

To the Graduate Council:

I am submitting herewith a dissertation written by Xin Luo entitled “Study on Infrastructure Materials Using Neutron Radiography and Diffraction.” I have examined the final electronic copy of this dissertation for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, with a major in Civil Engineering.

Dayakar Penumadu
Major Professor

We have read this dissertation
and recommend its acceptance:

Eric C. Drumm

Richard M. Bennett

Hahn Choo

Accepted for the Council:

Carolyn R. Hodges
Vice Provost and Dean of the Graduate School

(Original signatures are on file with official student records.)

**STUDY ON INFRASTRUCTURE MATERIALS USING
NEUTRON RADIOGRAPHY AND DIFFRACTION**

**A Dissertation
Presented for the
Doctor of Philosophy Degree
The University of Tennessee, Knoxville**

**Xin Luo
August 2007**

Copyright © 2007 by Xin Luo

All rights reserved

Dedication

This dissertation is dedicated to my wife Miao Chen
for her love and patience.

Acknowledgement

Firstly, I wish to thank all those who have supported and contributed to the completion of this research. I would like to thank my advisor Dr. Dayakar Penumadu for offering this opportunity to work with him, encouraging me throughout this journey, and providing help in my daily life. I wish to thank other members in my committee, Dr. Eric Drumm, Dr. Richard Bennett, Dr. Hahn Choo for their time and inputs in the review of the dissertation.

I wish to thank the Joint Institute for Neutron Science (JINS) Fellowship Program and the NSF International Materials Institute (IMI) Program for providing partial funding for the proposed research, travel support to neutron science related conferences, workshops and seminars. The research work undertaken at Oak Ridge National Laboratory (ORNL) was sponsored by the High Temperature Materials Laboratory User Program, ORNL. The research work carried out at the Spectrometer for Materials Research at Temperature and Stress (SMARTS) was benefited from the user program of the Los Alamos Neutron Science Center, Los Alamos National Laboratory. The training and technical support from the McClellan Nuclear Radiation Center (MNRC), UC Davis is appreciated.

Research projects as large as these would never be completed without teamwork. Special thanks go to those scientists who have provided tremendous supports and constructive inputs: Dr. Camden Hubbard, Dr. Ke An, Dr. Fei Tang, Dr. Thomas R. Watkins, Mr. W. Barton Bailey from Oak Ridge National Laboratory; Dr. Donald W. Brown, Dr. Bjorn Clausen from Los Alamos National Laboratory; Dr. Peter K. Liaw, Mr. Ken Thomas from the University of Tennessee, Knoxville; Mr. Hal Egbert and other

scientists and engineers from McClellan Nuclear Radiation Center, University of California at Davis; Dr. R. A. Winholtz from University of Missouri, Columbia.

I also would like to thank my friends and colleagues, Dr. Gang Zuo, Dr. Songqing Wen, Dr. Xueping Li, Dr. Xiang Yu, Dr. Amal Dutta, Dr. Xin Chen, Dr. Amit Prashant, Mr. Mathew Kant, Mr. Akawut Siriruk, Mr. Haitao Xu, Ms. Liang Ma, Mr. Caijun Su, Mr. Alex Davis-Smith, Mr. Bo Lu, for their wonderful friendship and kind help.

Last but not least, I wish to thank my wife and my parents for their constant encouragement and patience throughout the journey I have undertaken. Without their unwavering support I would never have reached this level.

Abstract

Advanced nondestructive neutron technology has been utilized to study fundamental issues in the Lost Foam Casting (LFC) process and in the mechanical behavior of infrastructure materials.

Time lapsed neutron radiography combined with digital image processing was used to investigate the real-time LFC process. Behavior and characteristics of the pyrolysis front in the LFC processes were discussed. Evidence shows that neutron radiography offers new insights into the pyrolysis front and the dynamics of the processes involved with the casting. Behavior and characteristics of the pyrolysis front and the molten metal interface in the LFC processes were revealed. The proposed approach will prove to be a powerful tool to characterize the degradation behavior of the expanded polystyrene foam during the LFC process and the interactions of liquid metal.

The stress-strain relationship of particulate materials is complex, and depends on the initial state of packing, past stress history, and the applied stress path. A novel in-situ study methodology has been developed using neutron scattering technique to obtain strains both globally and locally. The significant differences between the global deformation and the local lattice strain for silica sand have been found and discussed. The measured lattice strain was at least one order of magnitude smaller than the measured related global strain. However, the actual stress within the particles could be much higher than the applied global stress. Research results from this study will be useful for developing suitable elasto-plastic constitutive models of frictional granular materials.

Residual stress has a significant impact on the mechanical behavior materials. It is difficult to be measured or predicted using analytical methods, and can lead to premature failure of materials if not appropriately considered in design. Residual strains of identical steel tubular specimens after being subjected to either torsion or tension corresponding to a target equivalent strain invariant were probed using both reactor and spallation neutron sources. The lattice strains based on the hkl reflections that are reported to be both weakly and strongly affected by intergranular strain for tension stress path were investigated. The results indicate the essential difference between tension and torsion from the perspective of yield and failure criteria for materials.

An innovative approach has been developed to study the complete 3-D strain tensor using the 2nd Generation Neutron Residual Stress Facility at Oak Ridge National Laboratory. A procedure was also established to understand the mechanism and to analyze the errors of the calculated strain tensor. This newly developed approach makes it possible to study the strain/stress state in materials under complex conditions.

Contents

Chapter 1 Introduction to neutron radiography and neutron diffraction.....	1
What is neutron	2
Attenuation of neutron and neutron radiography	4
Bragg's Law and neutron scattering	10
Research tasks and objectives	12
References	15
 Chapter 2 Visualization of the Multiphase Flow in the Lost Foam Casting Process by	
Neutron Radiography and Image Processing.....	17
Abstract	18
Introduction to Lost Foam Casting	19
Fundamentals of Lost Foam Casting process	20
Neutron radiography for Lost Foam Casting	22
Neutron facility for studying the Lost Foam Casting	26
Image processing for neutron images	31
Neutron radiography results and discussion	33
Summary	49
Acknowledgement	50
References	51
 Chapter 3 Stress-Strain Behavior of Granular Materials under Confined Compression	
Using In-situ Neutron Diffraction.....	53
Abstract	54

Mechanical behavior of granular materials.....	55
Measurement of strains by diffraction method	58
Materials and sample preparation	59
Engineering stress mapping facility using neutrons	63
Experimental results and discussion	70
Summary	85
Acknowledgement	86
References.....	87

Chapter 4 Mapping of Residual Strains in Steel Tubes after Subjected to Either Torsion

or Tension Using Reactor Neutron Source	89
Abstract.....	90
Fundamentals of residual stress and strain.....	91
Generalized loading conditions by tension and torsion	94
Strain or stress measurement with diffraction method	96
Details of strain measurements using neutron diffraction	98
Results of residual strain mapping and discussion	105
Summary	117
Acknowledgement	117
References.....	119

Chapter 5 Residual Strains in Steel Tubes After Application of Either Torsion or Tension

Using Spallation Neutron Source.....	122
Introduction to residual stress	123

Diffraction based measurement using spallation neutron source	125
Materials and stress-strain history of steel tubular samples.....	127
Measurement of lattice strains by neutron diffraction	131
Summary	147
Acknowledgement	148
References.....	149
Chapter 6 Residual Strain Tensor for Steel Hollow Cylinders after Subject to Tension or Torsion Using Reactor Neutron Source	152
Introduction to measurement of residual stress	153
Stress and strain tensors, and Haigh-Westergaard space	155
Determination of strain tensor using Least Squares fitting.....	158
Measurement of lattice strains with Neutron diffraction	164
Error estimate of strain tensor based on neutron diffraction.....	175
Summary	181
Acknowledgement	182
References.....	183
Chapter 7 Summary and Future Work	186
Summary	187
Future work.....	190

List of Tables

Table 1.1 Neutron properties (Rauch and Waschkowski 2003)	3
Table 1.2 Neutron classification based on energy (Anderson 2007)	3
Table 2.1 Material properties and the calculated thickness for 50% attenuation	25
Table 3.1 Physical properties of Ottawa and Q-Rok silica sand.....	59
Table 3.2 Initial void ratios and relative density of the sand for the confined compression	64
Table 4.1 Typical compositions of 12L14 steel (ASM 1990)	99
Table 4.2 Mechanical properties of 12L14 steel (ASM 1990)	99
Table 4.3 Neutron scattering parameters used in the strain mapping	104
Table 4.4 Experimental parameters of the two levels of loading for tension and torsion	108
Table 6.1 Neutron diffraction parameters used in the strain tensor measurement.....	167
Table 6.2 Combination of the angles ϕ and ψ for different tilts in strain tensor measurement	170
Table 6.3 Strain tensor and the corresponding principal strains ($\mu\epsilon$) for the (110) and (211) reflections of the torsion and the tension samples.....	Error! Bookmark not defined.
Table 6.4 Comparison of the Least Squares fitting results for the neutron scattering measurements with different combinations of the ϕ and ψ angles	176
Table 6.5 Estimated standard deviation of the components of calculated strain tensor .	180

List of Figures

Figure 1.1 Interaction of neutrons with matter	8
Figure 1.2 Attenuation of Neutrons and X-rays for different elements (Berger 1965)	9
Figure 1.3 Elastic neutron scattering and Bragg's law	11
Figure 2.1 Neutron radiography facility at MNRC, UC Davis.....	28
Figure 2.2 MNRC radiography setup to study LFC	29
Figure 2.3 Three types of gating systems	30
Figure 2.4 A typical histogram of a digital image	32
Figure 2.5 An original neutron image prior to LFC process.....	34
Figure 2.6 A converted pseudo-color image prior to LFC process.....	34
Figure 2.7 Histograms of the EPS foam area (Side-gated, thickness 4 mm).....	35
Figure 2.8 Histograms of the EPS foam area (Side-gated, thickness 12 mm).....	36
Figure 2.9 Histograms of the EPS foam area (Side-gated, thickness 24 mm).....	37
Figure 2.10 Pixel intensity of the foam pattern area (side-gated).....	39
Figure 2.11 Bottom-gating casting with pattern thickness 4 mm (at 19 sec)	40
Figure 2.12 Side-gating casting with pattern thickness 4 mm (at 22 sec)	41
Figure 2.13 Neutron image of the side-gating with pattern thickness 24 mm (at 15 sec)	42
Figure 2.14 The Side-gating casting with pattern thickness 24 mm (at 15 sec)	42
Figure 2.15 Neutron image of the side-gating with pattern thickness 12 mm (at 15 sec)	43
Figure 2.16 The Side-gating casting with pattern thickness 12 mm (at 15 sec)	43
Figure 2.17 Neutron image of the side-gating with pattern thickness 4 mm (at 7 sec)	45
Figure 2.18 The Side-gating casting with pattern thickness 4 mm (at 7 sec)	45

Figure 2.19 The pyrolysis fronts of side-gating with thickness 24 mm (at 3, 6, and 15 sec)	47
Figure 2.20 The pyrolysis fronts of side-gating with thickness 4 mm (at 3, 6, and 15 sec)	48
Figure 3.1 Particle size distribution chart for Ottawa and Q-Rok silica sand	60
Figure 3.2 Experimental set-up of Geotest S5770 and the data acquisition system	62
Figure 3.3 Stress and void ratio behavior of Ottawa sand	65
Figure 3.4 Stress and void ratio behavior of Q-Rok sand	66
Figure 3.5 NRSF2 and in-situ load frame at HFIR, ORNL	67
Figure 3.6 Schematic of the continuous loading measurement in axial direction	68
Figure 3.7 Schematic of the continuous loading measurement in transverse direction	69
Figure 3.8 Schematic of the local strain mapping measurement in axial direction	71
Figure 3.9 The selected hkl reflection (-321) for the SiO_2	72
Figure 3.10 Typical results of fitting of Bragg's peak from neutron diffraction data	73
Figure 3.11 Global and lattice strains vs global stress in axial direction for Ottawa sand	75
Figure 3.12 Global and lattice strains vs global stress in axial direction for Q-Rok sand	78
Figure 3.13 Global and lattice strains vs global stress in transverse direction for Ottawa sand	80
Figure 3.14 Global and lattice strains vs global stress in transverse direction for Q-Rok sand	81
Figure 3.15 Variation of local lattice strains of Ottawa sand in longitudinal direction under different loads	83
Figure 3.16 Variation of local lattice strains of Q-Rok sand in longitudinal direction under different loads	84

Figure 4.1 Three types of residual stresses (Holden and Bowen 1991).....	93
Figure 4.2 Control of directions of principle stresses through torsion-axial loading system	95
Figure 4.3 Diffraction measurements for radial strains	100
Figure 4.4 Diffraction measurements for circumferential strains	101
Figure 4.5 Diffraction measurements for axial strains.....	102
Figure 4.6 BCC Fe and the lattice planes used in the neutron diffraction	103
Figure 4.7 Stress-strain history of the tubular specimen for the torsion test	106
Figure 4.8 Stress-strain history of the tubular specimen for the tension test.....	107
Figure 4.9 Residual strains in axial direction for the reflection (110) for samples subject to the same invariant strain in torsion or tension	110
Figure 4.10 Residual strains in axial direction for the reflection (211) for samples subject to the same invariant strain in torsion or tension	111
Figure 4.11 Residual strains in circumferential direction for the reflection (110)	113
Figure 4.12 Residual strains in circumferential direction for the reflection (211)	114
Figure 4.13 Residual strains in radial direction for the reflection (110).....	115
Figure 4.14 Residual strains in radial direction for the reflection (211).....	116
Figure 5.1 Stress-strain history of the sample for the torsion test	128
Figure 5.2 Stress-strain history of the sample for the tension test.....	129
Figure 5.3 Research facility at SMARTS, LANL.....	132
Figure 5.4 Experimental set-up measuring residual strain using spallation neutron source	134
Figure 5.5 An observed time-of-flight (TOF) diffraction pattern.....	135

Figure 5.6 A time-of-flight (TOF) diffraction pattern after normalization and Rietveld refinement	136
Figure 5.7 Individual peak fitting of the unstressed sample in radial direction (Peak# 1-4)	138
Figure 5.8 Individual peak fitting of the unstressed sample in radial direction (Peak# 5-7)	139
Figure 5.9 Residual strains of multiple (hkl) reflections at different heights in radial direction	140
Figure 5.10 Residual strains of multiple (hkl) reflections at different heights in circumferential direction.....	141
Figure 5.11 Residual strains in radial direction at various locations of the tension sample	142
Figure 5.12 Residual strains in radial direction at various locations of the torsion sample	143
Figure 5.13 Residual strains in circumferential direction at various locations of the tension sample.....	144
Figure 5.14 Residual strains in circumferential direction at various locations of the torsion sample	145
Figure 6.1 Decomposition of stress vector in Haigh-Westergaard space and Von Mises yield criterion	157
Figure 6.2 Relation between sample (S) and lab (L) coordinate systems.....	160
Figure 6.3 Stress-strain history of the tubular specimen for the torsion test	165
Figure 6.4 Stress-strain history of the tubular specimen for the tension test.....	166

Figure 6.5 Experimental set-up for the residual strain tensor measurement at HFIR, ORNL.....	169
Figure 6.6 An example of the experimental set-up for the d-spacing measurement (radial direction) in order to obtain strain tensor.....	171
Figure 6.7 d-spacing of the (110) reflection for the unstressed sample.....	172
Figure 6.8 d-spacing of the (211) reflection for the unstressed sample.....	173

Chapter 1 Introduction to neutron radiography and neutron diffraction

What is a neutron

Neutrons have both a wave and a particle state called wave-particle dualism. On the one hand, a neutron is a subatomic particle without net electric charge and with a mass slightly more than a proton. On the other hand, a neutron is a wave, and the wave-like nature explains its interference. Typical properties and classification based on energy of neutrons used in scientific research are given in Table 1.1 and Table 1.2. Thermal neutrons, whose energy is relatively low, have wavelengths comparable to interplanar d-spacings in crystal lattice and energy comparable to the collective vibration energy in condensed matter (Hutchings, Withers et al. 2005). This type of low-energy neutron is an important tool for the investigation of the static and dynamic properties of condensed matter. It can also be used for a detailed study of interaction of the neutron as an elementary particle with its surroundings.

Neutrons are produced in various nuclear reactions such as nuclear fission, or the spallation process. Those emission neutrons have high energies of several MeV, and can be slowed down to thermal energies through successive collisions at moderator. Such neutrons carry the nuclear chain reaction in most nuclear reactors and they can be extracted from the moderator by beam tubes and neutron guides. Monochromators are often used in reactor neutron source to filter out the thermal spectrum in order to obtain single wavelength neutron beams. Time-of-flight technique is used in pulsed sources such as OPNS (Richardson 1992), LANSCE (Bourke, Goldstone et al. 1992), or ISIS (Hull, David et al. 1992) to measure the energy and the energy change of neutrons after interaction with matter. Absorption, transmission and scattering of neutrons carry the

Table 1.1 Neutron properties (Rauch and Waschowski 2003)

Mass (kg)	$1.674928(1) \times 10^{-27}$
Spin	$\frac{1}{2}$
Magnetic moment ($J \cdot T^{-1}$)	$-9.6491783(18) \times 10^{-27}$
B-decay lifetime (s)	885.9 ± 0.9
Confinement radius (fm)	0.7
Quark structure	udd

Table 1.2 Neutron classification based on energy (Anderson 2007)

Type	Energy (meV)	Temperature (K)	Wavelength (nm)
Cold	0.1 – 10	1 – 120	0.4 – 3
Thermal	5 – 100	60 – 1000	0.1 – 0.4
Hot	100 – 500	1000 – 6000	0.04 – 0.1

information about the atomic structure and the dynamics of condensed matter.

Neutrons interact with matter through all four fundamental interactions (Dubbers 2003): the electromagnetic, weak nuclear, strong nuclear and gravitational interactions. While the electromagnetic interaction is primarily important in deep inelastic scattering and in magnetic interactions, the strong interaction is the most important force to neutrons as it plays the leading role when neutrons pass through matter. Unlike charged particles, a neutron goes on its way unchecked until it makes a head-on collision with an atomic nucleus.

Attenuation of neutron and neutron radiography

The scattering length of the neutron-nucleus system, which is of fundamental interest for both structure and dynamic investigations of condensed matter, nuclear research and other disciplines, is the basic quantity that describes the strength and the character of the interaction of low-energy neutrons with the individual nuclei. Due to the independence on the individual nuclei and atomic structures, the scattering length varies irregularly from one nucleus to another. Cross section is used to describe the likelihood of interaction between neutron and elements as an effective target size. It can therefore characterize the probability that a particular nuclear reaction will take place, or the statistical nature of scattering events. There are several types of cross sections, two of which are of principal interest to neutron radiography. Those are absorption cross section and the scattering cross section. The total cross section is the sum of the two (Von Der Hardt and Rottger 1981). The cross section is expressed as the unit of area, usually in

barn, which is 10^{-24} cm^2 . Cross section of elements vary with energy of the incident neutrons, and the higher the neutron energy, the lower the cross section.

The transmitted neutrons through a sample can be described by the rate at which the neutron intensity attenuates when it passes through the sample, as given by Equation (1.1) (Berger 1965)

$$\frac{d\Phi}{dx} = -\Phi \cdot \sigma \cdot N \quad (1.1)$$

where,

Φ – neutron intensity, the number of particles passing across unit area in unit time, $\text{n} \cdot \text{cm}^{-1} \cdot \text{s}^{-1}$;

x – thickness the neutron travels through, cm;

σ – target size, i.e., microscopic cross section, cm^2 ;

N – number of the target nuclei per unit volume, cm^{-3} ;

Integration of Equation (1.1) gives

$$\Phi = \Phi_0 e^{-\sigma \cdot N \cdot x} \quad (1.2)$$

where,

Φ_0 , Φ – neutron intensity, the number of particles before and after passing across unit area in unit time, $\text{n} \cdot \text{cm}^{-1} \cdot \text{s}^{-1}$;

σN – the total target size represented by a cubic centimeter of material, i.e., the macroscopic cross section, cm^{-1}

Neutron radiography is a powerful imaging technique for the internal evaluation of materials. Neutron intensity is attenuated through material, and the corresponding

spatial attenuation information is captured by various devices such as scintillator detectors, foil detectors, micro pattern gas counter, etc. (Oed 2003).

The interactions of neutrons and X-rays with matter are fundamentally different, which forms the basis of many unique applications using neutron radiography. X-rays interact with the electron clouds of atoms, and the cross sections for X-rays increase with atomic number. Neutrons interact with atomic nuclei, and the dependence on atomic number is not practically observed; the relationship between neutrons and atoms does not show any trend because each interaction between a neutron and an atom of a particular nuclide is unique. This fundamental difference enables neutron radiography to become a complementary technique to x-ray radiography, and in many cases the former considerably outperforms the latter. For x-ray radiography, it is very difficult to image low atomic number elements, such as hydrogen, carbon, oxygen, boron, or nitrogen. However, these elements are able to considerably attenuate neutron intensity, making themselves easily detected. Neutron radiography has the capability to reveal detailed components that are not visible in an x-ray image. Neutrons can penetrate many heavy materials such as titanium and lead. This allows for some unique applications, which is impossible with X-ray or Gamma-ray radiography. Thus, neutron radiography supplies a unique contrast mechanism not found with other imaging methods based on electrons, X-rays, or nuclear magnetic resonance. Elements having adjacent atomic numbers can have widely different absorptions of neutrons and it varies from element to element, even from isotope to isotope. Neutrons also provide high quality radiographs of highly radioactive components.

When neutrons encounter a matter, they are partly absorbed, partly scattered, and partly transmitted, as shown in Figure 1.1. Similar to an X-ray beam passing through a medium, neutron intensity follows the exponential attenuation law given by Equation (1.3).

$$I = I_0 e^{-\Sigma \cdot x} \quad (1.3)$$

where, Σ – the macroscopic cross section, cm^{-1} ;

x – the path length (distance the neutron beam travels), cm

I_0 – the intensity of incident neutron beam, i.e. the number of particles passing across unit area in unit time, $\text{n} \cdot \text{cm}^{-2} \cdot \text{sec}^{-1}$

I – the intensity of neutron beam passing through a distance x in the material, $\text{n} \cdot \text{cm}^{-2} \cdot \text{sec}^{-1}$

For a substance containing more than one element, the total macroscopic cross section is determined by the addition of its constituent elements. The attenuation coefficients of thermal neutrons are quite different for different elements as shown in Figure 1.2. The X-rays increase in attenuation with higher atomic numbers, but the neutrons are element dependent with no straightforward trend. With respect to neutron attenuation coefficients, hydrogen is about three orders of magnitude larger than aluminum, and oxygen about one order of magnitude larger than aluminum. Therefore, the contrast mechanism can be established for a neutron radiography image by the darker area that represents the greater attenuation and the lighter area representing the lower attenuation. Neutron radiography images can be produced with contrast set by different attenuation due to different elements.

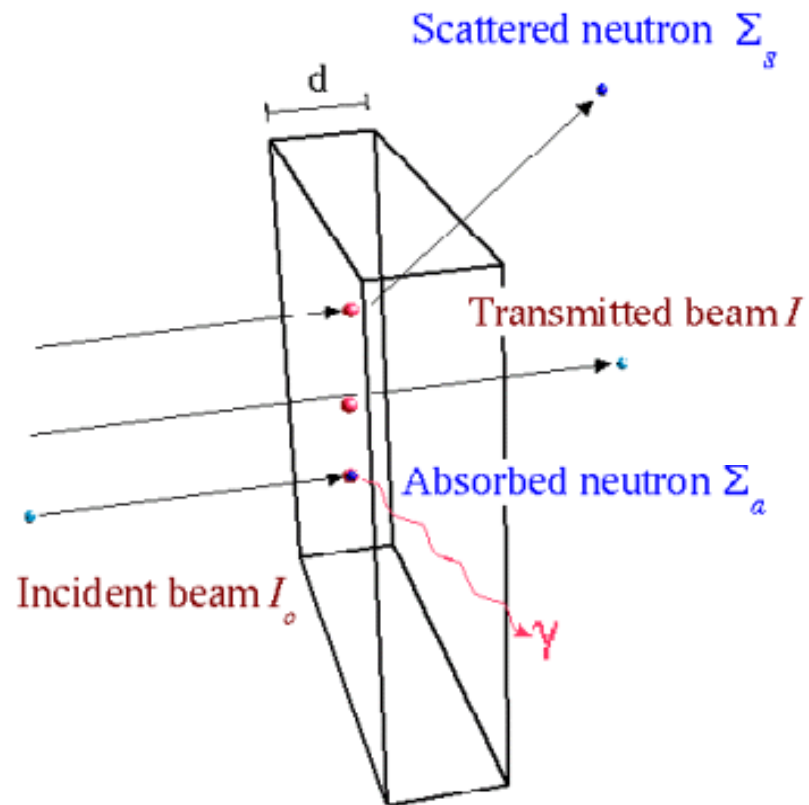


Figure 1.1 Interaction of neutrons with matter

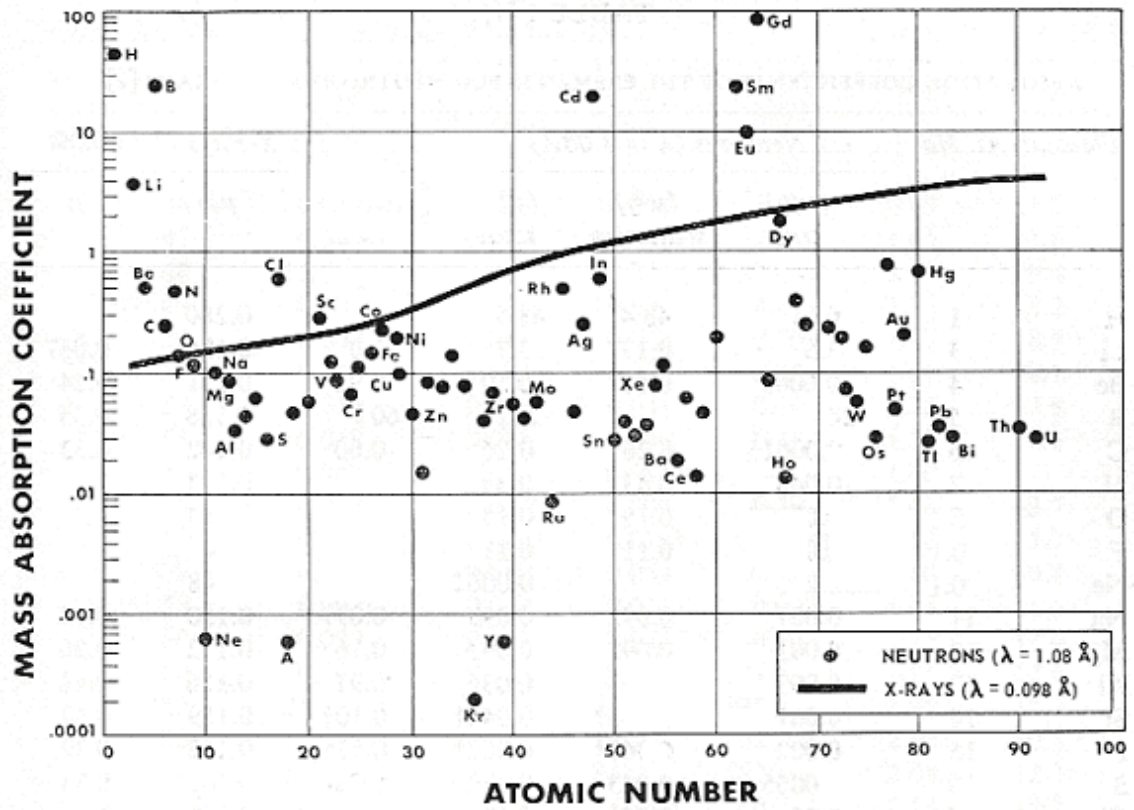


Figure 1.2 Attenuation of Neutrons and X-rays for different elements (Berger 1965)

Bragg's Law and neutron scattering

Diffraction based stress analysis techniques use the interplanar d-spacing, i.e., the distance between certain atomic planes, as an internal strain gage based on the Bragg's law. Elastic neutron scattering and Bragg's law are illustrated in Figure 1.3. Bragg's law can be described as that Bragg peaks in the intensity distribution occur when the scattering vector $\mathbf{Q}$ (the change in the wave vector of the radiation on scattering) coincides with a crystal reciprocal lattice vector $\mathbf{G}$.

$$\mathbf{Q} = \mathbf{G} \quad (1.4)$$

where,

$\mathbf{Q}$ – Scattering vector; for elastic scattering, $|\mathbf{Q}| = \mathbf{k}_0 - \mathbf{k}_i = 2\sin(\theta)/\lambda$

$\mathbf{G}$ – Reciprocal lattice vector for the given crystal in the reciprocal space

λ – Wavelength of the radiation

$\mathbf{k}_i, \mathbf{k}_0$ – Incident and scattered wave vectors; for elastic scattering $|\mathbf{k}_0| = |\mathbf{k}_i| = 1/\lambda$

θ – Angle of Bragg's peak

Reciprocal space is a derivative based upon the real space in which a three-dimensional array of lattice point of a crystal is defined. Reciprocal lattice is a very powerful description of all lattice planes in a three dimensional crystal. Reciprocal lattice and Ewald sphere construction are usually used to determine diffracted beam direction for a given crystal structure. Three vectors $\mathbf{b}_i$ ($i=1, 2, 3$) form a basis of this reciprocal space, and their relation to the real space lattice parameter $\mathbf{a}_i$ is given in Equations (1.5)-(1.7) (Cullity and Stock 2001).

$$\mathbf{b}_1 = \frac{\mathbf{a}_2 \times \mathbf{a}_3}{\mathbf{a}_1 \cdot \mathbf{a}_2 \times \mathbf{a}_3} \quad (1.5)$$

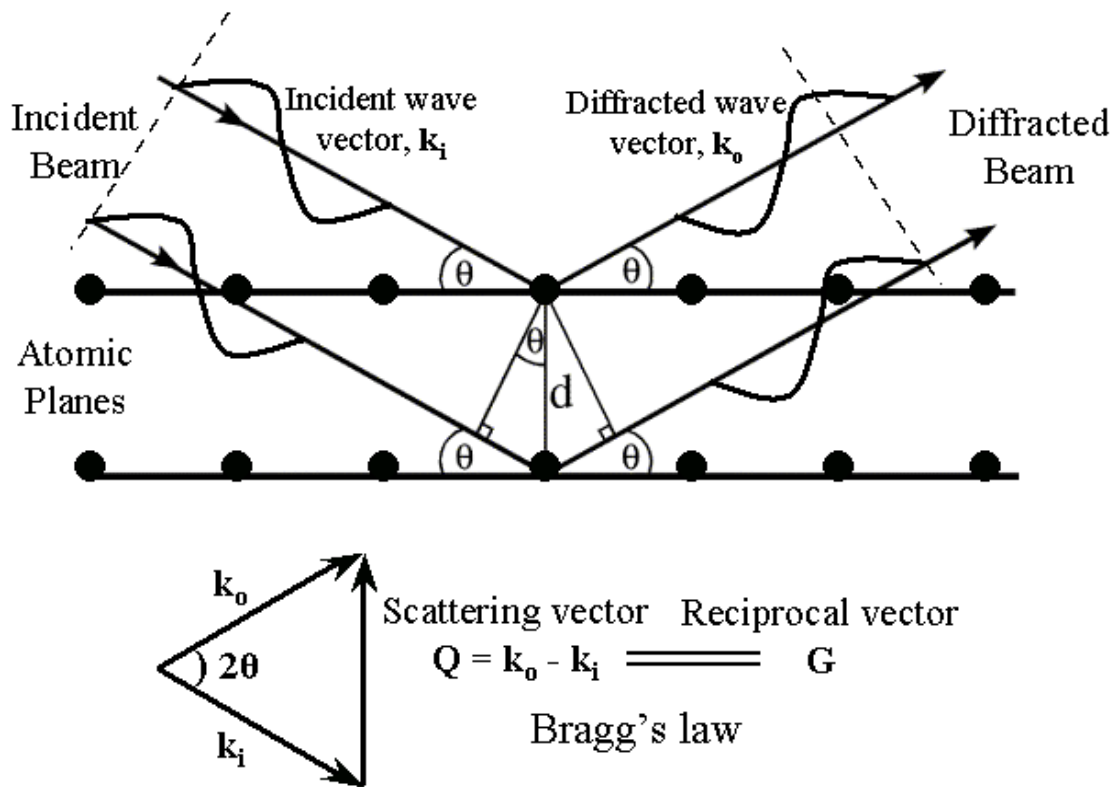


Figure 1.3 Elastic neutron scattering and Bragg's law

$$\mathbf{b}_2 = \frac{\mathbf{a}_3 \times \mathbf{a}_1}{\mathbf{a}_1 \cdot \mathbf{a}_2 \times \mathbf{a}_3} \quad (1.6)$$

$$\mathbf{b}_3 = \frac{\mathbf{a}_1 \times \mathbf{a}_2}{\mathbf{a}_1 \cdot \mathbf{a}_2 \times \mathbf{a}_3} \quad (1.7)$$

where, $\mathbf{a}_1, \mathbf{a}_2, \mathbf{a}_3$ – The unit vectors describing the lattice in the real space

$\mathbf{b}_1, \mathbf{b}_2, \mathbf{b}_3$ – The reciprocal unit vectors in the reciprocal space

Orientation and distance between parallel atomic planes are given by the Miller indices (hkl), which form the coordinates of the corresponding reciprocal lattice vector $\mathbf{G}_{\text{hkl}}$, as given in Equation (1.8).

$$\mathbf{G}_{\text{hkl}} = h \cdot \mathbf{b}_1 + k \cdot \mathbf{b}_2 + l \cdot \mathbf{b}_3 \quad (1.8)$$

In a crystal structure, $\mathbf{G}_{\text{hkl}}$ is normal to the (hkl) plane and has a length inversely proportional to the interplanar spacing of the planes, as shown in Equation (1.9).

$$|\mathbf{G}_{\text{hkl}}| = \frac{1}{d_{\text{hkl}}} \quad (1.9)$$

where, d_{hkl} is the interplanar d-spacing of the lattice plane (hkl)

Alternatively, for a given reflection (hkl) the lattice d-spacing can be determined by Bragg's law as given in Equation (1.10).

$$\lambda = 2d_{\text{hkl}} \sin(\theta_{\text{hkl}}) \quad (1.10)$$

where, θ_{hkl} is the Bragg's angle of the reflection (hkl)

Research tasks and objectives

This study intends to utilize advanced neutron techniques of radiography and diffraction to investigate fundamental issues in the field of infrastructure materials,

including both particulate and continuous materials with implication to engineering. The major tasks and significance of this study are summarized as following.

In Chapter 2, visualization and analyses by neutron radiography aims to develop a fundamental understanding of key issues governing the science in the Lost Foam Casting (LFC) process, which is difficult to reveal using traditional X-ray radiography. The proposed approach will prove to be a potentially powerful in-situ monitoring and visualization system for both quality control and improvement for LFC techniques. Extension of the proposed method for studying multi-phase flow through porous medium will be addressed.

In Chapter 3, experimental and analytical approaches have been developed for the first time providing a fundamental understanding of the globally external load-deformation behavior at the specimen boundaries, and the corresponding local force-chain elastic response under one-dimensional compression. The differences between the global strain and local lattice strain with stress were measured. Experimental data from this study will also have an implication for calibrating new computational methods that are non-continuum, such as discrete element method for particulate medium. This study will also be useful for many broad applications including fundamental understanding of sintered ceramics and metals.

In Chapter 4, residual strain mapping for tubular steel specimens in three orthogonal directions using reactor neutron source from High Flux Isotope Reactor (HFIR), Oak Ridge National Laboratory (ORNL) is presented. Torsion provides a unique opportunity to probe mechanical behavior of materials under pure shear state, and in combination with axial load provides a mechanism to rotate principal stresses in a

controlled fashion. This study is the first of its kind for evaluating residual strains in generalized loading conditions and also will help to demonstrate the need for having a multi-axial (axial and torsional) loading system for the anticipated engineering stress testing facility at Spallation Neutron Source (SNS).

In Chapter 5, residual strains in the carbon steel tubes, which were subject to a similar ultimate loading conditions in terms of deviatoric strain through either tension or torsion, were investigated using the pulsed neutron source facility – Spectrometer for Materials Research at Temperature and Stress (SMARTS) at Los Alamos National Laboratory. The residual strains based on the multiple (hkl) reflections, reported either strongly or weakly affected by the intergranular stress are discussed for both the tension and torsion samples. Effect of loading path (tension or torsion) on the residual strain will also be discussed.

In Chapter 6, the state of residual strain of the steel tubular samples which were subject to either tension or torsion with a similar ultimate loading conditions in terms of the deviatoric strain, were investigated using reactor neutron source are discussed. An approach using Least Squares fitting to obtain the tensor of residual strains based on six or over six independent measurements of lattice strains is presented. Error propagation of the strain tensor calculation based on the errors from both the measured lattice strains and the measured data in the unstressed reference sample will be analyzed. It also demonstrates that the study has helped the scientists from Oak Ridge National Laboratory to expand and establish an innovative research facility to measure strain/stress tensors at the 2nd generation Neutron Residual Stress Mapping Facility (NRSF2).

In Chapter 7, summary of the research and future work is provided.

References

- Anderson, I. (2007). Neutrons 101 - Basics on Production, Overview of Uses, and Summary of North American Availability. ASM International Oak Ridge Chapter Educational Symposium on Neutrons for Materials Science and Engineering, Oak Ridge, TN, Oak Ridge National Laboratory.
- Berger, H. (1965). Neutron Radiography Methods, Capabilities, and Applications. New York, Elsevier Publishing Company.
- Bourke, M. A. M., J. A. Goldstone, et al. (1992). Residual Stress Measurement Using the Pulsed Neutron Source at LANSCE. Measurement of Residual and Applied Stress Using Neutron Diffraction. M. T. Hutchings and D. Krawitz. Dordrecht, Kluwer Academic Publishers: 369-382.
- Cullity, B. D. and S. R. Stock (2001). Elements of X-Ray Diffraction. Upper Saddle River, Prentice Hall.
- Dubbers, D. (2003). The Neutron and Its Fundamental Interactions. Neutron Data Booklet. A.-J. Dianoux and G. Lander. Grenoble, France, Institut Laue-Langevin: 1.2-1 - 1.2-4.
- Hull, S., W. I. F. David, et al. (1992). Stress Measurement: Experience at ISIS. Measurement of Residual and Applied Stress Using Neutron Diffraction. M. T. Hutchings and D. Krawitz. Dordrecht, Kluwer Academic Publishers: 383-388.
- Hutchings, M. T., P. J. Withers, et al. (2005). Introduction to the characterization of residual stress by neutron diffraction. Boca Raton, CRC Press.

- Richardson, J. (1992). Residual and Applied Stress Measurements at IPNS. Measurement of Residual and Applied Stress Using Neutron Diffraction. M. T. Hutchings and D. Krawitz. Dordrecht, Kluwer Academic Publishers: 363-368.
- Oed, A. (2003). Detectors for Thermal Neutrons. Neutron Data Booklet. A.-J. Dianoux and G. Lander. Grenoble, France, Institut Laue-Langevin: 3.3-1 - 3.3-15.
- Rauch, H. and W. Waschkowski (2003). Neutron Scattering Lengths. Neutron Data Booklet. A.-J. Dianoux and G. Lander. Grenoble, France, Institut Laue-Langevin: 1.1-1 - 1.1-17.
- Von Der Hardt, P. and H. Rottger (1981). Neutron Radiography Handbook. Boston, D. Reidel Pub. Co.

Chapter 2 Visualization of the Multiphase Flow in the Lost Foam Casting Process by Neutron Radiography and Image Processing

This chapter is revised based on an invited paper submitted to Materials Science and Technology 2007 Conference and Exhibition:

X. Luo, D. Penumadu, Visualization of the Multiphase Flow in the Lost Foam Casting Process by Neutron Radiography and Image Processing, submitted to Materials Science and Technology 2007 Conference and Exhibition, Sept 16-20, Detroit, 2007

My primary contributions to this paper include: (i) development of the problem into a work, (ii) identification of the study areas, (iii) most of the gathering and reviewing of literature, (iv) sampling, processing, and analyzing neutron images from neutron radiography videos, (v) pulling various contributions into a single paper, (vi) most of the writing.

Abstract

An advanced experimental and analytical approach by applying the neutron radiography combined with imaging processing was developed to study the Lost Foam Casting (LFC) process. The background of the LFC, the research facility of neutron radiography, and the image processing techniques used in the analysis of the neutron images are introduced first. Experiments were conducted by using refractory coated Expanded Polystyrene foam patterns that have simple geometry with different gating and Aluminum alloy materials. Real-time neutron videos were recorded for the entire LFC process that includes material phase transitions. Still neutron images were extracted from the neutron videos, subsequently processed and rendered by image processing. Based on the results from neutron radiography combined with the image processing and analysis,

the multiphase flow and pyrolysis front were identified and isolated from the molten metal as well as the undecomposed polymer foam.

Behavior and the characteristics of the molten metal and the pyrolysis front in the LFC processes were also revealed. The proposed methodology and technique in this study help develop an understanding of fundamental issues governing the science in LFC process that is difficult to be revealed by conventional X-ray radiography. The proposed approach will prove to be a powerful in-situ monitoring and visualization system for both quality control and improvement for metal casting technique such as the LFC process.

Introduction to Lost Foam Casting

Casting is one of the oldest approaches of manufacturing dating back to 3000 B.C., and is often used because of its ability to produce large and durable volumes of complex shape castings with desirable features such as internal cavities and hollow sections. The Lost Foam Casting (LFC) also known as Expandable Polystyrene (EPS) Casting began its high production application in early 1980's (Lessiter and Kotzin 2003).

The LFC process has unique advantages when compared to competing technologies for metal casting. It has no size limit and the casting products from it can be obtained as near net shape, and complex shapes can be cast in one piece by using EPS foam patterns glued together. It is starting to be widely used as a replacement to the conventional casting processes such as green sand and investment castings, by many casting companies, especially in automobile industry. General Motors, Mercury Marine, Saturn, and Robinson Foundry have been applying the LFC for producing engine blocks and cylinder heads with complex shapes and internal pathways in order to improve

product quality. For example, General Motors had nine different products in the LFC products including aluminum cylinder head, aluminum cylinder block, aluminum driven sprocket supports for automatic transmission, iron crankshaft, iron differential case, and iron clutch housing for automatic transmission (Smith 1996). It has also been demonstrated that the LFC was capable of both high-quantity production and good quality by casting more than 5000 castings/day for General Motors (Foti 2000). Casting from this process can have surface finishes from 5-20 micrometer. Dimensional accuracy from this process is better than the traditional sand castings. After the LFC process, almost no further modification is required, the machining and assembly being eliminated or reduced (Campbell 2000). Unlike other casting processes, no parting lines, cores, or riser systems are needed. The process for the most part is inexpensive with the polystyrene, sand and containing units being relatively inexpensive. Shaping die could be costly, and low volume runs could be expensive. Compared with the conventional sand casting using resin-bonded sand, the LFC reduces environmental wastes because it uses unbonded and recycled sand. Bates (1999) gives a comparison of energy usage for both the LFC and the conventional sand casting, which shows that the LFC results in an energy savings of 27%, productivity increase of 46%, and material reduction of 7%. Research has also shown that the LFC could reduce energy use by as much as 27% compared with conventional sand casting (Dinwiddie 2004).

Fundamentals of the Lost Foam Casting process

Two major steps are involved in the LFC process. The first is to produce a polymeric foam pattern typically using expanded polystyrene. The second step is about

the process of casting, which is to replace the polymer pattern by molten metal. After the coated polymer pattern is ready, it is placed in a mould where silica sand is poured into to fill all the cavities. Molten metal is then introduced into the mould through the gating, and consequently the polymer foam pattern decomposes into gaseous and liquid pyrolysis products and escapes into the loose sand. The molten metal flow fills in the space originally occupied by the foam pattern to yield the target shape. The casting product is obtained by removing the sand and refractory coating. For the LFC process to be successful, there must be a high degree of process control both on raw materials including refractory coating, EPS foam patterns and the casting process itself. Several types of defects such as folds, porosity, and metal penetration have been observed to occur during the LFC process if the process control is not adequate.

In the LFC process, the EPS foam pattern degrades into liquid and gas products and escapes into the loose sand when the molten metal is introduced. During the process of metal filling, a gas layer that is also called the pyrolysis front mainly consisting of the thermal degradation products of styrene exists between the metal and the polymer fronts. Behavior of the pyrolysis front during casting process is important and has a significant impact on the quality of the casting products (Yao and Shivkumar (1997)). However, the pyrolysis front is very difficult to be identified using the traditional X-ray based radiography techniques, and consequently has not been studied in the past.

