THE USE OF PEARSON VII DISTRIBUTION FUNCTIONS IN X-RAY DIFFRACTION RESIDUAL STRESS MEASUREMENT

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ABSTRACT

The fitting of a parabola by least squares regression to the upper portion of diffraction peaks is commonly used for determining lattice spacing in residual stress measurement. When Kα techniques are employed, the presence of the Kα doublet is shown to lead to significant potential error and non-linearities in lattice spacing as a function of $Sin^2\psi$ caused by variation in the degree of blending of the doublet. An algorithm is described for fitting Pearson VII distribution functions to determine the position of the $K\alpha_1$ component, eliminating errors caused by defocusing of diffraction peaks of intermediate breadth. The method is applied to determine the subsurface residual stress distribution in ground Ti-6Al-4V, comparing directly the use of parabolic and Pearson VII peak profiles, and is shown to provide precision better than ±1% in elastic constant determination.

INTRODUCTION

Anomalous non-linearities in lattice spacing as a function of $Sin^2\psi$ have been reported in the literature and observed by the author [11] employing parabolic peak profile fitting Kα radiation techniques for residual stress measurement on machined or ground surfaces. The oscillations frequently are seen not in the highly deformed surface layers, but in the undeformed layers exposed by electropolishing, and are not explained by the residual stress models proposed by Marion and Cohen [12] or Dölle and Cohen.[13] The nature and degree of non-linearity can be altered by changing the x-ray optics to affect resolution of the doublet. The phenomenon now appears to be a result of experimental error associated with the least squares fit of parabolic profiles to the top of diffraction peaks of intermediate broadening in the presence of variable blending of the Kα doublet caused by defocusing as ψ is changed during stress measurement.

Many practical techniques for x-ray diffraction residual stress measurement employ Kα radiations to provide intense diffraction peaks at the high back reflection region. Kα techniques have the disadvantage of producing a diffraction peak doublet composed of the $K\alpha_1$ and $K\alpha_2$ components. The presence of the two closely spaced diffraction peaks, which generally cannot be separated instrumentally, can lead to significant uncertainty in the determination of the lattice spacing to the precision required for the calculation of lattice strain.

If the Kα diffraction peaks are extremely broad, as in hardened steels, the x-ray wavelength can be considered to be a weighted average for the doublet, and the fully blended doublet located precisely by calculating the vertex of a parabolic profile fitted to the top 15 percent of the combined doublet.[1] When the doublet can be resolved, a parabolic profile can be fitted to the top of the $K\alpha_1$ peak alone. However, for a broad class of specimens encountered in practice, the Kα doublet can be neither completely blended nor resolved for all ψ angles required for residual stress measurement. The position of the diffraction peak calculated from the vertex of the fitted parabola will then depend upon both the fraction of the doublet included in the regression analysis and the degree of blending of the doublet caused by defocusing as the sample is rotated in the incident x-ray beam.

The use of centroids or cross-correlation for peak location has the advantage of being independent of the shape of the diffraction peak, but requires integration of the diffracted intensity over the entire peak profile.[10] The accuracy of these integration methods is dependent upon the precision with which the intensity in the tails of the diffraction peak can be determined and upon the range of integration. The diffracted intensity must be measured at small angular increments to provide a precise definition of the entire diffraction peak profile. The Rachinger[2] correction can be applied to separate the Kα doublet without assuming a form for the diffraction peak

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profile. However, accurate correction again requires a large number of data points spanning the entire diffraction peak. Further, the method requires that the angular separation of the doublet be assumed prior to correction, which, in effect, assumes the diffraction angle being measured. The accuracy of the Rachinger correction diminishes rapidly on the $K\alpha_2$ side of the doublet, and may lead to inaccuracies in the determination of the position of the $K\alpha_1$ peak, too large to be tolerated in residual stress measurement.[3]

If the profile of the combined doublet can be adequately approximated by a suitable function for any degree of resolution, the peak position could be determined by least squares fitting of the function to intensity data collected at only a few angles, as in the case of the parabola method. To this end, the fitting of a combined Kα doublet peak profile derived from Pearson VII distribution functions was undertaken as proposed by Gupta and Cullity.[3]

