The Observation of Magnetic Domain Structures of the Intermetallic Compounds $SmCo_5$, $LaCo_5$ and $CeCo_5$

In general the observation of magnetic domain structures in metals and alloys by either the Bitter [1] or Kerr [2] method requires very careful preparation of the specimen. During mechanical polishing the surface of the specimen undergoes severe plastic deformation so that for strain-free equilibrium domains this surface layer must be removed. This is usually accomplished by electropolishing – a technique which has been used with considerable success for the ferromagnetic elements iron, nickel and cobalt and certain alloys, notably 3 to 4% silicon-iron [3]. Mechanically polished specimens give rise to the well known "maze" patterns [4].

The hexagonal lanthanide-cobalt intermetallic compounds, LCo_5, with the $CaCu_5$ structure (space group P6/mmm) have recently been shown to possess many interesting magnetic properties [5, 6]. In the case of $SmCo_5$ coercivities ~ 5000 to 10000 Oe or more can be produced relatively easily by grinding in air and subsequent alignment in paraffin wax by a field ~ 20 kOe. These extremely high coercivities have been attributed to the enormous uniaxial magnetocrystalline anisotropy field ~ 300 kOe [7], but a detailed theory of the mechanism of magnetisation reversal has yet to be presented.

The alloys used in the present work were prepared by melting very pure oxygen free samarium, lanthanum and cerium with cobalt in an induction furnace. They were supplied by Metals Research Limited, Melbourn, Cambridgeshire. The $SmCo_5$ and $CeCo_5$ were shown by X-ray diffraction analysis to be single phase in the as-cast state with the hexagonal $CaCu_5$ structure. The $LaCo_5$ required homogenisation for 4 days at 1000°C before the $LaCo_5$ single phase was obtained.

The intermetallic compounds are very hard and brittle at room temperature so that relatively little plastic deformation will be introduced during mechanical polishing (the specimens were polished down to 1 μm diamond paste). Thus, clearly defined domain structures can be observed without electro-polishing. The domain structures shown in figs. 1 to 7 were obtained by the Bitter technique using colloidal magnetite prepared by Elmore's method [8]. All the structures are typical of those for strongly uniaxial materials. The specimens were all polycrystalline and in general only one domain structure was observed on each grain.

The domain structure of the as-cast $SmCo_5$ shown in fig. 1 is typical of those on (0001) planes for hexagonal materials. Fig. 2 shows 180° wall structures on planes perpendicular to the (0001) plane, together with structures on planes which make angles between 0 and 180° with the hexagonal axis. Fig. 3 shows the structure on a specimen of $SmCo_5$ which was annealed for 4 days at 950°C, from which it appears that there is some preferential grain growth since nearly all the domain structures are typical of those to be expected for planes parallel to, or making a small angle with, the hexagonal axis. The structures for $LaCo_5$ shown in figs. 4 and 5 show typical 180° wall structures on planes which are parallel to, or make small angles with, the hexagonal axis. Several spikes of reverse magnetisation have been nucleated at the grain boundaries. Grain A of fig. 5 is probably orientated at an appreciable angle to the hexagonal axis since the domain structure is intermediate between those observed parallel and

Figure 1 Domain structure on cast $SmCo_5$ (× 260). Coercivity 550 Oe.

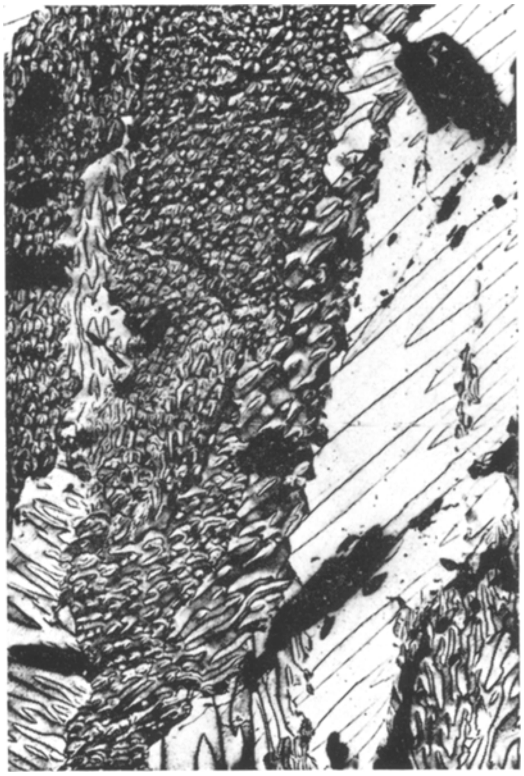

Figure 2 Domain structure on cast $SmCo_5$ (× 133). Coercivity 550 Oe.

