

PLASTIC DEFORMATION, RESIDUAL STRESS, AND CRYSTALLINE  
TEXTURE MEASUREMENTS FOR IN-PROCESS CHARACTERIZATION  
OF FCC METAL ALLOYS

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INTRODUCTION

The need for in-process characterization of metallic components is being recognized increasingly. In the field of x-ray analysis the x-ray fluorescent (spectroscopy) techniques have been successfully applied to in-process inspection, while successes in x-ray diffraction have been sparse. X-ray diffraction characterization techniques should be fast, non-contacting, and tolerant of detector to component distance variation. The Ruud-Barrett position-sensitive scintillation detector (R-B PSSD) is unique in its ability to satisfy these requirements, and has been successful in measuring plastic deformation, residual stress and crystalline texture in FCC metal alloys.

Plastic deformation, residual stress, and crystalline texture in metal alloys can be measured from the breadth, position, and intensity of the x-ray diffraction peaks (Figure 1). When metals are stressed, they deform both plastically and elastically [1]. Plastic deformation results in the distortion of individual crystallite lattices, and is manifested in peak broadening. Elastic strain (residual stress) occurs uniformly over several tens of grains, causing the d-spacing between elastically strained lattices to change, and the diffraction peak to shift. In addition, a preferred orientation of crystallites, i.e. texture, occurs in many manufacturing processes, which results in a greater intensity of diffraction by a given set of crystallographic planes at different orientations to the surface of a sample.

This paper will describe the results of several on-going investigations on the measurement of plastic deformation, residual stress, and crystalline texture in nickel, copper, and aluminum base alloys by x-ray diffraction techniques using the R-B PSSD.

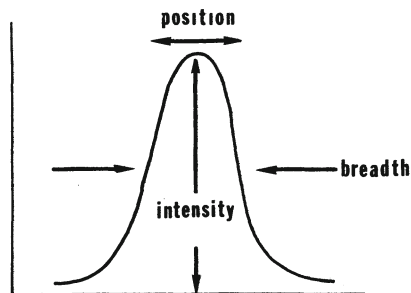


Figure 1. X-ray diffraction peaks are used to measure microstrain (breadth), residual stress (position), and crystalline texture (intensity).

## BACKGROUND

The Ruud-Barrett position-sensitive scintillation detector provides unprecedented x-ray diffraction measurement speed consistent with excellent accuracy. The R-B PSSD relies on the coherent conversion of the diffracted x-ray pattern to an optical signal (light); the conduction of this light signal over several linear centimeters of fiber optic bundles; the amplification of this signal by electro-optical image intensification; the electronic conversion of the signal; and the transfer of the electronic signal to a computer for refinement and interpretation. It has been described elsewhere [2,3], and its application to materials characterization discussed [4].

With the R-B PSSD, data collection times of less than one second are possible using modern x-ray tubes and constant potential power supplies -- a two order of magnitude improvement over conventional XRD instrumentation. A compact version of this instrument has been developed that is capable of making measurements on the inside of a pipe as small as 100 mm [5]. Also, the geometry of its x-ray optics allows stress readings in confined areas such as gear teeth, pipe and turbine vane bases.

## PLASTIC DEFORMATION: MICROSTRAIN

Plastic deformation, or microstrain, results in a distortion of the crystal lattice of individual crystallites and, subsequently, variability of the d-spacing of the lattice planes. The angle of x-ray diffraction for each plane will vary slightly, as well, yielding a broader peak than would an undeformed crystallite. However, peak broadening is also caused by small grain size, by imperfections in the detector, and by the non-parafocusing condition of the PSSD. Those effects can be subtracted from the raw data by a comparison of peaks with a reference material [6].

## Methods

Preliminary microstrain measurements were made on Inconel c-rings, with the intention of applying the techniques to the study of stress corrosion cracking in Inconel U-bend tubes. Strain measurements were

made on two c-rings, at 9 sites around the curvature of the bend. Diffraction peaks were analyzed for the (311) crystallographic plane with Cr K-beta radiation. The detectors were oscillated over the Debye ring in a 16° arc, to increase the number of grains contributing to diffraction.

Raw data were collected in the form of the diffraction peak integral breadth, B (area/height), and full width at half maximum height, 2w. Peak broadening data were adjusted for the non-parafocusing ( $\psi \neq 0$ ) condition of the PSSD, and for instrumental broadening. The integral breadth and full width at half maximum, that were attributed to structural broadening alone, were analyzed by the method of Delhez et al.. Peak broadening due to small grain size is approximated by a Cauchy distribution, and peak broadening due to microstrain is approximated by a Gaussian distribution. Deconvolution of the Gaussian profile from the whole peak yields an approximation of the peak due to microstrain alone. Microstrain is measured as percent strain from:

$$\tilde{e} = B / 4 \tan \theta \quad (1)$$

where e is percent microstrain, B is the broadening due to structural microstrain alone, and  $\theta$  is the x-ray diffraction angle [6].

### Results

The results of the microstrain measurements on a mill-annealed Inconel c-ring at a  $\psi$  angle of 12 degrees, are shown in Figure 2. Note that the greatest microstrain was detected at the site of greatest curvature of the ring at 0 degrees, as expected.

