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RESIDUAL AND APPLIED STRESS MEASUREMENTS AT IPNS

J. W. RICHARDSON, JR.
IPNS Division
Argonne National Laboratory
Argonne, IL 60439
USA

ABSTRACT. The Intense Pulsed Neutron Source (IPNS) at Argonne National Laboratory has operated as a user facility since 1981. From the early days on, highly precise measurement of structural parameters including lattice constants has been a major objective of developments on the powder diffractometers at IPNS. The General Purpose Powder Diffractometer (GPPD) combines excellant instrumental resolution - roughly independent of d-spacing - with good neutron flux to provide an exceptional tool for the measurement of residual and applied stresses.

Introduction

IPNS is a spallation neutron source, where diffracted intensities are measured as a function of Time-of-Flight (TOF) - related to neutron wavelength - at a series of fixed diffraction angles. Whereas all TOF diffractometers share the characteristic of simultaneously collecting data over an extensive range of d-spacing, the actual range depends on the wavelength spectrum of the source and the complement of detectors in use. These factors may vary from one instrument to another, so it is important to recognize and exploit the unique characteristics of each.

As the user program at IPNS has matured, the types of residual stress experiments performed have progressed from direct collaborations with individuals (MacEwen et al. (1983,1984)) to routine interactions with external researchers (Krawitz et al. (1986,1987), Kupperman et al. (1989), Majumdar et al. (1988, 1989)). As the tremendous opportunities offered by neutron diffraction have become more apparent to the general community, we have worked to enhance our capabilities and strengthen interactions with scientists active in the field. This is most visibly illustrated by increased acquisition of beamtime by a number of industrial and governmental concerns.

Instrumentation

A detailed description of the design characteristics and performance of the two powder diffractometers at IPNS, the SEPD and GPPD, is given by Jorgensen et al. (1989). On the SEPD and GPPD scattering is approximately in the horizontal plane (±7°). Both instruments have eight banks of detectors positioned at 2θ angles ranging from ±15° to ±150°, with varying numbers of detectors in each bank. On the GPPD the banks are centered at scattering angles ±148° (31 detectors), ±90° (20 det.), ±60° (12 det.), 30° (8 det.) and -20° (8 det.). Each detector is 15" high and spans a 2θ range of 0.5°.

Beam dimensions are adjustable via in-line collimation or additional slits, with a maximum allowable size of 2" high by 1/2" wide.

In addition to providing a nicely shaped gauge volume for all data, having banks of detectors at ±90° offers us the opportunity of measuring diffraction from the axial and radial directions of an oriented sample simultaneously, when the sample is positioned 45° to the beam. Furthermore, the ±148° and ±60° banks provide additional data for orientations between axial and radial, such that roughly 1/3 of the entire range is covered. The instrumental resolution ($\Delta d/d$) for ±148° is 0.25%, for ±90° it is 0.40% and for ±60° 0.77%.

Diffraction profiles typically cover the time-of-flight range 3-30 milliseconds which corresponds to roughly 0.4-4.0 Å d-spacing. Individual diffraction peaks on the GPPD have a well-defined asymmetry (Von Dreele et al. (1982)), due to the pulsed nature of the source, fittable by convoluting leading and lagging exponentials with a Gaussian. Broadening effects such as microstrains alter only the Gaussian full-width.

In order to develop our ability to routinely handle a wide variety of residual stress samples, we have emphasized ancillary equipment capabilities. On the GPPD, data can be taken at essentially all temperatures from 10K to 1600K in a variety of sample configurations. We have a specially-designed translator-rotator which can be utilized for computer-controlled depth-profiling and 3D orientation analysis.

Data Analysis

Residual strains are typically calculated from d-spacings and full-width-half-maximum values obtained from singlet or multiplet peak fitting of key reflections. One such fit is illustrated in Figure 1 for data from a 30%Si_3N_4-Al_2O_3 ceramic composite.

