factor $J_s^4 K_1^{-3/2}$ by which F_s is multiplied increases, however, rapidly with temperature which probably explains the peculiar temperature dependence of the coercive field in Fig. 3. The temperature dependence of η cannot be deduced as η is dependent also on those causes which lead to H_{co}.

My thanks are due to Dr. A. H. Qureshi for informing me of the results of his experiments prior to publication and to Prof. Dr.-Ing. M. Kersten for many stimulating discussions.

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DISCUSSION

C. D. Graham: It seems to me that you have a serious experimental problem in checking your theory. How are the Cu precipitate particle diameters measured to three significant figures in the range of 1000Å, and are the precipitates uniform in size so that it is sufficient to use one value of R?

H. D. Dietze: The size of precipitate particle is measured by electron microscope. The precipitates are not uniform in size.

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The Electron Microscope Examination of Small Ferromagnetic Precipitates*

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Small particles of cobalt precipitated from a Au 1.5% Co alloy and from a Cu 2.4% Co alloy have been examined using the thin film electron microscope technique. In the gold alloy the precipitates form in disc-like arrays on {100} planes of the gold matrix. In the copper alloy the particles are thought to be roughly spherical. It is suggested that the high coercivities observed in the gold alloy cannot be explained satisfactorily in terms of particle shape and particle interaction alone, but that particle shape and particle interaction could provide the main contribution to the lower coercivity of the copper alloy.

Introduction

The shape, size and distribution of small ferromagnetic particles precipitated from non-ferromagnetic matrices are currently being examined using the thin film technique in order to correlate these observations with the magnetic properties of such materials.

Gold 1.5% Cobalt Alloy

Some results have already been reported on

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the shape of cobalt particles precipitated in the gold matrix of a water quenched gold 1.5% cobalt alloy.[1] It had been suggested[2], that the high coercivity observed in these alloys (2,050 oe) after appropriate ageing treatments, could be explained if the cobalt was precipitated as small single domain particles of rod-like form with the f.c.c. structure. The electron micrographs suggest, however, that the particles are disc-like in shape.

Thin foils of the alloy about 0.2 mm thick were prepared by cold-rolling specimens previously used in magnetic measurements. The foils were solution treated at 880°C and quenched in water or air. The foils were then aged for various times at 565°C to produce the desired amount of precipitate. Thin films suitable for transmission electron microscopy, using a Siemens Elmiskop 1, were prepared by electropolishing in a solution of concentrated hydrochloric acid.

As reported by Gaunt and Silcox[1], elongated holes A and other contrast effects B as shown in Fig. 1 are seen in foils prepared from water quenched foils aged for 5 minutes at 565°C. Since these are not seen in unaged water quenched foils (Fig. 2), they are thought to be caused by precipitates : the holes correspond to particles etched out of the foil and the other effects correspond to particles left within the foil. The first feature of note is that many of the particles appear to be aligned in rows. From an analysis of photographs from foils having normals as shown in the stereographic triangle of Fig. 3 it was

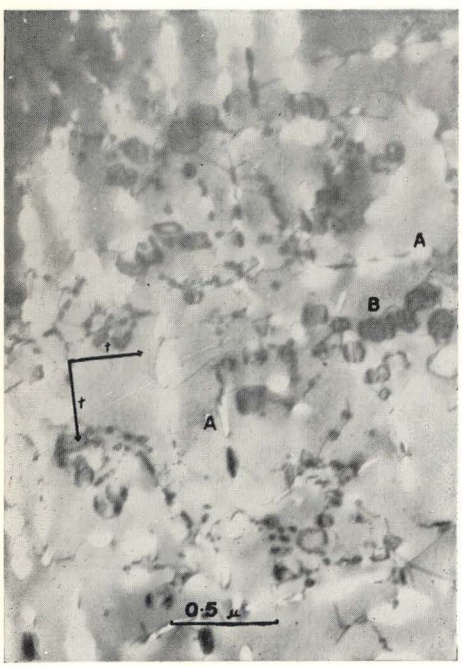

Fig. 1. Water quenched Au 1.5% Co alloy aged 5 mins. at 565°C. Foil normal near (100). The traces of {100} planes are marked *t*.

Fig. 2. Water quenched unaged Au 1.5% Co alloy.

