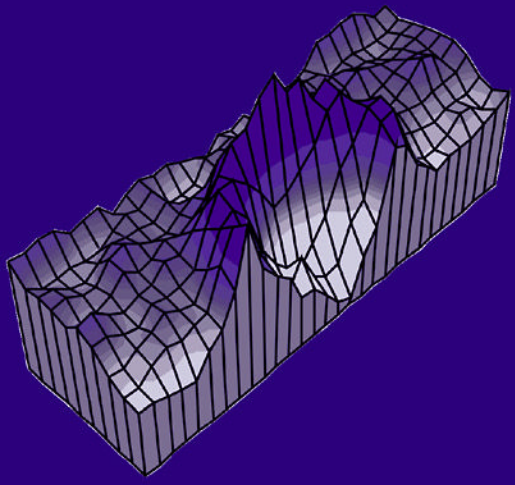

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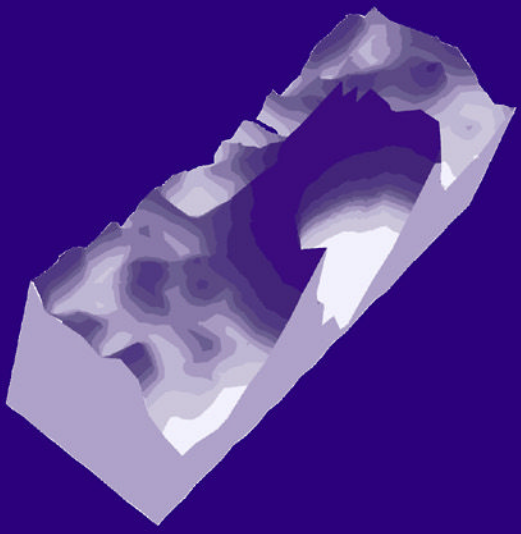

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Taylor & Francis
Taylor & Francis Group
LONDON AND NEW YORK

First published 2003
by Taylor & Francis
11 New Fetter Lane, London EC4P 4EE

Simultaneously published in the USA and Canada
by Taylor & Francis Inc,
29 West 35th Street, New York, NY 10001

Taylor & Francis is an imprint of the Taylor & Francis Group

This edition published in the Taylor & Francis e-Library, 2003.

British Library Cataloguing in Publication Data
A catalogue record for this book is available from the British Library

Library of Congress Cataloging in Publication Data
A catalog record for this book has been requested

ISBN 0-203-60899-2 Master e-book ISBN

ISBN 0-203-34593-2 (Adobe eReader Format)
ISBN 0–415–30397–4 (Print Edition)

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Preface

The presence of residual stresses in materials and components can have an important influence on their behaviour. Residual stresses can be introduced during manufacture and/or use by such processes as mechanical forming operations, welding and heat treatments. Generally those processes that result in near surface residual compression are beneficial and enhance resistance to failure whereas those that produce surface tension usually aid the onset of cracking which can lead to premature fracture.

Certain manufacturing operations, such as machining and welding, can produce undesirable surface residual tension. Often it is necessary to apply heat treatments to relax these stresses prior to use. For high-performance applications in, for example, the aerospace, automotive and chemical process industries where cyclic loading is involved, manufacturing operations such as shot peening, autofrettage, chemical surface treatment and laser surface hardening are often employed deliberately to introduce beneficial surface compression to enhance resistance to fatigue failure. In order to predict the influence of residual stress on material or component behaviour, it is essential to have an accurate knowledge of the magnitude of the stresses generated.

