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X-RAY DIFFRACTION PROFILE ANALYSIS OF HEXAGONAL COBALT IN METASTABLE COMPACT POWDERS

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Samples of pressed cobalt powder were investigated using *X*-ray diffraction line profile analysis. Stacking fault parameters and microstrains were obtained using the single profile method and then compared with previous results, derived from the Warren-Averbach method. Both methods give comparable results for stacking fault paramaters and root mean square strains. The results obtained are of the same order of magnitude as those measured in plastically deformed cobalt.

1. Introduction

Phase transformations in pressed cobalt powder have been investigated recently by means of *X*-ray diffraction line profile analysis and magnetometric measurements[1]. It was found that the α-(FCC) phase transformed rapidly into the ε-(HCP) phase up to 20 MPa, and more slowly at higher pressures[2]. The present paper reports on the determination of stacking fault parameters (SFP) and microstrains on the basis of *X*-ray diffraction line profile analysis of the same samples, using the single line profile method according to Mignot and Rondot[3].

2. Experimental procedure

The samples were prepared from technical cobalt powder of 99.8 wt. % purity, as used in hard metal production. They were compacted in a floating cemented carbide die, having the section of 22 × 12 mm², attached to a hydraulic press.

Pressure up to 660 MPa were applied at room temperature. *X*-ray diffraction analysis was taken of the upper surfaces of the sample. Diffraction line profiles were measured on a Siemens diffractometer using Bragg-Brentano focusing geometry. Radiation was monochromatized with a bent quartz crystal, which focused the Co/K_α radiation on the entrance slit of the counter. A scintillation counter was used with a single-channel pulse-height analyser. Samples of cobalt powder annealed in vacuum were used as calibration standards representing instrumental broadening.

The broadened diffraction line profiles were corrected for instrumental broadening using a computer program written in FORTRAN[1]. This procedure yields the Fourier coefficients of pure diffraction broadening.

3. *Theory*

The cosine Fourier coefficient of the pure diffraction line profile *hkl* can be represented as[4]

$$A(L, hkl) = A^p(L) \cdot A^s(L, h\,k\,l),$$

where $A^p(L)$ is the domain size-faulting component, and $A^s(L, h\,k\,l)$ the strain component of each coefficient. L is the distance normal to the reflecting planes (*hkl*) with interplanar spacing d_{hkl}, $L = n\,d_{hkl}$, where n is the order of the cosine coefficient.

$A^p(L)$ is independent of the diffraction line indices *hkl*, while $A^s(L, hkl)$ depends on *hkl*. This makes it possible to separate the two components of the Fourier coefficients, utilizing two or more orders of the reflections from the same lattice planes (Warren and Averbach[5]).

The distribution of the strain term $A^s(L, hkl)$ is Gaussian in L, for small L and strain ε_L:

$$A^s(L, hkl) = \exp\{[-2\pi^2 L^2/d_{hkl}^2]\,\langle\varepsilon_L^2\rangle_{hkl}\} \approx 1 - [2\pi^2 L^2/d_{hkl}^2]\,\langle\varepsilon_L^2\rangle_{hkl}\},$$

where $\langle\varepsilon_L^2\rangle_{hkl}$ is the mean square strain of the strain $\varepsilon_L = \dfrac{\Delta L}{L}$. The term $A^p(L)$ due to the domain size and faulting can be approximated for small L:

$$A^p(L) \cong -\frac{L}{D_e},$$

where D_e is the apparent (»effective«) crystallite (domain) size normal to the reflecting planes, consisting of the true crystallite size $\overline{D}$ and faultings.

In HCP crystals the (002) planes form close packed layers with regular stacking sequence *ABABAB*... The growth or twin stacking fault occurs when the stacking changes from *AB* to *BC* as in the sequence *ABABCBCB*... The deformation stacking fault with the sequence *ABABCACA*... introduces a small range with FCC stacking order. Warren[4] assumed that stacking faults occur on the (002)

planes independently and showed that the initial slopes of the plots of $A^p(L)$ vs. L are given as follows:

$$\text{for } h - k = 3N, l = 0,$$

$$-(\mathrm{d}A^p/\mathrm{d}L)_{L=0} = 1/\bar{D}$$

$$\text{for } h - k = 3N \pm 1,\ l \text{ even},$$

$$-(\mathrm{d}A^p/\mathrm{d}L)_{L=0} = 1/\bar{D}_e = 1/\bar{D} + (l \cdot d_{hkl}/c^2) \cdot (3\alpha + 3\beta), \tag{3}$$

$$\text{for } h - k = 3N \pm 1,\ l \text{ odd},$$

$$-(\mathrm{d}A^p/\mathrm{d}L)_{L=0} = 1/\bar{D}_e = 1/\bar{D} + (l \cdot d_{hkl}/c^2) \cdot (3\alpha + \beta). \tag{4}$$

Here α is deformation fault probability, β growth fault probability, c the lattice parameter along the c-axis, d_{hkl} interplanar spacing and N is an integer. The second terms in (3) and (4) represent the faulting contribution to the broadening, $1/D^F_{hkl}$.

Besides the method of Warren and Averbach, there is also the single line profile method according to Mignot and Rondot[2]. In their method one assumes that $\langle \varepsilon_n^2 \rangle = C/n$ and that the Fourier coefficients $A(n)$ can be written as follows:

$$A(n) = (1 - nx) \cdot (1 - n^2 y_n) = 1 - n(x + KC) + n^2 xKC$$

where

$$x = \frac{1}{2}[-a_1 \pm (a_1^2 - 4a\lambda)^{1/2}], \quad C = \frac{a_2}{xK}.$$

$A(n)$ may be approximated by the polynomial

$$P(n) = a_0 + a_1 n + a_2 n^2,$$

where a_0, a_1 and a_2 are the coefficients of the polynomial $P(n)$.

