Diffraction Notes

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PROBLEMS WITH NON-DESTRUCTIVE SURFACE X-RAY DIFFRACTION RESIDUAL STRESS MEASUREMENT

This article concerning errors in the measurement of the surface stress caused by the penetration of the x-ray beam is the third and final article in a series addressing the difficulties encountered in the measurement and interpretation of surface residual stress results obtained by x-ray diffraction.

The first article addressed the lack of correlation between surface residual stresses and the processes which produced them, and the second addressed the problem of stress variation in the plane of the sample surface. A copy of the entire series, to be published as a technical paper through the ASM, can be obtained by contacting Lambda Research.

Part 3:
Errors Due to Subsurface Residual Stress Gradients

For most materials and x-ray diffraction residual stress measurement techniques of practical interest, the effective depth of penetration of the x-ray beam is quite shallow. Nominally 50% of the diffracted radiation originates from a depth of less than 10 µm (0.0004 in.). However, the x-ray beam is attenuated exponentially as a function of depth. The rate of attenuation is governed by the linear absorption coefficient which depends upon the composition and density of the specimen and the radiation used.

Any "surface" measurement is, therefore, actually an exponentially weighted average of the stress at the surface and in the layers immediately beneath it. In developing the relationship between the observed strain in the crystal lattice and the stress at the sample surface, the assumption was made that the residual stress is constant throughout the depth of penetration of the x-ray beam. Unfortunately, for many samples of practical interest, the stress varies rapidly with depth beneath the surface, and the assumption of constant stress is violated. The result can be errors as large as 600 MPa (88 ksi).

The sign and magnitude of the potential error is dependent upon the subsurface stress gradient; i.e., the direction and rate of change of stress with depth into the sample surface. Because the depth of penetration of the x-ray beam also varies with the angles theta and psi, the apparent surface residual stress will depend upon the details of the technique chosen, specifically the radiation and angles selected, if a significant subsurface stress gradient exists.

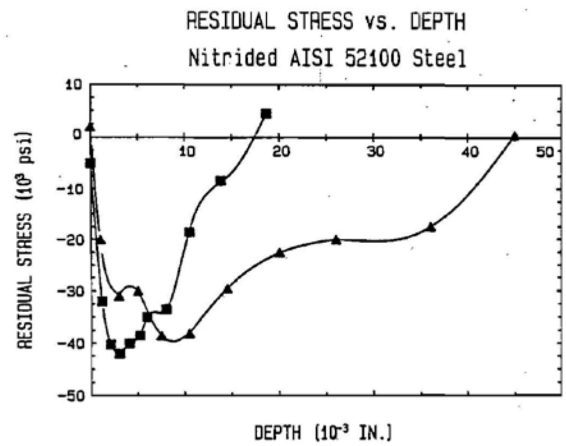

Fig. 1 Subsurface Stress Distributions Produced by Nitriding AISI 52100 Steel, Showing Pronounced Near-Surface Stress Gradient[2]