There are many factors that may have considerable impact on how well the LPC process works and affect the quality of the casting products. Mis-run is an incomplete fill of the cavity during metal flow filling process. Among other factors, this kind of defect occurs with increasing likelihood when the permeability of the coating is low enough to

prevent the transport of decomposed materials into the confining sand or mullite medium or when the thermal conductivity of the coating is such that abnormal solidification of molten metal front happens. Cold lap is a discontinuity on the casting surface resulted from the metal being too cold to fuse where the two streams of metal meet. Porosity often occurs if the metal flow solidifies before the decomposed materials escape through the coating. Metal penetration defect happens when coating is not strong enough to resist the pressure caused by compaction and thermal expansion during casting. Cracks will appear in the coating, causing the molten metal to break through the cracks in the coating and enter the surrounding sand (Goria, G. et al. 1986; Littleton, Miller et al. 1997; Liu, Ramsay et al. 1998).

Neutron radiography for Lost Foam Casting

Radiography has long been considered as a valuable tool for detecting internal features and flaws. With X-ray radiography, shrinks, blowholes, gas or slag inclusions and other flaws could be identified quickly and gating and risering appropriately adjusted (Turner 1972). The traditional radiography by using X-ray source has also been successfully used in practice to image aluminum flow for aiding gating design, turbulence reduction and determining flow patterns (Sirrell, Holliday et al. 1995; Cox, Harding et al. 2003). However, the X-ray radiography has difficulty in visualizing the polymer foam and the pyrolysis products due to its attenuation issue. It is crucial to characterize the behavior of pyrolysis fronts of polymer foam patterns when they recede during the LFC process. Then other issues involved in the LFC, such as the effect of the properties of the refractory coatings and unbonded granular materials on the filling

behavior of molten metal flows, are able to be investigated. Moreover, an in-situ quality control approach for the LFC can also be developed based on the neutron radiography technique.

Two types of important materials involved in the LFC are aluminum and EPS foam consisting of carbon, hydrogen and oxygen. X-ray radiography will be valuable to study the metal front, and the imaging technique as proposed in this research can be useful to study the degradation process of foam. In particular, neutrons are able to penetrate aluminum and yet are highly sensitive to hydro-carbons. Thus, neutron offers the potential to visualize degradation products from expanded polystyrene during the metal fill process.

Transmission of a neutron through a substance can be expressed in terms of the rate at which the neutron intensity reduces as it passes through the substance. Although neutrons, especially those traveling at relatively low velocities (thermal neutrons) are absorbed in matter according to laws that are very different from those governing the absorption of electromagnetic rays such as x- or gamma-rays, they actually follow a similar exponential attenuation law. To calculate cross section of compounds, an important assumption needs to be made, which is that the property of the nuclear species is unaffected by considerations of the molecular or crystal structure in which it resides. Thus, the macroscopic cross section for compounds can be calculated from the summation of the macroscopic cross sections of each nuclear species, as given in Equation (2.1).

$$\Sigma_c = \sum_i N_i \sigma_i \quad (2.1)$$

where,

Σ_c – the macroscopic cross section of compound, cm^{-1}

N_i – the number of nuclei per unit volume for i^{th} nuclear species, cm^{-3}

σ_i – microscopic cross section for i^{th} nuclear species, cm^2

The microscopic cross section σ_i is an indication of the probability of a specified interaction or reaction between an incident radiation and a target particle or system of particles. It is the reaction rate per target particle for a specified process divided by the flux of the incident radiation. Macroscopic cross-section is the cross-section per unit volume of a given material for a specified process. For a pure nuclide, it is the product of the microscopic cross-section and the number of target nuclei per unit volume; for a mixture of nuclides, it is the sum of such products.

Obviously,

$$N = \frac{\rho \cdot n_A}{M} \quad (2.2)$$

where,

ρ – density of compound, $\text{g}\cdot\text{cm}^{-3}$

n_A – Avogadro's number, $6.02214 \times 10^{23} \text{ mol}^{-1}$

M – atomic weight of compound, $\text{g}\cdot\text{mol}^{-1}$

Substitute Equation (2.2) into (2.1) to yield

$$\Sigma_c = \rho \cdot \frac{n_A}{M} \sum k_i \sigma_i \quad (2.3)$$

where,

k_i – the number of i^{th} nuclear species in the compound molecular

Equation (2.3) is the formula used to calculate the attenuation capability of a compound. Based on that, the macroscopic cross-section and the calculated thickness for 50% neutron attenuation of the typical materials involved in LFC process can be

calculated and listed in Table 2.1. It shows that there is remarkable difference in the attenuation between aluminum and styrene, which creates the contrast mechanism in neutron radiography for LFC.

Table 2.1 Material properties and the calculated thickness for 50% attenuation

Material	Molecular / Atom	Density (g/cm ³)	Molecular /Atomic weight (g/mol)	Macroscopic cross section (cm ⁻¹)	Thickness of 50% attenuation (cm)
Aluminum	Al	2.70	27	0.0984	7.05
Styrene	C ₈ H ₈	0.9	106	3.12	0.22
Water	H ₂ O	1.0	18	2.62	0.26
Silica	SiO ₂	2.65	60	0.259	2.68

Extensive work has been done in LFC research in the past, yet some fundamental issues demand more attention. One of the important aspects is to identify the molten metal and pyrolysis fronts during the LFC process, because the behavior of those fronts has an important effect on the quality of castings. Results have shown that typical defects are closely related to the behavior of the decomposed polymer foam and the complex property of the pyrolysis front during the LFC process (Goria, G. et al. 1986; Littleton, Miller et al. 1997; Liu, Ramsay et al. 1998). Yao and Shivkumar (1997) indicate that the filling behavior of the molten metal flow is essentially governed by the polymeric degradation process occurring at the metal front during the LFC process. Penumadu (2002) observed the presence of receding pyrolysis front and fast diffusion pathways in EPS foam during LFC process by using neutron radiography.

This research aims to take advantage of advanced and innovative technology provided by neutron radiography, through which the molten metal and pyrolysis fronts during the LFC process can be visualized so that the fundamental understanding of their behavior and its effect on the LFC process can be established.

Neutron facility for studying the Lost Foam Casting

Neutron sources used in neutron radiography can be either from continuous reactor sources like the TRIGA Reactor at McClellan Nuclear Radiation Center (MNRC), managed by University of California, Davis, or the High-Flux Isotope Reactor at Oak Ridge National Laboratory (ORNL), or from pulsed source like the new Spallation Neutron Source at ORNL. Each source offers special capabilities. For example, the pulsed source could permit high-speed stroboscopic imaging with 1 microsecond time

resolution. The reactor source could enable the best spatial resolution, especially with advanced neutron optics and new neutron detectors.

The neutron radiography experiments in this research were carried out at the research facility located in McClellan Nuclear Radiation Center (MNRC). As shown in Figure 2.1, MNRC has a two Megawatt TRIGA reactor with four neutron beam tubes all tangential to the reactor core. The neutron beam is approximately 9 inches (22.5 cm) in diameter and has an intensity of approximately $1 \times 10^7 \text{ n} \cdot \text{cm}^2 \cdot \text{s}^{-1}$. The beam angle is 20° from horizontal. The neutron spectrum is highly thermalized, which allows high quality imaging. The neutron beams are highly collimated with an L/D of 50 to 400, resulting in high spatial resolution from the digital still radiographs. The radiosopic systems use the CF Thompson tube as the neutron camera, which combined with the SIT (Silicon Intensified Tube) camera allows it to capture images at a rate of 30 frames/second. The radiography setup for LFC is shown in Figure 2.2. Charge Couple Device (CCD) based radiography was used in this study.

The polymer foams used in the study are expanded polystyrene (EPS) foams, with a density of 22.4 kg/m^3 . The casting metal used in this study is aluminum alloy. The aluminum alloy was heated to 1400 F before pouring into the EPS patterns. Real-time neutron radiography is conducted to visualize the molten metal and pyrolysis foam fronts during the whole LFC process. Geometries of all the casting samples are simple plates with the same size of 100 x 150 mm, various thicknesses (4, 12 and 24 mm), and various gating orientations (bottom, side and top), as shown in Figure 2.3.

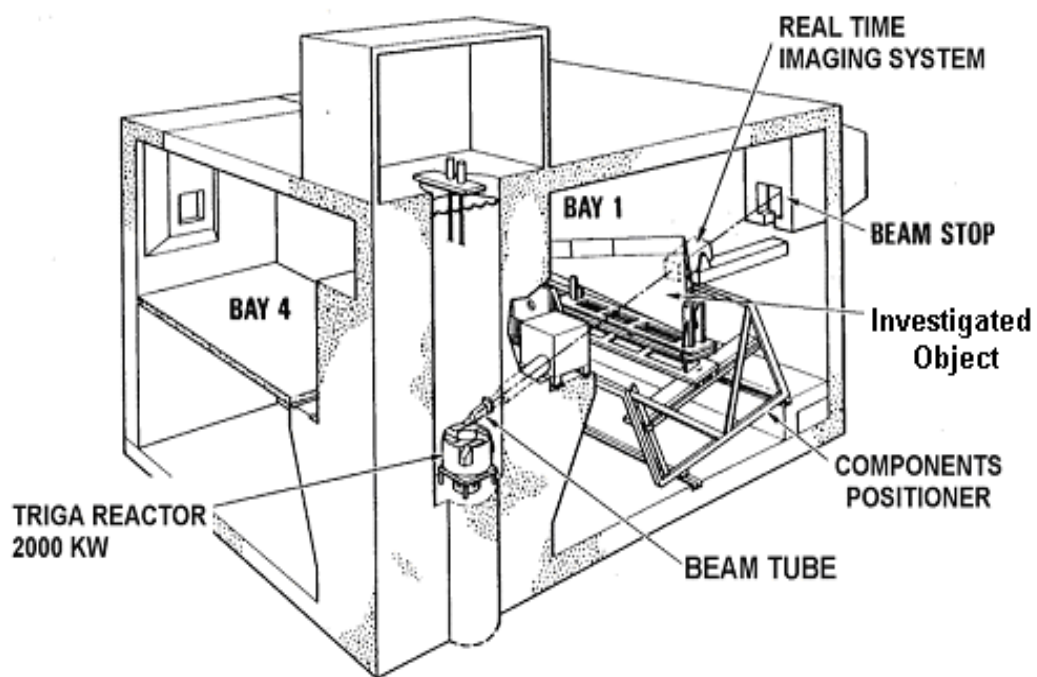


Figure 2.1 Neutron radiography facility at MNRC, UC Davis

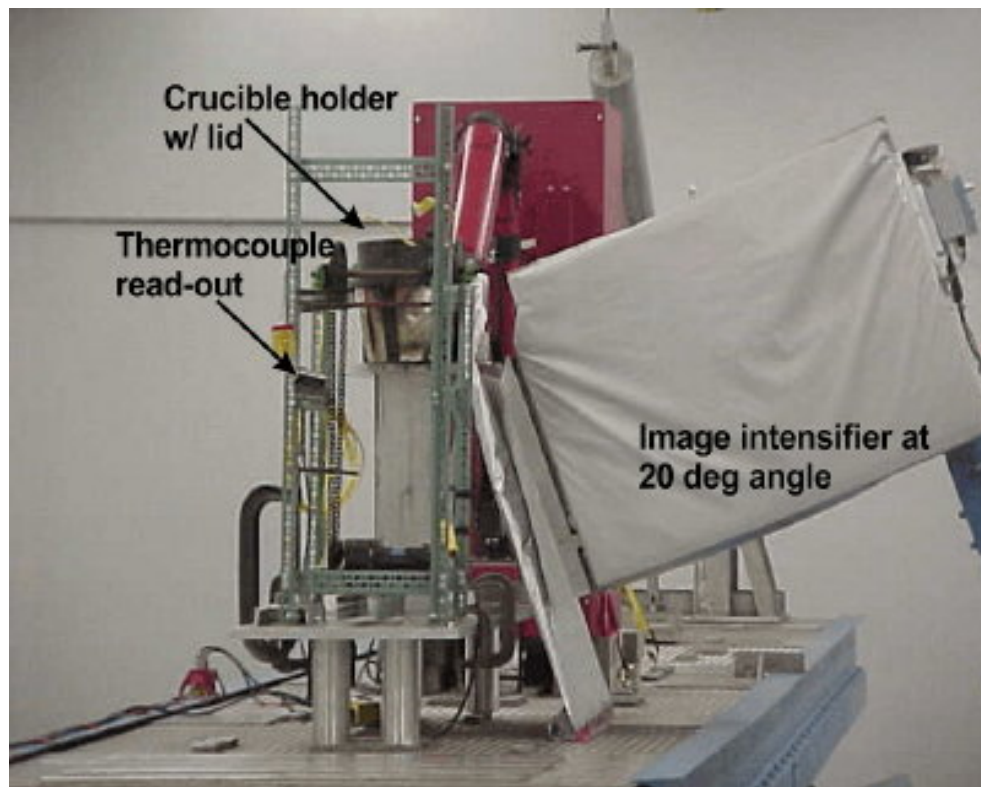


Figure 2.2 MNRC radiography setup to study LFC



(a) Top-gating



(b) Side-gating



(c) Bottom-gating

Figure 2.3 Three types of gating systems

Image processing for neutron images

A histogram measures and illustrates the characteristics of brightness and contrast of a digital image. In the histogram, the X-axis represents the intensity of a given pixel, and Y-axis represents the counts, i.e., the number of the pixels that possess that intensity in the image. As in 8-bit gray scale images, the X-axis in a histogram will represent the intensity range 0 through 255. Figure 2.4 shows a typical histogram, and the statistics of the pixel intensities are also provided.

The neutron images obtained originally from the real-time neutron videos are 24-bit RGB color images, which are also referred as true color image. It is well known that any color can be represented as a mixture of varying levels of three primary colors of light, i.e., red, green, and blue. Thus, the RGB image in which RGB stands for red, green and blue, respectively is the most straightforward way to represent color images by using those three primary colors of light. In a 24-bit RGB bitmap image, each pixel contains a 24-bit value called RGB triplet made of three separate 8-bit scales. Each scale represents the level of its respective color channel, i.e., red, green or blue. The brightness values represent levels with a 256-level scale, ranging from 0 (black) to 255 (brightest). Before the intensity analysis were performed, those RGB neutron images were transformed into 8-bit gray scale images by using the default procedure provided by the software package Image-Pro Plus 4.5 (Media Cybernetics 2001). Afterwards, those 8-bit gray scale images are processed with pseudo-color, which is used to visually amplify specific intensities that are difficult to distinguish from their surroundings by rendering those intensity values in different colors. The process of filtering of pixels is also used in order to choose

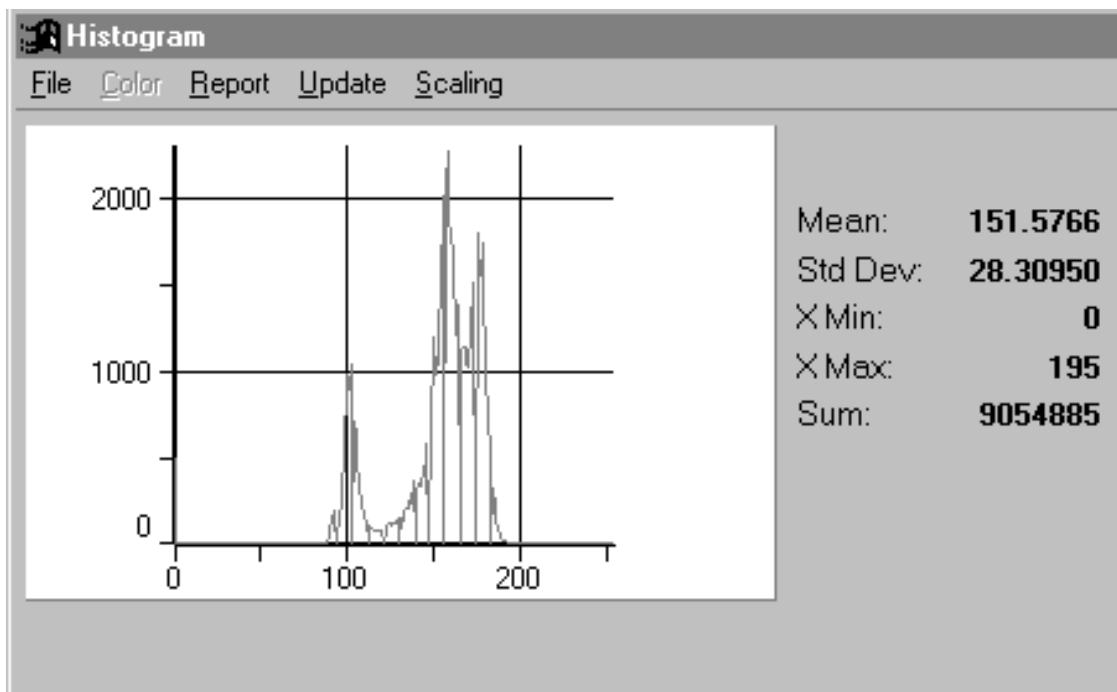


Figure 2.4 A typical histogram of a digital image

and distinguish the pixels of interest from their surrounding. In the processed still neutron images, the lower pixel intensity indicates the higher attenuation of neutrons at that location. For instance, in an 8-bit gray scale neutron image the pixel intensity of the area in the EPS foam pattern always has a lower value than that of the area in the molten metal flow.

Neutron radiography results and discussion

Real-time digital videos using the MNRC neutron radiography facility were recorded during the entire process of metal entry, foam decomposition, and casting solidification in the LFC process for all samples. Image of interest as a function of time were extracted from the digital videos of the LFC process. Then the extracted individual images were further processed, and analyses are performed to those extracted images by using the Image-Pro Plus V4.5 (Luo and Penumadu 2006).

Typical digital images extracted at different time from the neutron videos before the molten metal entered the EPS foam patterns are given in Figure 2.5 and Figure 2.6. Figure 2.5 gives a 24-bit color image showing the EPS foam pattern area before the metal flow was introduced. Figure 2.6 shows the same image after the original digital image was converted, and rendered with pseudo-color. As mentioned before, lower intensity pixels in the images are represented by colder colors such as blue, while higher intensity pixels are represented by warmer colors such as red. Figure 2.7 to Figure 2.9 give the histograms of the polymer foam area in the digital images from three typical LFC processes. These still neutron images are extracted at the time 0 and 3 before

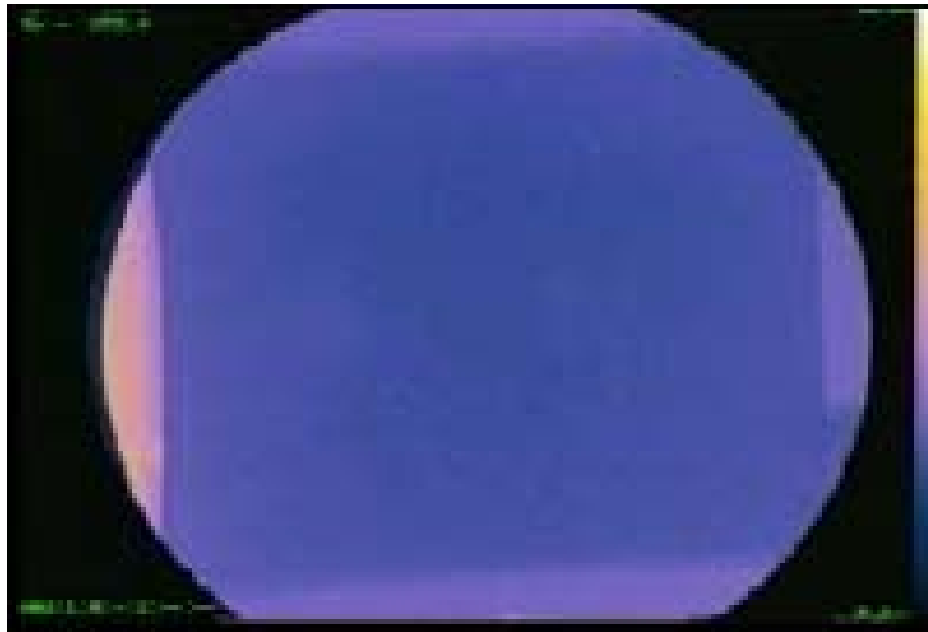


Figure 2.5 An original neutron image prior to LFC process

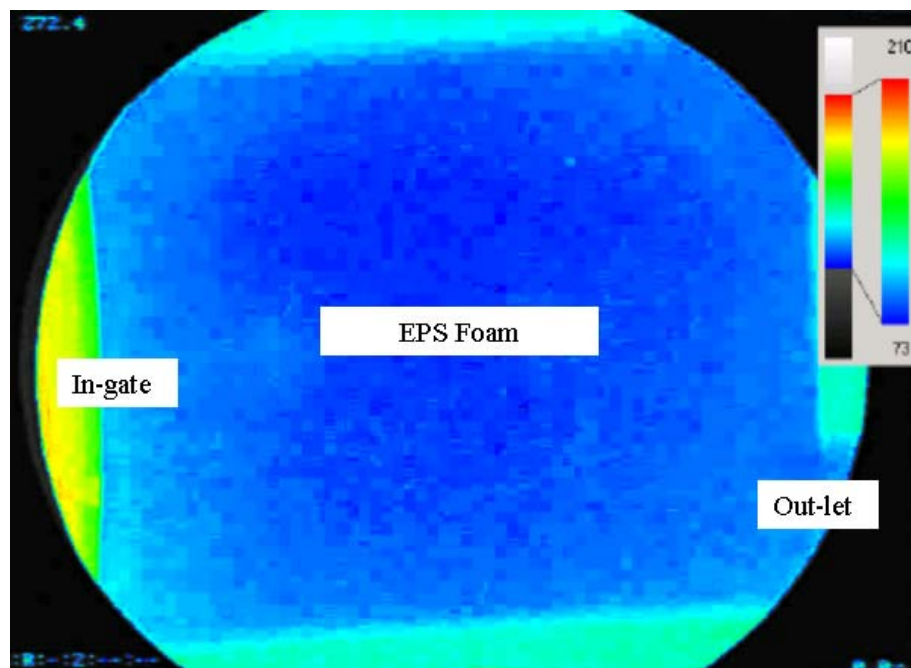
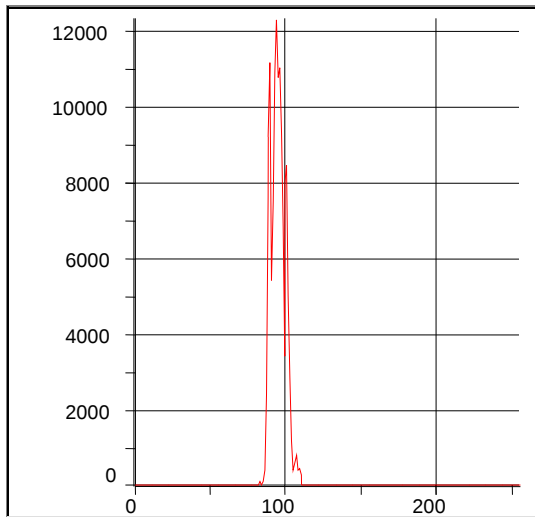
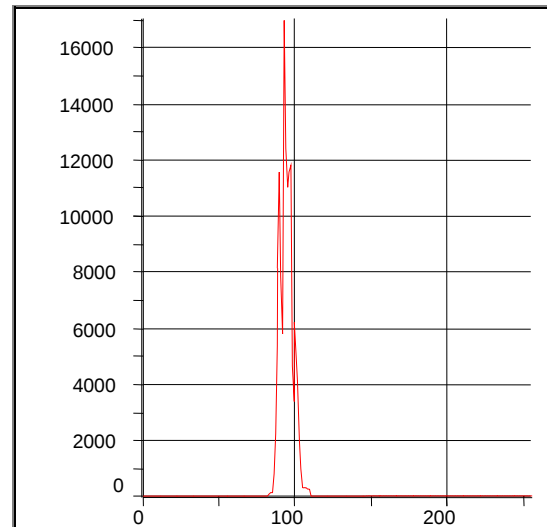


Figure 2.6 A converted pseudo-color image prior to LFC process

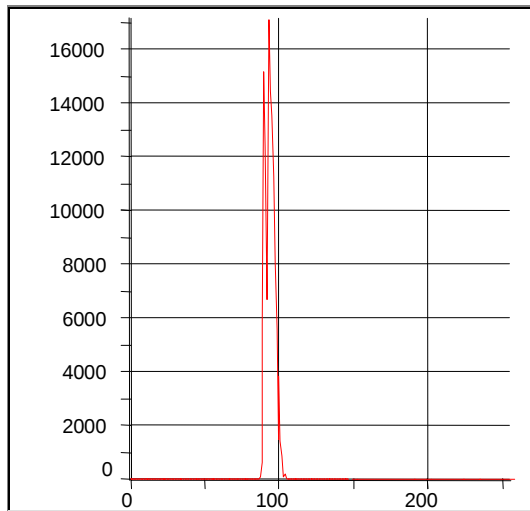


X Min: 83, X Max: 114
Mean: 94.95, Std Dev: 4.74
At 0 sec

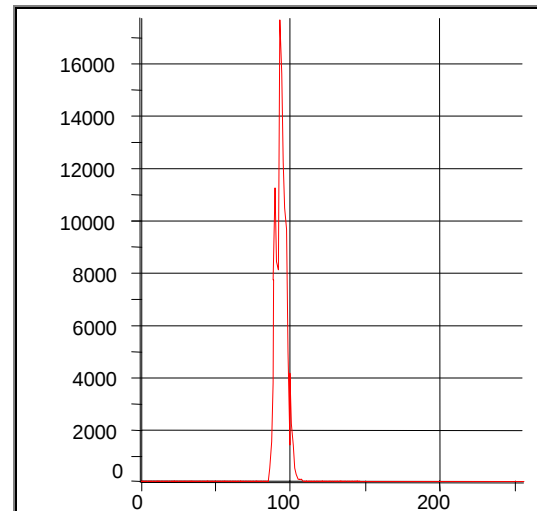


X Min: 82, X Max: 114
Mean: 94.47, Std Dev: 4.29
At 3 sec

Figure 2.7 Histograms of the EPS foam area (Side-gated, thickness 4 mm)

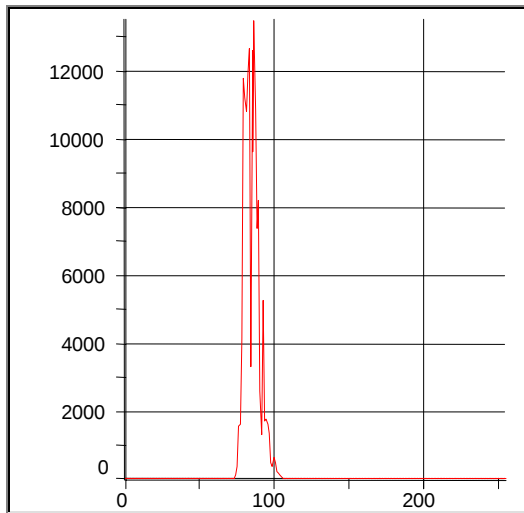


X Min: 87, X Max: 105
Mean: 93.76, Std Dev: 3.06
At 0 sec

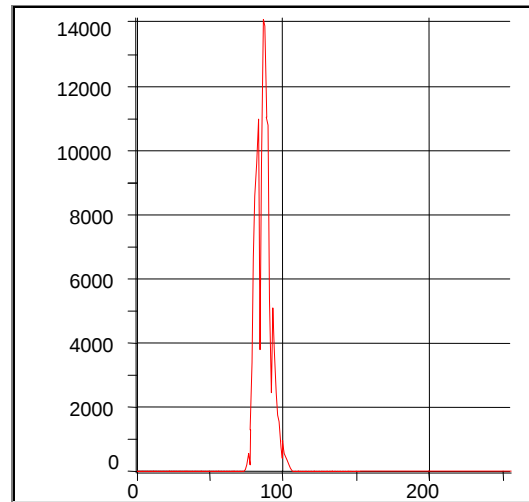


X Min: 80, X Max: 109
Mean: 93.75, Std Dev: 3.47
At 3 sec

Figure 2.8 Histograms of the EPS foam area (Side-gated, thickness 12 mm)



X Min: 70, X Max: 112
 Mean: 85.12, Std Dev: 5.08
 At 0 sec



X Min: 71, X Max: 111
 Mean: 86.83, Std Dev: 4.93
 At 3 sec

Figure 2.9 Histograms of the EPS foam area (Side-gated, thickness 24 mm)

the molten metal flows enter the EPS pattern area. The statistics of the pixel intensity are also given in the figures. Side-gating (location of metal entry) EPS foam patterns were used in those three LFC processes, and the thickness of the patterns varies from 4 to 24 mm, respectively.

It can be seen that the pixel intensity values are consistent with each other for a given polymer pattern before the high temperature molten metal flow enters the mold. The pixel intensity in the histograms is summarized in Figure 2.10, which shows that the pixel intensity values tend to decrease nonlinearly with increase in thickness of the EPS foam pattern. For the pattern thickness of 4 mm and 12 mm, the intensity values are around 93 and the difference between them is quite slight. For the thickness of 24 mm, lower intensity values are around 85.

Figure 2.11 and Figure 2.12 show the pseudo-color neutron images as well as the related photos of the casting samples that show mis-run during the LFC processes. It is of great interest that the defects of the casting samples can be detected by neutron radiography combined with the image processing and analysis. This can also be used as a powerful tool that is capable to capture what happens at the molten metal and pyrolysis fronts. Pertinent information from such analysis can be crucial to analyze the mechanisms leading to the occurrence of defects in metal castings and appropriate modifications required to make good castings to reduce scrap-rate and lead to significant economy and energy savings.

Figure 2.13 to Figure 2.16 give the pseudo-color neutron images extracted from

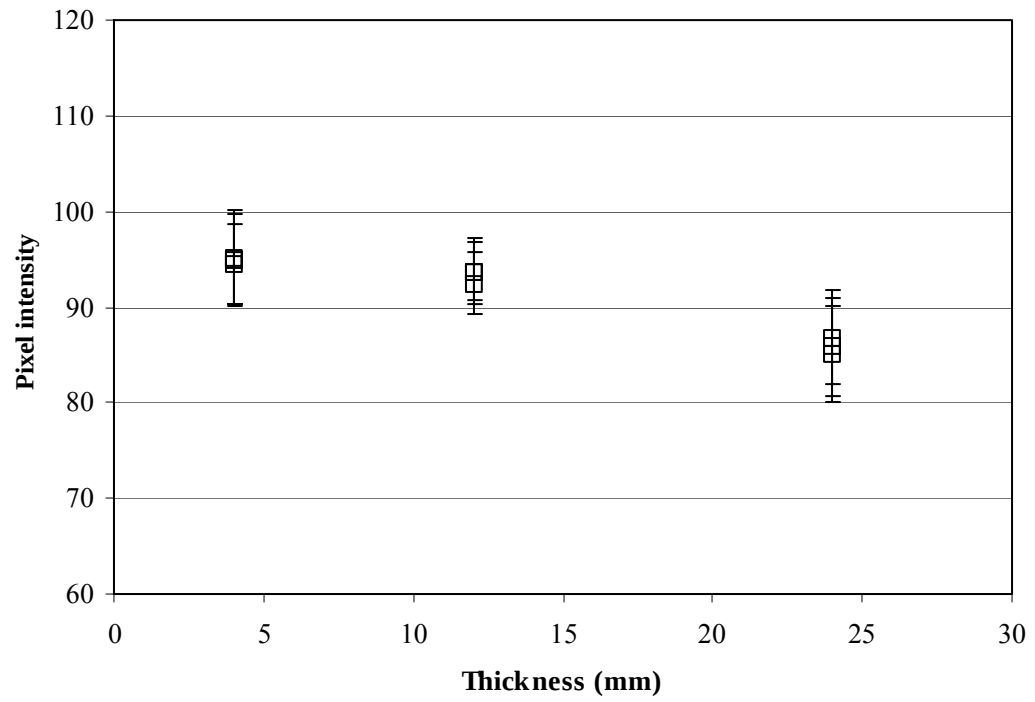
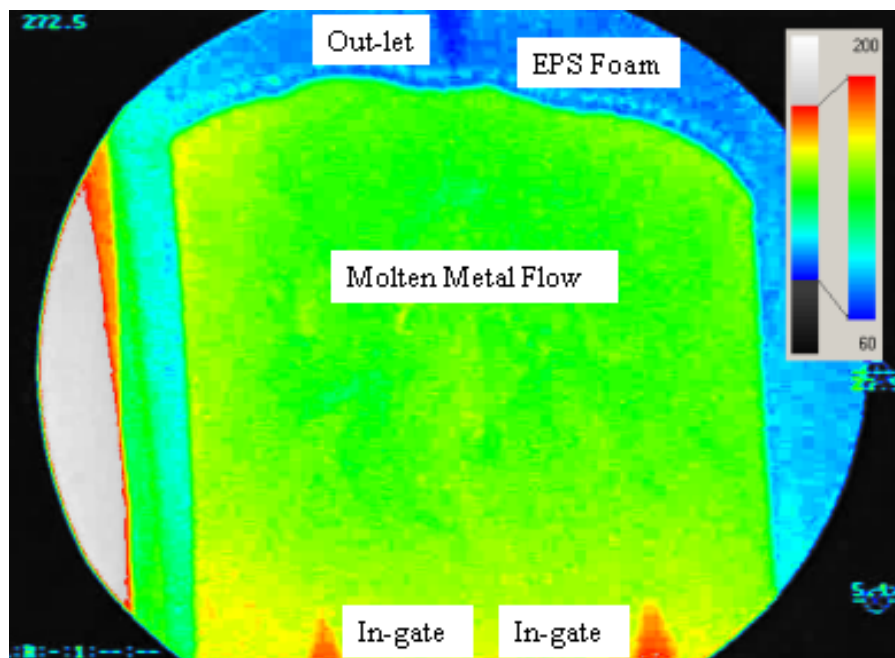


Figure 2.10 Pixel intensity of the foam pattern area (side-gated)

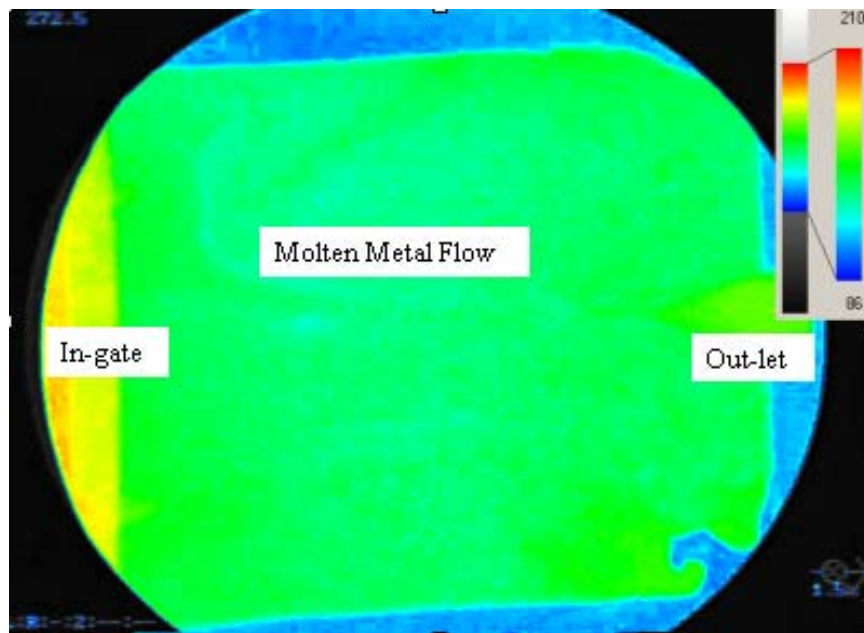


(a)

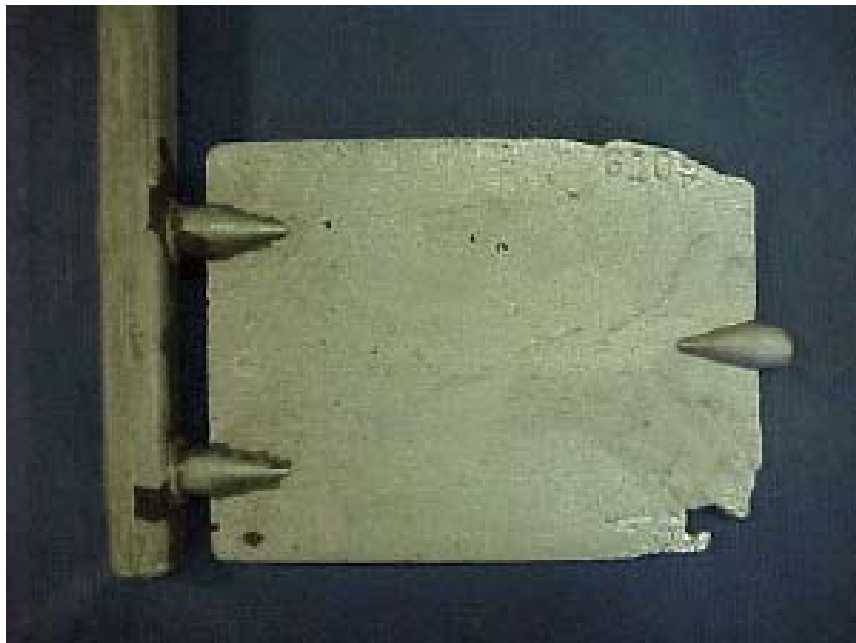


(b)

Figure 2.11 Bottom-gating casting with pattern thickness 4 mm (at 19 sec)



(a)



(b)

Figure 2.12 Side-gating casting with pattern thickness 4 mm (at 22 sec)

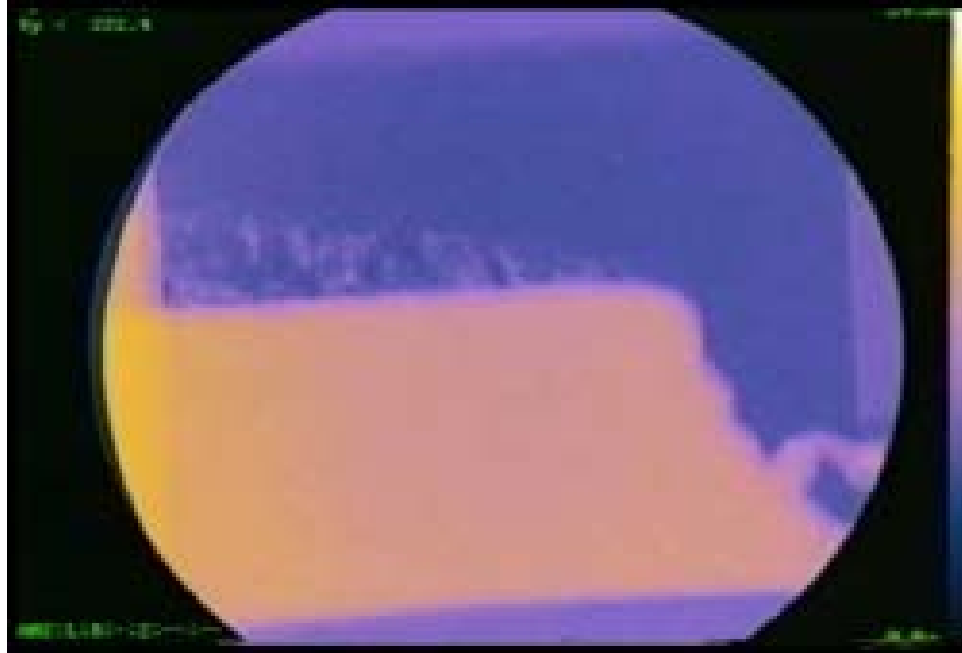


Figure 2.13 Neutron image of the side-gating with pattern thickness 24 mm (at 15 sec)

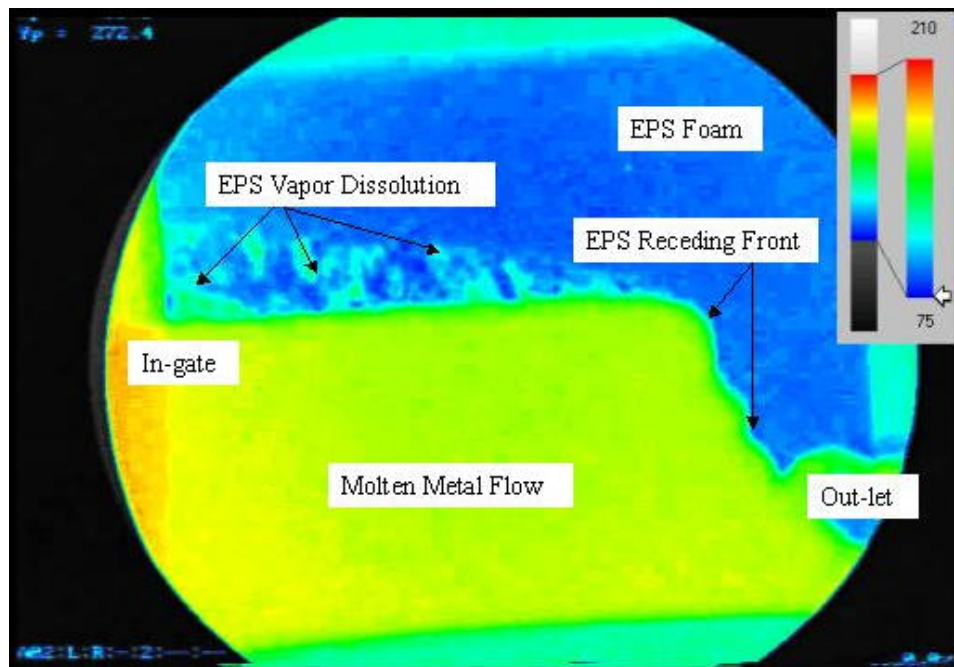


Figure 2.14 The Side-gating casting with pattern thickness 24 mm (at 15 sec)

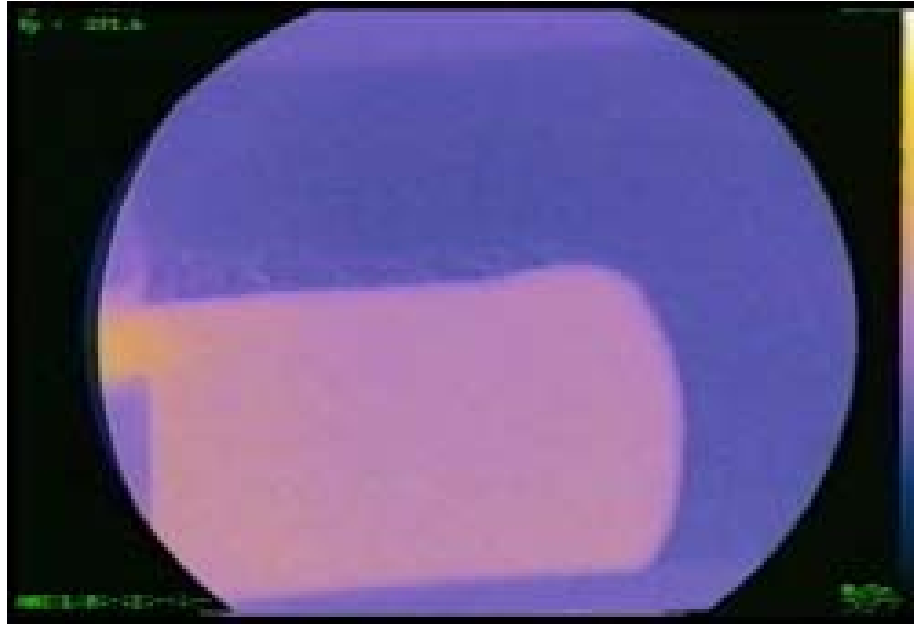


Figure 2.15 Neutron image of the side-gating with pattern thickness 12 mm (at 15 sec)

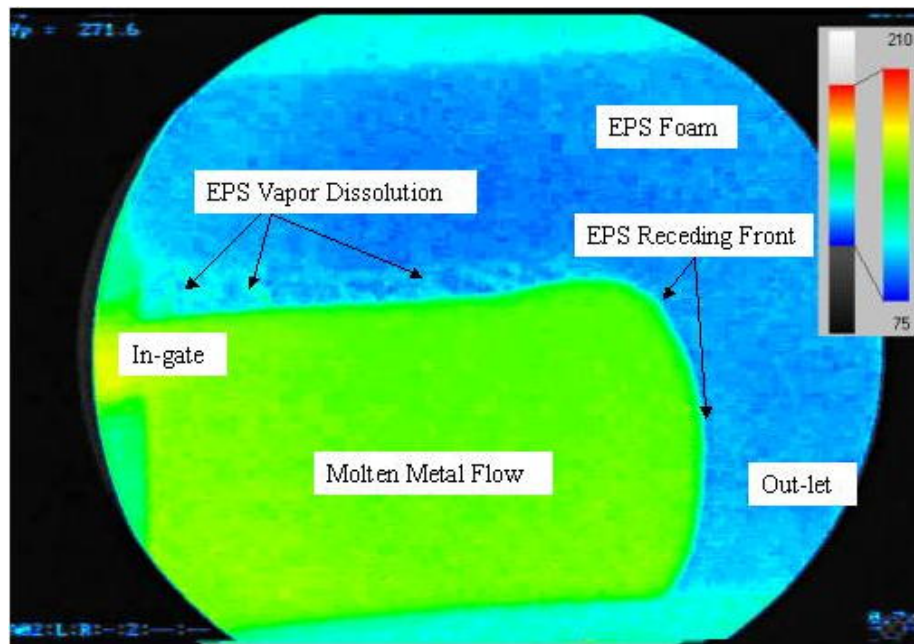


Figure 2.16 The Side-gating casting with pattern thickness 12 mm (at 15 sec)

the real-time neutron videos. Those images show the visualization at 15 seconds after the molten metal flow enters the EPS foam patterns with the thickness of 24, and 12 mm, respectively.