APPROXIMATION OF THE Kα DOUBLET USING PEARSON VII DISTRIBUTION FUNCTIONS

Pearson VII distribution functions have been used for years to approximate the form of diffraction peaks for a variety of purposes,[4,5,6] but are not commonly applied for the determination of peak position in residual stress measurement. The Kα doublet profile may be approximated as a summation of Pearson VII distribution functions, composed of a $K\alpha_1$ peak displaced by a fixed increment (dependent upon the difference between the Kα wavelengths) from a $K\alpha_2$ peak of identical shape, but having an intensity which is a fixed fraction of the $K\alpha_1$ intensity.[3]

If y(x) is the intensity as a function of angular position x, the general form of such a function describing the combined Kα doublet is

$$y(x) = A f\left(x - x_o\right) + CA f\left(x - x_o - \delta\right) \tag{1}$$

where f(x) is the Pearson VII distribution function, A is the intensity of the $K\alpha_1$ peak, x_O is the angular position of the $K\alpha_1$ peak, δ is the angular separation of the doublet, and C is some fixed fraction (typically 0.5) relating the intensity of the $K\alpha_1$ and $K\alpha_2$ peaks.

The general form of the Pearson VII distribution function, ranging from the extremes of Cauchy to Gaussian profiles, is

$$f(x) = \left[1 + K^2\left(x - x_o\right)^2 / M\right]^{-M} \tag{2}$$

where K governs the width of the profile, and M the rate of decay of the tails. For M = 1, the profile is purely Cauchy; for M = 2, a Lorentzian; and for M = infinity, the profile is purely Gaussian. A continuous range of profiles may be generated as a weighted summation of Cauchy and Gaussian components.

In order to locate the position of the $K\alpha_1$ line contributing to the unresolved doublet, the combined profile described in Equation (1) must be fitted to data points collected across the diffraction peak by non-linear least squares regression. The task is complicated if M is allowed to be a variable during the regression analysis. Fortunately, the diffraction peak profiles in the high back-reflection region, as are used for residual stress determination, may be closely approximated by a purely Cauchy profile.[7] This observation was verified by fitting both Cauchy and Gaussian profiles to diffraction peaks representing a variety of peak breadths and degrees of doublet resolution. Profiles fitted to data points with $N = 3 \times 10^5$ for a hardened steel and powdered iron are shown in Figures 1 and 2. Significant deviation from the Cauchy peak profile occurs primarily in the tails on the broadest peaks. Purely Gaussian profiles were found to drop away too rapidly in the tails of the diffraction peak for adequate approximation. The sum of the squares of the residuals was approximately twice as large for the Gaussian than for the Cauchy profiles for all of the samples examined. Therefore, a summation of purely Cauchy profiles with M = 1 was adopted as adequate for an approximation of the Kα doublet.

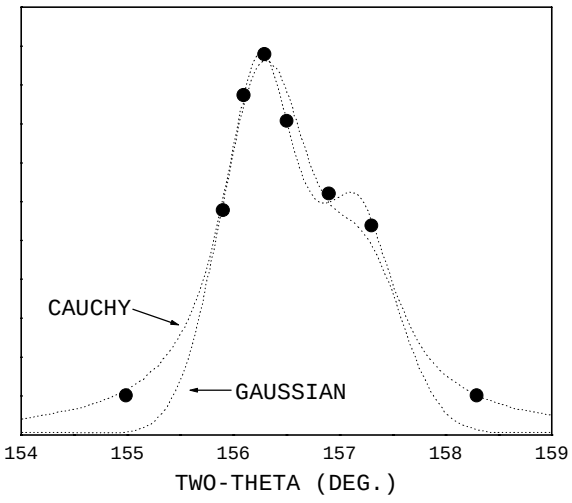

Fig. 1 - Cauchy and Gaussian Profiles, Powdered Iron (211) Cr Kα.

