THE MEASUREMENT OF SUBSURFACE RESIDUAL STRESS AND COLD WORK DISTRIBUTIONS IN NICKEL BASE ALLOYS

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ABSTRACT

A method of determining the diffraction peak width accurately and rapidly in conjunction with x-ray diffraction residual stress measurement using Pearson VII function profile analysis is described. An empirical relationship between the (420) diffraction peak width and the degree of cold work is developed for four nickel base alloys. The peak width produced was found to be independent of the method of deformation. Examples of the concurrent determination of subsurface residual stress and cold work distributions are presented for samples of abraded and shot peened Inconel 718 and ground Inconel 600.

X-RAY DIFFRACTION TECHNIQUES can be used to determine both macroscopic and microscopic residual stresses. Macrostresses, which extend over ranges large compared to the dimensions of the crystals in the material, are determined from the shift in the position of the diffraction peak. Microstresses, which extend over distances on the order of the unit cell, cannot be directly measured individually but can be quantified in the aggregate from the broadening of the diffraction peak which they produce.

Macrostresses are tensor properties measurable by mechanical means which are additive to applied stresses. The average magnitude of the microstress in a sample can be treated as a scalar property and is related to the hardness or degree of cold work of the material. As a metal is cold worked, the dislocation density increases, thereby reducing the size of the perfectly crystalline regions or crystallites (coherent diffracting domains) and increasing the average (root mean square) microstrain in the crystal lattice. The reduced crystallite size and increased microstrain both produce broadening of diffraction peaks which can be conveniently measured as a means of quantifying the degree to which the material has been cold worked.

A number of practical difficulties are encountered in determining the diffraction peak width in conjunction with macroscopic residual stress measurement. First, the $K\alpha$ radiation generally used for residual stress measurement produces two overlapping diffraction peaks. The $K\alpha$ doublet (consisting of peaks produced by the $K\alpha_1$ and $K\alpha_2$ radiations) can be separated to determine the width of the stronger $K\alpha_1$ peak using the Rachinger correction.(1) However, this method requires collection of a large number of data points to define the entire diffraction peak profile and may not provide sufficient accuracy for residual stress measurement.(2) Second, approximation of the diffraction peak profile by a parabola fitted to the top of the peak, a common method of peak location used for stress measurement in hardened steels,(3) may result in significant error when applied to diffraction peaks of intermediate width because the calculated peak position and width are dependent upon the portion of the diffraction peak included in the regression analysis.(4) Third, the background intensity must be known in order to calculate the diffraction peak width, but it may be difficult to measure accurately for broad or overlapping diffraction peaks. Fourth, the observed diffraction peak width is actually the convolution of broadening due to the cold work present in the specimen and instrumental broadening. The two components may be separated using Stokes' method,(5) but this approach requires extensive data collection to completely define the diffraction peak profile and a reference specimen which is free of cold work.

This paper describes a simple, accurate x-ray diffraction technique for determining the degree of cold work in nickel base alloys from the measured diffraction peak width. The (420) diffraction peak width is demonstrated for nickel base alloys to be both independent of the mode of plastic deformation and additive. An empirical relationship is described which

Residual Stress in Design, Process and Materials Selection,
ed. W. B. Young, ASM, Metals Park, OH, (1987), pp. 11-19.

allows the degree of cold work to be determined from the diffraction peak width measured in conjunction with the macroscopic residual stress. The method is rapid, requires minimum data collection, and is suitable for the routine analysis of surfaces deformed by machining, grinding, or shot peening where separation of the contributions to the diffraction peak width caused by crystallite size and microstrain[6] is not required.

PEARSON VII FUNCTION PEAK PROFILE FITTING

Most of the difficulties encountered in determining both the diffraction peak position and width can be overcome if the measured diffraction peak profile can be accurately described by a suitable function fitted by regression analysis. Pearson VII functions, which are bell-shaped curves ranging from Cauchy (Lorentzian) to Gaussian distributions, have been shown[2],[8] to accurately describe the profiles of diffraction peaks in the back-reflection region used for residual stress measurement.

