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# X-Ray Measurement of Residual Strains in Individual Grains of Polycrystalline Aluminum

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Shifts in peak position of {333} and {511} diffractions of cobalt $K\alpha_1$ x rays from individual grains of coarse-grained polycrystalline aluminum observed in the annealed condition and after 10 percent plastic extension revealed residual strains in each crystallite. These strains, however, did not conform to the strain quadric with a principal axis parallel to the axis of deformation, as is the case of observations from fine-grained metallic specimens that have been plastically deformed; nor was any consistency or meaningful average trend observed in the strains of the various grains. Irregularities of loading constraints by one grain upon its neighbors and the resulting great nonuniformity of deformation may account for the absence of systematic results.

## 1. Introduction

The angle of x-ray diffraction $\theta$ is related to the spacing $d_{hkl}$ between layers of atoms in a crystalline solid through Bragg's law,

$$\lambda=2d_{hkl}\sin\theta, \tag{1}$$

where $\lambda$ is the x-ray wavelength. The use of the shift of the diffraction angle as an indication of strain in the lattice structure of the solid is more than 30 years old. One of the first observations of this type was made by Lester and Aborn [1][1] in 1925 on the change of spacing of crystalline planes in steel subjected to stress. A comprehensive review article concerned with x-ray strain measurement as well as other aspects of quantitative x-ray diffraction observations on strained metal aggregates was published by G. Greenough [2] in 1952. Perhaps the most interesting aspect of these strains in the crystal lattice structure is the residual elastic strain observed in a metal specimen that has been plastically deformed and then unloaded. Recently the various theories attempting to explain these residual strains and stresses measured by x-ray diffraction have been evaluated by Vasil'ev and Smirnov [3] in 1961 in a review article discussing a variety of x-ray diffraction methods of investigating cold-worked metals.

It is generally accepted that the residual stresses arise on account of differences of "hardness" or resistance to plastic flow in various regions of the material. After the release of a uniform uniaxial deforming stress of a given sign, the weaker regions A will be constrained into a state of stress of the opposite sign by the greater amount of elastic strain recovery in the stronger regions B. Although the microscopic nature of these two regions has not been clearly determined, the model that seems to be most widely accepted today is based upon ideas first advanced by Smith and Wood [4]. They suggested that the soft regions A and the hard regions B are regions of low and high lattice structure distortion, respectively. This hypothesis has been supported by the observations of many recent workers [5, 6, 7], although there is some evidence that more than one mechanism may be contributing to the observed strains and stresses under certain circumstances [8]. The original idea that the distorted harder regions B were at the grain boundaries has gradually been generalized to include all regions of high dislocation density, such as slip planes, subgrain boundaries, and the dislocation tangles that constitute cell walls observed in some deformed metals [9]. Since the x-ray diffraction peak position is determined principally by the more perfect A material, the peak shift represents the elastic strain and the related stress in that material only.

Related to the question of the source of the residual elastic strains and stresses in the polycrystalline metal is the paradox of their observed quasi-isotropic behavior. The strains measured on a given surface are observed to satisfy the equation of a strain quadric, with one of the principal strains parallel to the axis of plastic deformation. This feature is implicit in most of the reports of this type of measurement and has occasionally been explicitly verified [10]. Most workers agree, moreover, that in practice it is permissible to use the gross average values of Young's modulus and Poisson's ratio as obtained from mechanical tests on polycrystalline specimens free of preferred crystallite orientation [11, 12] to relate the strains to a system of stresses using isotropic elastic theory; and there seems to be no question that the net observed behavior in the x-ray "powder" diffraction effects from the aggregate of individual anisotropic crystallites is itself isotropic.

There are at least two possible explanations for this isotropic behavior on the part of the diffracting regions A of the grains. First, these regions may be so constrained by their randomly oriented neighboring grains and by the hard, quasi-amorphous B material at grain or subgrain boundaries that the

[1] Figures in brackets indicate the literature references at the end of this paper.

strains are forced into an isotropic pattern relating to the applied deformation. Alternately, although the distribution of strain in any one crystallite might be itself quite anisotropic and unrelated to the geometry of the preceding deformation of the gross specimen, the strain indicated by the shift in the diffraction line, coming typically from hundreds of crystallites, might represent a nonzero average that exhibits the isotropic behavior.

