which included the refinement of the scales of the individual Weissenberg and precession layers, led to $R = 11{\cdot}3\,\%$, and after averaging the $|F_o|$ values of all common reflexions two further cycles reduced R to $9{\cdot}3\,\%$.

The revised coordinates and their e.s.d.'s are given in Table 1. The maximum change from the values of Belt & Baenziger is 0·030 Å; the effect of adding the precession data has been to reduce the changes. The anisotropic vibration parameters are not given, as they seem to be considerably affected by the neglect of absorption; however, they provide no hint of a need for rotational corrections. Table 2 shows the revised distances and angles. They are not significantly different from those given by Belt & Baenziger, so that their main results are confirmed despite the errors in the Weissenberg Lp corrections. They are also fairly consistent with the results obtained from the Weissenberg reflexions alone in the first stage of the refinement.

The N–O(1) bond length has changed from 1·51 ± 0·02 Å to 1·483 ± 0·017 Å, which agrees better with the values reported by Jerslev (1948) in hydroxylammonium chloride (1·47 Å) and by Meyers & Lipscomb (1955) in hydroxylamine (1·48 Å). The S–O lengths average 1·459 ± 0·007 Å and S–N is 1·687 ± 0·012 Å, as compared with 1·453 ± 0·005 and 1·662 ± 0·005 Å in the $[NH(SO_3)_2]^{2-}$ ion (Cruickshank & Jones, 1963). The slight lengthening of S–N may be correlated with a more pyramidal distribution of the valencies about nitrogen, for S–N–O(1) = 108·4° as compared with S–N–S = 125·5° in the imidodisulphate. Nevertheless it remains shorter than the formal S–N single bond length of 1·74 Å, so there is some measure of S(d)–N(p) π-bonding, though not so much as in $[NH_2SO_3]^-$ (1·60 ± 0·03 Å, Jeffrey & Stadler, 1951) or $SO_2(NMe_2)_2$ (1·623 ± 0·007 Å, Jordan, Smith, Lohr & Lipscomb, 1963).

The angle between the planes NSO(3) and SNO(1) is about 18°, so that with respect to the S–N bond O(1) is nearly *trans* to O(3). O(1) is 2·83 Å from O(2), 3·09 Å from O(4), but only 2·57 Å from S. The O(1)–S repulsion is therefore the main cause of the irregularities in the angles at S. In particular the fact that O(3)–S–N = 101° is the smallest angle is consistent with the *trans* position of O(1).

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Evaluations of particle size and strain values from X-ray line breadths and line shapes in α-AgCd alloys.

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In X-ray studies of cold-worked metals and alloys the conventional methods of line profile analysis involve estimates of various quantities (*e.g.* particle size and strain) from measurements of line shapes (Warren, 1959) and line breadths. The former analysis assumes a Gaussian peak profile while the latter assumes either a Gaussian or a Cauchy peak shape for both particle size and strain broadening. However, it turns out that particle size broadening tends to give a Cauchy line and strain broadening a Gauss or $1/(1+k^2x^2)^2$ line (Warren, 1959; Taylor, 1961). Recently, Schoening (1965) has given a method for evaluating particle size and strain values from line breadth analysis with the use of these more realistic profiles. In the present note Schoening's method has been considered and a comparative study of the peak broadening of cold-worked α-AgCd alloys by the Warren–Averbach method and integral breadth measurements has been made.

The experimental procedure and the results of Fourier analysis of line shapes in α-AgCd alloys have been reported earlier (Sen Gupta & Quader, 1966). The integral breadths of the 111, 200, 222 and 400 peaks of cold-worked and annealed materials were evaluated after the separation of the α_2 component by Rachinger's (1948) method and corrected for instrumental broadening by the parabolic relation (Wagner & Aqua, 1963) $b_{\text{true}} = B_{\text{CW}} - b_A^2/B_{\text{CW}}$. In the Cauchy peak shape, separation of particle size and strain has been effected by using the linear relation $b_t \cos\theta_0/\lambda = 1/D_C + 4\,\varepsilon_C \sin\theta_0/\lambda$ and in the Gaussian peak shape the separation has been made by using the square relation $(b_t \cos\theta_0/\lambda)^2 = 1/(D_G)^2 + 16\varepsilon_G^2\,(\sin\theta_0/\lambda)^2$, θ_0 being the peak maximum position. For Schoening's method both cases were considered and the separation of strain broadening from particle size has been done by the method outlined (Schoening, 1965). The values of the fault probabilities have been also deduced from the effective particle sizes (Wagner & Aqua, 1963) and the results are summarized in Tables 1 and 2.