Inherent to a TOF experiment, however, is the acquisition of many orders of those reflections defining unique crystallographic directions (e.g., (111), (222), etc.). These additional data can be used for confirmation of residual stress measurements from lowest order reflections, if systematic factors such as peak shape variation with TOF are understood. The Rietveld profile refinement technique (Rietveld (1968), Rotella (1986)) can be used to great advantage for this. A great number of characteristics such

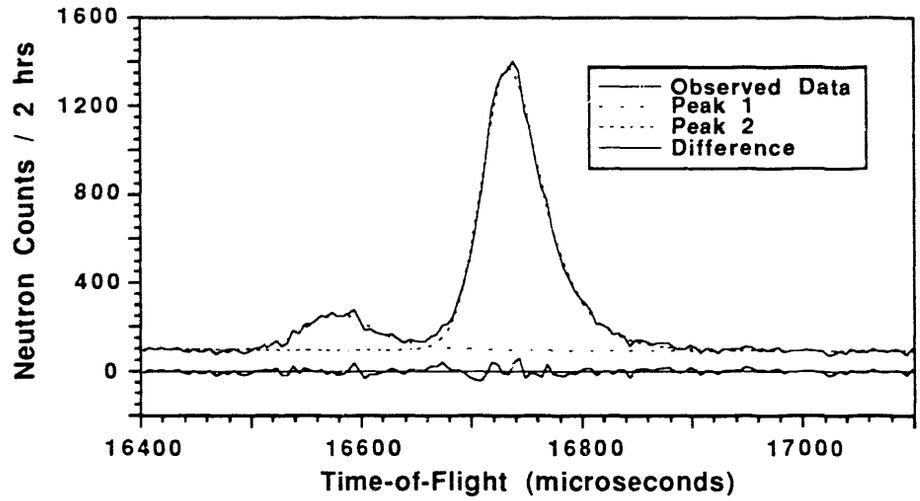

Figure 1. Typical least squares fit of two overlapping peaks (d~1.58 Å) from a Si_3N_4-Al_2O_3 composite. Note the slight asymmetry of the peaks which is inherent to the source.

as preferred orientation, anisotropic strain (varying peak breadth with crystallographic direction) and strain-induced phase transitions can also be modelled within the framework of the Rietveld formalism. The Rietveld profile fit for the Si_3N_4-Al_2O_3 composite is shown in Figure 2.

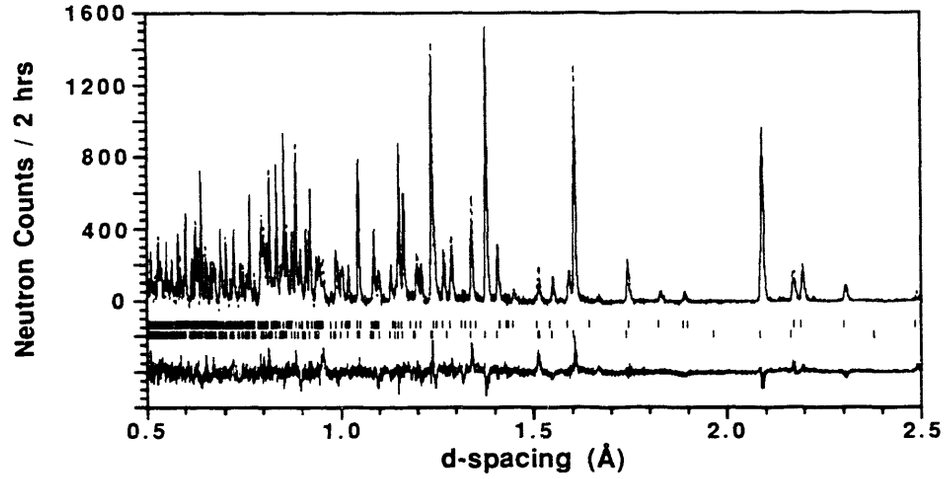

Figure 2. Rietveld profile fit for Si_3N_4-Al_2O_3 showing that: (a) both phases retain their structures, (b) peak breadths for both phases are modellable and (c) slight preferred orientation exists particularly in the second (Al_2O_3) phase.

