

MICROCOPY RESOLUTION TEST CHART  
NATIONAL BUREAU OF STANDARDS 1963-A

AD-A152 367

0

# NORTHWESTERN UNIVERSITY

DEPARTMENT OF MATERIALS SCIENCE

TECHNICAL REPORT # 17

February 1985

OFFICE OF NAVAL RESEARCH

CONTRACT # N00014-80-C-116

X-RAY DIFFRACTION STUDY OF CHANGES IN STRESS/STRAIN DISTRIBUTIONS  
DURING FATIGUE OF A TWO-PHASE ALLOY

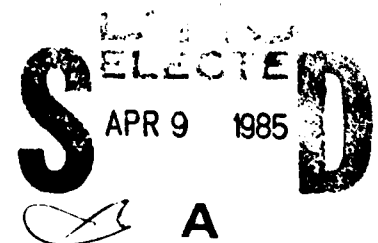
BY

I. C. Noyan and J. B. Cohen

Distribution of this document  
is unlimited

Reproduction in whole or in part  
is permitted for any purpose of  
the United States Government

DTIC FILE COPY



EVANSTON, ILLINOIS

85 03 21 171



## INTRODUCTION

Many engineering materials consist of more than one phase. Residual stresses, which play an important role in determining properties and the material's response to chemical and loading environments, can arise in two principal ways in such a material. Macro stresses develop when one part of a body is plastically deformed relative to another. Elastic constraints give rise to this component. As an example, consider shot-peening. Plastic flow due to the impinging shot occurs in the near surface regions and the extended material in this region applies a force to the undeformed bulk, and vice versa. Micro stresses (whose average values are sometimes called pseudo-macro stresses) arise between the phases on a microscopic scale due to differences in the elastic and plastic response of the different phases.

Recently <sup>1,2</sup> with modifications to the well-known x-ray methods for measuring stresses non-destructively, it has been shown how to separate these two components. In a shot-peened two-phase brass<sup>2</sup>, it was found that the micro stresses are much larger in the  $\beta$  phase than in the  $\alpha$  phase and are largest near the surface, whereas the macro stresses has its maximum value below the surface.

Changes in induced stress patterns during fatigue are of interest. Here we present a study of the stresses (and x-ray peak shape as well) to examine the changes after fatigue in an annealed and peened two-phase brass.

### EXPERIMENTAL PROCEDURES

The preparation of the flat annealed and peened polycrystalline 60-40 brass specimens is described in detail in<sup>2</sup>. The grain size was 40 microns, and the volume fraction of  $\beta$  was 0.22. The annealed specimens were peened first, then annealed, to produce the same surface roughness, ~6 microns in depth. Various depths below the surface were obtained by electropolishing in a phosphoric acid solution. This polishing was performed in a small area of the gauge length, and so corrections for stress relief were deemed to be unimportant.

Fatigue was carried out in a servo-hydraulic MTS machine. The grips were initially aligned by melting a Woods metal bath holding the lower grip and using a standard plate to move the grips in line, to minimize bending moments and assure uniaxial loading. Fatigue was in the load-control mode, with a fully reversed sinusoidal wave form and a frequency of 10Hz. The maximum applied load was  $\pm 175$  MPa,

which is the endurance limit for this brass<sup>3</sup>. The specimens were periodically removed and stress and x-ray peak shape obtained.

Stress determinations were made on a minicomputer-controlled Picker diffractometer, with a G. E. quarter circle goniometer as a sample holder and filtered  $\text{CrK}_{\alpha}$  radiation. The  $\phi$  rotation on this circle, and the separate  $\theta$  drive on the diffractometer were employed to establish the various required  $\phi$  and  $\psi$  tilts. Intensities were corrected on-line for background, absorption, the Lorentz-polarization factor and detector dead time. A peak's position was taken to be the apex of a parabola fit to the top 15 pct of a peak employing at least 7 points and background correction for the  $\alpha$  phase, 9-13 points for the  $\beta$  phase. The stationary slit method was employed, with corrections for changes in doublet resolution with  $\psi$  tilt<sup>5</sup>. The positions (of the  $(211)_{\alpha}$  and  $(220)_{\beta}$  peaks) were obtained to a precision of  $0.02^{\circ} 2\theta$ , which corresponds to  $\sim \pm 15$  MPa for both phases. Geometric errors due to sample misalignment and horizontal divergence were calculated to be 5 MPa at most.

The measurements consisted of obtaining the interplanar "d" spacings (from the peak  $2\theta$  values) vs.  $\sin^2\psi$ , for both phases, and for each phase at two  $\phi$  orientations ( $0^{\circ}$  and

90°) around the normal to the specimen face. The value of  $\phi = 0^\circ$  corresponds to stresses in the direction of the applied load. The x-ray elastic constants needed to convert the least squares fit to the slope of "d" vs  $\sin^2\psi$  to stress were calculated following Kröner<sup>6</sup> from single crystal values for both phases<sup>7</sup>. The stresses measured were the total values, e.g. at  $\phi = 0^\circ$ ,  $\sigma^t = [\langle\sigma_{11}\rangle^m + \langle\sigma_{11}\rangle^{p.m.} - \langle\sigma_{33}\rangle^{p.m.}]$ ; at  $\phi = 90^\circ$   $\sigma_{22}$  replaces  $\sigma_{11}$ .

The shape of the same x-ray peaks was monitored via the half-widths. For further experimental details see refs. 2 or 8.

## RESULTS

### a) Annealed State

Prior to fatigue the slope of "d" vs  $\sin^2\psi$  was essentially zero for both phases, indicating the absence of residual stresses. As fatigue proceeded oscillations developed in "d" vs  $\sin^2\psi$  for the  $\alpha$  phase, as can be seen by comparing Figs. 1a, b for the  $\alpha$  phase and Figs. 2a, b for the  $\beta$  phase. This absence of oscillations for the  $\beta$  phase persisted up until fracture at 312,000 cycles. There was no change in the peak half breadth for either phase. When 30

microns were removed there were no stresses and the oscillations in "d" vs  $\sin^2\psi$  for the  $\alpha$  phase disappeared. These data are summarized in Tables I and II.

b) Shot-Peened Specimens

Prior to the fatigue the various stresses obtained in  $d^2$  are summarized in Fig. 3. The "d" vs  $\sin^2\psi$  was linear and retained this linearity until  $\sim 50,000$  cycles, after which oscillations were observed for the  $\alpha$  phase only, Fig. 4. These increased with cycling until failure at 694,000 cycles. Note also that the relative intensity vs  $\sin^2\psi$  shows that texture is developing with cycling in the  $\alpha$  phase.

