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The Width of X-Ray Diffraction Lines From Cold-Worked Tungsten and α-Brass*

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The x-ray line width from specimens of cold worked α-brass and tungsten has been measured as a function of Bragg angle and of x-ray wave-length. The results are in agreement with a microstress theory of broadening. In α-brass the line width is found to depend on crystallographic direction in a systematic manner while with tungsten it does not. Both observations are explained on the basis of the elastic properties of the materials.

INTRODUCTION

ONE of the most striking phenomena connected with the cold working of polycrystalline metals is the broadening, in certain cases, of the x-ray diffraction lines. Thus, when well-annealed α-brass is cold worked by rolling to a 30 percent reduction in area, the width of the 420 diffraction line increases from 3×10^{-3} radian, to 30×10^{-3} radian, the width being defined as the full angular width at half-maximum intensity.

This x-ray phenomenon has received a great deal of scattered attention in the literature for a period of 25 years without a clear-cut decision being reached as to the cause of the broadening. At least two mechanisms present themselves as explanations of the broadening: the microstress theory and the particle size theory. The microstress theory pictures a system of internal stresses in the metal. In a general way these stresses are supposed to be random in direction, magnitude, and sign, and uniform only over distances very small compared with the dimensions of the sample. Such a system of stresses will produce a corresponding system of strains and hence random variations from crystal to crystal of any given interplanar spacing. Random variation of interplanar spacing will produce a broad x-ray diffraction line from the polycrystalline material. The particle size theory presumes that in the process of cold work, crystal fragmentation proceeds so far that the resulting small crystals have low resolving power. The width of the lines is then given by the Scherrer formula as $B=0.89\lambda\sec\theta/L$, where L is an average crystal dimension, λ is the x-ray wave-length, and θ is the Bragg angle.

It is difficult to show by a direct independent method either the existence in the metal of such a system of internal stresses, or the existence of crystals smaller than 2000A. This dimension is that which is just small enough to cause detectable broadening.[1] The weight of the opinion in the

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[1] B. E. Warren, J. App. Phys. **12**, 375 (1941).

literature, influenced by many indirect observations, has inclined toward the former point of view.

The x-ray observations themselves are capable of direct discrimination between these two theories, if numerous quantitative measurements are made and if their dependence on the Bragg angle θ, and the x-ray wave-length λ is examined.

Dehlinger[2] has suggested the test involving the data themselves. If the broadening is caused by small particle size, the breadth B should be proportional to sec θ or if comparable data of different x-ray wave-lengths is available, to λ sec θ. If a microstress mechanism is responsible the breadth should be proportional to tan θ and independent of x-ray wave-length, as logarithmic differentiation of the Bragg law will show.

The present work was originally an attempt to apply the Dehlinger test to α-brass. The systematic effects which are described appeared at once and the explanation in terms of elastic anisotropy presented itself. This explanation in turn suggested the experiments on tungsten. The work is discussed in the reverse order for the sake of convenience.

EXPERIMENTAL PROCEDURE

A side reflection focusing camera of 100-mm diameter was used in this work. This camera has the advantage that geometrical contributions to the line width can be computed and thus patterns, made of different materials and with different wave-lengths, can be made comparable. The camera was capable of registering effectively reflections from $\theta=30°$ to $\theta=80°$, a region which contains the majority of lines useful for the present purpose. A back reflection camera, which would be preferable from the point of view of precision, proved incapable of registering enough lines, as might be anticipated.

All patterns were made with $K\alpha$ radiation reflected from a self-focusing NaCl monochromator.[3] The use of crystal monochromated radiation is considered by the authors to be a "must" in work of this sort. It is only with such radiation that the background fluorescent scattering from the sample, caused by the continuous radiation from the x-ray tube, can be eliminated. A low background is absolutely necessary in order to secure significant width values from microphotometer readings. The self-focusing feature of the monochromator provides a wide horizontal angular distribution of radiation, all of which can be utilized in a camera of the focusing type, keeping exposures down to reasonable values. The x-ray tubes were of the common demountable hot filament type. Cu, Ni, Co, and Fe targets were used in the work on brass, while only Cu, Ni, and Fe were used on tungsten. From 400 to 1000 watts input was used, depending on the target. Exposures were about four hours. An important intensity factor with the softer radiations is the window of the x-ray tube. It was found, quite fortuitously, that the aluminium foil in which pre-war x-ray film was wrapped is satisfactory for windows. This foil is 0.0004 in. thick and is entirely without pinholes. The red paper if left on provides a convenient method for handling the foil.

