Magnetoresistance studies of spin-slip transitions in single crystal holmium

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Abstract

Previous studies of single crystal holmium using a variety of techniques including thermal expansion, magnetization, and X-ray and neutron scattering have confirmed the presence of"spin-slips" below the antiferromagnetic ordering temperature $T_N = 132$ K. Magnetization versus temperature studies taken below T_N showed several anomalies which occur above the ferromagnetic ordering temperature $T_c = 13$ K. These anomalies correspond to re-alignments of the moments within the magnetic unit cell as described in the spin-slip model developed by Bohr *et al.* Since magnetoresistance is sensitive to subtle changes in the magnetic structure, in this report we have studied the *H-T* phase diagram for applied fields along the b-axis of holmium up to 5.5 T and temperatures between 5 and 125 K using magnetoresistance (MR). Anomalies in the MR data correspond well with published transitions. Transitions are observed as either step increases or inflection points in magnetoresistance as the field is increased at a given temperature. Our data confirm previously published phase diagrams determined by magnetization and neutron diffraction.

1. Introduction

The first detailed studies of the magnetic structure versus temperature of holmium were carried out by Koehler *et al.* [1] using neutron diffraction. In these studies, holmium was found to be paramagnetic above $T_{\rm N}$ = 132 K. Below this temperature, the magnetic structure is an antiferromagnetic basal plane spiral in which the moments in a given plane are aligned along one of the six b-axis directions of the hexagonal close-packed structure of Ho. The magnetic spiral wavevector q_m was found to decrease with temperature. Below 20 K, a small c-axis moment was measured indicating a conical, ferromagnetic structure and q_m locked-in at a commensurate value of $0.167 = 1/6$ (in units of c^*). In this phase, the spiral makes one complete revolution every six unit cells, or 12 layers. Gibbs *et al.* [2] found other commensurate phases stable in the temperature region between 20 and 130 K. These phases have $q_m = 2/11$, 5/27 and 2/9 for lock-in temperatures of 20, 24 and 42 K, respectively. Bohr *et al.* [3] proposed a model to explain the lock-in behavior in which unpaired ferromagnetic planes are introduced. These planes are called "spin-slips". For example, if in the unit cell one out of every eleven planes is unpaired, then the spiral wavevector $q_m = 2/11$. Cowley *et al.* [4] carried out neutron scattering and ultrasonic attenuation studies which both confirmed and refined the spin-slip model. They also found an additional magnetic anomaly at 98 K which corresponds to a q_m lock-in value of 1/4 and an average spin-slip spacing of two layers.

In previous work [5], we have reported the results of magnetization versus temperature measurements of a Ho single crystal along the $a-$, $b-$ and c -axes. We observed anomalies in the magnetization data which we found corresponded well with the neutron diffraction and ultrasonic velocity results of Cowley *et al.* [4]. These anomalies were used to construct *H-T* phase diagrams for fields applied along each of the three axes.

In this report, we present data from magnetoresistance (MR) experiments performed on a single crystal of Ho for a magnetic field and current applied parallel to the b-axis. Previous MR studies [6,7] on Ho were carried out only at low temperatures and low fields. One of these studies [6] showed anomalies in the 40-80 K temperature range. We observed similar anomalies in our data and are able to confirm that these anomalies correspond to previously observed spin-slip and other magnetic structural transitions. We have used these anomalies to construct an *H-T* phase diagram.

2. Experimental details

The single crystal Ho sample in the shape of a long bar of mass 0.1364 g was prepared at Ames Laboratory.

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The dimensions of the sample were $1.15 \times 13.86 \times$ 1.0 mm³ along the a -, b - and c -axes, respectively. Four thin copper wires were spot welded to the sample to insure low contact resistance for the four-probe measurement. The sample was mounted at the end of a long probe such that the current direction would be parallel with the applied field. The sample leads were anchored to prevent movement in the magnetic field. The probe was then inserted into the sample chamber of a Quantum Design SQUID magnetometer and zerofield cooled. For the magnetoresistance measurements, only the 5.5 T superconducting magnet and temperature control system of the magnetometer were used. A 10 mA dc current was provided by a Keithley 220 current source and the voltage was measured using a Keithley 181 nanovoltmeter.

The magnetoresistance measurements were carried out as follows. The sample was cooled to the desired temperature under zero field and was allowed to reach thermal and electrical equilibrium. A field was applied from 0 to 5.5 T in steps of 0.01–0.05 T. After a given field was stabilized, three measurements of the sample voltage were made and averaged. The current was removed in between measurements to minimize sample heating. Under zero field, the sample was heated to 150 K (well above the Néel temperature, T_N) before beginning the next measurement cycle.