The principal line of attack in the present investigation was to measure the shift in the Bragg angle of diffraction from individual crystallites in a coarse-grained polycrystalline specimen that had been plastically deformed in tension. The purpose of the study was to see if there was an impressed residual stress-strain system, if it was isotropic with principal axes determined by the external deformation, as is the case with ordinary fine-grained material, and what the magnitude of the residual strain might be. It was hoped that such an investigation might throw some light on the alternate hypotheses of pseudo-isotropic behavior of the strains and possibly lead to further studies that might reveal some grain-size effects.

## 2. Experimental Procedures

The specimen was of 99.99 percent pure aluminum with threaded ends and a reduced section about 1¼ in. long with a square cross section ½ in. on a side. The specimen was supplied in a fully annealed, stress-free condition, surface etched, with grains ranging in mean diameter from about 1/16 in. to ¼ in., grown by the strain-anneal method. Prior to examination the specimen was further annealed for 24 hr at 150 °C and furnace cooled. The specimen in its initial condition may be seen in figure 1, a and b.

The x-ray diffraction measurements were obtained by a combination of film and counter methods on a commercial x-ray diffraction apparatus, employing auxilliary equipment designed and built at the National Bureau of Standards. The first step of the procedure was the determination of the crystallographic orientation by means of back-reflection Laue diffraction patterns of all the grains in the central ½ in. of each of the four faces of the reduced section of the specimen. The number of grains so oriented, countings duplicates around specimen edges twice, was 51. All of the {111} and {511} plane normals were located on the stereographic projection of the pattern from each grain, and the angular coordinates, azimuth $\alpha$ and co-altitude $\psi$, for all such poles within approximately 65° of the normal to the surface being studied were measured on a Wulff net and recorded.

After the determination of orientation of each grain, the specimen in its special holder, which may be seen in figure 2, was transferred to the diffractometer. By means of a collimator sighting adjustment, the surface of the specimen was placed in coincidence with the common vertical axis of the diffractometer and the holder; and while the specimen was observed with a low-powered microscope, a desired grain was translated into the incident collimated x-ray beam about 1 mm in diameter. The proportional counter was set at the expected $2\theta$ diffraction angle, which was 162.50° for both the {511} and {333} planes, for copper $K\alpha_1$ radiation. The alpha doublet was well resolved in all cases. The two angular adjustments on the specimen holder were then set, corresponding to $\alpha$ and $\psi$, in order to