The film which was used in these experiments was Eastman no-screen x-ray film. It is necessary to use the single-coated film because the x-ray beam in focusing cameras does not strike the film at normal incidence. This film, by test, has a linear density exposure relation for densities less than one. Thus the maximum density of all lines must be less than one, and further analysis of the precision of half-intensity width measurements by the photographic method showed that for best results the maximum density of the line should be held to the range of from 0.15 to 0.85. The x-ray diffraction lines used in these experiments varied in intensity, most markedly because of the multiplicity factor. In order to bring all lines on a given film to a common density level, aluminum absorbers were placed immediately in front of the film at the position of the strong lines. This procedure raised the relative intensity of the 400 and 222 lines and materially reduced the number of exposures necessary. Standard and uniform film treatment was used throughout.

The α-brass which was used here was ordinary 30 Zn 70 Cu material which was cold rolled to a 30 percent reduction in area from the well-annealed state. For use with the focusing camera a cylindrical surface on the specimen of 100-mm diameter is necessary. This surface was obtained

[2] P. Dehlinger, Zeits. f. Metallk. **31**, 231 (1939).
[3] C. S. Smith, Rev. Sci. Inst. **12**, 312 (1941).

by machining and the machined area was then etched electrolytically to a depth of about 0.005 in. The redeposition of copper could be visually detected by its color and was avoided by simple adjustment of the etching technique.

The tungsten, which was used was a section of an ingot prepared in the ordinary commercial way.[4] The ingot had been fused at the center and was obviously large grained. The diffraction pattern of the material in the condition in which it was received consisted of speckled lines. The problems of cold working the tungsten and of preparing a 100-mm diameter surface were solved simultaneously by grinding the surface with a fine 100-mm diameter wheel.

REDUCTION OF THE DATA

The films produced by the above process were microphotometered with a Hilger non-recording microphotometer of a type commonly used in spectrochemical analysis. The widest available scanning slit, approximately 0.1 mm, was used to reduce the otherwise prominent graininess fluctuations. The procedure used follows: The galvanometer deflection was adjusted to a standard δ_0 on either side of the line being measured. The line was scanned and the galvanometer deflection δ_m corresponding to maximum density was determined. The galvanometer deflection corresponding to half-maximum density was then computed by the relation $\delta_{\frac{1}{2}}=(\delta_0\delta_m)^{\frac{1}{2}}$. The two film positions corresponding to $\delta_{\frac{1}{2}}$ were then read off the scale and drum provided. Distances on the film could be read with a least count of 0.005 mm, which with lines one to six millimeters wide is more than good enough.

The measurements on each line were repeated several times with the film in slightly different positions perpendicular to the scanning direction. The reproducibility of width values on a given line on a given film was rarely worse than 1 percent. The same line measured on different films did not reproduce so well, differences of 10 percent being found. At least two and more often four and five films were available for each line. The average deviation from the mean among films carrying the same diffraction line may be set at a 5 percent maximum.

The result of the microphotometry is an average width for each line expressed in millimeters. For a 100-mm diameter camera this is quickly converted to B_m, the measured breadth expressed in radians, by dividing by 100. Consideration shows that the widening effect of oblique incidence on the film is just compensated by the correspondingly shortened specimen-film distance.

The measured breadth B_m is the result of a number of factors only one of which is cold work. These other factors are, in the present case, the natural wave-length width of the x-ray lines, the added widening caused by the presence of the α_2 component of the radiation and the width due to the finite size of the slit in the x-ray camera. In making these corrections it is important to note that they must be made as squared terms as pointed out by Warren.[1] The squared law for corrections may be verified by assuming that each element of an initial error curve distribution of intensity having width B_e is spread out into an error curve distribution having width B_c. Integration yields a new error curve distribution of width B_T where $B_T^2=B_e^2+B_c^2$. As will be noted a modified square law is valid for another type of distribution also.

