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PROFILE FITTING IN RESIDUAL STRESS DETERMINATION(U)
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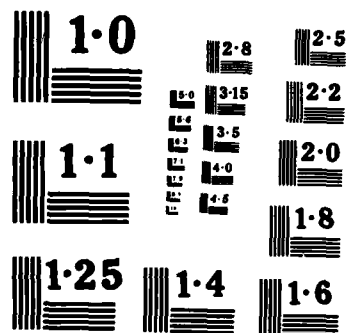
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NORTHWESTERN UNIVERSITY

DEPARTMENT OF MATERIALS SCIENCE

TECHNICAL REPORT #22

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PROFILE FITTING IN RESIDUAL STRESS DETERMINATION

by

T. J. DEVINE AND J. B. COHEN

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increasing interest in measuring the stresses in second phases as well as the matrix in multiphase materials, or in the strengthening phases in composites. Because of their low volume fraction, and their structure and its perfection, peaks with reasonable intensities are available only at intermediate angles, where the errors may be too large to obtain reliable stress values. Perhaps greater precision can be obtained by curve fitting the entire diffraction peak to some suitable function other than a parabola. Furthermore, the speed of stress measurements has been greatly reduced by the use of position sensitive detectors^{5,6}, by means of which an entire peak profile is recorded at the same time and no detector motion is required - a kind of digitized return to film! Why not use a fit to the entire peak? The data is already available in the same time it takes to obtain the information for a parabolic fit.

It is with these two points in mind that we have examined the use of profile fits in the measurement of a peak position.

FUNCTIONS

Two functions were chosen to compare to the parabolic fit. The first of these is a Modified Lorentzian, proposed by Mignot and Rondot⁷:

$$I_{\alpha_1}^{K_1}(2\theta_i) = I_0 \left[\cos \pi \frac{(2\theta_i) - 2\theta_p - \delta}{a} \right]^n \frac{K^2}{K^2 + (2\theta_i - 2\theta_p)^2} \quad (3)$$

Measurements are made at many $2\theta_i$ and a solution is sought for a , $2\theta_p$, I_0 the maximum intensity, K which is related to the peak width, δ to account for small peak asymmetry and n . The cosine term forces the function to fall more rapidly with 2θ in the tail of the peak than a pure Lorentzian function. This equation can be modified to include a $K_{\alpha_1} - K_{\alpha_2}$ doublet (with separation Δ):

$$I_i^{\text{total}} = I_i^{K_{\alpha_1}}(2\theta_i) + \frac{1}{2} I_i^{K_{\alpha_2}}(2\theta_i - \Delta) \quad (4)$$

The term δ was chosen as zero, because preliminary tests (multiple scans of peaks) indicated that it led to large variations in $2\theta_p$ without much improvement in the fit to the entire shape. Both singlet and doublet forms were tried.

The second function was a Pearson Type VII distribution⁸.

$$I_i(2\theta_i) = I_0 \left[1 + \frac{(2\theta_i - 2\theta_p)^2}{ma^2} \right]^{-m} \quad (5)$$

The terms I_0 and a are sought in the profile fitting procedure. The parameter m varies with peak shape. For narrow peaks $m = 1$ is appropriate, in which case Eq. 5 is a Lorentzian. When m approaches infinity it can be shown that Eq. 5 approaches a Gaussian function. Values of $m = 1-3$ and infinity were tried. Again, two terms can be added to form a doublet, and this was attempted for $m = 3$.

Background was subtracted. For the case of the parabolic fit, this was measured at $3^\circ 2\theta$ (before) the peak and this constant value was subtracted from all data points. This was adequate because only points in the

vicinity of the peak (the top 15 pct) were employed in the fit. For the other two functions a linear variation was assumed and the slope and intercept were sought in the solution, or the values were fixed from the data. If these were included in the solution, there were 7 variables for the Modified Lorentzian (these two, plus I_0 , K , a , n and $2\theta_p$). With the Pearson Type VII there were four, (two for background plus I_0 , and a).