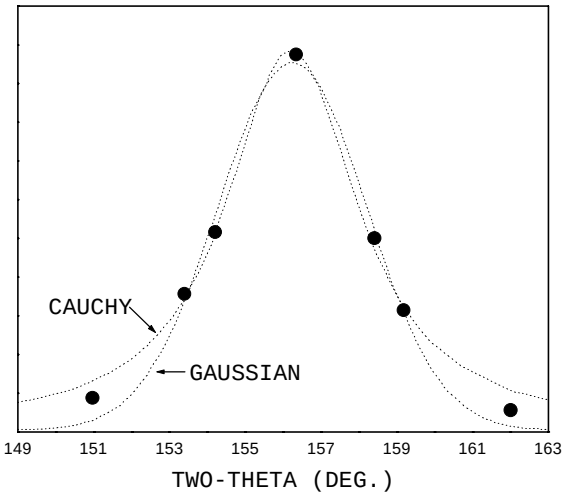

Fig. 2 - Cauchy and Gaussian Profiles, Carburized 9310 Steel, (211) Cr Kα.

An algorithm was developed for fitting the combined Cauchy doublet profile by non-linear least squares regression using the method of linearization and successive approximation. The program returns the values of K, A, x_O and δ for a Cauchy profile, using five or more inverse intensities measured to high accuracy, spanning the doublet. Between each iteration of the regression process, the spacing, δ, between the $K\alpha_1$ and $K\alpha_2$ peaks, is refined based upon the position of the $K\alpha_1$ peak determined in the previous iteration. The intensity of the $K\alpha_1$ and $K\alpha_2$ peaks, given by C in Equation (1), is held constant. The value of C was taken to be 0.5 for the results presented here, but could be adjusted to reflect actual measured intensities of the Kα components of the doublet. The program, running on a Z80 microprocessor system in compiled BASIC, will close to a solution with a variation of less than one part in 10^5 between iterations in less than ten iterations, requiring approximately 10 seconds. The method has been used for over 5,000 individual stress measurements on a broad range of alloys without failure to converge using data collected with both parafocusing diffractometers and a PSD.

COMPARISON OF PARABOLIC AND CAUCHY PROFILES FOR THE COMBINED DOUBLET

Assuming that a summation of Cauchy profiles provides an accurate representation of the (211) diffraction peak doublet produced using chromium Kα radiation for a simulated stress measurement in a steel specimen, a family of profiles representing a range of peak breadths is shown in Figure 3. The $K\alpha_1$ peak is assumed to be located at precisely 156.0 degrees, and the $K\alpha_1$ and $K\alpha_2$ wavelengths are assumed to be 2.28962 and 2.29351 Å, respectively. Combined profiles are shown for values of K ranging from 0.5 to 4.0, representing complete blending and separation of the Kα doublet. As seen in Figure 3, the vertex of the diffraction peak, which would be determined by fitting a parabola, shifts from 156.0 degrees for the resolved $K\alpha_1$ peak to approximately 156.3 degrees as the doublet is blended.

