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Neutrons for industry and engineering

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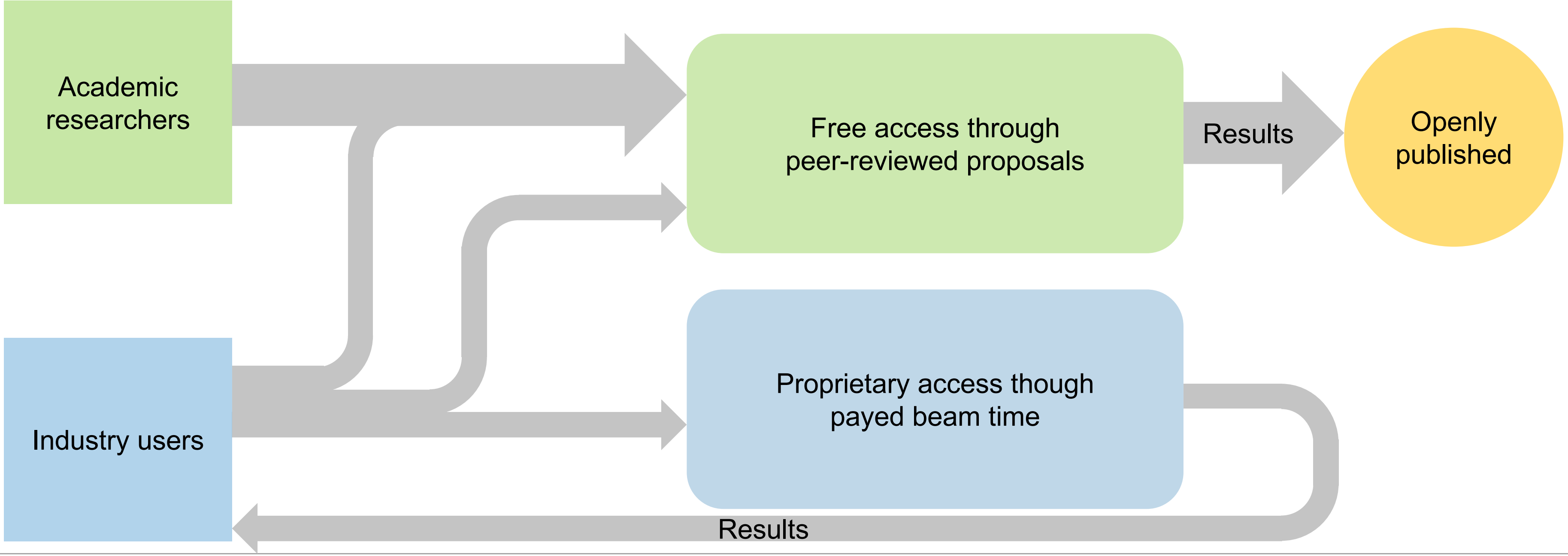
Neutrons for industry and engineering

Aim of this lecture: to provide an insight into how neutrons can be used to investigate engineering materials and components, in particular residual stress measurements, and give examples of how this is used in industrial research and development.

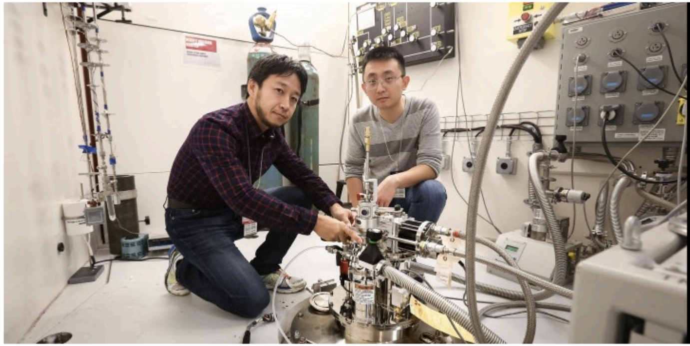
- Industry access to neutron sources
- Some examples of industrial use of neutrons
- Neutrons for engineering (materials)
- Residual stresses – origins and effects
- Residual stress measurements
- Examples of industry connected residual stress measurements using neutron scattering
- Brief outlook

