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Title:

Complementary Neutron Diffraction and Computational Micromechanics Studies - of Polycrystalline Deformation in Advanced Materials

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Abstract

Neutron diffraction provides a unique, non destructive method for studying deformation in polycrystalline materials. At pulsed sources like LANSCE the strain information typically comprises all possible lattice reflections. This is attractive because of its comprehensive description of the strain state. Indeed the gross aspects of the results, almost invariably, describe the long range or mean phase strain behavior reasonably well. However it is more of a challenge to interpret the data associated with the microscopic or intergranular strains which typically exceed our ability to model them. Research performed during this LDRD project addressed three areas that relate to the distinction of micro and macrostrains in engineering materials; 1) validation and development of self consistent models, 2) provision of a routine and reliable analysis method for "engineering" interpretation of data from a pulsed neutron source, & 3) assessment of problems in which strain, texture and phase fraction all evolve simultaneously.

Background and Research Objectives

The use of neutron scattering to examine highly anisotropic micro-scale strain fields in polycrystalline materials is limited by the ability to interpret all of the information contained in neutron diffraction patterns. This is especially true for studies of complex or graded microstructures or on heterogeneous materials, such as engineering composites. Even the types of experiments that are attempted are restricted by the fore-knowledge that, at present, interpretion of the results is limited to relatively simplistic bulk-averaging material theories and numerical tools based on continuum. At Los Alamos researchers have championed the use of the LANSCE pulsed neutron source to probe mean phase strains in disparate heterogeneous materials. However this has meant that we have needed to be aggressive in using the extensive microstructural information (implicit in a measurement at a pulsed source) to extract quantitative information on microstructural deformation.

The global behavior of bulk polycrystalline materials is determined by deformation patterns at length scales comparable to the microstructure. To better understand this it is necessary to document the deformation in grains in specific orientations with respect to a deforming axis. Although conventional X ray measurements do select individual grain directions but they are generally not representative of the bulk because of their limited penetration depth and the relaxation of constraint at the surface compared to the interior. Whereas synchrotron X-ray measurements do overcome this problem they are limited to lower Z materials and neutron diffraction is more appropriate for recording the average bulk behavior of families of grains in specific orientations.

Practical reasons for studying the development of deformation induced residual strains include their effects on both constitutive response and material failure resistance including ductility and fracture toughness. Phase transformation kinetics and issues such the relationship between texture and constitutive performance are also sensitive to the microstructural details of the polycrystalline deformation behavior.

Importance to LANL's Science and Technology Base and National R&D Needs

Experimental results from this project were used to enhance and validate the predictive capabilities of constitutive models for anisotropic materials and low symmetry materials for

which continuum mechanics calculations were insufficient to capture the describe the microstructural level deformations. These improved models will ultimately lead to a better understanding of microscopic deformation at grain level and are currently being applied to materials of direct LANL interest such as beryllium and a Uranium 6% Nb alloy. The ultimate goal is to improve the simulation of deformation-processing or remanufacturing. The studied materials were chosen either because of their relevance to LANL or because of their unique scientific interest. Neutron diffraction is playing an increasing role in studying materials within the context of the SBSS initiative. Studies of constitutive performance and of residual strain distributions will take place over the next few years using the new SMARTS spectrometer thus a fundamental understanding of deformation physics is critical to the interpretation of the data.

Scientific Approach and Accomplishments

This section comprises 3 sections. First it briefly discusses the LANL neutron scattering capability. Second it addresses the self consistent model (used to describe the experimental data). Finally it lists technical highlights extracted from referenced papers.