The stresses are given in Table III. The behavior is quite different for both phases. The residual stress in the  $\beta$  phase (which is primarily microstress) decayed to near zero (for both  $\phi$  directions) at  $10^4$  cycles. There was some decrease for the  $\alpha$  phase vs. N, followed by an increase. (The stresses in the  $\alpha$  phase are primarily macrostresses.) After 30 microns was removed there were oscillations in "d" vs  $\sin^2\psi$  for both phases.

The half breadths, Table IV, also indicated different behavior in both phases. That for the  $\alpha$ -phase remained

constant during cycling, while that for the  $\beta$  phase sharpened a bit with  $N$ , then remained constant. After removal of 30 microns these breadths were similar to those just prior to layer removal.

## DISCUSSION

### Formation and Variation of Residual Stresses in Both Phases of Annealed 60-40 Brass

Summarizing the results:

- i) No stresses developed in the  $\beta$ -phase at any point during fatigue loading.
- ii) Residual stresses developed in the  $\alpha$ -phase, which is softer, but oscillations occur in "d" vs  $\sin^2\psi$ .
- iii) The half-breadths of the measurement peaks, obtained at  $\psi = 0^\circ$ , remained constant during the experiment.
- iv) At 30 microns below the surface both phases exhibited linear "d" vs  $\sin^2\psi$  plots with negligible slope; thus, residual stresses were zero. The half-widths obtained after the etch were the same as the ones from the surface.

From these results, the following conclusions are derived:

- i) Plastic deformation caused by fatigue is inhomogeneously distributed in the  $\alpha$ -phase, (which is known to cause oscillations in "d" vs  $\sin^2\psi$ ).
- ii) The volume fraction of the deformed regions is small. This must be so since;
  - a) The half-breadth of the  $\alpha$ -peak, which is a measure of average deformation over all the grains sampled does not change.
  - b) There is no average balancing stress in the  $\beta$ -phase. As the volume fraction of  $\beta$  is much smaller than that for  $\alpha$ , any uniform distribution of stress in  $\alpha$  will be reflected as a larger change in the balancing stress in  $\beta$ . However, since deformation is inhomogeneously distributed, we have three (or more) phases;  $\beta$ -phase, deformed  $\alpha$ -particles and non-deformed  $\alpha$ -particles. Assume that all deformed  $\alpha$ -particles have the same deformation, and all other  $\alpha$ -particles have none. Then from the force balance

derived in ref. 1:

$$\sigma_{\alpha}^{\text{deformed}} \cdot f_{\alpha}^{\text{deformed}} + \sigma_{\alpha}^{\text{non-deformed}} \cdot f_{\alpha}^{\text{non-deformed}} + \sigma_{\beta} \cdot f_{\beta} = 0 \quad (1)$$

or,

$$-\sigma_{\alpha}^{\text{deformed}} \cdot f_{\alpha}^{\text{deformed}} = \sigma_{\alpha}^{\text{non-deformed}} \cdot f_{\alpha}^{\text{non-deformed}} + \sigma_{\beta} \cdot f_{\beta} \quad (2)$$

where  $f_i$  is the volume fraction. As we observed oscillations in "d" vs  $\sin^2 \psi$ ,  $(-\sigma_{\alpha}^{\text{deformed}} \cdot f_{\alpha}^{\text{deformed}})$  must be large. This must be balanced in large part by  $\sigma_{\alpha}^{\text{non-deformed}} \cdot f_{\alpha}^{\text{non-deformed}}$  since both  $\sigma_{\beta}$  and  $f_{\beta}$  are small. Thus, either  $\sigma_{\alpha}^{\text{non-deformed}}$  is large, or  $f_{\alpha}^{\text{non-deformed}}$  is large. If  $\sigma_{\alpha}^{\text{non-deformed}}$  is large, then  $f_{\alpha}^{\text{deformed}}$  is large but this will change the half-breadth; as it does not change,  $f_{\alpha}^{\text{non-deformed}}$  is large.

- iii) As shown in the above discussion, the stress in the deformed  $\alpha$ -particles is balanced by the stresses in non-deformed  $\alpha$ -particles and

$\beta$ -particles.

- iv) A deformed surface layer exists (only in the  $\alpha$ -phase) that extends  $\sim 30 \mu$  into the material, similar to that reported by Taira et al.<sup>10</sup> for 16% C steel. However, in this case, some  $\alpha$  grains are deforming in the surface, while others do not, while none deform below the surface.
- v) Even though the profile of residual stress with depth changes, there is no significant macrostress balance between surface and bulk because the volume fraction of deformed  $\alpha$  grains in the surface is small.

Behavior of Shot-Peening Stresses in Both Phases of 60-40 Brass Subjected to Fatigue Loading

To summarize the results, the stresses caused by shot-peening in the  $\beta$ -phase decayed to low values in  $10^4$  cycles, accompanied by a sharpening of the  $(211)_\beta$  peak. For the  $\alpha$ -phase, however, residual stresses persisted with linear "d" vs  $\sin^2 \psi$ , for both directions,  $S_1$ ,  $S_2$  (with the load being applied in the  $S_2$  direction). However, the change in  $\sigma_{22}$  was greater, a decay to 36% of its original value, while  $\sigma_{11}$  changed by 45%. There was also no change in the half-breadth

of the  $(220)_{\alpha}$  peak throughout the experiment. For this specimen, inhomogeneous deformation in the  $\alpha$ -phase started at  $90^{\circ}$  to the direction at  $10^5$  cycles, while "d" vs  $\sin^2\psi$  was linear in the load direction until  $388 \cdot 10^3$  cycles. This is in contrast to the annealed sample which exhibited oscillations in both directions early in the fatigue life. The magnitude of oscillations for both directions increased with increasing fatigue deformation and at fracture a linear regression line fitted to such oscillatory data had a rather large slope (although such a comparison is only qualitative).