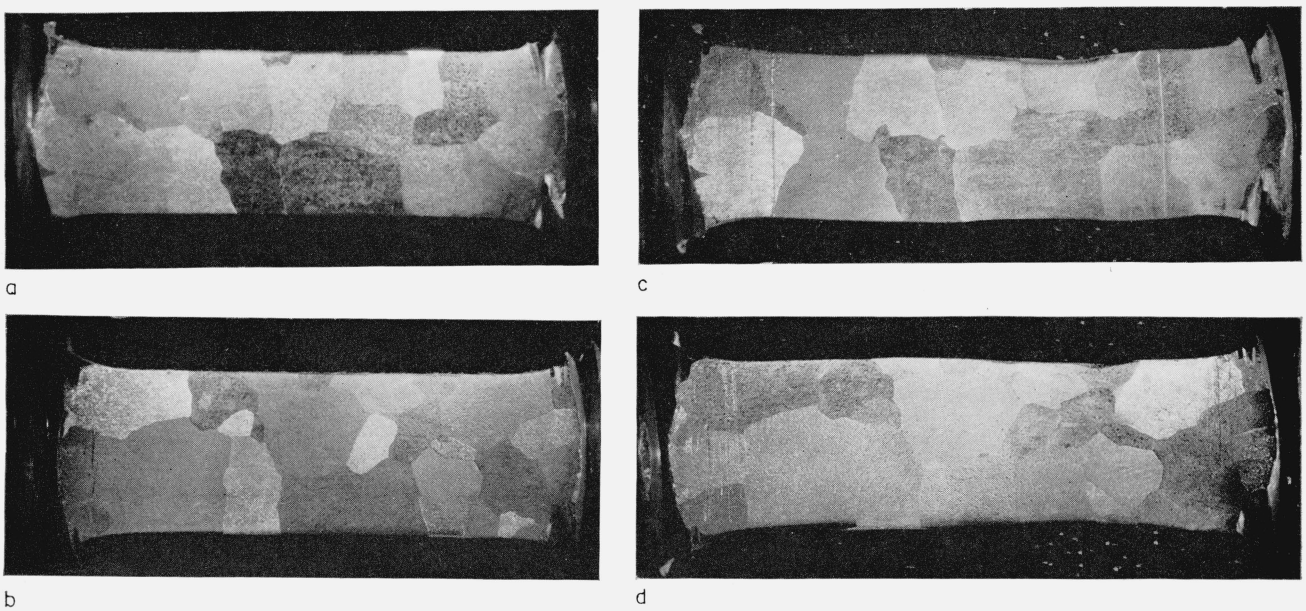


FIGURE 1. *Aluminum Specimen, Magnification 2 ×.*

a. Face A annealed condition.

b. Face B, annealed condition (at right angle to Face A).

c. Face A, after 10 percent plastic extension

d. Face B, after 10 percent plastic extension.

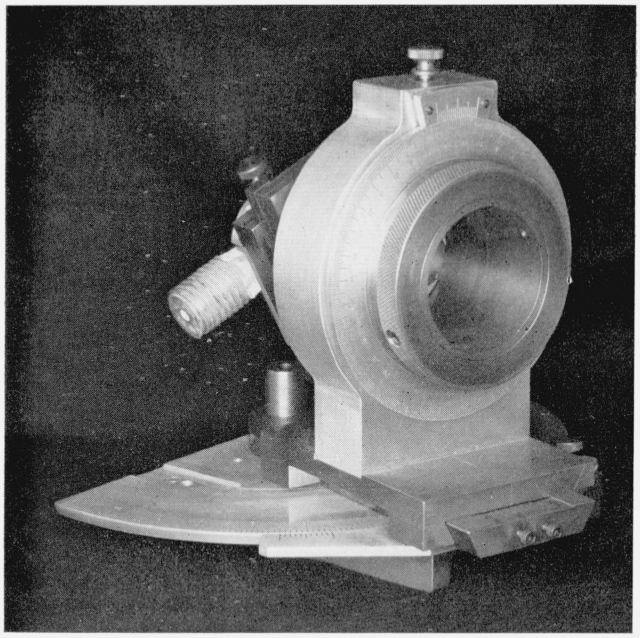

FIGURE 2. *Goniometer specimen holder for use on the x-ray diffractometer.*

place the desired pole of a diffracting plane in the horizontal plane of the diffractometer and in the position of bisector of the angle between the incident and diffracted rays. In order to minimize defocusing effects, the surface normal was always tilted away from the detector. All three angular adjustments were then "fine tuned" to give a maximum signal in the counter. The counter was then backed up a few hundredths of a degree and then "step-scanned" across the top of the diffraction peak. The steps were 0.01 of a degree apart and were held for a fixed time interval; the intensity in total counts was printed out at the end of each interval.

After the reference peak values of $2\boldsymbol{\theta}$ had been determined for all poles of interest in the specimen in the annealed condition, the specimen was strained in uniaxial tension at 23 °C to a final true strain of 10 percent. The cross-head speed for most of the deformation, including the latter part, was held at 0.001 in. per minute. The flow stress at the 10 percent plastic true strain was found to be approximately 4080 psi. At this strain, this coarse-grained specimen showed, as may be seen in figure 1 c and d, considerable inhomogeneity of strain, more than is usually the case with fine-grained material, but less than is typical with deformed single crystals.