Experimental studies using neutron diffraction

Neutron diffraction provides a non-destructive, bulk averaging phase and *hkl* discriminatory tool for recording the elastic strains in polycrystalline materials. Since thermal neutrons interact weakly with most materials, penetrations are typically millimeters to centimeters, and volumes of several cubic centimeters can be sampled. In a typical neutron scattering experiment elastic lattice strains are inferred from changes in inter-planar lattice spacing. The direction of strain measured is determined by a specimen's alignment with respect to the scattering geometry while the phase or grain orientation is determined by selection of appropriate lattice reflections. For prediction of the overall polycrystalline response the strain behavior of all *hkl*'s are needed in multiple directions with respect to a loading axis. At LANSCE all possible reflections are recorded simultaneously in each measurement. Each pulse of neutrons contains a continuous spectrum of energies, scanning a specimen in wavelength so that a complete diffraction pattern is obtained by a detector at any angle with *no movement* of specimen or detector. During *in situ*

loading measurements using a load frame (specifically designed for this work) bulk strain response is recorded for all crystallographic directions parallel, perpendicular and at 60° to a loading axis. The NPD spectrometer used for the measurements is a high resolution instrument with four detector banks. The load frame can apply up to 1 GPa in uniaxial compression or tension to specimens 10 mm in diameter.

Self consistent model (SCM)

In a SCM grains in a polycrystal are treated as ellipsoidal inclusions in a homogeneous equivalent medium (HEM), with the properties of the polycrystal as a whole. By applying a stress or strain state to the HEM the stresses and strains in the individual grains can be determined using the Eshelby solution for an ellipsoidal inclusion in an infinite homogeneous medium. The problem is solved iteratively for a macroscopic stress or strain increment, as the properties of the HEM is determined as the average over all the grains. Thereby the name 'self-consistent' model. A critical resolved shear stress is used for each plastic deformation mode (crystallographic slip only) available for the given crystal structure, and as the stress state in each grain is known, it is straight forward to determine the elastic and plastic strain in the grain. As plastic deformation takes place the grains follow an exponentially decreasing hardening law. Whereas the SCM is ideal for comparison with a neutron diffraction measurement the challenge comes from accurately describing the deformation mode and from incorporating the effects of texture.

Technical highlights

1 *Demonstration of Rietveld refinement as an averaging tool*

Macrostrain variations in engineering components are frequently performed using neutron diffraction. At both reactors and pulsed sources it is desirable to minimise the sampling volume in order to maximise the spatial resolution (although this increases the required measurement time). At reactors, macrostrain behaviour is inferred from a single lattice reflection (deemed to be representative of the bulk response). At a pulsed source, a complete diffraction pattern is recorded and accordingly it is natural to fit the entire diffraction spectra using a Rietveld refinement. This means that an idealised crystal structure is fit to the measured distorted crystal

structure, which includes deviation of the measured lattice reflections from the ideal due to hkl dependent elasto-plastic strain anisotropies. First we showed that for a (cubic) stainless steel, elastic macrostrains calculated from lattice parameter changes in Rietveld refinements, without accounting for hkl dependent anisotropies, are almost identical to the bulk elastic response (Fig 1). Then by incorporating a description of the *elastic* strain anisotropy expected in cubic materials into the Rietveld code, an empirical prediction of *plastic* strain history is possible [1]. The validity of these arguments is demonstrated by analysis of a uniaxial tensile load test and a re-analysis of previously reported data taken on a deformed stainless steel ring. The plastic strain predictions compare favourably with a finite element model. This study has been extended to a hexagonal crystal structure [2].

