Diffraction Notes

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X-RAY DIFFRACTION DETERMINATION OF SUBSURFACE COLD WORK DISTRIBUTIONS

Cold working of metals produces extensive dislocation networks within individual grains. The dislocation networks define "crystallites" which are nearly perfectly crystalline subgrains. As the percent cold work increases, the average size of the crystallites is reduced and the average (root mean square) microstrain within the crystallites increases. Both the reduced crystallite size and increased microstrain result in broadening of x-ray diffraction peaks.

Recent developments in Pearson VII function peak profile analysis have allowed the K-alpha diffraction peak width to be separated from the K-alpha doublet with high accuracy. Empirical relationships can be established between the K-alpha 1 diffraction peak width and known amounts of cold work. An empirical curve developed for the nickel base alloy, Rene 95, using specimens deformed in uniaxial tension and compression, and after first grinding or shot peening followed by tensile deformation, is shown in Figure 1. These data and similar results obtained for Inconel 718 demonstrate that the degree of broadening of the (420) diffraction peak for these nickel base alloys is independent of the mode of deformation, and is additive. The percent cold work is taken to be the absolute value of the true plastic strain.

The degree to which the material is cold worked is independent of the macroscopic residual stress. For example, a material may be highly cold worked by uniaxial tension or hydrostatic compression without inducing any macroscopic residual stresses. Many surface treatments such as machining, grinding, or shot peening develop macroscopic residual stresses as a result of nonuniform cold working of the surface layers. Using Pearson VII function peak profile analysis, the macroscopic residual stress and percent cold work distributions can be determined simultaneously as functions of depth.

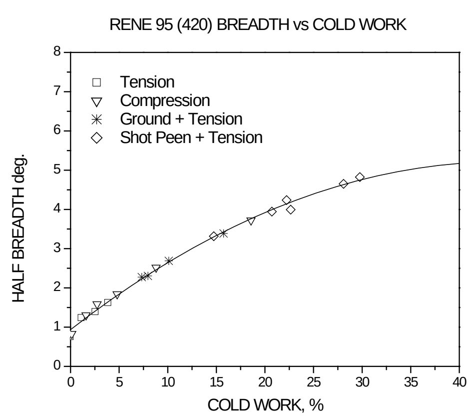

Figure 1

Figure 2 shows examples of macroscopic residual stress and cold work distributions produced near the surface of Inconel 718 samples by abrasive cutting and by moderate (6-8A) and heavy (5-7C) shot peening. The additional information provided by the cold work distribution can be a powerful tool in studying residual stress distributions. A process such as wire brushing may produce compressive surface stresses comparable to shot peening, but with much less cold work. Surface annealing during plasma spray coating or laser treatments can be detected as a reduction in peak width.

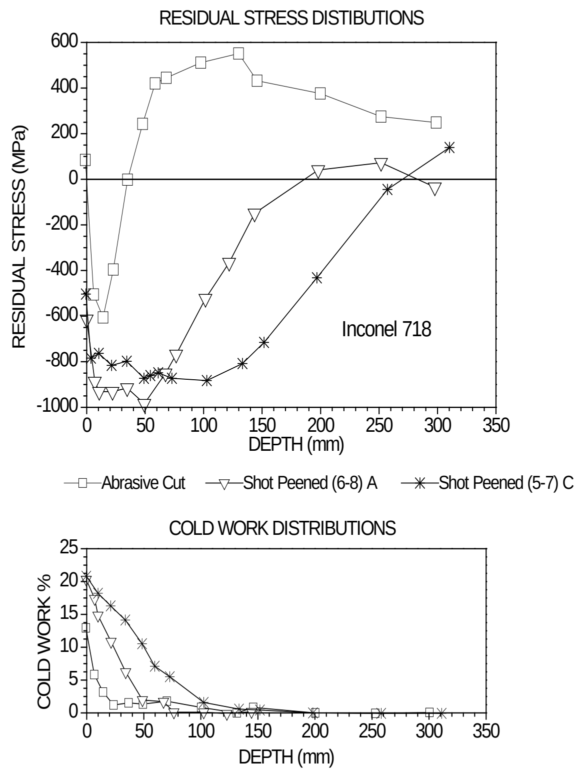

Figure 2

The yield strength of the cold worked surface layers may be altered as a result of the plastic strain induced. The yield strength at the surface of Inconel 600 tubing can be doubled by grinding.[1] The surface layer containing residual stress, cold work, and yield strength gradients may have a pronounced effect on residual stress distributions developed if the material is further plastically deformed, as in the bending of tubing or momentary overloads of shot peened components.