After the prescribed plastic strain, the specimen was realined in the diffractometer and the peak $2\theta$ values were redetermined for all of the {511} and {333} planes in the region of interest on two of the four faces. Since the diffraction peaks were somewhat broadened and considerably reduced in height after the deformation, the steps in the scanning were now spaced 0.02° apart and the time intervals of counting considerably lengthened. The peak position was determined analytically by a three-point parabola-fitting equation, with a precision estimated to be ±0.01° or better. The $\theta$-dependent corrections of the intensity often used in this type of peak determination were examined for a few cases in this study, but were not used, being negligible because the distance between first and last step positions of $2\theta$ on each side of the apex of a peak was only 0.04° in these single crystal diffractions, as compared to the several tenths or even whole degrees involved in the case of polycrystalline diffraction. Before the changes in $2\theta$ going from the annealed to the strained state were calculated, however, the individual values were corrected for the effects of thermal expansion from the temperature of measurement to a standard 25 °C. The handbook value of the coefficient used was $23.8\times10^{-6}$ per degree C for the lattice constant. This resulted in a temperature correction in the Bragg angle, in degrees, at $2\theta$ equal 162.50°, of

$$\delta(2\theta^\circ)=(-0.0177)\delta T, \tag{2}$$

where $\delta T$ is the difference in temperature in degrees C from the reference temperature.

If the measured strain is small, as it was in these cases, it is not necessary to calculate values of $d_{hkl}$, the lattice plane spacing, from the observed Bragg angles; it is more convenient simply to use $\Delta(2\theta)$, the change in Bragg angle, since it is directly proportional to the strain through the following equation:

$$\epsilon=\frac{\Delta d}{d}=-\frac{\cot\theta}{2}\Delta(2\theta)=(-1.343\times10^{-3})\Delta(2\theta^\circ). \tag{3}$$

The angle $\theta$ is approximately 81.25° in the case we are examining. Since only changes in Bragg angle need be observed, the question of absolute calibration of the diffractometer is avoided. For the sake of simplicity and directness, $\Delta(2\theta^\circ)$ values rather than actual strains are used throughout this paper.

If the uncertainty in a given $2\theta$ reading is ±0.01°, as estimated, the uncertainty in $\Delta(2\theta)$ should be about ±0.014°; hence the uncertainty in a strain calculated by equation (3) would be $\pm1.5\times10^{-5}$.

## 3. Results

On the two faces of the specimen, shown in figure 1, for which complete post-strain data were taken, changes in $2\theta$ were measured for an average of about nine planes on each of 25 grains. The data for two typical grains on Face A are tabulated in table 1. The $\Delta(2\theta)$ values, and hence the strains, are very small, but in most cases they are several times the estimated uncertainty in the measurement.

As stated in the introduction, residual strains measured by x rays on conventional polycrystalline material that has been plastically strained uniaxially satisfy the strain quadric equation

$$\epsilon=a_1^2\epsilon_1+a_2^2\epsilon_2+a_3^2\epsilon_3, \tag{4}$$

TABLE 1. *X-ray strain data (i.e., diffraction peak shift) from two typical grains*