The diffracted intensity at any angle, 2θ, can be approximated by a superposition of the $K\alpha_1$ and $K\alpha_2$ diffracted intensities and a linearly sloping background:

$$I(2\theta) = f(2\theta) + \alpha f(2\theta - \delta) + C \bullet 2\theta + D \quad (1)$$

where:

$$f(2\theta) = A\left(1 + \frac{B^2}{M}(2\theta - 2\theta_o)^2\right)^{-M} \quad (2)$$

is an optimized Pearson VII function describing the $K\alpha_1$ diffraction peak profile. Eq. (1) can be fitted to the diffracted intensity measured at a relatively small number of Bragg angles, 2θ, by non-linear least squares regression. The fit is optimized by letting M be a real number ranging from M = 1 for a Cauchy to M = ∞ for a Gaussian profile.

The regression parameters are as follows:

A	maximum net intensity of the $K\alpha_1$ peak
B	a peak width parameter
M	a decay rate parameter
$2\theta_o$	the $K\alpha_1$ peak position
C	linear background slope
D	linear background intercept.

The constant α is the ratio of the $K\alpha_2$ to $K\alpha_1$ peak intensities, typically fixed at 0.5, and δ gives the separation of the $K\alpha$ doublet which is dependent upon $2\theta_o$ and the x-ray wavelength.

Examples of Eq. (1) fitted to (420) diffraction peak profiles produced with copper $K\alpha$ radiation are shown in Figures 1 and 2, where the measured diffracted intensity is plotted as a function of the diffraction angle. The linear background, $K\alpha_1$ and $K\alpha_2$ components, which are combined to model the $K\alpha$ doublet peak profile, are shown separately. Figure 1 shows the relatively narrow diffraction peak obtained in the ψ = 45 deg. orientation on an electropolished Inconel 718 surface well beneath the deformed surface layers. Figure 2 shows the broad diffraction peak produced by the cold worked surface of a shot peened Inconel 718 sample in the ψ = 0 orientation.

In Figure 2, the (331) diffraction peak centered at a lower diffraction angle to the left of the figure produces overlap with the low angle side of the (420) peak. By eliminating the first six data points on the low angle side of the data set from the regression analysis, the effect of overlap of the adjacent diffraction peak has been effectively eliminated.

The Pearson VII function profile was fitted by linearization and successive approximation in seven iterations to the 26 and 28 data points included in the regression analysis for the diffraction peaks shown in Figures 1 and 2, respectively. The root-mean-square errors for the fitted profiles were 0.8 and 0.6 percent, respectively, which is on the order of the uncertainty in the measured intensity.

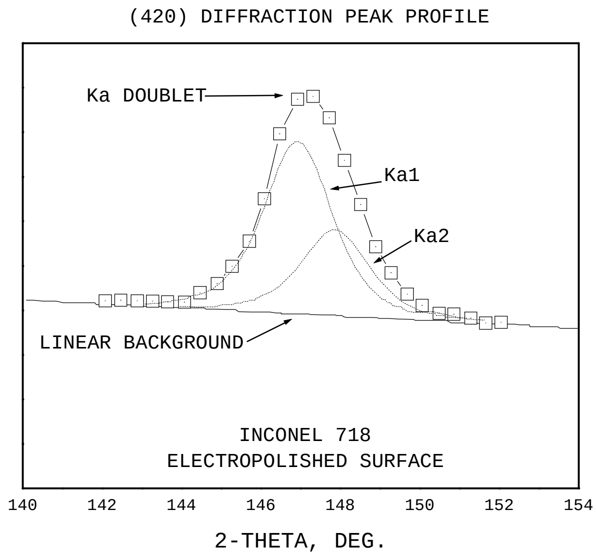

Fig. 1 - Pearson VII function profile fitted to the (420) peak from electropolished Inconel 718.