Figure 3 Domain structure on annealed $SmCo_5$ (× 170). Coercivity 550 Oe.

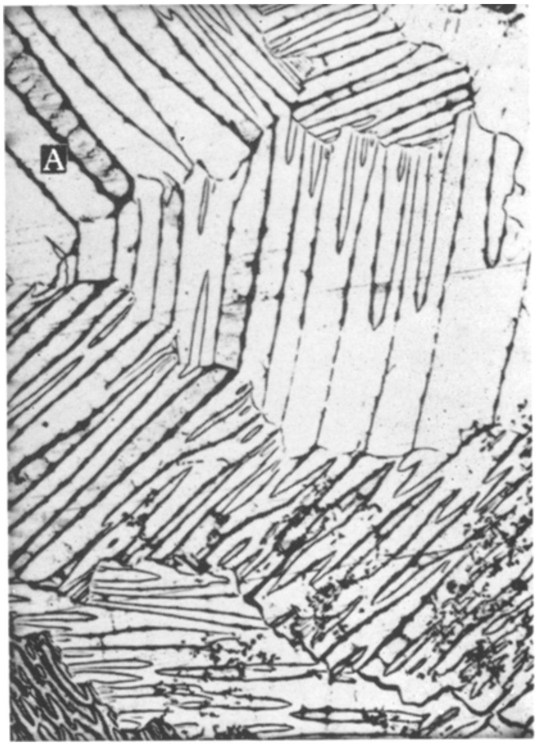

Figure 4 Domain structure on homogenised $LaCo_5$ (× 170). Coercivity <200 Oe.

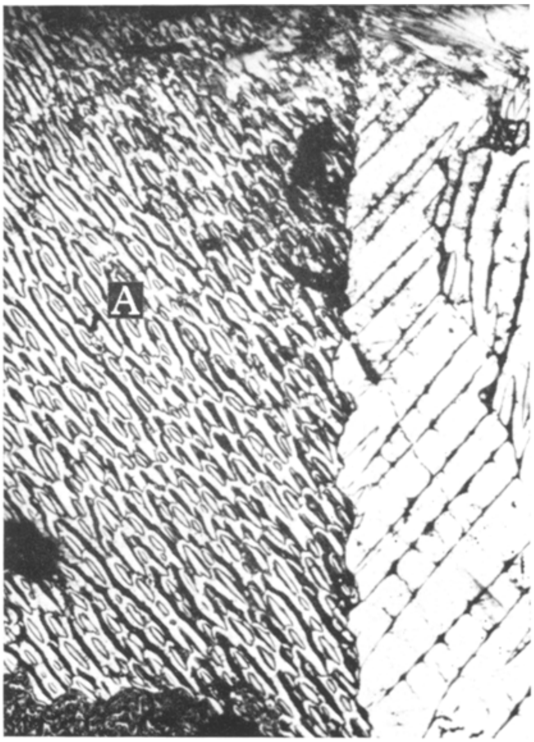

Figure 5 Domain structure on homogenised $LaCo_5$ (× 170). Coercivity <200 Oe.

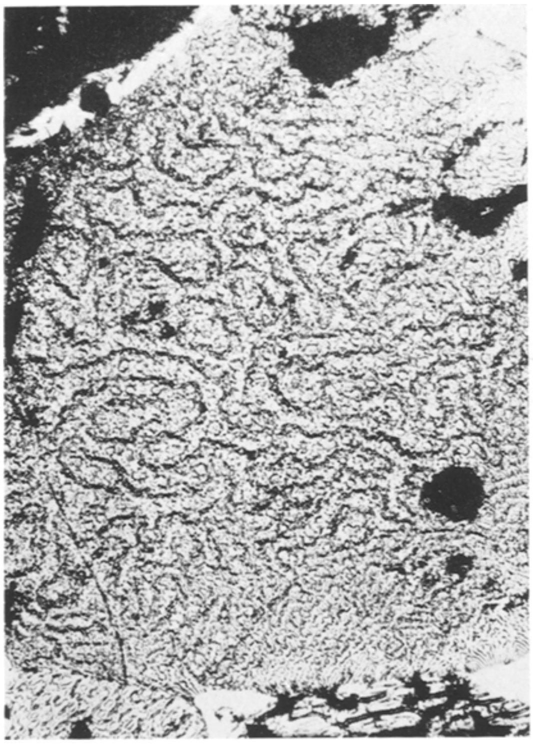

Figure 6 Domain structure on homogenised $LaCo_5$ (× 170). Coercivity <200 Oe.