Industry and large-scale facilities



Neutrons for industry

Examples of results from facility websites



Researchers Kensuke Takechi (left) and Ruidong Yang of TRI-NA test ionic liquid samples on VISION, SNS beam line 16B, to determine the molecular dynamics and interactions of a new electrolyte. This research may lead to the development of powerful new batteries with wide-reaching applications. (Image credit: ORNL/Genevieve Martin)

Toyota turns to neutrons to investigate new electrolyte for better batteries

March 29, 2017

Researchers from Toyota are using neutrons at the Spallation Neutron Source, located at the Department of Energy's Oak Ridge National Laboratory, to study the fundamental internal chemical reaction taking place in a newly developed electrolyte material they hope will one day lead to better batteries for a wide array of electronic applications.

"The electrolyte is the material used to transport ions in the battery, and we want to know the relationship between the ions' movement and solvation structure to develop better electrolytes," said Kensuke Takechi from the Toyota Research Institute of North America.

The electrolyte is an essential battery component that typically consists of a salt and multiple solvents. The precise combination of ingredients determines the power, lifetime, and safety properties of batteries.

VISION

Vibrational spectroscopy at VISION at SNS

OAK RIDGE
National Laboratory

Helping make hydrogen cars a reality

Toyota, who hope to release a hydrogen fuel cell vehicle in 2015, have been working with ISIS scientists to address a key challenge: hydrogen loss during cycling.

Science & Technology Facilities Council
ISIS

The race is on to produce the first commercially available hydrogen car. With the depletion of fossil fuels an alternative is needed, and hydrogen, provided it can be produced without using fossil fuels, is a promising option. But there are challenges in both producing and storing hydrogen that must be overcome before commercial hydrogen cars become reality.

The problem

One of the key requirements of materials for hydrogen storage is good cycling performance. Ti-V-Cr-Mo alloys have been proposed as a possible hydrogen storage system as they have the highest reversible storage capacity of materials of their type, but the capacity decreases with cycling. The exact location of hydrogen during cycling is key to understanding the loss in capacity. This is very difficult to achieve by X-ray diffraction as hydrogen is very light.

"Our work with ISIS has allowed us to develop *in operando* neutron powder diffraction techniques that has provided important insights into the nature and location of hydrogen in Ti-V-Cr-Mo alloy *for hydrogen storage* systems. This in turn provides new opportunities for the rational improvement of these materials for use as storage for future hydrogen cars."
Shin-ichi Towata, Toyota Central Research and Development Laboratories Inc

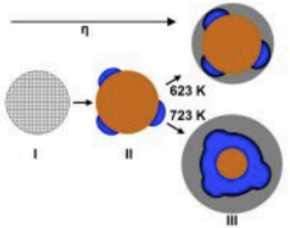
Diffraction at GEM

What do you do when the oil runs out? You make it!

It's all about the very large and the very small.

Neutrons have been used to understand Fischer-Tropsch chemistry, an industrial process for generating gasoline and diesel from a variety of carbon sources.

Syngas (or synthesis gas) is a mixture of carbon monoxide and hydrogen, which can be converted into clean gasoline and diesel by a process called Fischer-Tropsch catalysis. Importantly the carbon monoxide can come from any carbon source including biomass, coal and methane, so the process is very flexible. Sasol are pioneers and world leaders in this technology and their manufacturing plants in South Africa produce millions of litres of fuel a day, supplying about a third of the national demand. This massive scale of production relies on mastery of materials at the nanoscale. Scientists have been using ISIS to study Fischer-Tropsch catalysis at this molecular level, in order to better understand this vital process. The work was done in collaboration with Sasol Technology UK Ltd. and Glasgow University.



Schematic representation of restructuring of α-Fe2O3 precursor in the INS experiments on continuing exposure to a CO/ H2 mixture at elevated temperature. The reaction coordinate, η, increases from left to right of the figure. The colour scheme is as follows; chequered - α-Fe2O3, orange – Fe3O4, blue – iron carbide, black – hydrocarbonaceous overlayer, grey – amorphous carbon. Roman numerals indicate stages of the catalysts conditioning process [2].
View full-size image

Vibrational spectroscopy using inelastic neutron scattering at TOSCA

Breaking the Barriers to a solar Future

Researchers at the University of Sheffield, University of Durham and ISIS in collaboration with Start-up Company Ossila are using neutron reflectometry to look the formation of plastic solar cell films with the goal of developing devices which efficiently harness the power of the sun whilst being cheaper and easier to manufacture than the current silicon solar cells.