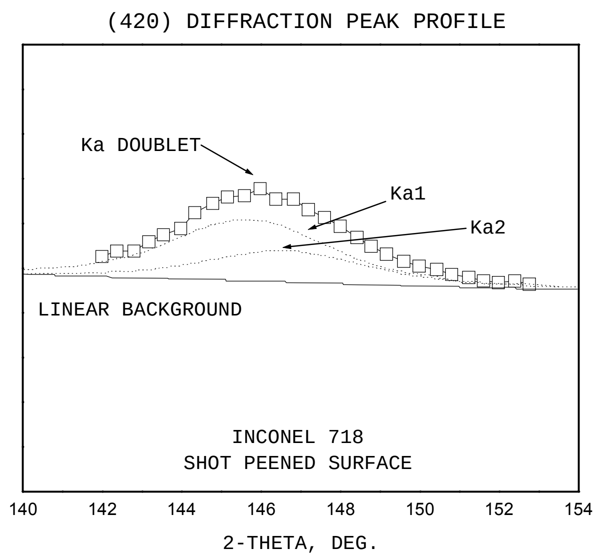

Fig. 2 - Pearson VII function profiled fitted to the (420) peak from the surface of shot peened Inconel 718.

Pearson VII function regression analysis offers a number of advantages over other methods of diffraction peak location. Because the diffraction peak can be accurately described with as few as seven data points, only the data used for routine macroscopic residual stress measurement is needed for simultaneous determination of the peak width. The absolute background intensity needs not be determined independently, allowing measurements to be made with the broad diffraction peaks commonly encountered on deformed metal surfaces, even when there may be partial overlap from adjacent peaks. Corrections for a linear background intensity and the superimposed $K\alpha$ doublet are achieved directly by the regression analysis. Finally, the $K\alpha_1$ diffraction peak position, width, and intensity can all be immediately determined algebraically from the fitted function.

Diffraction Peak Width

The diffraction peak width is usually taken to be either the width of the peak at some fraction of its height above background, or the integral breadth (the integrated intensity divided by the peak height). For most purposes, the choice is arbitrary. For the $K\alpha_1$ diffraction peak described by an optimized Pearson VII function, the width at half height (FWHM) is given by:

$$W_{\frac{1}{2}} = \frac{2}{B}\sqrt{M\left(2^{\frac{1}{M}} - 1\right)} \tag{3}$$

and the integral breadth is given by:

$$W_{\text{int}} = \frac{\sqrt{M\pi}}{B} \; \frac{\Gamma\left(M - \frac{1}{2}\right)}{\Gamma(M)} \tag{4}$$

where $\Gamma(x)$ is the Gamma function. For simplicity of calculation, the half width given by Eq. (3) was used in this investigation.

Separation of Instrumental Broadening

It is generally desirable to separate the contribution to the peak width due to instrumental effects, such as focal spot size, incident beam divergence, slit width, defocusing, etc. from the contribution caused by cold working of the specimen. Several approaches were considered. For purely Cauchy (M = 1), or perfectly Gaussian (M = ∞) peak profiles, the total peak breadth measured, B, can be shown to be a simple function of the instrumental broadening, b, and the specimen dependent broadening, β:

$$\text{Cauchy:} \quad B = b + \beta \tag{5}$$

Gaussian: $B^2 = b^2 + \beta^2$ (6)

Although the diffraction peaks encountered in residual stress measurement tend toward pure Cauchy, M may commonly vary from approximately 1 to 3. The applicability of Eq. (5) and Eq. (6) was tested by assuming a simple Cauchy or Gaussian peak shape, and calculating the specimen dependent broadening using different x-ray optics. The results demonstrated that the instrumental and specimen dependent distributions are not adequately approximated by either purely Cauchy or Gaussian profiles, and that Eq. (5) and Eq. (6) are not generally adequate for the separation of instrumental broadening.

If the variance of the distribution function is used as a measure of diffraction peak breadth, the instrumental and specimen contributions to peak broadening can be easily be separated.[(7)] However, the variance is not defined for a Pearson VII function with M < 1.5 due to the extended tails of the diffraction peak. Because many of the peak profiles encountered in stress measurement of deformed metals are best described by M approximately equal to 1, the variance cannot be used to quantify the peak width.