Figure 7 Domain structure on cast $CeCo_5$ (× 170). Coercivity <200 Oe.

perpendicular to the (0001) plane. The finely divided structure shown in fig. 6 will almost certainly be on an (0001) plane.

It is interesting to note that in fig. 4 there is a broadening effect on the 180° domain walls in the form of small peaks perpendicular to the direction of the wall. In area A, some of the peaks have almost joined to form a sub-domain structure in order, possibly, to reduce the magnetoelastic or magnetostatic energy associated with plastic deformation. Preliminary measurements of the Vickers microhardness show that for $LaCo_5$ there is a marked difference in microhardness on the (0001) plane and on planes perpendicular to this. The mean microhardness value on the (0001) plane was 440, but on planes parallel to the hexagonal axis the indentations were very anisotropic with mean values corresponding to microhardnesses of 180 and 620 in orthogonal directions. However, the latter effect is probably due to anisotropic recovery of the indentation and is often observed in hexagonal metals and alloys.

Preliminary hardness measurements on $SmCo_5$ and $CeCo_5$ gave values of 722 and 702 on the (0001) plane and 676 and 653 on planes perpendicular to this respectively. $SmCo_5$ and $CeCo_5$ are, therefore, appreciably harder than $LaCo_5$ and less anisotropic, so that plastic deformation should be correspondingly more difficult. Domain structures on the as-cast $CeCo_5$ are shown in fig. 7.

Since the grains on all the specimens were too small for their orientations to be conveniently determined by X-ray diffraction, the domain patterns provided a useful technique for the measurement of hardness as a function of orientation. More detailed measurements of Vickers and Knoop hardness are currently in progress.

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Dislocation Etching of GaS

The A^{III} B^{VI} compounds represent a new, interesting group of semiconductors of great photoconductivity. Their photo- and electroluminescence might usefully be studied at low temperatures. Most of them are *p*-type semiconductors whose charge carrier mobility is fairly low.

The *p*-type GaS is one of the most important compounds as well as the crystallographic basic model of the $A^{III}B^{VI}$ compounds. Its forbidden gap lies at 2.5 eV.

GaS crystallises in a hexagonal system. The crystals are built up of four-fold elementary layers of S-Ga-Ga-S [1, 2]. The Ga has a tetrahedral co-ordination in the layers, whereas the S atoms have a trigonal one. The presence of Ga-Ga metal bonds was proved by magnetic measurements [1].

The tetrahedral bonds of Ga sp^3 hybrids are formed by the s^2p and the *p* electrons of the sulphur. Within the four-fold layers the bonds are covalent, whereas the bonding between two adjacent layers is of van der Waals type.

Before it is possible to interpret the physical properties of the GaS its real crystal structure must be known. The lattice defects existing in GaS have not been studied in detail [3, 4]. The publications first of all dealt with the stacking faults since, due to the layer structure of the GaS crystal, many stacking faults might form by slipping and rotating. A defect structure similar to that of the graphite could be detected by transmission electron microscopy in GaS [5].

Since oxidising etching materials and hydrogen-halogenides had been successful for developing dislocations in II-VI compounds, hydrochloric acid, bromine-acetic acid mixture as well as aqueous chromous sulphuric acid were used as etchants for the GaS crystals.

The GaS crystals investigated were grown from Ga melt according to our previous publications [6]. Due to the high lateral solubility rate of the GaS treated with hydrochloric acid, only the lamellar crystal structure could be detected (fig. 1). Strong oxidising materials such as the acetic acid solution of bromine, which proved to be a good selective etchant for GaP [6] behaved in the same way as hydrochloric acid for GaS.

The etchant of medium oxidising effect described by Woods [7] (10 ml H_2SO_4, 10 g $K_2Cr_2O_7$, 70 ml H_2O) may also be used for GaS as a highly selective etching material. By applying it, two different kinds of etch pits could be observed on the (0001) planes of the GaS (fig. 2). One of them has a hexagonal pyramidal terraced shape, whereas the other shows conical character with indistinct hexagonal symmetry. The regular hexagonal etch pits occurred considerably more frequently than the other type. Etch pits appeared along low-angle grain boundaries that were perpendicular to the basal plane and parallel to $\langle 10\bar{1}0 \rangle$ and $\langle 1\bar{2}10 \rangle$ directions, respectively. Fig. 3 shows three low-angle grain boundaries running perpendicular to the basal plane.

Occasionally, evidence was seen of the movement of dislocations due to the low stresses that occurred during the etching process. This means that the dislocations have a considerable mobility at room temperature. Fig. 4 displays a dislocation shift of this kind. The great plane bottomed etch pit probably indicates the previous position of the dislocation marked by an arrow. Dislocations perpendicular to the lamellar structure form a new group of GaS crystal defects.

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