Residual stresses can be determined by experimental methods or by calculation from models of the processes responsible for their production. The experimental methods can be destructive or non-destructive. The destructive methods usually involve cutting or drilling operations to relax the residual stresses. The residual stresses are then calculated from the resulting dimension changes. Mostly the non-destructive methods use diffraction techniques. With this approach elastic strains are measured and residual stresses calculated from the elastic properties of the materials concerned. Diffraction techniques can employ conventional X-rays, synchrotron radiation or neutrons. In the former case only surface determinations of stress, to less than 100 μm deep, can be made unless progressive polishing away of the surface is used. With synchrotron radiation and neutrons, penetration depths of a few millimetres or centimetres, respectively, can be achieved in specific materials. The determination of residual stresses from models requires an accurate understanding of the processes responsible for the stresses and of the elastic and plastic properties of the materials being investigated. This book concentrates on the experimental determination of residual stress by the synchrotron radiation and neutron diffraction methods.

The book consists of individual chapters which are authored by an international collection of the foremost specialists in the field of residual stress measurement. The book is effectively split up into four parts dealing with, respectively, the principles of the techniques, how stresses are extracted from the strain measurements, the measurement procedures employed and applications to particular fields of study. The main emphasis is on the neutron diffraction method since this is the most established technique.

Both methods can only be applied to crystalline materials. Initially each method is discussed in turn. The theory of the interaction of neutrons with specific crystallographic planes to produce lattice strains is reviewed and procedures for generating neutrons covered. There is then a similar presentation of the synchrotron radiation method. It is described how the process provides a very intense beam of high-energy X-rays which can be used to determine elastic strains by diffraction in the same way as with neutrons.

Part 2 deals mainly with the factors that have to be taken into account in calculating stresses from the experimental measurements. It is described how three types of residual stress can be identified. These are separated into what are commonly called macro residual stresses (type I) and micro residual stresses (types II and III). The macro residual stresses are those which exist over the scale of the dimensions of a sample or component and are of most interest for engineering stress analysis and continuum mechanics applications. The type II stresses are those which exist across grains as a result of the anisotropy of grains and the constraints to deformation that occur between them. They can also exist between different phases in materials or composites. Type III microstresses are those residual stresses that are produced at the atomic level due to the presence of defects and precipitates in materials. The choice of crystallographic planes on which to make measurements for particular applications is discussed. Methods of extracting macro- and microstresses from the data are presented. The most appropriate elastic constants for use in calculating stresses in mono- and polycrystals are considered, as is the role of texture. The information needed to determine the full residual stress sensor is also discussed.

Specific experimental measurement techniques for obtaining residual stresses are examined in Part 3. Several types of instrument have been developed. Designs which employ a monochromatic beam of neutrons and those which use a pulsed polychromatic beam are both described. Detector systems and data analysis procedures are discussed. The principles of obtaining residual stresses from neutron transmission measurements of Bragg edges are also presented. Recent advances in the development of the synchrotron radiation technique are then outlined.

The final part considers a range of applications of the techniques. It is shown that neutron diffraction can be employed successfully for measuring residual stresses in single crystal and large grained materials, metals, ceramics and composites. It is demonstrated that it can also be used to measure stresses close to and through surfaces although precautions are required to avoid surface aberration effects. Manufacturing processes that are examined include shot peening and welding. It is noted that with welding, and other situations where metallurgical changes and/or gradients can exist, considerable care is needed in establishing the appropriate stress-free crystallographic lattice spacing to adopt when determining strains. The role of synchrotron radiation for performing rapid area strain scans and for identifying steep stress gradients is also examined.

Synchrotron radiation and neutron diffraction are complementary non-destructive methods for measuring residual stresses. The book is particularly timely. It is appearing at a time when the neutron diffraction technique is becoming established and when an international standard is to be issued recommending the experimental and data analysis procedures to be adopted to ensure reliable determinations of residual stress. It is also appearing at a time when dedicated synchrotron X-ray diffraction facilities are becoming available and more accessible for making residual stress measurements. The book is timely, particularly, because of the capability of modern computer analysis procedures. Sufficient computing power now exists to allow advanced stress analysis and modelling calculations to be carried out. They can be applied to components to account for the influence of residual stress on performance.

In addition they can be used in material science studies for predicting the residual stresses that are generated by different processes and within individual grains in polycrystalline materials. These stresses can then be compared to experimental measurements to validate the models.