| Grain | *hkl* | Annealed | | | | | Extended | | | | | $\Delta(2\theta^\circ)$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $\alpha$ | $\psi$ | $T$(°C) | $2\theta_T$ | $2\theta_{25^\circ}$ | $\alpha$ | $\psi$ | $T$(°C) | $2\theta_T$ | $2\theta_{25^\circ}$ | |
| 1 | 333 | 169.8 | 22.2 | 24.1 | 162.43 | 162.41 | 168.9 | 21.6 | 24.0 | 162.52 | 162.50 | +0.09 |
| | 333 | 323.9 | 49.9 | 24.1 | 162.44 | 162.42 | 324.1 | 50.2 | 23.6 | 162.51 | 162.49 | .07 |
| | 15$\bar{1}$ | 271.7 | 48.3 | 24.3 | 162.44 | 162.43 | 271.3 | 46.5 | 24.1 | 162.52 | 162.50 | .07 |
| | 151 | 241.8 | 40.7 | 24.2 | 162.45 | 162.44 | 241.8 | 40.0 | 24.5 | 162.53 | 162.52 | .08 |
| | 5$\bar{1}$1 | 64.3 | 47.1 | 24.4 | 162.46 | 162.45 | 64.3 | 47.7 | 27.7 | 162.48 | 162.53 | .08 |
| | 511 | 58.3 | 25.2 | 24.5 | 162.45 | 162.44 | 58.5 | 25.8 | 26.0 | 162.49 | 162.51 | .07 |
| | 5$\bar{1}\bar{1}$ | 37.1 | 54.1 | 24.5 | 162.45 | 162.44 | 38.2 | 54.9 | 26.1 | 162.44 | 162.46 | .02 |
| | 51$\bar{1}$ | 18.8 | 35.8 | 24.6 | 162.46 | 162.45 | 19.1 | 36.3 | 26.4 | 162.45 | 162.47 | .02 |
| | 115 | 154.9 | 60.2 | 24.8 | 162.43 | 162.43 | 154.4 | 60.0 | 26.4 | 162.49 | 162.51 | .08 |
| 2 | 333 | 64.1 | 47.8 | 23.2 | 162.52 | 162.49 | 66.4 | 47.8 | 22.5 | 162.54 | 162.50 | .01 |
| | 33$\bar{3}$ | 154.7 | 59.4 | 23.4 | 162.50 | 162.47 | 156.4 | 60.0 | 23.0 | 162.50 | 162.46 | −.01 |
| | 3$\bar{3}$3 | 237.4 | 62.8 | 23.6 | 162.51 | 162.49 | 242.2 | 64.7 | 23.6 | 162.65 | 162.63 | .14 |
| | 333 | 324.5 | 50.4 | 23.8 | 162.52 | 162.50 | 324.0 | 48.6 | 23.8 | 162.43 | 162.41 | −.09 |
| | 511 | 88.0 | 9.7 | 24.5 | 162.49 | 162.48 | 81.0 | 4.3 | 24.3 | 162.48 | 162.47 | −.01 |
| | 51$\bar{1}$ | 169.2 | 21.3 | 25.0 | 162.49 | 162.49 | 182.8 | 20.9 | 24.6 | 162.51 | 162.50 | .01 |
| | 5$\bar{1}\bar{1}$ | 229.4 | 23.1 | 25.1 | 162.50 | 162.50 | 229.2 | 25.4 | 25.1 | 162.58 | 162.58 | .08 |
| | 5$\bar{1}$1 | 299.5 | 13.1 | 25.4 | 162.47 | 162.48 | 293.6 | 20.1 | 25.1 | 162.52 | 162.52 | .04 |

where $\epsilon$ is the strain measured in some direction whose direction cosines are:

$$a_1=\sin\psi\cos\varphi$$

$$a_2=\sin\psi\sin\varphi$$

$$a_3=\cos\psi$$

and $\epsilon_1$, $\epsilon_2$, and $\epsilon_3$ are the principal strains, in the orthogonal directions identified in figure 3 with respect to the geometry of the specimen and its deformation. The instrumental azimuth angle $\alpha$ in this study was related to the usual coordinate $\varphi$ by

$$\varphi=270^\circ-\alpha$$

also illustrated in this figure. The direction cosines in terms of $\psi$ and $\alpha$ were

$$a_1=-\sin\psi\sin\alpha$$

$$a_2=-\sin\psi\cos\alpha$$

$$a_3=\cos\psi.$$

The direction cosines were calculated for all the directions in which the strains were measured in the two grains referred to in table 1, and selected sets of three simultaneous equations were set up from which sets of three principal strains were computed for a particular form of planes within each grain. In no case, however, was even an approximately consistent set of principal strains with this preassigned orientation found.