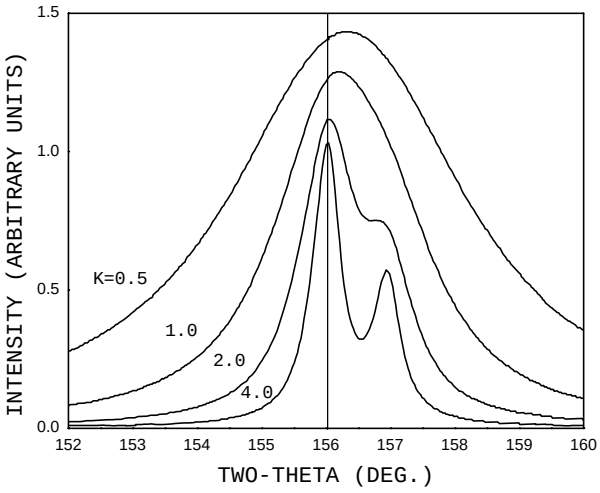

Fig. 3 - Cauchy profiles

For diffraction peaks of intermediate width ($0.5 < K < 2.5$), substantial error in determining the peak position may result from the use of a parabolic profile and a fixed weighted average wavelength from two sources. First, the peak breadth will vary as a result of defocusing as ψ is changed during the course of stress measurement even if parafocusing is used. The degree of defocusing and the resulting variation in K, encountered during a single stress measurement, will be a complex function of the incident beam divergence, silt and focal spot geometry, the range and sign of ψ tilts employed, and the diffraction angle.[8] In general, the degree of defocusing can be expected to increase rapidly as the diffraction angle decreases, and to be greater for a fixed slit than for a parafocusing technique. Second, the fraction of the upper portion of the diffraction peak included in the parabolic regression procedure, in practice, may be difficult to control. For diffraction from the surface of highly deformed specimens, where the diffracted intensity is low and the background intensity high, uncertainties in the precise background intensity may lead to significant variation in the percentage of the diffraction peak included in the analysis for different ψ angles.

Figure 4 shows the angular error in the determination of the $K\alpha_1$ peak position for a parabolic profile fitted

to the combined Cauchy doublet using from 10 to 35 percent of the diffraction peak in increments of 5 percent as a function of K. Figure 4 was derived by fitting a parabola by linear least squares regression to the data points shown in Figure 3 at increments for K of 0.1. The simulated intensity points are uniformly spaced at 0.0625 degree increments. No random error was assumed in the simulation. The number of data points included in the parabolic regression analysis ranged from 82 points for the 30 percent, K = 0.3 case, to a minimum of 4 points for the 10 percent, K = 4.0 case. The "+" symbols in Figure 4 represent the calculated positions for the diffraction peak, using the parabolic fit. The points have been simply connected by straight lines for presentation.

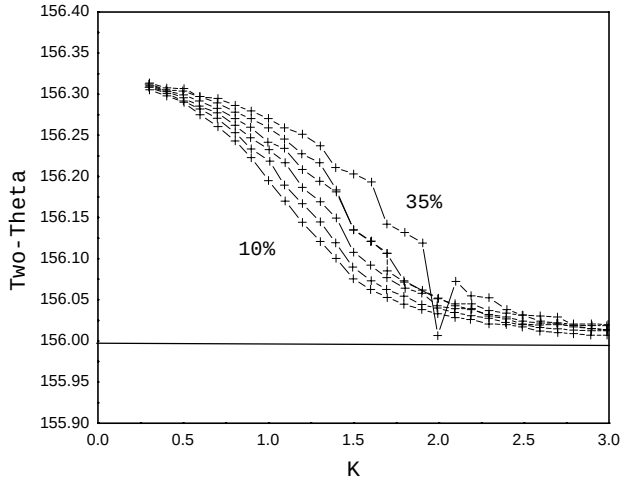

Fig. 4 - Angular error for parabolic profiles

For fully blended or separated doublets (K < 0.5 or K > 2.5), the position of the diffraction peak determined using parabolic regression becomes virtually independent of both peak width and the fraction of the profile included in the analysis. No significant error would result, provided the appropriate wavelengths were used ($K\alpha_1$ or weighted average) to calculate lattice spacing. For intermediate resolution (0.5 < K < 2.5), the position of the peak determined from the parabolic regression procedure is highly dependent upon both the peak breadth and the fraction of the peak included with maximum sensitivity at approximately K = 1.5. Significant error could result from variation in either parameter. As increased fractions of the peak are included in the parabolic analysis of partially resolved doublets (1.5 < K < 2.5), and the "shoulder" formed by the $K\alpha_2$ peak is included, the solution by parabolic regression becomes unstable for fractions in excess of 25 percent.

If the parabolic method of peak location is used to determine absolute rather than relative lattice spacings (as required to determine the full residual stress tensor), quite different results would be expected for d_0 determined on the surface of a specimen deformed by grinding or machining, where K is small due to line broadening, and beneath the surface where the material may be stress free and undeformed. The practice of determining d_0 beneath the deformed surface using parabolas could then lead to significant error.