Stokes method of deconvolution[(5)] to separate instrumental and specimen dependent broadening was not attempted because of the extensive data collection necessary, the dependence of the method on accurate definition of the tails of the diffraction peak, and the need for a cold work-free reference specimen.

Because no simple, accurate method of separating the instrumental and specimen contributions to peak breadth appeared feasible, a technique was developed using fixed x-ray optics to hold the instrumental contribution constant, and separation was not attempted.

EMPIRICAL RELATIONSHIP BETWEEN DIFFRACTION PEAK WIDTH AND PERCENT COLD WORK

To develop an empirical relationship between the amount of cold work present and the diffraction peak width, a series of coupons were prepared for each alloy by first heat treating or annealing, as appropriate for the alloy of interest, and then deforming the samples known amounts. The percent cold work was taken to be the absolute value of the true plastic strain calculated from changes in the dimensions of the specimens. For Inconel 718 and Rene 95, specimens were prepared by solution treating and aging, followed by deformation in compression or tension. For Inconel 600 and Inconel 690, specimens were annealed and deformed in tension only.

For Inconel 718 and Rene 95, it was observed that the diffraction peak width produced by a given amount of cold work was independent of the mode of deformation. Whether deformed in tension or compression, the data fell in the same curve, as shown in Figure 3. The points plotted are the mean values of from four to ten repeat measurements on each sample. The standard deviation about the mean peak width ranged from approximately 0.02 deg. to 0.2 deg., increasing with peak width. Further, the peak width was accumulative for additional plastic strain induced on an already deformed specimen.

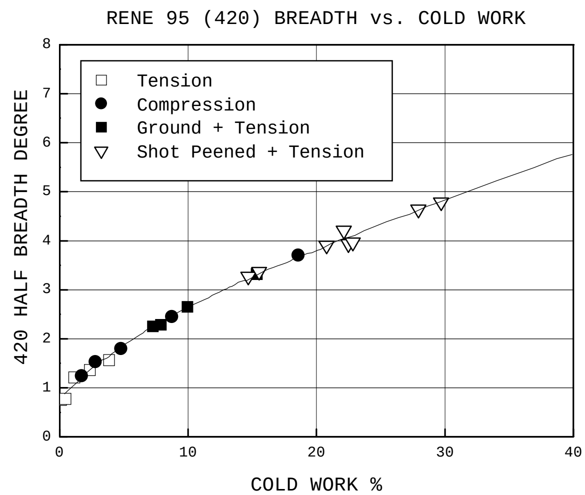

Fig. 3 - Empirical curve relating the (420) diffraction peak width to percent cold work for Rene 95 samples deformed by various means.

The empirical curve relating peak width to cold work could, therefore, be extended to levels of cold work beyond the range which could be produced directly in tension or compression. Specimens were prepared by grinding or shot peening the surface to induce a degree of cold work at the high end of the range previously covered by tension and compression samples. The initial surface percent cold work was first calculated from the empirical curve derived for the tension and compression samples. The ground or shot peened specimens were then deformed further in tension to extend the empirical curves for Inconel 718 and Rene 95 beyond 30 percent cold work.

For all four alloys investigated, it was observed that the peak width increased monotonically, but not linearly, with the percent cold work induced. The peak width initially increased rapidly with cold work, and then approached a linear dependence for a highly cold worked state. The dependence of peak width on the percent cold work can be described for the range of cold work investigated by a function consisting of exponential and linear terms:

$$W_{\frac{1}{2}} = A\left[1 - \exp\left(- B \bullet \in_p\right)\right] + C \bullet \in_p + D \tag{7}$$

where the cold work is expressed as the absolute value of the percent true plastic strain. Eq. (7) was fitted by non-linear least squares regression to determine the regression parameters A through D. Although the function describes the dependence of peak width on cold work accurately throughout the entire range of cold work investigated, the function is transcendental. Reverse solution to determine the degree of cold work from measured peak width was achieved using Newton's method. The empirical functions fitted to data sets for all four alloys investigated are shown on a common scale in Figure 4. The data points are eliminated for clarity.

