LA-UR -83-1724

Conf-830887--1

Los Alamos National Laboratory is operated by the University of California for the United States Department of Energy under contract W-7405-ENG-36.

LA-UR--83-1724

DE83 014118

TITLE:

EVIDENCE FOR STACKING FAULTS IN MULTIAXIAL STRAINED ALPHA-BRASS

AUTHOR(S):

R.B. Roof

SUBMITTED TO:

Combined Meeting of American Crystallographic Assn. and Denver X-ray Conference, Snowmass, CO Aug, 1983

DISCLAIMER

Los Alamos National Laboratory
Los Alamos, New Mexico 87545

FORM NO. 836 R4
ST. NO. 2629 5/81

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

EVIDENCE FOR STACKING FAULTS IN MULTIAXIAL STRAINED ALPHA-BRASS*

R. B. Roof

Los Alamos National Laboratory

Los Alamos, New Mexico 87545

INTRODUCTION

As part of a program studying the effects of large strain deformations resulting from multiaxial loading to a variety of materials, a thin-walled tube (0.46" O.D. x 0.02" wall thickness) of 70-30 Brass was subjected to strain deformation in the following directions 1) along the tube axis, $\varepsilon_z = 0.3393$; 2) circumferential around the tube surface, $\varepsilon_\theta = -0.0121$; 3) perpendicular to the wall thickness, $\varepsilon_R = 0.3514$. This report describes the results of an x-ray examination of the external surface of the tube by the line-broadening technique.

EXPERIMENTAL TECHNIQUES

Examination of the shape of an x-ray diffraction line profile is a technique whereby information can be obtained concerning the condition of the material on an atomic scale. Crystallite size, lattice strain, and lattice stacking faults are items that can be obtained. Since descriptions of the general procedures usually employed are available in standard literature references[1-4] the detailed procedures will not be described further in this report. A Fourier coefficient computer program was employed to aid in the analysis of the peak shape.[5-6] A unique feature of the program is the inclusion of equations due to Wilson[5-6] to calculate standard deviations of the Fourier coefficients directly in terms of the experimental intensity expressed as counts per second. The standard deviations were further propagated (with covariance included) through the complex division necessary to arrive at Fourier coefficients free from instrumental abberations.

The experimental sample used in the x-ray diffraction examination was formed as follows. Three pieces were cut from the strained tube approximately 0.50" long in the axial direction and 0.125" wide in the peripheral direction, mounted in plastic, and metallographically polished to provide a flat surface of 0.5" x 0.5" of the external face of the tube. Similar sections obtained from an unstrained tube were used as reference material.

*Work performed under the auspices of the Department of Energy.

α-brass is a face-centered-cubic material and the lattice constant, obtained from least-squares extrapolation techniques,[7] for the strained sample is a_o = 3.6851±3 Å. For the reference material the value is a_o = 3.6840±1 Å.

For the material under examination the reflections 111 and 220 are relatively sharp while 200 and 311 display broadening. The reflections 222 and 400 have extremely weak intensities, barely above background, and reliable measurements on these reflections cannot be obtained. The reflections 331 and 420 are available but with weak intensities and they are not included in the present analysis.

A full detailed analysis of all the diffraction data was made utilizing the general procedures presented previously.[8] From this analysis it was determined that the following approximations are valid for the present investigation: the double stacking fault probability, α", is essentially zero; the residual stress, σ, is virtually nil; and the crystallite size is in excess of approximately 2000 Å, the practical upper limit for determination by x-ray diffraction techniques. These assumptions/approximations allow considerable simplifications of the equations for determining the separation of the compound stacking fault parameters from one another.

DATA ANALYSIS

The normalized unfolded Fourier coefficients ($C = (A^2+B^2)^{\frac{1}{2}}$) for the four reflections examined are plotted in Figure 1. Two general sets are observed; those coefficients belong to the relatively sharp lines 111 and 220 and those in the second set belonging to broadened lines 200 and 311.

In Figure 2 the Fourier coefficients are plotted as a function of $h^2+k^2+l^2$ for constant L, with L = 100 Å as a typical example. It is to be noted that in Figure 2 the Fourier coefficients do not fall on a single curve. The departure from a common curve is indicative of stacking faults in the material. It is desired to fit the typical data of Figure 2 to the general exponential equation $y = a.\exp(bx)$. This is usually done for the different orders of reflections from the same set of parallel lattice planes. Since no second or higher orders are available the fitting is done with the selected pairs (111,220) and (200,311). Table 1 lists the intercepts and slopes obtained from the selected pairs. Also listed is the average slope.