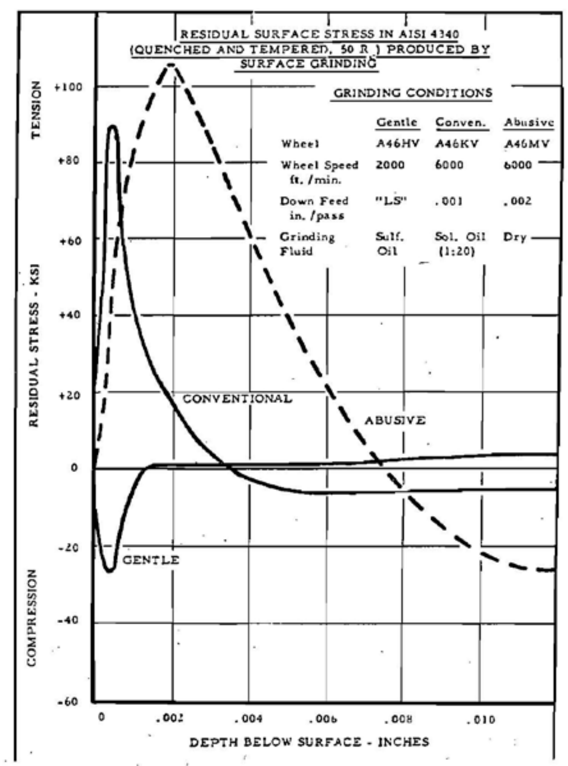

Fig. 2 Subsurface Stress Distributions Produced by Diverse Grinding Conditions in 4340 Steel[3]

Large stress gradients at the sample surface are common. Figure 1shows examples of large subsurface stress gradients produced by two different methods of nitriding 52100 steel. The grinding stress distributions shown in Figure 2 have large stress gradients at the surface, both positive and negative. Figure 3 shows a pronounced gradient in the "hook" commonly seen at the surface of shot peening stress distributions. Figure 4 depicts a complete reversal of the stress distribution within 50 microns of the surface observed on abrasively cut Inconel 718.

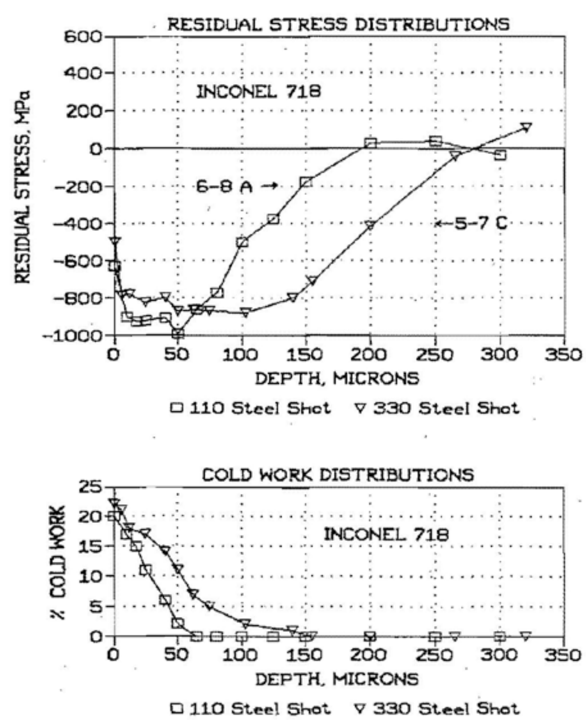

Fig. 3 Variation in Depth of the Stress Distributions Produced in Shot Peened Inconel 718, Showing Similar Surface Results(4)

It is possible to correct for the errors caused by the penetration of the x-ray beam into the stress gradient, provided subsurface measurements are made by electropolishing to remove layers with sufficient depth resolution to accurately establish the stress gradient. Koistinen and Marburger[1] developed a method of calculating the true residual stress by unfolding the exponential weighting caused by the penetration of the x-ray beam. Their often cited example of agreement between x-ray diffraction and mechanical methods of residual stress measurement in ground steel, reproduced in Figure 5, shows agreement only because the correction was applied, a fact commonly omitted when their work is referenced. The figure is reproduced exactly as it appears in their original publication.

Figures 6 and 7 show the effect of correction for positive and negative stress gradients, respectively. As seen in Figure 6, the uncorrected surface stress may even be of the wrong sign.

Non-destructive surface residual stress measurements cannot be corrected for errors caused by penetration of the x-ray beam into a varying stress field. Therefore, surface results must be interpreted with caution. The true surface stress frequently cannot be accurately determined by surface measurement alone.