For the side-gating system, the casting process of 24 and 12 mm thick samples shows very similar filling characteristics in the neutron images. As the molten metal flows are entering the foam patterns and filling the mould, some irregular vapor-like flows can be clearly visualized and observed by neutron radiography along the horizontal edge of the high temperature metal front.

The pixel intensities of those irregular vapor-like flows in the images are almost the same as those of the pyrolysis fronts, the very thin interfacial zones between advancing molten metal flow and the receding polymer foam. This indicates those irregular flows have the same characteristics as the pyrolysis fronts. Thus, the irregular vapor-like flows are part of the pyrolysis fronts, also referred as volatilized pyrolysis products. These high diffusion pathways are believed to be styrene monomer that polystyrene dissolves as they try to advance in various directions from the degradation front close to liquid metal front. These neutron radiography images are valuable to quantify the zone of dynamic pyrolysis front that is essential for modeling the complete process realistically in a computational fluid dynamics type approach or to establish phenomenological models.

Figure 2.17 and Figure 2.18 show other pseudo-color neutron images at 7 seconds after the molten metal flow enters the EPS foam pattern. The thickness of the foam pattern used in the LFC process is only 4 mm. This image shows much difference

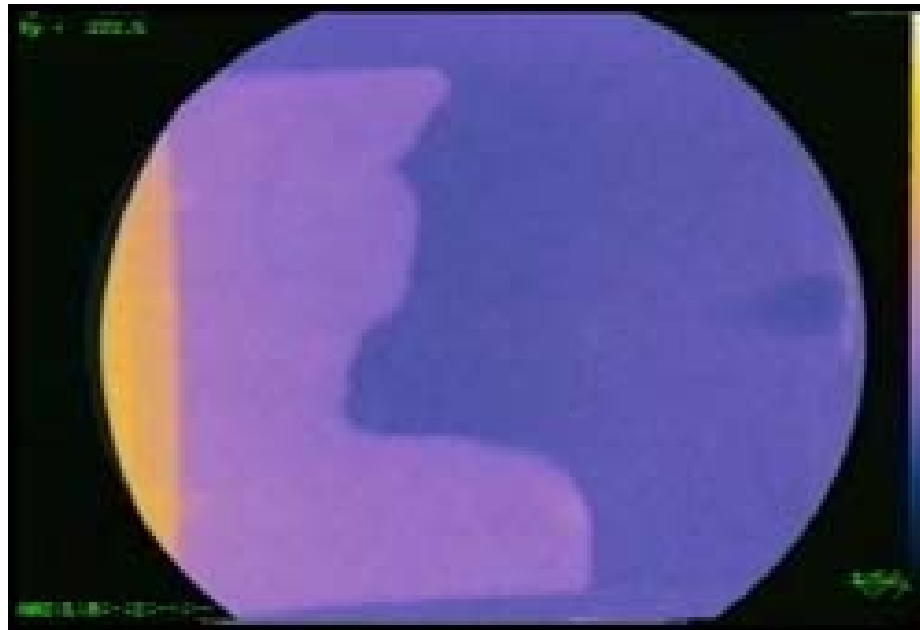


Figure 2.17 Neutron image of the side-gating with pattern thickness 4 mm (at 7 sec)

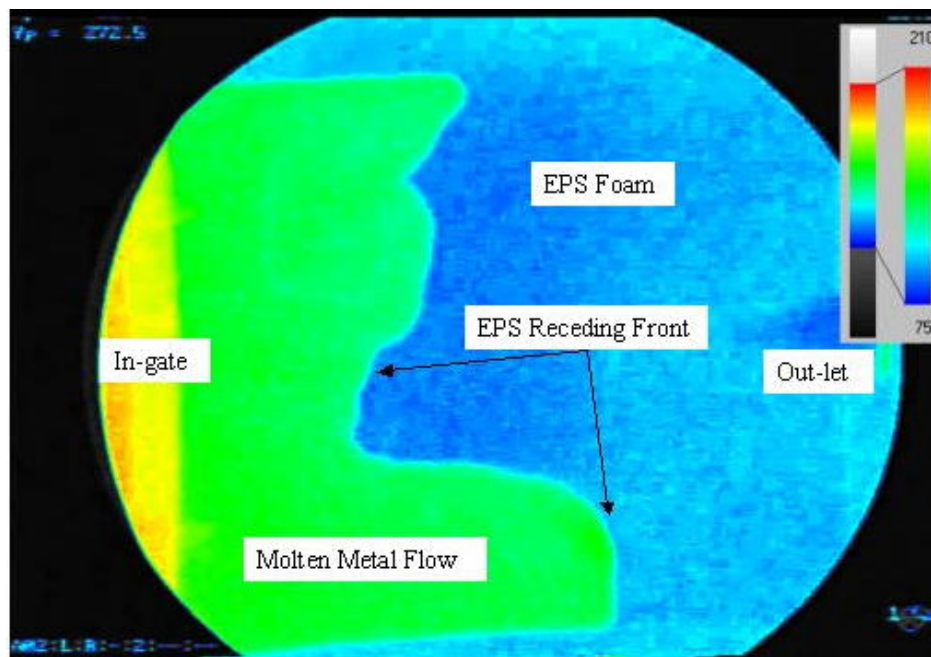


Figure 2.18 The Side-gating casting with pattern thickness 4 mm (at 7 sec)

from those with 24 and 12 mm foam patterns. Although both the advancing molten metal flow and the receding polymer foam can be clearly seen in the image, the irregular volatilized flows were not be observed during the LFC process. This demonstrates the complexity of degradation process and the unique advantage of neutron radiography to obtain fundamental science associated with EPS foam degradation with molten metal contact that is essential for improved understanding of this casting technique.

Based on the results from the image analyses, the irregular volatilized EPS pyrolysis fronts which are predominantly made of gaseous styrene monomer are more significant and control casting quality for shapes that include larger thickness EPS foam patterns. With increasing thickness, irregular volatilized polymer front was more pronounced as detected from neutron radiography. Those irregular degraded polymer pyrolysis products may not escape quickly and dissolve portions of un-decomposed foam as they travel leading to potential of trapped EPS products within setting metal. As the entrapped foam pyrolysis products are a major cause of the gas cavity defects (Sun, Littleton et al. 2003), the behavior of those irregular volatilized flows are of primary importance to determine the quality of casting products. This essential complexity of the volatilized flows, which could not be clearly revealed before by using the traditional X-Ray radiography, was visualized by neutron radiography combined with image processing.

Furthermore, by selecting different ranges of pixel intensity in a histogram, it is possible to identify the pyrolysis fronts and to distinguish them from the metal flows and the undecomposed polymer foam, as shown in Figure 2.19 and Figure 2.20. Figure 2.19

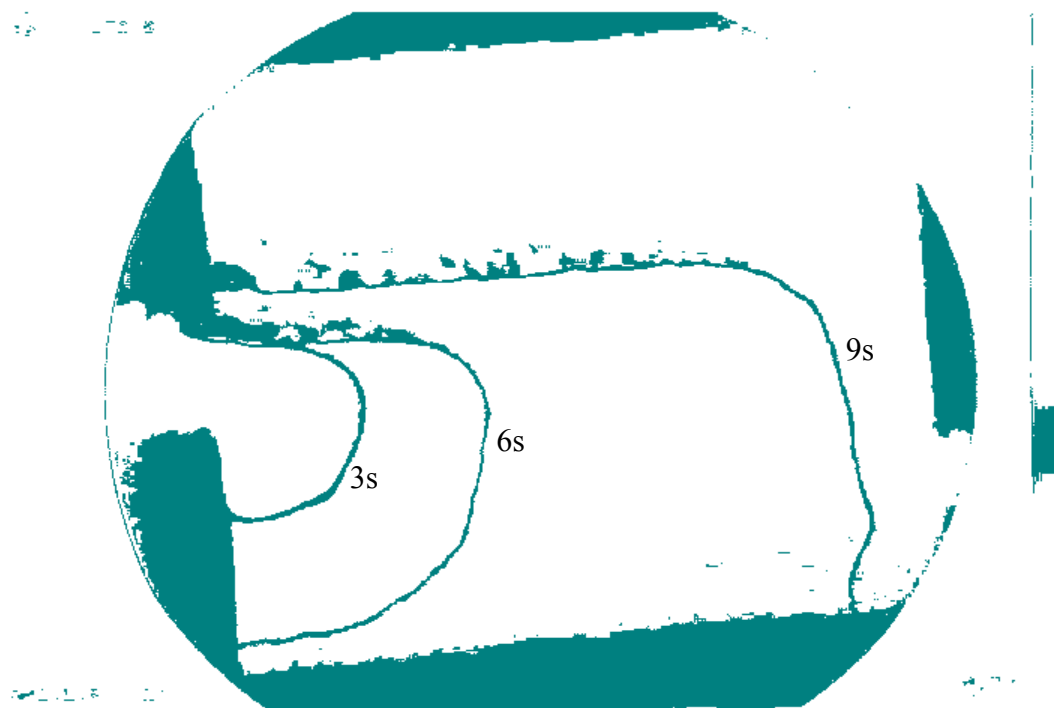


Figure 2.19 The pyrolysis fronts of side-gating with thickness 24 mm (at 3, 6, and 15 sec)

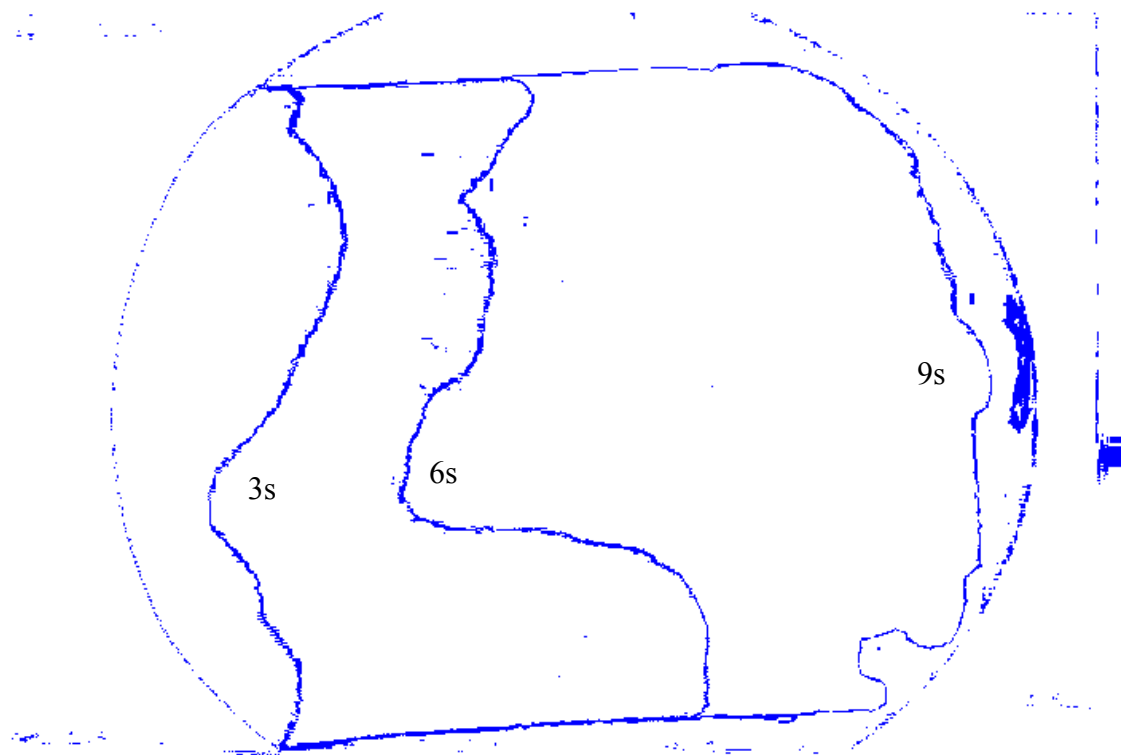


Figure 2.20 The pyrolysis fronts of side-gating with thickness 4 mm (at 3, 6, and 15 sec)

gives a processed image for the side-gating casting of the thickness 24 mm at 3, 6 and 15 seconds after the molten metal enters the polymer pattern. The pyrolysis front has its own corresponding intensity range, located between the lower intensity range representing the un-decomposed polymer foam and the higher intensity range representing the molten metal flow. In particular, the range of pixel intensity representing the pyrolysis front is between 105 and 130, while the range representing the undecomposed polymer pattern is between 80 and 105, and the range representing the molten metal flow is between 132 and 178. Figure 2.20 shows another example of processed image for the side-gating casting of the thickness 4 mm at 3, 6 and 15 seconds after the molten metal enters the polymer pattern. Those images show differences in the characteristics of the pyrolysis front and filling of molten metal due to the effect of thickness of the polymer pattern. This kind of visualization can be further used as a valuable tool for calibration of numerical models that can be used for predicting effectiveness of given gating type, coating properties, foam density and fusion levels for improved understanding of the lost foam casting process.

Summary

Time lapsed neutron radiography with digital image processing is presented using a reactor source and high resolution neutron detector at MNRC to study a metal casting technique. Neutron radiography was found to be a powerful tool to visualize the pyrolysis behavior of the expanded polystyrene foam interacting with liquid Aluminum metal during LFC process. Useful information, which is hardly obtained by other traditional techniques such as X-ray radiography, has been derived from neutron radiography due to

the unique neutron cross-section of the materials involved in the study. Based on the results from neutron radiography combined with the image processing and analysis of the neutron images, the pyrolysis front could be clearly identified and isolated from the molten metal as well as the undecomposed polymer foam. For the side-gating with a thickness of 24 mm, the range of pixel intensity representing the pyrolysis front is between 105 and 130, while the range representing the undecomposed polymer pattern is between 80 and 105, and the range representing the molten metal flow is between 132 and 178. Significant differences in the filling characteristics were observed and discussed during the LFC processes, in which the polymer patterns with different thicknesses and gating systems were used. The irregular volatilized pyrolysis products composed of gaseous styrene monomers were observed more pronouncedly for the thicker EPS foam patterns during the lost foam casting process.

Acknowledgement

The authors are grateful to the guidance, experimental collaborations, and interpretations over the past few years from the collaborators from General Motors Powertrain (GMPT) group and those who were formerly affiliated with Casting and Development Validation Center (CDVC). Also, the author would like to thank the technical support from the McClellan Nuclear Radiation Center (MNRC), UC Davis. The Joint Institute for Neutron Science (JINS) fellowship, which provided partial funding for the proposed research including travel support to the MNRC and the related training through neutron science related workshops and seminars, is greatly appreciated.

References

- Bates, C. E. (1999). Economic Evaluation, Energy Efficiency, and Environmental Impact of Lost Foam, University of Alabama, Birmingham.
- Campbell, J. (2000). "The concept of net shape for castings." Materials and Design **21**(4): 373-380.
- Cox, M., R. A. Harding, et al. (2003). "Optimised running system design for bottom filled aluminium alloy 2L99 investment castings." Materials Science and Technology **19**(5): 613-625.
- Dinwiddie, R. (2004). Refining Lost Foam Casting to Preserve American Industry. Science and Technology Highlights, Oak Ridge National Laboratory: p5.
- Foti, R. (2000). "Speakers Detail Lost Foam Casting Successes, Preventing Defects." Modern Casting **90**(5): 51.
- Goria, C. A., C. G., et al. (1986). "Coating Permeability: A Critical Parameter of the Evaporative Pattern Process." Transactions of the American Foundrymen's Society **94**: 589-600.
- Lessiter, M. J. and E. L. Kotzin (2003). "Timeline of Casting Technology." Engineered Casting Solutions: 76-80.
- Littleton, H., B. Miller, et al. (1997). "Process control for precision lost foam castings (part 2)." Foundry Management and Technology **125**(2): 41-44.
- Liu, J., C. W. Ramsay, et al. (1998). "A study of Foam-Metal-Coating Interaction in the LFC Process." Transactions of the American Foundrymen's Society **105**: 419-426.
- Luo, X. and D. Penumadu (2006). Application of Neutron Radiography to the Lost Foam Casting Process: Visualization and Characterization of the pyrolysis front.

- Imaging and Neutrons 2006 Workshop, Oak Ridge, TN, Oak Ridge National Laboratory.
- Media Cybernetics (2001). Image-Pro Plus V4.5 Reference Guide For Windows, Media Cybernetics.
- Penumadu, D. (2002). Characterization of EPS Foam and Visualization of Degradation Mechanism in Lost Foam Casting. American Foundry Society Division 11 Meeting, Birmingham, AL.
- Sirrell, B., M. Holliday, et al. (1995). The Benchmark Test 1995. 7th Conference on Modelling of Casting, Welding and Advanced Solidification Processes, London, UK.
- Smith, B. V. (1996). Developing Lost Foam Casting Processes. BCIRA International Conference, Viking, UK.
- Sun, W. L., H. E. Littleton, et al. (2003). "Formation mechanism of gas cavity defects in lost foam iron castings." International Journal of Cast Metals Research **16**: 549-553.
- Turner, R. E. (1972). "Radiography: Major contribution to the metalcasting industry." Modern Casting **61**(1): 44-45.
- Yao, X. and S. Shivkumar (1997). "Mould filling characteristics in lost foam casting process." Materials Science and Technology **13**: 841-846.

Chapter 3 Stress-Strain Behavior of Granular Materials under Confined Compression Using In-situ Neutron Diffraction

This chapter is revised based on a paper published by Xin Luo and Dayakar Penumadu:

X. Luo, D. Penumadu, K. An, F. Tang, C.R. Hubbard, Stress Strain Behavior of Particulate Materials Using Neutron Diffraction and Discrete Element Modeling, Materials Science and Technology (MS&T) 2006: Fundamentals and Characterization, Volume 1, 2006, pp.243-249

My primary contributions to this paper include (i) development of the problem into a work, (ii) identification of the study areas and objectives, (iii) design and conducting of the experiments, (iv) gathering and reviewing literature, (v) processing, analyzing and interpretation of experimental data, (vi) pulling various contributions into a single paper, (vii) most of the writing.

Abstract

Stress-strain relationship of particulate materials is complex, and depends on the initial state of packing, past stress history, and the applied stress path (compression, shear, or extension). The present study aims to develop an experimental approach to provide a fundamental understanding of the macroscopic stress-strain relationship to its microstructure (particle contacts and void size distribution) and the meaning of terms “stress” and “strain” in particulate materials subjected to various types of loading conditions. An assemblage of the particulate silica with controlled initial state was subjected to compression, and the deforming specimens were studied by in-situ neutron diffraction at the High Flux Isotope Reactor using the recently developed load frame associated with Neutron Residual Stress Facility (NRSF2). A monochromator of Si400

($\lambda=1.89 \text{ \AA}$) and the lattice plane SiO_2 (-321) with reflection at 2θ of 75.60° were used in this paper. Significant difference between the global or macroscopic strain and lattice strain-stress relationship was observed for confined conditions. The approach presented in this paper for studying particulate materials will be useful in wide ranging applications such as sintered ceramics and powder metallurgy.

Mechanical behavior of granular materials

Deformation response of a granular assembly to an external force is an essential feature of interest for studying particulate material systems. There is a need to establish the relationship between commonly observed overall or global stress strain response of granular materials with variations existing in local force and displacement at particle scale. For continuous materials, the stress strain relation can be well described macroscopically using continuum mechanics. However, for a granular assembly, it is well known that its macroscopic stress-strain relation is quite complex, and depends both on the initial state (such as local and global porosity and particle coordination numbers) of the assembly, past stress history, and its loading path.

A granular assembly has been found to show anisotropy, inhomogeneity, and non-linearity in the distribution of both the contact network and the contact forces among particles. Majmudar and Behringer (2005) studied the stress-induced anisotropy of contact forces in granular materials. Specially, they visualized internal stress in each grain and measured both normal and tangential grain-scale forces inside each disk by solving the inverse photoelastic problem for an assembly of photoelastic disks subject to pure shear and isotropic compression. They found that there are differences in terms of

contact network and contact forces between the two stress states; shear systems have long-range correlations in the direction of force chains while compressed systems show short-range correlations; anisotropy occurs in the spatial correlation of forces, forming the force chains. Otto et al. (2003) discussed a general approach to understand stress response in 2-D anisotropic granular layers. Based on both classical anisotropic elasticity theory and linear theory of anisotropic directed-force chain networks, they found that two-peak response functions can occur for classical anisotropic elastic materials. Krut (2003) investigated the probability density functions (PDF) of contact forces in anisotropic, cohesionless and frictional particles both numerically and theoretically. In his numerical study, he adopted discrete element simulations of biaxial deformation of a 2-D assembly of granular disks, and found that the PDF exhibits exponential decay; in his theoretical study, he used a maximum entropy method, and found that theoretical results correspond qualitatively to those obtained from the numerical study.

The “strain” experienced by a particulate assembly is a result of deformations both within each particle and relative motions between them. There are two fundamental deformation mechanisms in particulate materials; one is relative motion including rolling and sliding occurring between particles and the other is distortion and crushing of individual particles. A number of theoretical models have been developed to interpret the macroscopic stress strain behavior in terms of the interaction between particles. Deresciewicz (1958) and Scott (1963) described simplified models to analyze and predict the strain due to elastic distortion based on two elastic spheres in contact. Miller (1963) considered sliding within deformable spheres. Nevertheless, the motions within an assembly of particulate materials are too complex to be analyzed by any single and

simple model. For example, different mechanisms may be either acting in same parts of an assembly at different times or affecting different parts of the assembly at same time during the loading or applied deformation process. A number of attempts have been made to study the constitutive relations for granular materials (Lambe and Whitman 1969). Global stress and strain relation of granular materials has been discussed based on the applied load and displacement response at the macroscopic level. On the other hand, a theory of stress distribution based on the physics of force chains has recently been developed for granular materials (Behringer, Howell et al. 1999; Socolar, Schaeffer et al. 2002).

Understanding the relationship between stress and strain, particularly, the elastic response occurring in particles when the granular assembly is subjected to both elastic and inelastic loading process, represents an important aspect in granular mechanics. However, there is little experimental work to directly establish quantitative relationship between the macroscopic stress strain response and the strains at particle scale for particulate materials due to a lack of appropriate experimental approaches.

This study aims to demonstrate that the neutron scattering technique offers a unique opportunity to study such complex problems to provide a fundamental understanding of mechanical response of granular material for addressing relevant scientific issues in particle mechanics. Experimental data will also be very useful for developing suitable particle scale constitutive models for calibration and development of discrete element methods for modeling discontinuous medium.

Measurement of strains by diffraction method

In general, all diffraction based stress analysis utilizes the interplanar d-spacing, i.e., the distance between certain atomic planes, as an internal strain gage. For a given reflection hkl the lattice d-spacing can be determined by Bragg's Law as shown in Equation (3.1).

$$\lambda = 2d_{hkl} \sin(\theta_{hkl}) \quad (3.1)$$

where,

θ_{hkl} – The Bragg's peak of the reflection (hkl)

d_{hkl} – d-spacing of the reflection (hkl)

λ – Wavelength of the radiation

Unlike X-rays interacting with the electron shells of the atoms, neutrons interact with atomic nuclei and have the power of penetration that is two or three orders of magnitude larger for most materials than that of X-rays. Especially, in stress analysis, the X-ray technique is often limited to the region quite near the sample surface while the large penetration power makes neutrons a unique tool for probing strains and stresses in the interior of bulk materials in a non-destructive way. In this case, it is impossible to investigate the strain occurring in the granular assembly material by using the X-ray due to the significant attenuation, and neutron source appears to be viable option.

The lattice strain in terms of d-spacing change can be defined as shown in Equation (3.2).

$$\varepsilon = \frac{d_{hkl} - d_0}{d_0} \quad (3.2)$$

where,

ϵ – the lattice strain

d_{hkl} – the interplanar d-spacing of the reflection (hkl) at a given external loading

d_0 – the reference d-spacing of the reflection (hkl) under stress free conditions

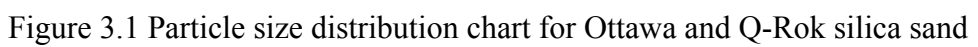
In practice, the lattice strain is actually an average based on the lattice strains measured within the sub-set of grains inside the scattering volume specified by both the incident and diffracted beams.

Materials and sample preparation

The particulate material used in this study is Ottawa and Q-Rok silica sand. Their material properties are listed in Table 3.1, and particle size distribution data are shown in Figure 3.1.

Table 3.1 Physical properties of Ottawa and Q-Rok silica sand

Type	Grain shape	Mineral	Specific Gravity	SiO ₂ %	Hardness	Ph	Mean grain size D ₅₀ (mm)
Ottawa	Round	Quartz	2.65	99.8	7	7	0.86
Q-Rok	Angular	Quartz	2.65	99.0	7	6.5	0.81



Mechanical behavior of the silica sand under the condition of confined axial compression was investigated by using Geotest S5770 and the pertinent data acquisition system, as given in Figure 3.2. An assemblage of the particulate silica with controlled initial state was subjected to compression. The particulate assembly had a diameter of 19 mm and a height of 19 mm confined by a cylindrical steel chamber and piston.

The relative density (D_r) is defined as the ratio of the difference between the void ratio of a cohesionless soil in the loosest state and any given void ratio, to the difference between the void ratios in the loosest and in the densest states (ASTMD653-97 2000), as given in Equation (3.3).

$$D_r = \frac{e - e_{\min}}{e_{\max} - e_{\min}} \times 100\% \quad (3.3)$$

Where,

$$e - \text{Void ratio of the sand, } e = \frac{V_v}{V_s} = \frac{1000V_T G_s}{W_s} - 1$$

$e_{\max}, e_{\min}$ – Maximum and minimum void ratio the sand can naturally obtain before compression test, respectively

V_v, V_s, V_T – Volume of void, solid, and the total in the assembly, respectively (m^3)

W_s – Weight of the solid in the assembly (kg)

G_s – Specific gravity of the silica sand

The relative density D_r is used to characterize how dense the packing of granular materials is, and D_r of a natural soil deposit very strongly affects its engineering behavior. Thus, it is important to conduct laboratory tests on samples of the sand at the same

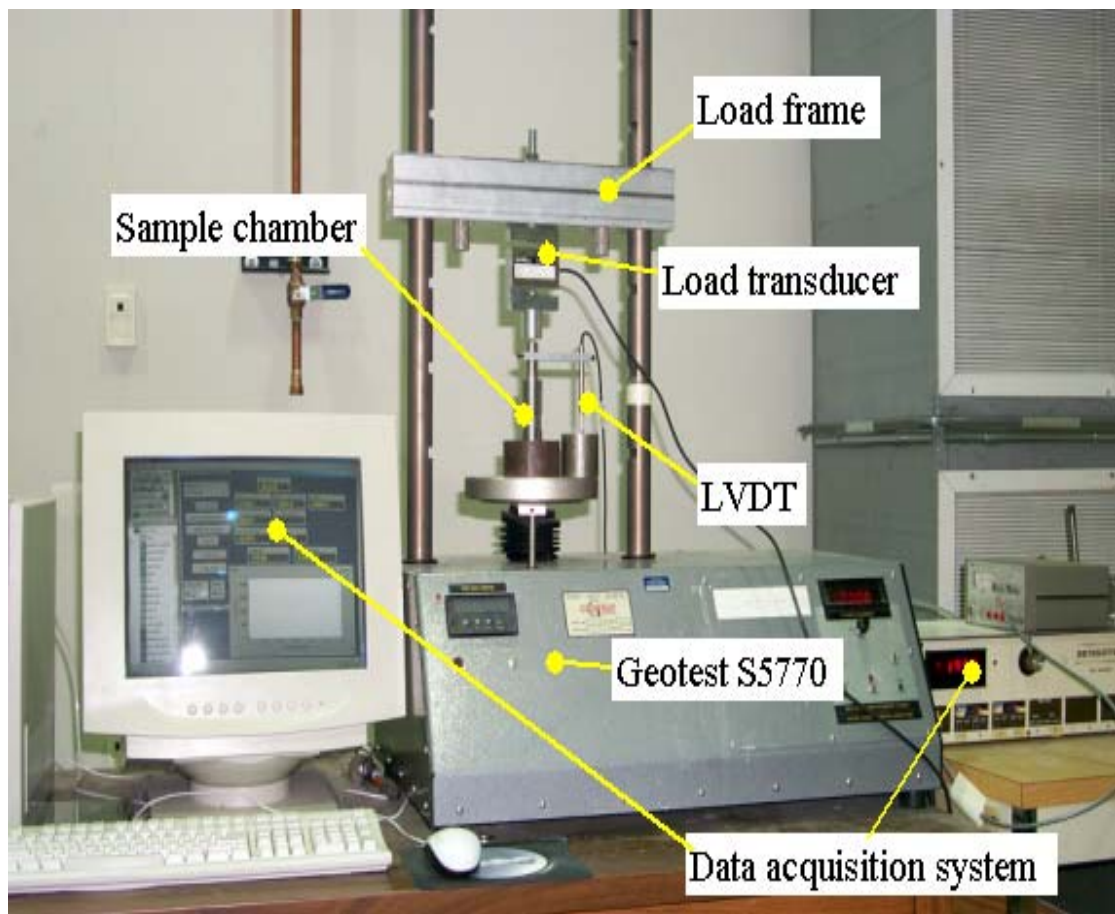


Figure 3.2 Experimental set-up of Geotest S5770 and the data acquisition system

relative density as in the field (Holtz and Kovacs 1981). The deformation of the two silica sands under stress at typical initial relative densities chosen in this study are given in Table 3.2, Figure 3.3 and Figure 3.4.

Engineering stress mapping facility using neutrons

An assemblage of the particulate silica with controlled initial state was subjected to compression. The particulate assembly had a diameter of 19 mm and a height of 19 mm confined by cylindrical steel chamber and piston, as shown in Figure 3.5 (Luo, Penumadu et al. 2005). The sample was subject to deformation using compressive load on the piston operated at a constant rate of movement. The assembly was oriented so that the strains could be measured along axial as well as transverse directions by in-situ neutron diffraction at the High Flux Isotope Reactor using the recently developed load frame associated with NRSF2, as shown in Figure 3.5 (Luo, Penumadu et al. 2005).

For continuous loading measurement, interplanar d-spacing was measured in the loading direction with an incident slit $5 \times 10 \text{ mm}^2$ and receiving slit 5 mm as shown in Figure 3.6 and Figure 3.7. Axial compressive loading was applied in displacement control mode using a deformation rate of 0.0005 mm/sec; the counting time for neutron intensity was 60 sec per strain measurement.

Table 3.2 Initial void ratios and relative density of the sand for the confined compression

Samples	Max void ratio $e_{\max}$	Min void ratio $e_{\min}$	Relative density chosen Dr (%)
Ottawa	0.71	0.57	0, 100
Q-Rok	1.11	0.83	0, 100

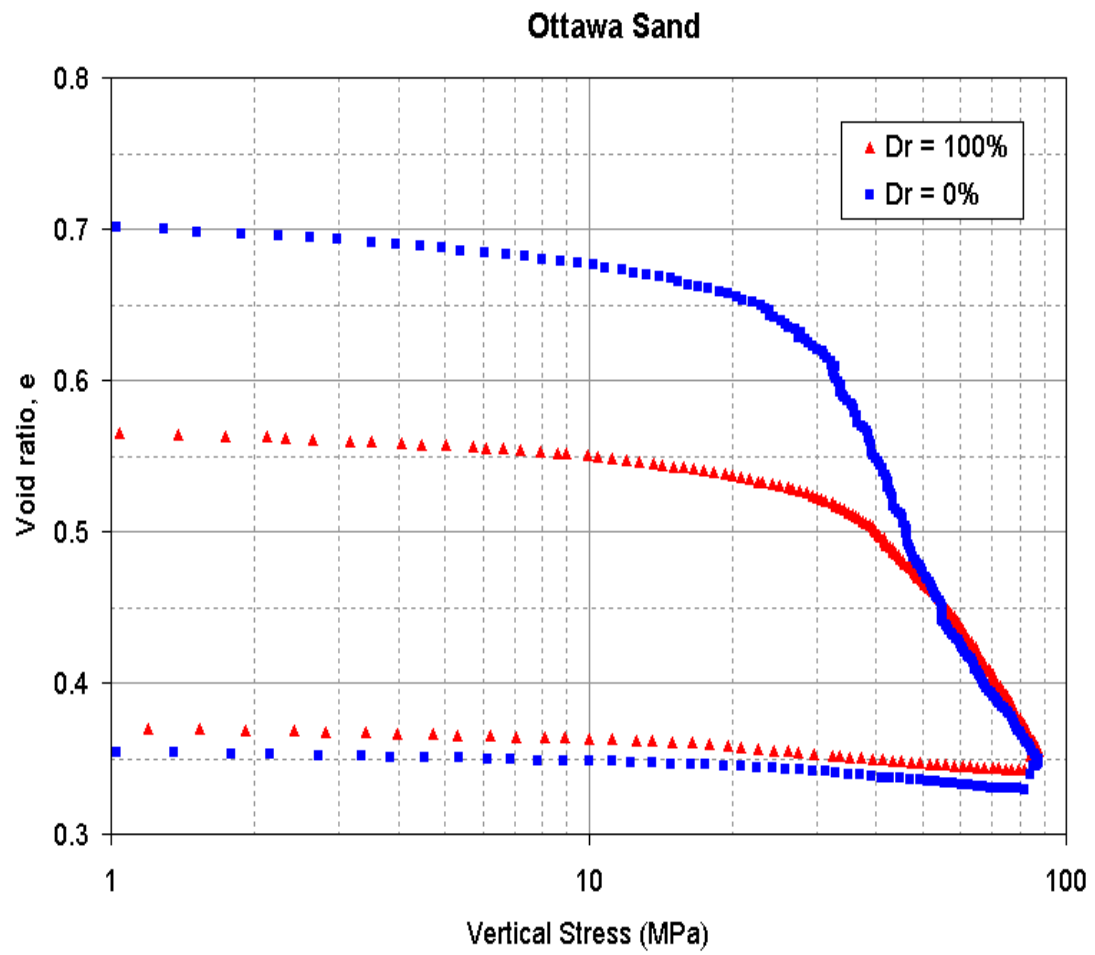


Figure 3.3 Stress and void ratio behavior of Ottawa sand

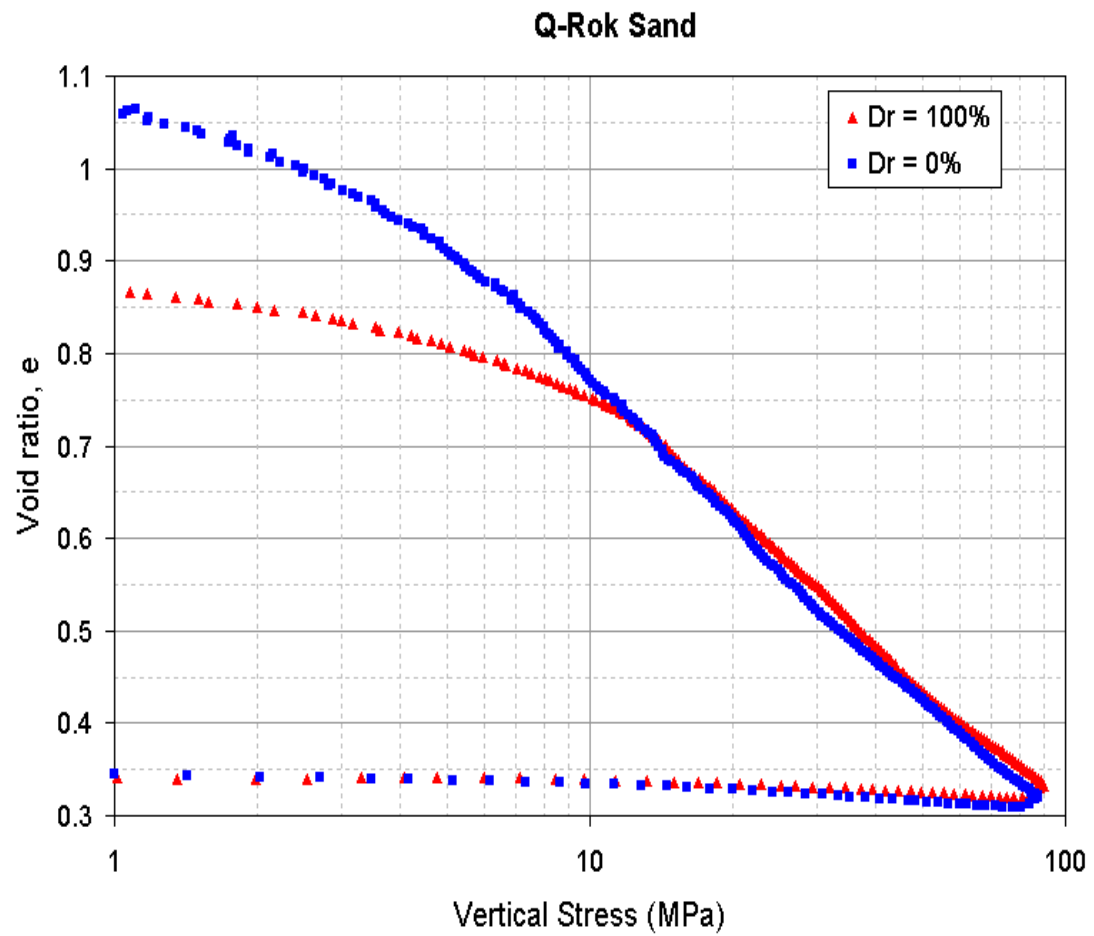


Figure 3.4 Stress and void ratio behavior of Q-Rok sand

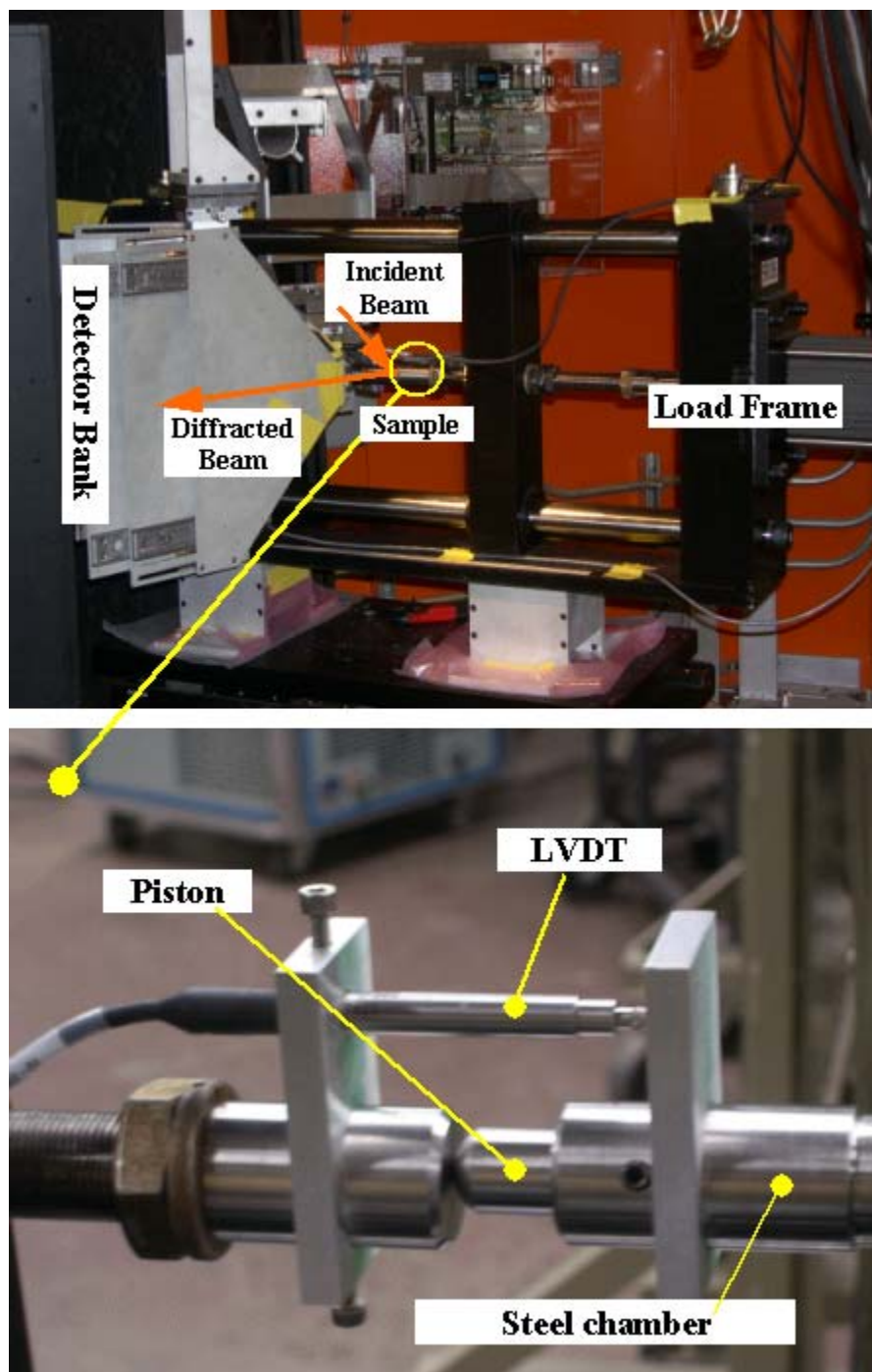


Figure 3.5 NRSF2 and in-situ load frame at HFIR, ORNL

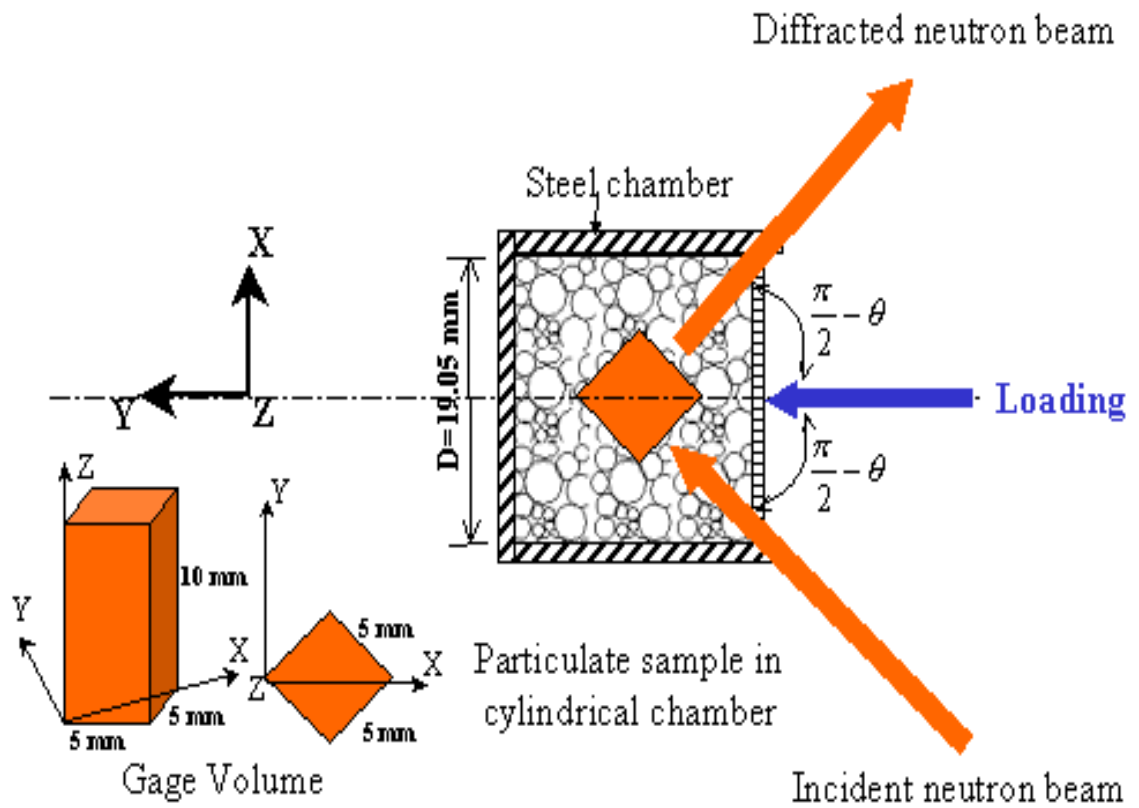


Figure 3.6 Schematic of the continuous loading measurement in axial direction

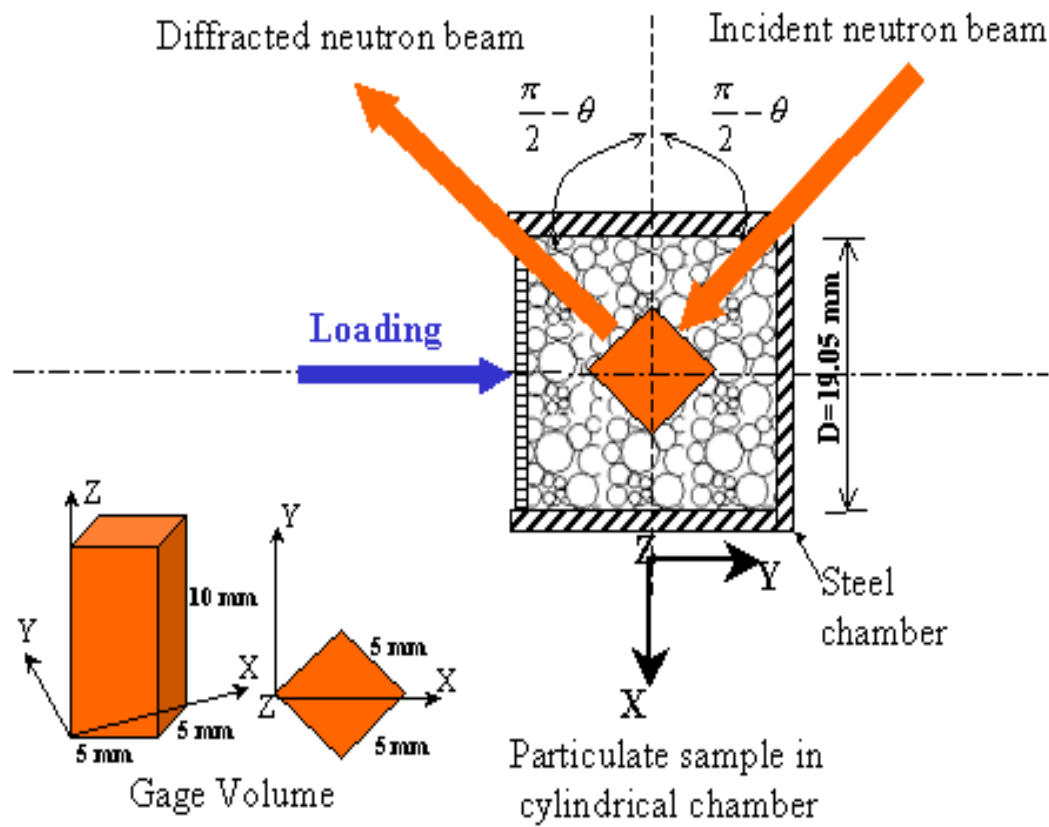


Figure 3.7 Schematic of the continuous loading measurement in transverse direction

For local strain mapping measurement, the gage volume with incident slit 2×5 mm² and receiving slit 2 mm were used as given in Figure 3.8. Axial displacement rate was 0.005 mm/sec; counting time was 40-70 min per strain measurement. Neutrons of wavelength 1.89 Å reflected from a Si400 monochromator were used to measure the positions of (-321) peak with a Bragg angle (2θ) of 75.60° for SiO₂, as shown in Figure 3.9

Experimental results and discussion

In-situ measurements of the strain response to the external loading were made on the automated load-frame system as given. During the loading process, the granular assembly in the steel chamber was exposed to the incident neutron beam as well. Scattered neutrons were collected by NRSF2 detector bank, and the neutron data in the form of counts versus angle were analyzed with a fitting algorithm using Gaussian method, which gave the peak position, intensity and line width, by the software NRSF2 Real Time Data Processing and Strain Mapping provided by Oak Ridge National Laboratory (An and Hubbard 2006). A typical fitting of Bragg's peak from neutron diffraction data for one strain measurement is shown in Figure 3.10.