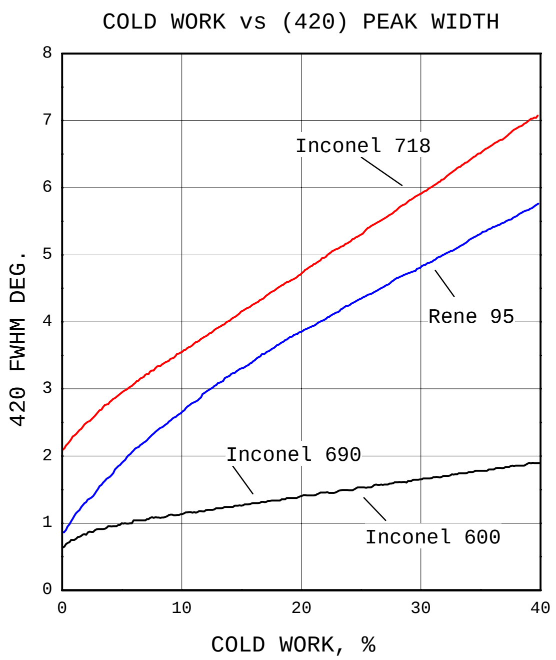

Fig. 4 - Empirical curves relating the (420) diffraction peak width to percent work for four nickel base alloys.

APPLICATIONS

The empirical relationship given by Eq. (7) has been applied in numerous investigations to determine simultaneously the cold work and macroscopic residual stress distributions as functions of depth induced in nickel base alloys by a variety of surface treatments.

Measurements were made on focusing horizontal diffractometers using copper Kα radiation and Si(Li) solid state detectors. The x-ray optics (focal spot size, take-off angle incident beam divergence, receiving slit, etc.) were the same as were used for the development of the empirical curve relating cold work to peak width. Material was removed for subsurface measurement by electropolishing. The residual stress results were corrected for both the penetration of the radiation into the subsurface stress gradient[9] and for stress relaxation caused by layer removal.[10] The x-ray elastic constants in the (420) direction were determined empirically.[11]

Inconel 718

Figure 5 shows the residual stress and cold work distributions near the surface of Inconel 718 produced by an abrasive cut-off saw. A layer of compression reaching a maximum of approximately -600 MPa exists to a depth of approximately 50 microns, followed by a deep tensile layer peaking in excess of approximately 500 MPa, and extending to at least 300 microns. The associated cold work distribution ranges from 12 percent at the surface to approximately 2 percent at a depth of only 20 microns. Some cold work is evident to a depth of approximately 200 microns

Figures 6 and 7 show the residual stress and cold work distributions produced in Inconel 718 by moderate and heavy shot peening intensities. Both peening intensities produced approximately 20 percent cold work at the surface. The cold work distributions differ primarily in the depth of the cold worked layer, which extends to approximately 70 microns for the 6 to 8 A Almen intensity, and to approximately 150 microns for the 5 to 7 C intensity. In both cases, the cold work distribution diminishes almost linearly near the surface of the sample and extends to a depth approximately equal to the depth of the uniform compressive layer. The cold work appears to be insignificant beyond approximately the maximum depth of uniform high compression.