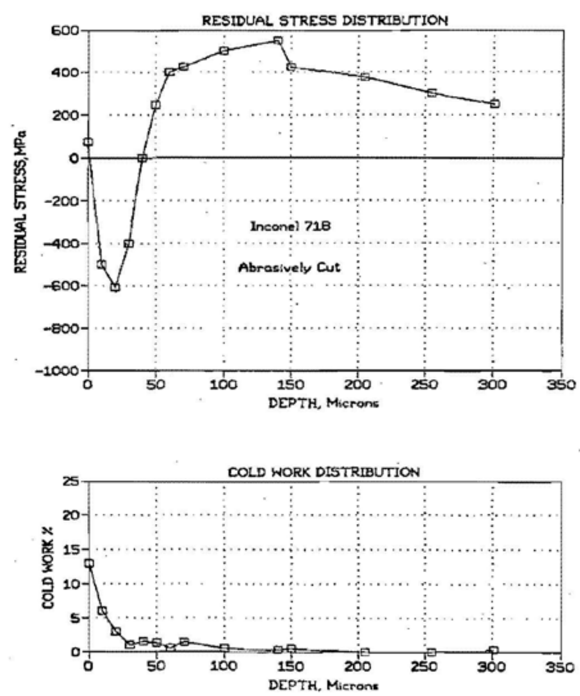

Fig. 4 Subsurface Stress Distributions in Abrasively Cut Inconel 718, Showing Complete Stress Reversal Near the Surface(4)

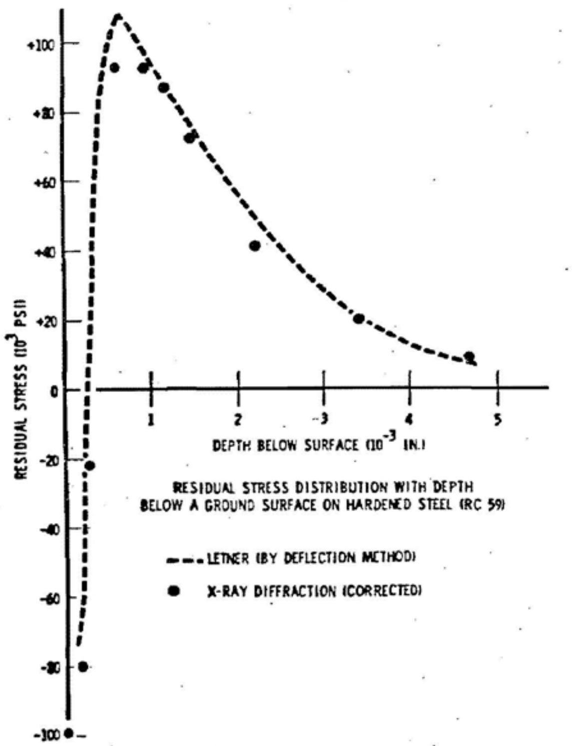

Fig. 5 Subsurface Stress Distribution in Ground Steel Measured by Mechanical and X-ray Diffraction Methods with Correction for the Near-Surface Stress Gradient[1]

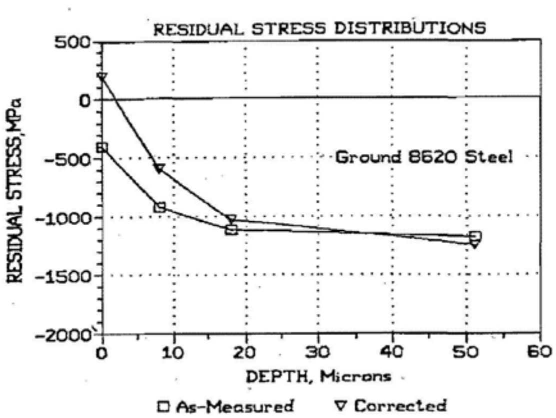

Fig. 6 Effect of Correction for Penetration of the Radiation into a Subsurface Stress Gradient, Showing a Change of Sign at the Surface

