IMPACT OF THERMAL AND MECHANICAL TREATMENT ON FAULTING IN HEXAGONAL COBALT

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Received November 15, 1964

I. Introduction

The reversible f.c.c. $\rightleftharpoons$ h.c.p. transformation that pure cobalt undergoes martensitically at 420° C. has been investigated over many long years, but many of its characteristics are yet to be clearly elucidated. Although the existence of stacking faults in the h.c.p. phase was first established[1] as early as 1942, quantitative measurements on cobalt specimens subjected to different thermal and mechanical treatments have been attempted only at greatly spread-out intervals. Wilson[1] measured faulting in one specimen of h.c.p. cobalt by X-ray line-breadth analysis, assuming that all the faults are of the growth type, but this interpretation has since been proved to be inadequate. Fresh measurements on the basis of both growth and deformation faults and utilising both X-ray line-breadth and line-shape analyses were made nearly fourteen years later by Anantharaman and Christian.[2] The next important measurements were made by Houska and Averbach,[3] who demonstrated by Fourier analysis that there are actually two regions in any transformed specimen, one containing only deformation faults and the other both deformation and growth faults. The effect of cold work and annealing on stacking fault densities has been most recently studied by Mitra and Halder.[4]

Although not numerous, the data collected so far on hexagonal cobalt have made it clear that the occurrence and distribution of stacking faults in hexagonal cobalt is definitely related to the nature of the martensitic transformation as influenced by the previous thermal and mechanical history of the specimen. Houska, Averbach and Cohen[5] have been the first to attempt an explanation of the impact of the structural change on the ultimate distribution and density of faults in hexagonal cobalt.

We propose here to re-examine the effects of thermal-mechanical treatment on faulting and its possible relation to the f.c.c.$\rightarrow$h.c.p. structural change in cobalt on the basis of more data on the densities of growth and deformation faults in various specimens.

II. Material and Methods

High-purity hydrogen-reduced powder (99·98% pure) or filings from solid cobalt obtained by melting the powder *in vacuo* in a high-frequency electric furnace were used throughout this study. The specimens were annealed at different temperatures for different periods. Debye-Scherrer patterns were obtained in a 9 cm. Unicam cylindrical camera with NiK_α radiation reflected from a crystal of pentaerythritol. The intensity profiles of individual reflections were recorded by accurate photometry.

The K_{α_1} peak positions were located by Rachinger's graphical method[6] as well as its analytical version suggested by Anantharaman and Christian.[7] The integral breadth due to pure diffraction (β) was calculated from the integral breadth of the powder reflections (B) with $H - K \neq 3n \pm 1$, where n is zero or an integer, which are affected by the incidence of stacking faults and that (b) obtained from the breadths of reflections with $H - K = 3n$, which are unaffected by the incidence of stacking faults, using the relation:

$$\beta = B - \frac{b^2}{B}$$

that was first suggested and employed by Anantharaman and Christian[2] and has since been verified by us[8] through Fourier analysis. The further analysis of the pure diffreaction integral breadths to calculate the densities of growth (α) and deformation (α') stackiug faults followed in this work has been described by us in an earlier paper.[9]

III. Experimental Results

Table I presents our data for cobalt annealed at 500° C. and 800° C. and also those further deformed mildly, well within the elastic limit, to varying amounts after this annealing treatment. This table also includes our results for fresh filings from a coarse-grained cast cobalt ingot and for the same annealed for a long time below the transformation temperature. The amount of hexagonal phase, the observed integral breadths of $10\bar{1}1$ and $10\bar{1}2$ reflections and the values of α and α', assuming that both types of faults are distributed at random, have been given in every case.