Analytical Concerns

The demand for very high precision and accuracy in residual stress analyses requires us to understand the absorption-weighted centroid of scattering for each sample. This is

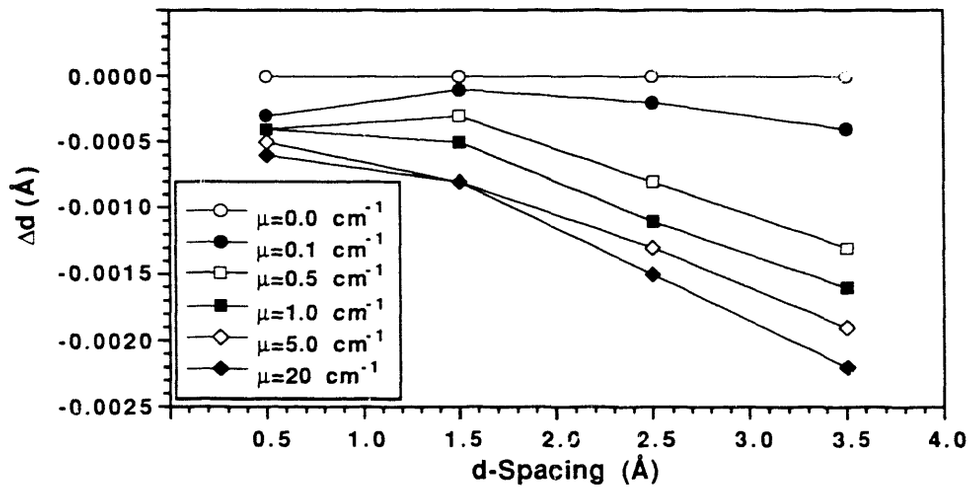

Figure 3. Monte Carlo calculations of the effect of absorption on apparent peak position on a time-of-flight diffractometer.

particularly critical when calculating stress-free parameters from standards with dramatically different bulk densities than the materials of interest. It is equally important to understand variations in sample position for different environments, e.g., a furnace versus a displex refrigerator. The importance of this can be calculated using Monte Carlo simulations of the instrumental characteristics. Shown in Figure 3 are plots of calculated Δd - representing shifts in peak positions - as a function of d-spacing for a series of possible sample linear neutron absorption coefficients. Such potential errors can be corrected either by using a stress-free standard with the same absorption characteristics or by applying an empirical correction. A similar effect can be expected when the sample diameter is changed as shown in Figure 4.

Although these errors are potentially severe, the Monte Carlo calculations appear to accurately reproduce the experimental behavior and empirical corrections appear to be sufficient when needed.

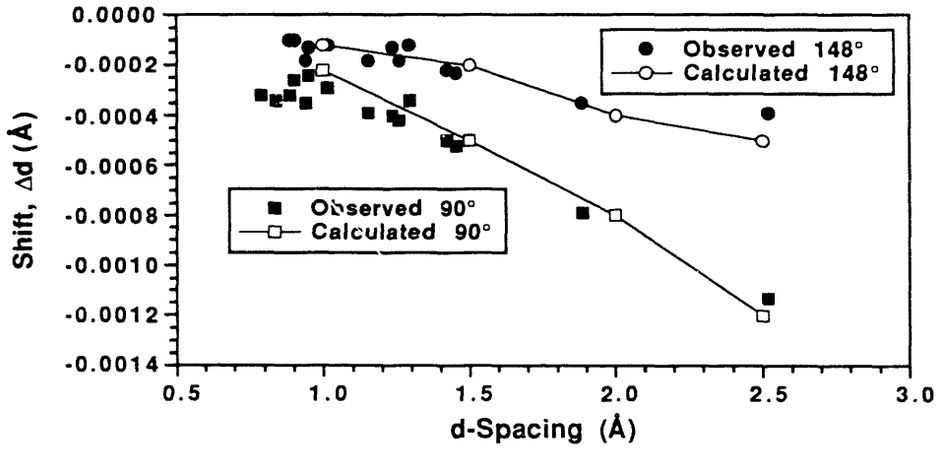

Figure 4. Apparent peak shifts due to changing from a $^1/_4$" diameter WC powder sample to a $^7/_{16}$" sample on the GPPD.