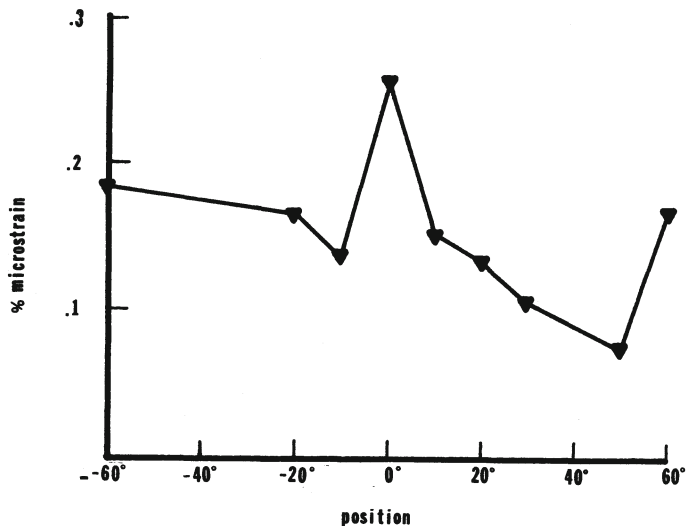


Figure 2. Microstrain variation versus position on an Inconel c-ring. The position of greatest curvature, as well as greatest strain, is at 0°.

## ELASTIC STRAIN: RESIDUAL STRESS

When a metallic crystalline material is stressed, the elastic strains in the material are manifested in the crystal lattices of its grains. The stress applied externally or residual within the material, if below its yield strength, is taken up by uniform interatomic macro-strain that is spread over several tens of grains. The distance between lattice planes, d-spacing, is thus changed, as well as the angle ( $2\theta$ ) the x-ray beam is diffracted, i.e., there is a shift in the peak position. Because x-ray diffraction techniques measure interatomic spacing (peak position), the elastic macrostrain experienced by the specimen can be quantified [7].

Residual stresses are induced in copper strips during the fabrication of electrical switches and contacts. The presence of residual stresses in copper strips can lead to warpage immediately after fabrication, or in later manufacturing operations.

### Method

The R-B PSSD uses two independent detection surfaces to collect data from two positions on the Debye ring simultaneously, thus providing a unique capability of precision stress measurement by the single exposure technique (SET) [7]. The SET method is based on the fact that a single incident x-ray beam is diffracted at a constant  $\theta$  angle such that a cone of diffracted radiation is formed. A plane perpendicular to the cone axis intercepts the cone as a circle when the specimen is unstressed, and as an ellipse, if stressed [1]. Deviation from a circle, then, is a measure of that stress. To read this deviation, detectors are placed  $180^\circ$  apart. Until the development of the R-B PSSD the SET method had been restricted to the use of film camera devices [6]. The stress measurement is made with:

$$\sigma = \left[ \frac{E}{1 + \nu} \right] \frac{1}{4R_0 \sin^2 \theta \sin 2\beta} (S_2 - S_1) \quad (2)$$

where  $R_0$  is the detector-to-specimen distance,  $\beta$  is the angle between the specimen surface normal and the incident x-ray beam,  $(S_2 - S_1)$  as shown in Figure 3, is a measure of distortion of the cone of diffracted radiation,  $E$  is the elastic modulus and  $\nu$  is Poisson's ratio.

For the x-ray method to be used as an absolute measure of the residual stress, the d-spacing of planes of the same Miller indices must be measured for at least two different orientations with the metal surface,  $\psi_1$  and  $\psi_2$ . Absolute measurement means that no previous or subsequent measurement of that metal piece need have been obtained in a zero-stress condition for comparison.

### Results

Residual stress measurements on copper alloy strips were performed by the single exposure technique (equation 1), using the (420) plane and Cu K-alpha x-radiation. The angle between the incident beam and the surface normal,  $\beta$ , was  $25^\circ$ , yielding psi angles of  $5^\circ$  and  $43^\circ$ .  $E$  (the elastic modulus) was  $16 \times 10^6$  psi, and  $\nu$  (Poisson's ratio) was 0.36. Preliminary results of residual stress measurements on the edge of a copper strip are shown in Fig. 4.

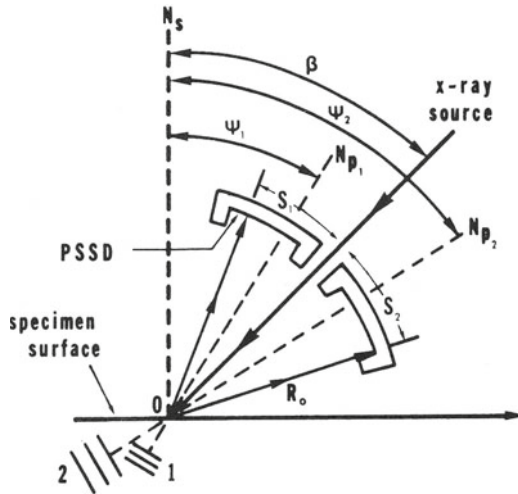


Figure 3. Drawing of the single exposure technique.  $S_1$  and  $S_2$  are the angles between the diffracted beams, at the two detectors, and the incident beam. Broken lines  $N_s$ ,  $N_{p1}$ , and  $N_{p2}$  are the normals to the specimen surface, and to the two crystallographic planes 1 and 2.  $\beta$  is the angle between the incident beam and the surface normal, and  $R_0$  is the distance between the point of incidence and the x-ray detection surface.  $\psi_1$  and  $\psi_2$  are the angles between the surface normal and the normals to the planes 1 and 2.

