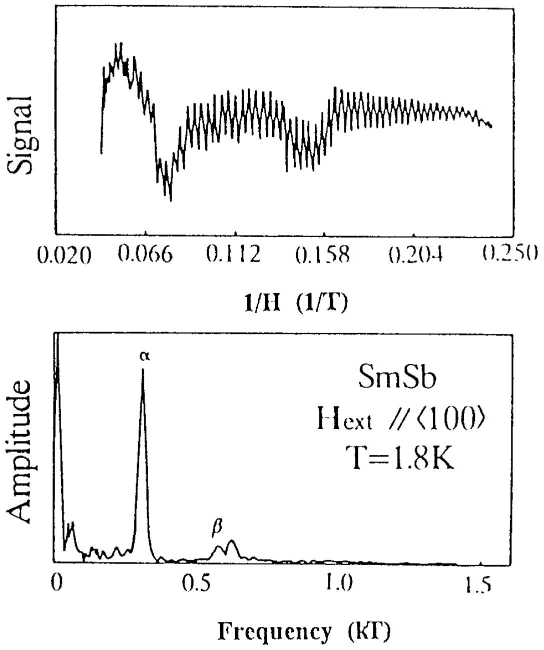

Fig. 6. The dHvA oscillation and its Fourier spectra in SmSb.

A non-magnetic singlet ground state is realized in rare earth pnictide PrSb due to the octahedral crystal field in the NaCl-type structure. The shape of Fermi surface of this material is similar to that of LaSb. When a high magnetic field is applied, a change of Fermi surface is expected because of the mixing of excited states to the ground state. However, the variation has not been detected in PrSb up to 10 T [16]. Up to now, the dHvA experiments in much higher field has been desired. Figure 7 shows the field dependence of the MEM spectral amplitude of PrSb at 1.6 K along the [111] direction. It was found that the α branch shifts to the higher frequency side by 5% and the γ branch shifts to the lower frequency side by 2 % at the high magnetic field range [6].

Much larger variation of Fermi surface may be expected in TmSb, because the magnetization of TmSb tends to saturate above 10T while that of PrSb increases continuously above 10 T. Recently, we succeeded for the first time to grow a high quality single crystal of TmSb and have

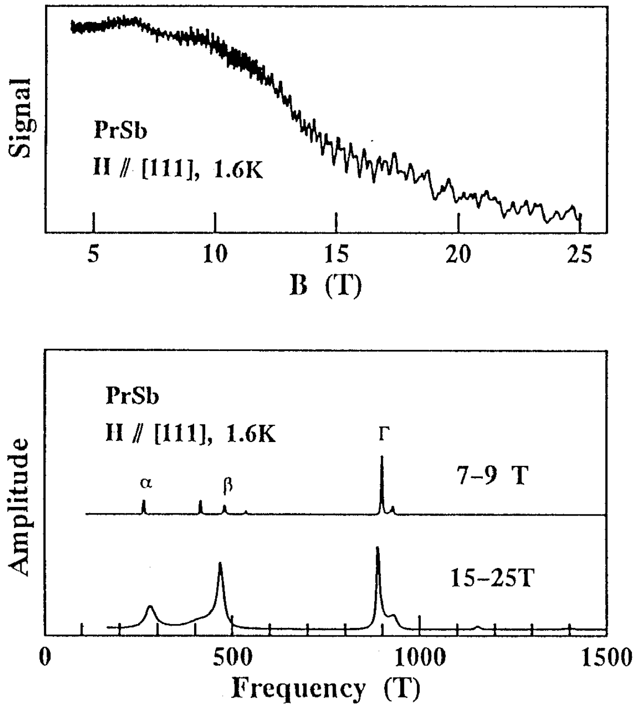

Fig. 7. The dHvA signal and spectral density of PrSb.

measured dHvA effect up to 23 T. Figure 8 shows the dHvA signal and the MEM spectral density of TmSb at 3.0 K along the [100] direction, where the label a is identical to the LaSb. We found a remarkably large spin splitting for a branch, which has never been seen in these family materials before.

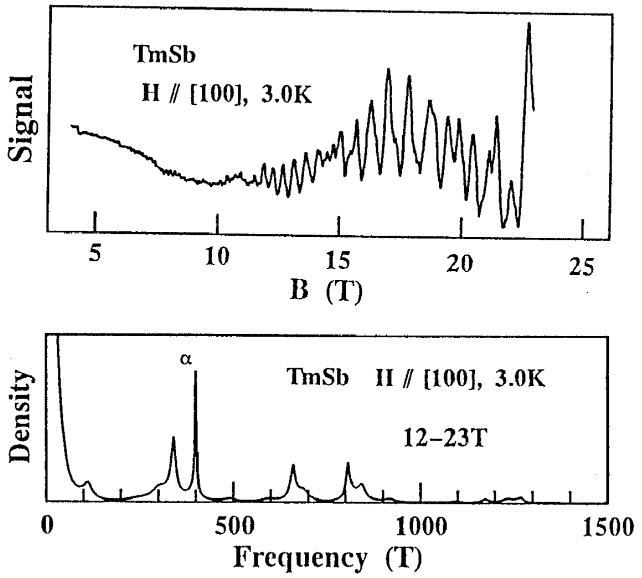

Fig. 8. The dHvA signal and spectral density of TmSb.

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