SIGNIFICANCE OF EXPERIMENTAL ERRORS IN RESIDUAL STRESS DETERMINATIONS USING Θ:Θ DIFFRACTOMETERS

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ABSTRACT

The restriction of the θ:θ diffractometers to tilt angles less than 40°, when measuring residual stress, can be overcome by rotating the specimen through an offset angle and recalibrating the zero points of the Ω and θ scales. The effectiveness of this offset is examined by measuring the residual stress in a PVD film of TiN deposited on a stainless steel substrate. An anticipated error associated with backlash in the gear as the detector, or X-ray tube, is traversed beyond the 90° vertical position, is shown to be insignificant. Temperature dependent measurements of residual stress in a cold worked specimen of Kanthal D heating alloy show that errors as great as 20% may result from the use of room temperature elastic constants, when making non-ambient residual stress measurements.

INTRODUCTION

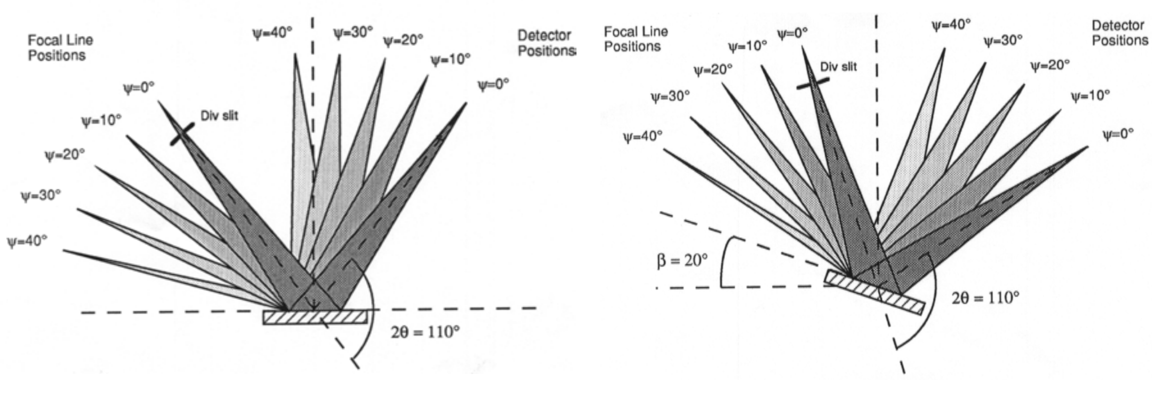
In the $\sin^2\psi$ X-ray method, residual stresses are determined by measuring the d-spacings of a selected diffraction plane when the sample surface is tilted through an angle ψ with respect to the zero angle plane of the diffractometer (1,2). Temperature dependent measurements are usually made with the θ:θ configuration, in which the sample is fixed with its surface aligned in the zero angle plane and the d-spacings of inclined planes are measured by successively displacing both the X-ray tube and the detector through an angle (ψ), as illustrated in Fig. 1A. Since diffraction measurements cannot be made when the either the X-ray tube, or the detector, approaches within ~10° of the zero angle plane, the maximum tilt angle is given by ψ_{max} = (θ - 10°) and the highest resolvable Bragg angle is thus recommended for residual stress determinations (1,2). However, the theoretical ψ_{max} of 45° for a diffraction peak at 110 °2θ can only be achieved in the θ:θ configuration if the detector (or the X-ray tube) is traversed 10° beyond the 90° vertical position, as shown in Fig. 1A. The greater theoretical ψ_{max} for higher angle peaks between 120-140 °2θ can only be achieved if the detector (or X-ray tube) is traversed even further, to 20-40° beyond the 90° vertical position.