The angular breadth, B_α, of an x-ray diffraction line which is due to the natural wave-length width of the α_1 component can be written $B_\alpha=2(\Delta\lambda_{\alpha 1}/\lambda)\tan\theta$, where $\Delta\lambda_{\alpha 1}$ is the half-maximum natural wave-length width which can be obtained from double crystal spectrometer data.[5]

The $\Delta\lambda_{\alpha 1}$ values all run about $\frac{1}{2}XU$ which gives a correction B_α^2 of from 1 to 10×10^{-6} radian^2. This correction is negligible compared with B_m^2 which ran from 100 to 3000×10^{-6} radian^2 but it was included for completeness.

The necessary correction $B_{\alpha 1\alpha 2}$ for the presence of the α_2 component of the doublet was determined by a graphical method developed by Warren.[6] In arriving at this correction the assumption is made that the intensity of the α_2 line is just one-half the intensity of the α_1 line. The

[4] The tungsten was obtained through the courtesy of Mr. W. P. Sykes, Cleveland Wire Works, General Electric Company, Euclid, Ohio.

[5] A. H. Compton and S. K. Allison, *X-Rays in Theory and Experiment* (D. Van Nostrand, New York, 1935), p. 745.

[6] This correction for doublet broadening, which is unpublished, was presented by B. E. Warren at the Gibson Island Symposium, July 28, 1941 in a paper entitled "Theory and Practise of Particle Size Determination."

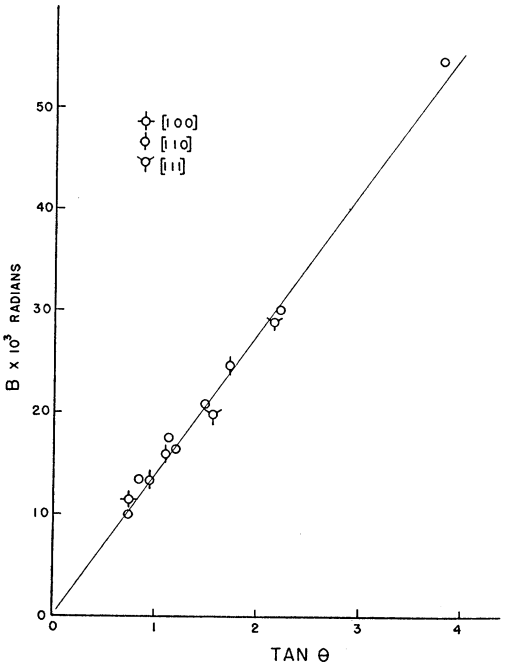

FIG. 1. Reduced breadth plotted against the tangent of the Bragg angle for tungsten. The data for the principal crystallographic directions are indicated.

lines are further assumed to possess error curve intensity distribution with some arbitrary width B_e. The separate curves are plotted a number of times with the two components separated by varying amounts S. The composite curve is drawn in each case by adding ordinates, and the width B_g at half the maximum intensity of the composite curve is measured. When the B_g^2 values are plotted against S^2 a linear relation results, giving $B_g^2 = B_e^2 + (1.225S)^2$. The necessary correction will be denoted by $B^2_{\alpha_1\alpha_2} = (1.225S)^2$ where S must be taken to be the angular separation of the doublet components

$$S = 2(\Delta\lambda_{\alpha_1\alpha_2}/\lambda) \tan\theta.$$

The final correction is then

$$B^2_{\alpha_1\alpha_2} = [2.45(\Delta\lambda_{\alpha_1\alpha_2}/\lambda) \tan\theta]^2.$$

The wave-length separation of the two doublet components is practically constant at $4XU$ for all wave-lengths. This correction is therefore large, $B^2_{\alpha_1\alpha_2}$ being roughly 20 percent of B_m^2 in all cases. It is interesting to note the $B^2_{\alpha_1\alpha_2}$ will be very much larger for short wave-length radiation such as Mo$K\alpha$ than it is for long wave-length radiation such as Fe$K\alpha$; in this case, $B^2_{\alpha_1\alpha_2}$ is 7 times larger.