The book should prove to be a valuable reference source for a wide audience. It is relevant to the fields of engineering and materials science. It is particularly appropriate to all students, researchers and industrialists with an interest in residual stress.

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Acknowledgements

In this book it has been our intention to provide a state-of-the art review of the application of diffraction techniques to the determination of residual stress in materials and components. Neutron diffraction has become well established over the last decade, and we are now beginning to see the rapid emergence of synchrotron X-rays as a useful and attractive technique.

For a book of this nature, thanks are due primarily to the authors who have supplied the chapters; the book would not exist without their efforts.

We wish to acknowledge the support of the Department of Materials Engineering at The Open University and the Laboratoire Léon Brillouin, Saclay, for secretarial support in aid of the production of the book. Particular thanks are due to Chantal Pomeau for styling and collating the final draft.

The cover images were provided by Dr J. Santisteban of The Open University.

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Part 1

General applications of neutron and synchrotron radiation to materials research

1 The use of neutrons for materials characterization

C. H. de Novion

1.1 Introduction and historical background

The neutron was discovered by Chadwick in 1932. The fission of the uranium nucleus, that is, its splitting in two fragments after having absorbed a neutron, was demonstrated in 1938 by Joliot-Curie and Hahn. The fission process leads to the emission of a few neutrons, making a chain reaction possible: nuclear reactors work on this principle.

As any moving particles, neutrons have a wave character (the "matter waves" first put forward by de Broglie around 1923). Because their mass is close to that of individual atoms, slow neutrons in thermodynamical equilibrium with a medium at ambient temperature (such as the moderator in a fission reactor), called "thermal" neutrons, have altogether a wavelength of the order of interatomic distances in condensed matter ($\sim$0.1 nm), and kinetic energies close to atomic vibration energies ($\sim 10^{-2}$ eV). They then give rise, like X-rays, to diffraction by crystalline solids; moreover, within the solid, they also can absorb or emit quanta of collective vibration energy ("phonons"), raising the possibility of spectroscopic studies (e.g. measurement of elastic wave dispersion curves) by inelastic neutron scattering. Both phenomena, diffraction and inelastic scattering, are observed by analysing the change of direction and speed of thermal neutrons "scattered" by a sample of solid or liquid matter.

Thermal neutron diffraction and inelastic scattering by crystals, theoretically predicted, were experimentally discovered in the late 1940s around the reactors built in the frame of the Manhattan project in the US. The 1994 Nobel prize in physics was awarded to C. Shull and B. Brockhouse for the development of neutron scattering techniques. Neutron scattering was found at that time to be a unique method to study lattice dynamics and (because the neutron bears a "spin" and an individual magnetic moment) to reveal the ordered magnetic structures of crystalline solids [1]. Dedicated single crystal and powder spectrometers for elastic (diffractometers) and inelastic ("triple-axis" instruments) scattering were installed on multipurpose reactors built in the 1950s and the 1960s, the largest of which being those in Oak Ridge, Brookhaven and Chalk River in North America.

A major progress was reached with the construction of the High Flux Reactor (HFR) of the Laue-Langevin Institute (ILL) in Grenoble (resulting from a French–German initiative, rapidly joined by the British), which opened in 1972, entirely dedicated to fundamental research with thermal neutrons, and specially designed for this purpose. ILL initiated an external user program, which progressively allowed it to receive a large number ($>$1000 per year) of scientists from many laboratories. The principle of an external user facility was then applied in several other centers (e.g. the NIST Center for Neutron Research, Gaithersburg, in the US).

Very important technical progress was made in the 1960s and 1970s (e.g. "cold" and "hot" neutron moderators, neutron guides, neutron polarization, etc.), and new neutron scattering techniques developed: small-angle neutron scattering (SANS), quasi-elastic scattering by time-of-flight (TOF), backscattering or spin-echo techniques, neutron reflectivity, etc. Progressively, neutron scattering broadened its applications to larger scientific domains: solid state chemistry, liquids, soft matter, materials science, geosciences, biology, etc. (for a general overview, see Ref. [2]).