Table II A summarises the results so far available for the impact of annealing temperature on the relative amounts of hexagonal and cubic phases and the degree of growth and deformation faulting therein. Table II B similarly gives the results for the impact of cold work and further annealing on the hexagonal phase and the densities of both types of faulting in several

TABLE I

Impact of thermal and mechanical treatment on the phase transformation in cobalt powder specimens

S. No.	Nature of Treatment	Hexagonal content %	β $10\bar{1}1$	β $10\bar{1}2$	α	α′
1	Annealed *in vacuo* at 500° C. for one day	52	0·041	0·064	0·026	0·020
2	No. 1 mildly cold-worked	73	0·036	0·055	0·021	0·018
3	Annealed *in vacuo* at 800° C. for one day	58	0·048	0·131	0·092	0·004
4	No. 3 mildly cold-worked	79	0·025	0·059	0·038	0·004
5	No. 1 heavily cold-worked	100	0·063	0·069	0·007	0·042
6	Cast cobalt filings	100	0·078	0·084	0·007	0·053
7	No. 6 annealed at 375° C. for 4 weeks	~100	negligible	negligible	negligible	negligible

TABLE II

Comparison of available results on faulting on cobalt powder specimens

A. *Impact of annealing temperature*

S. No.	Annealing temperature (° C.)	Annealing time (days)	Hexagonal phase			Cubic phase		Source
			Approx. content %	α	α′	Approx. content %	α′	
1	1100	5	50	0·066	0·009	..	..	Wilson[1] and present work
2	1000	..	75	..	..	25	0·007	Houska *et al.*[5]
3	900	3	75	0·035	0·003	..	..	do.
4	800	1	60	0·092	0·004	..	..	Present work
5	800	1	80	0·038	0·004	..	..	do.
6	600	3	75	0·054	0·030	25	0·013	Houska *et al.*[5]
7	600	3	80	0·044	0·018	..	..	do.
8	500	1	50	0·026	0·020	..	..	Present work
9	500	1	75	0·021	0·018	25	0·017	Houska *et al.*[5] and present work

B. *Impact of cold work and further annealing on the hexagonal phase*

S. No.	Nature of treatment	Hexagonal content %	α	α'	Source
1	Cold work of powder annealed at 500° C.	100	0·007	0·042	Present work
2	Filings of coarse-grained massive cobalt	100	0·007	0·053	do.
3	No. 2 annealed at 375° C. for four weeks	100	practically nil	practically nil	do.
4	Grinding massive cobalt and annealing at 300° C. for 2 hours	100	0·032	0·019	Houska *et al.*[5]
5	No. 4 annealed for 7 days at 300° C.	100	0·038	0·008	do.
6	No. 5 annealed for 2 days at 390° C.	100	0·036	0·004	do.
7	Grinding massive cobalt and annealing at 100° C. for a day	..	0·051	0·021	Mitra and Halder[4]
8	Grinding massive cobalt and annealing at 200° C. for a day	..	0·040	0·018	do.
9	Grinding massive cobalt and annealing at 300° C. for a day	..	0·036	0·012	do.
10	Grinding massive cobalt and annealing at 400° C. for a day	..	0·034	0·008	do.
11	Grinding massive cobalt and annealing at 500° C. for a day	..	0·030	0·004	do.

cobalt samples. The experimental data of Wilson[1] have been analysed afresh for this purpose as earlier described[9] and the results of Houska and Averbach[3] and Houska *et al.*[5] have been recalculated for random occurrence of faults throughout the specimens. Values of α and α' derived by Mitra and Halder[4] on the basis of a Gaussian assumption for strain distribution for separating the strain contribution to the observed broadening are also mentioned.

IV. Discussion of Results

The following points are clearly brought out by the results presented in Tables I and II:—

1. On mild deformation well within the elastic limit (by grinding with the fingers), the samples annealed at 500 and 800° C. record an increase in the hexagonal content by as much as 20 per cent. Concurrently there is a decrease in growth fault probability, while the deformation fault probability remains practically unaltered.

2. The density of deformation faults in the retained f.c.c. phase seems to be more or less the same as that in the transformed h.c.p. phase.