Before any external loading is applied, the pertinent neutron data was collected and fitted as aforementioned for obtaining reference d-spacing d_0 . Compression loading was applied to the particulate assembly via the piston, and the data was collected to

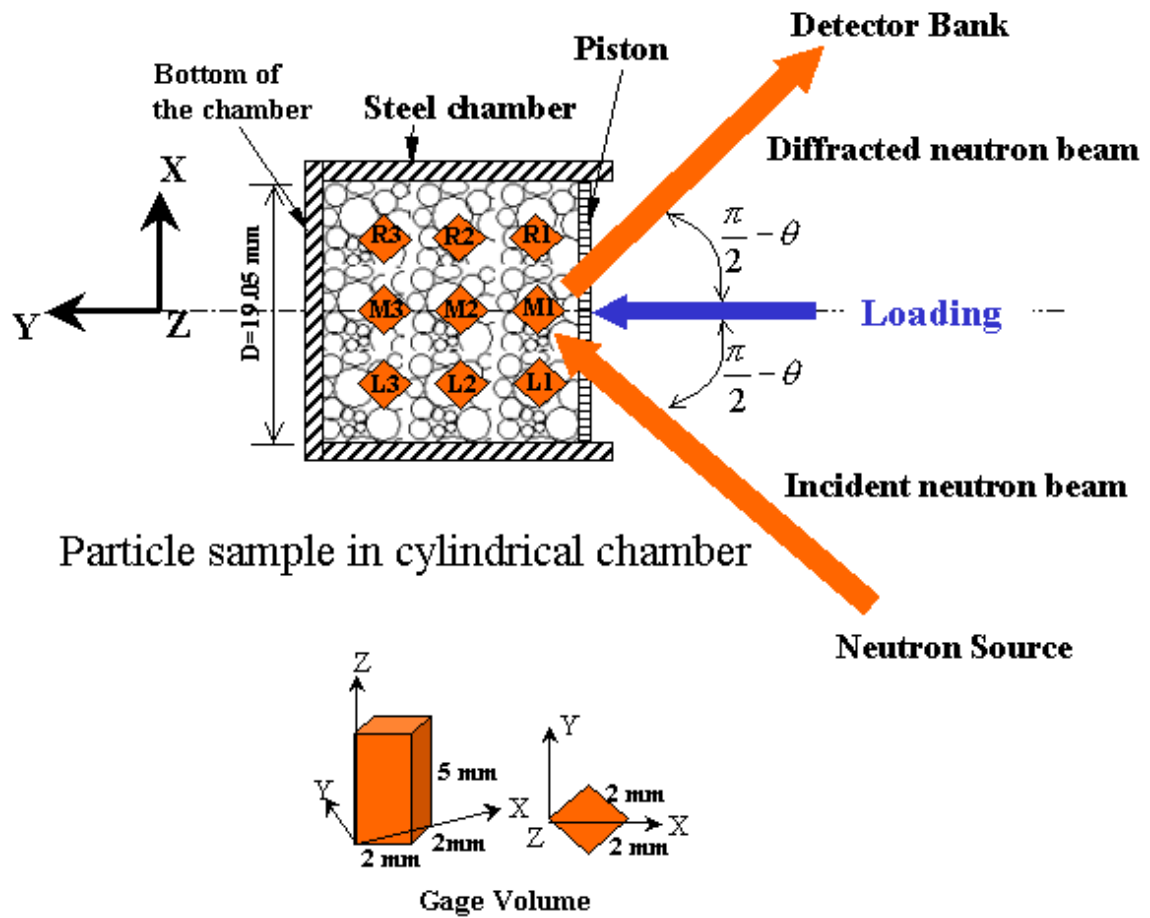


Figure 3.8 Schematic of the local strain mapping measurement in axial direction

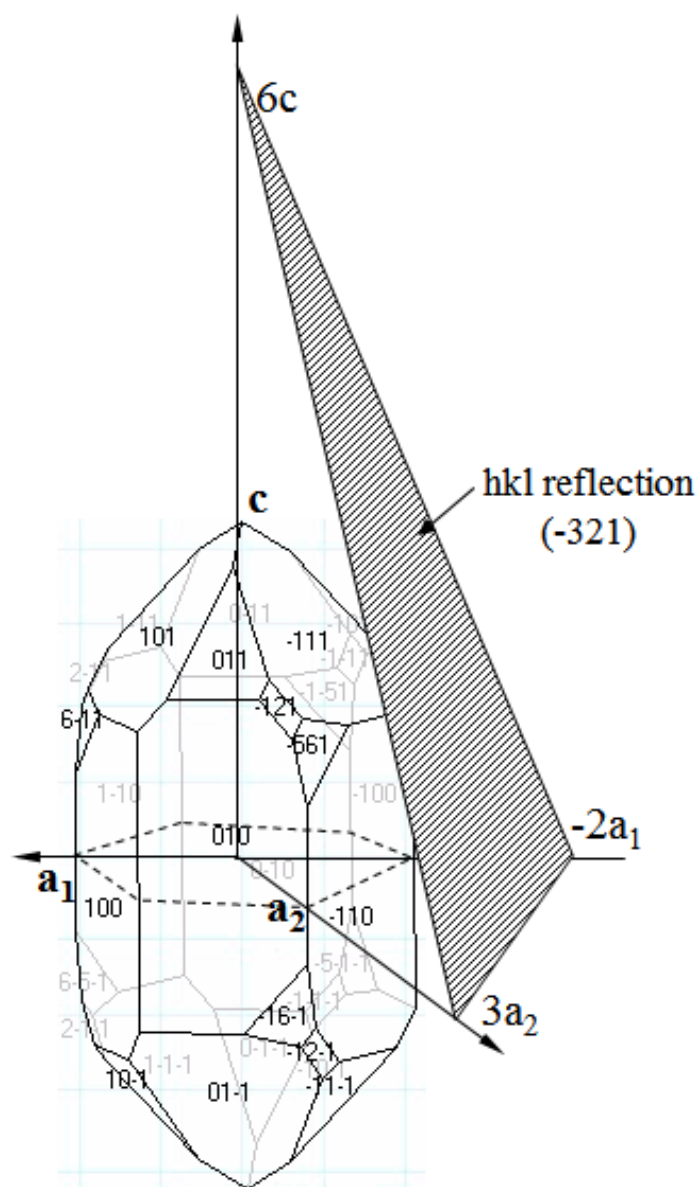


Figure 3.9 The selected hkl reflection (-321) for the SiO_2

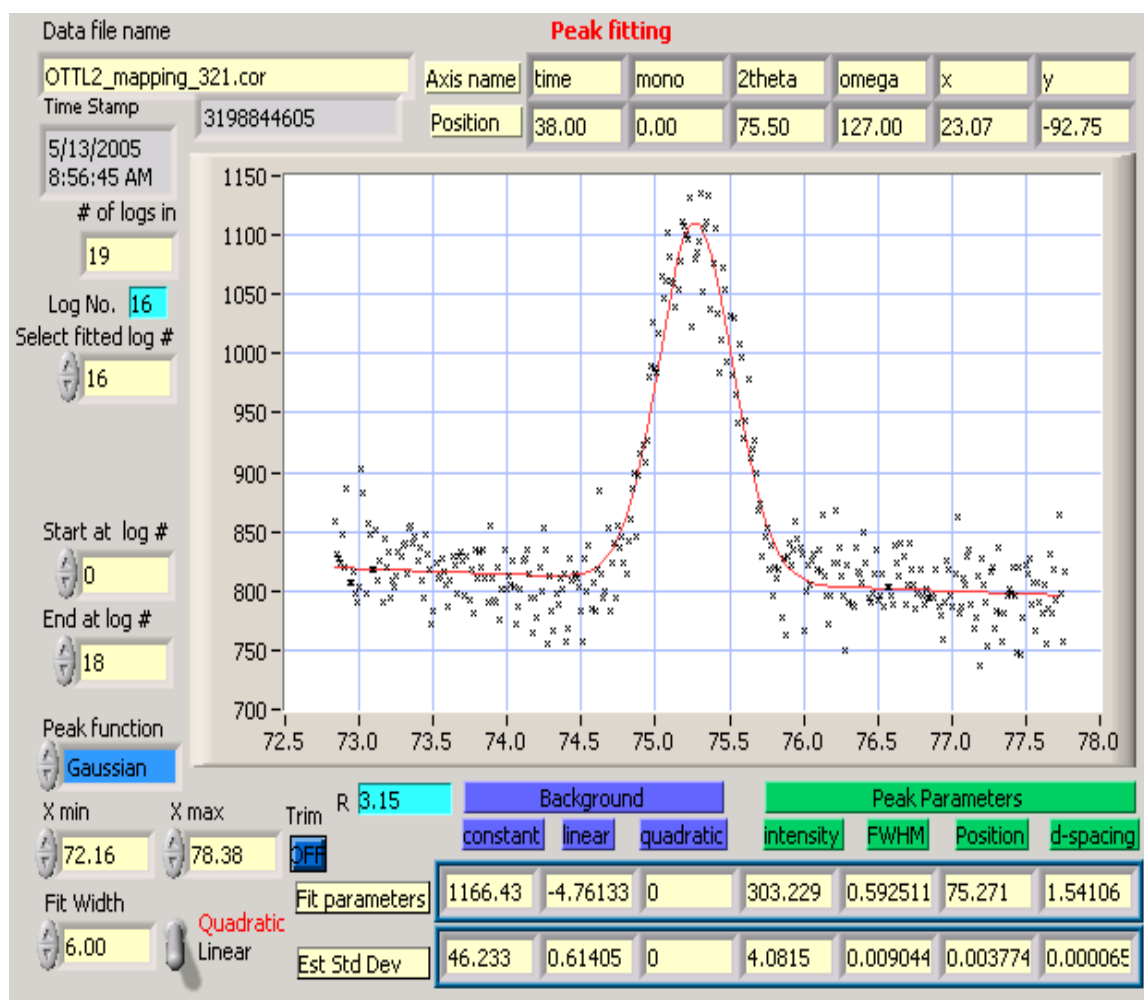


Figure 3.10 Typical results of fitting of Bragg's peak from neutron diffraction data

obtain global stress dependent d-spacing for the particles within the gage volume for the target hkl reflection. The d-spacing and corresponding lattice strains will also be calculated using Equations (3.1) and (3.2).

Figure 3.11 shows results for a complete loading and unloading history for a given initial porosity for the round shaped Ottawa sand particles (Luo, Penumadu et al. 2007). The horizontal axis represents applied global stress based on the externally applied compressive load and cross-sectional area of the specimen. The vertical axis on the left represents global strain based on the overall displacement of piston, and the lattice strain based on the neutron diffraction data is shown on the right.

The calculated global strain includes the combined effect of the elastic strain of individual particles, slippage between particles, crushing and compaction of particles in the assembly, while the lattice strain indicates only elastic strain within the particles. From Figure 3.11, considerable difference between measured global and lattice stress strain relation was observed. The lattice strain is at least one order of magnitude smaller compared to the global strain when subject to the same externally applied load. When the applied stress exceeded 40 MPa, significant particle breakage occurred resulting in a large increase in the rate of global strain variation. However, it is interesting to note that elastic strain did not change much when applied load exceeded the crushing stress and could be explained by the fact that due to particle breakage, circular silica particles had majority of through-particle cleavage type breaks, and did not significantly alter the atomic planar spacing in the process. Considering the error bounds for variation in the

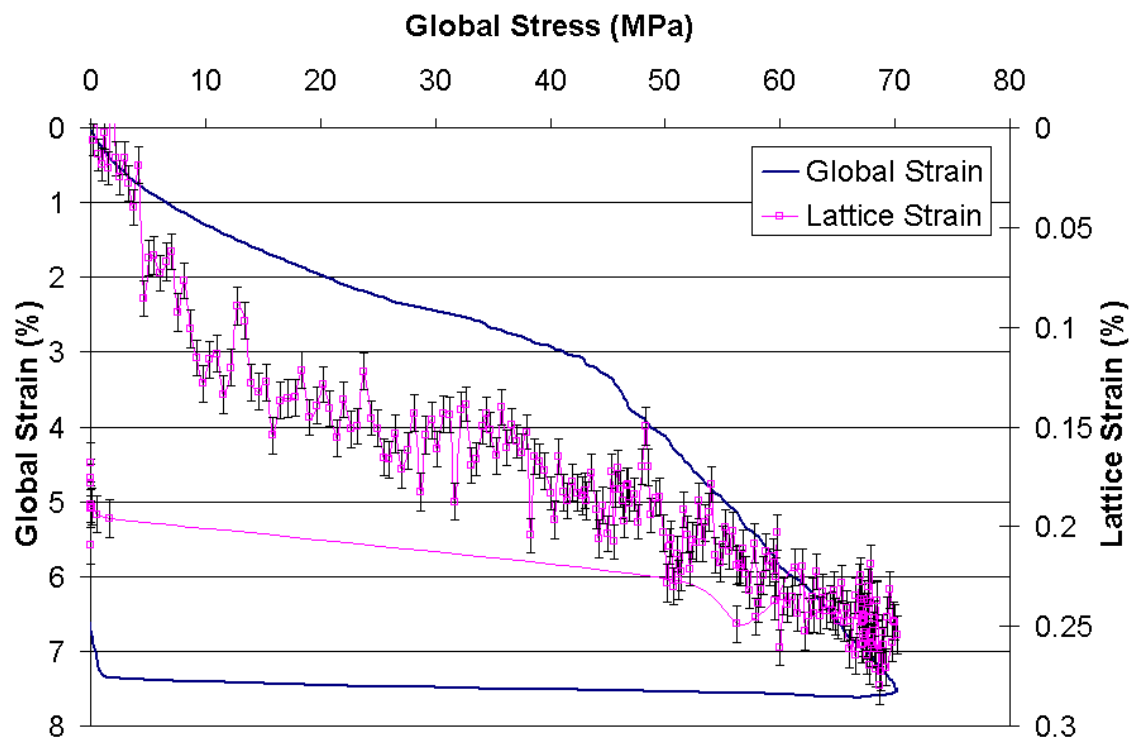


Figure 3.11 Global and lattice strains vs global stress in axial direction for Ottawa sand

interplanar d-spacing at a target stress, it is remarkable to note the parallel variation of both lattice level and global stress-strain curves during unloading. This confirms the common assumption that is often made to develop elastic parameters of particulate assembly using the unloading data typically under isotropic stress state.

For the global stress vs global strain curve, there are generally two stages observed during the loading process, as described by Lambe and Whitman (1969). From Figure 3.11, the first stage, which is also called “locking” stage, occurs when the applied global stress is less than around 20 MPa, and the stress-strain curve is concave to the stress axis, indicating that the stiffness of the particulate system is increasing gradually. This is due to the fact that the loose particles within the assemblage are squeezed and rearranged so that the system becomes more tightly packed. In this “locking” stage, some crunchy sound can also be heard when the global stress increases, which implies the crashing of the particles. The second stage, which is also called “yielding” stage, begins at the global stress of 10 to 20 MPa, and the stress-strain curve becomes concave to the strain axis, indicating that the global stiffness of the particulate system decreases gradually. In this stage, more and more particles start to get crashed with the increase in stress, which leads to large relative motions including sliding, compacting in particles. This stage is also called “yielding” stage. When the global stress reaches around 45 MPa, the stress-strain curve has a turning point, which means that there is a significant reduction in the stiffness of the particulate system. This is due to the further degradation of the particles and packing of the newly generated and remaining particles.

It is interesting to note that the curve of the global stress vs lattice strain behaves in a different way. There are also two stages in the stress-strain curve. In the first stage the relation between global stress and lattice strain is approximately linear, and the actual stress measured by neutron diffraction is much higher than the global stress. Young's modulus of 105 GPa for the particulate material (Dutta 2006) was used to estimate the actual stress within the particles. For example, when global stress reaches around 15 MPa, the global strain is around 1.6% while the lattice strain reaches around 0.15%, which corresponds to the stress about 157 MPa in the quartz particles. In the second stage, the relation between global stress and lattice strain is still approximately linear, but the "stiffness" in terms of global stress vs lattice strain becomes much larger. The global load where the second stage starts is around 15 MPa, which is close to the load where the second stage in the curve of global stress vs global strain starts. Therefore, the neutron scattering data confirmed the existence of the general two stages in the global stress vs global strain curve. When global stress reaches 70 MPa, the lattice strain reaches around 0.26%, which corresponds to the true stress about 265 MPa in the quartz particles. Therefore, there is a significant difference between the applied global stress and the true stress measured within the particles. Another interesting thing can be seen is that the stress relief due to the large motion and crashing of particles can be revealed and justified by the considerable decrease of the lattice strain.

From Figure 3.12 it can be seen that the maximum lattice strain is around 0.08%, much smaller than the corresponding global strain which is over 30%. The curve of global stress vs global strain for Q-Rok sand looks different from that for Ottawa sand

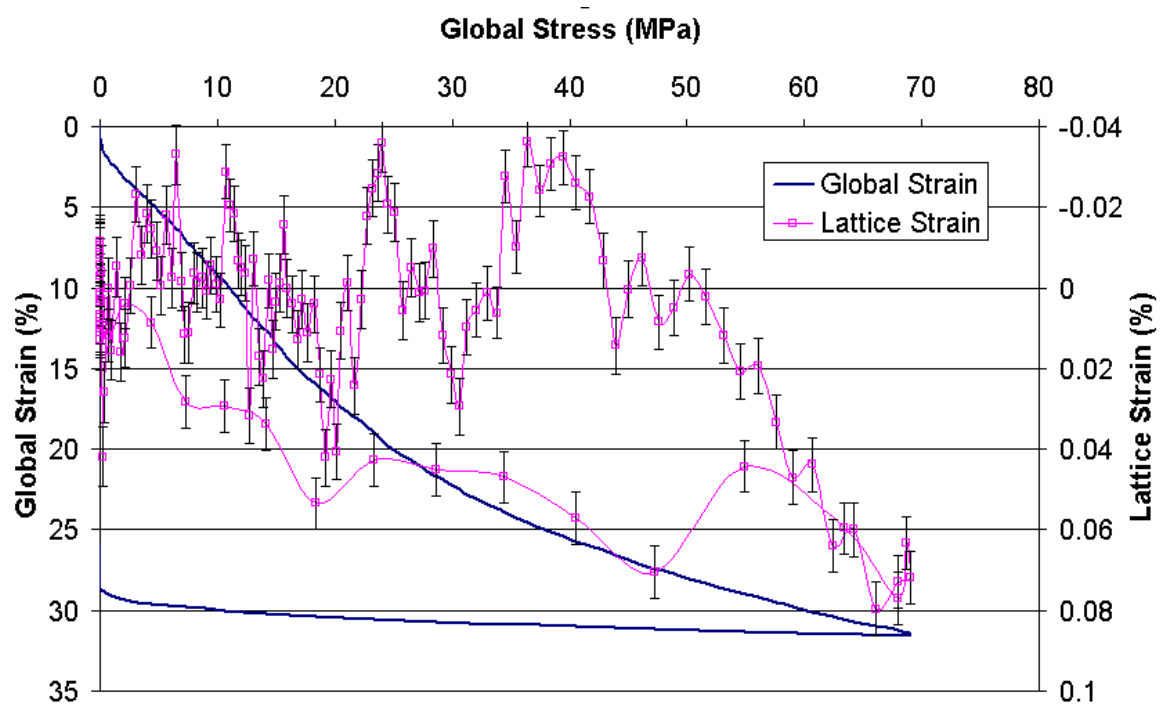


Figure 3.12 Global and lattice strains vs global stress in axial direction for Q-Rok sand

due to the morphology and nature of the particles. With increase in global stress, the system becomes stiffer and stiffer due to the locking, crashing and motions of the particles. The crunching sound can be heard as early as when global stress reaches around 3 MPa. When the global stress goes beyond 40 MPa, the global stress vs global strain is approximately linear.

As to the global stress vs lattice strain, there are several fluctuations observed in the curve of the global stress vs lattice strain before the global stress reaches around 40 MPa. In particular, during the loading the actual stress relief is observed at the global stress of about 12MPa, 20 MPa, and 30 MPa, which is justified by the decrease of the lattice strains. This also implies extensive fracturing, and large motions among the particles with increase of applied global stress. The maximum global stress applied to the particulate system is about 70 MPa, while the maximum lattice strain is around 0.08%, which corresponds to the estimated actual stress of 84 MPa. Therefore, the applied global stress is close to the actual stress within the particles. During the unloading, the global stress changes linearly with respect to global strain, and the lattice strain also decreases approximately linearly with respect to global stress.

The curves of the global stress vs lattice strain in transverse direction are different from that in axial direction, as shown in Figure 3.13 and Figure 3.14. The fluctuation pattern can be seen from the curve of global stress vs global strain. For example, while the global stress keeps increasing, the lattice strain decreases at around 10, 25 and 42 MPa, respectively, indicating the relief of the true stress within particles. Starting

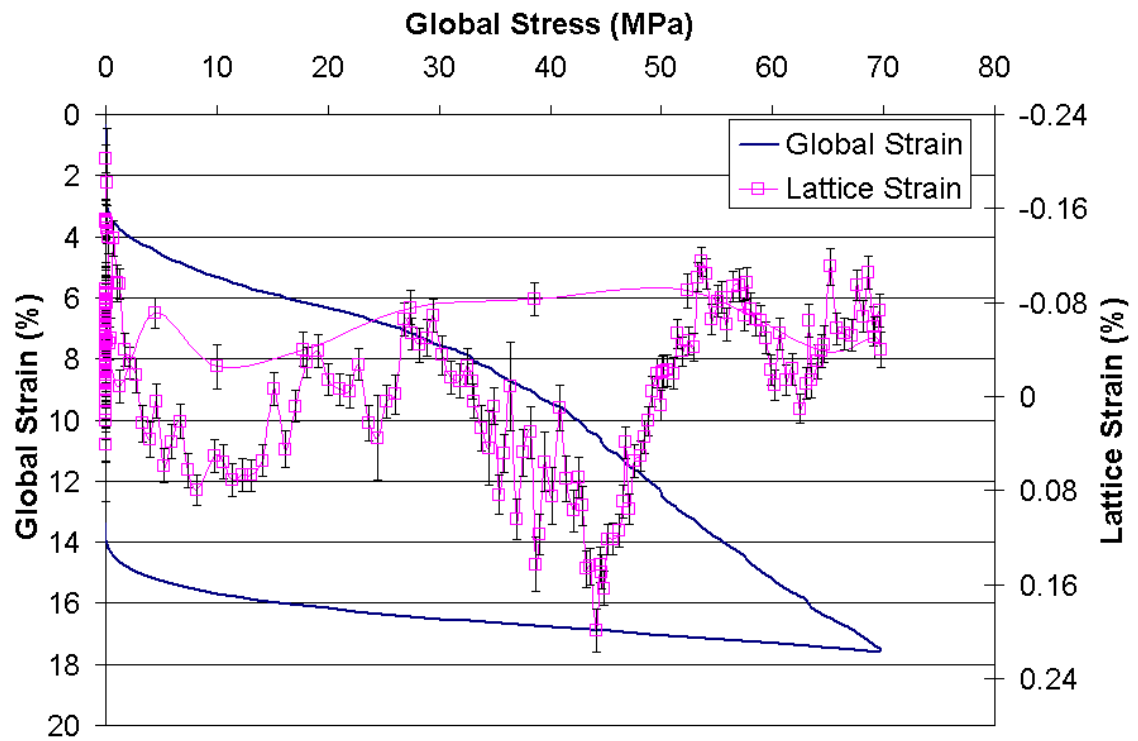


Figure 3.13 Global and lattice strains vs global stress in transverse direction for Ottawa sand

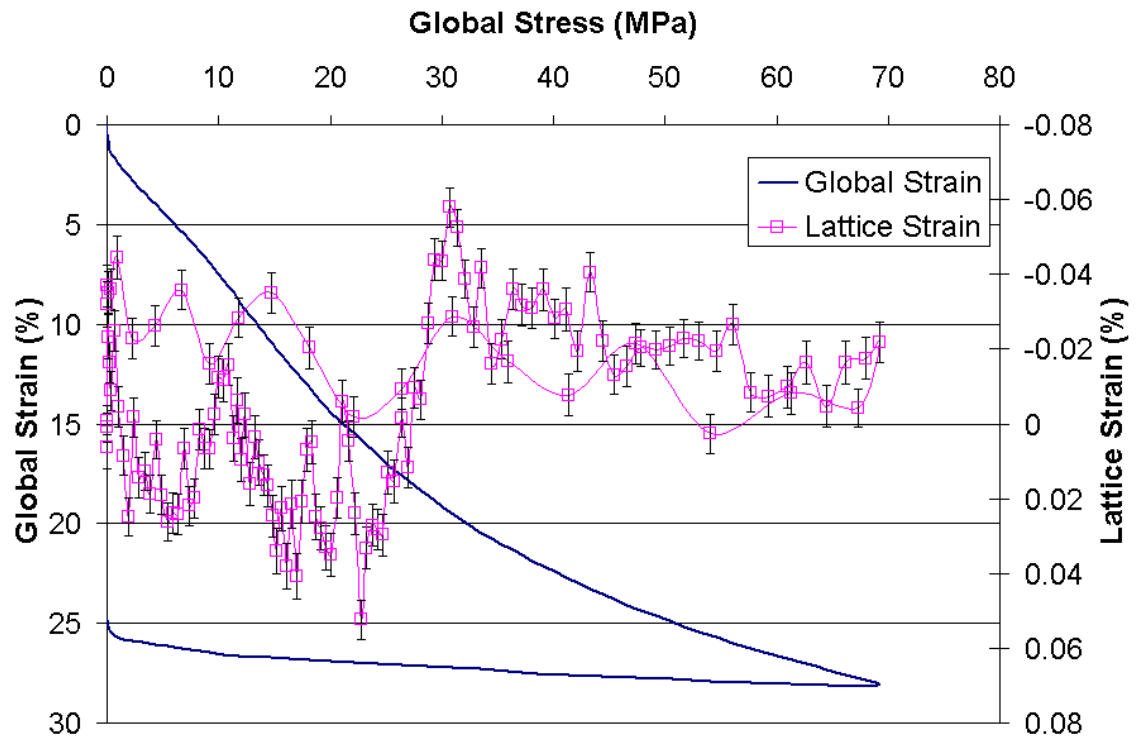


Figure 3.14 Global and lattice strains vs global stress in transverse direction for Q-Rok sand

from around 42 MPa of global stress, there is an approximate linear decrease of lattice strain with respect to the increase in global stress, indicating decrease of actual stress in particles. This could result from the crashing and the large motion of particles. A similar pattern of the global stress vs lattice strain in transverse direction for Q-Rok sand is observed to that for Ottawa sand. There is also fluctuation in the curve. Particularly, the relief of the actual stress in terms of the decrease in lattice strain occurred at around 5, 15, and 22 MPa respectively. There is a considerable decrease in lattice strain between 22 and 30 MPa, implying a major fracturing and compaction.

There is no evident trend of lattice strains observed in Ottawa particles for different applied global stress, as shown in Figure 3.15. The measured lattice strains are scattered, which implies a strong inhomogeneous distribution of the actual stress in particles. For example, when the applied global stress is 32 MPa, the lattice strain at the location of M1 (the location in the middle and close to piston) is about -0.13% , which corresponds to about 136 MPa in tension, while the lattice strain at the location of R3 (the location close to receiving slit and bottom of the steel chamber) is about 0.08% , which corresponds to around 84 MPa in compression. It is interesting to note that the local lattice strain could be significant at some locations even though the applied global stress is quite small. For example, when the global stress is 18 MPa, the lattice strain at R3 (a location close to receiving slits and bottom of the chamber) is about 0.07% , i.e., actual stress around 74 MPa equivalently. In Figure 3.16, three external loads (13, 30 and 65 MPa) were applied to the Q-Rok particulate system in the loading path, and the related

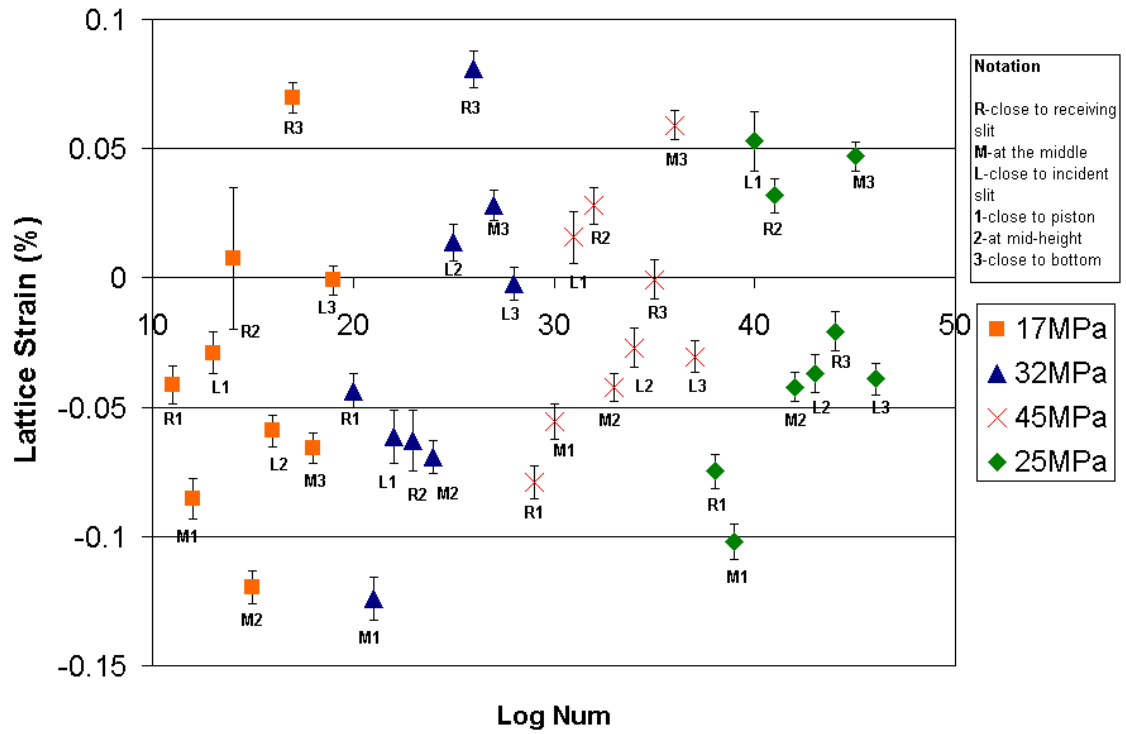


Figure 3.15 Variation of local lattice strains of Ottawa sand in longitudinal direction under different loads

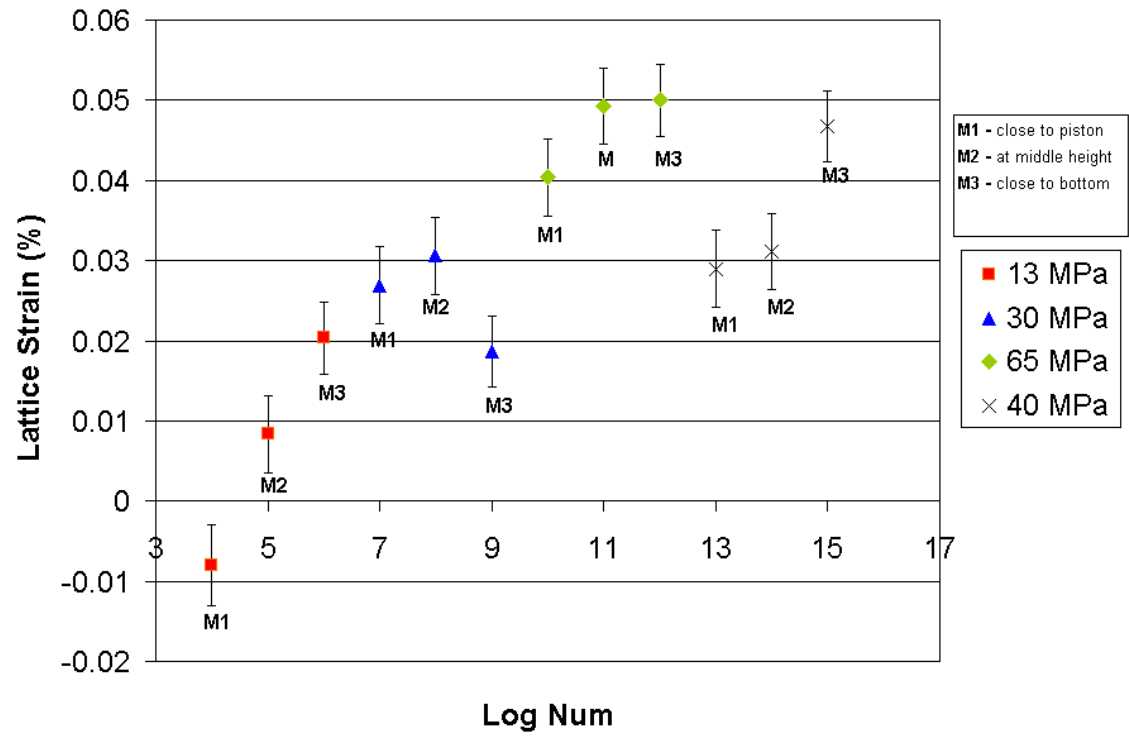


Figure 3.16 Variation of local lattice strains of Q-Rok sand in longitudinal direction under different loads

lattice strain at the middle height are around 0.01%, 0.03% and 0.05%, which corresponds to actual stress of 11, 32 and 53 MPa, respectively. These results are close to those from the in-situ measurement for the continuous loading for Q-Rok sand as shown in Figure 3.12, which on the other hand justifies the repeatability of the measurement of neutron scattering. It's interesting to note that the actual stresses within Q-Rok particulate system based on the lattice strain measured by neutron scattering are close to the applied global stress. This is different from the results of the Ottawa particulate system.

Summary

In this study, an innovative methodology was developed using the in-situ neutron diffraction technique to study particulate materials under laterally confined compression. Local lattice strains in terms of changes in interplanar d-spacing corresponding to the applied external loading were quantified using a customized compression chamber system and in-situ loading system at the 2nd Generation of Neutron Residual Stress Mapping Facility, the High Flux Isotope Reactor, Oak Ridge National Laboratory. The measured global strain includes the combined effect of the elastic strain of individual particles, slippage between particles, crushing and compaction of particles, while the lattice strain only reflects elastic strain within the particles. Significant differences were observed between the global and lattice strain-stress relationships. The measured lattice strain was at least one order of magnitude smaller compared to the global strain when subject to the same externally applied load. For the round Ottawa particles, when the applied load exceeded certain stress, significant particle breakage occurred resulting in a

large increase in the rate of global strain variation as a function applied compressive stress. The elastic strains did not change much when the applied load exceeded the crushing stress and the stress relief occurred due to breakage of particles. For the angular Q-Rok particles, multiple fluctuations in the curve of global stress vs lattice strain were observed, indicating multiple fracturing and stress relief within particles. The elastic stress within the Q-Rok particles are more close to the applied global stress compared to those within Ottawa particles during the loading process. Based on the lattice strain mapping data, there is no evident trend of lattice strains at different applied global stress levels, and the measured lattice strains are scattered, which implies a strong inhomogeneous distribution of the actual stress in particles. The research results presented in this paper will be useful for developing suitable elasto-plastic constitutive models of frictional granular materials, and can also be applied to powder metallurgy and sintered ceramics.

Acknowledgement

The authors would like to acknowledge the support from Joint Institute for Neutron Science (JINS) Fellowship program. Research at Oak Ridge National Laboratory (ORNL) was sponsored by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of FreedomCAR and Vehicle Technologies, as part of the High Temperature Materials Laboratory User Program, ORNL, managed by UT-Battelle, LLC, for the US Department of Energy under contract number DE-AC05-00OR22725.

References

- An, K. and C. R. Hubbard (2006). Neutron Residual Stress Facility (NRSF2) View Technical Manual Oak Ridge, TN, Oak Ridge National Laboratory.
- ASTMD653-97 (2000). Standard Terminology Relating to Soil, Rock, and Contained Fluids, American Society for Testing and Materials. **04.08**.
- Behringer, R. P., D. Howell, et al. (1999). "Predictability and granular materials." Physica D **133**(1-4): 1-17.
- Deresiewicz, H. (1958). "Mechanics of Granular Matter." Advances in Applied Mechanics **5**: 233-306.
- Dutta, A. (2006). Mechanical Properties of Granular Materials Using Nanoindentation and Modeling with Distinct Element Method. **Ph.D.** Dissertation, The University of Tennessee, Knoxville.
- Holtz, R. D. and W. D. Kovacs (1981). An Introduction to Geotechnical Engineering. Englewood Cliffs, Prentice-Hall.
- Kruyt, N. P. (2003). "Contact forces in anisotropic frictional granular materials." International Journal of Solids and Structures **40**(13-14): 3537-3556.
- Lambe, T. W. and R. V. Whitman (1969). Soil Mechanics. New York, NY, John Wiley & Sons.
- Luo, X., D. Penumadu, et al. (2007). Stress-Strain Behavior of Granular Materials under Confined Compression Using Neutron Scattering. ASM International Oak Ridge Chapter Educational Symposium on Neutrons for Materials Science and Engineering, Oak Ridge National Laboratory, Oak Ridge, TN.

- Luo, X., D. Penumadu, et al. (2005). Study of Granular Materials in Compression Using NRSF2. Spallation Neutron Source (SNS) - High Flux Isotope Reactor (HFIR) User Meeting, Oak Ridge, TN, Oak Ridge National Laboratory.
- Majmudar, T. S. and R. P. Behringer (2005). "Contact force measurements and stress-induced anisotropy in granular materials." Nature **435**(7045): 1079-1082.
- Miller, E. T. (1963). Stresses and strains in an array of elastic spheres, Department of Civil Engineering, MIT.
- Otto, M., J. P. Bouchaud, et al. (2003). "Anisotropy in granular media: Classical elasticity and directed-force chain network." Physical Review E **67**(3).
- Scott, R. F. (1963). Principles of Soil Mechanics. Reading, MA, Addison-Wesley Publishing Co.
- Socular, J. E. S., D. G. Schaeffer, et al. (2002). "Directed force chain networks and stress response in static granular materials (vol E 7, pg 353, 2002)." European Physical Journal E **8**(4): 453-453.

Chapter 4 Mapping of Residual Strains in Steel Tubes after Subjected to Either Torsion or Tension Using Reactor Neutron Source

This chapter is a revised version of a publication by Xin Luo and Dayakar Penumadu et al:

X. Luo, D. Penumadu, K. An, C.R. Hubbard, F. Tang, H. Choo, P.K. Liaw, Residual strains of steel tubular specimens after subjecting to torsion and tension, TMS 2007: Linking Science and Technology for Global Solutions, 2007

My primary contributions to this paper include (i) development of the problem into a work, (ii) identification of the study areas and objectives, (iii) design and conducting of the experiments, (iv) gathering and reviewing literature, (v) processing, analyzing and interpretation of experimental data, (vi) pulling various contributions into a single paper, (vii) most of the writing.