We observe that:

- i) For the shot-peened sample, the fatigue life is much longer (twice) than that of a specimen with identical surface finish but no residual stresses or cold-work.
- ii) Fatigue causes the  $\alpha$ -homogeneous surface deformation distribution produced by peening to become inhomogeneous. This inhomogeneity is more pronounced in the softer phase. However, some oscillations also occur for the harder ( $\beta$ ) phase, although they are not as large as in  $\alpha$ .
- iii) The inhomogeneity is larger in the cold-

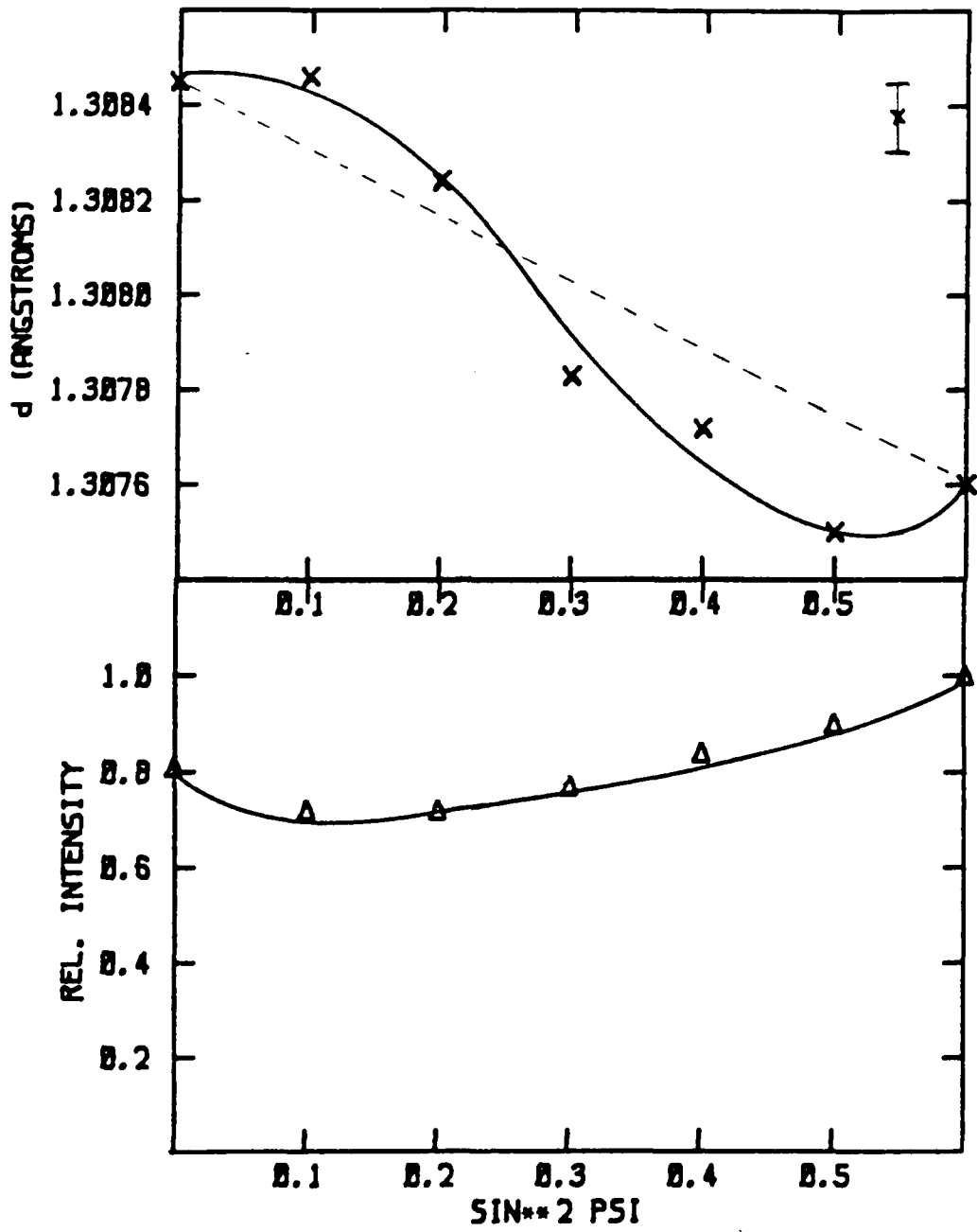


Figure 4a: I. C. Noyan and J. B. Cohen

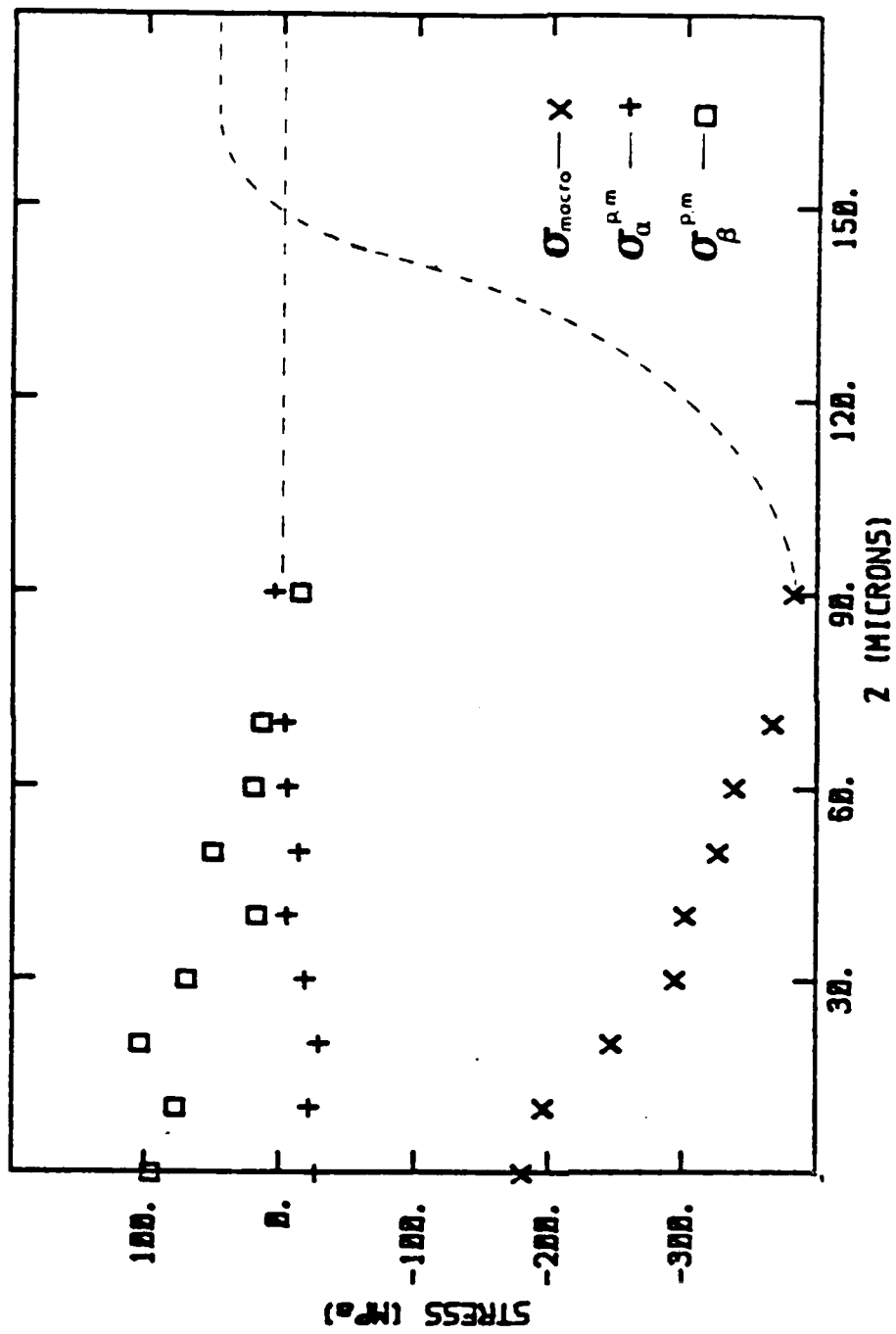


Figure 3b: I. C. Noyan and J. B. Cohen

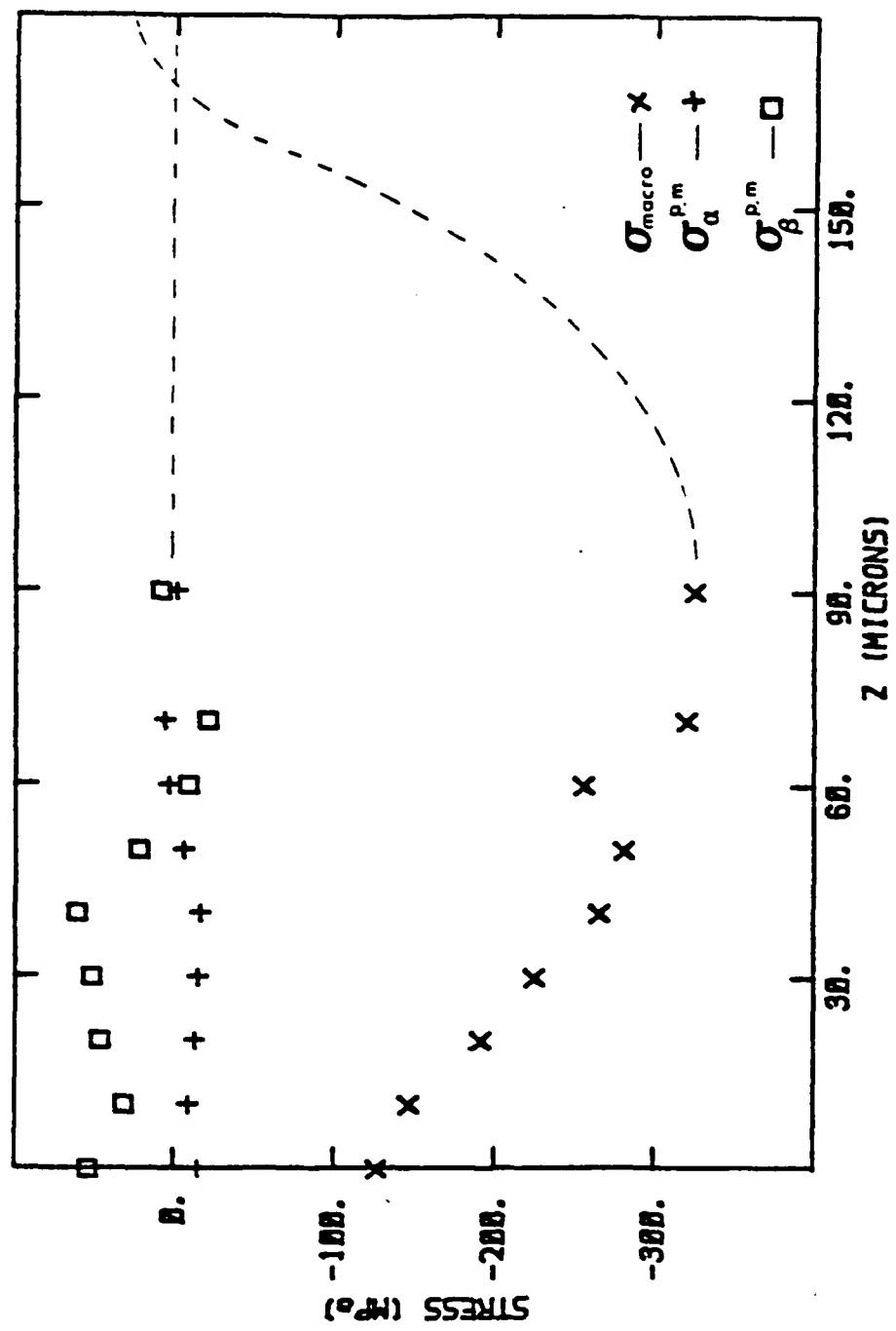


Figure 3a: I. C. Noyan and J. B. Cohen

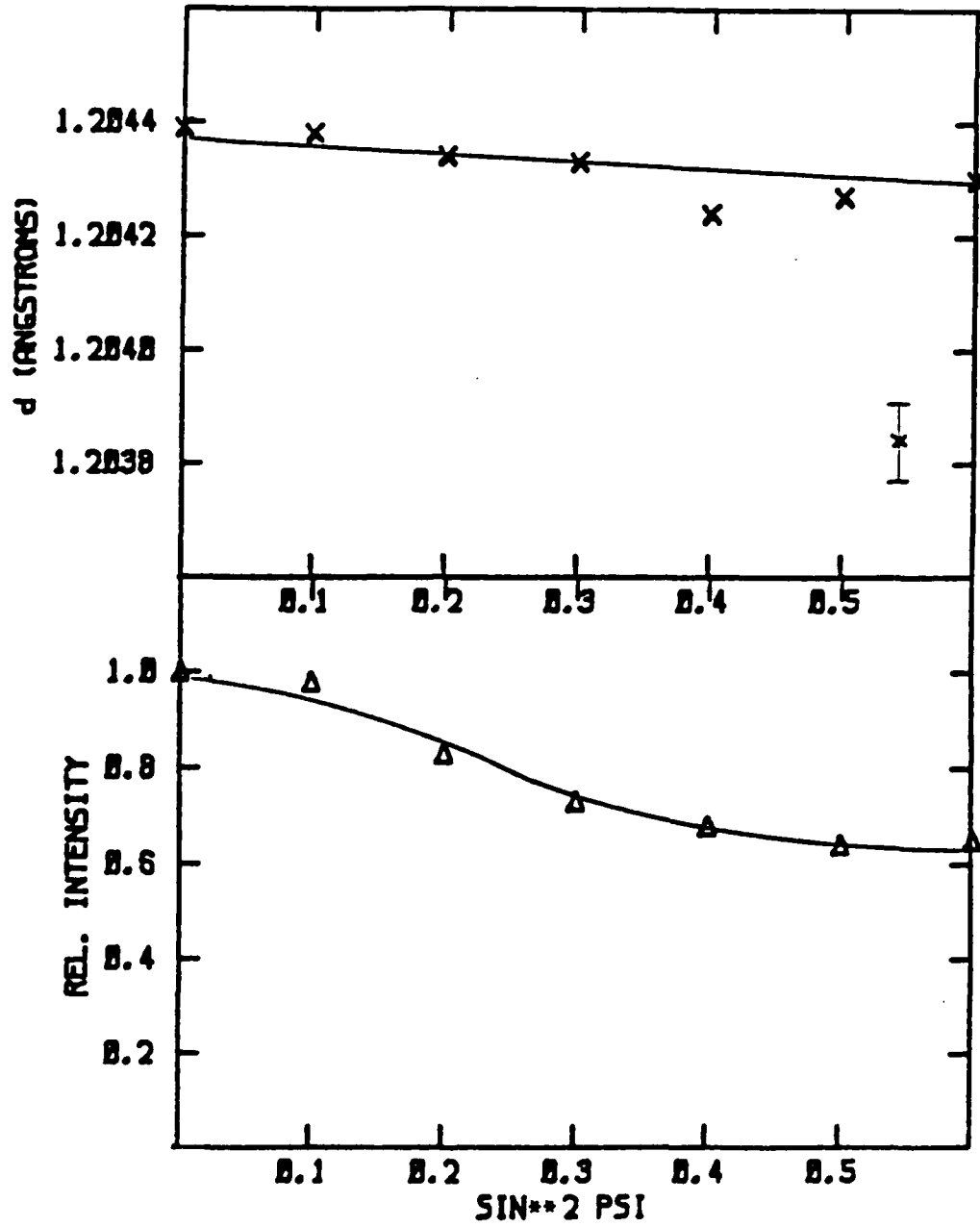


Figure 2b: I. C. Noyan and J. B. Cohen

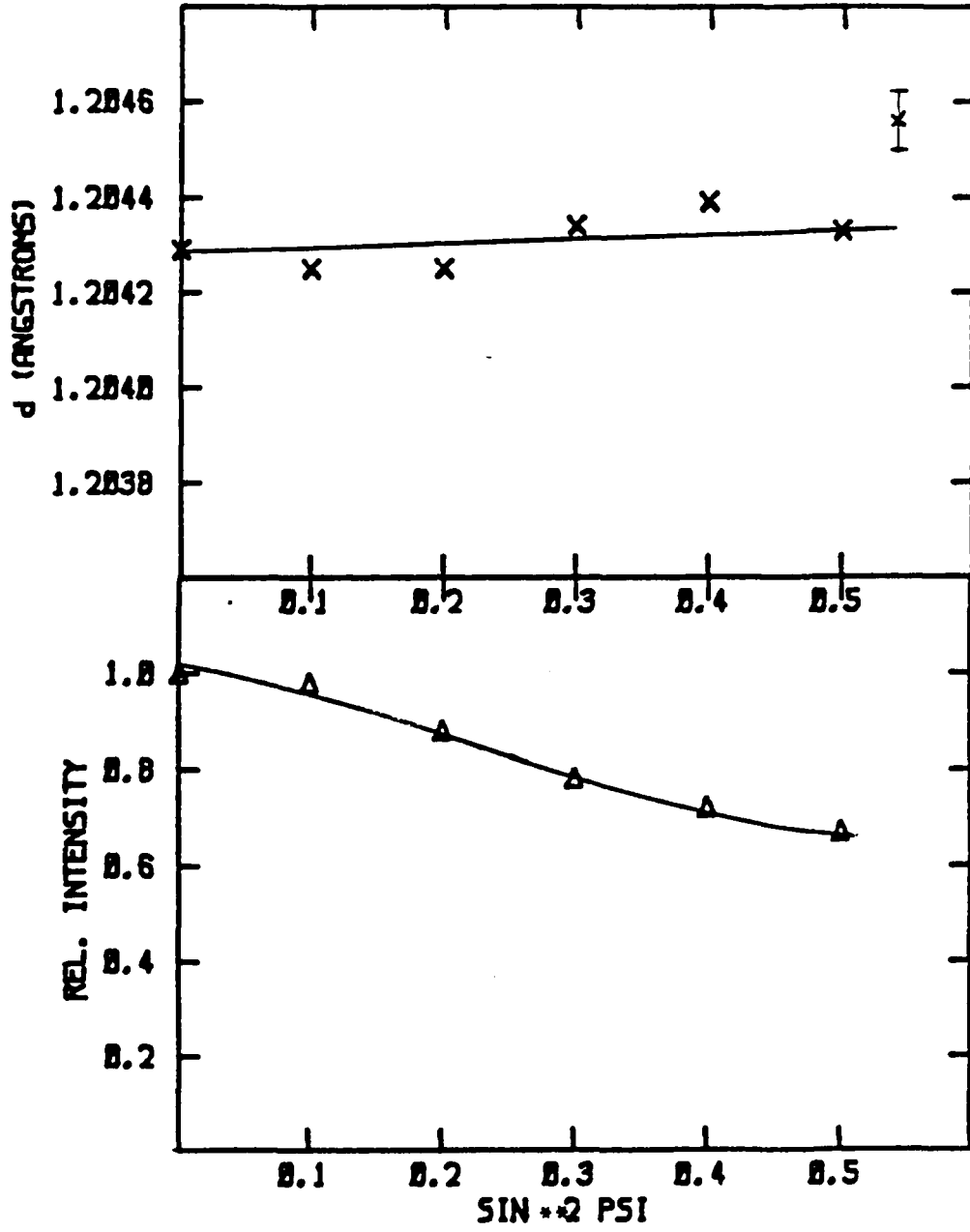


Figure 2a: I. C. Noyan and J. B. Cohen

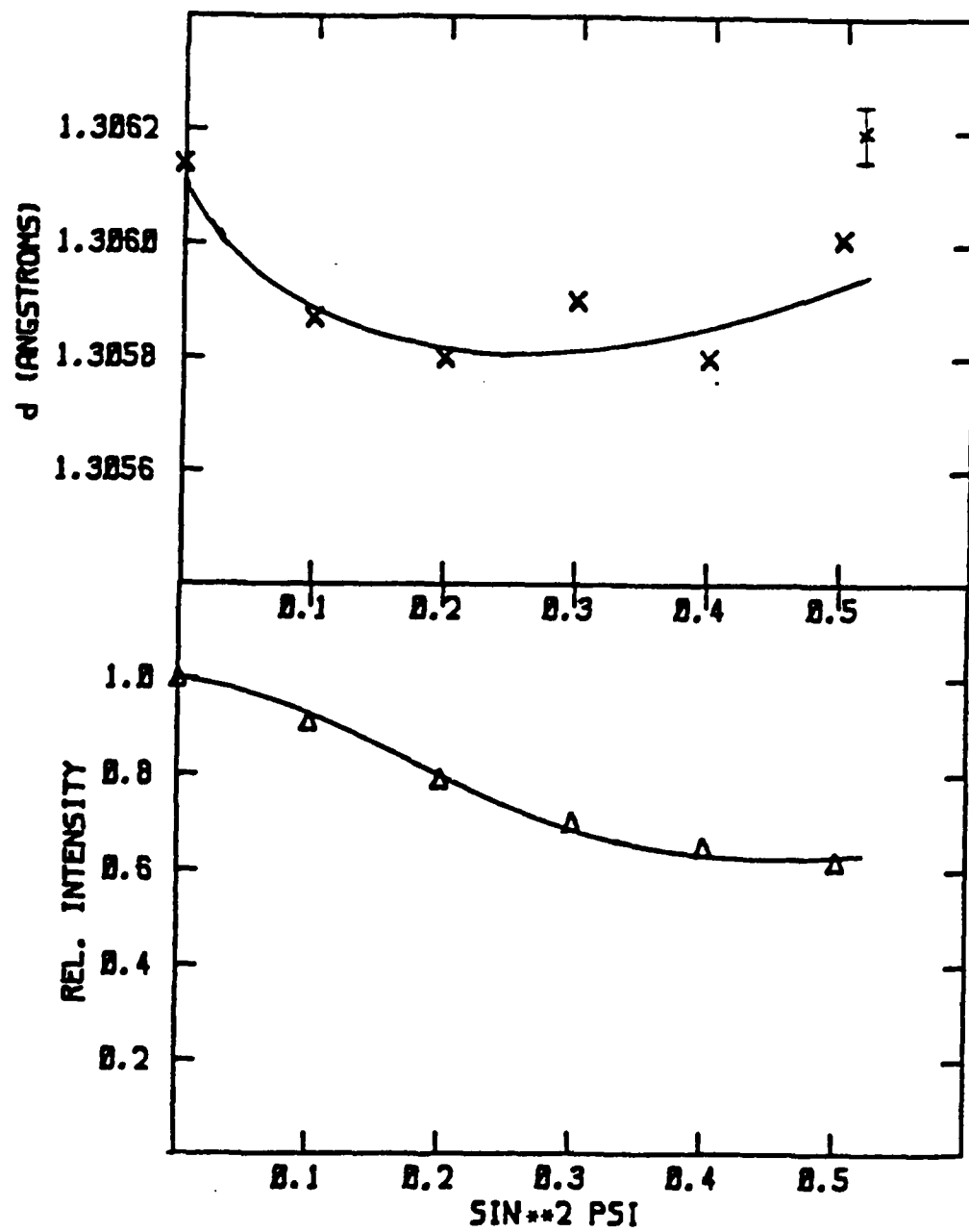


Figure 1b: I. C. Noyan and J. B. Cohen

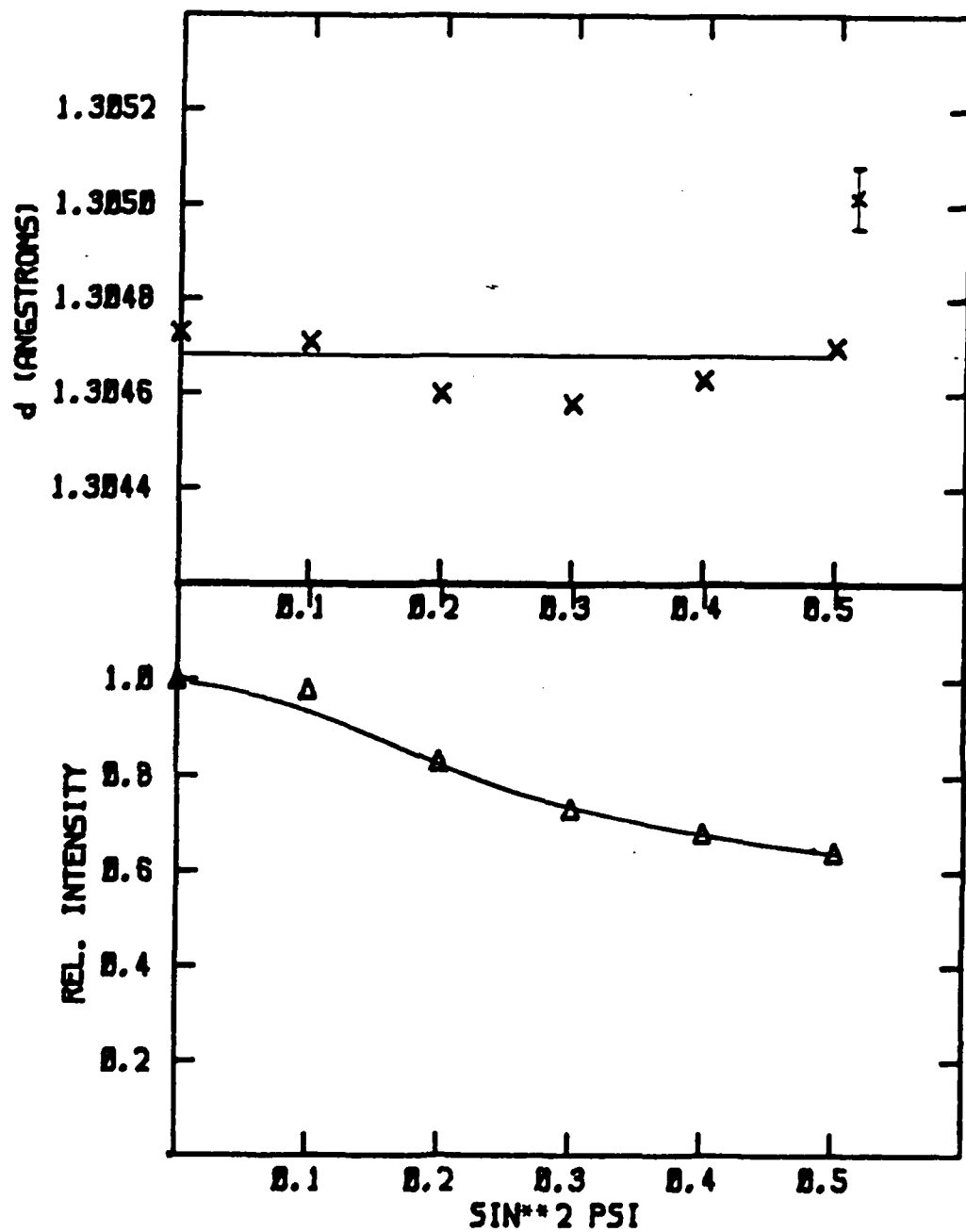