The root mean squared strain, ε_F^2, can be calculated from the average slope of the Fourier coefficient curve plotted as a function of $h^2+k^2+l^2$ utilizing the following equation

$$\varepsilon_F^2 = \frac{a_o^2}{L^2 \cdot 2\pi^2} \cdot (-\text{slope}) \qquad (1)$$

where a_o = 3.685 Å for α-brass.

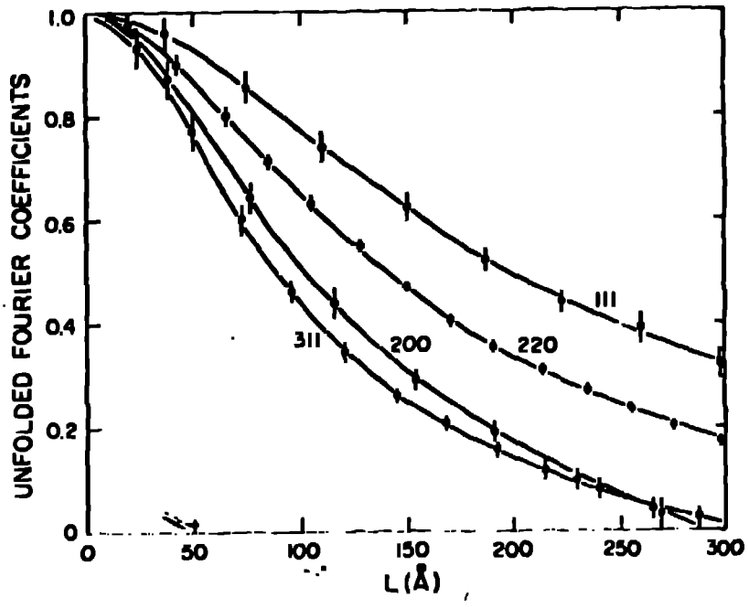

Figure 1. Normalized unfolded Fourier coefficients for four reflections for plane strained alpha-brass.

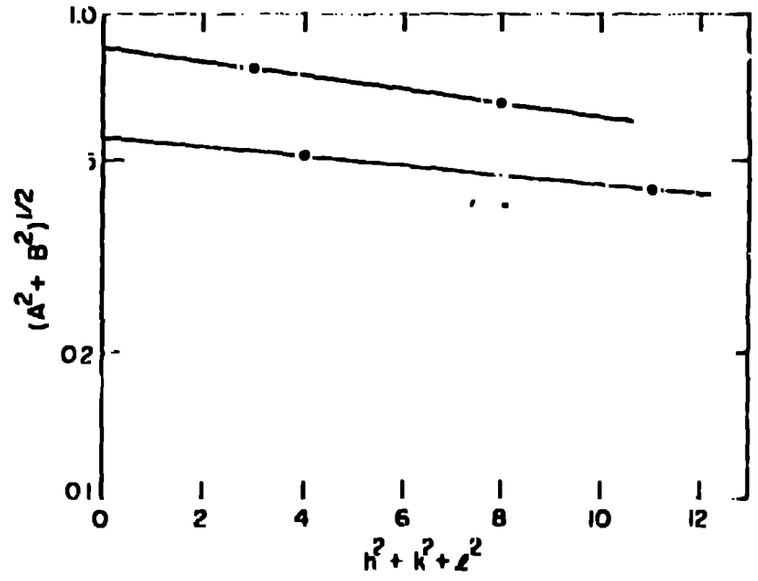

Figure 2. Logarithmic plot of normalized unfolded Fourier coefficients from plane strained alpha-brass. Results for L = 100 Å as a typical example.