Abstract

Residual stress is an important phenomenon that occurs commonly in materials due to thermal or elastic misfit at macro or micro level. Residual strain evolution under 1-D loading, typically uniaxial tension, by using neutron diffraction-based methods has been well studied. However, little research using neutron diffraction has been reported for pure torsion type loading or under 3-D multiaxial loading conditions. Torsion provides a unique opportunity to probe mechanical behaviors of materials under pure shear stress, and in combination with axial load, provides a mechanism to rotate principal stresses in a controlled fashion. Thus, complete 3-D mechanical behavior can be investigated by controlling the major principal stress rotation to characterize anisotropic materials. In this study, identical tubular steel specimens were first subjected to either pure torsion or uniaxial tension that corresponds to a target equivalent strain invariant. These specimens

then were examined using the Second Generation Neutron Residual Stress Mapping Facility (NRSF2) at Oak Ridge National Laboratory (ORNL). Residual strains based on the changes in the lattice spacing were measured in radial, circumferential and axial directions with a gage volume of $1 \times 2 \times 1 \text{ mm}^3$. Two wavelengths of 2.67 and 1.73 Å were used in this study. Results are analyzed for (110) and (211) reflections, reported to be weakly affected by intergranular strain for tension stress path. Results indicate considerable differences in the measured residual strain variation between the tubular specimens subjected to torsion versus tension. This study is the first of its kind for evaluating residual strains under generalized loading conditions, which also helped to demonstrate the need for having an in-situ multi-axial loading system (axial and torsional) for the anticipated residual stress testing facility (Vulcan) at the Spallation Neutron Source (SNS) in Oak Ridge, TN.

Fundamentals of residual stress and strain

Residual stress is one of the more common phenomenon occurring in materials. It arises during material process such as machining, grinding, rolling, heat treatment, and welding. Residual stress can be defined as self-equilibrating internal stress existing in a free body without constraints or external force added on the boundary (Mura 1987). Residual stress is also described as an elastic response of the material to an inhomogeneous distribution of inelastic strains such as plastic strains, precipitation, phase transformation, misfit, or thermal expansion strain (Noyan and Cohen 1987). Residual stress originates from shape misfit between the unstressed shapes of different parts, regions, or phases of the components (Hutchings, Withers et al. 2005).

Typically, from the perspective of the scale of misfit region, residual stresses are divided into three categories: Type I (σ^I), Type II (σ^{II}) and Type III (σ^{III}) stresses, as shown in Figure 4.1. Type I stress is also called macrostress that acts in a region comparable to the macroscopic dimension. Usually, this type of stress extends through the material on a scale of millimeters. Type I stress is often assumed to be continuous from grain to grain and even from phase to phase, and results in a detectable shift of the Bragg peaks. Type II stress acts in the region comparable to grain size of polycrystalline solids, usually a few tens of microns, and are discontinuous from grain to grain. Type III is referred to the stress that acts in the region within specific grains due to situations such as crystal defects of dislocations or vacancies. Both Type II and III have been referred to as microstresses, in which the latter accounts for the broadening of Bragg's peaks.

Residual stress has a significant impact on the mechanical behavior and durability of materials. Residual stress is not immediately apparent in many cases as it is difficult to be measured, predicted and it can lead to premature failure of materials if not considered in the design (Hutchings, Withers et al. 2005). For example, the presence of residual stress can reduce the elastic limit and increase the loss in stress-relaxation in cold drawn wires used in prestressed concrete structures. It is a critical issue to correctly measure and predict the residual stress in order to obtain the correct stress profile and prevent catastrophic failure (Atienza, Ruiz-Hervias et al. 2005). In many case residual stress may also increase the stress corrosion cracking and fatigue, leading to a significant reduction of service life (Okamoto and Nakamura 1990; Berkovits, Kelly et al. 1998; Komai 1998; Tait and Press 2001; Lee, Hwang et al. 2004), although in some cases residual stress does

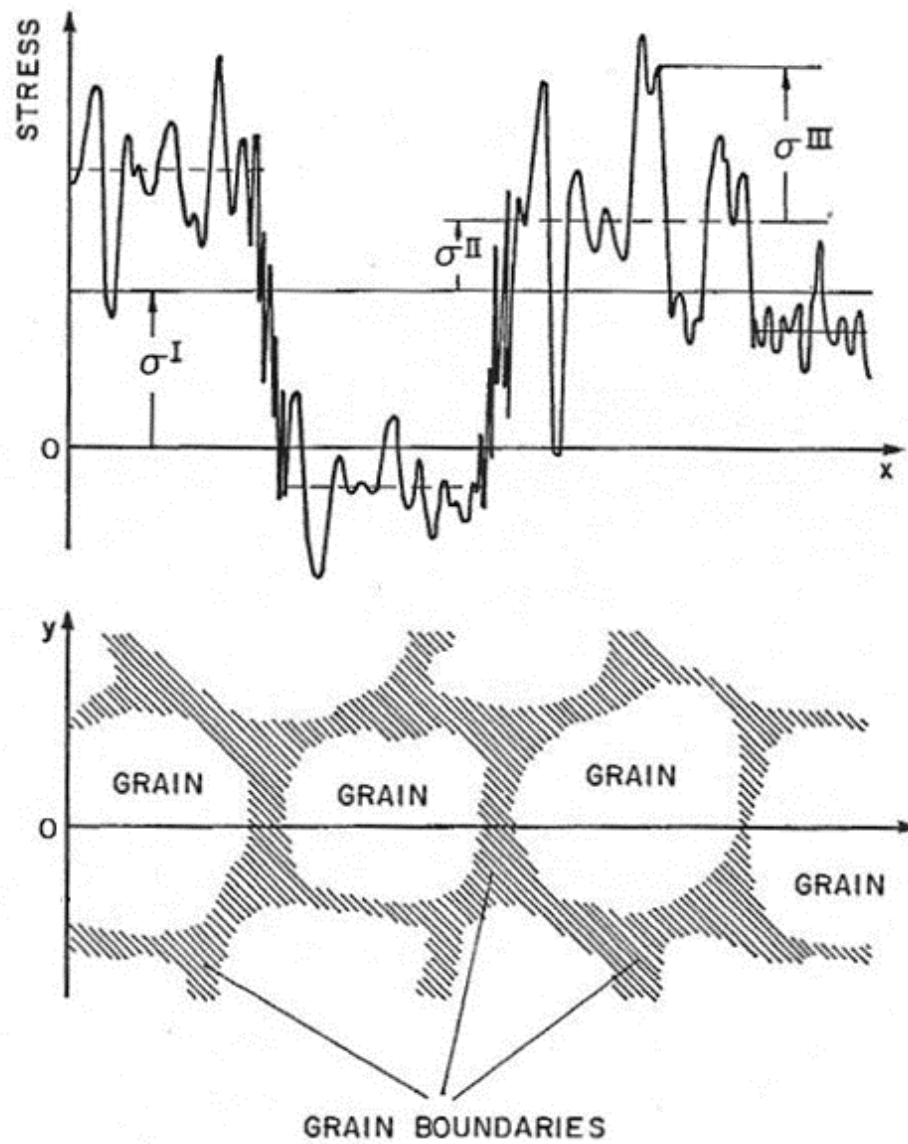


Figure 4.1 Three types of residual stresses (Holden and Bowen 1991)

bring benefits to mechanical properties of materials, such as delaying initiation of fatigue cracking in service (Grum 2003). However, while nearly all neutron diffraction based in-situ loading studies are for uniaxial loads, it is very rare to encounter an engineered component that is only subjected to a simple stress state such as uniaxial tension or compression. Instead, a multi-axial stress state is involved in most practical loading conditions. It is necessary to fully understand the mechanical properties of materials under such a realistic stress state in order to accurately analyze and more efficiently design components.

Generalized loading conditions by tension and torsion

Multi-axial situation can be achieved in the laboratory by applying internal pressure and uniaxial load on tubular samples, allowing independent control of the mean stress responsible for volume change and octahedral shear stress for shear distortions (Crawford and Lesser 2000). An alternative approach to achieve the multi-axial stress states is to use a combination of axial and torsional loading. This approach also comes with an extra advantage that allows the rotation of the major principal stress components with respect to the axial direction by applying normal and shear stress components simultaneously for a sample under isotropic confining stress. Vertical load and torque are applied simultaneously and the specimen is subject to a three-dimensional (3-D) stress state, as shown in Figure 4.2 (Saada 1988; Lin and Penumadu 2006), in which σ is the hydrostatic pressure, $\Delta\sigma$ is the change of the normal stress due to the axial load, and $\Delta\tau$ is the change of the shear stress due to the torque.

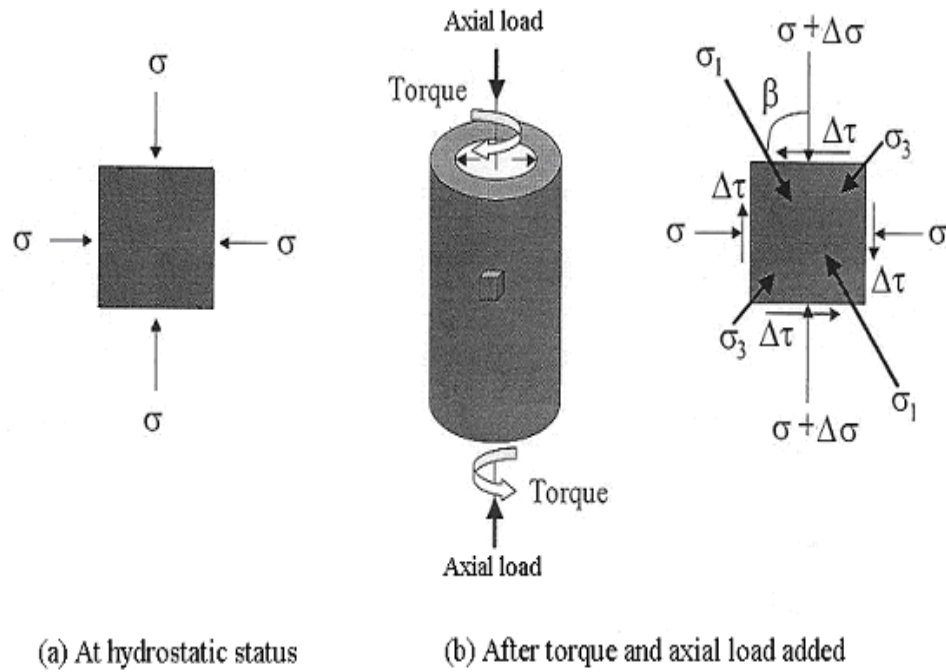


Figure 4.2 Control of directions of principle stresses through torsion-axial loading system

Because of the presence of the shear stress, the major and minor principal stresses rotate. The angle between the direction of the major principal stress and the axial direction is defined as β , and it can be obtained using the following Equation (4.1) (Lin and Penumadu 2006).

$$\tan(2\beta) = \frac{2\Delta\tau}{\Delta\sigma} \quad (4.1)$$

Typically, the situation when $\beta = 0$ and $\Delta\tau = 0$ represents triaxial compression test; the situation when $\beta = \frac{\pi}{2}$ and $\Delta\tau = 0$ represents triaxial extension test; and the situation when $\beta = \frac{\pi}{4}$ and $\Delta\sigma = 0$ represents pure torsion test. β other than 0, $\frac{\pi}{4}$, $\frac{\pi}{2}$ represents the situation where the specimens are subjected to a combination of axial and torsion loading simultaneously. Thus, complete three-dimensional mechanical behavior can be investigated by controlling the major principal stress rotation to characterize anisotropic materials.

Strain or stress measurement with diffraction method

Diffraction based stress analysis utilizes the interplanar d-spacing, i.e., the distance between certain atomic planes, as an internal strain gage. For any reflection (hkl), the lattice d-spacing can be determined by Bragg's Law as shown in Equation (4.2).

$$\lambda = 2d_{hkl} \sin(\theta_{hkl}) \quad (4.2)$$

where, λ is the wavelength of the radiation; θ_{hkl} is the angle of Bragg's peak of the reflection (hkl), and d_{hkl} is the interplanar d-spacing of the reflection (hkl).

Unlike X-rays interacting with the electron shells of the atoms, neutrons interact with atomic nuclei and have the power of penetration that is two or three orders of magnitude larger for most metallic materials than that of X-rays. Especially, in stress analysis, the X-ray technique is often limited to the region quite near to the sample surface while the large penetration power of neutrons provides a unique tool for probing strains and stresses in the bulk of materials in a non-destructive fashion.

The strain in terms of d-spacing change for a given (hkl) reflection can be defined as shown in Equation (4.3).

$$\varepsilon = \frac{d_{hkl} - d_0}{d_0} \quad (4.3)$$

where, ε is the lattice strain, d_0 is the interplanar spacing under stress free conditions. Substituting the Equation (4.2) to (4.3) yields (4.4)

$$\varepsilon = \frac{\sin(\theta_0)}{\sin(\theta_{hkl})} - 1 \quad (4.4)$$

where, θ_0 is the angle of Bragg's peak under stress free conditions

For the tensile strain, d is greater than d_0 , θ_{hkl} is smaller than θ_0 , which means the Bragg's peak shifts to smaller angle; for the compressive strain, d is smaller than d_0 , θ_{hkl} is larger than θ_0 , which means the Bragg's peak shifts to larger angle.

It should be noted that the strain is actually an average value based on the strains measured from all properly oriented grains within the gage volume, defined by the intersection of the incident and diffracted beams paths. Neutron scattering has been used extensively in the past several decades for (hkl) dependent residual strain measurements

in specimens subjected to 1-D loading (McClinton and Cohen 1982; Gurova and Teodosio 1997; Gurova and Teodosio 1998; Prime 1999; Kuboki 2001), typically in uniaxial tension or compression. To the authors' knowledge, residual strain or stress under pure torsion or 3-D loading conditions using neutron scattering has not been done yet. As stated earlier, study on torsion provides a unique opportunity to probe mechanical behavior of materials under pure shear stress, and in combination with axial load, provides a mechanism to rotate principal stresses in a controlled fashion. Thus, complete three-dimensional mechanical behavior can be investigated by controlling the major principal stress rotation to characterize anisotropic materials.

Details of strain measurements using neutron diffraction

Tubular specimens made of 12L14 steel with an outer diameter of 10.8 mm, an inner diameter of 6.8 mm, were used in the study. The 12L14 steel is an essentially resulfurized, rephosphorized, free-cutting carbon steel. The compositions and mechanical properties are listed in Table 4.1 - Table 4.2. The experiments were carried out using the second generation neutron residual stress mapping facility (NRSF2) at Oak Ridge National Laboratory (ORNL) (An and Hubbard 2006).

The interplanar d-spacings were measured using a gage volume 1 mm $\times$ 1 mm in the scattering plane and 2 mm tall in the vertical direction. The gage volume position was moved from the outer to inner surface with an increment of translation in gage volume center by 0.25 mm, as shown in the Figure 4.3 - Figure 4.5.

The crystal structure of BCC iron and the chosen hkl reflections, namely, (110) and (211), for this neutron scattering study, are shown in Figure 4.6. The neutron

Table 4.1 Typical compositions of 12L14 steel (ASM 1990)

AISI Number	Chemical compositions (%)				
	C max	Mn	P	S	Pb
12L14	0.15	0.85-1.15	0.04-0.09	0.26-0.35	0.15-0.35

Table 4.2 Mechanical properties of 12L14 steel (ASM 1990)

AISI Number	Mechanical properties				
	Tensile strength (MPa)	Yield point (MPa)	Elongation (%)	Reduction in area (%)	Brinell hardness
12L14	603	517	15	50	163/179

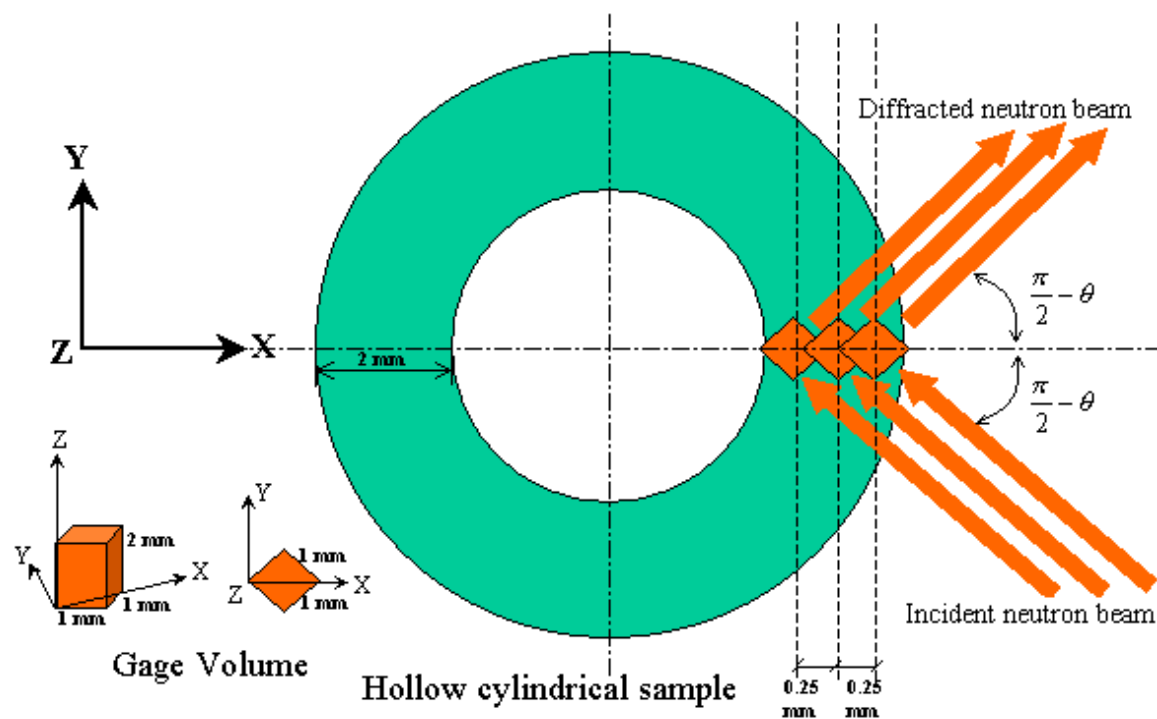


Figure 4.3 Diffraction measurements for radial strains

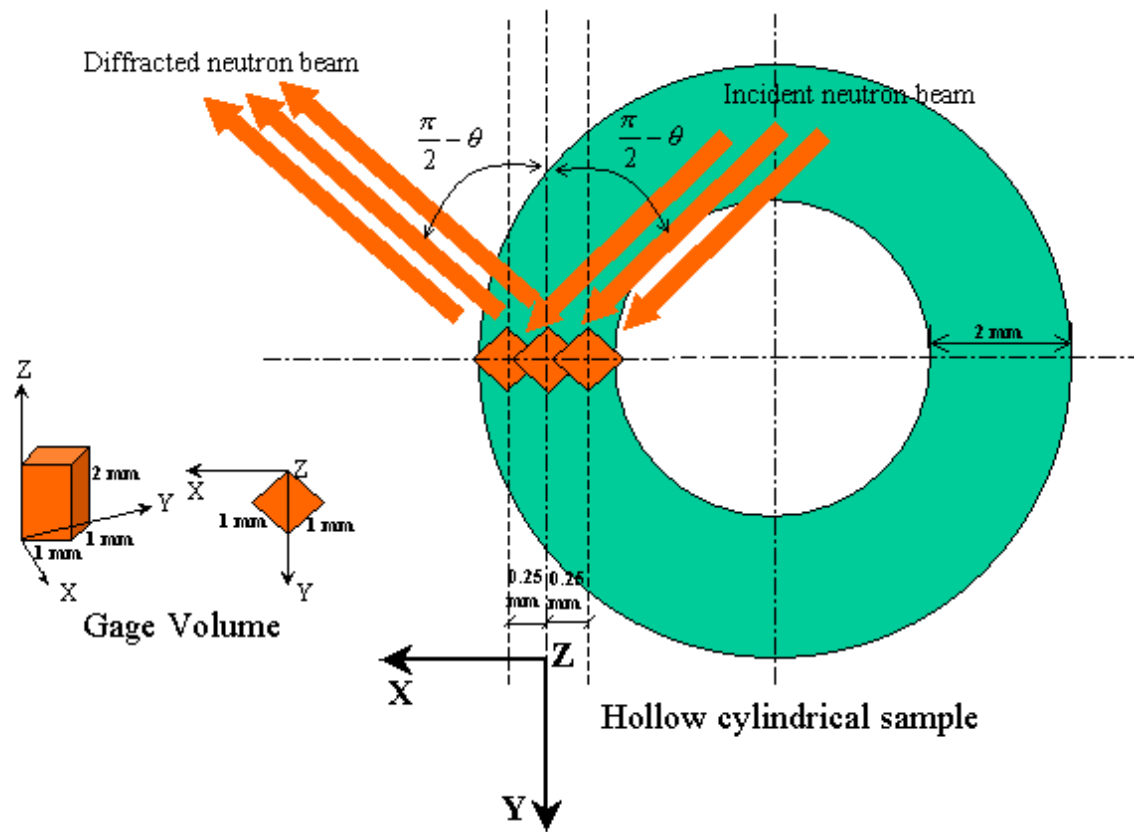


Figure 4.4 Diffraction measurements for circumferential strains

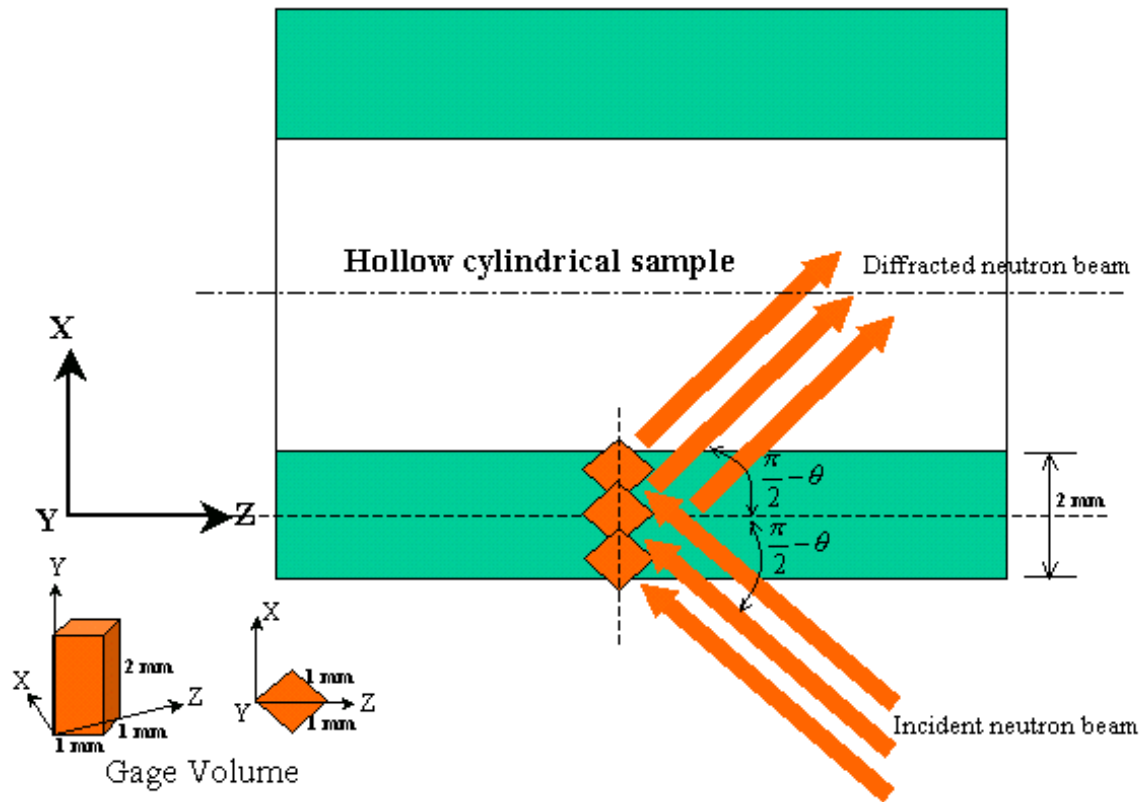


Figure 4.5 Diffraction measurements for axial strains

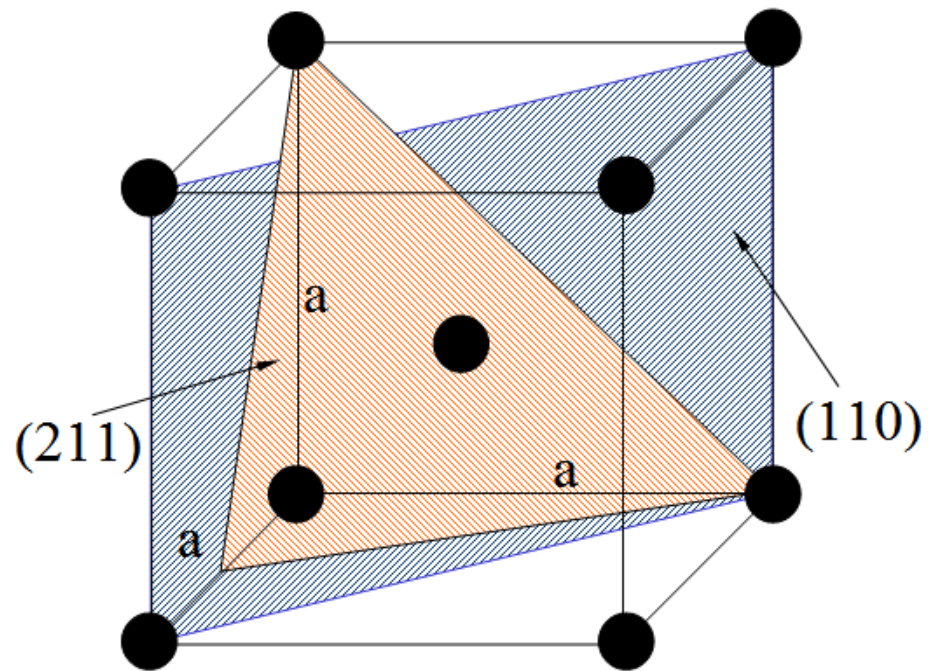


Figure 4.6 BCC Fe and the lattice planes used in the neutron diffraction

scattering parameters including the Si monochromator settings (Stoica, Popovici et al. 1999), the wavelengths of the neutron beams, and the nominal Bragg angles for the chosen lattice planes of iron are listed in Table 4.3.

The stress-free d-spacing (d_0) was obtained by mapping the d-spacing in the radial, circumferential, and axial directions, respectively, through the wall-thickness of the as-received tubular sample without subject to either torsion or tension. Averaged values of d_0 in the pertinent directions were used. The experimental results of the reflections (110) and (211), both of which have been reported as weakly affected by intergranular strain (Hutchings, Withers et al. 2005), will be discussed in order to explore and reveal the essential difference in residual strain between the specimens subjected to uniaxial tension and pure torsion.

Table 4.3 Neutron scattering parameters used in the strain mapping

Monochromators	Neutron wavelength λ (Å)	Bragg's Peak 2θ (°)	Lattice planes
Si220	2.67	82.23	Fe (110)
Si331AF	1.73	95.37	Fe (211)

Results of residual strain mapping and discussion

The tubular specimens were first subjected to either pure torsion or uniaxial tension that corresponds to a similar target value of similar deviatoric strain, which is essentially the octahedral shear strain as given by Equation (4.5) (Saada 1983).

$$\gamma_{oct} = \frac{2}{3} \sqrt{(\varepsilon_{11} - \varepsilon_{22})^2 + (\varepsilon_{22} - \varepsilon_{33})^2 + (\varepsilon_{33} - \varepsilon_{11})^2 + 6(\varepsilon_{12}^2 + \varepsilon_{23}^2 + \varepsilon_{13}^2)} \quad (4.5)$$

where,

γ_{oct} – octahedral shear strain

ε_{ij} –the components of strain tensor (i, j =1, 2, 3)

For torsion samples, the average shear stress and strain are calculated with the assumption that work done by the applied torques is equal to the sum of the work done by the stresses and strains involved (Saada 1988). In addition, the average shear strain for the hollow cylinder is given by the Equation (4.6) (Saada 1988).

$$\gamma_{\theta z} = \frac{2\theta(R_o^3 - R_i^3)}{3H(R_o^2 - R_i^2)} \quad (4.6)$$

where,

R_o, R_i – Outer and inner radius of the hollow sample cylinder, mm

θ – twisted angle, rad

H – twisted length, mm

In this study, the laboratory stress-strain history for the tubular specimens is shown in Figure 4.7 - Figure 4.8 (Penumadu, Luo et al. 2005; Luo, Penumadu et al. 2006), and the experimental parameters are summarized in Table 4.4. For tension samples, the maximum tensile strains were reached in the uniaxial tension test. As to

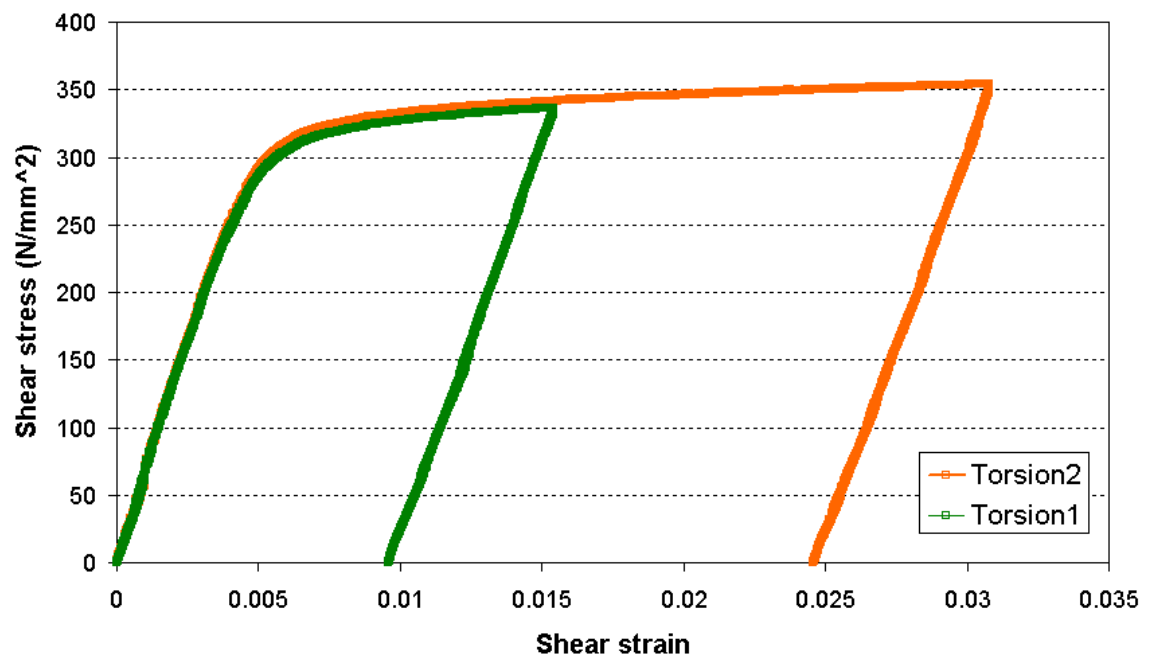


Figure 4.7 Stress-strain history of the tubular specimen for the torsion test

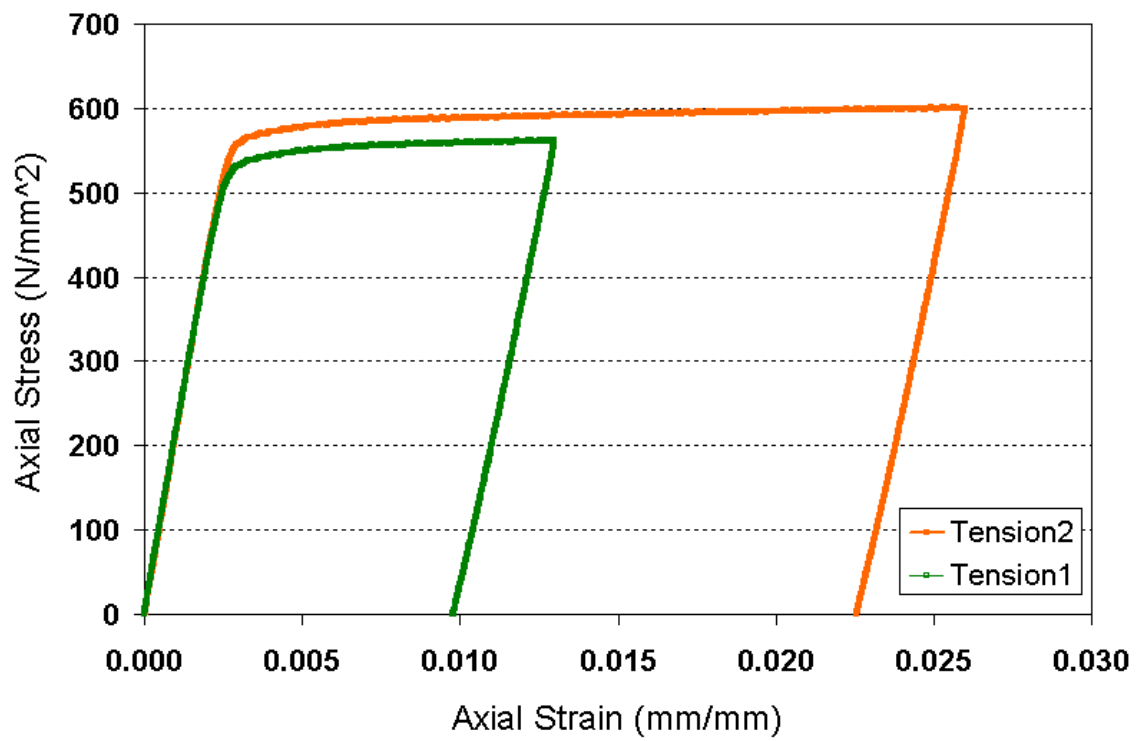


Figure 4.8 Stress-strain history of the tubular specimen for the tension test

Table 4.4 Experimental parameters of the two levels of loading for tension and torsion

Loading history	Tension samples		Torsion samples	
	Tensile strain	Octahedral shear strain	Twisted angle (deg)	Octahedral shear strain
Loading 1	0.0130	0.0123	10.0	0.0121
Loading 2	0.0260	0.0245	20.0	0.0242

torsion samples, the twisted length is 52.8 mm, and the maximum twisted angles were reached. The corresponding octahedral shear strains are calculated based on the Equation (4.5).

Subsequently, the specimens were studied using the Neutron Residual Stress Mapping Facility 2 (NRSF2) at Oak Ridge National Laboratory (ORNL). The residual strains of the tubular steel samples in the axial direction are given in Figure 4.9 - Figure 4.10, in which the horizontal axis represent the distance from the inner surface of the tubular specimens, and the vertical axis represent the lattice strain calculated using Equation (4.3). The residual strains from the two torsion samples are quite close, although they were subject to two different levels of ultimate shear strains in torsion. The maximum shear strain of the sample Torsion 2 is 0.0296, twice as large as that of the sample Torsion 1. A similar situation occurs for the two tension samples (Tension 1 and 2). However, there is significant difference between the tension and torsion samples in terms of their residual strains. For the (110) reflection, the residual strains are quite different for the tension and torsion samples. Compared to the torsion samples which have small residual strains in tension, the tension samples have considerable residual strains around 400 $\mu\epsilon$ in compression. In addition, the residual strains for both tension and torsion samples are uniform through out the wall thickness of the samples. For the reflection of (211), the differences in residual strains between tension and torsion samples were reduced in general. At the middle of the wall thickness, the two tension samples show slight compressive residual strain around 50 $\mu\epsilon$, and the two torsion samples show slight tensile residual strain around 50 $\mu\epsilon$.

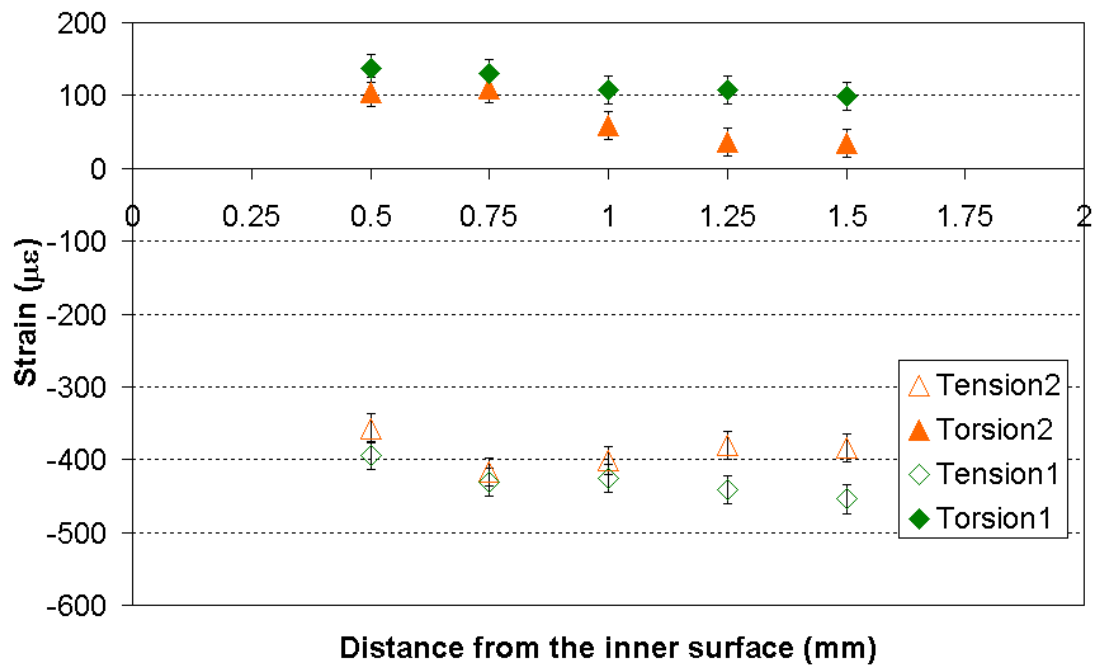


Figure 4.9 Residual strains in axial direction for the reflection (110) for samples subject to the same invariant strain in torsion or tension

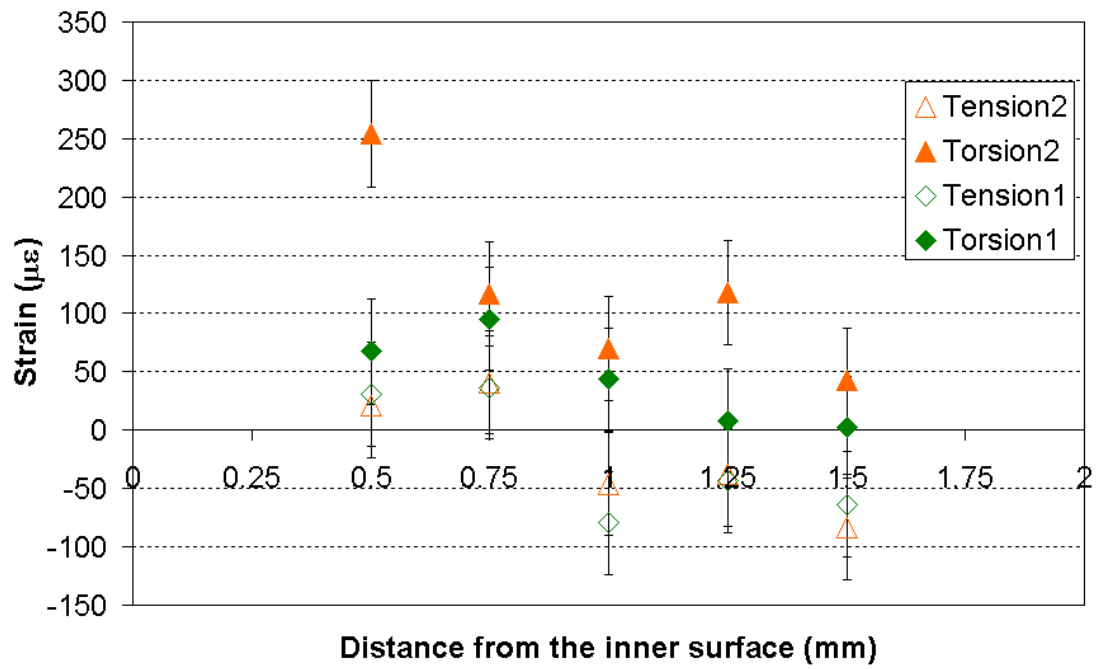


Figure 4.10 Residual strains in axial direction for the reflection (211) for samples subject to the same invariant strain in torsion or tension

The residual strains of the tubular steel samples in the circumferential direction are given in Figure 4.11 - Figure 4.12. The tubular samples exhibit a similar variation of their circumferential residual strains for both (110) and (211) reflections. A considerable change from tension to compression strain for the (110) reflection is observed for both of the samples as the distance from the inner surface increases. However, the patterns of the circumferential residual strains based on the (211) reflection are different for tension and torsion samples. There is a similarity between tension and torsion samples after subjected to the Loading 2, but remarkable difference between tension and torsion samples after subjected to Loading 1 exists.

The residual strains of the tubular samples in the radial direction are given in Figure 4.13 - Figure 4.14. The residual strains in torsion samples after subjected to two different loading histories are quite close to each other, so are those of tension samples. However, for the (110) reflection, the difference in residual strains between tension and torsion samples is considerable in the radial direction. Similar to the situation in axial direction, the residual strains are compressive for the tension samples, while there are not considerable residual strains left for the torsion samples. For the reflection (211), both tension and torsion samples exhibit similar strain patterns and those strains trend from compression to tension when the measurement locations move from near the inner surface toward the outer surface.