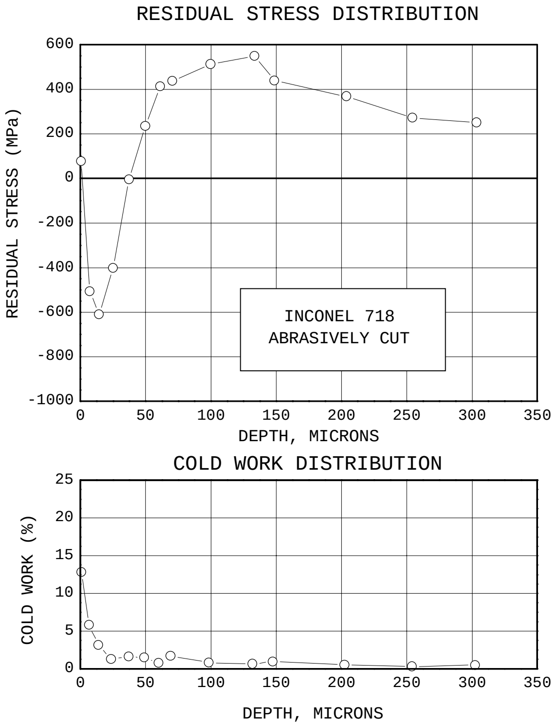

Fig. 5 - Residual stress and cold work distributions produced in Inconel 718 by an abrasive cut-off saw.

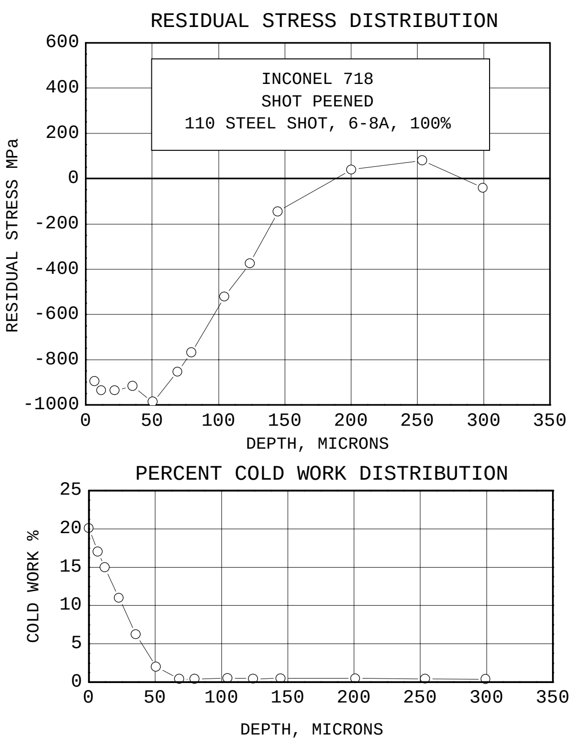

Fig. 6 - Residual stress and cold work distributions produced by moderate (6-8 A, 100%) shot peening of Inconel 718.

Residual stress distributions produced by shot peening of nickel base alloys typically exhibit the "hook" seen in Figures 6 and 7, with lower magnitude compression at the immediate surface after moderate to heavy shot peening. The reduced compressive stress achieved at the surface may be related to the extensive cold working of the material and a resulting increase in yield strength at the surface. The surface may be cold worked by prior machining or by the shot peening operation itself.

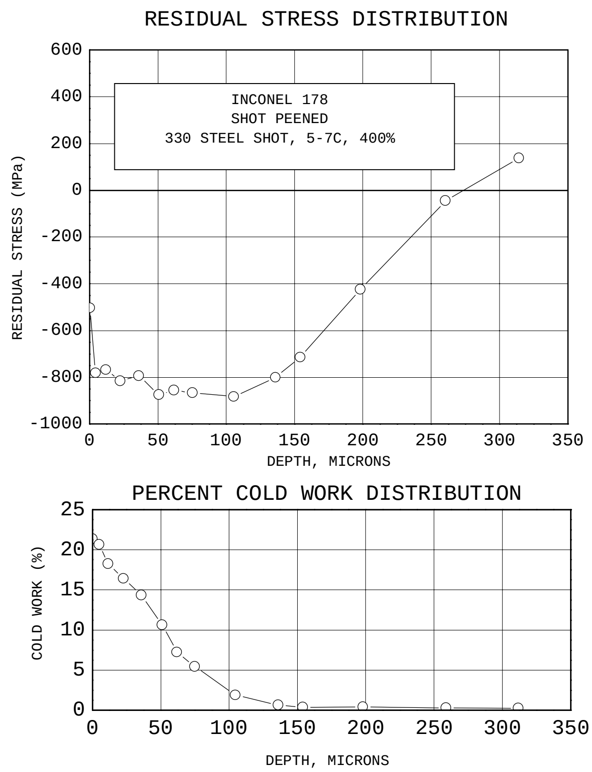

Fig. 7 - Residual stress and cold work distributions produced by high intensity (5-7 C, 400%) shot peening of Inconel 718.

