and minimum amplitudes as a function of φ, and $\varphi_{\max}$ being the azimuthal angle for the principal axis with the larger amplitude. The temperature factor is the product of two terms $T_{\max_H}$ and $T_{\min_H}$, and its derivatives with respect to $\Delta_{\max}$, $\Delta_{\min}$, and φ are

$$T_{\min_H}\frac{\partial T_{\min_H}}{\partial \Delta_{\max}}, \quad T_{\max_H}\frac{\partial T_{\min_H}}{\partial \Delta_{\min}}$$

and

$$T_{\min_H}\frac{\partial T_{\max_H}}{\partial \varphi_{\max}} + T_{\max_H}\frac{\partial T_{\min_H}}{\partial \varphi_{\max}}$$

respectively. The partial derivatives have the same form as the corresponding terms in Table 1.

The most important case of three degrees of freedom is rigid body libration. If correlated motions are excluded nine parameters are required. It is convenient to choose three of these as coordinates r_1, r_2 and r_3 specifying the point of intersection of the principal axes in a right handed system parallel to these axes. Three r.m.s. amplitudes Δ_1, Δ_2 and Δ_3 and three azimuthal angles φ_1, φ_2, and φ_3, defined in a similar manner to the analogous quantities in two dimensions, are also required. The temperature factor is the product of three terms T_{1H}, T_{2H}, and T_{3H}, and its derivatives with respect to r_1, Δ_1 and φ_1 are

$$T_{1H}\left(T_{2H}\frac{\partial T_{3H}}{\partial r_1} + T_{3H}\frac{\partial T_{2H}}{\partial r_1}\right), \quad T_{2H}\,T_{3H}\frac{\partial T_{1H}}{\partial \Delta_1}$$

and

$$T_{1H}\left(T_{2H}\frac{\partial T_{3H}}{\partial \varphi_1} + T_{3H}\frac{\partial T_{2H}}{\partial \varphi_1}\right)$$

respectively. The remaining derivatives are obtained by symmetry. The partial derivatives are identical in form with the corresponding terms in Table 1.

The procedure can also be extended to include the effect of correlated motions. It has been shown recently by Schomaker & Trueblood (1966, 1968) that a general treatment of rigid body motion should allow for correlation between vibrations and librations. The effect of the component of the correlated vibration in the same plane as a libration may be approximated closely by a lateral displacement of the rotation axis, while that of the perpendicular, or screw component, may be described by a rotation of the true rotation axis about the radius vector. The amount of the rotation is inversely proportional to the length of the radius vector. The lateral displacement and the constant of proportionality can be included in the least-squares refinement as parameters for the in plane and screw components respectively.

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Structural Imperfections in Hexagonal Gold–Cadmium Alloys

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The structural imperfections in hexagonal gold–cadmium alloys containing 18, 19 and 20 wt.% Cd have been investigated by X-ray diffraction methods. An analysis of X-ray diffraction line breadths of fault-broadened as well as unbroadened reflexions has been carried out. The stacking fault probability is greatest in 18 wt.% Cd alloy and decreases as the cadmium content increases. Evidence for the clustering of faults has been found.

X-ray diffraction provides a good method of studying structural defects in metals and alloys. The broadening of lines in the X-ray powder spectrum of deformed hexagonal metals and alloys is anomalous (Edwards & Lipson, 1942). The anomalies are such that only those reflexions for which $h-k=3t\pm1$, t being an integer, and $l \neq 0$ are broadened by stacking faults. Other reflexions, for which $h-k=3t$, show no broadening due to stacking faults and the only factors causing broadening are domain size and lattice strain.

In earlier work on hexagonal gold–cadmium alloys by Owen & Roberts (1940), there was no evidence for the occurrence of stacking faults in the lattice. Massalski (1957) determined the lattice spacings of close-packed hexagonal gold–cadmium alloys as a function of composition. X-ray diffraction patterns obtained from these alloys showed progressive broadening and splitting of certain close-packed hexagonal lines. The broadened reflexions were those with $h-k=3t\pm 1$ ($l \neq 0$), thus confirming to the presence of faults. Rivlin, Hume-Rothery & Ryder (1962) have studied the equilibrium diagram of the system gold–cadmium by thermal microscopic and X-ray diffraction methods and found that hexagonal alloys contain heavy faulting. The aim of this work was to estimate the fault probability in three hexagonal gold–cadmium alloys containing 18, 19 and 20 wt.% Cd. This investigation also seeks to determine the nature of any stacking fault density variations in hexagonal alloys.

The alloys used in the present investigation were prepared from spectroscopically standardized materials supplied by Johnson, Matthey and Co. Ltd., London. All alloys were cast in small ingots of two grams in weight, by melting carefully the weighed amounts of component metals in sealed, thin walled tubes of fused quartz under reduced pressure of helium. The ingots weighed 2·000 g before casting and the loss in weight after casting was less than 0·1%.

The alloys were homogenized for one month at 450°C and were examined under polarized light. They were all homogeneous polycrystalline hexagonal phase alloys.

All measurements were conducted with fine grained powder obtained from ingots. For eliminating the instrumental broadening samples annealed at 350°C in an electric furnace under vacuum were used. X-ray powder diffraction photographs were taken with copper radiation using a nickel filter and an 11·46 cm diameter camera. The intensity distribution of the powder lines was determined with the use of a microphotometer. The α_2 component was eliminated by the method due to Rachinger (1948).