Table 1. Intercepts and slopes as a function of L for plane stained alpha-brass. Obtained by fitting data of Figure 1 to the general equation $y = a.\exp(bx)$

L, (Å)	(111,220) Intercept	(111,220) Slope	(200,311) Intercept	(200,311) Slope	Average Slope
25	0.992	-0.00412	0.962	-0.00304	-0.00358
50	0.968	-0.01334	0.840	-0.00910	-0.01122
75	0.921	-0.02488	0.702	-0.01737	-0.02113
100	0.852	-0.03388	0.559	-0.02272	-0.02830
125	0.800	-0.04680	0.443	-0.02533	-0.03607
150	0.742	-0.05700	0.343	-0.02841	-0.04271
175	0.671	-0.06621	0.265	-0.03037	-0.04829
200	0.621	-0.07906	0.190	-0.02773	-0.05340
225	0.571	-0.08680	0.133	-0.02604	-0.05645
250	0.533	-0.09965	0.076	-0.02202	-0.06084

The strains determined in this manner are plotted in Figure 3 as a function of L. From the variation in Figure 3 it is estimated that the local strain is equal to 0.00225 at L = 0.

The Fourier crystallite size coefficients, the intercepts of Table 1, are plotted as a function of L in Figure 4. The intersection on the L axis of a line drawn through the linear portion of the curve yields an effective crystallite size, D_e. For the pairs in Figure 4, D_e = 450 Å for (111,220) and 200 Å for (200,311).

The separation of compound stacking faults and true crystallite size from the effective crystallite size is accomplished by plotting $1/D_e$ as a function of V(hkl). V(hkl) is a constant for a given value of hkl and reflects the contribution to the total observed line profile of the various signed permutations of hkl that are affected by stacking faults. Tables of V(hkl) are available.[2-3]

In Figure 5 $1/D_e$ is plotted versus V(hkl) for 111 and 200, the first hkl's of the selected pairs of reflections. The intercept in Figure 5 is the reciprocal of the true crystallite size, D; by fixing it at zero we satisfy the assumption that the crystallite size exceeds 2000 A. The slope of Figure 5 is equal to $(1.5\,\alpha' + 1.5\,\alpha'' + \beta)/n$ where α' = the probability of finding a single stacking fault between neighboring 111 planes; α'' = the double stacking

fault probability; and β = the twin fault probability. With α'' assumed to be negligible the slope reduces to $(1.5\ \alpha' + \beta)/a_o$. From Figure 5 the value of $(1.5\ \alpha' + \beta) = 0.01850 \pm 12$.

A value for a stacking fault combination has been obtained and a second value from a different equation is needed to separate the stacking faults. The lattice parameter, a_{hkl}, calculated from a peak position of the individual hkl reflections of fcc materials depends on the true lattice parameter, a_o, the difference $(\alpha' - \alpha'')$, the residual stress, σ_2, and the geometrical abberations of the diffractometer. It has been shown[2] that these quantities are related according to

$$a_{hkl} = a_o + (S_1)_{hkl}^{A} \cdot a_o \cdot \sigma + G_{hkl} \cdot a_o \cdot (\alpha' - \alpha'') + m.f(\theta) \qquad (2)$$

where $(S_1)_{hkl}^{A}$ and G_{hkl} are constants which depend on the planes hkl. Tables of G_{hkl} are available.[3-4] With σ and α'' assumed to be negligible equation (2) reduces to

$$a_{hkl} = a_o + \alpha' \cdot a_o \cdot G_{hkl} + m.f(\theta) \qquad (3)$$

or

$$y = P_1 x_1 + P_2 x_2 + P_3 x_3$$

where $x_1 = 1.0$, $x_2 = G_{hkl}$, and $X_3 = \cos\theta\cot\theta$ for diffractometer focusing geometry. Values for a_{hkl} (i.e., y) were calculated from 2θ values representing the centroids of the observed experimental intensity data. Values for the remaining terms (i.e., x_j, $j = 1,3$) were calculated or taken from published tables.[3-4] The x,y values are elements of a matrix and these are arranged in an array in Table 2.