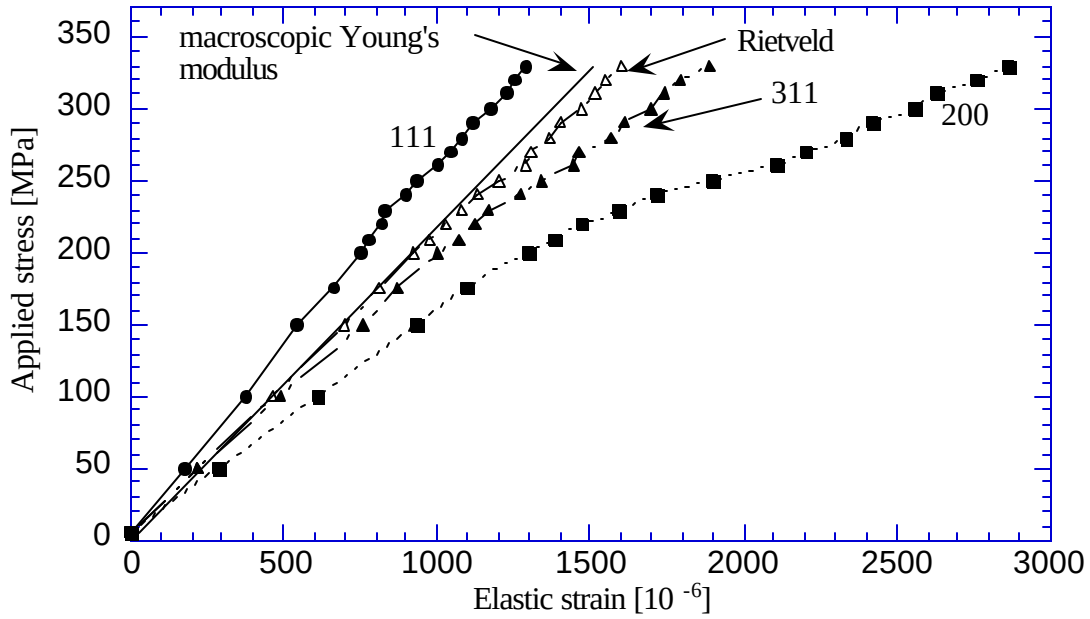

Figure 1. The stress-strain responses of the 111, 200 and 311 planes, parallel to the load axis, compared with strains determined using the lattice parameter from a Rietveld refinement that does not incorporate any elastic strain anisotropy. The bulk elastic response is also shown.

2 Application of the self consistent model to a strongly textured system

The applicability of the SCM was demonstrated on a randomly texture stainless steel [3]. Then to assess its viability on a textured problem tensile specimens were machined from a heat treated austenitic stainless steel plate prior to and after a 70% reduction by uni-directional rolling[4]. Measurements were performed on specimens cut from the rolled plate, with their

axes parallel and perpendicular to the rolling direction. *In situ* measurements of the strain response (to macroscopic plastic strains of around 1%) were recorded. The experimental results were then compared with predictions from a self-consistent Hill-Hutchinson model. It transpired that the effect of this level of rolling proved to be relatively small and the model was reasonably successful in describing the *qualitative* nature of the strain anisotropy (hardening meant that the yield stress was increased by a factor of 4) (Fig 2). By far the largest effect proved to be on the residual strains left by the rolling process.