While a least-squares solution is possible for a parabolic fit, this is not as simple for the other two functions (except for the simpler Pearson Type VII forms). A modified simplex method was adapted in this study^{9,10}. Convergence was tested against the significance ratio between the best and worst points of the simplex, whose points were defined in terms of a goodness of fit, or reliability index, R :

$$R = \frac{\sum_{i=1}^n [I_i^{obs} - I_i^{calc}]^2}{\sum_{i=1}^n [I_i^{obs}]^2} \times 100. \quad (6)$$

In the simplex procedure, a multidimensional R space is formed ($n + 1$ dimensions, where n is the number of unknowns), one point from initial guesses at parameters and the others from fractional changes in each of the "guesses". Changes are then made in these values in a systematic way to reduce the range of these R values until by some test it is found that all values are essentially identical. This was judged by forming r , the ratio of the reliability index of the worst point in the simplex, to that of the best. This ratio was subjected to an F test¹¹, that is r was calculated as:

$$r_{p,n-p,\alpha} = \left[\frac{p}{n-p} F_{p,n-p,\alpha} + 1 \right]^{1/2} \quad (7)$$

Here p is the number of parameters, and α is the probability of incorrectly concluding that the best and worst points in the simplex are different. Iterations were continued until $r < r_{p,n-p,\alpha}$. The test value was 1.00018, which corresponds to an α value less than 0.005. Values even closer to unity were attempted, but beyond this value the error in $2\theta_p$ was not substantially improved. It is important in using the simplex procedure to make appropriate first guesses at the change in variables, so that the R values are far apart, and to accelerate convergence only changes in the parameters which decreased R were accepted; if an improvement occurred, the change was increased in the same direction. In effect, the point with the worst R is moved through the centroid of the ($n + 1$) sided polygon of R values to lower R . Finally, the entire simplex was contracted by moving all points in any iteration half the length of a side of the polygon toward the best point. Even with all these precautions, typically nearly 5 minutes were required on PDP 11/34 to accomplish the ~150 iterations in the case of a Modified Lorentzian with 7 parameters. This was reduced to ~1.5 minutes for 5 parameters, and was ~45 seconds for the Pearson Type VII (with 5 parameters).

STATISTICAL ANALYSIS

Our procedure was to remeasure a peak many times (typically 10) and to examine the variation in fitting parameters. Accordingly, we employed various statistical tests to judge the results. The mean of $2\theta_p$ and R for any set (n) of peaks fit by a single method was obtained, as well as the observed variance, S^2 .

$$S^2 (\langle 2\theta_p \rangle) = \frac{1}{n-1} \sum_i (2\theta_i - \langle 2\theta_p \rangle)^2, \quad (8)$$

and as well the true variance $\sigma^2 = S^2/n$. The range around the mean with 95% confidence limits was formed as $\langle 2\theta_p \rangle \pm 1.96\sigma(2\theta_p)$. The confidence limits for the variance was established with a chi-squared test (C) at the $1-\alpha$ confidence level as follows:

$$\frac{\sigma^2}{C_{1-\alpha}^2} < \sigma^2 < \frac{\sigma^2}{C_{1-\alpha}^2}. \quad (9)$$

For a given peak, the use of different profile fitting methods could lead to different $2\theta_p$. A test was made to ascertain if such differences were real or due to counting statistics. Let ℓ be the number of different profile methods for any one peak. Then the "profile method" variance was defined as:

$$S_{\ell}^2 (\langle 2\theta_p \rangle) = \frac{1}{\ell-1} \sum_{\ell} (2\theta_{\ell p} - \langle 2\theta_p \rangle)^2, \quad (10)$$

where $\langle 2\theta_p \rangle = \frac{1}{\ell} \sum_{\ell} 2\theta_{\ell p}$; ℓ is the number of peak positions for the different methods for the same peak.

The pooled variance for all methods is defined as:

$$S_p^2 = \frac{1}{\ell} \sum_{\ell} S^2 \quad (11)$$

With n peaks for each method, there are then $(n-1)$ degrees of freedom for each method, and $\ell(n-1)$ for S_p (and $[\ell-1]$ for S_{ℓ}). The ratio:

$$F = \frac{nS_{\ell}^2}{S_p^2}, \quad (12)$$

was then tested. If Eq. 12 is less than the tabulated F value for some confidence level, the difference in mean position is due to random counting statistics (with that confidence).