Inconel 600

Figure 8 shows the subsurface residual stress distributions in both the longitudinal and circumferential directions produced by grinding the surface of Inconel 600 tubing. The residual stress distributions are of similar form, with compression in both directions near the surface and substantial tension beneath. The circumferential stress distribution is shifted toward tension, and both distributions show substantial tensile stress beneath a depth of approximately 100 microns. The associated cold work distribution reveals a highly cold worked surface, estimated in excess of 40 percent, requiring extrapolation of the empirical relationship beyond the range which could be produced in test coupons. The material is seen to be significantly deformed to a depth of 100 microns, approximately the extent of the compressive layer.

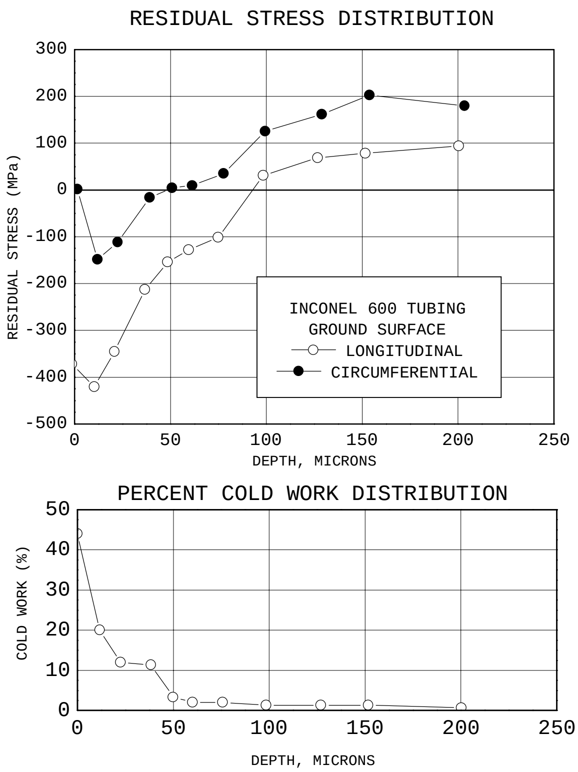

Fig. 8 - Residual stress and cold work distributions produced by grinding the O.D. surface of Inconel 600 tubing.

Cold working of nickel base alloys to the extent observed in Figures 5 through 8 can result in significant work hardening and alteration of mechanical properties, such as yield strength, in the near surface layers.

From a true stress-strain curve for the alloy, it is possible to estimate the yield strength distribution from the cold work distribution. Figure 9 shows a true stress-strain curve measured at room temperature for Inconel 600 tubing in the condition supplied by the mill. Because the percent cold work was taken to be the true plastic strain, the resulting yield strength at each depth can be estimated from Figure 9. Figure 10 shows the estimated subsurface yield strength distribution produced by grinding the surface of the tubing. At the surface, the yield strength is estimated to be approximately twice the value for the core material. The distribution drops at first nearly linearly to a depth of approximately 50 microns and then more gradually to reach the yield strength of the undeformed material at a depth of approximately 150 microns.

