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NEUTRON DIFFRACTION STUDY OF THE *p–T* PHASE DIAGRAM FOR ERBIUM

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Electrical resistivity measurements performed on a single crystal of erbium as a function of temperature and hydrostatic pressure have provided a preliminary *p–T* phase diagram. The results have been interpreted in terms of a model for the magnetic structures of Er deduced from neutron diffraction studies at ambient pressure. This model predicted the existence of a magnetic structure with a wave vector of $Q = 2/7\,c^*$ at 4.2 K, when the applied pressure is larger than 3 kbar. This paper reports a neutron diffraction study of erbium made in the temperature range of 4 to 100 K, at pressures between 0.5 and 6 kbar. We have observed the predicted suppression of the low temperature conical ferromagnetic phase and the emergence of a new phase with $Q = 8/33\,c^*$. The neutron diffraction measurements has enabled us to identify the various phases that develop from the cycloidal phases previously observed at atmospheric pressure.

Keywords: Rare-earth; Pressure; Neutron; Diffraction; Magnetic structures; Phase-diagram

INTRODUCTION

In recent years erbium has attracted considerable attention due to its complex magnetic phase diagram. One of many studies has been the neutron diffraction investigation of erbium in a *c*-axis field by McMorrow *et al.* [1]. These studies reveal three basic types of magnetic structure. Below $T_C = 18$ K the structure is described as *conical* with moments ordered antiferromagnetically in the basal plane and ferromagnetically parallel to the *c*-axis. The ordering wave vector in this phase is $Q = 5/21\,c^*$, where c^* is the reciprocal lattice vector parallel to the *c*-axis [1]. For temperatures between $T = 18$ and 52 K the moments are constrained to the *a*-*c* plane and ordered antiferromagnetically along both axes. This elliptically polarized *cycloidal structure* locks to the lattice period in a number of commensurable phases; the reader is referred to [1] and references therein. Between $T = 52$ and 85 K the magnetic structure is *c-axis modulated* (CAM), with the moments constrained to the *c*-axis and the size of the moment varying sinusoidally from one basal plane to the next. The ordering wave vector in this phase is close to $Q = 2/7\,c^*$. An early magnetostriction study by Rhyne and Legvold [2] revealed sharp changes in the values of the strain parameters at T_C. Using these strain para-

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meters, Jensen and Mackintosh [3] predicted that the magnetoelastic energy would cause the cone structure to become unstable when a hydrostatic pressure of approximately 2.5 kbar is applied. We have earlier investigated the form of the *p–T* phase diagram of erbium using electrical resistivity measurements [4]. In this paper we present, results for the ordering wave vector as function of hydrostatic pressure derived from neutron defraction studies.

EXPERIMENTAL

A single crystal cylinder of Er (0.367 g) was spark-cut with its axis parallel to the real space *b*-axis and mounted on a vanadium pin using a low temperature glue. The pin was secured to a Bridgman seal composed of two lead seals with a copper support on a maraging steel obdurater. This assembly was then inserted into a Ti–Zr helium pressure cell. The cell was attached to the centre stick of an Orange cryostat and initially pressurised at 1.5 kbar using helium, whilst at room temperature, in order to form the seal. Thermometers were attached at both ends of the cell with the heater close to the entry point of the helium capillary to prevent blockage during cooling. The pressure was determined using a pressure transducer attached to the top of the centre stick allowing a constant monitor of the pressure as the sample assembly was cooled to the required temperature. The cooling rate for the cell through the liquidus/solidus phase line of helium was approximately 0.02 K/s to ensure that the helium solidified evenly throughout the cell.

The cryostat was mounted on the PRISMA neutron spectrometer at the ISIS Facility, UK, with measurements being made in the *a*-*c* horizontal scattering plane. The collimation before the detector was $30':30'$. Erbium has a large neutron absorption cross section and therefore PRISMA was configured in an elastic mode with the detector bank centred at $-40°$, such that the energies of the neutrons diffracted from the (1 1 0) and (0 0 2) nuclear Bragg peaks were 55 meV (1.2 Å) and 22 meV (1.9 Å) respectively. At these energies the absorption cross section is approximately 120 barns.