In many θ:θ systems, the high voltage cable, water cooling tubes and counterweights restrict the traverse of the X-ray tube or a Peltier cooled detector to a maximum Ω or θ angle of ~95°, while the likely spillage of liquid nitrogen limits the traverse of a liquid nitrogen cooled detector to 72

°θ. If the available residual stress software only permits the detector (as opposed to the X-ray tube) to be progressively displaced to higher angles, as shown in Fig. 1A, the effective ψ_{max} for a Bragg peak at 120 °2θ would be only 12° when using a liquid nitrogen cooled detector, while no ψ tilts would be possible for a Bragg peak at 140 °2θ. Another concern of the θ:θ configuration, is that when either the X-ray tube, or the detector, passes through the 90° vertical position, the respective counterweight acts in the opposite direction, so that the driving gear is pressed against the other side of the teeth of the main goniometer gear. A Bragg angle error related to an unknown degree of backlash in the driving mechanism may thus occur (3) and cause plots of $\Delta d/d_n$ vs. $\sin^2\psi$ to exhibit non-linearity at higher values of ψ.

The geometrical and physical restrictions on the traverse of the X-ray tube and/or detector, when using the θ:θ configuration for determining residual stress, can be counteracted by recalibrating the zero angle positions of both the Ω and θ scales after the sample surface has been rotated through an offset angle (β) in the opposite sense (-ψ) to the effective tilt angle ψ. The geometrical conditions of the β angle offset, and the consequent displacements of the X-ray tube and detector to obtain a wider range of ψ angles, are illustrated in Fig. 1B. While the change in incident angle of the X-ray beam with increasing ψ is not affected by the β offset, the practical maximum tilt angle can be increased closer to the theoretical ψ_{max}, without traversing the detector beyond the 90° vertical position. The effectiveness of an β offset in the Ω and θ scales is evaluated by determining the residual stress in a PVD TiN coating under controlled experimental conditions.

Although considerable care is usually taken to calculate specific X-ray stress coefficients for a relevant *hkl* diffraction plane from the bulk elastic properties of the specimen (4,5), the temperature dependence of the elastic modulus is often ignored when conducting *in situ* high temperature experiments. The significance of such errors is investigated by determining the temperature dependence of residual stress in a cold worked strip of a Kanthal resistive heating alloy.



A **B**

Figure 1. Geometry of the θ:θ diffractometer method for determining residual stress.
A. Tilt angles induced by moving the X-ray tube and detector
B. Higher tilt angles obtained (without passing through the 90° vertical position) by offsetting the zero angle plane through an angle β .

EXPERIMENTAL

Residual stress experiments were performed in a Buehler HDK 2.3 high temperature furnace mounted on a Scintag XDS 2000 X-ray diffractometer, equipped with a Peltier cooled drifted Si detector. The diffractometer was set up in the θ:θ configuration with a copper target X-ray tube, which was operated at 45 kV and 40 mA. For the room temperature evaluation of the β angle offset, the specimens were composed of 10 µm thick PVD coatings of TiN deposited by the Liburdi reactive ion plating process on 1mm thick strips of 316 stainless steel (6). Specimens 1 cm wide were cut from the coated strips and mounted in the Buehler furnace with special stainless steel clips, which gave an exposed surface length of 36 mm (7).

The DMSNT Scintag software only permits residual stress measurements to be made at increasing tilt angles ψ by progressively displacing the detector to higher angles, and the X-ray tube to lower angles. As indicated in Fig. 1, the size of the divergent slit should be successively decreased with increasing ψ angle, in order to keep the irradiated length of sample constant. As the diffractometer is not equipped with a variable slit assembly, a 2 mm (1.43°) divergent slit was used for all measurements, since this gave a maximum irradiated length of 32 mm at the highest ψ angle used in the present experiments. As resolution is not a concern with stress-broadened profiles, the diffracted intensity was increased by removing the Soller slits and using a 3 mm receiving slit. Selected diffraction peaks were step-scanned over a range of at least 10 °2θ, using a step width of 0.3° and a dwell time of 20 s, to obtain a minimum of 30 data points per diffraction profile. The peak positions of diffraction profiles at selected tilt angles were determined with a profile fitting program based on a Pearson VII function, and used to calculate the interplanar spacings d_ψ of planes inclined at an angle ψ to the sample surface. Using the interplanar spacings (d_n) of planes that lie parallel to the surface as a reference, the strain ε_ψ within the inclined planes was determined in terms of $\Delta d/d_n$, where $\Delta d = (d_n - d_\psi)$. The biaxial surface residual stress σ_ϕ was determined from linear slopes fitted to plots of $\Delta d/d_n$ versus $\sin^2\psi$, in accordance with the relationship:

$$\varepsilon_\psi = \Delta d/d_n = {}^1/_2 S_2 \sigma_\phi \bullet \sin^2\psi + S_1 (\sigma_{11} + \sigma_{22}) \qquad \text{equation (1)}$$

where S_1 (= $-\nu/E$) and S_2 (= $2(1 + \nu)/E$ are the X-ray stress constants, and σ_{11} and σ_{22} are the principal stresses in the biaxial system (1,2). S_1 and S_2 for the 422 Bragg peak were obtained by the weighting analysis of Perry (4), based on the model of Reuss (5), using values of Poisson's ratio (ν) = 0.30 and Young's modulus (E) = 640 GPa for the TiN the specimen (8).

For the high temperature measurements to investigate the significance of errors resulting from the use of X-ray stress coefficients based on room temperature elastic properties, the specimen was composed of a 45 mm length of a 1 cm wide cold rolled strip of Kanthal D ferritic Fe-Cr-Al resistive heating alloy. This alloy was selected because the temperature dependence of its Young's modulus has been well characterized over the temperature range from 20-1000 °C (9) and previous experiments have shown that it can be rolled into thin strip with electrical resistance similar to the normal Pt-13%Rh heater strip (10). The Kanthal strip specimen was heated by the passage of a DC current and its temperature was measured with a Pt/Pt-10%Rh (type S) thermocouple, welded to its lower surface, and controlled ±1 °C using the installed Micristar PID controller. The surround heater was also used to improve the temperature uniformity along the heated length of the specimen. The X-ray stress constants for the Kanthal 511 diffraction plane were calculated in accordance with equation 1, using the room temperature values of 0.30 for Poisson's ratio and 220 MPa for Young's modulus, and values of Young's modulus specific to the different experimental temperatures from data supplied by the Kanthal Company (9).

RESULTS AND DISCUSSION

A specimen of the TiN coating on the 316 stainless steel substrate was used to determine the significance of any changes in d-spacings that might be attributed to controllable experimental conditions. The 422 TiN peak which occurs at 126 °2θ was selected, because it is not overlapped by any of the peaks from the diffraction pattern of the stainless steel substrate. Using no β angle offset, the profile of the 422 peak was scanned four consecutive times at tilt angles up to $\psi = 40°$, in increments of 10°, without changing any of the instrumental settings. As shown in Fig. 2, the four resulting plots of $\Delta d/d_n$ versus $\sin^2\psi$ overlap very closely and exhibit only minor deviations from strict linearity. Since, at the tilt angle of $\psi = 40°$, the 422 peak occurs at (63 + 40 = 103°) on the theta scale, the pronounced linearity of these plots confirms that the anticipated backlash errors associated with the traverse of the detector beyond the 90° vertical position are not significant in the present experimental set up. The compressive residual stress in the TiN coating determined from linear slopes fitted by computer to the individual plots was -5.81 ± 0.05 GPa, which means that the instrumental and software errors inherent in the present apparatus enable residual stresses to be determined with a reproducibility of ±0.86%. On the basis of this finding, any changes in residual stress that occur after making intentional changes in experimental conditions can only be regarded as significant if they are greater than 1.0%.

