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The determination of the lattice parameter for $GdCo_2$

In this paper we describe the result of the X-ray analyses of specimens of the cubic Laves phase compounds $GdCo_2$ which were manufactured for pulsed nuclear magnetic resonance investigations of the hyperfine field acting on cobalt nuclei. The specimens were manufactured using an electron beam furnace. Both stoichiometric and non-stoichiometric proportions of gadolinium and cobalt were used in the fabrication of the specimens. Originally non-stoichiometric proportions were used because it was thought than an excess (5 to 15%) of gadolinium would compensate somewhat for the loss of gadolinium from the mixture during the melting process, the gadolinium having a much higher vapour pressure than cobalt at that temperature. However, in the pulsed NMR experiments, and in the X-ray analyses described in this paper, there was no detectable difference between the stoichiometric and the non-stoichiometric specimens. The specimens were formed from an intimate mixture of 4N purity gadolinium powder and 5N purity cobalt powder which were pressed firmly in an extrusion press to form a pellet of 10 mm diameter. This was then placed on the water-cooled hearth of a G.D. Planer electron beam furnace and the furnace was evacuated to a pressure of less than 10^{-6} Torr. The electron beam was than focused on the pellet to melt the specimen. After a temperature of 1100 K was reached the electron beam was turned off and the specimen cooled quickly to room temperature. The melted button was then turned over and remelted several times to ensure that a homogeneous sample was prepared. After manufacture the buttons were filed under alchohol using a diamond hone. Some of the resulting powder specimens were wrapped in tantalum foil and vacuum annealed for 96 h at 1000 K. Others were analysed without annealing.

The X-ray analysis was made using two separate X-ray systems. One comprised a Rigaku SG-7 horizontal goniometer, a Rigaku D9C generator fitted with a fine-focus chromium X-ray tube, and a Xenon proportional detector coupled to a standard Rigaku circuit panel. The second system consisted of a mains stabilized Philips PW1120 generator fitted with a fine focus molybdenum tube. The sample was mounted in a Rigaku 2122B3 vertical gonimeter and the scattered radiation was detected with a Xenon proportional detector coupled to an Ortec counting system. Because the positions of the Bragg reflections had to be known very accurately the diffractometers were calibrated each day using both a silicon powder specimen and a bulk aluminium specimen. Both step-scan and continuous rotation measurements were made. Scans were made in both the clockwise and the anti-clockwise directions.

In an earlier paper [1] it was shown that, for a related Laves phase compound $GdFe_2$, the Bragg reflections were shifted from their expected positions because of deformation faulting. A similar effect is observed for $GdCo_2$ specimens.

Warren [2] has shown that the deviation of the position of the peak of a reflection from the true position is given by:

$$\Delta(2\theta) = \frac{90\sqrt{3}\tan\theta}{\pi^2 h_0^2 (u+b)} \cdot \alpha \cdot \Sigma [\pm] L_0 \quad (1)$$

where θ is the Bragg angle for a reflection of type hkl, L_0 equals $(h+k+l)$, h_0^2 equals $(h^2+k^2+l^2)$, and $(u+b)$ is the total number of combinations of hkl for the reflection. Those components corresponding to $L_0 = 3M$ give rise to unbroadened lines (u). Those corresponding to $L_0 = 3M \pm 1$ are broadened and L_0 is taken only for those broadened components.

The directions and magnitudes of these shifts can be calculated; e.g. the shift for 1 1 1 is + 0.25 for 2 0 0 is − 0.5; the shift for 2 2 0 is + 0.25 and so on.

The following procedure was used for the determination of the lattice parameter. A plot of the latttic parameters $a(hkl)$ calculated for the hkl reflections were plotted as a function of $\cos\theta_B \cot\theta_B$. Here θ_B is the observed Bragg angle of the hkl reflection. This is the procedure suggested by Adler and Wagner [3] for the elimination of systematic errors arising from misalignment of the specimen and the diffractometer. All the low-order Bragg reflections were included in the plot. Using least-squares analysis a straight line was fitted to the experimental points and the intercept of this line was taken to be an approximation to the value of a_0.

The distribution of the low-order reflections about the fitted line was examined. If the sequence of shifts of the experimental points from the "true" position had the same pattern as that expected from a faulted specimen (i.e., + for 1 1 1, − for 2 0 0, + for 2 2 0 etc.) the data was analysed to take into account the faulting. In all the samples of $GdCo_2$ the pattern of peak shifts was consistent with the existence of deformation faults in the samples.

By using Equation 1 to the values of α for all the low-angle reflections was determined, and the mean of these values $\bar{\alpha}$ was taken to be representative to the degree of faulting in the specimen. From this value of $\bar{\alpha}$ the shifts $\Delta(2\theta)$ were computed for each reflection. The position of each Bragg reflection was then corrected for the shift due to faulting and the values of $a(hkl)$ were computed from this value of Bragg angle. The procedure was then repeated starting with a new $\cos\theta \cot\theta$ plot and finishing with refined values of a_0. The value of $\bar{\alpha}$ was then determined using the new value of a_0.