An indirect method of correcting for blending of the $K\alpha$ doublet using Pearson VII functions has been described by Dölle and Cohen.[13] A parabola is fitted to the upper portion of the combined doublet and the position of the $K\alpha_1$ peak is calculated from an error function similar to those shown in Figure 4 derived from combined Gaussian profiles representing the $K\alpha_1$ and $K\alpha_2$ components of the doublet. There are several difficulties inherent in this method which are eliminated by fitting the Pearson VII profiles directly. First, the form of the peak profile used to generate the correction function must be assumed (Gaussian for Dölle and Cohen). The correction function is highly dependent upon the form assumed. Although only Cauchy profiles are presented here, the algorithm has been refined to fit generalized Pearson VII functions where M is a variable in the regression analysis. Second, the use of a fixed correction function presupposes both the separation of the doublet and the portion of the diffraction peak included in the parabolic regression analysis, which as seen from Figure 4, is itself a source of error. Third, the width of the diffraction peak, which is also a sensitive function of the fraction of the peak included in the parabolic regression analysis, must be determined separately in order to apply the correction, introducing an additional source of experimental error.

The use of parallel beam optics would reduce the variation in blending of the doublet as a function of ψ tilt during measurement. Whether the variation in the degree of blending would be less than observed with parafocusing or eliminated entirely with the low incident beam divergence achievable is beyond the author's experience. Errors due to variation in the portion of the diffraction peak included in the parabolic regression analysis would still remain.

The method of diffraction peak location by fitting Pearson VII functions proposed here would still be subject to error in the event of defocusing so severe as to cause asymmetry in the individual doublet component profiles, as would peak profile approximation using any symmetrical function. The

method also requires accurate determination of the background intensity, which is not required for peak location using simple parabolic regression.

APPLICATION TO RESIDUAL STRESS MEASUREMENT IN GROUND TI-6AI-4V

A direct comparison was made using both parabolic and Cauchy profiles on the identical five data points collected in the top 20 percent of the (21.3) diffraction peak produced with Cu Kα radiation for a positive ψ tilt $\sin^2\psi$ technique on a sample of ground Ti-6Al-4V, assuming the plane stress model. Data were collected at the surface and as a function of depth to a maximum of 0.175 mm, removing material for subsurface measurement by electropolishing. Measurements were made on a horizontal GE goniometer, with a 1.0 degree incident beam divergence and a 0.2 degree receiving slit, using a parafocusing technique, Si(Li) detector, and six positive ψ tilts, to a maximum of 45 degrees at even increments of $\sin^2\psi$. The elastic constant, E/(1+v) = 84.1 ± 0.5 GPa, was determined empirically using the four-point bending technique previously described. [9]

The inverse intensity was determined at five points in the top 20 percent of the diffraction peak, for N = $3x10^4$, and corrected for a linearly sloping background intensity, Lorentz polarization and absorption, prior to fitting the parabolic and Cauchy profiles. The lattice spacings for the parabolic profiles were calculated using the weighted average of the Kα doublet, while the Cauchy profiles were calculated for the $K\alpha_1$ wavelength alone. The results shown have been corrected for penetration of the radiation into the subsurface stress gradient.

Plots of d (21.3) and the value of K, derived from the fitted Cauchy profile describing the peak breadth, are plotted as functions of $\sin^2\psi$ for selected depths in Figures 5 through 8. The stress derived by fitting a straight line by least squares regression through d as a function of $\sin^2\psi$ is shown for both the parabolic and the Cauchy profiles with the error indicated by one standard deviation, based upon the uncertainty in the slope of the fitted line.