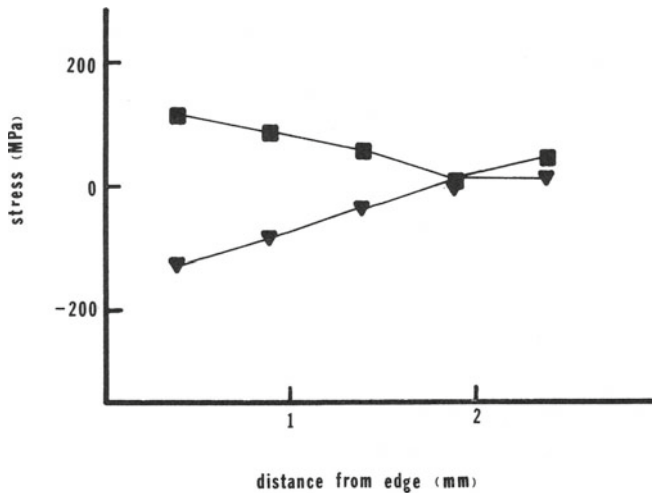


Figure 4. Plot of the residual stresses in copper alloy strip as a function of distance from the edge of the strip. Measurements were made on both concave (■) and convex (▼) surfaces.

#### CRYSTALLINE TEXTURE

Preferred orientation of crystallites is often produced in metals by manufacturing methods such as wire drawing or sheet rolling. The presence of a preferred orientation in aluminum can stock results in a distorted can, i.e. "earring" texture. Crystalline texture is detected in

the surface of the aluminum sheet by variation in peak intensity from one psi angle to another.

### Method

Twelve samples of aluminum alloy can sheet stock were selected for texture evaluation. The samples varied in thickness from 0.128" to 0.0124", and in texture from 2.8% to 11.0% as measured by a cup drawing "earing" test. The (333,511) planes and both Cu K-alpha and Cr K-alpha radiation sources were used. The x-ray source was tilted at two beta angles (18° and 30°), about an axis oriented at three different orientations ( $\phi$ ) to the rolling direction of the samples. The specimen-to-detector was allowed to vary about 1.0 mm about the ideal of 45 mm.

### Results

Data were collected from diffraction patterns in the form of relative full width at half peak breadth ( $b/b_p$ ), relative intensity ( $I/I_p$ ), and the peak intensity area function percent ( $b/b_p \times I/I_p \times 100$ ), from the two diffracted x-ray peaks at the two beta angles. The  $b_p$  and  $I_p$  values were used to normalize the  $b$  and  $I$  values from the textured samples and were obtained from the aluminum powder specimen.

The data are plotted in Figure 5. A linear regression line is drawn through the points, with a correlation coefficient of 0.958. This is an excellent fit despite the fact that the samples came from two different manufacturers, were different gages, and were irradiated by two different sources.

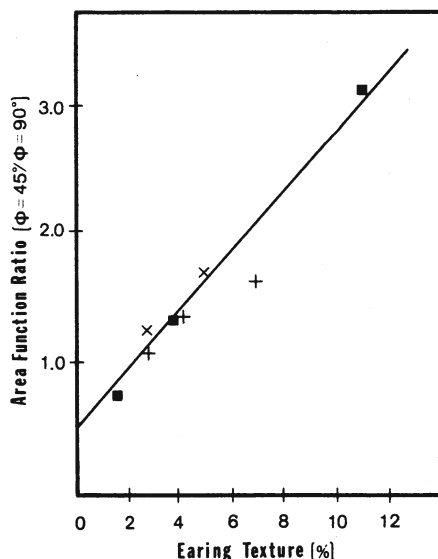


Figure 5. Plot of the area function ratio ( $\phi=45^\circ/\phi=90^\circ$ ) for psi angle of  $28^\circ \pm 2^\circ$  versus percent earing in aluminum can stock. Sheet thickness varied from 0.012" (+), to 0.08" (■), to 0.127" (x). The regression line drawn through the points has a slope of 0.22, with a correlation coefficient of 0.958; there was a 0.4% uncertainty percent earing for the gage between 0.080" and 0.125".

## CONCLUSIONS

X-ray diffraction techniques have been shown to be effective in the measurement of plastic deformation, residual stress, and crystalline texture in FCC metals, from the breadth, position, and intensity of the x-ray diffraction peaks. The Ruud-Barrett position-sensitive scintillation detector has been demonstrated to be fast, non-contacting, and tolerant of detector to component distance variation -- necessary requirements for cost-effective in-process inspection of materials.

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