In 90 minutes enough sun hits the earth to provide the entire planet's energy needs for a year. Credit: Dreamstime
View full-size image

The Problem

A sustainable energy supply - it's a huge challenge but one we need to conquer in order to support the rapidly rising population, under the shadow of depleting fossil fuels. The sun, proposes a bright solution; within just 90 minutes enough sun hits the earth to provide the entire planet's energy needs for a year. But we need better technology to efficiently harness it.

Reflectometry at CRISP

Science & Technology Facilities Council
ISIS

Science & Technology Facilities Council ISIS

Neutrons could reveal how pesticides protect crops

Scientists have created a model of a leaf's waxy surface, similar to those found in wheat crops, in a project supported by the agrochemical company, Syngenta. They are now using the model at ISIS to study how surfactants, a key component in pesticide formulations, interact with the leaf surface to get into the plant and take effect. The results could lead to the fine-tuning of pesticide formulations to further increase crop yields in an attempt to meet the demand of feeding an ever growing global population.

This research is another step towards fine-tuning the chemicals used in agriculture to optimise crop yields, without damaging the plants. Credit: Emily Mobley
View full-size image

Waxy cuticles are essential for the well-being of all plants. The cuticle, made up of a thin coating of wax on plant leaves, acts as a protective shield against attack from pests, prevents the loss of nutrients and water from the plant, and is involved in transporting water and nutrients across the plant surface for plant growth.

To add an additional layer of protection against pests, farmers use pesticides on their crops in an attempt to maximize their yields. However, the structure of wax cuticles and the route pesticides take to cross the wax barrier and get inside are not fully understood.

Reflectometry at INTER

The Linde Group launches revolutionary carbon nanotube ink with help from ISIS

SANS at LOQ

The Linde Group, a world-leading gases and engineering company, has launched SEERe-Ink, a revolutionary ink based on carbon nanotubes for use in flat screen TVs, touchscreens and solar cells.

The team behind the research used the LOQ instrument at ISIS to gain vital information on the structure of the ink.

The Problem

Transparent conductive thin films used in high tech displays and touch screens rely primarily on the use of Indium Tin Oxide. However, this is expensive to process and brittle, making it unsuitable for touchscreens and flexible displays. Single walled carbon nanotubes (SWNTs) present an ideal alternative, but to be useful it must be possible to isolate individual, purified nanotubes with specific optoelectronic properties. Existing techniques using sonification or ultracentrifugation limit scalability and introduce damage into the SWNT structure.

Honeywell and NASA are studying residual stress using VULCAN

November 11, 2016

Robert Carter from NASA's Glenn Research Center (left) and Daira Legzdina from Honeywell Aerospace (right) examined high temperature nickel alloy samples containing linear friction welds using VULCAN, Oak Ridge National Laboratory's Spallation Neutron Source beam line 7.

Nickel alloys are often used in critical rotating applications, like turbines, and welded components are typically stress relieved through standard heat treatment processes, often without understanding the actual final stress state. Potential remaining residual stress can lower the life of the component dramatically, in some cases leading to premature field failure. Measuring the stress state of welded components using neutron diffraction could help designers manufacture more reliable aircraft components and other aerospace-related materials.

Through this research, Honeywell and NASA are working to understand the fundamental parameters that drive performance improvement in advanced nickel alloy bonding.

Strain mapping at VULCAN at SNS

Unlocking Potential of 3D Printed Rocket Parts with Neutrons

September 13, 2016

The process of 3D printing, or additive manufacturing, holds promise for advancements in almost every industry, including even rocket science. Engineers from NASA's Marshall Space Flight Center in Huntsville, Alabama, used neutrons recently to help understand the potential benefit of additive manufactured rocket engine components.

The team used the Neutron Residual Stress Mapping Facility at Oak Ridge National Laboratory's (ORNL) High Flux Isotope Reactor (HFIR), beam line HB-2B, to study residual stress in additive manufactured materials with hopes of qualifying them for flight, which could significantly reduce cost and schedule of flight hardware component manufacture.