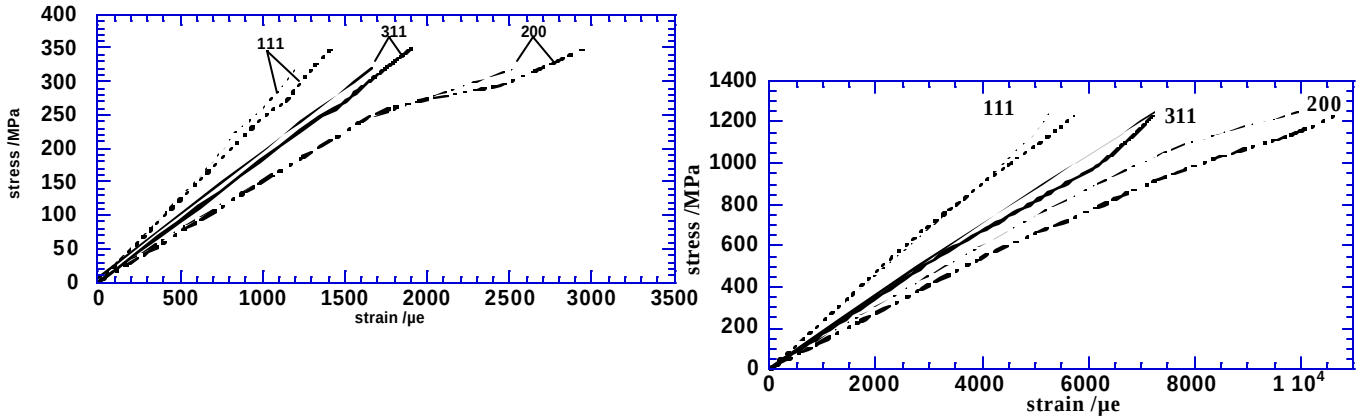

Figure 2. Left- Experimental and modeled lattice strain for a uniaxial tensile test on untextured nitronic stainless steel. Right - Experimental and modeled lattice strain $\varepsilon \| \sigma$ (The SCM assumes spherical grains and includes a pre-strain for the textured sample). Thick lines are model data, thin lines are experimental.

3 Studies on a NiTi shape memory alloys

One class of materials that are uniquely suited for studies using neutron diffraction are shape memory alloys. At a pulsed source characterization of a transformation is always performed with a complete diffraction pattern. Since the transformations are associated with texture effects this is easily discernable when data are collected simultaneously in several scattering geometries they provide an excellent system with which to explore the intricacies of strain-texture relationships. One system that was extensively studied within this LDRD is near-equiatomic nickel-titanium alloys and NiTi-titanium carbide composites[5,6,7]. NiTi-based alloys exhibit a thermoelastic phase transition between a low-temperature, monoclinic (B19') martensitic phase and a high-temperature, cubic (B2) austenitic phase. The allotropic nature of the alloys and the

reversible deformation mechanism give rise to an effect called "superelasticity," in which the alloy can deform elastically by as much as 8%, with all strains being recoverable on unload [8]. An example of the measured data is given in Fig 3.

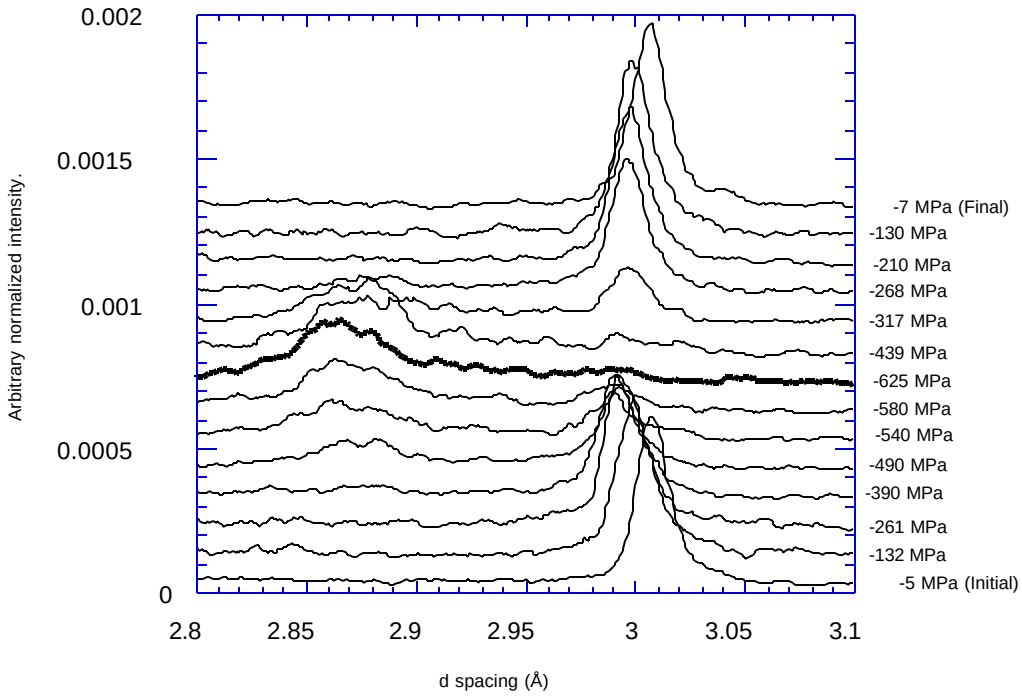

Figure 3 ; Displaced spectra showing a short section of neutron diffraction patterns obtained during a load test. The austenitic [100] at 3.0Å reduces in intensity to approximately 0 at -625 MPa. Conversely the martensitic [100] at 2.86Å becomes visible at -490 MPa reaching a maximum in intensity at -625 MPa (bold line). The shift to shorter d spacings is caused by the increasing elastic compressive strain. The data is smoothed and displaced vertically for clarity.