A correction for instrumental broadening was applied by the method due to Stokes (1948). The breadths of the instrumental corrected profiles of reflexions which are broadened in the presence of stacking faults were corrected for domain size and strain broadening in order to obtain the stacking fault broadening. The domain size D and strain which were determined from reflexions unaffected by faulting *viz.* 0002, 0004, $10\bar{1}0$ and $20\bar{2}0$ were used to calculate the domain size and strain broadening, the respective broadening being $\lambda/D\cos\theta$ and $2\varepsilon\tan\theta$, where λ is the X-ray wavelength and θ, the Bragg angle. The anisotropy factor in these alloys is not large and so the mean domain size and strain obtained from fault unbroadened reflexions can be used for finding the broadening due to faults in the reflexions $10\bar{1}1$, $10\bar{1}2$ and $10\bar{1}3$. Fault probabilities have been determined from line widths assuming only the presence of deformation faults (Stratton & Kitchingman, 1965).

The results are summarized in the Table 1, which gives the values of domain size and strain determined from fault unbroadened reflexions 0002, 0004, $10\bar{1}0$ and $20\bar{2}0$. The faulting probability is the greatest in 18 wt.% Cd alloy. Electron microscope observations (Hirsch, Kelly & Menter, 1955) show that extended dislocations of equilibrium width are rare in heavily deformed materials and therefore no attempt has been made to determine the absolute values of stacking fault energies from the X-ray method which is based upon the assumption that faults consist of ribbons of extended dislocations of equilibrium width. Conclusions can however be drawn about the relative stacking fault energies in these alloys. The 18 wt.% Cd alloy having the greatest fault probability will have the lowest stacking fault energy and the 20 wt.% Cd alloys having the lowest fault probability will have the greatest stacking fault energy.

Table 1. *Fault probabilities in gold–cadmium alloys*

Alloy	Domain size (Å)	Mean strain	Fault probability
18 wt.%Cd	325	0·0051	0·059
19 wt.%Cd	360	0·0041	0·051
20 wt.%Cd	395	0·0036	0·039

The face-centered cubic phase boundary in the gold–cadmium system lies at 16·7 wt.% Cd. Therefore the alloy whose composition is nearer to the composition of the face-centered cubic phase boundary has the greatest stacking fault density. The plots of fault probability

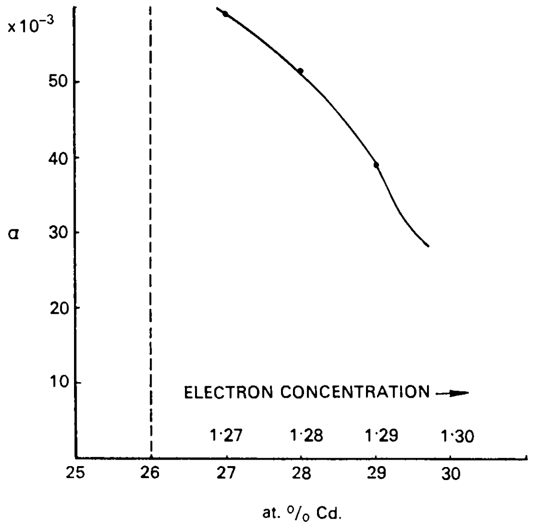

Fig. 1. The variation of stacking fault probability with electron concentration and wt.% cadmium in Au–Cd alloys. The dotted line shows the position of the phase boundary.

versus electron concentration are shown in Fig. 1. The fault probability is seen to decrease with electron concentration as in the results of Stratton & Kitchingman (1965). This suggests that the faulting probability depends upon the relative free energies of the hexagonal and face-centered cubic phases and electron concentration is only important in so far as it affects these energies. The fault probability is therefore not a function of electron concentration but depends upon the relative free energies of hexagonal and face-centered cubic phases.

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Structural Investigation of Hydrogen Bonds in Deuterated Pentaerythritol by Nuclear Magnetic Resonance

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A single crystal of pentaerythritol with deuterium substituted on the hydroxylic hydrogen positions was investigated by means of nuclear magnetic resonance. The quadrupole splittings of the deuterium magnetic resonance lines were measured as a function of the orientation of the external field with respect to the crystal axes. The electric field gradient tensors at the positions of the deuterium nuclei were evaluated and compared with the results obtained by Chiba in other hydrogen-bonded systems. The results provide evidence for a configuration obtained by taking the oxygen and carbon parameters from Shiono, Cruickshank & Cox (*Acta Cryst.* (1958), **11**, 389) and the hydrogen parameters from Hvoslef (*Acta Cryst.* (1958), **11**, 383).

1. Introduction

Pentaerythritol, $C(CH_2OH)_4$, is probably the only organic compound with exclusively alcoholic hydroxyl groups which can be rather easily obtained in large single crystals. The symmetry of pentaerythritol has been a point of discussion for a long time, but was established as being tetragonal with space group $I\bar{4}$ by Llewellyn, Cox & Goodwin (1937). The external symmetry of the crystals suggests the existence of a fourfold rotation axis, which is, however, not compatible with the internal structure.

The atomic positions were derived by, among others, Llewellyn, Cox & Goodwin (1937) and Shiono, Cruickshank & Cox (1958) from X-ray measurements, while Hvoslef (1958) has performed a neutron-diffraction study. The central carbon atoms are situated at the corners and in the centre of the tetragonal unit cell (Fig. 1). Four molecules whose central carbon atoms form a square in the (001) plane are linked by hydrogen bridges between the oxygen atoms. Four oxygen atoms are situated near the (001) plane at the corners of a distorted square. Between two oxygen atoms, but not exactly on the line connecting these oxygen atoms, a hydrogen atom is found (Hvoslef, 1958). It follows from the space group symmetry that the four hydrogen bridges are crystallographically equivalent. Quantitatively the results of Shiono, Cruickshank & Cox differ considerably from those of Hvoslef, more particularly in so far as the values of the z coordinates are concerned (Table 1). These discrepancies are reflected particularly in the orientations of the hydrogen bonds quoted by the two authors. According to Hvoslef the line connecting the two oxygen atoms of a hydrogen