Conclusions

The GPPD powder neutron diffrctometer at IPNS combines good resolution with reasonable neutron flux to accomodate routine measurement of residual and applied stresses. Time-of-flight diffraction at fixed angle allows measurements from a geometrically fixed gauge volume throughout the d-spacing range. Lower time-average neutron fluxes relative to reactor sources can be partially overcome by examining changes in many reflections to obtain more complete strain information. This will be especially important as new mixed phase materials such as composites are studied in detail. Successfully exploiting some of the unique aspects of time-of-flight diffraction for stress measurements has broadened the scope of neutron diffraction as an non-destructive examination technique.

Acknowledgments

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References

Jorgensen, J. D., Faber, Jr., J., Carpenter, J. M., Crawford, R. K., Haumann, J. R., Hitterman, R. L., Kleb, R., Ostrowski, G. E., Rotella, F. J. and Worlton, T. G. (1989) 'Electronically Focused Time-of-Flight Powder Diffractometers at the Intense Pulsed Neutron Source', *J. Appl. Cryst.*, **22**, 321.

Krawitz, A. D., Roberts, R. and Faber, Jr., J (1986) 'Residual Stress Relaxation in Cemented Carbide Composites', Science of Hard Materials, Institute of Physics Conference Series No. 75, pp. 577-589.

Krawitz, A. D., Reichel, D. G. and Hitterman, R. L. (1987) 'Residual Stress and Stress Distribution in a WC-Ni Composite', *J. of Mater. Sci. Eng.*, **A119**, 127-134.

Kupperman, D. S., Singh, J. P., Faber, Jr., J. and Hitterman, R. L. (1989) 'Application of Neutron Diffraction to the Characterization of Residual Strains in YBa_2 Cu_3 O_{7-x} / Ag', *J. Appl. Phys.*, **66**, 3396-3398.

MacEwen, S. R., Faber, Jr., J. and Turner, A.P.L. (1983) 'The Use of Time-of-Flight Neutron Diffraction to Study Grain Interaction Stresses', *Acta Metall.*, **31**, 657-67.

MacEwen, S. R., Faber, Jr., J. and Turner, A.P.L. (1984) 'The Influence of Texture on the Interpretation of Diffraction Data to Determine Residual Stress', *Scripta Metall.*, **18**, 629-33.

Majumdar, S., Kupperman, D. S. and Singh, J. P. (1988) 'Determination of Residual Thermal Stresses in a SiC/Al_2O_3 Composite Using Neutron Diffraction', *J. Am. Ceram. Soc.*, **71**, 858-863.

Majumdar, S. and Kupperman, D. S. (1989) 'Effects of Temperature and Whisker Volume Fraction on Average Residual Thermal Strains in a SiC/Al_2O_3 Composite', *J. Am. Ceram. Soc.*, **72**, 312-313.

Rietveld, H. M. (1968) 'A Profile Refinement Method for Nuclear and Magnetic Structures', *J. Appl. Cryst.*, **2**, 65-71.

Rotella, F. J. (1989) 'Users Manual for Rietveld Analysis of Time-of-Flight Neutron Powder Diffraction Data at IPNS', Argonne National Laboratory, USA.

Von Dreele, R. B., Jorgensen, J. D. and Windsor, C. G. (1982) 'Rietveld Refinement with Spallation Neutron Powder Diffraction Data', *J. Appl. Cryst.*, **15**, 581-589.

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