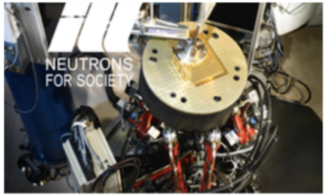
"Using the additive manufactured version of one engine component we are studying could reduce the amount of time needed to build a space-flight ready engine by years," said Stacey Bagg of NASA Marshall.

Such a significant time reduction means major cost savings for the project. But before a component can be used for space flight, the team needs to be sure it's safe.

OAK RIDGE
National Laboratory

Strain mapping at
NRSM at HFIR

Neutrons point the way to optimised crash-tolerant automotives



- **Boron steel has crucial applications in the automotive industry; it is attractive due to the reduced automobile weight and increased passenger safety it provides**
- **Understanding the effect spot welding has on residual stress in boron steel is crucial for informing new welding methods that have a less damaging impact on the material and its lifetime, leading to the production of optimised automotive components**
- **The correlation between spot welding and residual stress in boron steel was experimentally determined for the first time with neutron diffraction experiments conducted at the Institut Laue-Langevin (ILL)**

Press-hardened boron steel is an ultra high-strength steel used across a variety of industries, with a particularly important application in the automotive industry. A large proportion of car manufacturers use boron steel for structural components and anti-intrusion systems in automobiles, as it provides high strength and weight-saving potential, allowing for stronger yet lighter cars, with increased passenger safety.

In the automotive industry, a major joining method is resistance spot welding, with several thousand welds being made on a single car. Spot welding exposes the boron steel sheet directly underneath to very high temperatures, causing the metal to exceed melting temperature and then rapidly solidify upon cooling. This results in a heat-affected zone, where surrounding material contracts and microstructures are altered.

It is important to understand the exact effects spot welding has on boron steel, as the heat-affected zones can exhibit reduced hardness, which can in turn shorten the material's lifetime. To investigate the correlation between hardness and residual stress, measurements must be taken on the same spot weld. Therefore, a non-destructive method of measuring residual stress must be used so that the weld can be sectioned afterwards for the hardness tests. Non-destructive methods include electron, X-ray and neutron diffraction; the latter was the chosen method as the neutron beam has a cubic gauge volume, which is the most suited for the given sample's geometry.

Strain mapping at SALSA

NEUTRONS
FOR SOCIETY

Building safer ships with Lloyd's Register

Lloyd's Register have been using ENGIN-X to investigate UP welded joints in the marine industry. Credit: Dreamstime
View full-size image

Ultrasonic peening (UP) is a technique for improving the fatigue performance of welded joints. Little research has been done on how UP-treated welds behave when they are subjected to real world conditions such as compressive overload or variable amplitude loading. Lloyd's Register provides quality assurance to the marine industry, and they have been using ENGIN-X to investigate UP welded joints in these conditions. Understanding the process and its benefits will allow improved control of fatigue cracking, lower maintenance costs, and extending the life of welded connections in marine and other industries.

The problem

Ultrasonic peening has been used since the 1970s to improve the fatigue strength of welded joints. Lloyd's Register has accepted the benefits of the technique in welded joints, with mild steels seeing a fatigue improvement factor of 2.5, and up to 3.5 in high tensile steels. The challenge now is to assess what happens when the welds are subject to compressive overload or variable amplitude loading.

Optimising machining strategies for Boeing

Boeing, with the Advanced Manufacturing Research Centre (AMRC), have been using ENGIN-X to investigate the evolution of residual stresses in aerospace structures. Credit: Dreamstime
View full-size image

Understanding the development and distribution of residual stresses caused by machining is key to improving machining processes. The Advanced Manufacturing Research Centre (AMRC), with Boeing, at the University of Sheffield have been using Engin-X at ISIS to study the evolution of residual stresses in AA7050 – an aluminium alloy commonly used in aerospace structures - as it is heated and then machined. This understanding will enable them to reduce non-conformance in the manufacturing process, and significantly reduce costs.

Testing new welding techniques for the nuclear industry with AREVA

AREVA has designed the new European Pressurised Reactor (EPR)
View full-size image

Introduction of new designs, novel fabrication methods or modifications to existing plant in the nuclear power generation industry are subject to intense scrutiny to ensure that safety is not compromised. Multi-national corporation AREVA has designed the new European Pressurised Reactor (EPR) to meet stringent demands for increased safety and reduced cost of electricity generation. A twin EPR power station at Hinkley Point in Somerset is planned and will be constructed using modern welding technology.

