

Discussion of Paper by S. Weissmann

The Observation and Measurement of Substructures  
In Crystals by X-Ray Techniques

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We would like to point out that another x-ray technique, the Fourier analysis of shapes of diffraction peaks, is useful in characterizing substructures. This method was originally proposed by Warren and Averbach<sup>1</sup> and was recently reviewed by Warren<sup>2</sup>. One obtains from the coefficients of a Fourier series representing a peak the mosaic size ( $D_{\text{eff}}^{\langle hkl \rangle}$ ), determined by substructure and fault density, and the rms strains  $\langle \varepsilon_{x\dot{A}} \rangle_{\langle hkl \rangle}^{1/2}$  averaged over various distances ( $X\dot{A}$ ) normal to the diffracting planes, i. e., in specific crystallographic directions. Additional information concerning faulting and long-range strains can also be obtained from peak positions and peak asymmetry<sup>2, 3</sup>. The information obtained is averaged over the area under the x-ray beam and thus good statistical data is obtained to relate to properties which do not depend on a local state of imperfection (such as in a failure or in recrystallization). In addition to being non-destructive and not very restrictive as to sample geometry, it is also especially helpful after moderate deformation, in which case present resolution with the electron microscope is not adequate. In fact, we have even found it useful after only a few percent elongation in tension of fine grained materials. Apparently the method has not been used extensively because of the work involved in obtaining the Fourier coefficients. However, this can be done quickly and cheaply with computers<sup>4</sup>; in fact it is possible to feed the x-ray data directly to

punched cards or tape, or to use a chart reading device with the computer and hence to eliminate almost all the time for analysis.

Two recent theoretical investigations have shown the nature of some of the errors in the analysis and how to simply correct for these<sup>5, 6</sup>.

In order to demonstrate the usefulness of the method, we wish to briefly present two examples:

- 1) Relationships between the substructure and properties of electro-deposits are not at all clear, for example, it is known that macro-residual stresses and hardness do not correlate well<sup>7</sup>. In Table I data is presented concerning the effect of potassium ethyl xanthate (KEX) additions on the substructure of Ag electrodeposits, as determined by the Fourier technique, and some of the properties of the deposit<sup>8</sup>. These results clearly illustrate that in this case the mosaic size and twin fault probability ( $\rho$ ) control the hardness but are not important in determining the residual stresses, especially after large additions of KEX.
- 2) The fatigue life of steels seems to increase and pass through a maximum with increasing hardness<sup>9</sup>. It is also known that introducing surface compressive stresses, for example by peening, will improve fatigue life<sup>10</sup>. In Table II, some x-ray data on a 1045 steel is presented for two heat treatments and for the effects of peening<sup>11</sup>. At low hardness levels, peening affects both the long-range residual stresses and the substructure (mosaic size and microstrains) whereas at a high hardness level, only the long-range stresses are strongly affected. This data suggests that in low hardness steels, the substructure is important in retarding ductile fracture. However, this substructure also lowers cleavage strength or raises the flow stress above the cleavage strength; hence as hardness increases cleavage fracture becomes more important, resulting in the maximum in fatigue life. The role of long-range compressive residual stresses at high hardness is then to

retard cleavage fracture.

It is also worth pointing out that with polycrystalline b. c. c. metals and alloys (such as low carbon steel) the diffraction pattern contains a third order of one peak, the 440, which does not overlap with any other peak. (This does not occur with polycrystalline f. c. c. materials.) As a result of this, enough data is available in the diffraction pattern to obtain the actual strain distribution without assumptions<sup>1</sup> and thus to see how the distribution is affected by treatment. The results can be compared with distributions arrived at from theoretical considerations. For example, if the strain distribution is thought to be Gaussian, the standard deviation ( $\sigma$ ) is simply related to the microstrain and the long-range residual strains:

$$\sigma^2 \langle hkl \rangle \text{ at } X \text{ \AA} = \langle \epsilon^2 \rangle_{xA} \langle hkl \rangle - \langle \epsilon \rangle^2 \langle hkl \rangle$$

where  $\langle \epsilon \rangle \langle hkl \rangle$  is obtained from peak shifts.

Thus it is possible to calculate the Gaussian distribution from some of the Fourier data and to compare it to the actual distribution determined from the Fourier analysis, and perhaps with electron microscopy as well, to understand why and how the distribution is affected.

Finally, we would like to point out that electron microscopy could be especially useful in establishing the exact meaning of the mosaic size determined by x-ray diffraction. Some work along these lines has been done<sup>12</sup> but much more is needed.

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TABLE I  
STRUCTURE AND PROPERTIES OF  
SILVER ELECTRODEPOSIT\*

Conc. KEX mg/l	0.00	0.34	0.84	1.9	3.5
$\langle \epsilon^2_{50\text{\AA}} \rangle^{1/2}_{\langle 111 \rangle}$	$1.4 \times 10^{-3}$	1.7	1.4	1.7	1.8
$\langle \epsilon^2_{50\text{\AA}} \rangle^{1/2}_{\langle 200 \rangle}$	$1.9 \times 10^{-3}$	1.6	1.7	1.75	-
Surface Stress (in psi) error: $\pm 2560$ psi	+ 3750	1250	10,750	11,900	9,000
VHN error $\approx \pm 7$ pct.	92	103	95	111	116
$\beta$	0.001	0.011	0.005	0.015	-
$D_{\text{eff}} \langle 111 \rangle$	560A	250	340	150	170
$D_{\text{eff}} \langle 200 \rangle$	260A	140	180	85	-
$D_f \langle 220 \rangle^+$	4000A	370	820	280	-
Plating Efficiency	75%	85%	76%	85%	89%
* Plating Conditions: 1.4N KCN + 0.4N KCN, 10 ma/cm <sup>2</sup> , room temperature.					

+ Calculated contribution of  $\beta$  to  $D_{\text{eff}}$

TABLE II  
EFFECTS OF PEENING ON THE SUBSTRUCTURE  
AND PROPERTIES OF SAE 1045 STEEL

	Not Peened		Peened*	
	RC - 21	RC - 50	RC - 21	RC - 50
$\langle \epsilon_{4\text{\AA}}^2 \rangle_{\langle 110 \rangle}^{1/2}$	$4.5 \times 10^{-3}$	$10.7 \times 10^{-3}$	$8 \times 10^{-3}$	$11 \times 10^{-3}$
$\langle D_{\text{eff}} \rangle_{\langle 110 \rangle}$	1900Å	315Å	420Å	260Å
$\langle \epsilon_{\text{av.}} \rangle_{220}$ peak shift	$0.2 \times 10^{-3}$	$-0.5 \times 10^{-3}$	$0.9 \times 10^{-3}$	$1.3 \times 10^{-3}$
$\langle \epsilon_{\text{av}} \rangle_{\text{sine}}$ coeff. 220	$0.2 \times 10^{-3}$	$0.4 \times 10^{-3}$	$1.1 \times 10^{-3}$	$1.6 \times 10^{-3}$
Surface residual macro-stresses	-15,000psi	-1200psi	-67,000psi	-95,000psi
*Peened to 0.010 A-2 intensity				