Figure 1a: I. C. Noyan and J. B. Cohen

## FIGURE CAPTIONS

- Figure 1a: Interplanar spacing, "d", vs  $\sin^2 \psi$  and relative intensity for  $\alpha$ -phase of annealed 60-40 brass before fatigue cycling.
- Figure 1b: Interplanar spacing, "d", vs  $\sin^2 \psi$  and relative intensity vs  $\sin^2 \psi$  for the  $\alpha$ -phase of 60-40 brass after 6000 fatigue cycles.
- Figure 2a: Interplanar spacing, "d", vs  $\sin^2 \psi$  and relative intensity vs  $\sin^2 \psi$  for  $\beta$ -phase of annealed 60-40 brass before fatigue cycling.
- Figure 2b: Interplanar spacing, "d", vs  $\sin^2 \psi$  and relative intensity vs  $\sin^2 \psi$  for the  $\beta$ -phase annealed 60-40 brass at 6000 fatigue cycles.
- Figure 3a: Separated macro and micro (p.m.) components as a function of depth ( $\varphi = 0^\circ$ ).
- Figure 3b: Separated macro and micro(p.m.) components as a function of depth ( $\varphi = 90^\circ$ ).
- Figure 4a: Interplanar spacing, "d", vs  $\sin^2 \psi$  and relative intensity vs  $\sin^2 \psi$  for the  $\alpha$ -phase of shot-peened 60-40 brass at 694,000 fatigue cycles.