EXPERIMENTAL METHODS

Samples were chosen to represent a typical range of peak intensities and peak widths, and to include second-phase peaks. Their preparation and the peak characteristics are described in Table I. The diffraction conditions are in Table II. The data were obtained on a microprocessor controlled diffractometer. The parabolic fit was performed on line, and data were obtained in a three stage point-counting procedure described in Ref. 2, to locate the peak and the range of 2θ covering the top 15 pct. Seven points in this range were employed. For the other functions, data was obtained with the same system (without removing the specimen) at $.02^\circ 2\theta$ intervals (each counted for 15 seconds) across the entire profile, and transferred to a PDP 11/34 minicomputer for data processing. The data were not corrected for the Lorentz-polarization factor or scattering factor variation, as would be needed in actual stress analysis; only the fit to the shape and the value (and error) of $2\theta_p$ were of interest here.

RESULTS AND DISCUSSION

As with the parabolic fit, the Pearson Type VII distribution fits only the main part of the peak, but falls more rapidly in the tails than the observations, the difference being larger as "m" increases. (The region fit was actually similar to that for a parabola.) On the other hand, the Modified Lorentzian fits the entire shape quite well. A comparison of the various fits with ten recordings of three different peaks is given in Table III, and samples of the fits are shown in Fig. 4. A fixed linear background was employed for the results in this table; solutions with a variable background had worse errors in peak position, although with the significance tests at the 95 pct level these apparent differences were not necessarily real. More importantly, the errors in the fitting parameters showed drastic decreases, and the values of K and I_0 approached the measured peak width and intensity when the background was fixed prior to the solution for the other parameters.

It is evident from the table, that the Modified Lorentzian and parabolic fit provides similar error values for all peak shapes. This is true of the Pearson Type VII function as well but the value of m is different for each peak type. Thus, some knowledge of the peak shape would be required prior to fitting, which would make automation more difficult than for the other functions. Also, the Goodness-of-Fit values are quite high with this function.

Comparing the method and pooled variances indicated that the slight differences in peak positions in Table III for the various functions are significant in all cases except for the broad peaks. In this latter case the low intensity leads to larger scatter so that any difference is masked.

Note especially that the parabolic fit gives the lowest errors of any method for weak or broad peaks.

The 60/40 brass sample exhibited a doublet whose resolution varied with tilt of the sample to the x-ray beam (as would be done in a stress measurement). Examples of fits to this peak are given in Fig. 5, and a summary of results in Table IV. The singlet forms of the Modified Lorentzian and Pearson Type VII functions gave values of $\Delta 2\theta[(\psi = 0^\circ) - (\psi = 45^\circ)]$ closer to the values of the parabola, despite the fact that the $\langle R \rangle$ values were lower with the doublet form. From the analysis of variance at both angles, the differences in peak positions with each fit technique in this table were significant.

Possible limitations in the data that could occur in practice were also explored. Firstly, it may not be possible to record the entire peak; another one nearby may overlap on one side, or the equipment itself could preclude recording the entire peak. Some results are shown in Table V. With a Modified Lorentzian and a sharp peak the "correct" (parabolic) peak position, a low error in this value, and a low R value are all obtained with data that only just reaches the peak's maximum. Similar tests with a broad profile showed that a wider range was necessary, but only a few tenths of a degree 2θ beyond the maximum is adequate. Analysis of variance tests confirmed that beyond 68.82° , any change in peak position is solely due to random fluctuations, and the peak values obtained with data up to and beyond this angle are not distinguishable from the value for the entire peak, at 95 pct confidence.

Next, with the Modified Lorentzian, the effect of time per data point and the number of data points were explored, Table VI. The error is not statistically different at the shorter counting time, or smaller number of points.

TABLE I.
TYPICAL TEST CURVE FEATURES

MATERIAL	PREPARATION ¹	PEAK ² INTENSITIES (CPS)	BACKGROUND ³ INTENSITIES (CPS)	PK/BKD RATIO	FWHM (° 2θ)	PEAK TYPE
STEEL						
1008, 110 _a	As rolled and grit blasted	1221	427	2.86	.65	Broad
1008, 110 _a	Same + 2 hrs. 723°K	1546	250	6.18	.42	Sharp
1074, 021 _c } Fe ₃ C 112	Cast, grit blasted + 723°K, 2hrs.	151	122	1.23	.40	Weak
BRASS						
60/40, 211 _β }	Grit blasted + 1 hr. 673°K	398	167	2.38	.48	Doublet φ = 0°
60/40, 211 _β }		256	114	2.24	1.02	Doublet φ = 45°

1. All samples were polished with 600 grit paper to remove oxidation.

2. Peak intensity is that for K_{α1} radiation.

3. Background intensity is the cps value at the peak, obtained from a straight line regression using intensity values beyond each tail.