It must be emphasized that this method of correcting for the α_2 component is valid only if the two components are completely unresolved, as was the case in these experiments.

The correction B_s made necessary by the finite width of the camera slit was the most troublesome one in the present work. The NaCl monochromator may be adjusted to focus the $K\alpha$ radiation to a rather narrow beam at the focal point, but within this narrow beam the α_1 and α_2 components will be relatively displaced because of the small wave-length difference. In order to make sure that the intensity of the $K\alpha_1$ line was just twice that of the $K\alpha_2$ line it was necessary to use a larger slit, about one-half millimeter wide and two millimeters high, than would have been necessary otherwise. Both the width and the height of the slit must be considered in this correction.

The height of the slit may introduce a widening on two counts, the permitted height of parallel rays and the vertical divergence allowed. It was felt that the use of the crystal monochromators and the resulting large distance between the tube focal spot and the camera slit reduced the widening due to vertical divergence to a negligible amount.

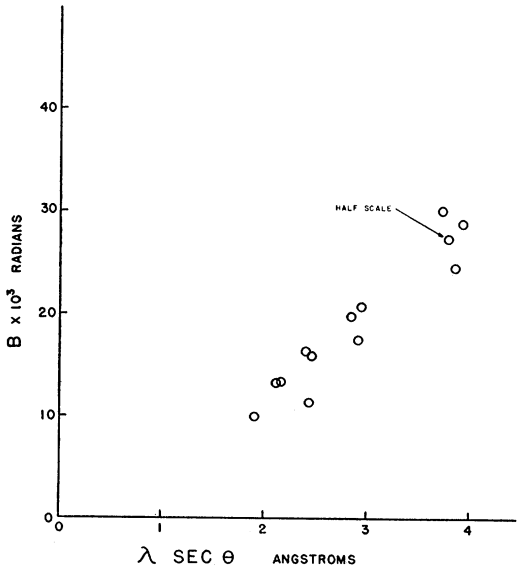

FIG. 2. Reduced breadth plotted against the function $\lambda \sec\theta$ for tungsten.

The width due to a finite height of parallel rays is important only in regions of θ near to 0° or 90°, and where the sample-film distance is small. This correction, for example, vanishes at $\theta=45°$ where angle of opening of the cone of diffracted radiation is 90°. In the side reflection camera the sample to film distances are large except in just the region $\theta \lesssim 45°$. Furthermore, this type of slit height width can usually be detected visually, the lines being wider at the ends than in the center. For these reasons the entire correction due to the height of the slit was neglected.

A correction appropriate to the width of the slit was deduced by a graphical method. This was first done for the case of the error introduced into the measurements by the finite microphotometer slit width. This problem is mathematically just the reverse of the problem for the camera slit width, and results in exactly the same form of correction. For convenience the derivation of the correction is explained in terms of the microphotometer slit width. If a diffraction line of any given form of density distribution and of width B is scanned with a slit of finite width ω, an observed density distribution will result which will be of slightly different analytical form and which will certainly have width B' greater than B. It was assumed that the lines dealt with here had the shape of error curves of width B_e. With the help of tables of $2/\sqrt{\pi}\int_0^x \exp(-x^2)dx$ the distribution which results from scanning an error curve with a slit of width ω can be computed. The new distribution can be plotted and the width B_g measured. This was done for a series of ω values and the resulting widths were plotted as B_g^2 against ω^2. A straight line of the form $B_g^2=B_e^2+0.45\omega^2$ closely agrees with the points for $\omega<0.3B_e$. This relation therefore gives $B_s^2=0.45\omega^2$.

In the case of the microphotometer slit $\omega=0.1$ mm while the measured breadth of the lines was 1 mm or greater. The maximum correction of 0.5 percent in the squared terms was neglected. In the case of the camera slit, quantities must be expressed in angular measure and ω can easily be shown to be the angle subtended by the slit at the sample. The average sample-slit distance was 50 mm. In the work on tungsten a 0.50-mm slit was used and in the work on brass a 0.40-mm slit. The squared corrections that were applied were therefore 45×10^{-6} radian^2 and 29×10^{-6} radian^2, respectively. This correction is of course a constant. The order of magnitude of this correction was checked by diffraction patterns made of large grained powdered material. It must be admitted that this correction is unsatisfying on several counts. It is a constant correction, however, and is not large enough to affect any conclusions drawn from the present experiments.