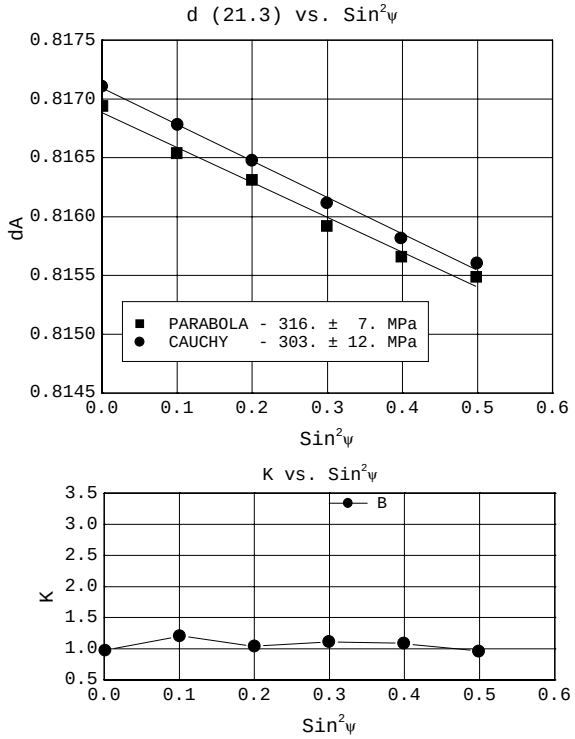

Fig. 5 - Ground Ti-6Al-4V surface

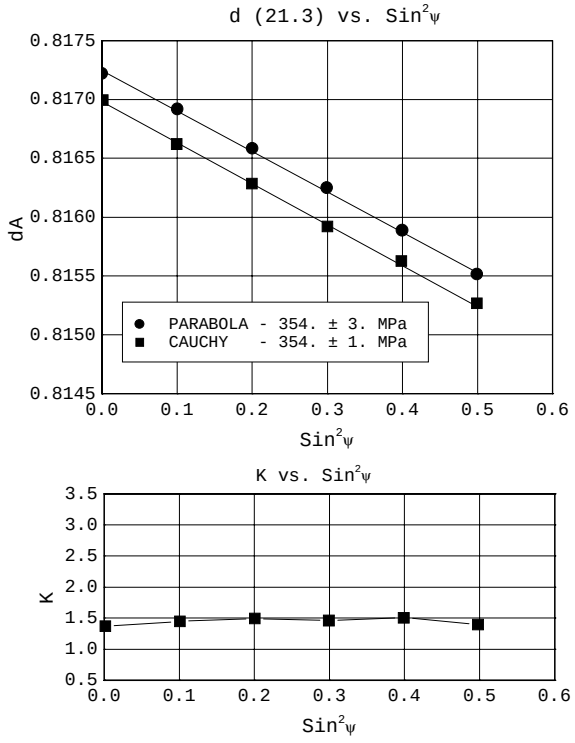

Fig. 6 - Ground Ti-6Al-4V 0.010 mm.

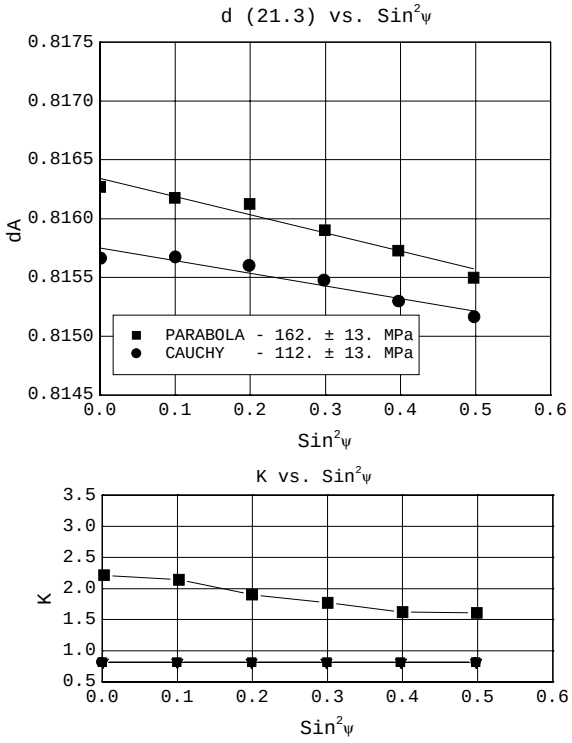

Fig. 7 - Ground Ti-6Al-4V 0.025 mm.

