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X-RAY STUDY OF RADIATION DAMAGE

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Introduction

This report describes work done between July 1, 1953 and January 31, 1954 at the Massachusetts Institute of Technology on Contract AT(30-1)-858 for and the X-Ray Study of Radiation Damage. The aim of this program is to develop x-ray diffraction methods for measuring and evaluating the nature of damage in materials, and to apply these methods to typical metals, alloys and inorganic compounds.

I. Publication of Technical Reports

Report NYO-3733 "Measurement of Stacking Faults in Cold Worked Alpha Brass" was published in the Journal of Applied Physics, 24, 951, July 1953.

In collaboration with C. J. Dienes of Brookhaven National Laboratory "X-Ray Measurement of Radiation Damage in Black Phosphorus" was published in the Journal of Applied Physics, 24, 1251, Sept. 1953.

Report NYO-3735 "Monochromatic X-Rays for Single Crystal Diffuse Scattering" has been submitted for publication in the Journal of Applied Physics.

II. Radiation Damage in Single Crystals of LiF

Measurements were made on cleavage slabs irradiated two days and eight days in the Argonne pile. Irradiation produced an increase of about 50 percent in the integrated intensity of 002, but within experimental error, there is no observable change for 004, 006, 008 and 0010. The increase for 002 is presumably a reduction in extinction.

In a previous report NYO-3734, it was stated that the (00ℓ) peaks shift toward small angle corresponding to an average expansion, and that the fractional change in sin θ is greatest for 002, decreases with increasing order and then increases again for 0010. It now appears that this effect was due to an incomplete correction for the shift toward small angle resulting from the low absorption in the irradiated sample. When properly corrected, the peak shifts give an expansion $+0.012$ percent, but the peak shifts are so small that the values are scarcely outside of the experimental error.

The peak breadths have been expressed in terms of their Fourier coefficients and instrumental broadening corrected by the Stokes' method. Using log plots to separate the particle size and the distortion broadening, particle size values of 500 A or greater were obtained, indicating that particle size broadening was very small. The peak breadths expressed in terms of 2θ were closely proportional to tan θ. In terms of a length L perpendicular to the (00ℓ) planes, a distribution function $P(\Delta L)$, for the change in L due to the distortion, is synthesized from the Fourier coefficients. Allowing for the small expansion 0.012 percent, the functions are symmetrical, corresponding to equal probability of expansion or compression. The function $P(\Delta L/L)$ is independent of L, and the root mean square strains $\langle \epsilon^2 \rangle^{1/2} = \langle (\Delta L/L)^2 \rangle^{1/2}$ are independent of the length L over which they are averaged up to values of L = 120A. The root mean square strain values were 0.0035 and 0.0044 for the 2 day and 8 day irradiations.

The constant values of the strains over distances as great as L = 120 A indicate that the distortion cannot be due to isolated vacancies or interstitials. Rather large regions of the crystal must be in uniform compression or expansion. For ionic crystals such as rock salt, the ratio of the breaking strength to Young's Modulus S/E varies between a theoretical value of 0.10 and measured values as low as 2×10^{-4}. The measured root mean square strains for LiF fall in this range.

The results suggest that interstitial atoms collect along certain 002 planes, spreading them apart and producing a microcleavage over areas whose diameters may be several hundred angstroms. The regions above and below such areas will be in uniform compression and the cylindrically surrounding regions will be in tension. The microcleavage is a way of partially relieving the strains and it can proceed to such an extent that the strains are kept down to values corresponding to the ratio S/E. From this picture it is not surprising that the strains for the 8 day irradiation are only slightly greater than those for the 2 day irradiation. The postulate of microcleavages is in keeping with the fragile character of irradiated material.

Since compressions and extensions normal to the cleavage planes have been postulated, additional measurements should be made on samples cut parallel to (110) and (111). The single crystal measurements should also be compared with measurements on powders since the effects may not be the

same. Additional work is needed to obtain a reliable picture of radiation damage in LiF.

III. Nature of Cold Work in Alpha Brass

Measurements have been made on compositions 90-10, 80-20, 70-30 and 65-35. Filings were used as the cold worked samples, and additional measurements made after various annealings. The peak broadening was analyzed to give particle size and distortion. From the peak shifts, stacking fault probabilities were obtained independently. Stacking faults produce a peak broadening which is similar to that of particle size, and the results indicate that a large part of the measured particle size broadening is actually due to stacking faults. In cold worked alpha brass there are stacking faults on the 111 planes, there is some true particle size broadening the coherent regions being 500A or more in size, and there are locked in strains. The root mean square strains are related to the particle sizes.

$\langle \epsilon^2 \rangle^{1/2} = \delta/L$ where δ is a length of the order of 1A and therefore of the magnitude of a half dislocation displacement. The stacking fault probability α is small for 90-10 and increases on approaching the alpha phase boundary. Values for the 100 direction from (200) and (400) and for the 111 direction from (111) and (222) for the cold worked filings are listed below.

	Size		RMS Strain		
Comp.	L(100)	L(111)	(100)	(111)	1/α
65-35	62A	128A	0.0080	0.0035	25
70-30	89	153	0.0067	0.0036	41
80-20	100	170	0.0057	0.0034	58
90-10	145	360	0.0033	0.0032	200

A complete report of this work will be prepared soon for publication.

IV. Nature of Cold Work in Body Centered Crystals

The significance of the small particle size value L = 200A found from cold worked tungsten is still obscure. To try to get additional information about the nature of cold work distortion in a body centered cubic metal, the alloy AuZn was prepared. This alloy has a beta brass structure and it remains in

long range order up to the melting point. Due to the difference in scattering power of the two atoms, the superstructure reflections are easily measured. It was hoped that slip might affect the breadths or the intensities of the superstructure reflections differently from those of the fundamentals and thus give additional information. It was found that the ratio of the integrated intensity of the annealed sample to that of the cold worked P(Ann)/P(CW) is about unity for high order reflections and drops below unity for decreasing sin θ. The drop is greater for the fundamentals than for the superstructure reflections, and the effect is presumably just a reduction in extinction by cold work. Both superstructure and fundamental reflections broadened by about the same amount. There is no evidence for any appreciable loss in long range order as a result of cold work, and no difference in the effect of cold work on a superstructure reflection from which to draw any conclusions.

Measurements are now being made on cold worked filings of beta brass, using the new methods for separating particle size and distortion broadening. The elastic anisotropy in beta brass is large, and it is hoped that the effect of the anisotropy on the particle size and the strain values in different directions may give additional information which could not be obtained from an isotropic metal such as tungsten.

V. Irradiated Cu-2 percent Si Single Crystals

Single crystals of the alloy Cu-2 percent Si were cut with 100, 110, and 111 faces and irradiated in the Hanford pile. Measurements are not yet far enough along to give any information about the radiation damage. However, one interesting effect has been observed. An $FeCl_3$ plus HCl etch applied to an irradiated sample produces a tremendous peak broadening and slight shift toward small angle whereas the same etch applied to an unirradiated sample gives no such effect. For the Cu-2 percent Si samples, measurements are being made of peak broadening, peak shift, and change in integrated intensity.

VI. The work reported here has been carried out by B. Borie, O. Guentert, D. Keating, E. Warekois, and B. E. Warren. We are indebted to Professor B. L. Averbach for help in preparing the alloy specimens.