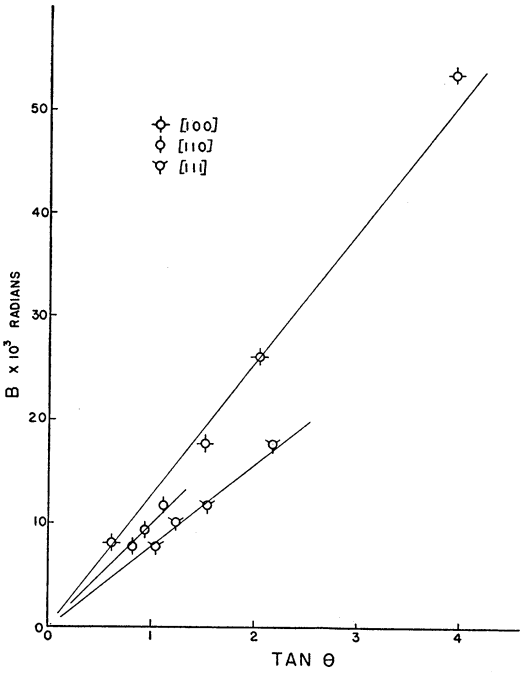

FIG. 3. Reduced breadth plotted against tan θ for the three principal crystallographic directions in α-brass.

In summary, then, the measured widths were corrected by the relation $B_m^2=B^2+B_\alpha^2+B_{\alpha_1\alpha_2}^2+B_s^2$ where B is the width due to cold work alone.

RESULTS

The reduced data for tungsten are plotted in Fig. 1 against tan θ and in Fig. 2 against λ sec θ. In Fig. 1 the data fall on a straight line drawn through the origin within ±5 percent. In the λ sec θ plot, not only is the scatter of the data noticeably greater, but any straight line presumed to represent the data would necessarily have a negative intercept on the B axis. Such a negative intercept is of course impossible physically; furthermore, of the two important corrections

made to the data, B_s is not large enough to eliminate this situation and $B_{\alpha_1\alpha_2}$, if reduced or neglected would worsen the situation. These results show that the data are proportional to tan θ and are not proportional to λ sec θ.

The data from tungsten include the following reflections: (211), (220), (310), (222), and (321) with Cu$K\alpha$ radiation; (211), (220), (310), and (222) with Ni$K\alpha$ radiation; and (200), (211), (220), and (310) with Fe$K\alpha$ radiation. The data from planes (100), (110) and (111) are indicated in Fig. 1. In the cubic system these planes are normal to the principal crystallographic directions [100], [110], and [111], respectively. It is evident from Fig. 1 that B does not depend on the crystallographic direction of the reflecting planes by an amount more than the experimental error.

The data for brass included reflections from the following planes: (311), (222), (400), (331), and (420) with Cu$K\alpha$ radiation; (220), (311), (222), (400), and (331) with Ni$K\alpha$ radiation; (220), (311), (222), and (400) with Co$K\alpha$ radiation; and (200), (220), (311), and (222) with Fe$K\alpha$ radiation. It may be noted that the (200) and (400) planes are both normal to the [100] direction. On a tan θ plot similar to Fig. 1 these data scatter considerably compared with those for tungsten, but inspection shows that the scatter is systematic depending particularly on the crystallographic direction normal to the reflecting planes. Figure 3 shows the data for the principal directions, [100], [110], and [111], while Table I shows values of $B/\tan\theta$ for all reflections. For each crystallographic direction B is proportional to tan θ to within a ± 10 percent deviation from the individual mean and the systematic trend among the various directions is unmistakable. The values of the orientation function Γ are included in Table I for reference below.

It is evident from comparison of Figs. 1, 2, and 3 that the data for α-brass, taken as a whole or taken one direction at a time, are not proportional to λ sec θ.

TABLE I. Values of $B/\tan\theta$ in units of 10^{-3} radian and of the orientation function Γ for different crystallographic directions in α-brass.