Neutrons can also be produced by another type of nuclear reaction, called "spallation," obtained by hitting a target with protons (see below). Unlike the reactors, which produce continuous neutron flux, spallation sources generate pulses of neutrons. Spallation sources for neutron scattering were developed in the 1980s (first in Tsukuba, Japan and in Argonne, US); presently, the most intense is ISIS, at Abingdon in the UK. Generally, it is estimated that the potential of progress of pulsed spallation sources is much larger than that of reactors. Several regional new pulsed spallation sources are either in construction (SNS at Oak Ridge in the US), decided (in Japan) or planned (in Europe): they should allow major progress in the structural and spectroscopic study of materials in the coming decades.

1.2 Characteristics of the neutron particle

The neutron is a particle of zero electric charge, of mass $m = 1.67 \times 10^{-24}$ g, of radius $r_0 = 6 \times 10^{-16}$ m, with a spin of 1/2 and a magnetic moment $\mu = -1.9\mu_N$ (nuclear magnetons). One may note that the mass of the neutron is practically equal to that of the hydrogen atom (the proton and the neutron are two different charge states of the "nucleon"), and its radius is 5–6 orders of magnitude smaller than the average size of an atom ($\sim 10^{-10}$ m).

The following relationships can be written between the properties of the neutron, considered either as a particle or as a wave: velocity $\mathbf{v}$, energy E, momentum $\mathbf{p}$, wavelength λ, wavevector $\mathbf{k}$:

$$\hbar\mathbf{k} = m\mathbf{v} = \mathbf{p}, \quad E = \frac{1}{2}m\mathbf{v}^2 = \frac{h^2}{2m\lambda^2} = \frac{\hbar^2\mathbf{k}^2}{2m}$$

($\hbar = h/2\pi$: reduced Planck constant).

For example, thermal neutrons with a wavelength of 0.4 nm (=4 Å) have a speed of 10^3 m/s and a (kinetic) energy of 5 meV.

It is interesting to note that the wave nature of the neutron has been directly demonstrated by neutron interferometry experiments, quite analogous to light interferometry [3].

1.3 Production of neutrons

Neutrons are particles difficult and expensive to produce: one reason for this is the necessity of biological protection and of safety procedures to prevent accidents.

Presently, two types of sources produce beams of thermal neutrons: nuclear fission (steady state) reactors and neutron spallation (pulsed) sources. They are schematically shown in Figures 1.1 and 1.2, respectively. The major neutron sources for fundamental research, and some of their characteristics, are listed in Table 1.1.