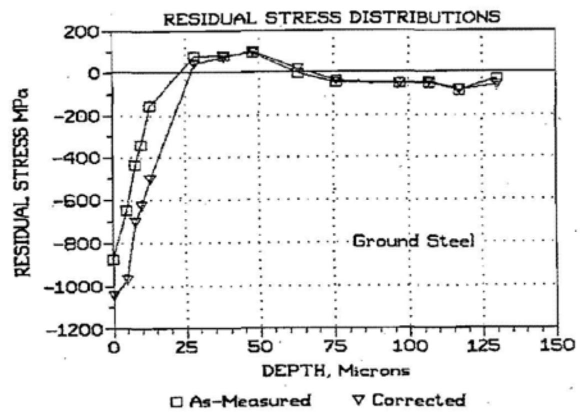

Fig. 7 Effect of Correction for Penetration of the Radiation into a Stress Gradient in Ground Steel

CONCLUSIONS

The limitations inherent in the use of surface x-ray diffraction residual stress measurements have been shown to result in three areas of concern which must be considered before non-destructive surface results may be used reliably.

First, there frequently is no correlation between the surface residual stress and the method of processing which produced the stress distribution. Subsurface stresses often differ significantly from the surface value.

Second, the surface stresses produced by many material removal processes, particularly machining and grinding, will often vary significantly over short distances. The surface stress measured is then dependent upon the details of the measurement technique, such as the irradiated area size and psi tilt used.

Third, many processes of practical interest produce a rapid change in residual stress immediately beneath the surface, within the depth of penetration of the x-ray beam. This results in errors which can approach 600 MPa (88 ksi) and even alter the sign of the apparent results. The effects of penetration of the x-ray beam can only be corrected if subsurface results are obtained.

REFERENCES:

1. KOISTINEN & MARBURGER, Trans. ASM, Vol. 51, p. 537, 1959.
2. KOISTINEN & MARBURGER, Trans. ASM, Vol. 67, 1964. .
3. KOSTER, w.P. et al~ "Surface Integrity of Machined Structural Components," US Air Force Materials Laboratory Technical Report No. 70-11 p. 112, 1970.
4. PREVEY, P.S., "The Measurement of Subsurface Residual Stress and Cold Work Distributions in Nickel Base Alloys," Residual Stress in Design, Process and Materials Selection, ASM International, pp. 11-19, 1987.

GANDOLFI CAMERA CAPABILITY

Lambda Research has installed a Gandolfi Debye-Scherrer 114mm camera system. The camera allows qualitative analysis to be performed on extremely small sample sizes. Debye-Scherrer patterns can be obtained on sample volumes on the order of O.1mm^3 ideal for corrosion products, failure analysis, etc. The Gandolfi mechanism allows high quality powder patterns to be obtained on coarse-grained specimens or even single crystals with high symmetry.

LAMBDA RESEARCH TO PARTICIPATE IN ASTM F04.02 ON HYDROXYLAPATITE

Paul Prevey and Robert Rothwell of Lambda Research will be attending the meeting of ASTM Committee F04.02 on Medical and Surgical Materials and Devices to be held in San Diego, CA on November 6-8. Lambda Research will be contributing to the development of a new standard for quantitative phase analysis of hydroxylapatite under the auspices of Task Group F04.02.03.08 9n Crystallographic Characteristics of Calcium Phosphates chaired by Floyd Larson. Anyone interested in x-ray diffraction quantitative phase analysis of this material being widely applied in medical applications is urged to attend.

LABORATORY AUDIT

Lambda Research has been audited by the American Association for Laboratory Accreditation (A2LA) as required biannually to maintain our accredited status. After an exhaustive two day audit of our organization, management, laboratory procedures, calibration and maintenance records, safety system, and technician training, we are pleased to announce that there were no findings of deficiencies. Lambda Research maintains its accreditation by the A2LA in accordance with internationally recognized ISOIIEC Guide 25, your assurance of good laboratory practice, documented procedures, and traceable calibrations.