The problem

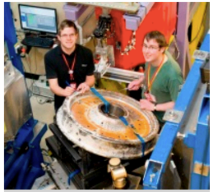
AREVA has developed new welding procedures for joining materials, but before deploying them, the very high levels of qualification and validation required by the nuclear industry and regulators must be met. Together with the Open University (OU), AREVA has been using Engin-X to map residual stress in mock-ups of welded nuclear components for the purpose of validating models simulating their new processes for over five years.

ISIS helps UK magnesium producer with a cracking problem

Scientists from ISIS have worked in partnership with UK company Magnesium Elektron to solve manufacturing challenges.

Magnesium Elektron is a world leader in magnesium technology and alloy development. The company, who first produced magnesium in Manchester in 1936, specialises in the development, manufacture and supply of magnesium products to technology industries worldwide. Their alloys are extensively used for applications in the aerospace and automotive industries. "Magnesium is 30% lighter than aluminium, and therefore it could be used for components in the transport industry to help reduce emissions. For magnesium to be a financially viable alternative to aluminium, we need to be able to mass produce it. However, we were unable to mass produce it as large slabs because they cracked during casting," explained Dr Mark Turski, a senior metallurgist at Magnesium Elektron.

Letting the train take the strain



Robert Jones and David Crosbee from Huddersfield University with a train wheel on Engin-X.
View full-size image

A consortium of train manufacturers, operators and standards bodies have used Engin-X to understand cracking in train wheels.

Every five years or so, every wheel on every train in the UK has to be replaced. Maintenance and renewal of train wheels make up a significant proportion of the cost of our rolling stock. In everyday use the wheels are subjected to stresses and strains that can cause cracks to develop, particularly in the wheel rims. A group from the University of Huddersfield has been using Engin-X at ISIS to study new and used train wheels to understand how cracks begin and spread. Their work is funded by a consortium including the Rail Safety and Standards Board, the Association of Train Operating Companies, Siemens, Lucchini, and EPSRC.

The manufacturing process of train wheels is designed to try and minimise the likelihood of cracks appearing. A process of heating and cooling the wheel hardens the rim and puts it under compressive strain, making it difficult for cracks to start.

Strain mapping at ENGIN-X

Science & Technology Facilities Council
ISIS

Rolls-Royce has applied for a patent for a new material for use in higher-efficiency jet engines, after collaborating with University of Cambridge scientists to study new alloys using neutron beams.

Source: Canadian Neutron Beam Centre (CNBC)
Contact: cnbc@cnl.ca
Image: Takeoff of a test flight for Rolls-Royce's latest and most energy-efficient jet engine, the Trent XWB. (Image: Airbus)

Developing more energy-efficient transportation is a global priority—and one that was highlighted in Mission Innovation, a commitment made by more than 20 countries to double spending on clean energy research in order to meet the targets set out in the 2015 Paris Agreement on climate change.

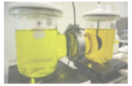
One area that has a lot of potential for energy savings is air travel. "Making jet engines one percent more efficient would translate into avoiding 500 to 1000 tonnes of carbon dioxide for each plane every year," says Katerina Christofidou, a post-doctoral researcher at the Rolls-Royce University Technology Centre at the University of Cambridge in the United Kingdom. "That's like taking 100 to 200 cars off the roads for each plane with a better engine."

MEEIR Technologie Inc. is seeking to commercialise energy-saving technology following research that accessed the CNBC.

Source: Canadian Neutron Beam Centre (CNBC)
Contact: cnbc@cnl.ca

Canada's sodium chlorate industry is the largest in the world, with revenues of over $350 million per year. Sodium chlorate is exported to the United States and is used by Canada's $8.8-billion-per-year paper manufacturing industry to bleach its paper products. These industries are challenged by the strong Canadian dollar, declining demand for newsprint, and by the rising prices of energy.