[For further details the reader is referred to Ref. 12.]

CONCLUSIONS

1. The parabolic fit has the best overall ability to determine peak positions over a wide range of shapes.
2. A Modified Lorentzian can be quite helpful if only a part of a peak can be explored, especially for sharp peaks.
3. The errors in a Modified Lorentzian fit are not very sensitive to counting time. With a position sensitive detector precision comparable to that for a parabolic fit can be obtained in about one tenth the time. As all of the data are recorded for either fit with this type of detector, a considerable saving in measurement time is possible with a Modified Lorentzian function. Unfortunately, the fit itself takes the order of 1 minute on a miniprocessor. If this time could be reduced with a specially designed microprocessor for this purpose, this function offers a way of drastically reducing the time for stress measurements in the field, beyond that already achieved by the use of a PSD.
4. Goodness-of-Fit is not necessarily an accurate gage of error in peak position. (For example, in Table III, part (c), similar values of R are associated with widely different peak locations.)

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TABLE II
OPERATING CONDITIONS

FEATURE	SETTING	SAMPLES
Divergent Slit	1°	All
Receiving Slit	.15°	Annealed 1008, 1074, Brass
Beam Size on Sample at $\phi = 0^\circ$ (approximate)	2.5mm x 2.5mm	All
Tube Target	Cr	All
Tube Voltage- Current	40kv - 10ma 35kv - 10ma 40kv - 15ma 35kv - 10ma	Annealed 1008 Deformed 1008 1074 Brass
Filter	Vanadium Oxide	All
Soller Slits	None used	All

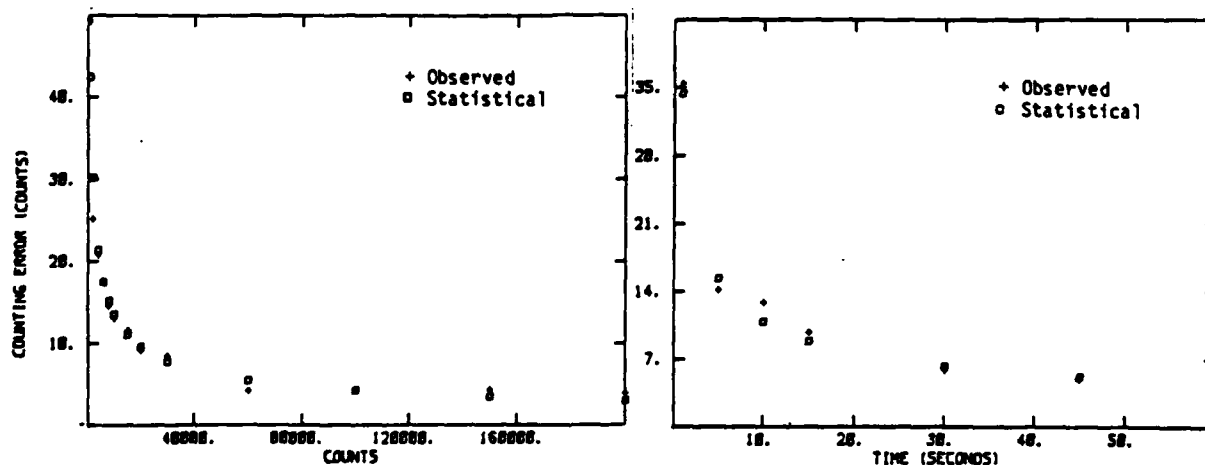


FIG: 1. Comparison of statistical error in intensity measurements with observed error. Left: Fixed counts, Eq. 2a, Right: Fixed time measurements, Eq. 2b. Twenty five replications of a point on a 211 ferrite reflection from a 1008 steel was employed with filtered CuK_α radiation.

TABLE III.