TABLE I Lattice parameter a_0 and stacking fault parameter α for $GdCo_2$

Author		a_0 (Å)	α
This study		7.247 ± 0.008	0.35 ± 0.15
Skrabek	[4]	7.22 ± 0.05	—
Nooy *et al.*	[5]	7.3	—
Wernick	[6]	7.255 ± 0.005	—
Ross	[7]	7.257 ± 0.002	—
Baeziger	[8]	7.258 ± 0.003	—

In Table I values of a_0 and $\bar{\alpha}$ are recorded for a number of $GdCo_2$ samples manufactured at R.M.C. in the electron beam furnace. It should be mentioned that a sample manufactured by Mr A. Vass of Monash University in an argon arc furnace yielded values of a_0 and $\bar{\alpha}$ which are within the limits of error quoted in Table I. The limits of error quoted there do not represent the limits of error for a particular sample but rather they are the limits of error which result from the analysis of a large number of different $GdCo_2$ samples.

The wide spread of lattice parameters is evidence that some problem exists in either the technique of preparation of the sample or in the method of analysis of the X-ray diffraction data. One might expect that there would be some difficulty in the production of $GdCo_2$ samples since the compound is formed at high temperature and its structure is retained by means of a rapid quench to room temperature. It might be expected therefore that variations in such factors as the rate of quenching would cause minor variations in both the lattice parameter and the stacking fault probability.

Values of the lattice parameter quoted in the literature range from 7.22 to 7.26 Å. The value of the lattice parameter determined in this study is slightly lower than those quoted by Wernick [6], Ross [7] and Baeziger [8]. All these authors used Nelson–Reilly–Taylor–Sinclair extrapolation procedures without any consideration of the effects of deformation faulting in the specimen. It was demonstrated in our paper [1] that the use of such extrapolation procedures without consideration of crystallographic sources of line shifts can

lead to significant errors in the determination of lattice parameters.

The stacking fault probability is high, and every third or fourth plane contains a stacking fault. The $GdCo_2$ lattice (Structurbericht C15) is an fcc lattice containing eight atoms per unit cell. The cobalt atoms lie in sheets parallel to the {1 1 1} planes. For such an fcc structure the stacking faults lie in the {1 1 1} planes. Thus a high proportion of cobalt atoms lie in regions containing stacking faults and experience hyperfine fields which differ from those which one would expect for a perfect crystal. At the time of writing no $GdCo_2$ specimen has yielded a unique hyperfine field in a pulsed nuclear magnetic resonance experiment.

Assuming that the spacing between stacking faults is approximately three times the {1 1 1} interplanar spacing one can use Ayling's data for the yield strength to estimate the stacking fault energy. A value of about 2×10^{-4} J m^{-1} results from such a calculation. This is comparable with the value calculated for $GdFe_2$ using similar assumptions.

In this paper we have established that it is necessary to consider the possibility of crystallographic sources of line shifts as well as the well known mechanical sources of line shifts if a proper assessment of lattice parameters is to be made.

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The stability of equilibrium surface topography developed by sputtering

In a series of recent papers the present authors and others [1] have considered the development of topography upon isotropic, homogeneous solids resulting from the variation of erosion rate induced by energetic ion sputtering as a function of the angle of incidence of the ion flux. These studies have shown that a steady state topography will develop after high fluence irradiation where the surface is either (a) planar or (b) faceted, with facet angles inclined at $(\pi/2) - |\theta_p|$ to the ion flux incident direction, but except in the case of computational simulation [2, 3] have not distinguished the relative stability of these end forms. It is the purpose of this communication to explore the stability criteria.

As in earlier studies [1] we assume a surface generator in the $x0y$ plane to be irradiated with a uniform ion flux Φ in the $-$y-direction. If the sputtering yield (number of atoms eroded per incident ion) is denoted by S, then, frequently, the variation of S with the angle θ between the ion flux and the normal to a surface element, is given by the functional form shown in Fig. 1a, i.e. $S = S_0$ at $\theta = 0$, rising to maxima at $\theta = \pm\theta_p$ and declining, thereafter to $S = 0$ at $\theta = \pm(\pi/2)$.

If the surface generator is described, at time t, by the form

$$y(t) = f(x, t) \tag{1}$$

then, in order that this generator should maintain a time independent profile, Equation 1 must satisfy the general equation of motion for a wave, travelling in both x- and y-directions, with constant shape (the "soliton" [4, 5] solution), i.e.

$$y + v_y \cdot t = f(x - v_x \cdot t) \tag{2}$$