RESULTS AND DISCUSSION

Figure 1a shows the electrical resistivity as a function of pressure at $T = 4.5$ K (filled circles) previously reported in [4]. The resistivity exhibits a rapid increase between 0.5 and 3.5 kbar, with some suggestion of an intermediate phase. The solid line is the predicted resistivity based upon our model [4]. For pressures greater than 3.5 kbar the magnetic structure was predicted to have a wave vector of $Q = 2/7\,c^*$. In addition the model predicted the suppression of the cone phase and the emergence of a structure described as *tilted cycloidal.* This new structure is characterised by a small oscillating component parallel to the real space *b*-axis and no ferromagnetic moment parallel to the *c*-axis. Figure 1b presents the results of scans made along [0,0,*L*] in order to determine the ordering wave vectors at various pressures. We include the results of our experiments performed on PRISMA together with results reported by Kawano *et al.* [5]. Our results from PRISMA demonstrate self-consistency in two experiments, and reveal only a modest change in the wave vector at the transition shown in Figure 1a between 0.5 and 3.5 kbar. The wave vector does not approach the predicted value of $Q = 0.286\,c^*$ ($2/7\,c^*$) in [4] but instead adopts a value of $Q = 0.242 \pm 0.001\,c^*$. This latter value is equivalent to a commensurate wave vector of $8/33\,c^*$: this structure persists until 4.8 kbar where Q is seen to increase rapidly. Since the ferromagnetic moment in erbium is parallel to the *c*-axis scans parallel to [0,0,*L*] cannot detect changes in this component of

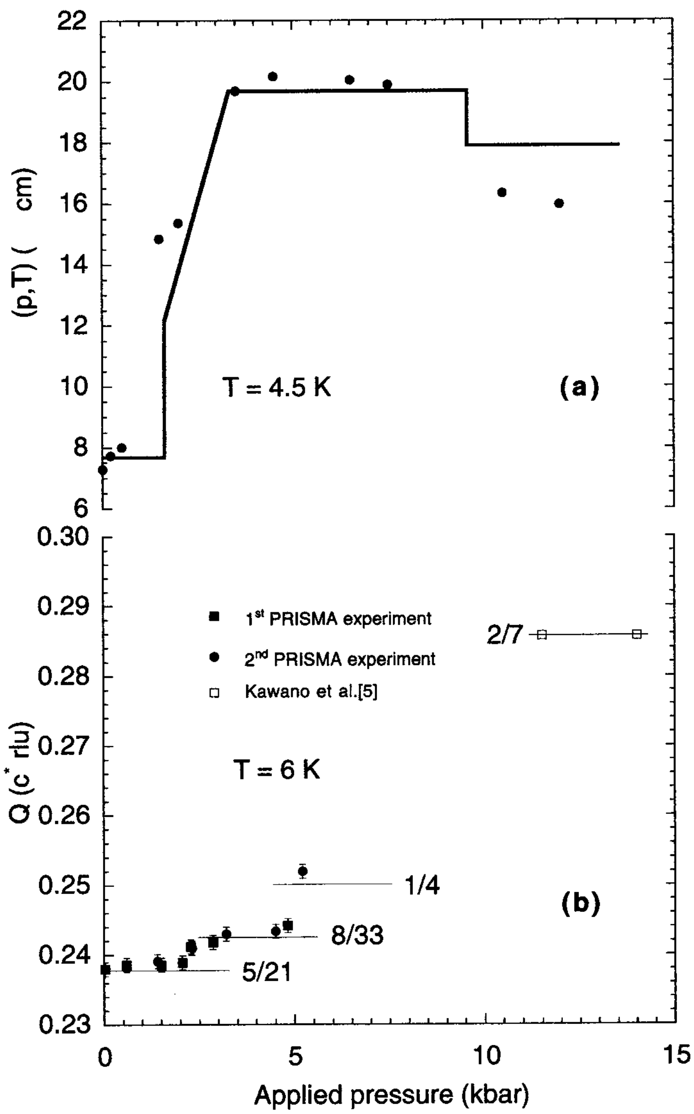

FIGURE 1 (a) The electrical resistivity at $T = 4.5$ K measured for increasing pressure (solid circles) plotted with calculated values [4] (solid line). (b) Shows the pressure dependence of the wave vector Q at $T = 6$ K measured in this study (filled symbols) and previous measurements [5] (open squares). The horizontal lines with fractions indicate the positions of commensurate magnetic structures.

the structure. Hence, we performed scans along the [1,1,L] direction (not shown) where the ferromagnetic structure factor for the c-axis is finite. These measurements showed that the ferromagnetic moment persists for pressures up to 4.8 kbar, whilst at 5.2 kbar the ferromagnetic moment collapses dramatically; at this pressure a small residual moment was observed at the (1,1,0) reciprocal lattice point.