The effective crystallite size is $D_e = \dfrac{\delta}{x}$, where δ is distance the magnitude of which is inversely proportional to the Fourier period, $K = 2\pi^2 \delta^2/d_{hkl}^2$, where d_{hkl} is the distance between the (hkl) planes. The results were selected applying the following conditions:

$$a_0 \approx 1 \quad a_2 > 0 \text{ and } \frac{1}{N} \sum_{n_0}^{N} [A(n) - P(n)]^2 = \textit{minimum},$$

where N is the total number of the Fourier coefficients used in the calculation, and n_0 the smallest utilized value of Fourier's harmonics.

4. *Results and discussion*

The experimental results for three samples are shown in Table 1 and Table 2. Table 1 shows the effective crystallite sizes $D_{hkl} \to D_e$ for diffraction lines 101, 102, 103, obtained by analysing results according to the single line profile method. The values $(3\alpha + \beta)$, $(3\alpha + 3\beta)$ and true crystallite size $\overline{D}$ were calculated from Eqs. (3) and (4). The values of SFP α and β were then calculated. These values were compared with the values $\alpha^{1)}$ and $\beta^{1)}$ obtained in Ref. 1 from the Warren-Averbach (*W. A.*) analysis and from peak shift measurements. From Table 1 one can see that SFP α and β which were obtained utilizing different methods, are in fairly good agreement. The ε-phase shows relatively high SFP α at all pressures. There is a tendency of increasing SFP α with the applied external pressure, while SFP β is decreasing at the same time.

TABLE 1.

p/MPa	(hkl)	D_{hkl}/nm	$(3\alpha + \beta)$	$(3\alpha+3\beta)$	$\overline{D}$/nm	α	β	$\alpha^{1)}$	$\beta^{1)}$
	101	14.3	0.0433		50.5	0.013	0.0038	0.016	0.032
66.2	103	9.2							
	102	9.0		0.0509					
	101	13.8	0.0444						
221	103	8.8			47.4	0.0102	0.014	0.022	0.017
	102	6.7		0.072					
	101	8.3	0.075						
552	103	5.26			29.7	0.025	0.0003	0.022	0.006
	102	5.9		0.0757					

Effective crystallite sizes $D_{hkl} \to D_e$, true crystallite sizes $\overline{D}$ and stacking fault parameters α and β obtained from single line profile method for different external pressures p.

TABLE 2.

p/MPa	(hkl)	$\langle \varepsilon_L^2 \rangle^{1/2} \cdot 10^3$	$\langle \varepsilon_L^2 \rangle^{1/2} \cdot 10^3$	$\overline{D}\,(WA)$/nm	$\overline{D}$/nm
	101	3.7		89.0	50.5
66.2	103	2.3	2.5		
	102	14.4			
	101	4.3			
221	103	9.6	3.1	84.0	47.4
	102	7.8			
252	101	3.2			
	103	1.6	2.7	71.4	29.7
	102	2.3			

Root mean square strains $\langle \varepsilon_L^2 \rangle^{1/2}$ averaged at the distance $L = 2$ nm and true crystallite sizes $\overline{D}$, obtained from single line profile method, compared with $\langle \varepsilon_L^2 \rangle^{1/2}$ at $L = 2$ nm and $\overline{D}\,(WA)$, calculated from Warren-Averbach analysis.

In Table 2 the root mean square strains $\langle \varepsilon_L^2 \rangle_{hkl}^{1/2}$ (RMSS) at $L = 2$ nm in directions normal to the planes (101), (102) and (103), obtained from the single line profile method, are compared with values $\langle \varepsilon_L^2 \rangle^{1/2}$ obtained from the *W. A.* analysis for all diffraction lines. The mean value obtained for three lines should be compared with $\langle \varepsilon_L^2 \rangle^{1/2}$ value obtained from the *W. A.* method at the same L. We may say that the results are in good agreement as the obtained order of magnitude is the same for both methods.

In Table 2 the true crystallite sizes $\bar{D}$ which are independent of the diffraction line indices *hkl*, were obtained utilizing relations (3) and (4), and compared with $\bar{D}$ (*WA*) values obtained from the *W. A.* analysis.

We might conclude that the RMSS and the SFP obtained in Tables 1 and 2 are of the same order of magnitude as those obtained in plastically deformed cobalt[5].

We also conclude that the single line profile method and the Warren-Averbach method give comparable results and that the single line profile method might be applicable even when the Warren-Averbach method does not apply.

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ANALIZA RENDGENSKIH DIFRAKCIJSKIH PROFILA HEKSAGONSKOG KOBALTA U UZORCIMA TLAČENOG PRAHA

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Ispitivani su uzorci tlačenog kobaltnog praha analizom profila rendgenskih difrakcijskih linija. Parametri α i β pogrešaka u slaganju mrežnih ravnina, te mikrodeformacija, dobiveni su metodom profila jedne difrakcijske linije i uspoređeni s rezultatima dobivenim Warren-Averbachovom metodom. Obje metode daju usporedive rezultate za parametre pogrešaka u slaganju i relativne prosječne deformacije. Dobiveni rezultati su istog reda veličine kao i oni u uzorcima plastično deformiranog kobalta.