The cost of electricity represents more than 45% of the production cost of sodium chlorate, which is made by an electrochemical process. One way to combat rising electricity prices is to conserve energy by designing more efficient production methods. Specifically, Hydro-Québec's research institute, IREQ, has conducted research over the past decade in developing new cathodes that could save substantial amounts of power for the industry, estimated at $6 million annually in Quebec alone.



CINS
CANADIAN INSTITUTE FOR NEUTRON SCATTERING

Neutrons help General Motors to accelerate engine development.

> *The CNBC's measurements clearly falsified a hypothesis for GM concerning which of two manufacturing methods would be more effective in reducing residual stress in cylinder heads.*

General Motors (GM) uses neutron beams to accelerate the development of engine heads and blocks. These projects span three primary research areas: (1) evaluating the effectiveness of heat treatment and quenching methods; (2) directly observing phase precipitation during solidification; and (3) creep testing to make better predictions of reliability over the long term.

In the first area of evaluating the effectiveness of heat treatment and quenching methods, the CNBC's measurements clearly falsified a hypothesis for GM—specifically, that the air quenching of cylinder heads would provide a benefit over water quenching because of an overall reduction in residual stresses. The results showed that, with air quenching, significant stresses remained deep inside the cylinder heads, at a depth of about one centimetre.

A GM cylinder head

Operators of nuclear power plants use stress data from the CNBC to ensure safe, reliable and economic operations.

Source: Canadian Neutron Beam Centre (CNBC)
Contact: cnbc@cnl.ca

One of the ways that electricity generators ensure safety is by making conservative decisions to resolve even the slightest safety concern. Rare and unexpected issues—even minor ones—in nuclear power reactors around the world can sometimes result in temporary outages to investigate and respond. When reactor operators choose to forego electricity production, which can represent tens or sometimes *hundreds* of millions of dollars, it shows how important safe operations are to them.

Neutrons help the Ford Motor Company to investigate riveting techniques for lightweight vehicles.

The CNBC has an ongoing research project with the Ford Motor Company to examine new ways of joining dissimilar materials for use in lightweight vehicles. For example, self-piercing riveting (SPR) is a leading alternative to traditional welding methods and has been widely used by Audi, Mercedes, BMW and Jaguar on their aluminum cars and SUVs. SPR joints have excellent mechanical properties and high fatigue resistance. Prior to the CNBC's involvement, however, the 3D residual stress field in a mixed metal SPR joint had never been experimentally studied, making predictions for the fatigue life of such SPR joints difficult. Thus, Ford turned to the CNBC's neutron beam capability because other ways of determining stresses were too difficult due to the complex geometry and number of different materials involved. Ford plans to use the results from the neutron analysis to validate its existing residual stress prediction method and to document these findings to inform broader manufacturing processes.

Cross-sectional image of a self-piercing rivet joining sheets of steel and alumninum

Hydro-Québec is using stress data from the Canadian Neutron Beam Centre (CNBC) to improve electricity generation from hydroelectric dams.

Source: Canadian Neutron Beam Centre
Contact: cnbc@cnl.ca

The water flow in hydroelectric dams makes turbine runners turn, and this motion is used to produce electricity. These critical parts are expensive, costing up to $10 million each, and if one fails, the lost electricity production can be very costly.

To ensure reliability, Hydro-Québec asks turbine manufacturers to show that their turbines will last at least 70 years. But how can that be done without having to wait 70 years?