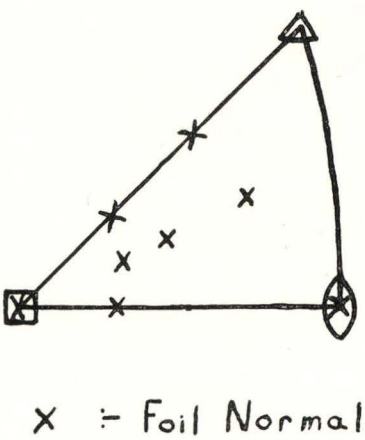

Fig. 3. Normals to Au 1.5% Co foils in which {100} plane traces were analysed.

found that the directions of the rows were always along the traces of {100} planes in the plane of foil. It was therefore concluded that the particles are clustered in the form of sheets in the {100} planes of the gold matrix, the disc-like contrast effects of Fig. 1 being due to sheets parallel to the foil. The determination of the shape of the individual precipitates is complicated by contrast effects presumably due to strain, but a similar analysis can be made on specimens in which the precipitates have not been etched out. These observations are fewer in number but suggest that the precipitates are discs on {100} planes. This is also supported by the fact that the direction of the elongation of the holes left by the etched out precipitates also lies along the trace of a {100} plane in the plane of the foil. Deviations from this model are observed but it is estimated that the amount of precipitate that could be in rod-like form is less than 5%.

The observations on the air quenched gold cobalt are similar qualitatively, (see Fig. 4), but differ primarily in that the precipitates have far fewer nucleation sites (as suggested by Gaunt[2]). Almost all the precipitate is clustered in the form of sheets, usually on {100} planes. Deviations from the sheets on {100} planes can be interpreted as being due to the presence of other suitable nucleation sites (*e.g.* grain boundaries and dislocations).

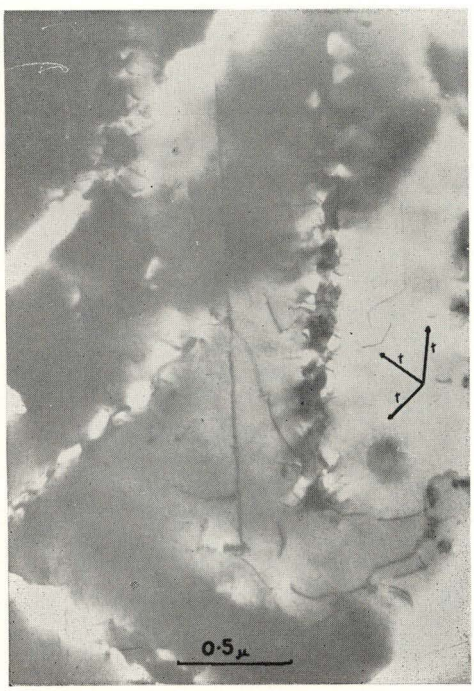

Fig. 4. Air quenched Au 1.5% Co alloy aged 10 mins. at 565°C. Foil normal near (112). The traces of {100} planes are marked *t*.

Recent work by Campbell and Muldawer[3] on an Au 5% Co alloy has been reported in which the observations on the shape and arrangement of the precipitates differ from those given in this paper. However, the present observations were made on a more dilute alloy and at higher ageing temperatures than those reported by Campbell and Muldawer and it is felt that serious disagreement may not exist.

The theory of Stoner and Wohlfarth[4] predicts that there will be no contribution to the coercivity of an array of small isolated ferromagnetic particles from those particles which are isotropic and disc-like in shape. Measurements of the remanent magnetization (Gaunt[2]) suggest that, for example, in a water quenched alloy aged for 5 minutes at 565°C, 25% of the total magnetisation is associated with precipitate having an intrinsic coercivity greater than $\sim 5I$ (7,500 oe), where I is the saturation magnetization per unit volume of precipitate. Although deviations from the effects interpreted as disc-like particles are observed, it is estimated that the total amount of precipitate which could be in rod-like form (with intrinsic coercivities as high as $6I$) is less than 5%. The contribution to the coercivity due to interaction between the discs in the {100} sheets is estimated to be $\sim I$ (Gaunt and Silcox[1]). It is, therefore, likely that a significant contribution to the high coercivity of this alloy is due to either magneto-crystalline anisotropy or to uniaxial strain-induced anisotropy. In the first case the precipitates could be h.c.p. cobalt, which has a high uniaxial magneto-crystalline anisotropy, rather than the f.c.c. form, or the precipitates could be in the form of a metastable intermediate phase. It is therefore, of interest to determine the crystal structure of the precipitate. Gaunt[2] reported that no extra lines attributable to the precipitate were found in Debye-Scherrer photographs of aged and over-aged specimens. In the present experiments selected area diffraction patterns were obtained from a large

number of areas but only in a few cases were spots observed which were not due to the matrix. In these few cases the interpretations have suggested the possibility that both the f.c.c. and the h.c.p. phases of cobalt may be present, but the evidence is not conclusive. In connection with the second possible cause of anisotropy, *i.e.* strain induced anisotropy, it is pointed out that the precipitates could possibly be deformed because of the strain required at the interface to accommodate the difference in atomic diameter, (of about 12%), between gold and cobalt. Without more satisfactory diffraction patterns there appears to be no way of estimating the state of strain of the precipitates. While, therefore, the observations reported here appear to rule out shape anisotropy as the cause of the high coercivity of these alloys it has not yet proved possible to establish that the other two types of anisotropy make a substantial contribution to the observed coercivity.