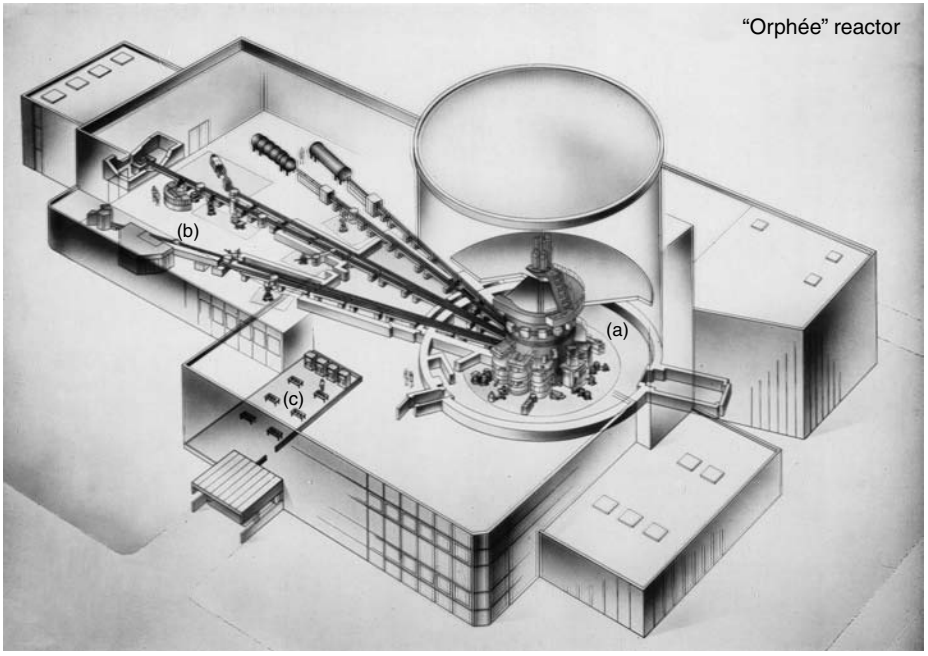

Figure 1.1 Perspective view of a fission reactor for thermal neutron beam production (the Orphée reactor, located in Saclay, France; note that not all instruments have been drawn, for clarity): (a), reactor hall: (b), guide hall: (c), computing facilities. (See Colour Plate I.)

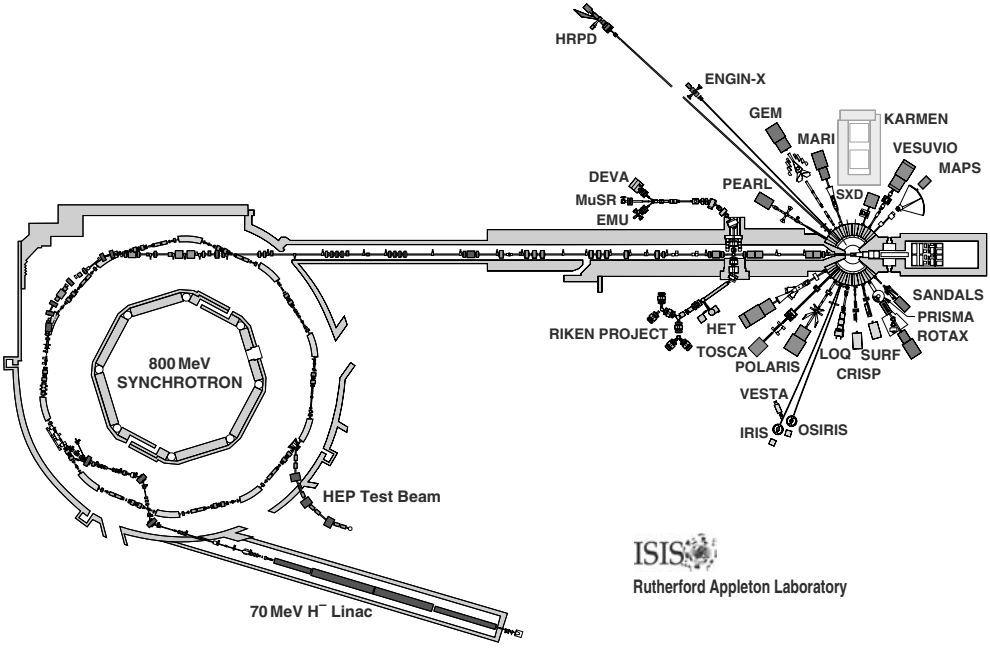

Figure 1.2 Plan of the ISIS facility (spallation source and instruments), located in Abingdon, UK. (See Colour Plate II.)