It is apparent from Tables 1 and 2 that, in general, the Warren–Averbach technique and line breadth measurements lead to different values of particle sizes and strains. But it can be seen from Table 1 that there exists a close agreement between the particle size values $[D_e]_{hkl}$ and $[D_G]_{hkl}$ obtained from two methods, $[D_G]_{hkl}$ being slightly higher. The values obtained from the Cauchy relation are larger ($[D_C]_{hkl} \sim 2[D_e]_{hkl}$) and there exists a fair agreement between all the Cauchy values. Schoening's method gives lower values and for the case where the strain assumes a Gaussian form values of $[L]_{hkl}$ approach those of $[D_G]_{hkl}$. Regarding compound fault probability $(1{\cdot}5\alpha+\beta)$ evaluated from particle size values and from peak-shift and peak asymmetry measurements, line shape analysis gives good agreement and a fair agreement has been observed in the values obtained with the Cauchy assumption. The strains

Table 1. *Particle size and faulting probabilities in cold-worked* α-AgCd *alloys*

Alloys (at. %)	$(1{\cdot}5\alpha+\beta)_{P+A}$	Line shape measurements (Warren & Averbach's method)			Integral breadth measurements Gaussian		
		$[D_e]_{111}$ (Å)	$[D_e]_{100}$ (Å)	$(1{\cdot}5\ \alpha+\beta)$	$[D_G]_{111}$ (Å)	$[D_G]_{100}$ (Å)	$(1{\cdot}5\ \alpha+\beta)$
Ag—4·8 Cd	0·020	208	116	0·028	378	164	0·050
Ag—9·6 Cd	0·023	190	101	0·034	250	122	0·061
Ag-19·3 Cd	0·048	160	75	0·052	206	113	0·058
Ag-29·2 Cd	0·060	130	66	0·055	192	91	0·085

Integral breadth measurements

Alloys (at. %)	Cauchy			Schoening's Method Case I			Schoening's Method Case II		
	$[D_C]_{111}$ (Å)	$[D_C]_{100}$ (Å)	$(1{\cdot}5\ \alpha+\beta)$	$[L]_{111}$ (Å)	$[L]_{100}$ (Å)	$(1{\cdot}5\ \alpha+\beta)$	$[L]_{111}$ (Å)	$[L]_{100}$ (Å)	$(1{\cdot}5\ \alpha+\beta)$
Ag—4·8 Cd	625	294	0·026	544	237	0·034	480	210	0·038
Ag—9·6 Cd	392	192	0·038	305	162	0·042	305	149	0·049
Ag-19·3 Cd	312	172	0·038	251	156	0·035	232	141	0·041
Ag-29·2 Cd	278	151	0·044	233	125	0·055	216	114	0·061

Table 2. *Strains in cold-worked* α-AgCd *alloys*

Alloys (at. %)	Line shape measurements (Warren & Averbach's Method)		Integral breadth measurements Gaussian	
	$[\langle\varepsilon^2{}_{L=50\,\text{Å}}\rangle]_{111}{}^{\frac{1}{2}}$	$[\langle\varepsilon^2{}_{L=50\,\text{Å}}\rangle]_{100}{}^{\frac{1}{2}}$	ε_{G111}	ε_{G100}
Ag—4·8 Cd	0·00271	0·00310	0·00399	0·00682
Ag—9·6 Cd	0·00287	0·00317	0·00458	0·00771
Ag-19·3 Cd	0·00220	0·00316	0·00444	0·00907
Ag-29·2 Cd	0·00289	0·00377	0·00476	0·01105

Integral breadth measurements

Alloys (at. %)	Cauchy		Schoening's method Case I		Schoening's method Case II	
	ε_{C111}	ε_{C100}	e_{111}	e_{100}	e_{111}	e_{100}
Ag—4·8 Cd	0·00346	0·00574	0·01398	0·02331	0·01447	0·02397
Ag—9·6 Cd	0·00370	0·00516	0·01657	0·02536	0·01540	0·02565
Ag-19·3 Cd	0·00329	0·00744	0·01367	0·03041	0·01417	0·03102
Ag-29·2 Cd	0·00353	0·00900	0·01471	0·03685	0·01518	0·03736

obtained from line breadth measurements are larger than the r.m.s. strain components at an average distance $L=50$ Å (Table 2). The strain values are much higher in the case of Schoening's method and are closer to the values of r.m.s. strains for $L=0$. It may be noted that Schoening's method gives rise to nearly identical strain values in the two cases though they were obtained from different assumptions. However, in the light of these results we may draw the conclusion that besides the most satisfactory and widely used method of Warren (1959) which unfolds the respective contributions to the line contour of particle size, lattice-strain and stacking faults, Schoening's method (1965) also proves to be quite useful in evaluations of particle size and strain from integral breadth measurements, and should be taken into consideration as it deals with the more realistic line profiles.

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