Table 2. Matrix elements for the solution of equation(3) for experimental values obtained from plane strained alpha-brass.

hkl	y (Å)	x_1	x_2	x_3
111	3.68635	1.0	-0.035	2.398
200	3.68850	1.0	0.069	1.974
220	3.68594	1.0	-0.035	1.099
311	3.68578	1.0	0.013	0.748

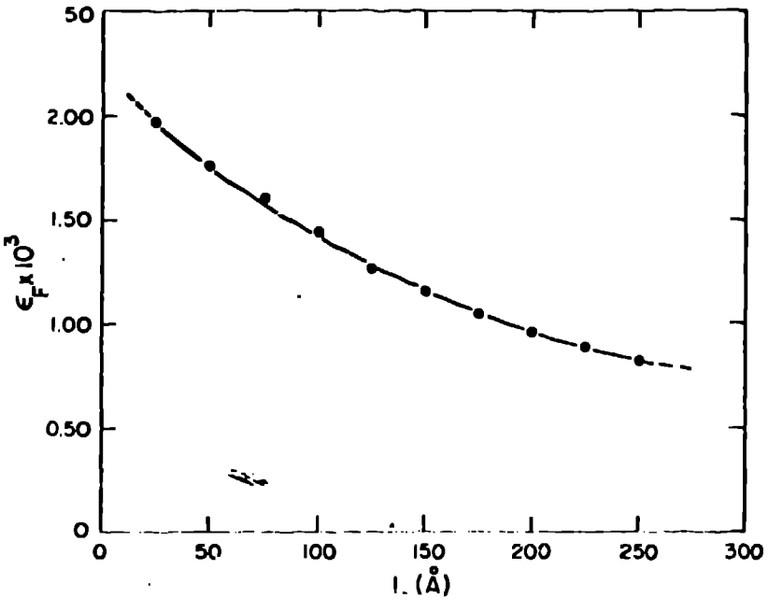

Figure 3. Variation of strain, ϵ_F, as a function of L for plane strained alpha-brass.

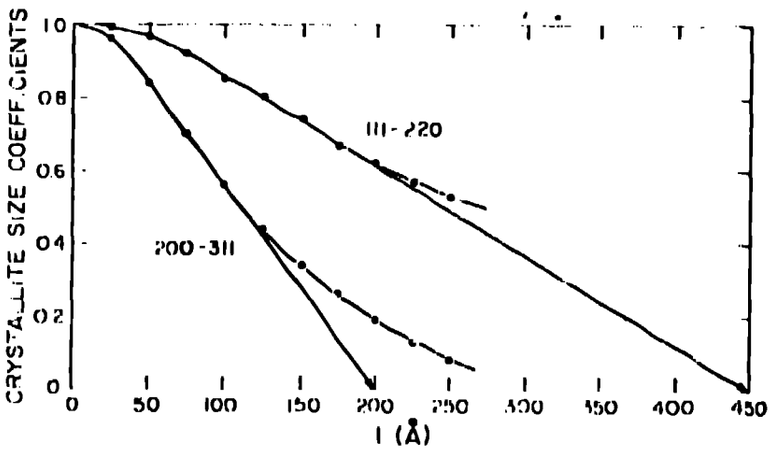

Figure 4. Crystallite size via the Fourier coefficient technique for plane strained alpha-brass.

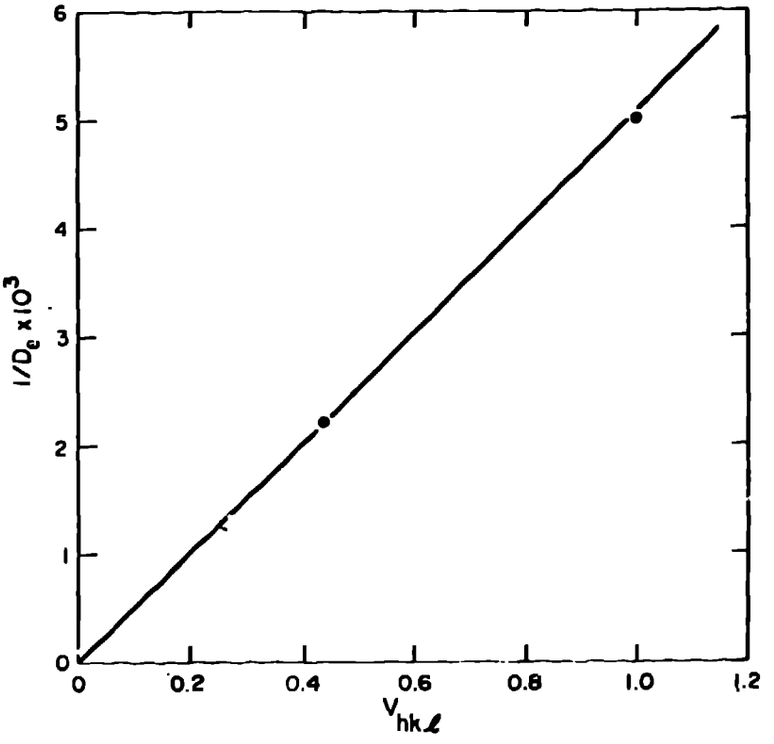

Fig. 5. Separation of true crystallite size and compound stacking fault parameter from effective crystallite size. See text.