Figure 2 presents the p–T phase diagram of erbium. Filled circles represent points derived from the resistivity data, and the thick solid and dashed lines represent trends described in the earlier study [4]. Our new investigation on PRISMA was focused on the temperature region below $T = 30$ K, where we have mapped the boundaries of the phases we detected. The thin

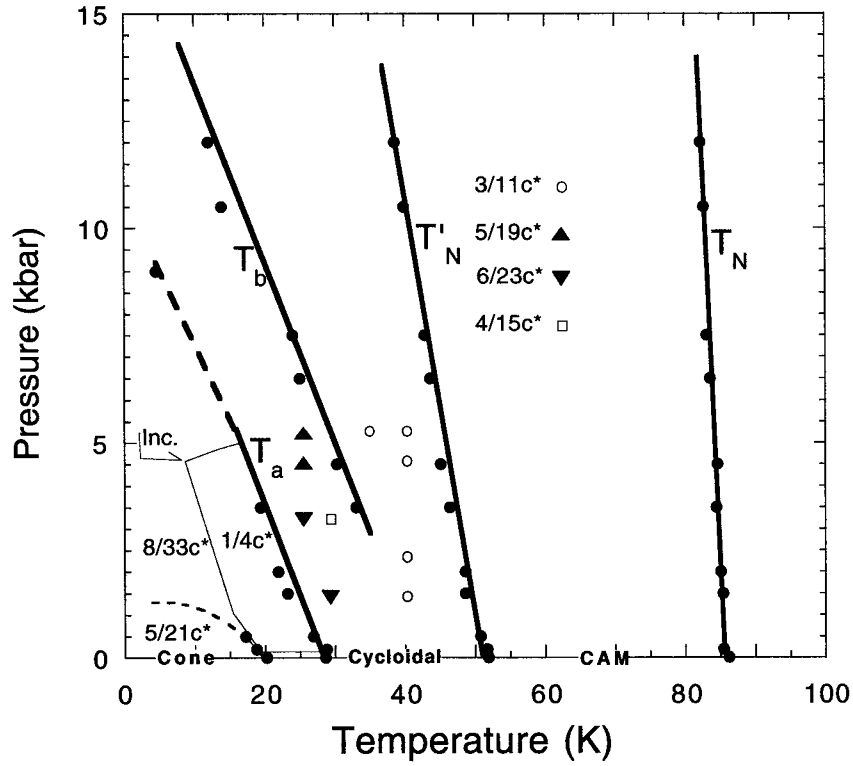

FIGURE 2 The *p–T* phase diagram for erbium. The nomenclature is described within the main text.

lines are approximate guides that require further verification. The commensurable value of the ordering wave vector $8/33\,c^*$ may occur both in the cone (an effective 11 layer period of the helix) and in the cycloidal (the 44444445-structure) phases. However, our neutron diffraction experiments indicate that the value of $Q = 8/33\,c^*$ is connected to the cone, and that the phase transition from the cone to the cycloid is accompanied by a change of the wave vector from this value to $Q = 1/4\,c^*$.

Our results show that at 6 K, the cone phase is not destroyed until a pressure of about 5 kbar is applied. In comparison, the resistivity measurements (see Fig. 1a) indicate that the first-order transition between the cone and the cycloidal structure in this sample is smeared out between 0.5 and 3.5 kbar. It is important to note that the total jump in the resistivity is twice as large as that observed at T_C at ambient pressure, which was attributed to a major pressure-induced change of the ordering wave vector [4]. However, our present results demonstrate that this does not occur. An alternative explanation is that the Fermi-surface topology becomes modified due to changes in the lattice constants. Analysis of the lattice parameters as a function of pressure at $T = 6$ K reveals an overall decrease in the c-axis parameter by approximately 0.5% between 1 bar and 3.2 kbar. The strain-induced modification of the Fermi surface then implies that the reduction of the superzone effect, present at ambient pressure and proportional to the change of the wave vector away from $2/7\,c^*$, no longer occurs. In other words, at ambient pressure a large number of the superzone boundaries lie close to the Fermi surface, whereas when the applied pressure exceeds about 3 kbar the boundaries lie at sufficiently large distances away from the tangential planes to the Fermi surface, so that the decrease of the wave vector no longer has the effect of reducing the number of energy gaps at the Fermi surface.

In summary, we have determined that changes in the low temperature resistivity of erbium are due to changes of the lattice parameters as well as the changes in the magnetic structure and this has provided an important reassessment of the model used earlier [4]. These studies have also provided important information on the magnetoelastic properties of erbium. Further experiments are planned to investigate the phase diagram at pressures above 6 kbar and establish the nature of the phase boundaries marked T_a and T_b in Figure 2.

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