- Figure 4b: Interplanar spacing, "d", vs  $\sin^2 \psi$  and relative intensity vs  $\sin^2 \psi$  for the  $\beta$ -phase of the shot-peened 60-40 brass specimen at 694,000 fatigue cycles.

TABLE IV: Variation of Total Residual Stress With Number of Fatigue Cycles in the Respective Phases of Shot-Peened 60-40 Brass Specimen

N.10 <sup>3</sup>	$\psi = 0^\circ$		$\psi = 90^\circ$	
	$\sigma_\alpha^t$ (MPa)	$\sigma_\beta^t$ (MPa)	$\sigma_\alpha^t$ (MPa)	$\sigma_\beta^t$ (MPa)
0	-141	-75	-207	-84
10	-118	-16	-109	0
50	-78	-15	-75	0
100	-94*	-28	-60	13
238	-89*	0	-56	38*
388	-75*	0	-52*	36*
694	-126*	31	-113*	-13*
694 (@ 30 $\mu$ )	-149*	13*	-103*	-54*

\* These stress values are approximate. For these cases oscillations occur in the "d" vs  $\sin^2 \psi$  data and the stresses are obtained by fitting a straight line to the oscillatory data.

TABLE III: Variation of the Peak-Breadth at Half the Maximum Intensity with Number of Fatigue Cycles (N) for the (220)<sub>α</sub> and (211)<sub>β</sub> Reflections Obtained from Shot-Peened 60-40 Brass

$N \cdot 10^3$	$PBHMI_{\alpha}^* (\text{°}2\theta)$	$PBHMI_{\beta}^* (\text{°}2\theta)$