3. On heavy cold work, the retained f.c.c. phase changes over completely to the h.c.p. phase with a considerably reduced growth fault density value,

but with heavy deformation faulting. The highest deformation fault density ($\alpha' = 0\cdot053$) so far recorded for cobalt was observed in fresh filings from a coarse-grained cobalt ingot.

4. The deformation faults in the h.c.p. phase anneal out rapidly on heating below the transformation temperature.

5. When fresh filings from a cast ingot are annealed at 375° C. for about 4 weeks, the hexagonal phase shows practically no faulting and a negligibly small percentage of the same transforms to the cubic phase. Such a sample is ideal for accurate evaluation of lattice parameters of hexagonal cobalt and has been in fact used for this purpose by Anantharaman.[11] The high growth fault density in the specimens of Houska *et al.*[5] and Mitra and Halder[4] annealed at 300–390° C. is rather surprising in this context. It has, however, to be stated that our coarse-grained cobalt ingot seemed to be practically free from stacking faults as examined by the X-ray oscillation technique.

6. With, increasing temperature of anneal above the transformation temperature the deformation fault probability decreases definitely. There does not seem to be any pronounced increase in growth fault probability with increase in annealing temperature, but this effect is definitely modified by the further transformation induced as referred to above by mild deformation of the specimen during handling, sieving specimen preparation, etc. The deformation fault density, however, is not affected by this mild deformation.

The present results are generally in agreement with those obtained earlier and confirm certain trends, which could only be tentatively deduced from earlier data. The only serious difference concerns the growth fault density values in specimens totally transformed by deformation, the present data showing negligible or no growth fault density and the results of Houska *et al.*,[5] and Mitra and Halder[4] indicating as high an α value as $\sim 0\cdot03$, after annealing at 300 and 390° C.; the rather high value of α ($0\cdot051$) in the cold-worked specimen of Mitra and Halder[4] is particularly surprising. In the absence of clear informatiion in these earlier investigations on the previous history of the massive specimen from which the powder was prepared, we shall confine ourselves to an interpretation of only the present results in this regard.

On the justifiable assumption that a higher annealing temperature leads to a higher grain size and a less imperfect f.c.c. phase, the effects of the deformation accompanying the transformation on cooling can be visualised to be more drastic in the case of specimens annealed at lower temperature with more grains per unit volume, more imperfections and greater possibilities for the operation of different dislocation mills leading to the transformation. Such a state of affairs will lead to a larger α' value at lower tempearature of anneal and also more or less the same α' value for both the transformed h.c.p. and untransformed f.c.c. phase in the the same specimen. These conclusions are generally borne out by the data in Table II A.

The rapid annealing out of deformation faults above room temperature and below the transformation temperature is easily understood. As explained by Houska *et al.*,[5] a deformation fault can be unslipped merely by the passage of a partial dislocation along a single close-packed plane.

The percentage of the hexagonal phase formed by spontaneous transformation seems to have a bearing on the growth fault density in specimens annealed at the same temperature. Obviously the transformed specimen is in a state of delicate metastable equilibrium and can easily be subjected to further transformation by the mildest of deformations (Table I). Such transformation is found to bring down the α value without in any way affecting the α' value in the same specimen. This is in general agreement with one aspect of the data on specimens completely transformed by plastic deformation, *viz.*, the growth faults are partly removed by some mechanism during the transformation induced by stress. Obviously cold work induces dislocation motions which annihilate growth faults by further transformation. Considering that growth faults seem to be generally crowded in certain regions of the transformed specimens,[3-4] this effect seems quite probable.

V. Summary

New results on the impact of thermal and mechanical treatment of pure cobalt on the content of the hexagonal phase and densities of growth as well as deformation stacking faults have been presented. The significance of these results along with those obtained by earlier investigators has been discussed *vis-a-vis* the spontaneous as also stress-induced f.c.c.→h.c.p. transformation in cobalt.

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