COMPARISON OF FIT METHODS¹

METHOD	MEAN 2 p	OBSERVED ERROR ($\times 10^{-2}$ degrees)	95% LIMITS ON ERROR ⁴ ($\times 10^{-2}$ degrees)	MEAN GOODNESS-OF- FIT
A) Curve Type: Sharp (Annealed) Ferrite 110, 1008 Steel				
Parabolic ²	68.82193	.287	.197, .514	29.91
Pearson Type VII ³				
m = 3	68.81368	.473	.326, .863	30.24
m = 2	68.82818	.172	.118, .314	32.43
m = 1	68.82902	.282	.194, .515	41.77
m =	68.80705	.172	.118, .314	33.43
Modified Lorentzian ²	68.83298	.135	.093, .246	1.75
B) Curve Type: Broad (deformed) Ferrite 110, 1008 Steel				
Parabolic	68.53719	.173	.199, .316	1.16
Pearson Type VII				
m = 3	68.53853	.511	.352, .870	20.53
m = 2	68.54362	.334	.229, .609	9.95
m = 1	68.53595	1.40	.963, 2.56	17.07
m =	68.54447	.277	.191, .506	11.21
Modified Lorentzian	68.54165	.351	.242, .641	1.88
C) Curve Type: Weak Fe₃C 112 + 021, 1074 Steel				
Parabolic	57.42794	1.05	.723, 1.92	2.79
Pearson Type VII				
m = 3	57.35688	.263	.181, .480	26.5
m = 2	57.36416	1.78	1.22, 3.25	17.2
m = 1	57.36556	.785	.540, 1.43	6.98
m =	57.40937	5.15	3.54, 9.40	4.49
Modified Lorentzian	57.40813	1.28	.881, 2.34	2.11

1. 15 Seconds per point, 10 peaks per mean, Fixed background - 5 points either side of peak used to fix line (for parabola, see ref. 2).
2. Parabolic fit is 7 pt. fit to top 15%. A single-valued background is subtracted before fit.
3. All Pearson Type VII and Modified Lorentzian utilized 90 pts. taken at $.02^\circ$ 2 increments, except for Fe₃C, for which 58 points were measured.
4. Values listed are low and high limits respectively.

TABLE IV.
DOUBLET ANALYSIS¹

FITTING METHOD	PREDICTED SHIFT WITH TILT	$\psi = 0^\circ$				$\psi = 45^\circ$			
		$\langle 2\theta_p \rangle$ ($^\circ 2\theta$)	ERROR ($\times 10^{-2}$)	$\langle R \rangle$	$\langle 2\theta_p \rangle$	ERROR ($\times 10^{-2}$)	$\langle R \rangle$		
Parabolic	.082	144.07920	.555	.291	143.99692	.802	3.74		
Pearson Type VII ($m = 3$)									
W/Doublet	.078	144.04743	6.206	11.34	143.96992	.659	4.45		
W.O./Doublet	.133	144.19838	4.015	12.34	144.06610	8.56	6.81		
Modified									
Lorentzian									
W/Doublet	.088	144.05093	2.665	4.45	143.96318	5.49	3.95		
W.O./Doublet	.020	144.07462	1.576	7.86	144.05425	1.19	4.62		

1. 60/40 Brass, 211 peak, CrK_α Radiation, 10 scan replications, 78 points per peak).

TABLE V.

PARTIAL FIT OF SHARP PEAK (1008 STEEL) TO MODIFIED LORENTZIAN

TWO THETA VALUE OF LAST POINT	$\langle 2\theta_p \rangle$ ($^\circ 2\theta$)	OBSERVED ERROR	$\langle R \rangle$
68.70	68.77712	.296E-1	1.48
68.74	68.79541	.584E-2	1.47
68.78	68.82561	.341E-1	1.70
68.82	68.83322	.101E-2	1.74
68.86	68.83303	.836E-3	1.74
68.90	68.83298	.135E-2	1.75
68.94	68.83226	.113E-2	1.75
68.98	68.83322	.110E-2	1.74
69.02	68.93300	.937E-3	1.72

10 second count per point, $0.02^\circ 2\theta$ intervals.

Lowest two theta value in all cases was 67.50° .

'Value of Last Point' indicates the highest two theta value used in analysis.

Peak value, as determined from the full angular range fit, is $68.83299 \pm .00135^\circ 2\theta$.

A fixed background, obtained from 10 data points on the low angle side of the curve, was used.

TABLE VI

EFFECT OF NUMBER OF POINTS-MODIFIED LORENTZIAN, BROAD CURVE (1008 STEEL)

TIME PER POINT (SEC.)	NUMBER OF POINTS ¹	MEAN PEAK POSITION ($^\circ 2\theta$)	OBSERVED ERROR ($\times 10^{-2}$)	$\langle R \rangle$
1	22	68.54718	.572	3.09
	90	68.54763	.434	3.59
15	22	68.54224	.360	2.07
	90	68.54165	.351	1.88

1. The 22 points were selected as every fourth point of the full 90 point fit. Background line determined from 5 points on either side of the peak.