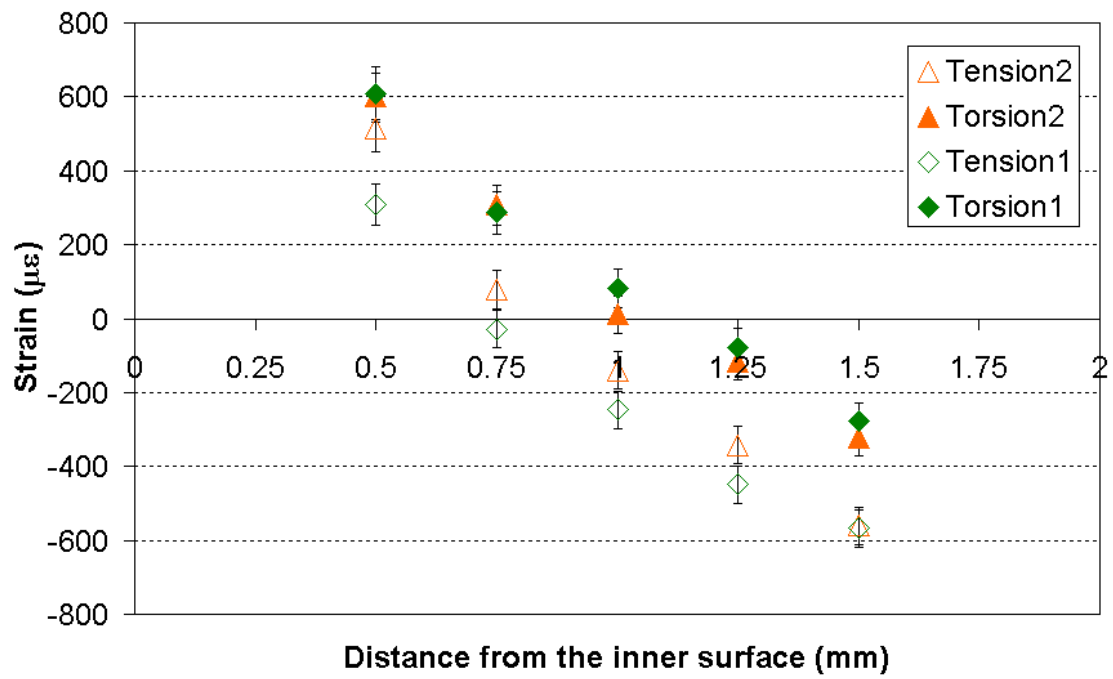


Figure 4.11 Residual strains in circumferential direction for the reflection (110)

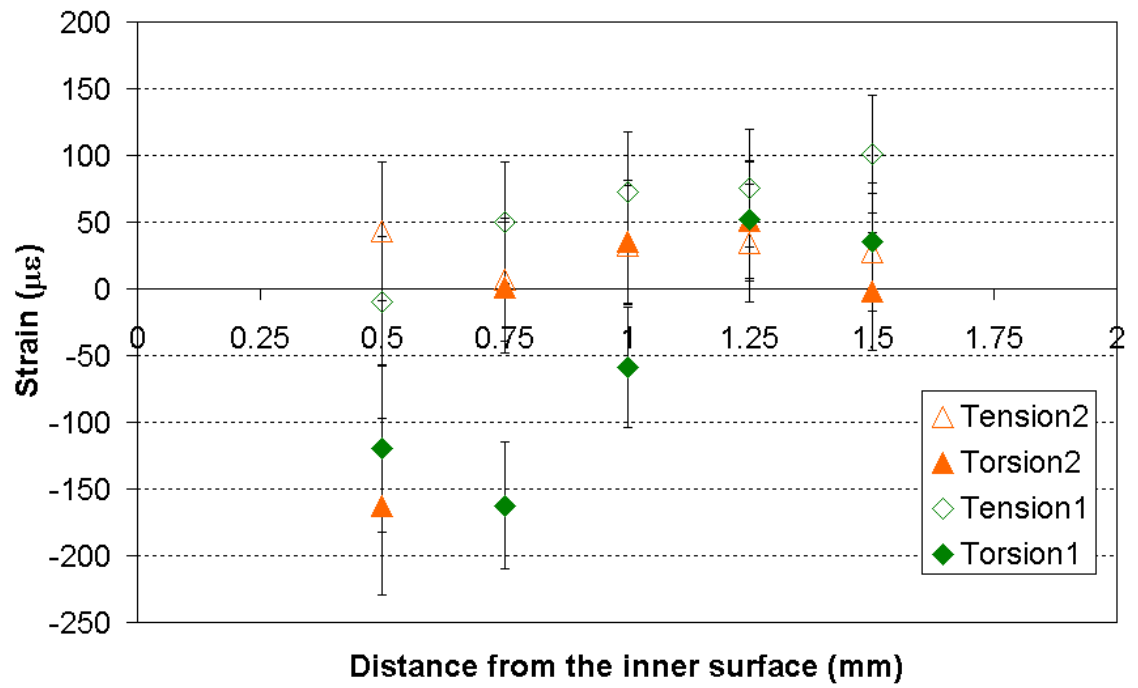


Figure 4.12 Residual strains in circumferential direction for the reflection (211)

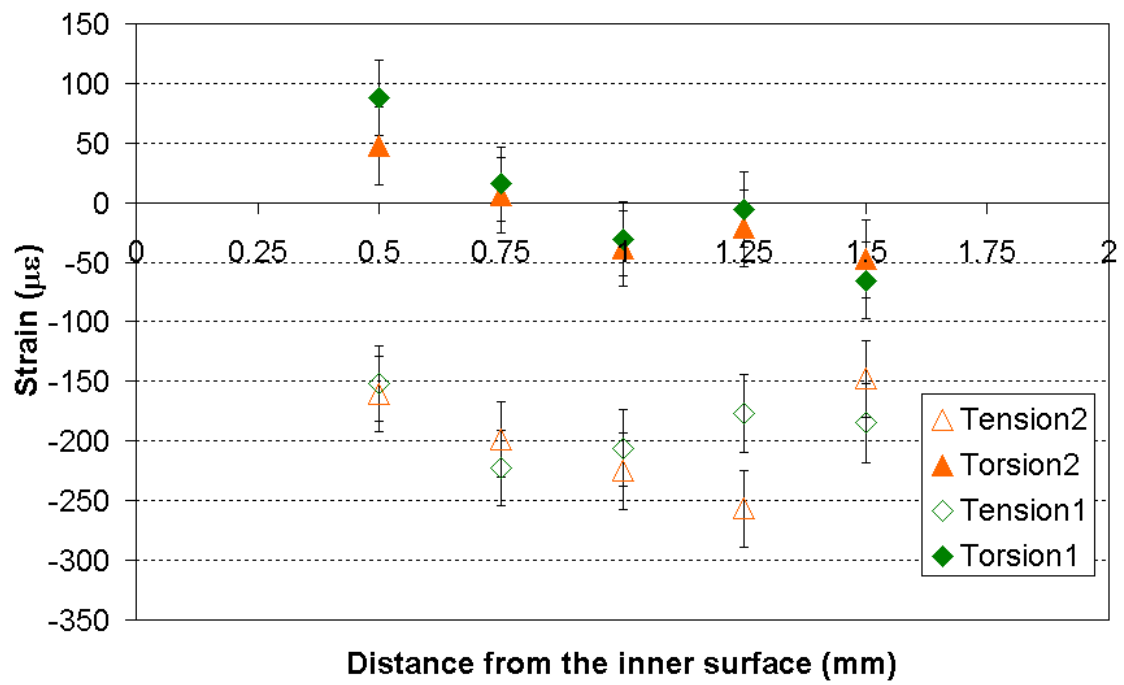


Figure 4.13 Residual strains in radial direction for the reflection (110)

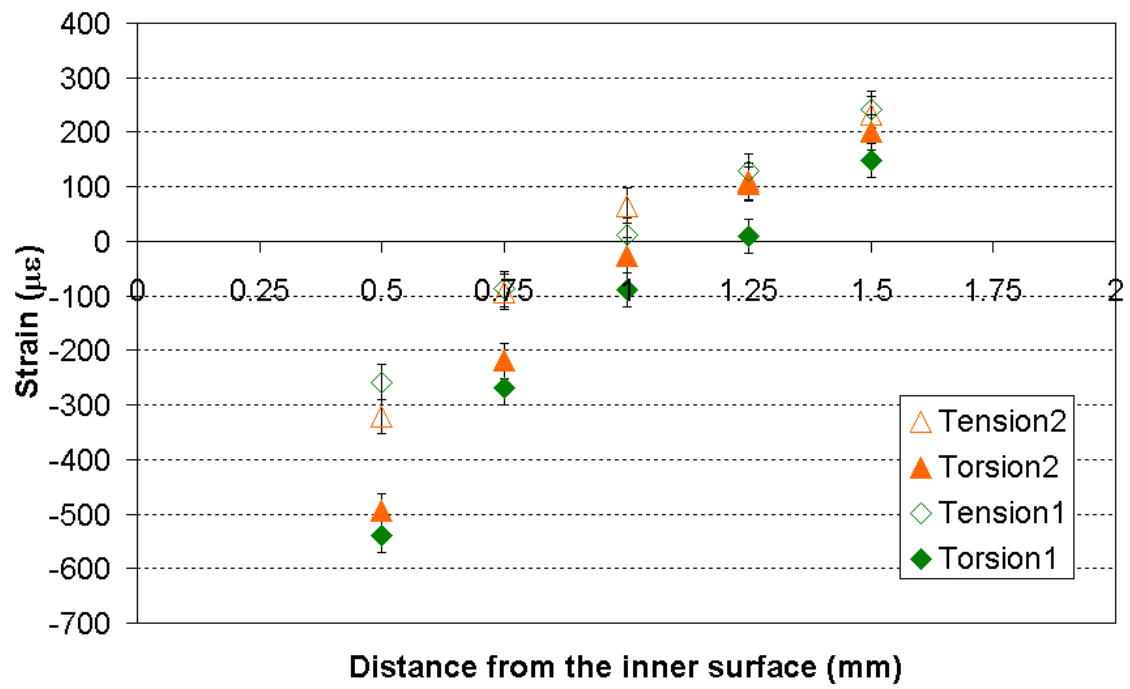


Figure 4.14 Residual strains in radial direction for the reflection (211)

Summary

Residual stress has a significant impact on the mechanical behavior materials. It is difficult to be measured or predicted using analytical methods, and can lead to premature failure of materials if not appropriately considered in design. A nondestructive strain mapping method based on neutron diffraction was used to obtain three orthogonal lattice strain values for the tubular steel specimens subjected to either tension or torsion using the Second Generation Neutron Residual Stress Mapping Facility (NRSF2) at Oak Ridge National Laboratory. The (110) and (211) reflections, which have been reported as weakly affected by intergranular strain based on tension tests, were used to study the effect of tension versus torsion on the residual strains for the carbon steel after being subjected to the same target strain invariant. Similarities in the residual strain at various spatial locations were observed for the tension and torsion samples after they were exposed to different loading levels. Significant differences in the residual strains of the given reflections were found and discussed for torsion versus tension tests. The analysis of the differences will require further experiments and modeling. Most importantly, this study demonstrates the need for an in-situ tension-torsion loading system for studying materials under realistic loading conditions.

Acknowledgement

The authors would like to thank the support from the NSF IMI program (DMR-0231320) and JINS Fellowship program. Research at Oak Ridge National Laboratory (ORNL) was sponsored by the Assistant Secretary for Energy Efficiency and Renewable

Energy, Office of FreedomCAR and Vehicle Technologies, as part of the High Temperature Materials Laboratory User Program, ORNL, managed by UT-Battelle, LLC, for the US Department of Energy under contract number DE-AC05-00OR22725.

References

- An, K. and C. R. Hubbard (2006). Neutron Residual Stress Facility (NRSF2) View Technical Manual Oak Ridge, TN, Oak Ridge National Laboratory.
- ASM (1990). Metals handbook (10th ed.) Volume 1 Properties and Selection: Iron, Steel, and High Performance Alloys. Materials Park, OH, ASM International.
- Atienza, J. M., J. Ruiz-Hervias, et al. (2005). "Residual stresses in cold drawn pearlitic rods." Scripta Materialia **52**(12): 1223-1228.
- Berkovits, A., D. W. Kelly, et al. (1998). "Considerations of the effect of residual stresses on fatigue of welded aluminium alloy structures." Fatigue & Fracture of Engineering Materials & Structures **21**(2): 159-170.
- Crawford, E. and A. J. Lesser (2000). "Mechanics of rubber particle cavitation in toughened polyvinylchloride (PVC)." Polymer **41**(15): 5865-5870.
- Grum, J. (2003). "Analysis of residual stresses in main crankshaft bearings after induction surface hardening and finish grinding." Journal of Automobile Engineering(217): 173-182.
- Gurova, T. and J. R. Teodosio (1997). "Study of the residual stress state during plastic deformation under uniaxial tension in a 5.0Cr and 0.5Mo steel." Scripta Materialia **36**(9): 1031-1035.
- Gurova, T. and J. R. Teodosio (1998). "Model for the variation of the residual stress state during plastic deformation under uniaxial tension." Journal of Strain Analysis for Engineering Design **33**(5): 367-372.
- Holden, T. M. and A. W. Bowen (1991). Interpretation of Residual Stress Measurements: Summary of Discussion Session. NATO Advanced Research Workshop on

Measurement of Residual and Applied Stress Using Neutron Diffraction, Kluwer Academic Publishers.

Hutchings, M. T., P. J. Withers, et al. (2005). Introduction to the characterization of residual stress by neutron diffraction. Boca Raton, CRC Press.

Komai, K. (1998). "Failure analysis and prevention in SCC and corrosion fatigue cases." International Journal of Fatigue **20**(2): 145-154.

Kuboki, T. (2001). "Influence of back-tension on effect of skin pass in minimising residual stress levels after cold rod drawing." Ironmaking & Steelmaking **28**(2): 117-121.

Lee, H. C., Y. H. Hwang, et al. (2004). "Failure of aircraft propeller assembly." Engineering Failure Analysis **11**(3): 305-312.

Lin, H. and D. Penumadu (2006). "Strain localization in combined axial-torsional testing on kaolin clay." Journal of Engineering Mechanics-Asce **132**(5): 555-564.

Luo, X., D. Penumadu, et al. (2006). Residual strain evolution in 3-D stress conditions under torsion and tension for steel using a spallation neutron source. American Conference on Neutron Scattering, St. Charles, NSSA.

McClinton, M. and J. B. Cohen (1982). "Changes in Residual-Stress During the Tension Fatigue of Normalized and Peened Sae 1040 Steel." Materials Science and Engineering **56**(3): 259-263.

Mura, T. (1987). Micromechanics of Defects in Solids. Hague, the Netherlands, Martinus Nijhoff Publishers.

Noyan, I. C. and J. B. Cohen (1987). Residual Stress Measurement by Diffraction and Interpretation. New York, NY, Springer-Verlag.

- Okamoto, A. and H. Nakamura (1990). "The Influence of Residual-Stress on Fatigue Cracking." Journal of Pressure Vessel Technology-Transactions of the ASME **112**(3): 199-203.
- Penumadu, D., X. Luo, et al. (2005). Residual Strains Under Torsion Using Hollow Cylinder Steel Specimens and Neutron Diffraction. Spallation Neutron Source (SNS) - High Flux Isotope Reactor (HFIR) User Meeting, Oak Ridge, TN, Oak Ridge National Laboratory.
- Prime, M. B. (1999). "Measuring residual stress and the resulting stress intensity factor in compact tension specimens." Fatigue & Fracture of Engineering Materials & Structures **22**(3): 195-204.
- Saada, A. S. (1983). Elasticity:Theory and Applications. Malabar, FL, Krieger Publishing.
- Saada, A. S. (1988). Hollow Cylinder Torsional Devices: Their Advantages and Limitations. Advanced Triaxial Testing of Soil and Rock. R. T. Donaghe, R. C. Chaney and M. L. Silver. Philadelphia, PA, American Society for Testing and Materials: 766-795.
- Stoica, A. D., M. Popovici, et al. (1999). Neutron Monochromators for Residual Stress Mapping at the New HB-2 Beamport. Oak Ridge National Laboratory.
- Tait, R. B. and J. Press (2001). "An experimental study of the residual stresses, and their alleviation, in tube to tube-sheet welds of industrial boilers." Engineering Failure Analysis **8**(1): 2001.

**Chapter 5 Residual Strains in Steel Tubes After Application of Either
Torsion or Tension Using Spallation Neutron Source**

Introduction to residual stress

Residual stress can be defined as the stresses remaining in the body of a component after processing or manufacture in the absence forces. Residual stress can be classified in several ways based on causes, scales, or measurement methods (Hutchings, Withers et al. 2005). Mechanical residual stresses are produced during various manufacturing processes such as machining, heat treatment, and deformation processing (Rudd 1992; Borland 1997; Withers and Bhadeshia 2001). The magnitude and distribution of residual stress sometimes is a key factor to design engineering components. In most of cases, tensile residual stress in surface or near surface region in a component is undesirable, because this kind of tensile residual stress might be the major reason for corrosion cracking or fatigue failure (Clapham, Krause et al. 1997; Hayashi, Ishiwata et al. 2000). Other examples of undesirable surface tensile residual stresses include deep deformation operation such as rod or wire drawing, turning or milling operation such as welding, and harsh conditions such as grinding. Residual stresses could result from non-uniform thermal process such as heating or cooling. Quenching of metallic materials may lead to very high compressive residual stresses on the surface, which is usually beneficial to the increase of resistance to fatigue and stress-corrosion cracking (Inoue, Kawashima et al. 2000). Residual stresses also can develop due to volume change relating to precipitation or phase transformation. For instance, some surface treatment or coating by chemical processes can cause substantial residual stresses in the surface layers in an engineering component. A very high compressive strength of the order of 6 – 8 GPa or higher was measured at the interface of some thermal barrier coatings (Kandil, Lord et al. 2001).

From the perspective of the region or length scale where the stresses act, there are two kinds of residual stresses, one of which is macrostresses, and the other is microstresses (Holden and Bowen 1992; Withers and Bhadeshia 2001). The macrostresses are also referred as Type I residual stresses, which vary and self-equilibrate within the body of a component over a range much larger than grain size. Microstresses resulted from differences within microstructure of materials can be further classified as Type II and Type III residual stresses. Type II is also referred to as intergranular stresses, which vary on the scale of grain size (Withers and Bhadeshia 2001). Such stresses are expected to exist either in single-phase materials due to anisotropy in behavior of each grain or in multi-phase materials due to the different properties of the different phases. Type III is referred to the residual stresses existing within a grain as a result of the crystal defects such as dislocation.

X-ray and neutrons are usually used as non-destructive tools to measure stress or even stress profile in crystalline materials in terms of changes in Bragg's peaks (Noyan and Cohen 1987; Jones 1989; Lu and Rehr 1998; Cullity and Stock 2001). Unlike traditional X-ray, neutrons have a significantly larger penetrating depth in most of the engineering materials, because they interact with atomic nuclei and are independent of atomic number, which makes neutrons a unique tool to acquire otherwise inaccessible information during strains probing in the interior of bulk materials in a non-destructive way (Hutchings 1992). As the non-destructive measurement of residual stress using X-ray or neutrons is based on the shifts of the Bragg's peaks, some special cautions need to be considered. For example, stacking fault density can be changed by plastic

deformation, and solute could be removed by heat treatment (Krawitz and Winholtz 1994). All of those could shift the Bragg's peaks, leading to errors in stress calculation and estimate.

Diffraction based measurement using spallation neutron source

Like other diffraction based methods, the time-of-flight (TOF) can be described by Bragg's law, as given in Equation (5.1).

$$\lambda = 2d \sin(\theta) \quad (5.1)$$

where,

λ – The wavelength of the radiation

θ – The angle of Bragg's peak of the reflection (hkl)

d – The interplanar d-spacing of the reflection (hkl)

For a spallation neutron source with a fixed angle and multiple wavelengths, the following holds:

$$\Delta\lambda = 2\Delta d \sin(\theta) \quad (5.2)$$

Substituting Equation (5.1) for $\sin(\theta)$

$$\varepsilon = \frac{\Delta d}{d} = \frac{\Delta\lambda}{\lambda} \quad (5.3)$$

where,

ε – The lattice strain

The TOF technique is base on the combination of the wavelength-sorting with position-sensitive detectors (PSDs). The neutrons produced at an accelerator-based pulsed neutron source are created at a known time (t_0) at which the high energy particle

beam hits the neutron-producing target. As a consequence of the nature of neutrons, the velocity of each neutron is related to its wavelength. By recording the arrival time (t) of each neutron, and having a known distance from the source to the detectors, it is possible to relate the time-of-flight (t-t₀) to the velocity (v), and hence to the wavelength (λ).

The momentum of the neutron is related to its mass, velocity, and wavelength, as given in Equation (5.4) (Hutchings, Withers et al. 2005).

$$p = mv = \frac{h}{\lambda} \quad (5.4)$$

where,

p, m, and v – Momentum, mass, and velocity of the neutron, respectively

h – Plank's constant.

$$v = \frac{L}{t} \quad (5.5)$$

where,

L – The total flight path distance between the moderator and detector

t – The time of the flight, the time from the instant at which the pulse of neutron is generated in the moderator to the instant at which the neutron is captured in the detector

Substituting Equation (5.5) into (5.4) yields

$$\lambda = \frac{ht}{mL} \quad (5.6)$$

For a given reflection (hkl), the lattice strain is obtained by substituting Equation (5.6) into (5.3)

$$\varepsilon_{hkl} = \frac{\Delta t_{hkl}}{t_0} = \frac{t_{hkl} - t_0}{t_0} \quad (5.7)$$

where, t_{hkl} – The measured TOF for the reflection (hkl) of the stressed sample

t_0 – The measured TOF for the reflection (hkl) of the reference stress-free sample

This also shows that the strain measured at a spallation neutron source is a function of the change in TOF of the diffracted beam with a fixed angle θ and flight path length L .

Brief information of the LANSCE user facility was given by Lisowski and Jones (2002). The spallation neutrons are produced by an 800-MeV linear accelerator system that accelerates up to 100 kW of negative hydrogen ions with pulsed beam timing patterns suitable for a wide variety of experimental programs. At the Manuel Lujan Jr. Neutron Scattering Center sixteen flight paths utilize pulsed cold, thermal and epithermal neutrons produced at 20Hz by intense 0.13 μ s bursts of protons incident on a tungsten spallation target and moderated by water or liquid hydrogen.

Materials and stress-strain history of steel tubular samples

Hollow cylindrical samples were prepared from steel 12L14, which is an essentially resulfurized, rephosphorized, free-cutting carbon steel with lead added. The steel met the chemistry requirements of AISI 12L14, namely, C max 0.15 wt%, Mn 0.8-1.5 wt%, P 0.04-0.09 wt%, S 0.26-0.35 wt% and Pb 0.15-0.35 wt%. This steel also shows the characteristics of a yield point of 517 MPa, tensile strength of 603 MPa, and elongation of 15%. The geometry of the samples is a hollow cylinder, with outer diameter (O.D.) and inner diameter (I.D.) of 10.8 and 6.8 mm, respectively.

The stress-strain history for the tension and torsion samples are shown in Figure 5.1 and Figure 5.2 (Luo, Penumadu et al. 2006). Those tubular steel samples were subject

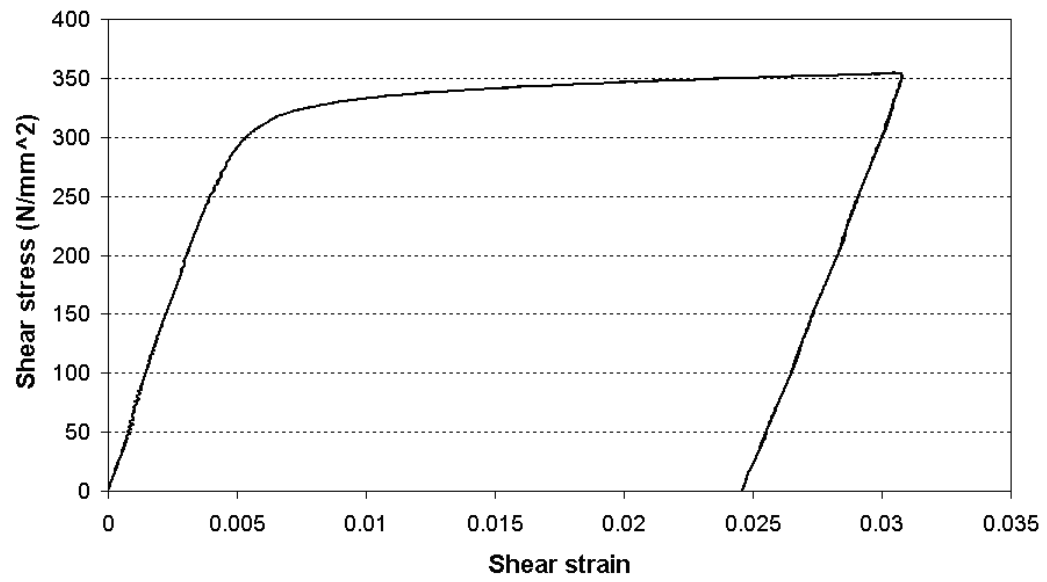


Figure 5.1 Stress-strain history of the sample for the torsion test

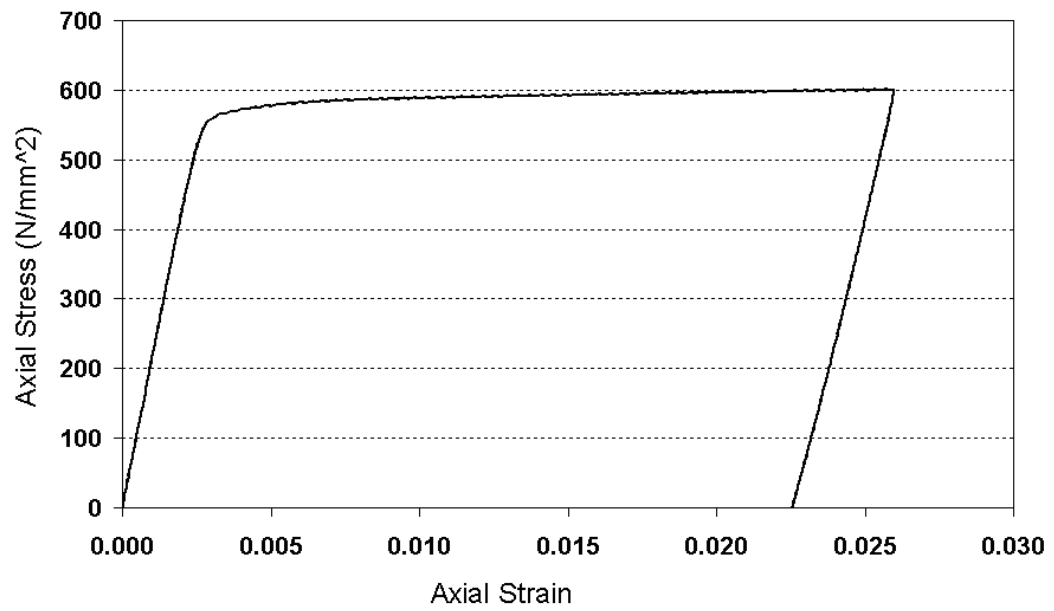


Figure 5.2 Stress-strain history of the sample for the tension test

to either tension or torsion first, that corresponds to a target value of invariant strain, i.e., the octahedral shear strain, as given in Equation (5.8) (Saada 1983).

$$\gamma_{oct} = \frac{2}{3} \sqrt{(\varepsilon_{11} - \varepsilon_{22})^2 + (\varepsilon_{22} - \varepsilon_{33})^2 + (\varepsilon_{33} - \varepsilon_{11})^2 + 6(\varepsilon_{12}^2 + \varepsilon_{23}^2 + \varepsilon_{13}^2)} \quad (5.8)$$

where,

γ_{oct} – octahedral shear strain

ε_{ij} –the components of strain tensor (i=1,2,3 and j=1,2,3)

The maximum tensile strain 0.026 was reached for the tension sample in the uniaxial tension test, therefore, the corresponding octahedral shear strain is 0.0245 based on Equation (5.8).

For the torsion sample, the average shear strain for the hollow cylinder is given by Equation (5.9) (Saada 1988).

$$\gamma_{\theta z} = \frac{2\theta(R_o^3 - R_i^3)}{3H(R_o^2 - R_i^2)} \quad (5.9)$$

Where,

R_o, R_i – Outer and inner radius of the hollow sample cylinder, mm

θ – twisted angle, rad

H – twisted length, mm

In this study, the maximum twisted angle for the torsion sample is 20° and the twisted length is 52.8 mm, therefore, the corresponding average shear strain is 0.0296, and the octahedral shear strain is 0.0242.

Measurement of lattice strains by neutron diffraction

After subjected to tension or torsion, those tubular steel samples were studied using the time-of-flight (TOF) technique at the pulsed neutron source facility – Spectrometer for Materials Research at Temperature and Stress (SMARTS) at Los Alamos Neutron Science Center, as shown the Figure 5.3. SMARTS was set up to measure the d-spacing of different (hkl) reflections simultaneously. The incident neutron beam was defined by boron nitride apertures; the scattered neutrons were determined by radial collimators through which the gage volume can be viewed by two detector banks (± 90 degree) (Larsson, Holden et al. 2005).

As indicated by Equation (5.3), the interplanar d-spacing can be used as an internal strain gage, and can be determined based on the diffraction spectra at different spatially locations in the sample. Lattice strain then can be obtained in terms of the change in d-spacing of stressed and stress-free samples. In this study, residual strains are measured in radial and circumferential direction using a gage volume of $1 \times 1 \times 5 \text{ mm}^3$. The residual strain results based on typical lattice planes that have been reported to be weakly or strongly affected by intergranular strain for tension stress path from prior literature are discussed in the following.

The samples were placed on a stage where the x, y, z translation and rotation with respect to the center of the stage are controlled by computer. The samples were positioned and vertically aligned with an accuracy of 0.1mm using two specialized telescopes using triangulation. The scattering vector Q_R goes through the center of the circular cross section of the specimen so that the detector bank (90 degree) measured d-spacing in radial direction; the scattering vector Q_H is normal to Q_R so that the detector

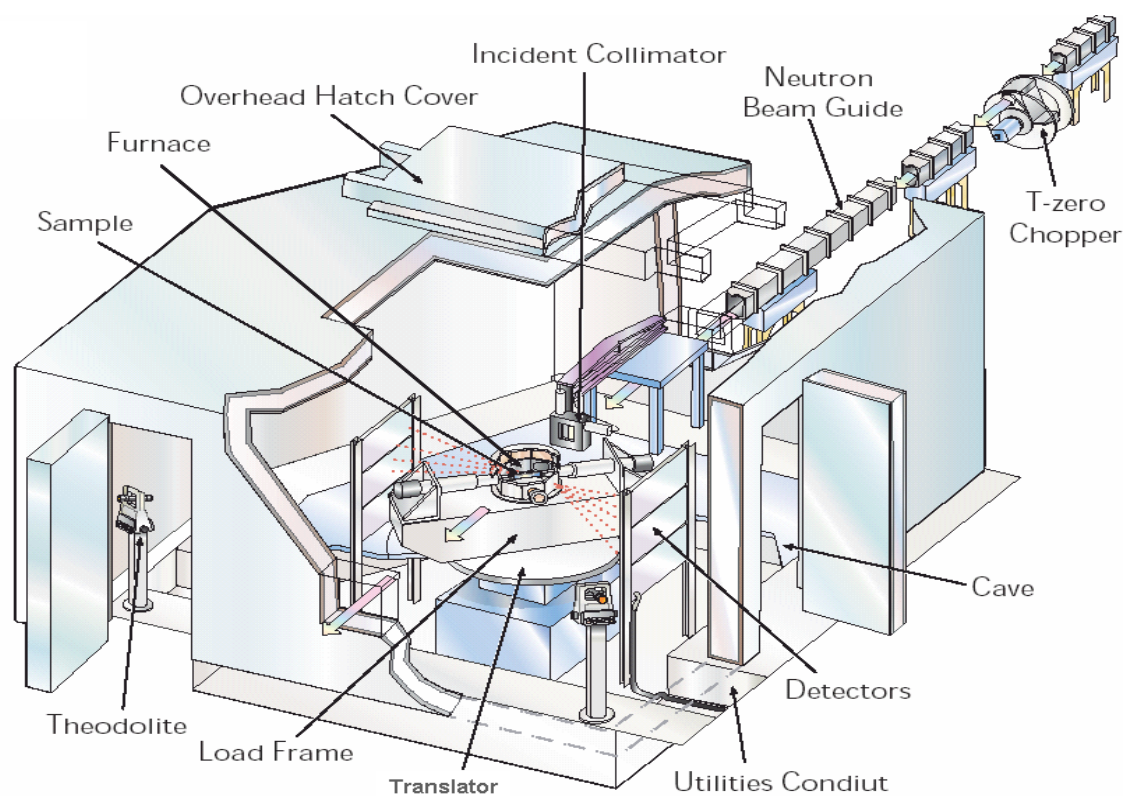


Figure 5.3 Research facility at SMARTS, LANL

bank (-90 degree) measured d-spacing in circumferential direction as shown in Figure 5.4.

Neutron scattering measurements were conducted at different locations such as mid-height, 10 mm and 20 mm from mid-height in unstressed, tension and torsion samples. Each measurement took about 4 hours. All the measured points are at the middle of the wall thickness of the hollow cylindrical samples.

The raw data from the spallation neutron scattering can be processed in two ways. The first approach is to fit the whole neutron spectra using Rietveld refinements, and the other is to fit individual (hkl) reflection like the way to process raw data from reactor neutron source.

The Rietveld procedure optimizes the lattice parameters which include lengths and angles in a unit cell until the calculated spectrum shows the best least squares fit to the measured spectrum. A typical time-of-flight (TOF) diffraction pattern in the radial direction at the mid-height of the tension sample is given in Figure 5.5, in which the crosses are the measured neutron data (Luo, Penumadu et al. 2006). The time-of-flight (TOF) diffraction pattern was normalized, and processed using a Rietveld refinement program named General Structure Analysis System (GSAS) (Larson and Von Dreele 2000), as shown Figure 5.6, in which seven (hkl) reflections of interest were calculated based on the Rietveld least square fit. The difference curve between the refinement and measurement were also shown beneath the data.

The individual peak fitting method deals with the particular reflection (hkl) individually. The computer program (Clausen 2003) developed based on the GSAS code

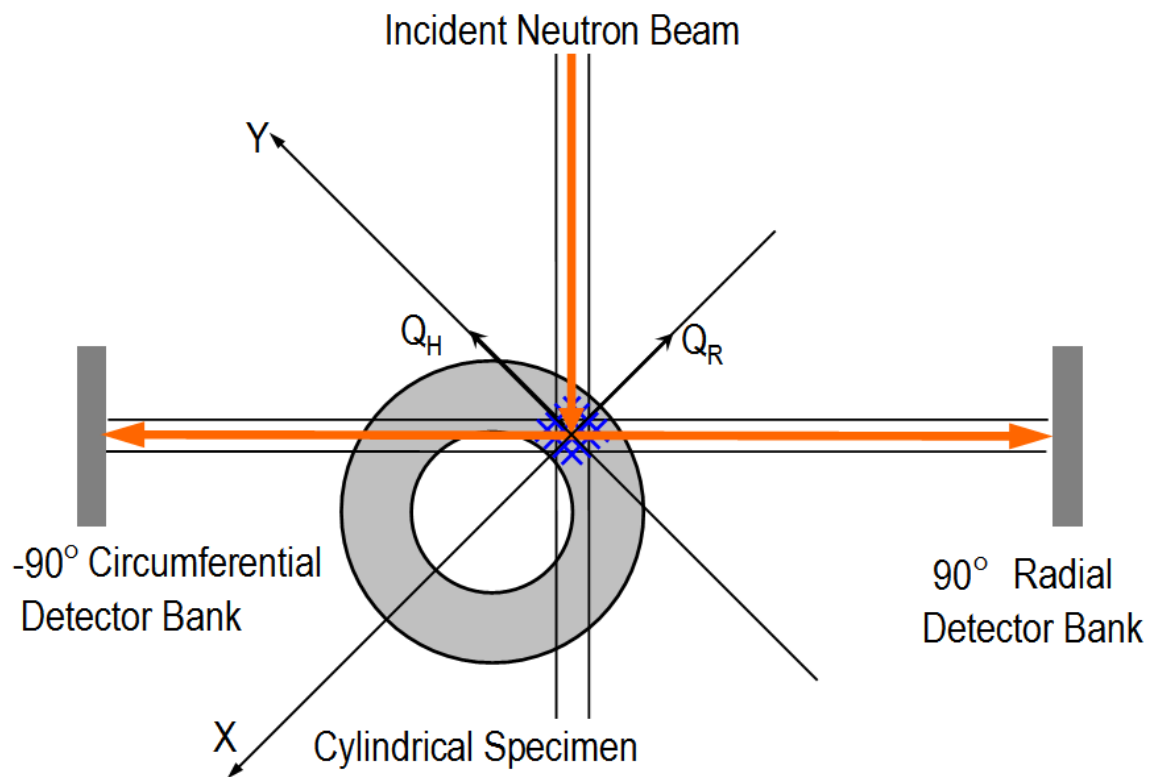


Figure 5.4 Experimental set-up measuring residual strain using spallation neutron source

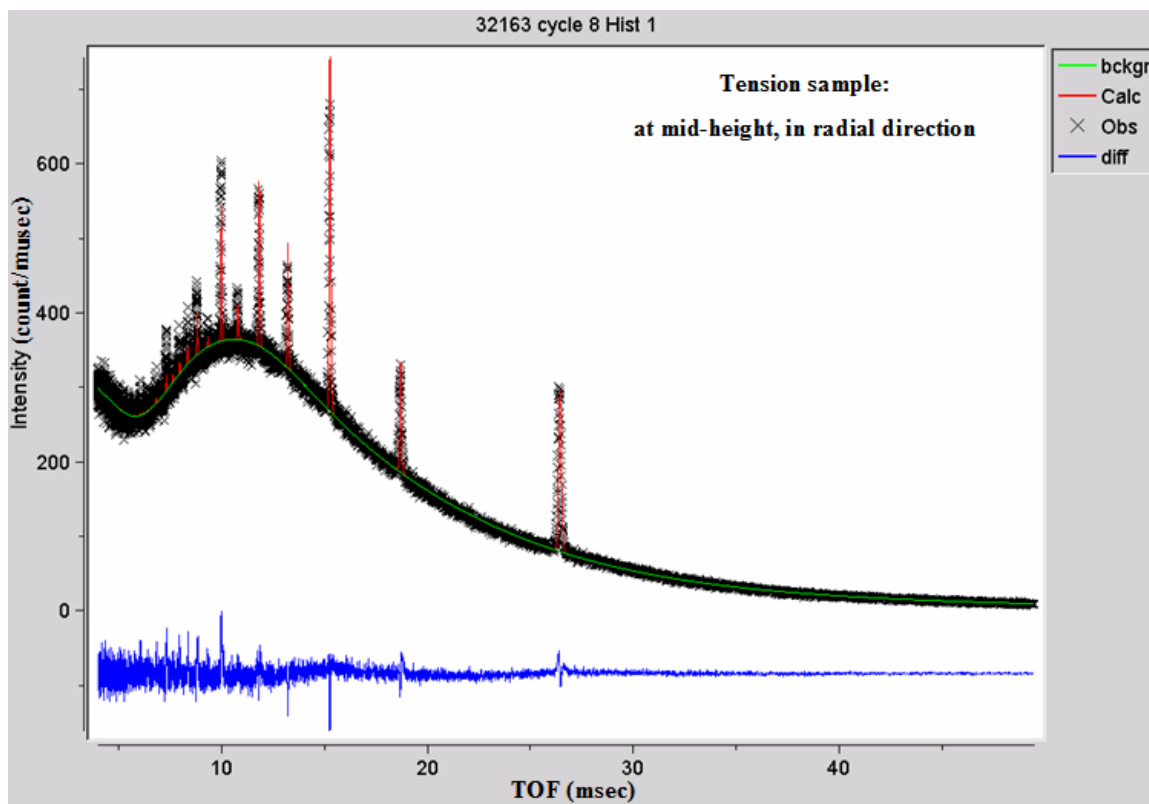


Figure 5.5 An observed time-of-flight (TOF) diffraction pattern

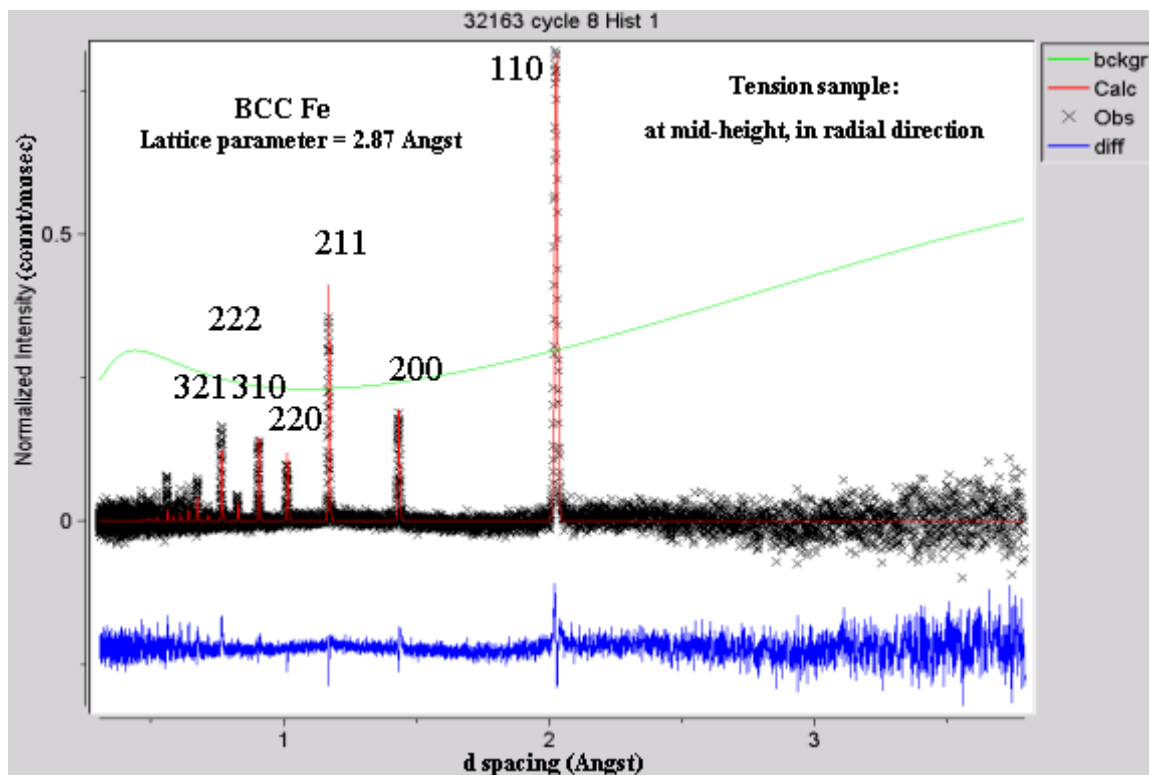


Figure 5.6 A time-of-flight (TOF) diffraction pattern after normalization and Rietveld refinement

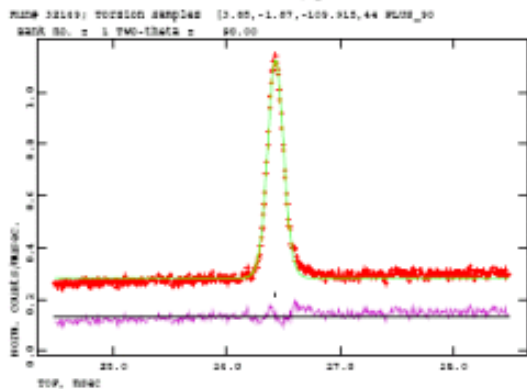
was used to fit the reflections (110), (200), (211), (220), (222), (310), and (321) in neutron scattering spectra, respectively. In this study, the individual peak fitting method was used and expected to provide better resolution. Representative individual peak fitting results for the unstressed sample in the radial direction are shown in Figure 5.7 and Figure 5.8.

Figure 5.9 – Figure 5.10 show the residual strains of multiple (hkl) reflections for the torsion and tension samples at three different heights in both radial and circumferential directions. For example, for the tension sample in radial direction, the left data points represent the location at the mid-height of the sample, the middle points represent the location of the mid-height plus 10 mm, and the right side points represent the location of the mid-height plus 20 mm. In general, there is much difference in terms of the residual strains between the seven reflections. Some reflections such as (321), (211), and (110) show relatively small residual strains in radial direction in the tension sample, while other reflections such as (220) and (200) show considerable residual strains in the same sample. There is also remarkable difference in the residual strains for the tension and torsion samples in both radial and circumferential directions.

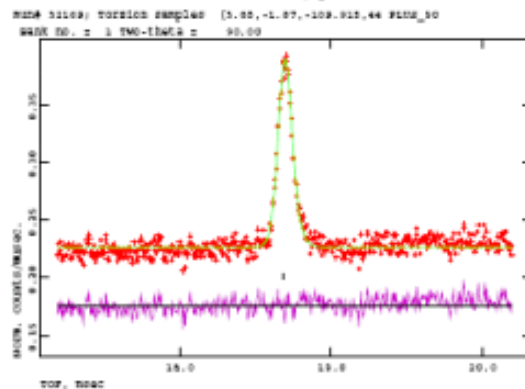
Figure 5.11 – Figure 5.14 show that most of the measured residual strains at different heights are quite similar for each reflection in both tension and torsion samples. This indicates the uniform residual strains along the longitudinal axis of the samples and the repeatability of the neutron diffraction measurement.