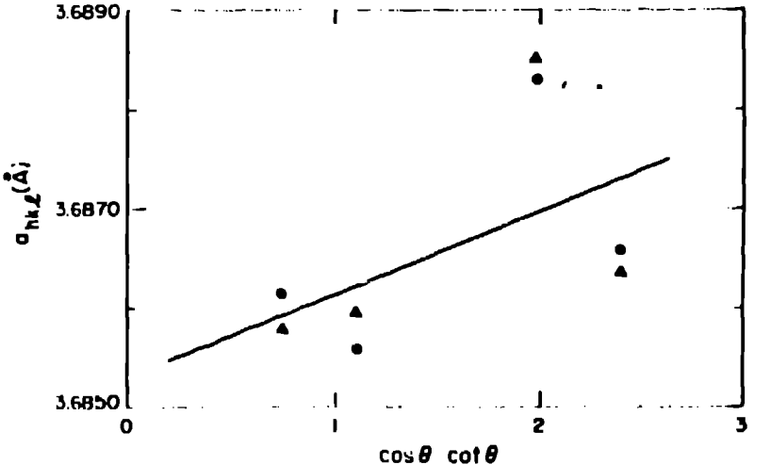

Figure 6. Calculated lattice constant, a_{hkl}, versus $\cos\theta\cot\theta$ for plane strained alpha-brass. ▲ = experimental; ● = effect of $\alpha' = 0.005$.

The data presented in Table 2 is usually plotted as y versus x_3 with deviations from a straight line representing the contributions of stacking faults of the lattice parameter (see Figure 6). Using the data of Table 2 and solving equation (3) by least-squares techniques results in P_1 = 3.6853±8, P_2 = 0.020±8, and P_3 = 0.00083±50.

The stacking fault parameter of interest, α', is obtained from the ratio, P_2/P_1, and equals 0.0054±22. From $(1.5\,\alpha' + \beta)$ = 0.0185±1, β is calculated to be 0.0104±22.

CONCLUSIONS

Two different techniques of X-ray diffraction have been applied to the examination of multiaxial strained alpha-brass. From an examination of the unfolded Fourier coefficients describing the shape of the diffraction profile it has been determined that the true crystallite size probably exceeds 2000 Å, a practical upper limit for determining crystallite size by x-ray methods. The localized strain is approximately 0.225% and a combined stacking fault probability $(1.5\,\alpha' + \beta)$ = 0.0185±1.

From lattice constant variations the single stacking fault probability, α', has been determined to be 0.0054±22. The twinning stacking fault, β, is thus 0.0104±22. The reciprocal of the probability is the number of planes of atoms between the indicated stacking fault. The magnitude of the numbers indicates that twinning is twice as common as single stacking faults.

REFERENCES

1. H. P. Klug and L. E. Alexander, "X-ray Diffraction Prcoedures", (Wiley, New York, 1954), p. 491.

2. C. N. J. Wagner, in "Local Atomic Arrangements Studied by X-ray Diffraction", edited by J. B. Cohen and J. E. Hilliard, (Gordon and Breach, New York, 1966), p. 219.

3. B. E. Warren, "X-ray Diffraction", (Addison-Wesley, Reading, Massachusetts, 1969), p. 251.

4. N. C. Halder and C. N. J. Wagner in "Advances in X-ray Analysis", Vol. 9, (Plenum Press, 1966), p.91.

5. A. J. C. Wilson, Acta Cryst. 23 (1967), p. 888.

6. Idem, Ibid A25 (1969), p. 584.

7. R. E. Vogel and C. P. Kempter, Acta Cryst. 14 (1961), p. 1130.

8. R. B. Roof and R. O. Elliott, J. Mat. Sci. 10 (1975), p. 101.