Table 1.1 Major thermal neutron beam sources

Continuous sources

Country	*Source*	*Location*	*First operation*	*Power* (MW)	*Thermal flux* (10^{14} n/cm^2/s)	*Special moderator*[a]	*Scattering instruments*	*Stress diffractometer*
Australia	HIFAR	Lucas Heights	1958	10	1.4	No	7	0
Canada	NRU	Chalk River	1957	120	3.0	No	6	1
Denmark[b]	DR3[b]	Risø	1960	10	1.5	1 C	8	1
France[c]	HFR-ILL[c]	Grenoble	1972–1994	58	12.0	2 C, 1 H	32	([d])
France	Orphée	Saclay	1980	14	3.0	2 C, 1 H	25	1
Germany	BER-2	Berlin	1973–1986	10	2.0	1 C	16	1
Germany	FRJ-2	Jülich	1962	23	2.0	1 C	16	0
Hungary	BNC	Budapest	1959	10	1.6	1 C	7	1
Japan	JRR-3	Tokai	1962	20	2.0	1 C	23	1
Korea	Hanaro	Taejon	1996	30	2.8	No	6	1
Sweden	R-2	Studsvik	1960	60	1.0	No	5	1
Switzerland	SINQ	Villigen	1996	1000 kW (spallation source)	2.0	1 C	13	1
USA[b]	HFBR[b]	Brookhaven	1965	30	4.0	1 C	14	1
USA	HFIR	Oak Ridge	1960	85	12.0	1 C	9	1
USA	NBSR-NIST	Gaithersburg	1969	20	2.0	1 C	17	1
Under construction								
Germany	FRM-II	München	2003[e]	20	7.0	1 C, 1 H	17	1

Pulsed sources

Country	*Source*	*Location*	*First operation*	*Beam power* (kW)	*Pulse length (proton) and repetition rate*	*Thermal peak flux* (10^{14} n/cm^2/s)	*Moderators*[a]	*Scattering instruments*	*Stress diffractometer*
Japan	KENS-KEK	Tsukuba	1980	3	0.1 μs; 20 Hz	3	1 C, 1 T	16	0
Russia	IBR2	Dubna	1984	2000 (fission)	305 μs; 5 Hz (thermal neutrons)	100	1 C, 3 T	11	1
UK	ISIS	Abingdon	1985	160	0.4 μs; 50 Hz	20–100	2 C, 2 T	19	1
USA	LANSCE	Los Alamos	1985	56	0.27 μs; 20 Hz	34	1 C, 3 T	7	([d])
USA	IPSN	Argonne	1980	7	0.1 μs; 30 Hz	5	3 C	13	([d])
Under construction									
USA	SNS	Oak Ridge	2006[e]	2000	1 μs; 60 Hz	200	2 C, 2 T	10	1

Source: Richter and Springer [7].

Notes

Two other new reactors have been approved and should start in the next decade: the PIK reactor in Gatchina, St. Petersburg (Russia), with performances close to those of ILL, and a new medium flux multipurpose reactor HIFAR II (which will replace the present HIFAR) in Lucas Heights, Australia.

Apart from the Spallation Neutron Source (SNS) in the US presently in construction, two other large regional spallation sources are planned, with a power of 5 MW, a neutron scattering facility within the JHP/NSRP multipurpose Joint Project in Tokai (Japan), which has recently been decided and should start to operate in 2007, and the European Spallation Source (ESS) which is not decided yet, but has an anticipated start date around 2013. A medium-flux spallation source in Central Europe is also in project (AUSTRON, 0.5 MW).

It is worth mentioning that high-quality stress diffractometers are also found in four continuous sources not mentioned in the table: at the High Flux Reactor in Petten (The Netherlands), at the FRG reactor in Geesthacht (Germany), at the Rez reactor near Prague (Czech Republic) and at the reactor MURR of the University of Missouri (Columbia, USA).

a Moderator – C, cold; T, thermal; H, hot.
b The Brookhaven and Risø reactors have been shut down since 1998 and 2000, respectively.
c The Institut Laue-Langevin (ILL), located in Grenoble (France), is a multinational institute, managed by a consortium of three major associates (France, Germany, UK) and several minor Scientific Member countries (Switzerland, Spain, Italy, Russia, Austria and Czech Republic).
d Stress experiments performed on a general purpose high-resolution powder diffractometer.
e For sources in construction (FRM-II reactor and SNS spallation source), these are anticipated starting dates.