Analysis of X-ray Line Broadening in a Face-Centred-Cubic Copper-Silicon-Manganese Alloy due to Cold-Work at Room Temperature

It is now well established that the broadening of X-ray diffraction lines from a cold-worked metal or alloy is to be attributed to the small size of the coherently diffracting domains, lattice strains and stacking faults. Shifts in the peak positions of diffraction lines [1], and the angular separation of the centre of gravity from the peak position of certain diffraction lines [2] are the most convenient and reasonably accurate methods of determining the deformation faulting parameter (α) and twin faulting parameter (β) respectively. The magnitudes of the coherently diffracting domains and the microstrains in the lattice can be obtained either by the method of Warren and Averbach [3] using Fourier techniques, or from measurements of the integral breadths of several diffraction lines corrected for the contribution due to stacking faults [4]. This communication gives details of results obtained by using the latter method with a copper-silicon-manganese alloy.

An alloy with a composition of approximately 6.7 at. % silicon and 1.3 at. % manganese, remainder copper (corresponding to about 3.5 wt % silicon, 1.1 wt % manganese, remainder copper), known to have a face-centred-cubic structure and a tendency towards profuse faulting [5] was deformed by filing at room temperature. A portion of the filings was compacted straight away into a diffractometer pellet while another portion was annealed *in vacuo* at a temperature of 700° C for $1\frac{1}{2}$ h prior to compacting, so as to provide the diffraction pattern of the undeformed state of the alloy. Copper Kα radiation and a Siemens goniometer with a scanning speed of $\frac{1}{8}$°/min in 2θ were employed to record the profiles of the diffraction lines. Using the shifts in the peak positions of the lines (111), (200) and (220), the deformation faulting parameter α was evaluated as 0.022. From the integral breadths (B) of the cold-worked reflections and the integral breadths (b) of the annealed reflections, the integral breadths due to deformation only (β) can be calculated by using the relation [6]:

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

This broadening however includes not only the contributions due to domain size and lattice strain but also that due to stacking faults. Anantharaman [7] has described a method whereby the contribution due to stacking faults can be included in the pure geometrical broadening b. Designating this combined value as b_0, the new relation would be:

$$\beta' = B - \frac{b_0^2}{B},$$

where β' is the deformation-broadening due only to domain size and lattice strain. The domain size value can now be obtained by plotting $(\beta' \cos\theta/\lambda)^2$ against $(\sin\theta/\lambda)^2$ for two orders of the same reflection [5]. The intercept at $(\sin\theta/\lambda)^2 = 0$ gives the value of the square of the reciprocal of the domain size, while the slope of the line is proportional to the square of the strain. Had the factor β been used instead of β' in the plot, one would have obtained a fictitious value of the domain size which would have included the contribution due to stacking faults.

Table I gives the values of β, β' and the domain size values obtained by using each of these. The values of β and β' for the (400) reflection could not be obtained with much accuracy as the reflection in the cold-worked state was very weak. Domain size and strain values in the [100] direction, especially the latter, are therefore only approximate, as is usual in this type of work with alloys showing profuse faults. It will be noticed from the table that the domain sizes are, in both directions, larger after correction for fault-broadening, as is to be expected. The strain values remain unaffected.

In table II, the values β and β' are given for the first five reflections of the alloy and an

TABLE I Domain sizes and lattice strains before and after correction for stacking fault-broadening.

Crystallographic direction	β_{hkl} (111) or (200) rad. × 10^3	$\beta_{2h_2k_2l}$ (222) or (400) rad. × 10^3	β'_{hkl} (111) or (200) rad. × 10^3	$\beta'_{2h_2k_2l}$ (222) or (400) rad. × 10^3	Domain size obtained using β	Domain size obtained using β'	Lattice strain obtained using β	Lattice strain obtained using β'
[111]	12.05	22.61	10.40	21.11	163 Å	205 Å	0.004	0.004
[100]	23.76	67.20	17.79	60.90	105 Å	400 Å	0.009	0.009

TABLE II Analysis of the first five reflections of the alloy.

Reflection	$\beta = B - \frac{b^2}{B}$ rad. $\times 10^3$	$\eta = \frac{\lambda}{\beta\cos\theta}$ Å	$\epsilon = \frac{\beta}{4\tan\theta}$ $\times 10^3$	Deviation from mean value of η %	Deviation from mean value of ϵ %	$\beta' = B - b_0^2/B$ rad. $\times 10^3$	$\eta' = \frac{\lambda}{\beta'\cos\theta}$ Å	$\epsilon' = \frac{\beta'}{4\tan\theta}$ $\times 10^3$	Deviation from mean value of η' %	Deviation from mean value of ϵ' %
(111)	12.05	138	7.61	50	9	10.40	159	6.57	54	10
(200)	23.76	72	12.65	22	52	17.79	96	9.47	7	29
(220)	23.58	82	7.83	11	6	22.73	85	7.55	18	3
(311)	33.11	66	8.32	28	0	32.54	67	8.18	35	12
(222)	22.61	101	5.19	10	38	21.11	108	4.84	5	34
Mean value	—	92	8.32	—	—	—	103	7.32	—	—
Mean deviation from mean value	—	—	—	24	21	—	—	—	24	18

apparent domain size (η, η') and *apparent* strain (ϵ, ϵ') are calculated, assuming the *entire* deformation-broadening to be caused solely by either one of these factors. The mean deviation from the mean value is then calculated for both. If either one of these factors were to be *preponderantly* responsible for the broadening then the mean deviation from the mean value would be much less for this one than for the other [4]. In the present case it is seen that the mean deviation from the mean value is large in both cases and no appreciable variation is observed even after correcting for faults. This seems to indicate that both of these factors contribute in comparable amounts to the line-broadening.

Before concluding it may be worthwhile to compare our results with those of Welch and Otte [5]. The values quoted by these authors, $\alpha = 0.025$, domain sizes and strains along $\langle 1\,1\,1\rangle$ and $\langle 1\,0\,0\rangle$ = 142 Å, 92 Å, 0.003 and 0.006 respectively, without correcting for stacking fault-broadening, agree reasonably well with our own values of $\alpha = 0.022$, 163 Å, 105 Å, 0.004 and 0.009 respectively, for work of this nature.

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Internal Friction in Vanadium After Heating at Low Air Pressure

Edington and Smallman [1] have studied, using electron microscopy techniques, polycrystalline samples of vanadium after heating at low air pressures. They report the existence of a body-centred tetragonal phase similar to the β-phase with a high concentration of twins. They also observed, on cooling from 1300° C, a martensitic