For the torsion sample in the radial direction, the lattice strains based on the (110), (211) and (200) are within a relatively narrow range at the different heights of the sample. For example, the lattice strains are around 125 $\mu\epsilon$ for the (110) reflection, 30 – 60 $\mu\epsilon$ for

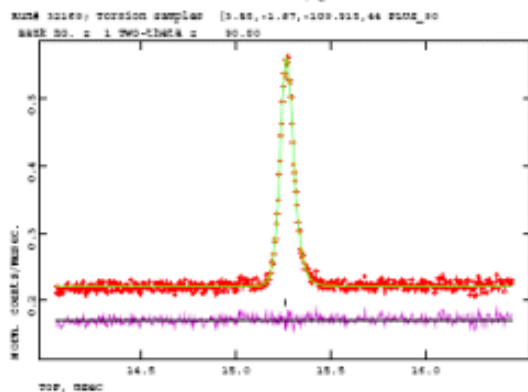
Run 32169 Peak 1 $\chi^2 = 2.330$



Run 32169 Peak 2 $\chi^2 = 0.782$



Run 32169 Peak 3 $\chi^2 = 0.693$



Run 32169 Peak 4 $\chi^2 = 0.709$

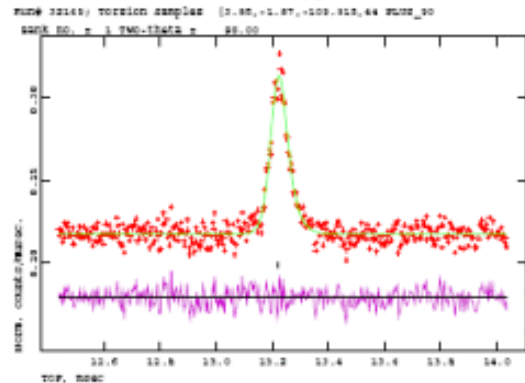
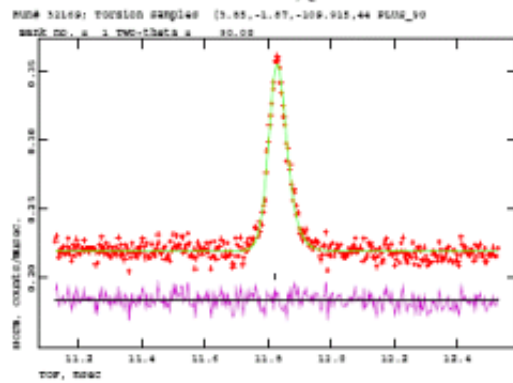
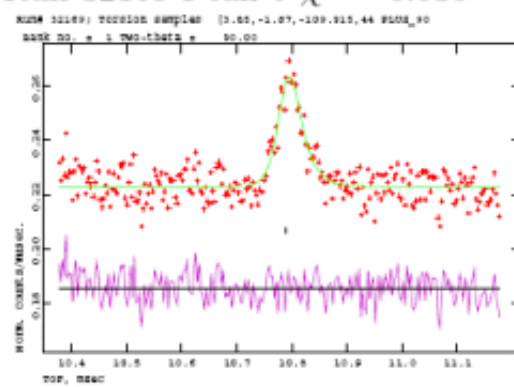


Figure 5.7 Individual peak fitting of the unstressed sample in radial direction (Peak# 1-4)

Run 32169 Peak 5 $\chi^2 = 0.644$



Run 32169 Peak 6 $\chi^2 = 0.636$



Run 32169 Peak 7 $\chi^2 = 0.687$

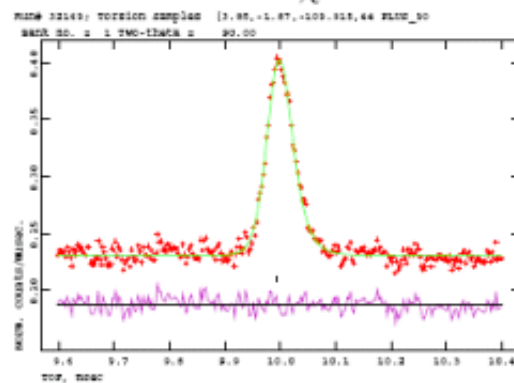


Figure 5.8 Individual peak fitting of the unstressed sample in radial direction (Peak# 5-7)

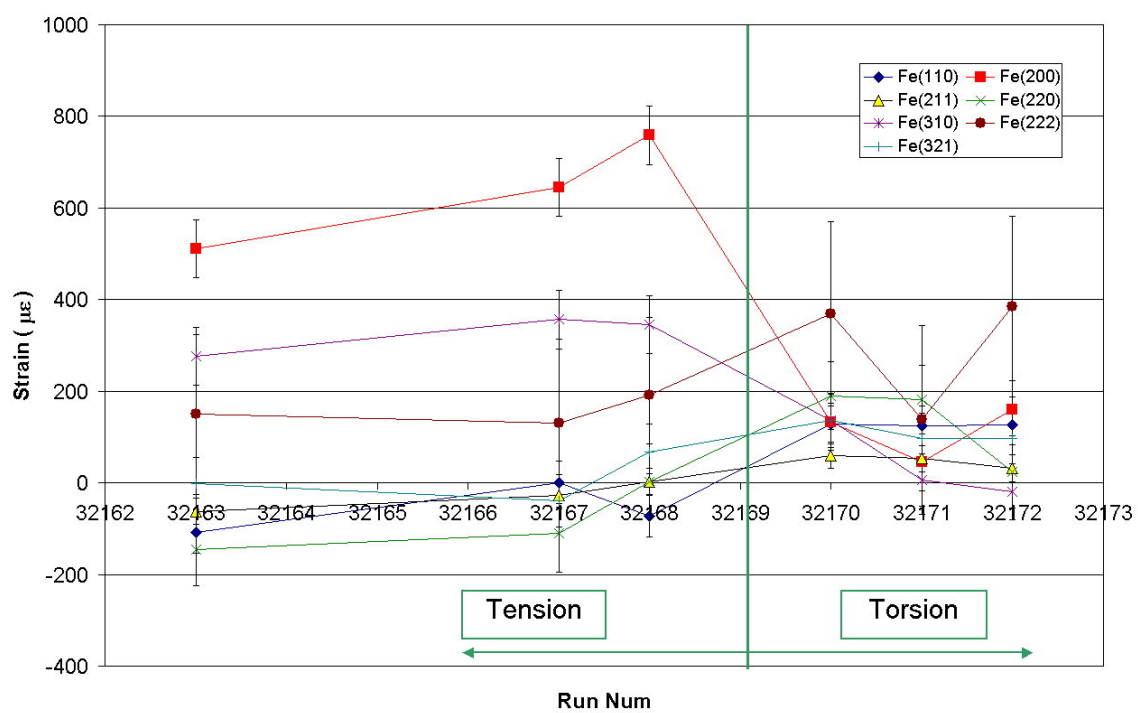


Figure 5.9 Residual strains of multiple (hkl) reflections at different heights in radial direction

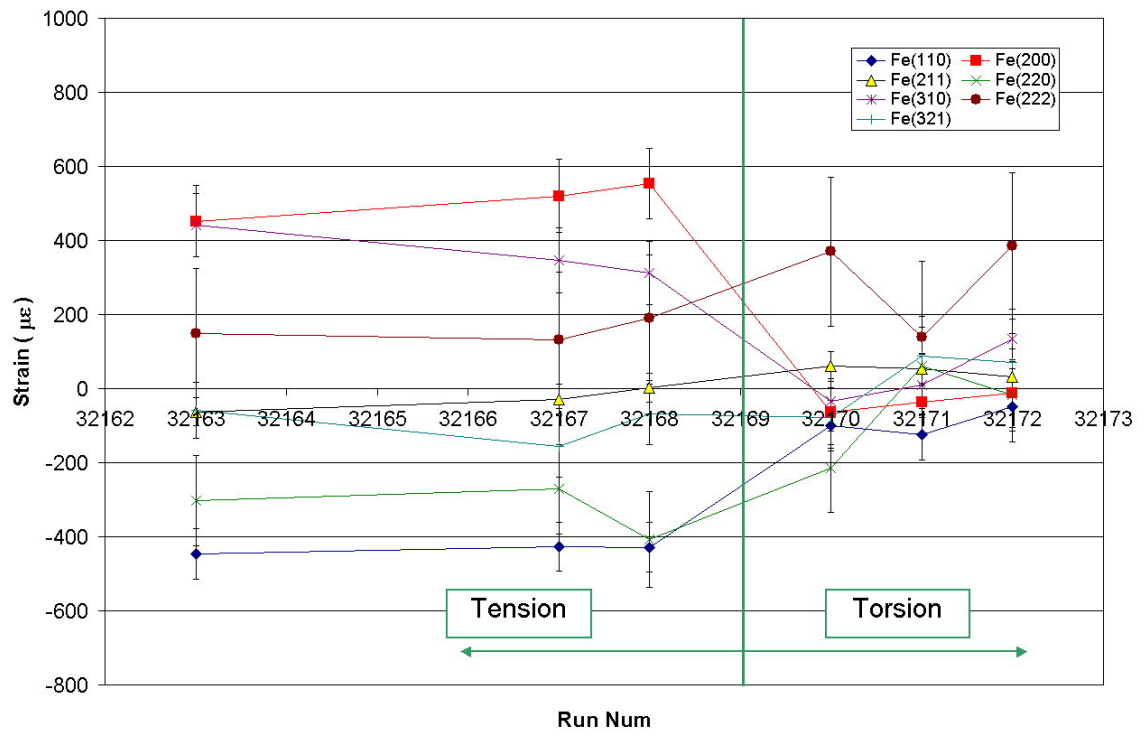


Figure 5.10 Residual strains of multiple (hkl) reflections at different heights in circumferential direction

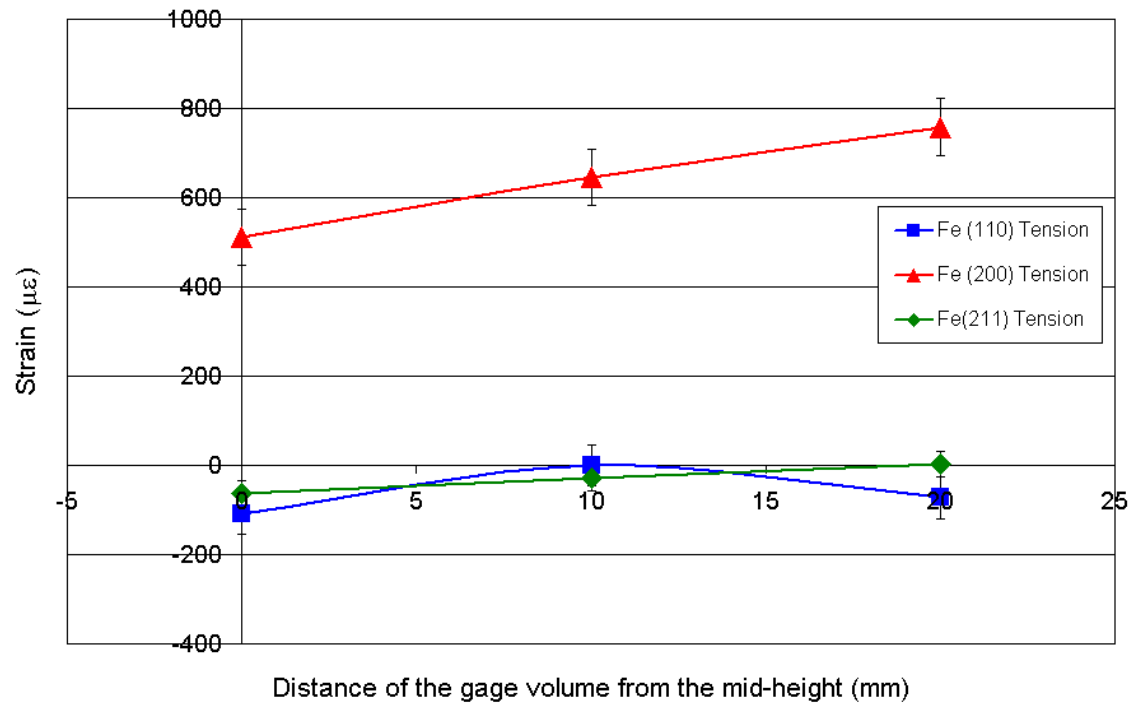


Figure 5.11 Residual strains in radial direction at various locations of the tension sample

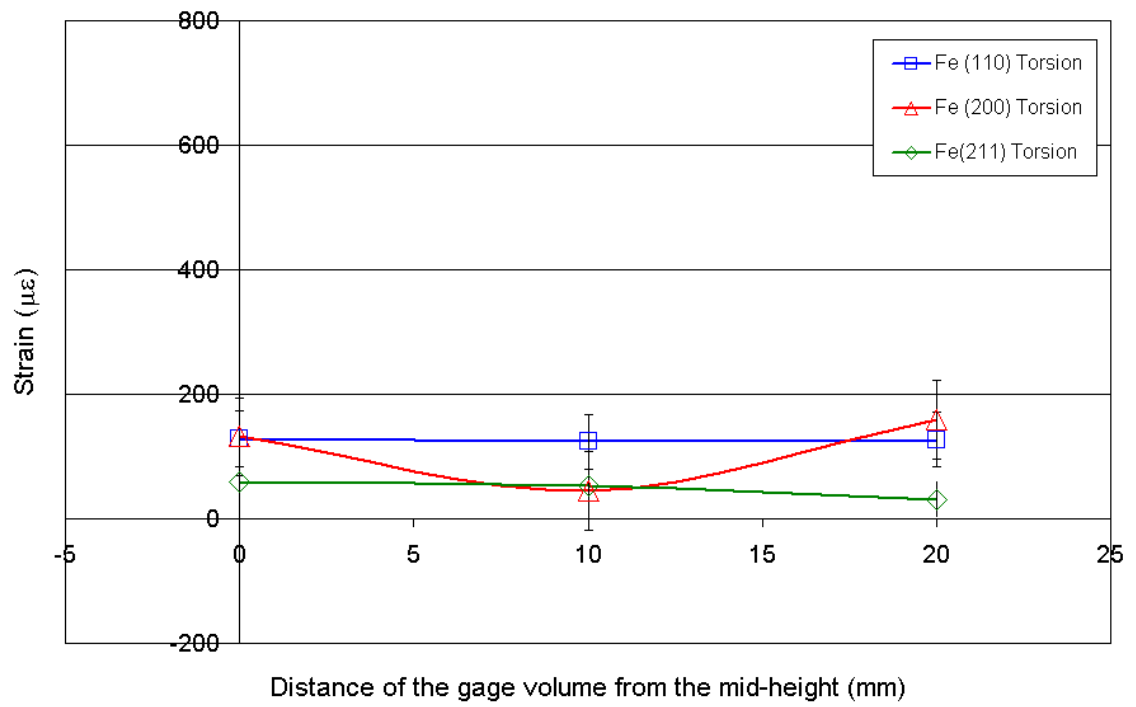


Figure 5.12 Residual strains in radial direction at various locations of the torsion sample

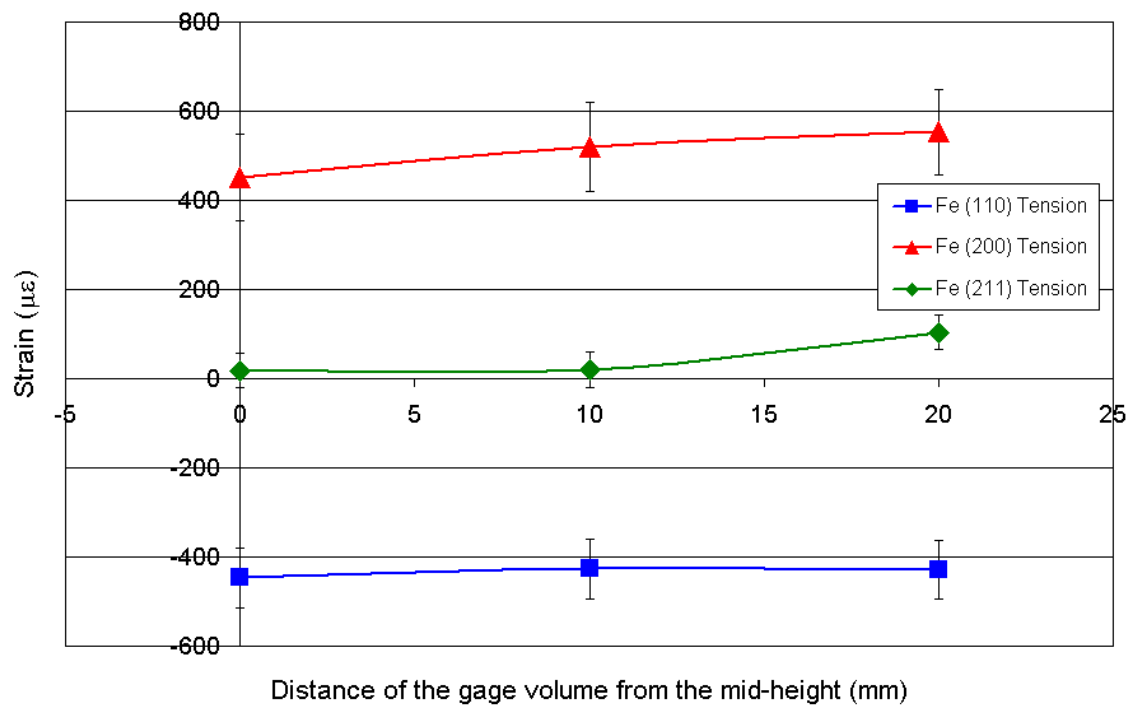


Figure 5.13 Residual strains in circumferential direction at various locations of the tension sample

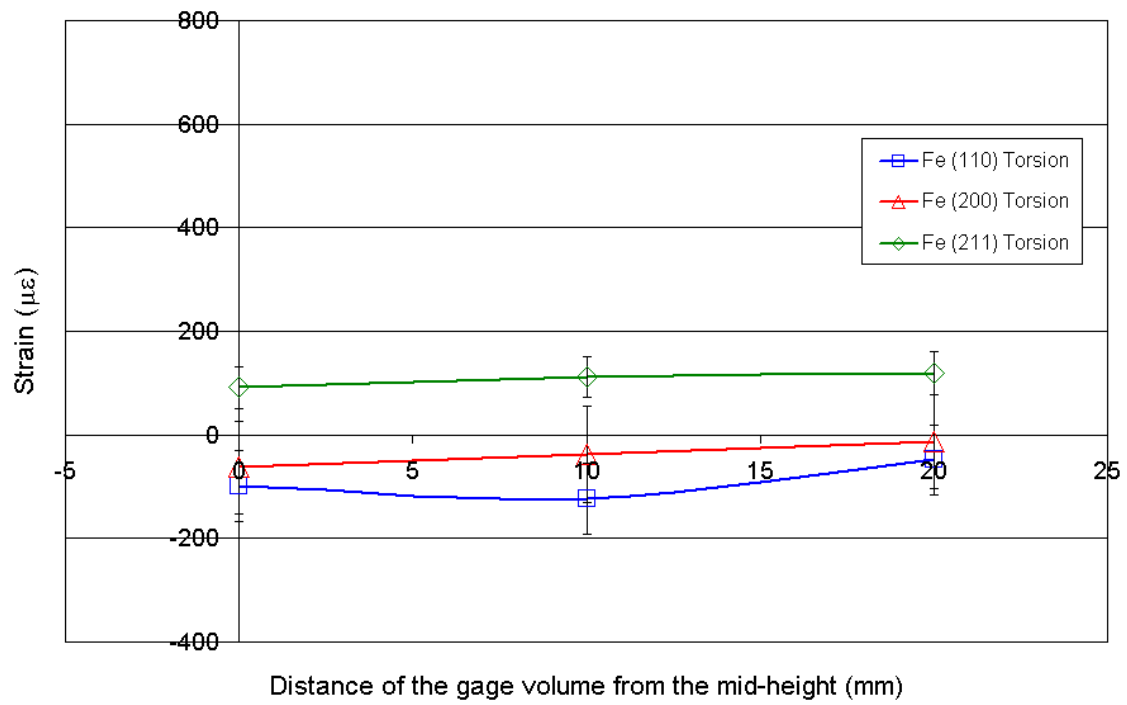


Figure 5.14 Residual strains in circumferential direction at various locations of the torsion sample

the (211) reflection, and 45 – 160 $\mu\epsilon$ for the (211) reflection. However, the residual strains based on the (200) reflection show a significant difference from both (110) and (211) reflections in the tension sample. In particular, the measured lattice strains of the (200) range from 500 – 750 $\mu\epsilon$ in the tension sample, compared to 44 – 160 $\mu\epsilon$ in the torsion sample. A similar situation occurs in the residual strains in the circumferential direction for both the tension and torsion samples, in which the (200) reflection shows significant residual strain in the tension sample but small residual strains in the torsion sample.

It has been reported that (211) and (110) are weakly affected by the intergranular strains and (200) are strongly affected by the intergranular strains in polycrystalline materials (Pang, Holden et al. 1998). From this study, it is observed that the (200) reflection does show significant residual strains in the tension sample, but quite smaller residual strains in the torsion sample. It appears that the intergranular strain effect is associated not only with the particular reflection, but also with loading paths even though the tension and torsion samples are subject to the similar deviatoric strain (octahedral shear strain).

In Figure 5.13 – Figure 5.14, the (110) and (211) also show considerable difference in terms of the residual strain. For the tension sample, while the residual strains are near zero at some locations in the (211) reflection, the residual strains are over 400 $\mu\epsilon$ in the (110) reflection. For the torsion sample, the difference between (110) and (211) reflections is reduced although it still exists. Like the lattice strains of the (200) reflection in radial directions, the lattice strains of the (110) in the circumferential direction are greater than the torsion sample.

Summary

Residual strains of carbon steel tubes, which were subject to a similar deviatoric strain through either tension or torsion, were investigated using the pulsed neutron source facility – Spectrometer for Materials Research at Temperature and Stress (SMARTS) at Los Alamos National Laboratory.

Experimental results show the uniform residual strains measured along the longitudinal axis of the samples and the repeatability of the neutron diffraction measurement. The reflection of (200) reported strongly affected by intergranular strains does show significant residual strains in the tension sample, but quite smaller residual strains in the torsion sample. The reflections of (110) and (211), both reported weakly affected by intergranular strains show considerable difference in terms of residual strain in circumferential direction. It appears that the intergranular strain effect is not only associated with particular reflections, but also strongly associated with loading paths through which the tension and torsion samples are subject to the similar deviatoric strain. Although the tension and torsion samples were subject to the similar deviatoric strain, the residual strains resulted from the tension and torsion show a considerable difference in both radial and circumferential directions.

This study is the first of its kind for evaluating residual strains in generalized loading conditions and also will help to demonstrate the need for in-situ tension-torsion load frame at Spallation Neutron Source for studying materials under realistic loading conditions.

Acknowledgement

This research was support by the JINS Fellowship program and NSF IMI program (DMR-0231320). This experimental work carried out at SMARTS was benefited from the user program of the Los Alamos Neutron Science Center (LANSCE), Los Alamos National Laboratory (LANL).

References

- Borland, D. W. (1997). Residual stress measurement - methods, limitations and significance. The 2nd Australian International Conference on Surface Engineering, Adelaide.
- Clapham, L., T. W. Krause, et al. (1997). "Machining of simulated defects in pipeline steels: a neutron diffraction study of local stress effects." Journal of Strain Analysis for Engineering Design **32**(5): 325-334.
- Clausen, B. (2003). SMARTSware Manual: Software package for SMARTS users to facilitate data reduction and analysis. Los Alamos, NM, Los Alamos National Laboratory.
- Cullity, B. D. and S. R. Stock (2001). Elements of X-Ray Diffraction. Upper Saddle River, Prentice Hall.
- Hayashi, M., M. Ishiwata, et al. (2000). "Residual stress distribution in carbon steel pipe welded joint measured by neutron diffraction." Materials Science Research International **6**(4): 287-294.
- Holden, T. M. and A. W. Bowen (1992). Interpretation of Residual Stress Measurements: Summary of Discussion Session. Measurement of Residual and Applied Stress Using Neutron Diffraction. M. T. Hutchings and D. Krawitz. Dordrecht, Kluwer Academic Publishers: 223-231.
- Hutchings, M. T. (1992). "Neutron diffraction measurements of residual stress fields - the engineer's dream come true?" Neutron News(3): 14-19.
- Hutchings, M. T., P. J. Withers, et al. (2005). Introduction to the characterization of residual stress by neutron diffraction. Boca Raton, CRC Press.

- Inoue, K., H. Kawashima, et al. (2000). "Neutron diffraction measurement and finite element method calculation of residual stress of a heat treated steel pipe." Japanese Journal of Applied Physics Part 1-Regular Papers Short Notes & Review Papers **39**(12A): 6652-6657.
- Jones, A. M. (1989). Residual stresses: A review of their measurement and interpretation using X-ray diffraction, Harwell Laboratory, Materials Development Division.
- Kandil, F. A., J. D. Lord, et al. (2001). A Review of Residual Stress Measurement Methods - A Guide to Technique Selection. Middlesex, UK, NPL Centre.
- Krawitz, A. D. and R. A. Winholtz (1994). "Use of Position-Dependent Stress-Free Standards for Diffraction Stress Measurements." Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing **185**(1-2): 123-130.
- Larson, A. C. and R. B. Von Dreele (2000). General Structure Analysis System (GSAS). Los Alamos, NM, Los Alamos National Laboratory.
- Larsson, C., T. M. Holden, et al. (2005). "Measurement and modeling of residual stress in a welded Haynes (R) 25 cylinder." Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing **399**(1-2): 49-57.
- Lisowski, P. W. and K. W. Jones (2002). Status of the Los Alamos Neutron Science Center and Plans for the Future. The 21st Linear Accelerator Conference, Gyeongju, Korea.
- Lu, J. and D. Reintant (1998). "A review of recent developments and applications in the field of X-ray diffraction for residual stress studies." Journal of Strain Analysis for Engineering Design **33**(2): 127-136.

- Luo, X., D. Penumadu, et al. (2006). Residual strain evolution in 3-D stress conditions under torsion and tension for steel using a spallation neutron source. American Conference on Neutron Scattering 2006, St. Charles, Neutron Scattering Society of America.
- Noyan, I. C. and J. B. Cohen (1987). Residual Stress Measurement by Diffraction and Interpretation. New York, NY, Springer-Verlag.
- Pang, J. W. L., T. M. Holden, et al. (1998). "The development of intergranular strains in a high-strength steel." Journal of Strain Analysis for Engineering Design **33**(5): 373-383.
- Rudd, C. O. (1992). Residual stresses and their measurement. The 1st International Conference on Quenching and Control of Distortion, Chicago, IL.
- Saada, A. S. (1983). Elasticity: Theory and Applications. Malabar, FL, Krieger Publishing.
- Saada, A. S. (1988). Hollow Cylinder Torsional Devices: Their Advantages and Limitations. Advanced Triaxial Testing of Soil and Rock. R. T. Donaghe, R. C. Chaney and M. L. Silver. Philadelphia, PA, American Society for Testing and Materials: 766-795.
- Withers, P. J. and H. Bhadeshia (2001). "Overview - Residual stress part 1 - Measurement techniques." Materials Science and Technology **17**(4): 355-365.
- Withers, P. J. and H. Bhadeshia (2001). "Overview - Residual stress part 2 - Nature and origins." Materials Science and Technology **17**(4): 366-375.

**Chapter 6 Residual Strain Tensor for Steel Hollow Cylinders after
Subject to Tension or Torsion Using Reactor Neutron Source**

Introduction to measurement of residual stress

Residual stress in an engineering component is inherently of 3-D in nature. However, due to the complex nature of residual stress, very limited study on the stress or strain state has been carried out under 3-D conditions, although extensive studies have been performed in the area of uniaxial and biaxial stress analysis (Holden, Root et al. 1995; Noyan, Huang et al. 1995; Pang, Holden et al. 2000; Oliver, Daymond et al. 2002; Brown, Bourke et al. 2003; Larsson, Clausen et al. 2004). For example, the $\sin^2(\psi)$ method, which is based on assumption that a biaxial stress state exists in the near-surface region of a stressed component, has been widely used for many years to measure the reference interplanar d-spacing by X-ray techniques (Noyan and Cohen 1987). A pioneering study of residual stress state under a 3-D triaxial conditions for welded cylindrical segments using reactor source neutron scattering was conducted by Krawitz et al (1994). They measured the lattice strains in six independent orientations of the tilted sample, solved stress tensors at different spatial locations in the weld, and revealed the stress states at the corresponding locations.

X-ray technique is usually used as a non-destructive tool to measure surface stress in a component (Noyan and Cohen 1987; Jones 1989; Lu and Retraint 1998; Cullity and Stock 2001). In combination with certain layer removal technique, a stress profile in a component can be obtained. Unfortunately, there are several major disadvantages of the traditional X-ray based stress measurement. Problems will occur if the surface of the sample is too rough. A surface treatment such as polishing is often needed, which inevitably changes the original conditions of the near-surface region in materials. Use of X-ray in stress profiling in a component requires stepwise removal of surface layers, and

plastic deformation may be induced during the removal procedure, causing new stresses and influencing the results for the stress profiling (Pintschovius 1992). Sample size could be another problem. Depending on material and source, X-ray usually has a penetration depth of around 10-30 μm and a spatial resolution of 1-2 mm down to tens of microns (Kandil, Lord et al. 2001).

Although X-ray might be the best or the only approach in the near surface region of materials, neutron technique is much more competitive for stress analysis at depths of over 100 μm (Webster 1992). Compared to traditional X-ray, neutrons have a significant larger penetrating power in most of the engineering materials, because they interact with atomic nuclei and are independent of atomic number. This makes neutrons a unique tool to acquire otherwise inaccessible information for strains probing in the interior of bulk materials in a non-destructive way, so “the engineer’s dream come true” (Hutchings 1992). For example, neutrons can measure bulk component of up to 100 mm in aluminum or 25 mm in steel (Kandil, Lord et al. 2001). The large penetration power of neutron is particularly helpful in the exploit of grain interaction stresses, as it allows the determination of interplanar d-spacing vs $\sin^2(\psi)$ up to $\sin^2(\psi)$ equal to 1, which is especially important to the study of strongly textured materials (Pintschovius 1992). Other merits of neutron scattering used for stress analysis include that neutrons can be used to monitor the evolution of residual stress or strain in realistic loading and environmental conditions, and neutron scattering can provide adjustable spatial resolution for strain mapping in order to resolve stress gradients in bulk engineering components (Hutchings, Withers et al. 2005).

Stress and strain tensors, and Haigh-Westergaard space

Stress can be represented by vector that is defined as the intensity of forces acting on the given infinitesimal part within a body. The state of stress at a point is therefore defined as the totality of all stress vectors at that point, and is represented by a second-order symmetric tensor σ_{ij} , which has six independent components due to symmetry, in a right-handed Cartesian coordinate system. The stress tensor σ_{ij} is expressed as:

$$\sigma_{ij} = \begin{bmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{21} & \sigma_{22} & \sigma_{23} \\ \sigma_{31} & \sigma_{32} & \sigma_{33} \end{bmatrix} \quad (6.1)$$

where,

$\sigma_{11}, \sigma_{22}, \sigma_{33}$ – normal stress components

$\sigma_{12}, \sigma_{21}, \sigma_{13}, \sigma_{31}, \sigma_{23}, \sigma_{32}$ – shear stress components, and $\sigma_{12}=\sigma_{21}, \sigma_{13}=\sigma_{31}, \sigma_{23}=\sigma_{32}$

Stress tensor acting at a point depends on orientation of the body. If planes of the body are oriented such that the stress vectors act in the normal direction of the planes on which there are no shear stresses, the stresses are principal stresses ($\sigma_1, \sigma_2, \sigma_3$). The principal stresses are orthogonal to each other, and construct a 3-D stress space called Haigh-Westergaard stress space or the principal stress space. For a given state, the stress can be decomposed into a hydrostatic stress component and deviatoric stress component in Haigh-Westergaard stress space, as given in Equation (6.2).

$$\sigma_{ij} = \frac{\sigma_{kk}}{3} \cdot \delta_{ij} + s_{ij} \quad (6.2)$$

where,

σ_{kk} , – Addition of the normal stress components, $\sigma_{kk} = \sigma_{11} + \sigma_{22} + \sigma_{33}$

δ_{ij} – Kronecker delta function, $\delta_{ij} = 1$ if $i=j$, and $\delta_{ij} = 0$ if $i \neq j$

s_{ij} – Deviatoric stress components

The decomposition of a given stress state ($\sigma_1, \sigma_2, \sigma_3$) can also be visualized by Figure 6.1, in which coordinates of the point P represents the given stress state in Haigh-Westergaard stress space, thus,

$$\mathbf{OP} = \mathbf{ON} + \mathbf{NP} \quad (6.3)$$

where,

OP – The vector representing the given stress

ON – The vector representing the hydrostatic component

NP – The vector representing the deviatoric component.

Yield criteria are used to predict whether the material's response is elastic or plastic. Von Mises criterion is one of the important yield criteria, and used to characterize the yield behavior for most metal materials, which are considered hydrostatic-pressure-independent, which implies that the yield only depends on deviatoric stress components, as shown by Equation (6.4). Von Mises yield criterion states that the yielding of material would occur when the maximum shear strain energy reaches a critical value specified by material parameter k . This also can be visualized as a cylinder with its longitudinal axis coincident with hydrostatic axis in Haigh-Westergaard space, as shown in Figure 6.1.

$$J_2 - k^2 = 0 \quad (6.4)$$

where,

J_2 – The second invariant of the deviatoric stress component, $J_2 = \frac{1}{2} s_{ij} s_{ij}$

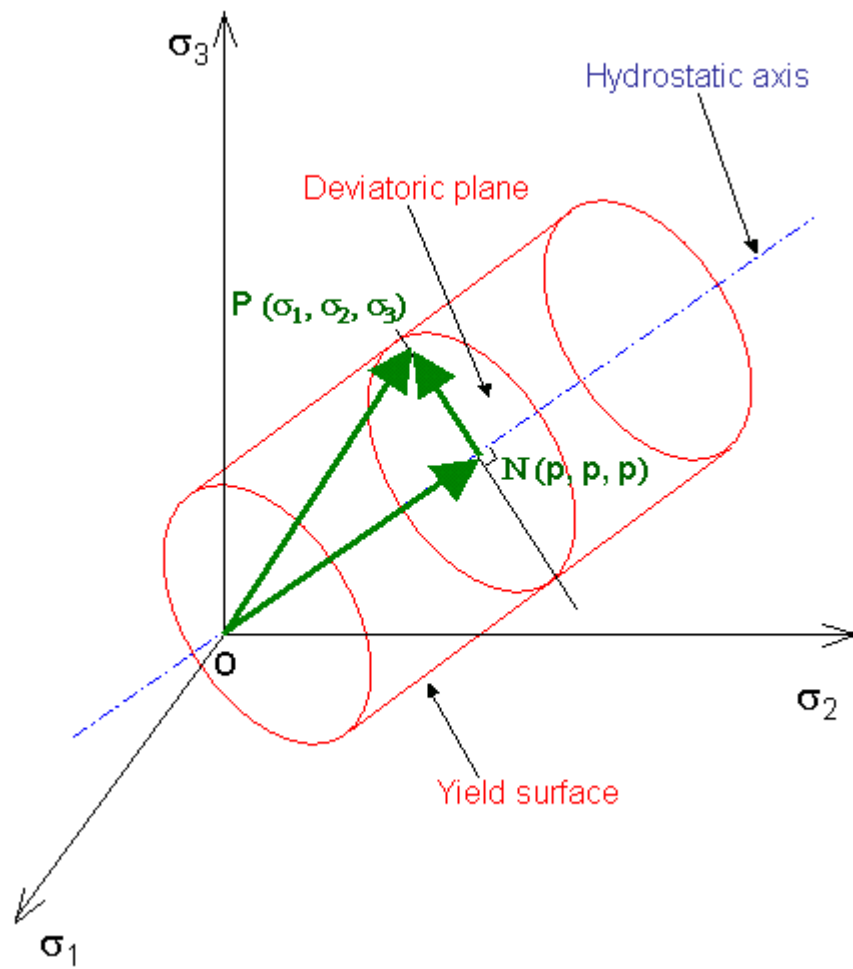


Figure 6.1 Decomposition of stress vector in Haigh-Westergaard space and Von Mises yield criterion

k – Material parameter depended on loading conditions. For example, $k = \frac{\sigma_0}{\sqrt{3}}$ for

uniaxial tensile test, where σ_0 is the uniaxial tensile strength; $k = \tau_0$ for pure

shear test, where τ_0 is the shear strength

Similarly, the state of strain at a point in a deformed body can be described by a second-order symmetric tensor ϵ_{ij} , which is also related to a set of three principal strain ϵ_1 , ϵ_2 , and ϵ_3 , as given by Equation (6.5)

$$\epsilon_{ij} = \begin{bmatrix} \epsilon_{11} & \epsilon_{12} & \epsilon_{13} \\ \epsilon_{21} & \epsilon_{22} & \epsilon_{23} \\ \epsilon_{31} & \epsilon_{32} & \epsilon_{33} \end{bmatrix} \quad (6.5)$$

where,

ϵ_{11} , ϵ_{22} , ϵ_{33} – normal strain components

ϵ_{12} , ϵ_{21} , ϵ_{13} , ϵ_{31} , ϵ_{23} , ϵ_{32} – half of the shear strain components, i.e., $\epsilon_{ij} = \frac{1}{2}\gamma_{ij}$,

where γ_{ij} is the related shear strain, and $\epsilon_{12} = \epsilon_{21}$, $\epsilon_{13} = \epsilon_{31}$, $\epsilon_{23} = \epsilon_{32}$

In this study of residual stress, it is of great interest to understand how the similar ultimate loading conditions, particularly, the hollow cylindrical samples were subjected to a target deviatoric strain (octahedral shear strain) which indicated a similar strain history in both torsion and tension, will affect the final residual stress/strain state.

Determination of strain tensor using Least Squares fitting

In order to obtain the stress or strain tensor, two coordinate systems are involved in measurement. One is the laboratory coordinate system L_i ($i = 1, 2, 3$), which consists of the axes with respect to what types of diffraction measurements are made. The other is

the sample coordinate system S_i ($i = 1, 2, 3$), which consists of axes with respect to how the sample is placed. These two systems are related by angles ϕ and ψ , as shown in Figure 6.2.

For a given hkl diffraction, the axis L_3 is always normal to that lattice plane (hkl) and L_2 is coplanar with axes S_1 and S_2 . The relation of lattice strain $\epsilon_{\phi\psi}$ in L_3 direction (with angles ϕ and ψ) to the six independent components of the strain tensor ϵ_{ij} in the sample coordinate system can be described by Equation (6.6).

$$\begin{aligned}\epsilon_{\phi\psi} = & \epsilon_{11} \cos^2 \phi \sin^2 \psi + \epsilon_{22} \sin^2 \phi \sin^2 \psi + \epsilon_{33} \cos^2 \psi \\ & + \epsilon_{12} \sin 2\phi \sin^2 \psi + \epsilon_{13} \cos \phi \sin 2\psi \\ & + \epsilon_{23} \sin \phi \sin 2\psi\end{aligned}\tag{6.6}$$

There are six unknowns (namely, ϵ_{11} , ϵ_{22} , ϵ_{33} , ϵ_{12} , ϵ_{13} , ϵ_{23}) in Equation (6.6), and one can determine the lattice strain ($\epsilon_{\phi\psi}$) along six independent directions. However, during the measurement of neutron scattering, errors coming from both the experimental system and measurement process inevitably affect the final results. Therefore, more than six measurements should be done, and the Least Squares fitting is used in order to obtain the 6 unknowns so that the random errors are reduced.

Least Squares fitting (LSF) is a procedure for finding the best-fitting curve to a given set of points by minimizing the sum of the squares of the offsets or residuals of the points from the curve. The offsets of the points with respect to the fitting curve can be defined in two ways: one is vertical offset, and the other perpendicular offset. Winholtz and Cohen (1988) introduced a generalized least squares determination of triaxial stress

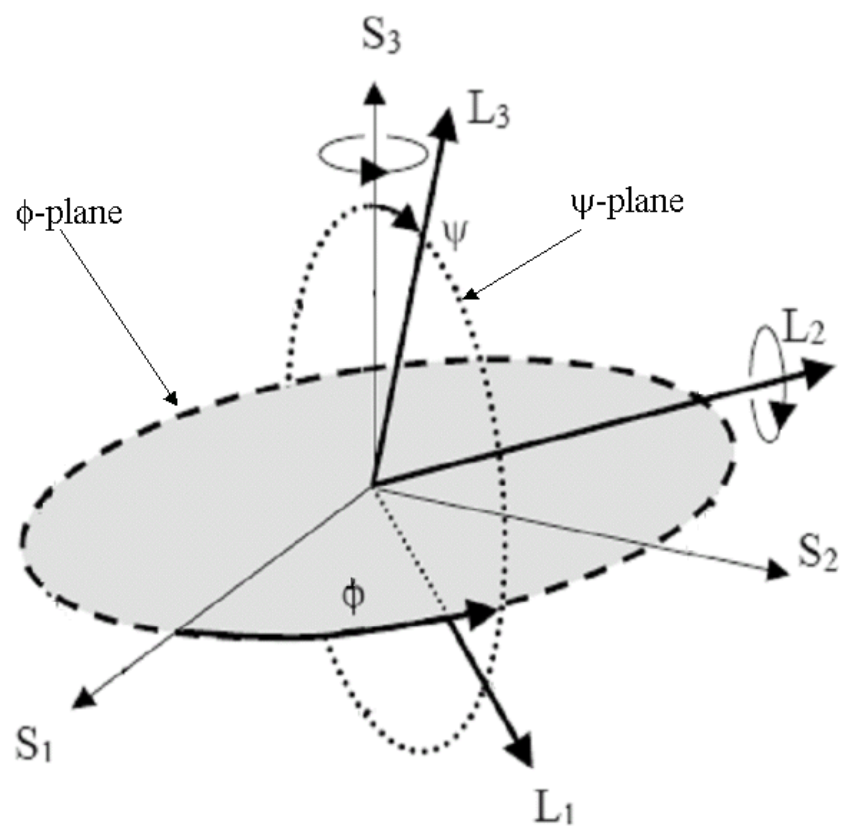


Figure 6.2 Relation between sample (S) and lab (L) coordinate systems

states by X-ray diffraction. They considered the inverse of the variance associated with each measured lattice strain as the weight for the corresponding residual square in a total weighted sum of the squared error. In this study, however, a simplified and general LSF adopting the vertical offsets as the residual was used as given in Equation (6.20). In order to utilize the LSF to solve a system of equations based on the Equation (6.6), the following definitions are made. The measured lattice strains are defined as

$$\varepsilon_i = \varepsilon_{\phi_i \psi_i} \quad (6.7)$$

where,

$$i = 1, 2, \dots, N$$

N – The times that independent measurement is made

The transformation coefficients are defined as

$$G_{i1}(\phi_i, \psi_i) = \cos^2 \phi_i \cdot \sin^2 \psi_i \quad (6.8)$$

$$G_{i2}(\phi_i, \psi_i) = \sin^2 \phi_i \cdot \sin^2 \psi_i \quad (6.9)$$

$$G_{i3}(\phi_i, \psi_i) = \cos^2 \psi_i \quad (6.10)$$

$$G_{i4}(\phi_i, \psi_i) = \sin 2\phi_i \cdot \sin^2 \psi_i \quad (6.11)$$

$$G_{i5}(\phi_i, \psi_i) = \sin \phi_i \cdot \sin 2\psi_i \quad (6.12)$$

$$G_{i6}(\phi_i, \psi_i) = \cos \phi_i \cdot \sin 2\psi_i \quad (6.13)$$

The unknown six components of the strain tensor are defined as

$$e_1 = \varepsilon_{11} \quad (6.14)$$

$$e_2 = \varepsilon_{22} \quad (6.15)$$

$$e_3 = \varepsilon_{33} \quad (6.16)$$

$$e_4 = \varepsilon_{12} \quad (6.17)$$

$$e_5 = \varepsilon_{23} \quad (6.18)$$

$$e_6 = \varepsilon_{13} \quad (6.19)$$

The residual between the calculated and measured lattice strain is

$$r_i = \sum_{j=1}^6 e_j \cdot G_{ij}(\phi_i, \psi_i) - \varepsilon_i \quad (6.20)$$

The sum of the squared residual for the N measurements is

$$R^2 = \sum_{i=1}^N r_i^2 = \sum_{i=1}^N \left(\sum_{j=1}^6 e_j \cdot G_{ij}(\phi_i, \psi_i) - \varepsilon_i \right)^2 \quad (6.21)$$

Based on LSF, the condition for R^2 to be a minimum is that

$$\frac{\partial R^2}{\partial e_k} = 0 \quad (6.22)$$

where, $k = 1, 2, \dots, 6$

So

$$\sum_{i=1}^N \left(\sum_{j=1}^6 e_j \cdot G_{ij}(\phi_i, \psi_i) - \varepsilon_i \right) \cdot G_{ik}(\phi_i, \psi_i) = 0 \quad (6.23)$$

$$\sum_{j=1}^6 e_j \sum_{i=1}^N G_{ij}(\phi_i, \psi_i) G_{ik}(\phi_i, \psi_i) - \sum_{i=1}^N \varepsilon_i \cdot G_{ik}(\phi_i, \psi_i) = 0 \quad (6.24)$$

This can be rewritten in a form by defining the following

$$A_{jk} = \sum_{i=1}^N G_{ij}(\phi_i, \psi_i) G_{ik}(\phi_i, \psi_i) \quad (6.25)$$

$$B_k = \sum_{i=1}^N \varepsilon_i \cdot G_{ik}(\phi_i, \psi_i) \quad (6.26)$$

This leads to

$$\sum_{j=1}^6 e_j A_{jk} - B_k = 0 \quad (6.27)$$

In matrix form

$$\mathbf{A} \cdot \mathbf{e} = \mathbf{B} \quad (6.28)$$

Therefore

$$\mathbf{e} = \mathbf{A}^{-1} \mathbf{B} \quad (6.29)$$

where,

$\mathbf{e}$ – The 6×1 vector of the six unknown strain components

$\mathbf{A}$ – The 6×6 matrix of the transformation defined by Equation (6.25)

$\mathbf{B}$ – The 6×1 vector of the modified measured lattice strain defined by Equation (6.26)

Once the six independent components of the strain tensor ϵ_{ij} are resolved, the principal strains can be calculated from below.

$$\epsilon_1 + \epsilon_2 + \epsilon_3 = I_1 \quad (6.30)$$

$$\epsilon_1 \epsilon_2 + \epsilon_2 \epsilon_3 + \epsilon_3 \epsilon_1 = I_2 \quad (6.31)$$

$$\epsilon_1 \epsilon_2 \epsilon_3 = I_3 \quad (6.32)$$

where,

$\epsilon_1, \epsilon_2, \epsilon_3$ – the principal strains of the strain tensor ϵ_{ij}

I_1, I_2, I_3 – the strain invariants of the strain tensor ϵ_{ij}

$$I_1 = \epsilon_{11} + \epsilon_{22} + \epsilon_{33} \quad (6.33)$$

$$I_2 = \begin{vmatrix} \epsilon_{22} & \epsilon_{23} \\ \epsilon_{32} & \epsilon_{33} \end{vmatrix} + \begin{vmatrix} \epsilon_{11} & \epsilon_{13} \\ \epsilon_{31} & \epsilon_{33} \end{vmatrix} + \begin{vmatrix} \epsilon_{11} & \epsilon_{12} \\ \epsilon_{21} & \epsilon_{22} \end{vmatrix} \quad (6.34)$$

$$I_3 = \begin{vmatrix} \epsilon_{11} & \epsilon_{12} & \epsilon_{13} \\ \epsilon_{21} & \epsilon_{22} & \epsilon_{23} \\ \epsilon_{31} & \epsilon_{32} & \epsilon_{33} \end{vmatrix} \quad (6.35)$$

Therefore, the principal strains ϵ_1 , ϵ_2 , and ϵ_3 can be obtained by solving Equations (6.30) - (6.35).