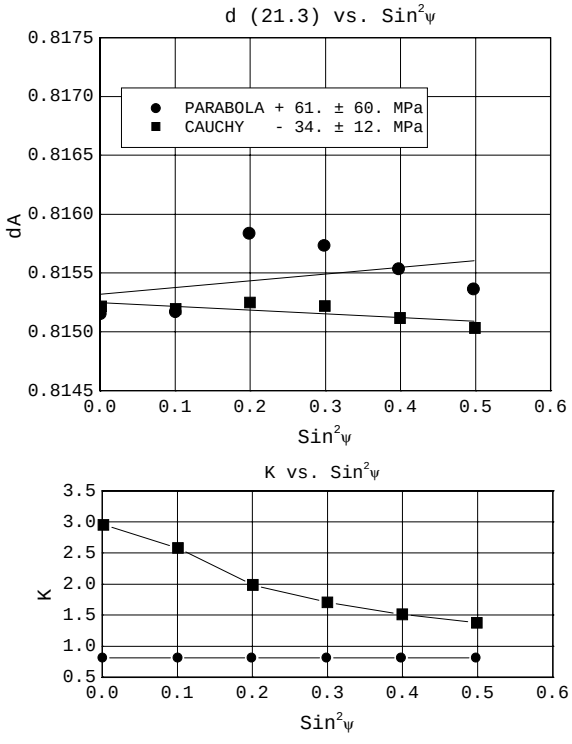

Fig. 8 - Ground Ti-6Al-4V 0.175 mm.

No significant differences are observed in the residual stresses derived from the Cauchy and parabolic profiles at the surface or at 0.01 mm. At these depths, shown in Figures 5 and 6, line broadening is dominated by the plastic deformation of the alloy, and the diffraction peak width, indicated by K, does not vary significantly with ψ. At 0.025 mm, shown in Figure 7, a significant difference is observed in the stress derived from the parabolic and Cauchy profiles, although no pronounced anomalies are evident. The discrepancy of nominally 40 MPa between the results accompanies a linearly varying peak breadth due to defocusing. At the remaining three depths, from 0.048 to 0.175 mm, pronounced anomalies in the d-Sin^{2}ψ plots derived from the parabolic profile are evident. Typical results are shown for the maximum depth in Figure 6. The Cauchy results yield a constant stress of nominally -34 MPa at the three depths, while the parabolic results give values ranging from + 86 to + 61 MPa, with the standard deviation approaching the magnitude of the stress measured.

The residual stress distributions, showing the mean value and standard deviations, are presented for both parabolic and Cauchy profiles as functions of depth in Figure 9. The sample was in the form of a thick walled tube approximately 10 cm in diameter with a 3.8 cm wall. The stresses measured were in the circumferential direction parallel to the grinding direction. Failure to achieve equilibrium in the range of depths examined is attributed to hoop stresses of low magnitude in the tube.

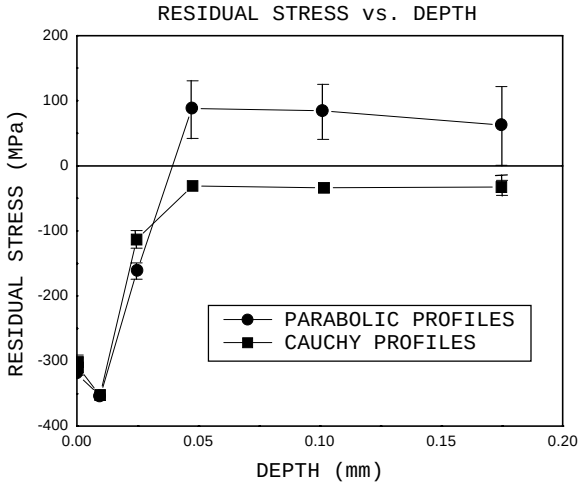

Fig. 9 - Ground Ti-6Al-4V

X-RAY ELASTIC CONSTANT DETERMINATION

The Cauchy profile procedure for locating the $K\alpha_1$ peak position has been applied to the determination of x-ray elastic constants by the four-point bending