Line	Radiation	$B/\tan\theta$	Direction	Γ
200	Fe	13.09	[100]	0.000
400	Cu	11.65		
	Ni	12.72		
	Co	13.49		
Average		12.74		
420	Cu	10.91	[210]	0.160
311	Cu	11.05	[311]	0.157
	Ni	10.55		
	Co	10.72		
	Fe	11.41		
Average		10.93		
220	Ni	9.40	[110]	0.250
	Co	10.00		
	Fe	10.45		
Average		9.95		
331	Cu	9.23	[331]	0.274
	Ni	9.65		
Average		9.44		
222	Cu	7.44	[111]	0.333
	Ni	8.15		
	Co	7.55		
	Fe	8.07		
Average		7.80		

DISCUSSION

The results for cold-worked tungsten show that the breadth of the x-ray diffraction lines is proportional to tan θ and is not proportional to λ sec θ. These results are consistent with a microstress mechanism of broadening and are inconsistent with a particle size mechanism. It may be concluded on the basis of this result alone that particle size makes no significant contribution to the breadth in the case of tungsten and that the microstress picture may be accepted as a simple explanation of the facts.

The results for α-brass are similarly inconsistent with a particle size theory, but they are consistent with a microstress picture only if this picture is capable of explaining the systematic effect observed with the brass and of explaining simultaneously its absence in the case of tungsten. Such an explanation may be found in the elastic properties of the two materials.

Cubic crystals are in general anisotropic in their elastic properties. The Young's modulus, defined as the ratio of normal force per unit area to deformation in the direction of the force per unit of length in the same direction, is given by,

$$1/E = A - B\Gamma \qquad (1)$$

for a cubic crystal. In this equation A and B are constants of the material, and Γ is the orientation function given by $\Gamma = \gamma_1^2\gamma_2^2 + \gamma_2^2\gamma_3^2 + \gamma_3^2\gamma_1^2$, where

γ_1, γ_2, and γ_3 are the direction cosines of the specimen axis with respect to the cubic crystal axes.[7] For α-brass of composition 72 percent Cu, 28 percent Zn, A is 19.4×10^{-13} cm^2 dyne^{-1} and B is 41.6×10^{-13} cm^2 dyne^{-1}. The modulus in the [111] direction is then a maximum and the modulus in the [100] direction is the minimum with a ratio of $E_{[111]}/E_{[100]}=3.5$. For tungsten $A=2.573\times10^{-13}$ cm^2 dyne^{-1} and $B=0$. This latter value means that tungsten is elastically isotropic, the only known example of a crystalline material which is so.[8]

A given system of microstress in a metal will produce a system of strains, the magnitude of which will depend on the elastic properties of the material. In an anisotropic material one would expect the strain magnitude to be a function of crystallographic direction since the modulus is a function of crystallographic direction. Since the breadth of a diffraction line, from this point of view is simply a measure of the strain magnitude one would expect the breadth to be a function of the crystallographic direction, and further, inspecting Eq. (1), a function which monotonically decreases with increasing Γ. In Table I this may be seen to be the case within the errors of experiment for α-brass.

Tungsten, which was chosen for these experiments because of its elastic isotropy, shows no dependence of breadth on direction in agreement with the above point of view.

The above conclusions are semi-quantitative only, the results being only in the predicted sequence for α-brass and exhibiting no sequence for tungsten as expected. It is not possible to make quantitative conclusions from the present experiments since the details of the stress distribution are unknown. It is not correct to predict that the breadths should be quantitatively proportional to tan θ/E for an anisotropic material, where E is given by Eq. (1). The modulus in Eq. (1) is the stress strain ratio for a single crystal under a uniaxial stress, i.e., with free boundaries, which is certainly not the case for the crystals in the interior of a massive piece of cold-worked polycrystalline metal. As a simple example it may be pointed out that a single cubic crystal under hydrostatic pressure would exhibit strain which would be independent of Γ.