4 Yield stress prediction in an intermetallic composite using measured residual stresses

NiAl-based hybrid composites containing 5, 15 and 30 volume percent of the Al_2O_3 fibers in addition to the AlN dispersion particles were fabricated via powder metallurgy route. The strengthening mechanism(s) associated with the short Al_2O_3 fiber in NiAl-$(AlN)_{dispersion}$-$(Al_2O_3)_{fiber}$ composite was studied with emphasis on the effect of the thermal residual stress on the compressive strength at room temperature and 1300K. At 300K, the yield strength of the composites was predicted using a linear superposition of monolithic yield strength, direct fiber

strengthening and tensile matrix residual stresses. The prediction shows good agreement with the measured data (see Fig 4). The majority of the room temperature strengthening is attributable to the residual stress, and the measurements required separation of the residual stresses in the 3 phases which was a tour de force in interpretation of the data [9,10]. At 1300K, the strengthening was achieved only by the load sharing of the fibers and there is no direct influence from the process-induced thermal residual stresses as they have completely relaxed at this temperature.

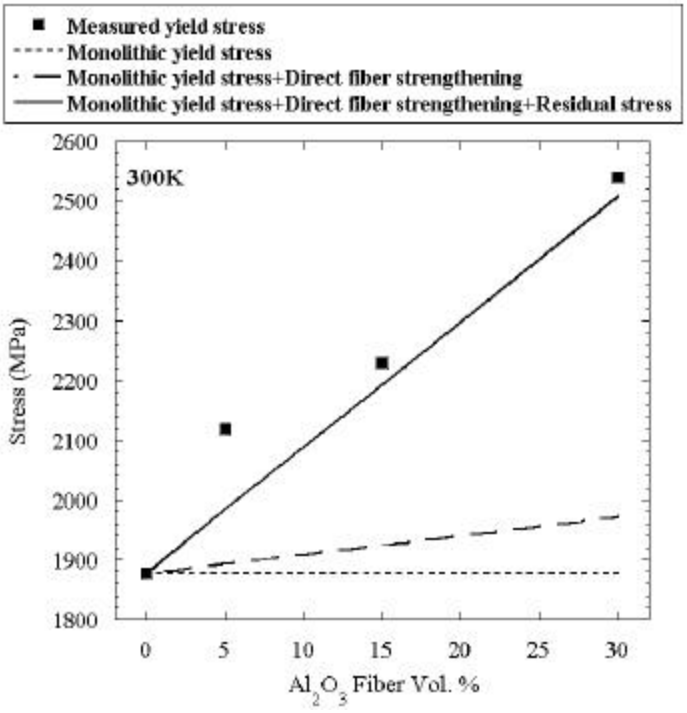

Fig 4 ; Measured (symbols) and predicted yield stress in NiAl AlN Al2O3 composites using residual stresses (solid line) measured by neutron diffraction.

5 Other results

Research in this LDRD contributed to an MS [11] theses and two PhD [12,13] theses.
Experimental measurements were performed on two co-deforming systems CuMo and BeAl [14,15,16] and on Al2O3 SiC nanocomposites [17].