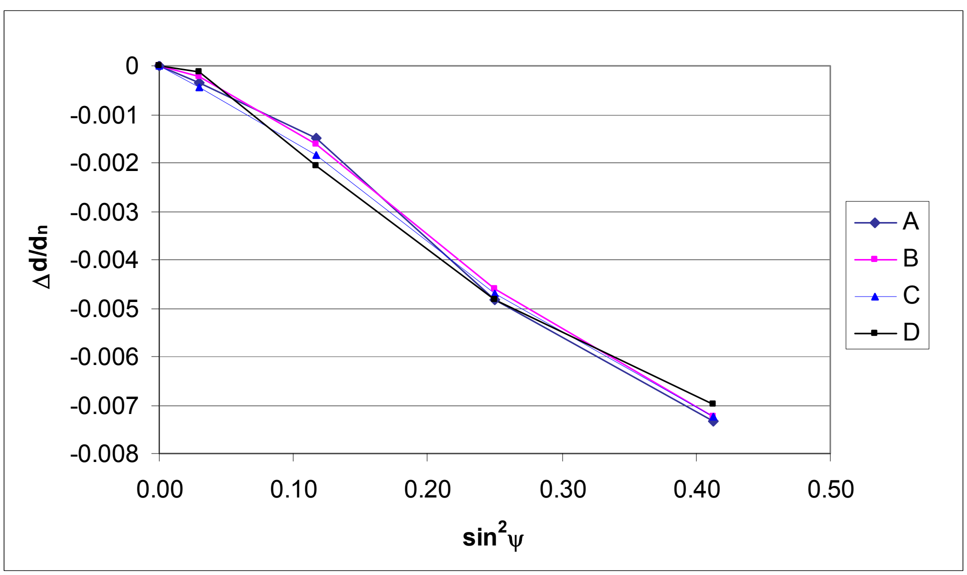


Figure 2. Reproducibility of $\Delta d/d_n$ vs. $\sin^2\psi$ results for a TiN coating on 316 stainless steel.

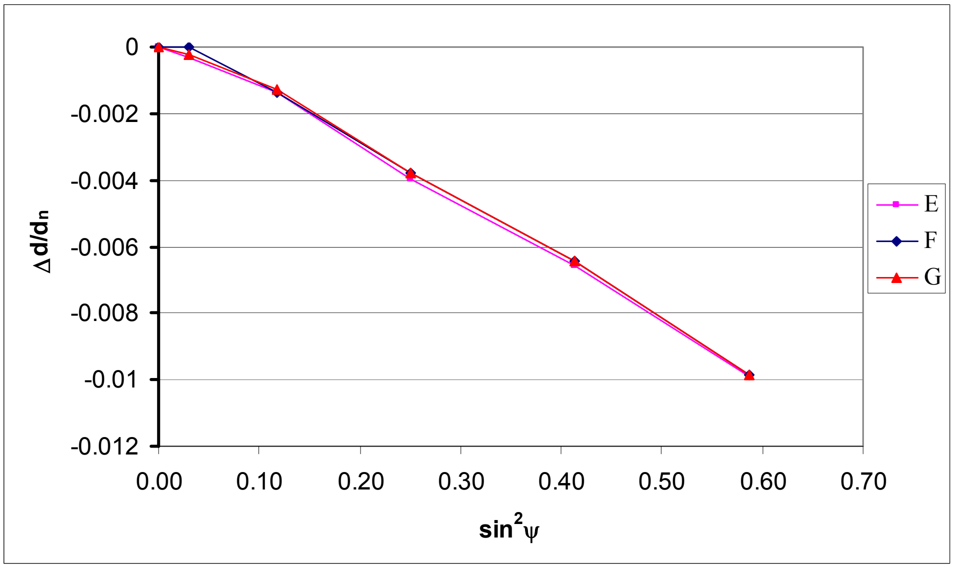


Figure 3. $\Delta d/d_n$ vs. $\sin^2\psi$ results for a TiN coating on 316 stainless steel for ψ angles up to 50° using a 20° β offset.