0	2.6	3.8
10	2.5	3.1
50	2.6	2.9
100	2.6	2.8
238	2.6	2.9
388	2.5	3.0
694	2.5	3.0
694 (@ 30μ)	2.5	3.0

\* All data ± .2° in 2θ

TABLE II: Variation of Peak-Breadth at Half the Maximum Intensity With Number of Fatigue Cycles (N) for the  $(220)_\alpha$  and  $(211)_\beta$  Reflections Obtained From 60-40 Brass

$N \cdot 10^3$	$PBHM I_\alpha^* (\circ 2\theta)$	$PBHM I_\beta^* (\circ 2\theta)$
0	.66	.50
6	.65	.50
26	.68	.52
126	.64	.48
312	.64	.46
312 ( $\text{@ } 30\mu$ )	.66	.48

\* All data  $\pm .04^\circ$  in  $2\theta$

TABLE I: Variation of Total Residual Stress With Number of Fatigue Cycles in Both Phases of Annealed 60-40 Brass

Cycles, $N \cdot 10^3$	$\psi = 0^\circ$		$\psi = 90^\circ$	
	$t_{\sigma_a}^t$ (MPa)	$t_{\sigma_b}^t$ (MPa)	$t_{\sigma_a}^t$ (MPa)	$t_{\sigma_b}^t$ (MPa)
0	-8	14	-8	14
6	-16*	-15	-42*	-28
26	-27*	18	-32*	11
126	-61*	-6	-89*	-9
312	-60*	-6	-66*	1
312 ( $\phi$ 30 $\mu$ ) <sup>o</sup>	-20	4	-12	10

\* These values are obtained by fitting a straight line to oscillatory "d" vs  $\sin^2 \phi$  data and are therefore, approximate.