Project publications

1. *Daymond, M.R. Bourke, M.A.M. VonDreele, R.B. Clausen, B. Lorentzen T. "Use of Rietveld refinement for elastic macrostrain determination and for the evaluation of plastic strain history from diffraction spectra"J. App Phys 82 pp 1554 1997 LAUR 97-1219*

2. *Daymond, M.R. Bourke, M.A.M., Von Dreele, R.V. "Use of Rietveld refinement to fit hexagonal crystal structures in the presence of elastic and plastic anisotropy" J . Appl. Physics Vol 85 (2) pp 739 1999 LAUR 98-3189*

3. *Clausen, B., Lorentzen, T., Bourke, M.A.M. & Daymond, M.R. "Lattice Strain Evolution During Uniaxial Loading of Stainless Steel" Materials Science & Engineering A, Vol. 259(1), pp. 17-24, 1999, LAUR 97-4780*

4. *Daymond, M.R., Bourke, M.A.M. and Tome, C. "Effect of Texture on hkl Dependent Intergranular Strains Measured In-Situ at a Pulsed Neutron Source" Proceedings International Conference on Residual Stresses V, Linkoping, Sweden pp 577-585 1997 LAUR 97-2955*

5. *Vaidyanathan, R. Bourke, M.A.M. Dunand D.C. "Stress Induced martensitic transformations in NiTi and NiTiTiC composites investigated by neutron diffraction" Materials Science and Engineering A (In Press) 1999 LAUR 98-3567*

6. *Vaidyanathan, R. Bourke, M.A.M. Dunand D.C. "Analysis of neutron diffraction spectra obtained during stress induced phase transformations in superelastic NiTi" J. Applied Physics (In press) 1999 LAUR 98-2586*

7. *Dunand, D.C., Fukami-Ushiro, K.L., Mari, D., Roberts, J.A., Bourke, M.A.M. "Mechanical Properties of NiTi-TiC Shape Memory Alloys" Mat.Res.Soc Symp. Proc. Vol 459, pp 131- 141 1997 LAUR n/a*

8. *Bourke, M.A.M., Vaidyanathan, R., and Dunand, D.C. "Neutron-Diffraction Measurement of Stress-Induced Transformation in Superelastic NiTi" Applied Physics Letters 69:17 pp 2477-2479. 1996 LAUR 96-1959*

9. *Choo, H. Bourke, M.A.M. Nash P. "Effect of thermal residual stress on the mechanical properties of NiAl based composites" High temperature ordered intermetallic alloys VIII (MRS Proceedings) (Submitted) 1998 LAUR 98-5647*

10. *Choo, H. Bourke, M.A.M. Nash. P. Daymond, M.R. Shi N. "Thermal residual stresses in NiAl-AlN-Al_2O_3 composites measured by neutron diffraction" Materials Science and Engineering A (In Press) 1999 LAUR 98-1257*

11. *Lund, C. "A study of deformation in metal matrix composites at room and elevated temperature using neutron diffraction" M.S. Thesis, Massachusetts Institute of Technology, 1998*

12. *Choo, H. "Synthesis processing and characterization of NiAl-AlN-Al2O3 composites", PhD thesis Illinois institute of technology, 1998*

13. *Vaidyanathan, R. "Mechanical properties of superelastic and shape memory NiTi and NiTiTiC composites investigated by neutron diffraction", PhD Massachusetts institute of technology, 1998*

14. *Clausen, B. Bourke, M.A.M. "Microstructural stress/strain inhomogeneity in MMC's" Nondestructive evaluation (NDE) and materials properties IV Edited by P.K. Liaw, R.J. Arsenault , R.E. Green Jr. et al . The Minerals Metals and Materials Society, pp 15 – 19, 1999. LAUR 98-5968*

15. *Daymond, M.R. Lund, C. Bourke, M.A.M. Dunand D.C. "Elastic phase strain distribution in a particulate-reinforced metal matrix composite deforming by slip or creep" Acta Mat (Submitted) 1999 LAUR 97-4677*

16. *Carter, D.H. Bourke M.A.M. "Neutron diffraction study of the co-deformation behavior of beryllium aluminum composites" Acta Mat. (submitted) 1999 LAUR 96-4695*

17. *Todd, R.I., Bourke, M.A.M., Borsa, C.E., Brook, R.J. "Neutron diffraction measurements of residual stresses in alumina/sic nanocomposites" Acta Mater. Vol 45, No 4 pp 1791-1800, 1997 LAUR 96-0074*

Figure captions (repeat)

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