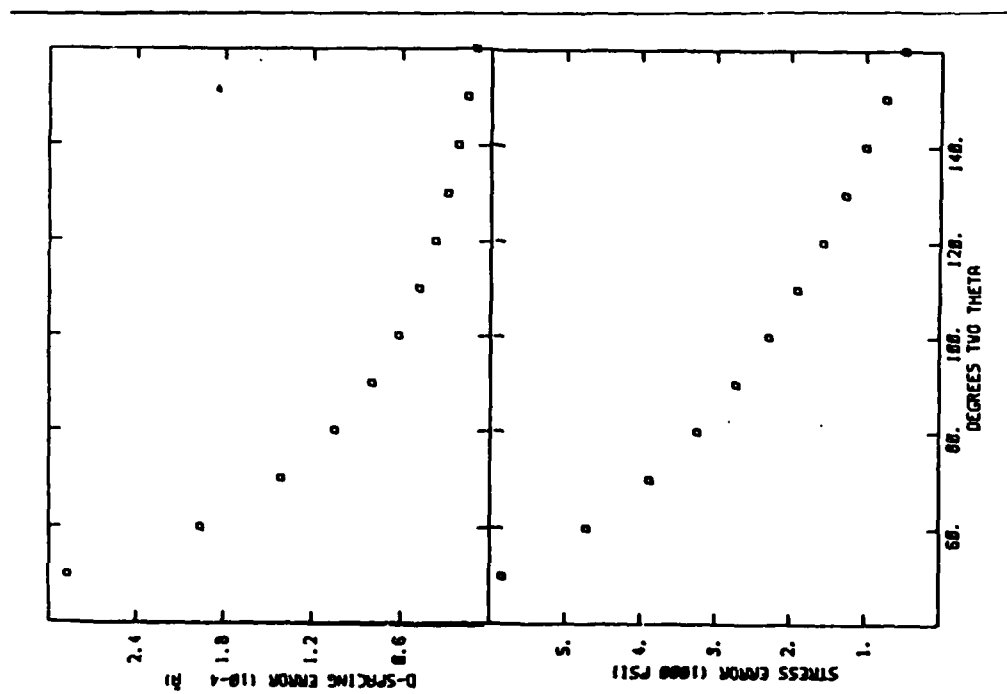


FIG. 2: Estimated statistical error in: a) "d"-spacing and b) stress, as a function of diffraction angle. A seven point parabola was assumed.

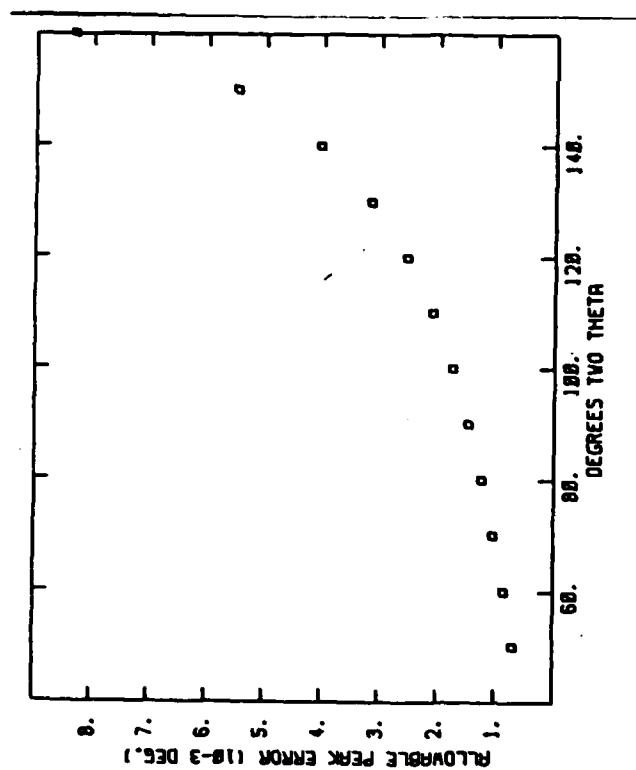


FIG. 3. Allowable error in peak 2θ for a maximum 1000 psi error in stress, as a function of diffraction angle. Seven point parabolic fit.