<sup>o</sup> 30  $\mu$  removed.

fulfillment of the requirements for a Ph.D. degree, August 1984.

#### REFERENCES

- 1) I. C. Noyan, *Met. Trans.*, 14A (1983) 1907.
- 2) I. C. Noyan and J. B. Cohen, *Mat. Sci. and Eng.*, in press.
- 3) D. E. Minar and J. B. Seastone, *Handbook of Engineering Materials* (1955) Wiley, New York, NY
- 4) M. R. James and J. B. Cohen, *Adv. in X-ray Analysis*, 20 (1977) 291.
- 5) H. Dölle and J. B. Cohen, *Met. Trans.*, 11A (1980) 159.
- 6) H. Dölle, *Jrnl. Appl. Cryst.*, 12 (1979) 489.
- 7) H. Wang and G. Simmons, "Single Crystal Elastic Constants and Calculated Aggregate Properties" (1971) MIT Press, Cambridge, MA.
- 8) I. C. Noyan, Ph. D. Thesis (1984), Northwestern University, Evanston, IL
- 9) I. C. Noyan and J. B. Cohen, *Adv. in X-ray Analysis*, 27 (1984) 129.
- 10) S. Taira and K. Hayashi, *Proc. 9th Japan Congr. on Testing Materials, The Soc. Mat. Sci. Japan* (1966) 1.

worked specimen than in an annealed one, since oscillations in "d" vs  $\sin^2\psi$  were much more pronounced than those in an annealed specimen subjected to the same load.