Neutrons peer into a running engine

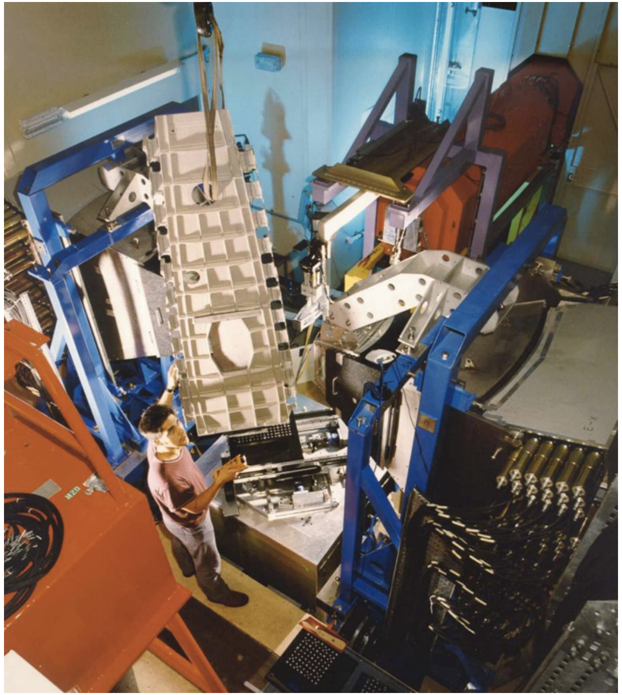
July 26, 2017

In a first-of-a-kind experiment, researchers used neutrons to investigate the performance of a new aluminum alloy in a gasoline-powered engine—while the engine was running.

A team from the Department of Energy's Oak Ridge National Laboratory worked with industry partners to perform the test, which looked at whether a high-performance alloy that is promising for automotive applications held up under the heat and stress of an internal combustion engine.

The feat was a first for the Spallation Neutron Source, said Ke An, lead instrument scientist for the facility's VULCAN instrument. "This was the first time an internal combustion engine has been run on our diffractometer, and, as far as we know, on any other," he stated.

Airbus wing prototype investigated for residual stresses at ENGIN-X, ISIS.



Neutrons for engineering (materials)

What can we do?

Neutron scattering for *engineering materials*

Diffraction

- Texture
  - Note, often easier ways to measure texture (lab X-rays, EBSD, …).
- Phase distribution/transformations
  - Lab X-rays or microscopy is usually sufficient, and synchrotron radiation has much better time resolution for kinetic/in-situ studies
- Strain/stress
  - **Most common application**. Need to know stresses/strains inside large components without sectioning. Benefits from large sampling volumes and cubic gauge volumes. Complicated geometries possible to handle. Possible to do in-situ measurements.

Small-angle scattering

- Precipitation/decomposition
  - Large sampling volumes, contrast complementary to SAXS, plus magnetic scattering. Low flux limits in-situ measurements to systems with slower kinetics. Small cluster sizes.

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journal homepage: www.elsevier.com/locate/actamat

ELSEVIER

Full length article

Effect of neutron flux on the characteristics of irradiation-induced nanofeatures and hardening in pressure vessel steels

CrossMark

A. Wagner [a], F. Bergner [a,*], R. Chaouadi [b], H. Hein [c], M. Hernández-Mayoral [d], M. Serrano [d], A. Ulbricht [a], E. Altstadt [a]

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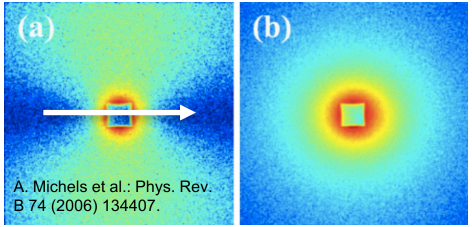
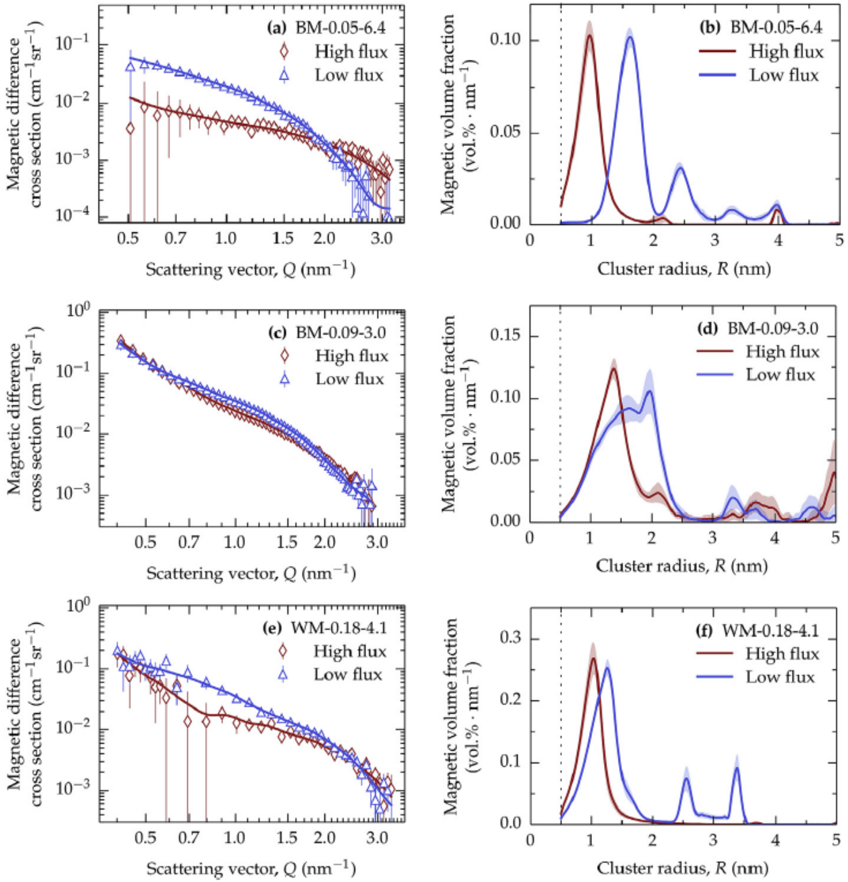
$$I(Q) = N_p V_p^2 \Delta\rho^2 P(Q) S(Q)$$