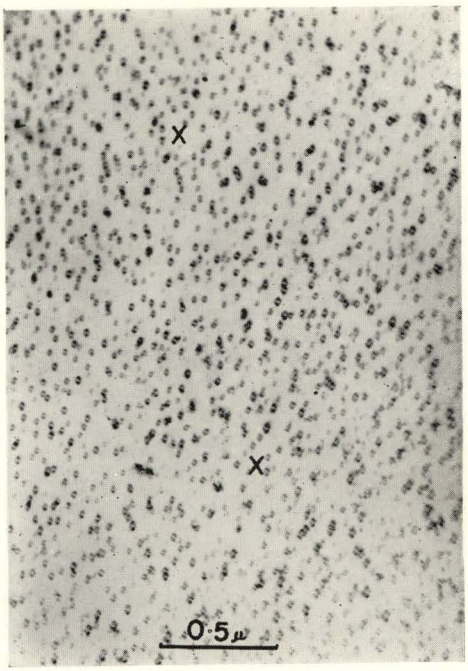

Fig. 5. Water quenched Cu 2.4% Co alloy aged 33 mins. at 705°C.

Copper 2.4% Cobalt Alloy

It is also thought to be of interest to report some preliminary observations made on a copper 2.4% cobalt alloy made by melting Johnson Matthey & Co. Ltd. spectroscopically standardised metals in a carbon mould under helium. A subsequent 36 hours homogenizing anneal at 960°C was given to the specimens. Electropolishing was carried out in a bath of 80% methyl alcohol, 20% nitric acid mounted in a dewar vessel containing liquid nitrogen.

Fig. 5 and 6 show areas of specimens given 30 min. and 451 mins. ageing at 705°C showing round contrast effects X and other effects Y. Since these are not seen in unaged specimens, they are thought to be due to precipitates. The behaviour on ageing also suggests this as, at the shorter time of ageing, there are many small precipitates, while the longer ageing time produces fewer but larger precipitates. The effects X are thought to be precipitates in the matrix and the effects Y to be precipitates on dislocations (since the arrays of effects Y are similar to the dislocation arrays seen in quenched unaged specimens). Apart from the precipitates Y, there appears to be no evidence so far for the presence of needles or disc-shaped particles (although the details of the contrasts mechanism have not yet been fully worked

Fig. 6. Water quenched Cu 2.4% Co alloy aged 451 mins. at 705°C.

out).

These preliminary results suggest that the conclusion of Becker[5] from magnetic measurements; that the precipitates were almost spherical, was essentially correct, but a detailed correlation of his predicted particle diameters and the observed contrast effects has not yet been attempted. The maximum coercivity of ~275 oe observed by Becker in similar alloys aged at 700°C is much lower than that observed in the gold cobalt alloys and may be explicable almost entirely in terms of particle shapes and interaction between the chains of particles precipitated on dislocation arrays.

Acknowledgements

The authors wish to thank Professor N. F. Mott and Professor W. Sucksmith for their interest and encouragement and Dr. P. B. Hirsch and Dr. R. B. Nicholson for useful discussions.

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DISCUSSION

C. D. Graham: I would like to point out that the precipitate particle shape in 2% Co-Cu has been studied by ferromagnetic resonance in our laboratory (D. S. Rodbell, J. Appl. Phys. **29** (1958) 311; Beau, Livingston, and Rodbell, Acta Met. **5** (1957) 682). The conclusion was that the precipitate particles were face-centered cubic, coherent with the copper matrix and accurately spherical (within 1%) for particle size below 1000 Å dia. A slight plate-like shape (a few percent deviation from sphericity) developed as the particle grew larger.

Victor Phillips of our laboratory is currently carrying out electron microscope observations on this system and his preliminary results confirm these findings.

W. Sucksmith: I am glad to hear that magnetic resonance experiments support the conclusion that the cobalt precipitate particles in Co-Cu alloy are approximately spherical.

P. Rhodes: Might it not be possible to obtain evidence on the origin of the particle anisotropy by measuring the variation of coercivity with temperature?

W. Sucksmith: The measurement of coercivity on a range of temperature would certainly answer your question but we have avoided the formidable difficulties of measurements above room temperature. On the other hand I do not think low temperature measurements would be unduly difficult and should be attempted.

E. P. Wohlfarth: H_c is only zero for strictly oblate spheroids. For flat plates in the shape of general ellipsoids H_c is finite and its value could be estimated from the dimensions. For Cu-Fe-Ni alloys of H_c~600 oersted, Beederman and Kneller also found plate-shaped particles.

W. Sucksmith: It is, as you say, probable that the plane of the disc may be elliptical and not circular, but I feel that the photographs rather tend to be negative for too much ellipticity and I doubt whether quantitatively there would be sufficient elongation to account for the experimental data.