Some work reported in a recent letter by Stokes, Pascoe, and Lipson[9] is of interest in connection with the above. These workers have studied the breadth of diffraction lines from copper *filings* and have found that the quantity $EB/\tan\theta$ is a constant for their data, or in other words, that the breadth is proportional to tan θ/E, where E is presumably computed from Eq. (1). It is quite conceivable that Eq. (1) might apply to the crystals in a filing more rigorously than to crystals in a massive piece of metal and this is probably the explanation of their result. Moreover the present writers cannot check the modulus values quoted in this letter, finding $E_{[100]}$ high by 10 percent and $E_{[111]}$ low by the same amount. The conclusion that $EB/\tan\theta$ is constant would thus be incorrect by 20 percent. The constants A and B in Eq. (1) may have come from different sources however. In any event it seems in order to comment that experiments on the connection between elastic properties and breadth may prove a profitable way in which to study the microstress distribution as a function of the method of cold work.

Elastic anisotropy and its relation to the well-known method of x-ray stress analysis has been discussed at length in the German literature.[10,11] A definite effect is claimed but it is apparently not large enough to cause trouble in stress measurements on aluminum and steel, to which the method is usually applied.

The difference between the α-brass and tungsten found in the present experiments might very possibly be accounted for on grounds other than that of the elastic properties. The materials differ in two important respects: They are of different crystal structure and they have been cold worked by quite different methods. The writers can find no obvious explanation based on either of these two differences. Experiments with controlled crystal structure and controlled cold-work method can decide this point.

The present data for tungsten and α-brass are not strictly comparable quantitatively because

[7] E. Schmid and W. Boas, *Kristallplastizität* (Julius Springer, Berlin, 1935), p. 23.
[8] See reference 7, page 200.
[9] R. S. Stokes, K. J. Pascoe, and H. Lipson, Nature **151**, 137 (1943).
[10] R. Glocker, Zeits. f. tech. Physik **19**, 289 (1938).
[11] H. Moeller and G. Martin, Mitt. Kaiser-Wilhelm Inst. Eisenforsch, Duesseldorf **21**, 213 (1939).

TABLE II. Comparison of root-mean-square strains with maximum strains.

	$\Delta d/d$	U.T.S./E
Tungsten	12×10^{-3}	12×10^{-3}
α-brass	5	7
Permalloy	4	3

the method and amount of cold work is different in the two cases. However, since Haworth[12] has shown that the breadth rapidly approaches a limiting value with increasing amounts of cold work an attempt has been made to compare these data with those of Haworth on Permalloy. For this purpose a microstress mechanism is assumed to be responsible for the broadening in all cases and the root-mean-square strain $\Delta d/d$ is computed by help of the relation given by Haworth, $(\Delta d/d)^2=0.55(B/\tan\theta)^2$. The r.m.s. strain may be compared with the maximum strains computed by dividing the ultimate strength by the modulus. Handbook values were used in the latter case. Table II shows the results of doing this.

The rough agreement of the two columns of Table II and the order in which the $\Delta d/d$ values arrange themselves may be considered to be further support for the microstress mechanism.

It is interesting to see what conclusions result when the evidence in favor of a microstress mechanism recorded here is disregarded and a particle size theory is assumed. If the reduced breadths for tungsten are introduced directly into the Scherrer formula and an average computed from all the data the number resulting is 140A. α-brass yields a value of 180A while the data of Stokes, Pascoe, and Lipson for copper yield 310A. These values are so small that they are hardly credible.

There is other evidence that the major portion of cold-work broadening is due to strains in the metal. This evidence has been summarized elsewhere.[13] The possibility of broadening mechanisms other than the two examined here cannot be ruled out by the present results.

CONCLUSIONS

The results of measurements of x-ray line width in cold-worked tungsten and α-brass are in agreement with a microstress theory of broadening and are in disagreement with a particle size theory. The former hypothesis explains the observed dependence of the breadth on the Bragg angle and on the x-ray wave-length; it explains in a simple manner the systematic dependence of breadth on Miller indices found with α-brass and it explains the absence of this effect in tungsten; the mechanism yields reasonable quantitative results for the strains presumed to be present in the metal.

ACKNOWLEDGMENT

The authors have had several interesting discussions with Professor C. S. Barrett of the Carnegie Institute of Technology during the course of the present work. They wish to express their appreciation for his interest in these experiments.

[12] F. E. Haworth, Phys. Rev. **52**, 613 (1937).

[13] F. Niemann and S. T. Stephenson, Phys. Rev. **62**, 330 (1942).