Measurement of lattice strains with Neutron diffraction

Hollow cylindrical samples were prepared from steel 12L14, which is resulfurized, rephosphorized, free-cutting carbon steel with lead added. The steel met the chemistry requirements of the AISI 12L14, namely, C max 0.15 wt%, Mn 0.8-1.5 wt%, P 0.04-0.09 wt%, S 0.26-0.35 wt% and Pb 0.15-0.35 wt%. This steel also shows the characteristics of a yield point of 517 MPa, tensile strength of 603 MPa, and elongation of 15% (ASM 1990). The geometry of the samples is hollow cylinder, with outer diameter (O.D.) and inner diameter (I.D.) of 10.8 and 6.8 mm, respectively.

The loading history in terms of stress vs strain for both the torsion and tension samples is shown in Figure 6.3 and Figure 6.4. The tubular steel specimens are first subjected to either pure torsion or simple tension that corresponds to a target of similar deviatoric strain, i.e., the octahedral shear strain as given in Equation (6.36) (Saada 1983):

$$\gamma_{oct} = \frac{2}{3} \sqrt{(\epsilon_{11} - \epsilon_{22})^2 + (\epsilon_{22} - \epsilon_{33})^2 + (\epsilon_{33} - \epsilon_{11})^2 + 6(\epsilon_{12}^2 + \epsilon_{23}^2 + \epsilon_{13}^2)} \quad (6.36)$$

where,

γ_{oct} – octahedral shear strain

ϵ_{ij} – the components of strain tensor (i, j = 1, 2, 3)

For the tension sample, the maximum tensile strain 0.0130 was reached in the uniaxial tension test, therefore, the corresponding octahedral shear strain is 0.0123. For the torsion

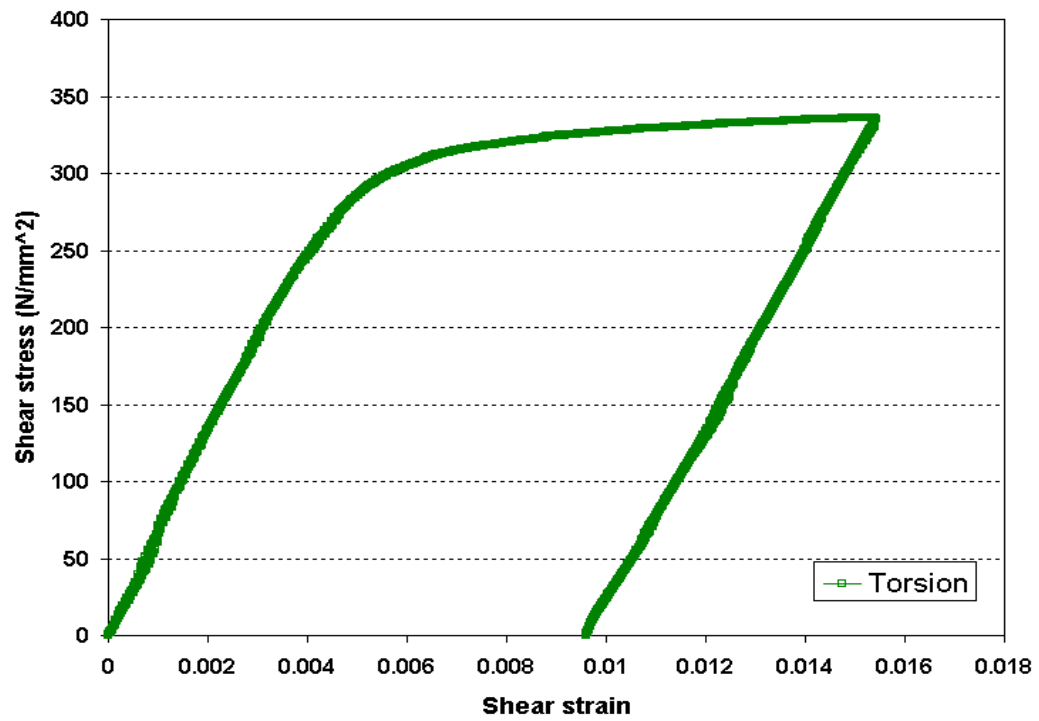


Figure 6.3 Stress-strain history of the tubular specimen for the torsion test

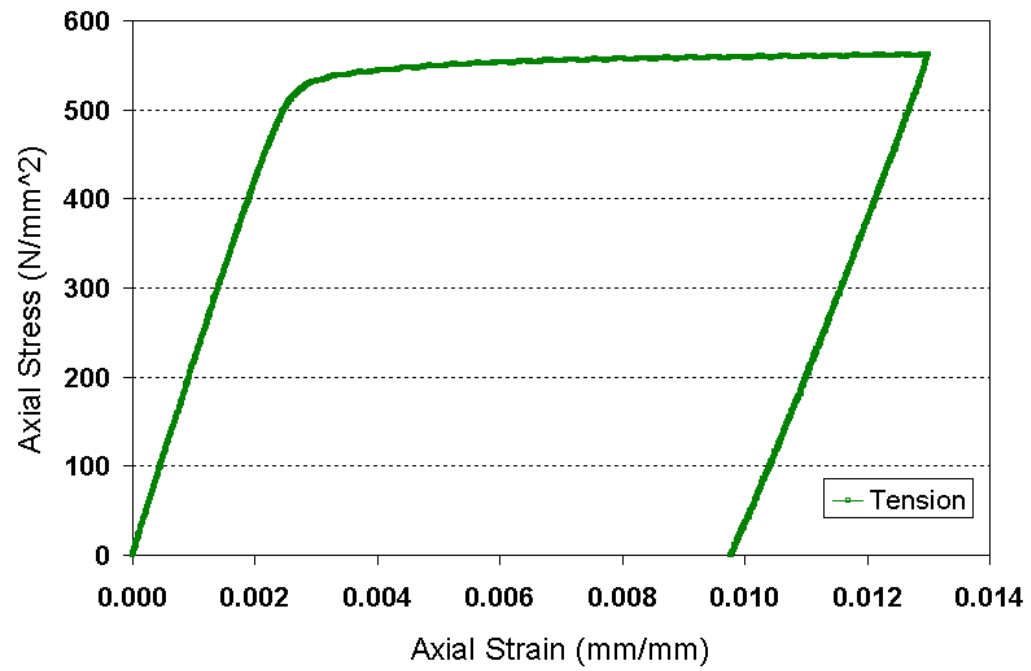


Figure 6.4 Stress-strain history of the tubular specimen for the tension test

sample, the average shear strain for the hollow cylinder is given by the Equation (6.37) (Saada 1988).

$$\gamma_{\beta z} = \frac{2\beta(R_o^3 - R_i^3)}{3H(R_o^2 - R_i^2)} \quad (6.37)$$

where,

R_o, R_i – The outer and inner radius of the hollow sample cylinder, mm

β – The twisted angle, rad

H – The twisted length, mm

The maximum twisted angle for the torsion sample is 10 degree and the twisted length is 52.8 mm, therefore, the corresponding average shear strain is 0.0150, and the octahedral shear strain is 0.0121.

Neutron scattering experiment was carried out at High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL). The neutron diffraction parameters including the Si monochromator settings (Stoica, Popovici et al. 1999), the wavelengths of the neutron beams, and the nominal Bragg angles for the chosen lattice planes (110) and (211) are listed in Table 6.1.

Table 6.1 Neutron diffraction parameters used in the strain tensor measurement

Lattice planes	Monochromators	Neutron wavelength λ (Å)	Nominal Bragg's Peak 2θ (°)
Fe (110)	Si220	2.67	82.23
Fe (211)	Si331AF	1.73	95.37

Lattice d-spacing of the reflections (110) and (211) for the tension and torsion samples in six or more independent directions were measured using Huber Circle at High Flux Isotope Reactor (HFIR) as shown in the Figure 6.5. The interplanar d-spacings were measured using a gage volume, defined by the incident slit $1 \times 1 \text{ mm}^2$ and receiving slit 1 mm. Extreme cautions were paid to make sure that the center of the gage volume is both at the center of the rotation of ϕ -plane of the goniometer and at the center of the rotation of the ψ -plane of the Huber Circle. Combination of the angles ϕ and ψ for different tilt in strain tensor measurement is given in Table 6.2.

In this study, the sample coordinate system S_i ($i = 1, 2, 3$) is oriented so that the axis S_1 is in radial direction, S_2 in circumferential direction, and S_3 in axial direction, as shown in Figure 6.6, where the measured lattice strain is in radial direction and the corresponding ϕ and ψ are 0 and 90 degree, respectively. Measurement of d-spacing of the (110) and (211) reflections for the unstressed reference samples were carried out using 20 combinations of the ϕ and ψ angles from Table 6.2, and the results are shown in Figure 6.7 and Figure 6.8. Average values of the unstressed d-spacing were obtained for further use in calculation of the lattice strains.

The measured and calculated strain tensor as well as the principal strains based on the (110) reflection for the torsion sample is given in Table 6.3. As 20 different combinations of the ϕ and ψ angles were used to calculate the six independent components in the strain tensor, it is of great importance to understand the effect of the quality and quantity of the neutron scattering measurements on the strain state calculated at the end. Typical examples of Least Squares fitting results and the corresponding calculated strain state in terms of the principal strains for the (110) reflection of the

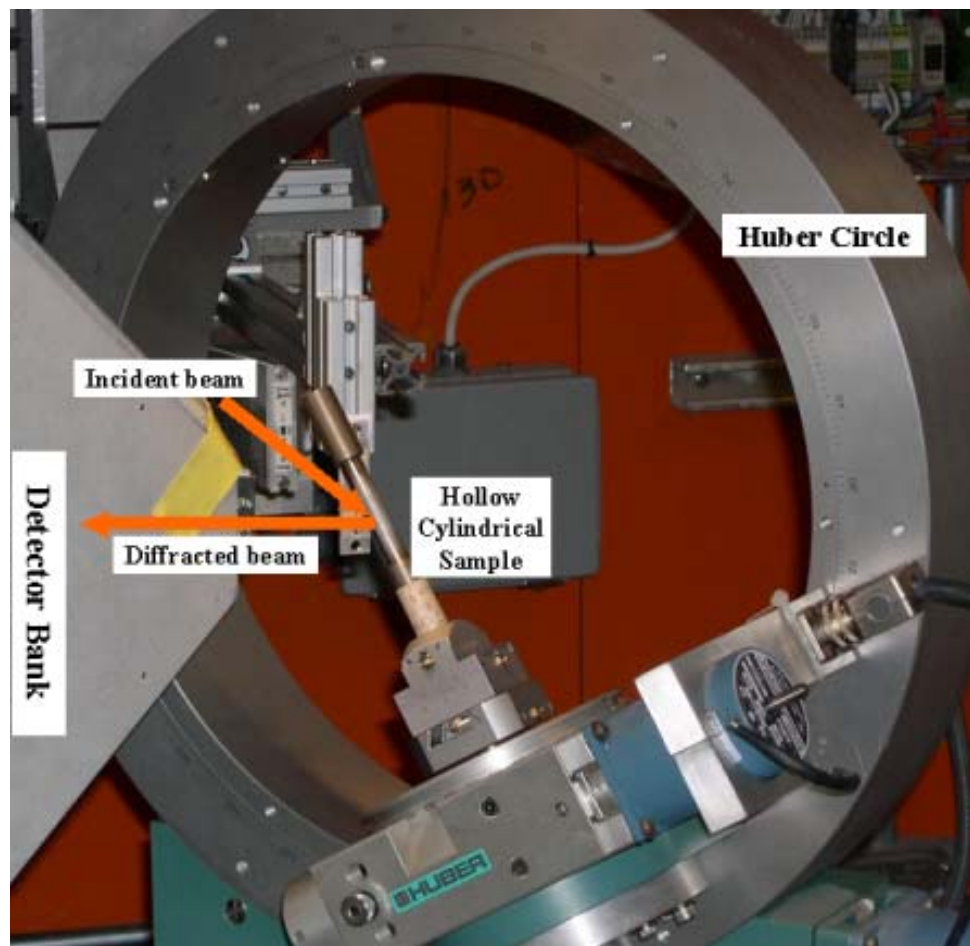


Figure 6.5 Experimental set-up for the residual strain tensor measurement at
HFIR, ORNL

Table 6.2 Combination of the angles ϕ and ψ for different tilts in strain tensor measurement

Run Number	Φ angle (deg)	ψ angle (deg)
1	0	90
2	30	90
3	60	90
4	90	90
5	0	60
6	30	60
7	60	60
8	90	60
9	0	45
10	30	45
11	60	45
12	90	45
13	0	30
14	30	30
15	60	30
16	90	30
17	0	0
18	30	0
19	60	0
20	90	0

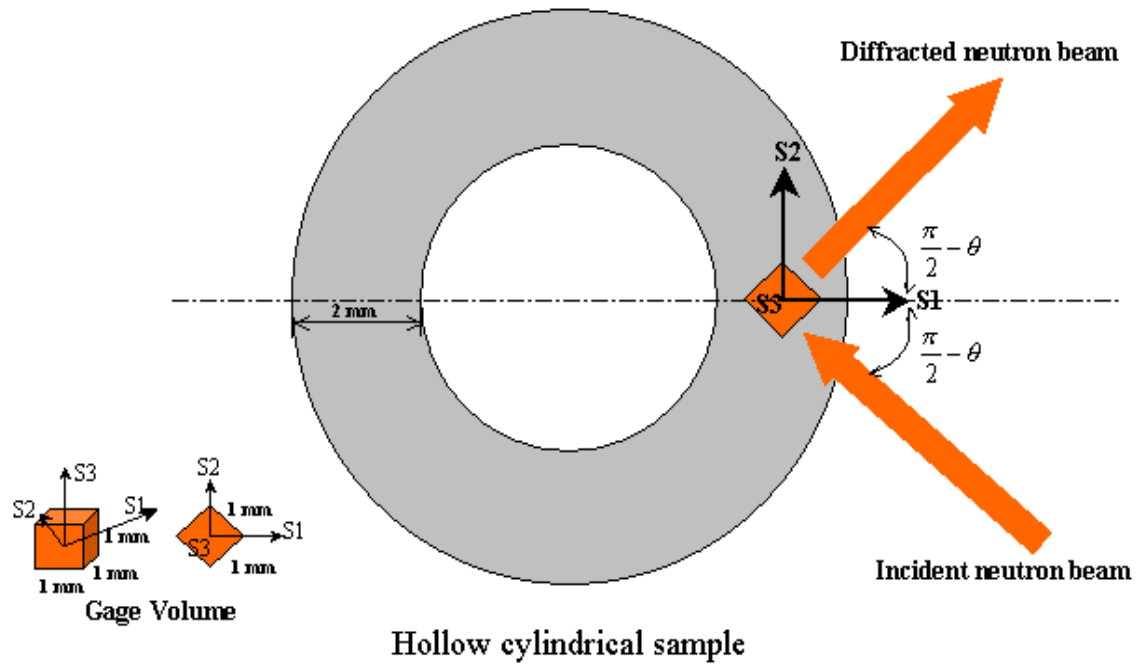


Figure 6.6 An example of the experimental set-up for the d-spacing measurement (radial direction) in order to obtain strain tensor

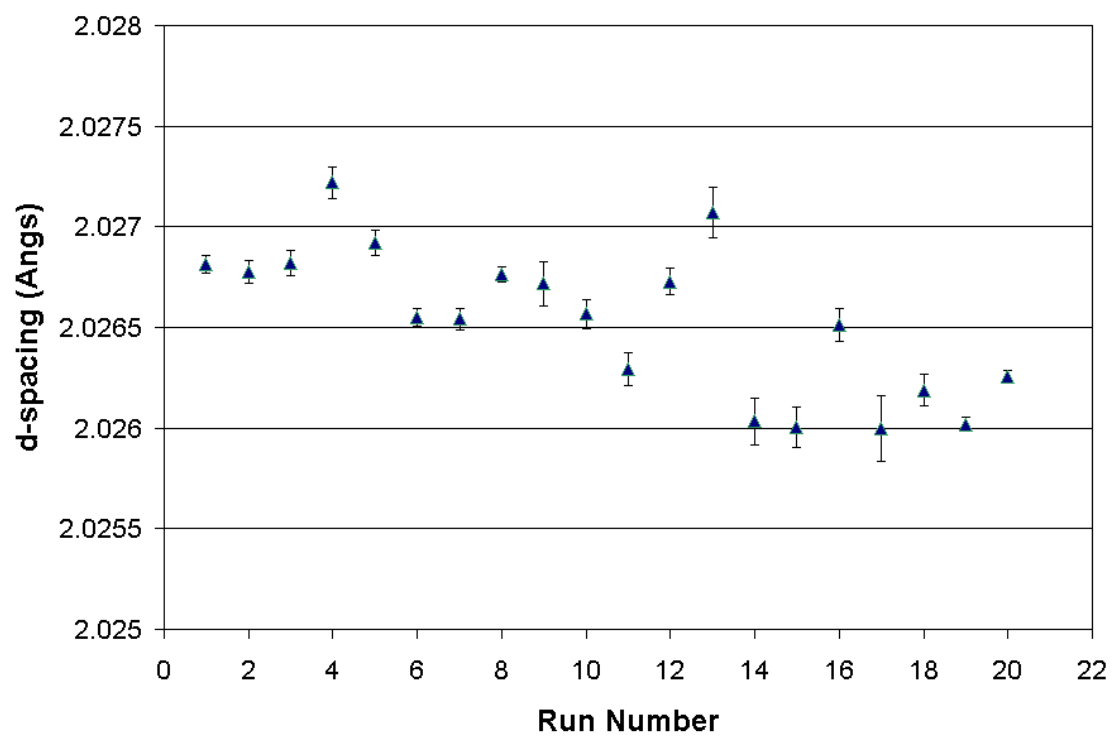


Figure 6.7 d-spacing of the (110) reflection for the unstressed sample

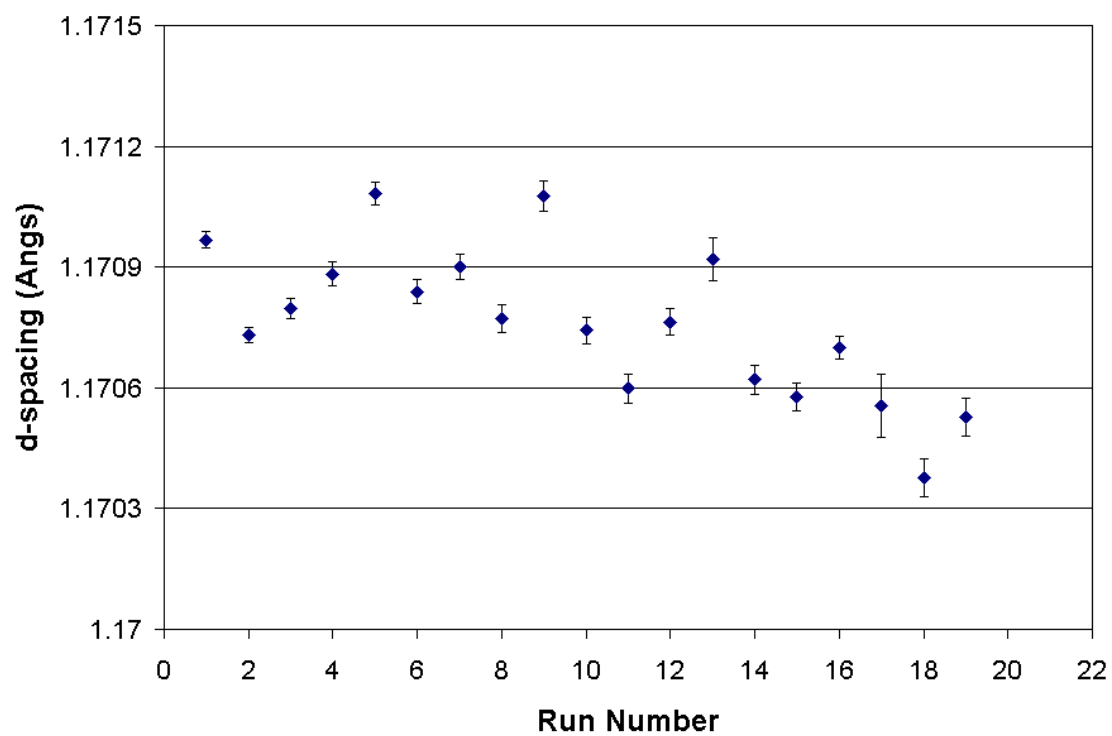


Figure 6.8 d-spacing of the (211) reflection for the unstressed sample

Table 6.3 Strain tensor and the corresponding principal strains ($\mu\epsilon$) for the (110) and (211) reflections of the torsion and the tension samples

	(110) Reflection	(211) Reflection
Torsion sample	$\varepsilon_{ij} = \begin{pmatrix} 825 & -317 & -314 \\ -317 & 385 & -12 \\ -314 & -12 & -53 \end{pmatrix}$ $\begin{pmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \varepsilon_3 \end{pmatrix} = \begin{pmatrix} 1060 \\ 275 \\ -178 \end{pmatrix}$	$\varepsilon_{ij} = \begin{pmatrix} 344 & -191 & 20 \\ -191 & 9 & 132 \\ 20 & 132 & -323 \end{pmatrix}$ $\begin{pmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \varepsilon_3 \end{pmatrix} = \begin{pmatrix} 432 \\ -380 \\ -23 \end{pmatrix}$
Tension sample	$\varepsilon_{ij} = \begin{pmatrix} 241 & -334 & 331 \\ -334 & 363 & 103 \\ 331 & 103 & -454 \end{pmatrix}$ $\begin{pmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \varepsilon_3 \end{pmatrix} = \begin{pmatrix} 660 \\ -639 \\ 128 \end{pmatrix}$	$\varepsilon_{ij} = \begin{pmatrix} 86 & -301 & 284 \\ -301 & 293 & -227 \\ 284 & -227 & -174 \end{pmatrix}$ $\begin{pmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \varepsilon_3 \end{pmatrix} = \begin{pmatrix} 657 \\ -358 \\ -94 \end{pmatrix}$

torsion sample are shown in the Table 6.4, in which the different Least Squares fittings using part or whole set of the lattice strain measurements to obtain a strain tensor were listed. The results show that the principal strains based on the different Least Squares fitting utilizing from 6 to 20 independent measurements also show the consistency. In particular, the calculated major principal strains are around $1100 \mu\epsilon$ in tension, and the minor principal strains around $150 \mu\epsilon$ in compression using the different Least Squares fitting, except for the fitting of using 6 independent measurement. This justifies on the other hand that the measurements to obtain the strain tensor are robust and consistent. Similarly, the calculated strain tensors and principal strains for the (110) reflection of the torsion sample, (211) and (110) reflections of the tension samples are provided in Table 6.3. The regression results from the Least Squares fitting show remarkable difference in the residual strain between (110) and (211) in the torsion and tension samples. On the other hand, for a given specific reflection, there is also considerable difference in the torsion and tension samples even though they were subject to the similar target deviatoric strain. The results show agreement in actually measured lattice strains in radial, circumferential, and axial directions for (110) and (211) reflections of both the tension and torsion samples, which indicates that the measured neutron diffraction data were consistent and the fitting procedure was robust.

Error estimate of strain tensor based on neutron diffraction

Error estimate, in particular, error propagation for the strain tensor is as much important as the measurement and calculation of strain/stress tensor itself. In some cases, the estimated standard deviation of the strain could be twice as large as the corresponding

Table 6.4 Comparison of the Least Squares fitting results for the neutron scattering measurements with different combinations of the ϕ and ψ angles

Run number included in the Least Squares fitting	Calculated principal strains ($\epsilon_1, \epsilon_2, \epsilon_3$) ($\mu\epsilon$ or $\times 10^{-6}$)
1 – 20	(1060, 275, -178)
1 – 19	(1060, 278, -176)
1 – 16	(1084, 393, -149)
1 – 12	(1168, 363, -124)
1 – 6	(1064, 560, -359)
5 – 20	(1130, 139, -161)

measured components in that strain tensor, or the error in measured reference cell parameter could be up to 700 MPa in some stress tensor components (Krawitz and Winholtz 1994). Therefore, it is of great interest to understand how the errors occurring in the neutron diffraction measurements propagate during the process in which those measurements are used to calculate the components of the strain or stress tensor. Effective efforts cannot be made to quantify and control those errors until the mechanism of the error propagation is fully understood.

One source of error results from the system of experimental facility, and a typical system error is due to measurement of the Bragg's peak by detectors. Such system error can be estimated by differentiating Bragg's Law equation, as given in Equation (6.38) (Hutchings, Withers et al. 2005).

$$\frac{\Delta d}{d} = \frac{\Delta \lambda}{\lambda} - \cot \theta \cdot \Delta \theta \quad (6.38)$$

where,

d – Interplanar d-spacing for the given reflection (hkl)

λ – Wavelength length of the radiation

θ – Angle associated with the Bragg's peak

If the error from wavelength of the radiation is not considered, then the error of the measured lattice strain can be represented by

$$\frac{\Delta d}{d} = -\cot \theta \cdot \Delta \theta \quad (6.39)$$

For example, at the 2nd Generation Neutron Residual Stress Mapping Facility (NRSF2), a typical peak position fitting precision is ± 0.003 degrees in 2θ . Therefore, for the d-

spacing measurement with the nominal Bragg's peak of 90 degree, the precision of lattice strain is around $\pm 26.2 \mu\epsilon$ nominally.

Another source of error results from the approach to obtain the strain tensor using neutron diffraction itself. In the process to obtain the strain tensor, statistical errors are produced when the Bragg's peaks are determined by regressing the neutron diffraction data, which are the neutron counts with respect to angles according to the position sensitive detectors (PSD). Thus, it is of great interest to understand how the statistical error from the obtained neutron data will affect the calculated strain tensor.

A general formula for uncertainty of a function $z(x, y)$ is Equation (6.40) (Taylor 1982).

$$\Delta z = \left| \frac{\partial z}{\partial x} \right| \Delta x + \left| \frac{\partial z}{\partial y} \right| \Delta y \quad (6.40)$$

where, Δx and Δy are uncertainties or errors from the quantities x and y

When the uncertainty Δx and Δy are independent and random, the variance of the function $z(x, y)$ can be represented by

$$\text{Var}(z) = \left(\frac{\partial z}{\partial x} \right)^2 \text{Var}(x) + \left(\frac{\partial z}{\partial y} \right)^2 \text{Var}(y) \quad (6.41)$$

The error of the function $z(x, y)$ will be estimated based on the standard deviation of the variance of z . The variance of the measured six components (e_j) of strain tensor can be represented in terms of the measured lattice strains (ϵ_i) at N times, as given in Equation (6.42).

$$\text{Var}(e_j) = \sum_{i=1}^N \left(\frac{\partial e_j}{\partial \epsilon_i} \right)^2 \text{Var}(\epsilon_i) \quad (6.42)$$

Based on Equation (6.29),

$$e_j = \sum_{k=1}^6 (\mathbf{A}^{-1})_{jk} \cdot \mathbf{B}_k \quad (6.43)$$

Substituting Equation (6.26) into (6.43) yields

$$e_j = \sum_{k=1}^6 (\mathbf{A}^{-1})_{jk} \cdot \left[\sum_{i=1}^N \varepsilon_i \cdot G_{ik}(\phi_i, \psi_i) \right] \quad (6.44)$$

So

$$\frac{\partial e_j}{\partial \varepsilon_i} = \sum_{k=1}^6 (\mathbf{A}^{-1})_{jk} \cdot G_{ik}(\phi_i, \psi_i) \quad (6.45)$$

Substituting Equation (6.45) into (6.42) yields

$$Var(e_j) = \sum_{i=1}^N \left(\sum_{k=1}^6 (\mathbf{A}^{-1})_{jk} \cdot G_{ik}(\phi_i, \psi_i) \right)^2 Var(\varepsilon_i) \quad (6.46)$$

The lattice strain (ε_i) measured using neutron diffraction can be defined as

$$\varepsilon_{\phi\psi} = \frac{d_{\phi\psi} - d_0}{d_0} \quad (6.47)$$

where, d_0 and $d_{\phi\psi}$ are the measured d-spacing for the unstressed and stressed sample at the combination of tilt angles (ϕ, ψ), respectively

The error of the measured lattice strain is estimated by differentiating Equation (6.47), so the variance of the lattice strain (ε_i) at the i^{th} ($i=1, 2, \dots, N$) time is

$$Var(\varepsilon_i) = \left(\frac{1}{d_0} \right)^2 Var(d_{\phi\psi}) + \left(\frac{d_{\phi\psi}}{d_0^2} \right)^2 Var(d_0) \quad (6.48)$$

where, d_0 and $d_{\phi\psi}$ are the related i^{th} measurement

However, in actual strain tensor measurement, if the average value of d-spacing for the unstressed reference sample is used, the variance in d_0 will use the averaged variance in d_0 of the N time measurement, given that all the measurements are

independent with each other. The error of the components (e_j) of strain tensor will be estimated with the standard deviation, the square root of the variance based on Equations (6.46). The estimated standard deviations calculated based on the above procedure for the (110) and (211) reflections of both the torsion and tension samples are give in Table 6.5. The results show that the estimated standard deviation for the calculated strain tensor are within the same magnitude of those for the individual measurement of the lattice strain measurement using the different combination of the φ and ψ angles. This also justifies that the neutron diffraction measurements are stable and consistent.

Table 6.5 Estimated standard deviation ($\mu\epsilon$) of the components of calculated strain tensor

	(110) Reflection	(211) Reflection
Torsion sample	$\begin{pmatrix} 69 & 71 & 57 \\ 71 & 71 & 57 \\ 57 & 57 & 37 \end{pmatrix}$	$\begin{pmatrix} 36 & 38 & 34 \\ 38 & 38 & 35 \\ 34 & 35 & 37 \end{pmatrix}$
Tension sample	$\begin{pmatrix} 67 & 72 & 57 \\ 72 & 76 & 60 \\ 57 & 60 & 39 \end{pmatrix}$	$\begin{pmatrix} 35 & 39 & 34 \\ 39 & 39 & 35 \\ 34 & 35 & 33 \end{pmatrix}$

Summary

The state of residual strain of the carbon steel tubular samples which were subject to either tension or torsion with a similar ultimate loading conditions in terms of the deviatoric strain, were investigated using neutron diffraction. An approach of the Least Squares fitting to obtain the tensor of residual strain based on six or over six independent measurements of lattice strains is presented. Multiple Least Squares fittings with input data from different combination of independent lattice strain measurements have shown the robust and consistency in the calculated strain state. The results also show the significant difference between the two samples, although the tension and torsion samples were subject to the similar deviatoric strain.

It is of great importance to quantify and control the errors of the calculated components of strain or stress tensor. Effective efforts cannot be made to quantify and control those errors until the mechanism of the error propagation is fully understood. A novel procedure was developed to understand the mechanism and to analyze the errors of the calculated strain tensors based on the errors occurring the neutron diffraction measurements. Error propagation of the strain tensor calculation based on the errors from both the measured lattice strains and the measured data in the unstressed reference sample was analyzed. The results show that the estimated standard deviation for the calculated strain tensor are within the same magnitude of those for the individual measurement of the lattice strain measurement using the different combination of the φ and ψ angles.

Acknowledgement

The support from JINS Fellowship program and NSF IMI program (DMR-0231320) is greatly appreciated. Research at Oak Ridge National Laboratory (ORNL) was sponsored by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of FreedomCAR and Vehicle Technologies, as part of the High Temperature Materials Laboratory User Program, ORNL, managed by UT-Battelle, LLC, for the US Department of Energy under contract number DE-AC05-00OR22725.

References

- ASM (1990). Metals handbook (10th ed.) Volume 1 Properties and Selection: Iron, Steel, and High Performance Alloys. Materials Park, OH, ASM International.
- Brown, D. W., M. A. M. Bourke, et al. (2003). "A neutron diffraction and modeling study of uniaxial deformation in polycrystalline beryllium." Metallurgical and Materials Transactions a-Physical Metallurgy and Materials Science **34A**(7): 1439-1449.
- Cullity, B. D. and S. R. Stock (2001). Elements of X-Ray Diffraction. Upper Saddle River, Prentice Hall.
- Holden, T. M., J. H. Root, et al. (1995). "Neutron-Diffraction Measurements of Stress." Physica B **213**: 793-796.
- Hutchings, M. T. (1992). "Neutron diffraction measurements of residual stress fields - the engineer's dream come true?" Neutron News(3): 14-19.
- Hutchings, M. T., P. J. Withers, et al. (2005). Introduction to the characterization of residual stress by neutron diffraction. Boca Raton, CRC Press.
- Jones, A. M. (1989). Residual stresses: A review of their measurement and interpretation using X-ray diffraction, Harwell Laboratory, Materials Development Division.
- Kandil, F. A., J. D. Lord, et al. (2001). A Review of Residual Stress Measurement Methods - A Guide to Technique Selection. Middlesex, UK, NPL Materials Centre.
- Krawitz, A. D. and R. A. Winholtz (1994). "Use of Position-Dependent Stress-Free Standards for Diffraction Stress Measurements." Materials Science and

Engineering a-Structural Materials Properties Microstructure and Processing
185(1-2): 123-130.

Larsson, C., B. Clausen, et al. (2004). "Measurements and predictions of strain pole figures for uniaxially compressed stainless steel." Scripta Materialia **51**(6): 571-575.

Lu, J. and D. Retraint (1998). "A review of recent developments and applications in the field of X-ray diffraction for residual stress studies." Journal of Strain Analysis for Engineering Design **33**(2): 127-136.

Noyan, I. C. and J. B. Cohen (1987). Residual Stress Measurement by Diffraction and Interpretation. New York, NY, Springer-Verlag.

Noyan, I. C., T. C. Huang, et al. (1995). "Residual-Stress Strain Analysis in Thin-Films by X-Ray-Diffraction." Critical Reviews in Solid State and Materials Sciences **20**(2): 125-177.

Oliver, E. C., M. R. Daymond, et al. (2002). Stress induced martensitic transformation studied by neutron diffraction. Ecrs 6: Proceedings of the 6th European Conference on Residual Stresses. Zurich-Uetikon, Trans Tech Publications Ltd.
404-4: 489-494.

Pang, J. W. L., T. M. Holden, et al. (2000). "The generation of intergranular strains in 309H stainless steel under uniaxial loading." Acta Materialia **48**(5): 1131-1140.

Pintschovius, L. (1992). Grain Interaction Stresses. Measurement of Residual and Applied Stress Using Neutron Diffraction. M. T. Hutchings and A. D. Krawitz. Dordrecht, Kluwer Academic Publishers: 189-204.

- Pintschovius, L. (1992). Neutron Versus X-rays. Measurement of Residual and Applied Stress Using Neutron Diffraction. M. T. Hutchings and A. D. Krawitz. Dordrecht, Kluwer Academic Publishers: 577-580.
- Saada, A. S. (1983). Elasticity: Theory and Applications. Malabar, FL, Krieger Publishing.
- Saada, A. S. (1988). Hollow Cylinder Torsional Devices: Their Advantages and Limitations. Advanced Triaxial Testing of Soil and Rock. R. T. Donaghe, R. C. Chaney and M. L. Silver. Philadelphia, PA, American Society for Testing and Materials: 766-795.
- Stoica, A. D., M. Popovici, et al. (1999). Neutron Monochromators for Residual Stress Mapping at the New HB-2 Beamport. Oak Ridge, TN, Oak Ridge National Laboratory.
- Taylor, J. R. (1982). An Introduction to Error Analysis: The study of Uncertainties in Physical Measurements. Mill Valley, University Science Books.
- Webster, P. J. (1992). Spatial Resolution and Strain Scanning. Measurement of Residual and Applied Stress Using Neutron Diffraction. M. T. Hutchings and A. D. Krawitz. Dordrecht, Kluwer Academic Publishers: 235-252.
- Winholtz, R. A. and J. B. Cohen (1988). "Generalized Least-Squares Determination of Triaxial Stress States by X-Ray-Diffraction and the Associated Errors." Australian Journal of Physics **41**(2): 189-199.

Chapter 7 Summary and Future Work

Summary

In this research, advanced neutron techniques of radiography and diffraction have been utilized to investigate fundamental issues in the Lost Foam Casting process and in mechanical behavior of infrastructure materials such as granular silica and carbon steel.

Time lapsed neutron radiography with digital image processing has been used to investigate the real-time Lost Foam Casting (LFC) process, which was carried out at McClellan Nuclear Radiation Center, University of California, Davis. Evidence has been presented that neutron radiography is able to overcome the difficulty of other traditional techniques such as X-ray radiography due to unique neutron cross-section, and offer new insights into the pyrolysis of foam and the dynamics of casting. Based on the results from radiography combined with the image processing and analysis of the still images extracted from the real-time neutron videos, the pyrolysis front can be clearly identified and isolated from the molten metal as well as the undecomposed polymer foam. Behavior and characteristics of the molten metal and the pyrolysis front in the LFC processes were revealed. The proposed approach will prove to be a powerful tool to characterize the pyrolysis front of the expanded polystyrene foam during LFC process during the interaction with liquid metal.

Stress-strain relationship of particulate materials is complex, and depends on the initial state of packing, past stress history, and the applied stress path. A novel methodology has been developed using neutron scattering technique to obtain strains both globally and locally for assemblies of granular materials. The significant difference between the global macroscopic deformation and the local interplanar lattice strain has been observed during the in-situ study of the granular assemblies under confined

compressive loading at the High Flux Isotope Reactor (HFIR), Oak Ridge National Laboratory. The measured lattice strain within particles was at least one order of magnitude smaller than the measured global strain when the granular materials were subject to same external loading. For the round Ottawa particles used in this study, when the applied load exceeded certain stress, significant particle breakage occurred resulting in a large increase in the rate of global strain variation as a function applied compressive stress. For the angular Q-Rok particles, multiple fluctuations in the curve of global stress vs lattice strain were observed, indicating multiple fracturing and stress relaxation within particles. Based on the lattice strain mapping data, there is no evident trend of lattice strains at different applied global stress levels, and the measured lattice strains are scattered, which implies a strong inhomogeneous distribution of the actual stress in particles. Research results in this study will be useful for developing suitable elasto-plastic constitutive models of frictional granular materials, and can also be applied to powder metallurgy and sintered ceramics.

Residual stress is one of the more common phenomenon occurring in materials, and has a significant impact on the mechanical behavior and the durability of materials. Residual stress is not immediately apparent in many cases; it is difficult to be measured, difficult to be predicted and can lead to premature failure of materials if not appropriately considered in design. Torsion provides an opportunity to probe mechanical behaviors of materials under pure shear stress, and in combination with axial load, provides a mechanism to rotate principal stresses in a controlled fashion. Residual strains of identical steel tubular specimens were investigated after subjected to pure torsion or uniaxial tension that corresponds to a target equivalent strain invariant using both reactor

neutron source from the High Flux Isotope Reactor at Oak Ridge National Laboratory and spallation neutron source from the Spectrometer for Materials Research at Temperature and Stress at Los Alamos National Laboratory. The lattice strain based on the reflections that have been reported to be both weakly and strongly affected by intergranular strain for tension stress path was investigated. The results have shown the promise to reveal the essential difference between tension and torsion from the perspective of yield criteria for materials. This is the first study of its kind for evaluating residual strains in generalized loading conditions and also will help to demonstrate the need for having a multi-axial loading system for the anticipated under construction engineering stress testing facility at Spallation Neutron Source.

An innovative approach has been developed to investigate the strain/stress state in terms of strain/stress tensors by using Huber Circle and diffractometer at the 2nd Generation Neutron Residual Stress Facility at Oak Ridge National Laboratory. A computer program has also been developed, which is capable of processing the raw data from neutron scattering to obtain the strain state in stressed materials. It is also of great interest to understand how the errors occurring the neutron diffraction measurements propagate during the process in which those measurements are used to calculate the components of strain or stress tensor. A procedure was developed to understand the mechanism and to analyze the errors of the calculated strain tensors based on the errors occurring the neutron diffraction measurements. Error propagation of the strain tensor calculation based on the errors from both the measured lattice strains and the measured data in the unstressed reference sample was analyzed. The results show that the estimated standard deviation for the calculated strain tensor are within the same magnitude of those

for the individual measurement of the lattice strain measurement using the different combination of the ϕ and ψ angles. The newly developed approaches make it possible to study the strain/stress tensor in materials under completely three-dimensional conditions.

Future work

Future work could be performed in the following aspects.

- Quantitative analysis and modeling relating to the pyrolysis front's complex behavior associated with various basic casting parameters such as coating type, gating system, and foam thickness during the Lost Foam Casting is needed in order to better understand the fundamentals which is the foundation for quality control for Lost Foam Casting process.
- Macroscopic load vs. deformation relationship in elastic particulate materials, which were considered as breakage free, has been studied using the distinct element method (DEM). However, there is no research work of establishing the relation between the simulated load-deformation behavior at macroscopic level and the measured lattice strains at microscopic levels. In particular, for the silica sand under compression, a distinct element method could be used to model the elastic portion of the experiment and contact dynamics. The influence of particle elasticity could be possibly separated from the effect that particle rearrangements have on the macroscopic stress-strain behavior found in the compression testing.
- Due to limited time, the effective measurements of residual strains based on multiple reflections were conducted only at the middle of the wall thickness of both tension and torsion samples in the radial and circumferential directions.

Extension of this study on residual strain from 2-D to 3-D at different spatial locations through the wall thickness of the tubular samples using the spallation neutron source at the Los Alamos National Laboratory or Spallation Neutron Source would reveal more details about the residual stress/strain state from the different reflections either sensitive or insensitive to the intergranular strain/stress.

- This research has already pushed the boundary of the experimental research on both in-situ and ex-situ measurement of interplanar d-spacing at the neutron scattering facilities – High Flux Isotope Reactor from Oak Ridge National Laboratory and Spectrometer for Materials Research at Temperature and Stress Los Alamos National Laboratory. It also demonstrates a need that future work should use the more powerful Spallation Neutron Source (SNS) in engineering material research.
- For the study of residual strain under generalized load condition such as the combination of torsion and tension, numerical modeling using elasto-plastic self-consistent (EPSC) model with the specialty for incorporation with the important concept of intergranular strain/stress in the polycrystalline materials is needed. Yield and failure criteria such as Von-Mises or Mohr-Coulomb criteria are of importance because the prediction of the residual strain will be significantly affected using different failure criteria.
- More studies on the stress/strain state in materials subject to generalized, controlled loading conditions are needed in order to better understand the fundamentals of the failure behavior of engineering materials. Neutron diffraction

seems to be a very viable and highly effective technique to explore stress/strain tensor in engineering materials.

Vita

Xin Luo was born in the southwest of China on February 16, 1973. He was raised in Chongqing and attended grade school and high school there. He attended Southeast University in Nanjing, Jiangsu Province, and received a bachelor's degree in Civil Engineering in 1995 and a master's degree in Materials Science and Engineering in 1998. He worked as a research and teaching assistant at the Department of Civil and Structural Engineering, the Hong Kong Polytechnic University during 1998 – 1999. He later joined Wuxi TYFO Engineering Composites Co. Ltd. as a senior engineer providing consulting services for the patented FRP composite systems used in structural strengthening and retrofitting. In August 2003, he started his new journey, pursuing his doctoral degree in Civil Engineering at the University of Tennessee, Knoxville. His research has been focused on the study of the infrastructure materials using innovative nondestructive techniques such as neutron radiography and neutron diffraction.