The effect of a β offset of 20° is illustrated by the plots of $\Delta d/d_n$ vs. $\sin^2\psi$ in Fig. 3, which refer to three successive scans at tilt angles up to 50° of the 422 peak of a TiN coating on stainless steel. In this case, the 422 peak at the highest tilt angle of ψ = 50° occurs at (63 + 50 -20 = 93°) on the theta scale, so is minimally affected by possible backlash errors. The three plots are again seen to overlap very closely, to give a residual stress of 5.93 ± 0.03 GPa, which means a reproducibility of 0.51%. However, if data points are only used up to ψ = 40°, which is the maximum tilt angle available with zero offset, the calculated residual stress is found to be reduced to 5.63 ± 0.08 GPa, i.e. by 5.5% . Since this difference in stress is about ten times greater than the demonstrated reproducibility, it is evident that the slope of the $\Delta d/d_n$ vs. $\sin^2\psi$ plot, on which the residual stress is based, is essentially governed by the high angle data points, so that the accuracy of the $\sin^2\psi$ method can be significantly improved by employing a β offset, when using a θ:θ diffractometer. A further possible contribution to the accuracy in these residual stress determinations, which is also under investigation at present in the authors' laboratory, is the size of the increment in the tilt angle ψ.

The significance of errors in the calculation of high temperature residual stress when using room temperature elastic moduli is indicated in Fig. 4, which refers to stresses calculated from shifts in the Kanthal D 511 peak, that occurs at 136 °2θ. Both sets of calculated compressive stresses are coincident at room temperature (as would be expected), but as the Young's modulus of Kanthal decreases with temperature (9) residual stresses calculated using the "at temperature" Young's modulus are significantly smaller than values calculated using the room temperature elastic constant. The maximum discrepancy of 100 MPa between the two sets of results, which occurs at 500 °C, represents an error of the order of 20%, which is clearly unacceptable. These results show that, while *in situ* high temperature determinations of residual stress based on room temperature elastic constants may be used to identify temperatures associated with anomalous

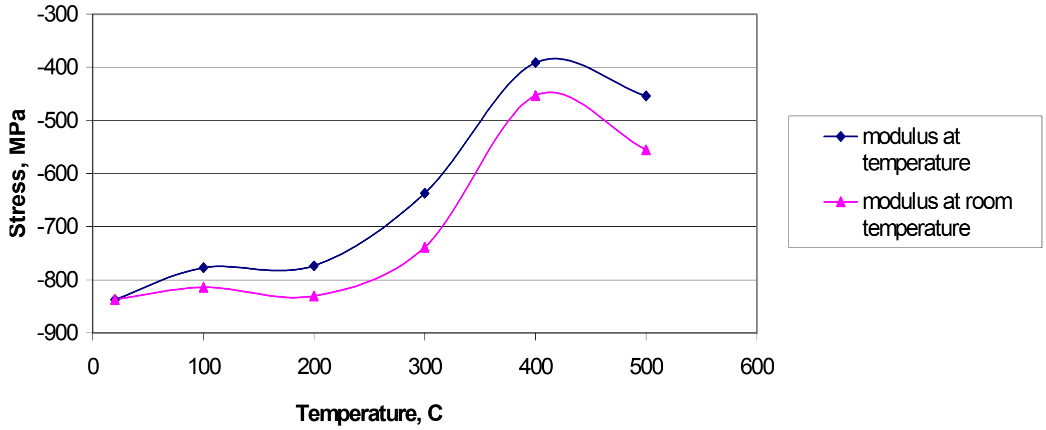


Figure 4. Effect of using room temperature and "at temperature" elastic coefficients for calculating residual stress in Kanthal D heating alloy.

changes in residual stress, due to structural or microstructural changes, etc. (as occurs in the present specimen between 400 and 500 °C) the specific values of residual stress calculated in this manner are subject to an error that increases with temperature of measurement above the ambient. From a practical point of view, this means that the temperature dependence of residual stress in a component cannot be meaningfully determined unless the temperature dependence of the Young's modulus of the material is known over the intended experimental temperature range. The measurement of the temperature dependence of the Young's modulus of TiN is thus in progress in the authors' laboratory.

ACKNOWLEDGEMENTS

This work was supported in part by an operating grant from the Natural Sciences and Engineering Research Council of Canada. The authors express their appreciation to Mr. D.R. Nagy of Liburdi Engineering, Dundas, ON, for providing the PVD TiN specimens and to Dr. E.A. Payzant for his assistance with the cold rolling of the Kanthal heater strip.

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