In the isotropic analysis of strains in fine-grained material, a plot of strain versus $\sin^2\psi$ is found to be linear when the directions of measured strain are confined to a plane normal to the surface of the specimen [7]. In an attempt to find analogous "cooperative" behavior from the coarse-grained material, two plots of $\Delta(2\theta)$ versus $\sin^2\psi$ were prepared for each of the two faces A and B. By restricting $\alpha$ to $90^\circ\pm10^\circ$ and $270^\circ\pm10^\circ$, values of strain from very many grains were measured for various $\psi$ values close to a longitudinal plane ($x_1x_3$) normal to the surface and parallel to the axis of deformation. By restricting $\alpha$ to $0^\circ\pm10^\circ$ and $180^\circ\pm10^\circ$, similar data were obtained near a transverse plane ($x_2x_3$). These four plots of $\Delta(2\theta)$ versus $\sin^2\psi$ are shown in figure 4. There does not appear to be any relationship of the strain to $\sin^2\psi$ in any of the four cases examined. Indeed, no self-consistency or general trend or "average" behavior among the grains was anywhere in evidence among the 229 strain determinations on the two faces of this specimen.

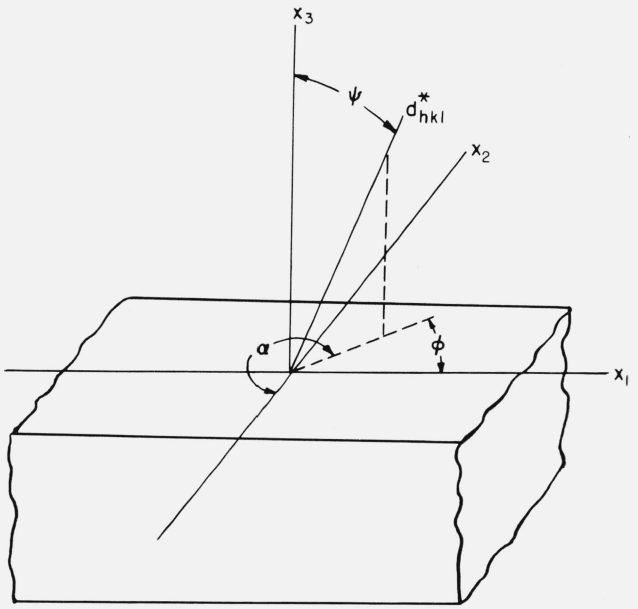


FIGURE 3. *Diagram illustrating directions of axes and defining angles with respect to the geometry of the specimen and its deformation.*