3. Results and discussion

Magnetoresistance (MR) data were collected for the following temperatures: 18, 22, 39, 45, 60, 70, 75, 85, 95, 103, 110 and 125 K. Figure 1 presents five of these data sets which are considered to be representative. Data are plotted as a percent change in resistance relative to the zero-field value, $\Delta R(H)/R(0)$, versus the applied field. MR data taken at 39 K are shown in the lower part of the figure. The vertical scale has been expanded to show the inflection point anomalies which are indicated by filled arrows. Similar inflection point anomalies were observed in the 18 and 22 K data. The inset shows the midpoint of a transition occurring near 1 T. For temperatures $T>45$, step-like increases in resistance are observed. The midpoints of these transitions are indicated by either filled or open arrows for the corresponding data points plotted. As data were taken at higher temperatures, these step-like transitions occur at higher fields. As the applied field is increased further, the change in resistance achieves a maximum before another transition occurs. Then, the magnetoresistance gradually decreases. Arrows are used to indicate the midpoints of these transitions for the 45 and 70 K data. These two transitions, along with the one shown in the inset for the 39 K data, are believed

Fig. 1. Magnetoresistance data are plotted as the percent change in resistance as a function of applied field at constant temperature. Arrows indicate the midpoints of step-like transitions and inflection points which are found to correspond to "spin-slip" or other magnetic structural transitions as described in the text.

to be due to the transition from the spiral antiferromagnetic to the conical ferromagnetic structure.

Figure 2 plots the positions of anomalies in MR data as filled circles. The solid lines represent the phase diagram determined from our previously published magnetization measurements [5]. In that study, magnetization was measured as a function of temperature with a constant field applied along the b-axis. Figure 3 shows magnetization data for four fields ranging from 0.01 T to 3T. The anomalies, seen as a small rise in magnetization, are indicated by arrows. The data collected by magnetoresistance correspond well with our phase diagram determined by magnetization even though the measurements were performed on two different single crystals.

The dashed lines in Fig. 3 represent the phase diagram determined by the neutron diffraction studies in fields up to 2 T and for temperatures below 70 K of Jahan *et al.* [8]. We now wish to combine the results from refs. 4, 5, 8 and 9 to label the various phase lines and regions of the *H-T* phase diagram. Jahan *et al.* [8] found region I to be primarily the $q_m = 1/6$ commensurate phase, region II to be a mixture of the 1/6 phase and the 2/11 spin-slip structure, region 1II to be a mixture of the ferromagnetic phase and a helifan structure

Fig. 2. The phase **diagram of holmium for magnetic fields** H (T) **applied along the** b-axis as a **function of temperature** T (K). The filled **circles are the positions of the anomalies found in magnetoresistance measurements.** These data **are compared with published** phase diagrams determined by **neutron diffraction** (dashed lines), magnetization (solid lines) and mean-field calculations. The labels **for the phase regions and lines are defined in the text.**

Fig. 3. Anomalies **in magnetization** data as a **function of** temperature such as **those indicated by arrows were used to determine the solid phase lines shown in** Fig. 2. The anomalies **shown here correspond to a spin-slip transition with a lock-in wavevector** $q_m = 2/9$. This anomaly moves towards higher temperature as the **field is increased.**

(predicted by ref. 9), region IV to be a mixture of the spiral and helifan phases, and region V to be a spiral phase with $0.195 < q_m \le 0.25$. Jensen *et al.* [9] carried **out mean-field calculations which predict the presence of a fan structure in region VI. In region VII, Koehler** *et al.* **[1] observed the spiral antiferromagnetic structure and found that qm varies from 0.28 to 0.25 with decreasing**

temperature. The MR data points plotted in the region above the line marked "a", are points which were previously described as inflection points in the data. The inflection points observed for fields below 2 T seem to correspond well with the (dashed) phase lines determined by Jahan *et al.* **[8]. Inflection points in this region above 2 T may indicate the presence of other magnetic structural changes and the need for further investigation.**

The solid lines labeled a-f determined from magnetization data [5] correspond to wavevector lock-in values as follows: line a (below the dashed line), 1/6; b, 2/11; c, 5/27; d, 2/9; e, 1/4. Line f is the paramagnetic-antiferromagnetic transition and is observed to split into two lines above 1 T. The magnetic structure in the region of the phase diagram above this split has not been determined.

In this paper, we have reported results of magnetoresistance studies on single crystal holmium for magnetic fields ranging from 0 to 5.5 T applied along the b-axis and temperatures between 18 and 125 K. Observed anomalies in the MR data correspond well with those observed previously in magnetization, neutron diffraction and other studies. We have confirmed many of the phase boundaries in the *H-T* **phase diagram and have pointed out the areas that require further study.**

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