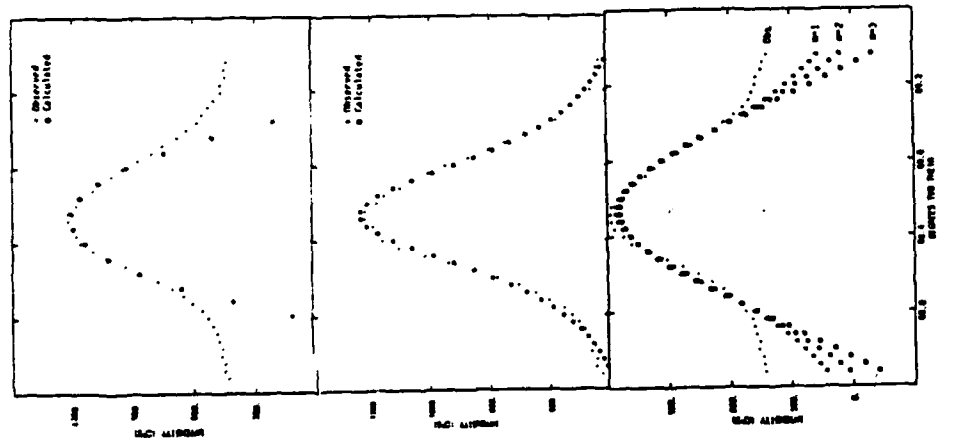


FIG. 4. Various fits to the 110 ferrite peak, 1008 steel, 15 seconds counting per point. Top: 7 point parabolic fit, top 15 pct. Middle: Modified Lorentzian function. Bottom: Pearson Type VII distributions.

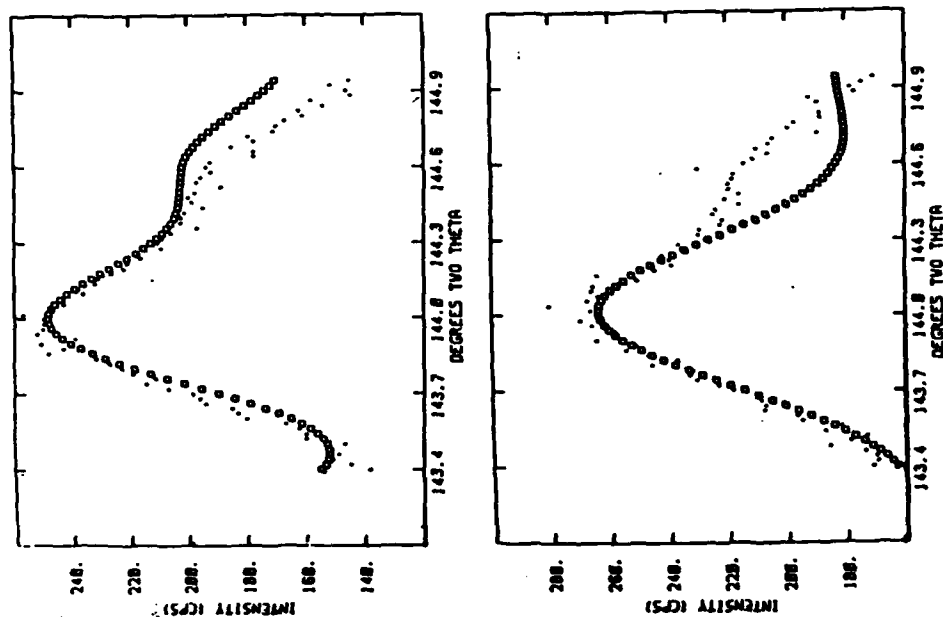


FIG. 5. Fit of the Modified Lorentzian function to the 211 β reflection from 60/40 brass, $\psi = 45^\circ$. a) doublet equation, b) single function. Dots are observations, open figures are fit.

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13. ABSTRACT It is shown that there is no improvement over a parabolic fit in peak position determinations by profile fitting with a modified Lorentzian function or a Pearson Type VII function. Furthermore, error evaluation is more difficult. The modified Lorentzian does offer certain advantages: 1) much increased speed, 2) portions of the peak can be eliminated with no loss of precision.			

14. KEY WORDS	LINK A		LINK B		LINK C	
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