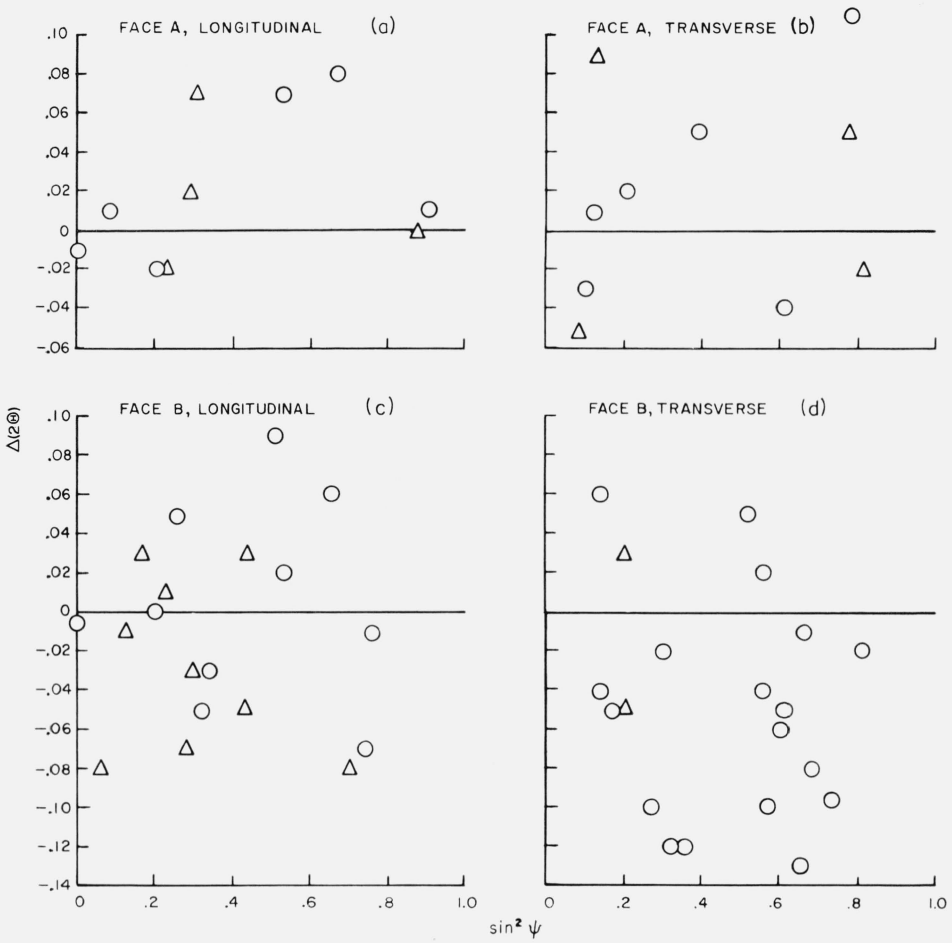


FIGURE 4. *Plots of Strain in Terms of* $\Delta(2\theta)$ *versus* $\sin^2\Psi$.

a. Longitudinal Plane Normal to Face A.
b. Transverse Plane Normal to Face A.
c. Longitudinal Plane Normal to Face B.
d. Transverse Plane Normal to Face B.
Circles—{511} diffraction data.
Triangles—{333} diffraction data.

## 4. Discussion

The results of this investigation show that it is possible to detect directed residual strains by the peak shift of x-ray diffractions in single crystals within a coarse-grained polycrystalline aggregate that has been plastically deformed in tension. Even when the material, however, is aluminum, a metal which is not so strongly anisotropic as many others, these individual crystallite strain values do not conform to the type of isotropic elastic behavior observed with ordinary fine-grained polycrystalline material after plastic deformation; at least such was the case for the specimen studied here.

It may be assumed that the strain data from any one of the grains in this study could be subjected to a rigorous anisotropic elastic analysis, such as that of Imura, Weissman, and Slade [13] in their work with divergent beam diffraction from single crystals. It is doubtful, however, that the information return in this case of highly irregular loading constraints would justify the involved computations. Perhaps such an analysis of residual strains measured by x rays in plastically deformed specimens that were true single crystals would yield meaningful information. The author is not aware that results of this type have as yet been published.

It is interesting to consider whether the anisotropic result obtained with the coarse-grained material in this study is more consistent with the "constraint" hypothesis or the "averaging" hypothesis of the isotropic behavior of the fine-grained material. The change in grain size involved is from that of a few millimeters in the present case to a few hundredths of a millimeter or less in the typical fine-grained case. This change of scale is relevant to the consideration of either hypothesis. It will change drastically the ratio of the volume of soft A-type regions, discussed in the Introduction, to the volume of hard B-type regions, if the regions near grain

boundaries are of paramount importance to the latter. This consideration, along with the change of average distances over which forces would act, should account for marked changes in behavior with size if the "constraint" hypothesis is valid. On the other hand, it must be admitted that the "averaging" of strain behavior is also improved in the statistical sense when the grain size is reduced by two orders of magnitude. However, one might have expected, in this case, that even a relatively small sampling of 16 grains, as on Face B of our specimen, might have revealed some trace of a consistent trend, and this was not the case. It is believed, therefore, that, while neither hypothesis is clearly tested by the results of this investigation, the picture of the effect of constraints upon the diffracting material when the grain size is small is the more favored one.

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