Further analysis of x-ray data from such specimens (in order to determine where the stresses are being balanced and to what extent) is not possible currently since techniques are not yet available to analyze oscillatory "d" vs  $\sin^2\psi$  data. It is clear that it is possible to use "d" vs  $\sin^2\psi$  plots as a tool for determining the onset of in-homogeneous plastic deformation by observing the presence and magnitude of oscillations.

#### ACKNOWLEDGEMENTS

This research was funded by ONR under grant No. N00014-80-C0116. The experiments were performed in the X-ray Diffraction Facility at Northwestern University, which was supported in part by the NSF-MRL program under grants No. DMR 7923573 and No. DMR 8216972. One of us (I. C. Noyan) also thanks the Turkish Scientific and Technical Research Foundation (TUBITAK) for a NATO grant enabling him to study in the U.S.A. This research constitutes a portion of a thesis submitted to Northwestern University (by I. C. Noyan) in partial

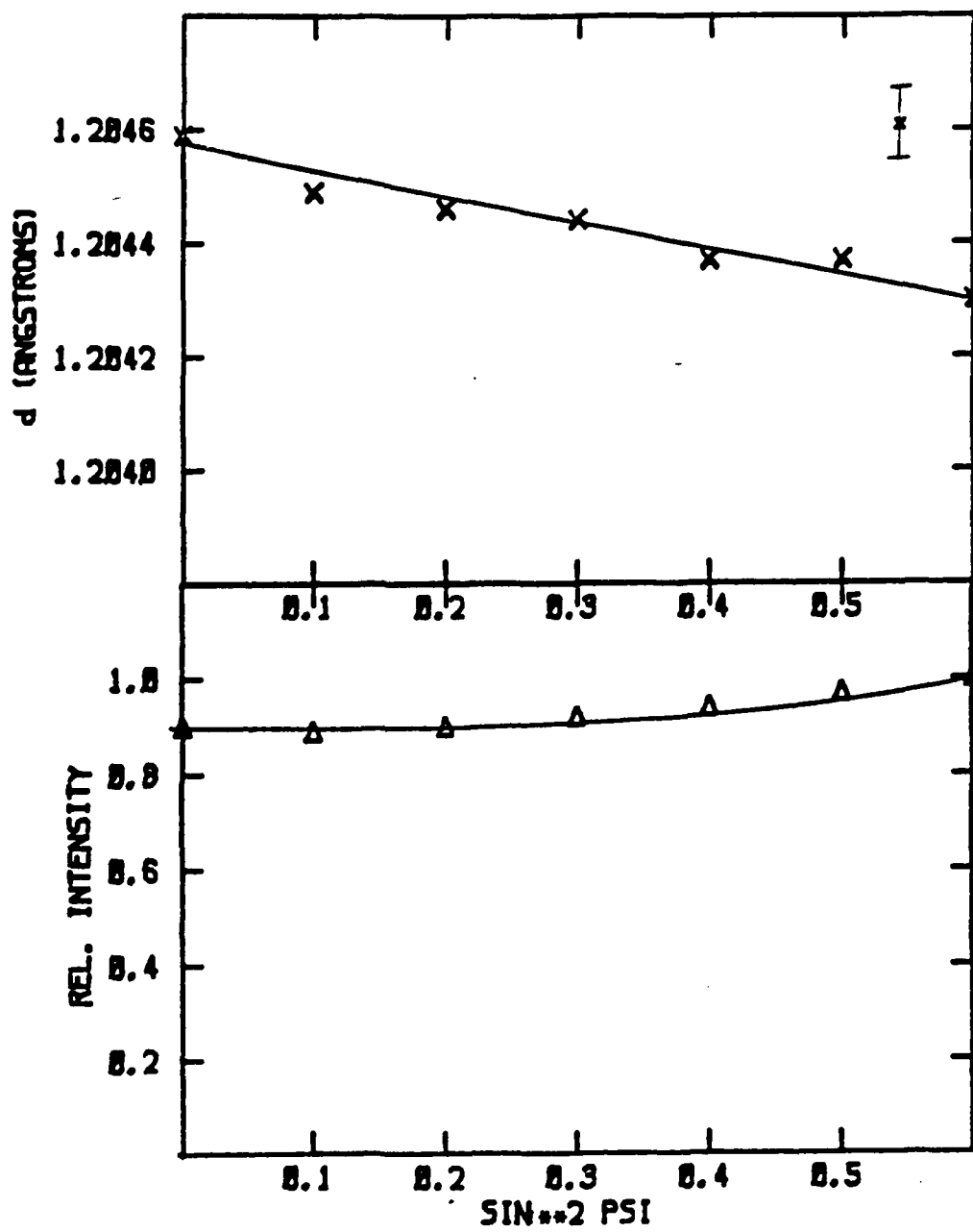


Figure 4b: I. C. Noyan and J. B. Cohen

## DOCUMENT CONTROL DATA - R &amp; D

Security Classification of title, body of abstract and indexing annotation must be entered when the overall report is classified

1. ORIGINATING ACTIVITY (Corporate author) J. B. Cohen Northwestern University Evanston, IL 60201		2a. REPORT SECURITY CLASSIFICATION	
		2b. GROUP	
3. REPORT TITLE <b>X-RAY DIFFRACTION STUDY OF CHANGES IN STRESS/STRAIN DISTRIBUTIONS DURING FATIGUE OF A TWO-PHASE ALLOY</b>			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) <b>Technical Report #17</b>			
5. AUTHOR(S) (First name, middle initial, last name) Ismail C. Noyan Jerome B. Cohen			
6. REPORT DATE February 1985		7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
8a. CONTRACT OR GRANT NO N00014-80-C-116		9a. ORIGINATOR'S REPORT NUMBER(S) 17	
b. PROJECT NO		9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
c.			
d.			
10. DISTRIBUTION STATEMENT Distribution of document is unlimited			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Metallurgy Branch Office of Naval Research	
13. ABSTRACT <p>In annealed two-phase brass, push-pull fatigue at loads near the endurance limit produces residual stresses only in the softer majority <math>\alpha</math> phase, not in <math>\beta</math>. These stresses are balanced between undeformed <math>\alpha</math>-phase regions, and a small fraction of deformed <math>\alpha</math>; the <math>\beta</math> phase is not involved in this balance. The uniform deformation produced by peening becomes inhomogeneous during fatigue, especially in the softer <math>\alpha</math>-phase. This inhomogeneity of deformation is greater after fatigue of the peened specimens, than after fatigue of annealed material.</p>			

14

KEY WORDS

LINK A

LINK B

LINK C

ROLE

WT

ROLE

WT

ROLE

WT

Residual stresses  
 Fatigue  
 Two-phase alloys  
 Brass

**END**

**FILMED**

**5-85**

**DTIC**