Empirical relationships similar to Figure 2 have been developed for several nickel base alloys to date. A more detailed discussion of the subject of simultaneous determination residual stress and cold work distributions will be published shortly in the Proceedings of the ASM Conference, “Residual Stress – In Design, Process, and Material Selection,” held in Cincinnati, Ohio, April, 1987.

REFERENCE:

1. P.S.Prevey, “Surface Residual Stress Distributions in As-Bent Inconel 600 U-Bend and Incoloy 800 90-Degree Bend Tubing Samples,” EPRI Workshop Proceedings, 1980.

QUALITY ASSURANCE PROGRAM

Lambda Research has implemented a comprehensive laboratory quality control and assurance program conforming to ACIL, AALA (G4062386), ASTM (E548-84), ANSO (Z34.2-1987) and military (MIL-Q-9858A) specifications. While we have consistently improved and monitored all laboratory functions, we now have in place a formal QA/QC manual clearly defining all testing, sample preparation, calibration and maintenance procedures.

Calibration and maintenance schedules have been established for all laboratory equipment. Calibration and instrument alignment checks and versions of both data collection and reduction software are documented to be traceable to individual projects. All records are kept permanently in a fireproof vault.

EXPANSION AND GROWTH

This year marks the tenth anniversary of Lambda Research providing x-ray diffraction laboratory services. To date, we have reported over 1,700 projects, including over 32,000 residual stress measurements, 1,500 retained austenite determinations, 400 qualitative, and 1,300 quantitative phase analyses as well as many other services. In the startup years through 1980, Lambda had performed nearly 1,600 residual stress measurements each year, and from 1981 through 1986, performed nearly 5,000 stress measurements annually. From a one-room laboratory with one diffractometers, Lambda has grown into a beautifully refurbished three-story building of over 6,000 square feet of laboratory and office space with a full time staff of eight employees. The laboratory is equipped with 4 computer-controlled diffractometers instrumented with solid-state detectors or monochromators, and position sensitive detector apparatus for stress measurement in massive steel components. Strain gaging, machining, electro-polishing and heat treatment facilities are available for complete support of residual stress studies.

IMPROVEMENTS IN QUALITATIVE PHASE ANALYSIS

In the past several months, Lambda Research has developed enhanced software for the presentation and reduction of qualitative phase analysis data. The new software packages provide complete diffraction patterns presented in an 8 ½ x 11 format on a dot matrix printer. The data reduction software uses first and second derivative peak searching algorithms after background subtraction and Golay smoothing. The patterns are reduced to tables of peak positions. Lattice spacings, net intensities and their uncertainty, and diffraction peak widths, corrected for systematic error using the NBS silicon standard. Comprehensive qualitative analysis includes identification of the components present in the sample by matching patterns in the JCPDS powder diffraction file. For repetitive analysis, at considerable cost savings, the reduced data and computer generated diffraction patterns can be provided for the client’s own analysis.

X-RAY FLUORESCENCE ANALYSIS

Energy dispersive x-ray fluorescence apparatus has recently been placed in service. The equipment, built at Lambda Research, uses a Si(Li) solid-state detector system and vacuum chamber to allow detection of elements ranging from sodium to uranium. The device uses direct x-ray excitation to minimize background intensity. Regenerative filters can be used to enhance the detection limits for specific elements. The instrument is currently used for qualitative elemental analysis in support of x-ray diffraction phase analysis. Quantitative analysis software, including correction for primary and secondary absorption effects and instrumental losses, is under development.