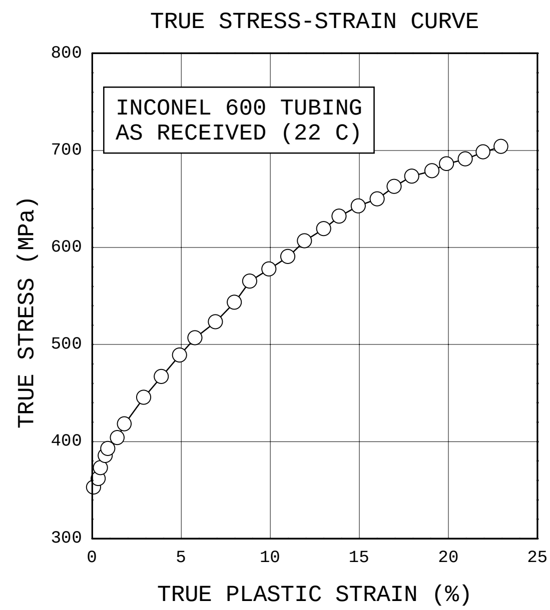

Fig. 9 - Monotonic true stress-strain curve for Inconel 600 tubing in the mill annealed condition at room temperature.

Figures 8 and 10 show that the ground tubing consists of a thin shell of compressively stressed, highly cold worked material on the O. D. surface with a yield strength substantially higher than the core, which is in residual tension. This inhomogeneity induced in the tubing by grinding the O. D. surface can have a significant effect upon the residual stress distributions developed in subsequent forming operations, such as expansion and bending of the tubing during fabrication.

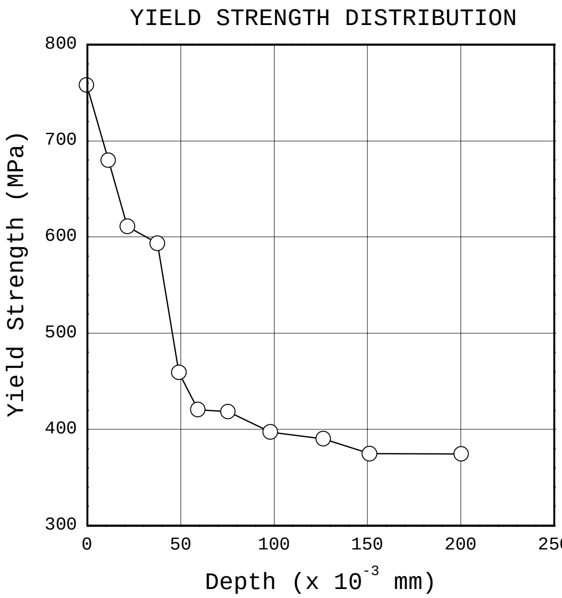

Fig. 10 - Estimated yield strength distribution for the O.D. surface of ground Inconel 600 tubing.

SUMMARY AND CONCLUSIONS

A rapid, accurate method of determining the diffraction peak width simultaneously with the peak position during residual stress measurement has been developed using Pearson VII function peak profile analysis. The method provides a number of advantages over methods previously used:

1. Separation of the Kα doublet and background subtraction are provided directly by the regression model.

2. Independent measurement of the background intensity is not necessary, allowing the use of broadened or partially overlapping diffraction peaks.

3. Only the data routinely collected for the determination of diffraction peak position during the measurement of macroscopic residual stresses are required.

An empirical relationship has been developed relating the (420) diffraction peak width to the percent cold work present in the nickel base alloys, Inconel 718, Rene 95, Inconel 600 and Inconel 690. For all four materials, the diffraction peak width was found to approach a linear dependence upon the amount of cold work for highly cold worked material. The peak width produced for a given amount of cold work was found to be independent of the manner in which the cold work was induced, whether by sample uniaxial tension or compression, or as a result of the complex deformation produced during shot peening or grinding.

The method has been used to determine simultaneously the residual stress and cold work distributions produced on abrasively cut and shot peened surfaces of Inconel 718, and on the ground surface of Inconel 600 tubing. A method of estimating the subsurface yield strength distributions from a true stress-strain curve and the measured cold work distributions has been demonstrated for ground Inconel 600 tubing.

ACKNOWLEDGEMENT

The author wishes to acknowledge the assistance of the Metal Improvement Company in providing the shot peened and abrasively cut Inconel 718 specimens and the Babcock & Wilcox Corporation for providing the true stress-strain data for Inconel 600 tubing shown in Figure 9.

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