technique.[9] The results indicate a significant reduction in the random error in the determination of the lattice spacing and, therefore, improvement in the precision with which the elastic constant, E/(1 + v) can be determined, compared to the parabolic regression method previously used. Typical results for titanium alloy IMI-679, showing the change in lattice spacing for the (21.3) planes as a function of applied stress, are shown in Figure 10. A total of 13 data points are plotted, five at the maximum and minimum loads, and three at the intermediate load, to test linearity. The uncertainty shown is one standard deviation based upon the line fitted by least squares regression. The x-ray elastic constants for the (211) direction for seven steels, determined using the Cauchy profile method, are presented in Table I. The random error, due primarily to uncertainties in peak location, is 1.06 or less as indicated by the standard deviations and represents a reduction in random error to approximately half that routinely achieved with parabolic regression.

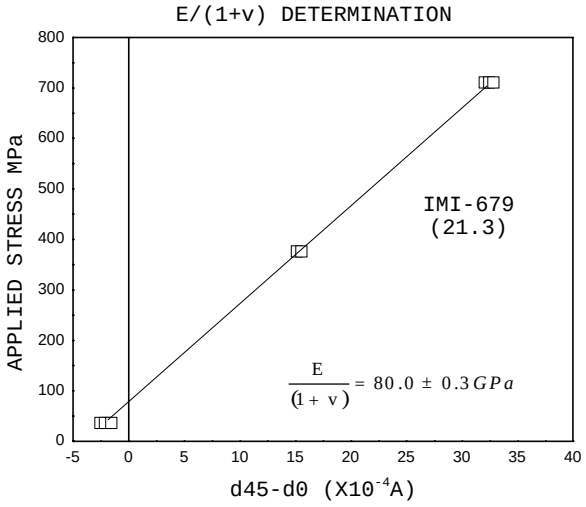

Fig. 10

TABLE I

X-RAY ELASTIC CONSTANTS FOR SELECTED STEELS
Determined Using the Cauchy Peak Profile Method
(211) Cr Kα1 2θ = 156 deg.

Steel	Hardness (Rc)	E/(1+) ($x10^6$ psi)	GPa	S.D.%
SAE 1050	56.	26.72 ±0.27	184.2 ±1.9	1.0
SAE 5150	34.	26.98 ±0.08	186.0 ±0.5	0.3
AISI 15B48	44.	25.86 ±0.10	178.2 ±0.7	0.4
AISI 15B48	15.	28.12 ±0.05	193.4 ±0.3	0.2
MIL-S-46850	50.	23.24 ±0.07	160.2 ±0.5	0.3
High C Tool (1.3 C)	27.	28.64 ±0.08	197.5 ±0.5	0.3
AISI 402 SS	22.	26.32 ±0.10	181.5 ±0.7	0.4

CONCLUSIONS

A method of locating the diffraction peak in the high back-reflection region for x-ray diffraction residual stress measurement, using the Cauchy sub-class of Pearson VII distribution functions to approximate the combined Kα doublet, has been developed. The position, intensity, and breadth of the $K\alpha_1$ peak can be determined from the intensity measured for as few as five points spanning the Kα doublet. The method has been demonstrated to provide a more reliable method of determining lattice spacing in residual stress measurement than the method of fitting parabolic profiles by least squares regression for diffraction peaks of intermediate breadth. Anomalies in d vs. $Sin^2\psi$, which result from variable blending of the doublet caused by defocusing and variation in the portion of the peak included in the analysis, appear to be essentially eliminated by the method.

If the diffraction peak breadth happens to vary nearly linearly with $Sin^2\psi$, significant error may result from the use of parabolic regression, even though anomalies are not evident in the $Sin^2\psi$ plots. Determination of the unstressed lattice spacing, d_0, using parabolic regression in subsurface material for studies of triaxial stresses in deformed surfaces, may result in significant error.

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