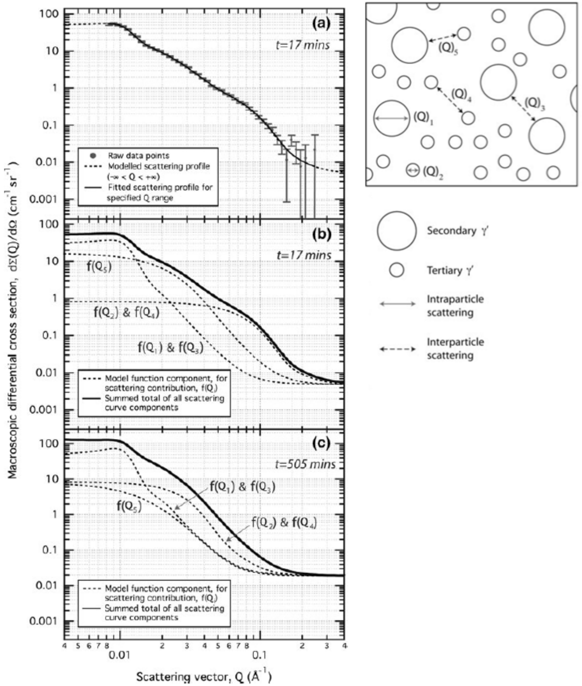
$$\frac{d\sigma}{d\Omega} = \frac{d\sigma_n}{d\Omega} + \sin^2\alpha \frac{d\sigma_m}{d\Omega}$$



METALLURGICAL AND MATERIALS TRANSACTIONS A VOLUME 42A, JANUARY 2011—49

Characterization of Gamma Prime (γ') Precipitates in a Polycrystalline Nickel-Base Superalloy Using Small-Angle Neutron Scattering

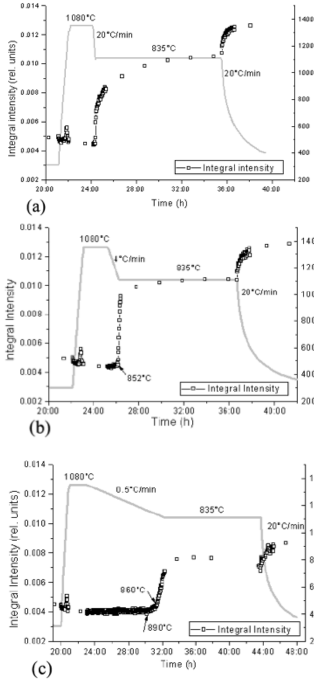
D.M. COLLINS, R.K. HEENAN, and H.J. STONE



J. Phys.: Condens. Matter 20 (2008) 104220 (9pp) doi:10.1088/0953-8984/20/10/104220

Microstructural characterisation of a Ni–Fe-based superalloy by *in situ* small-angle neutron scattering measurements

D Mukherji[1], D Del Genovese[1], P Strunz[2,3], R Gilles[4], A Wiedenmann[5] and J Rösler[1]



Journal of Alloys and Compounds 589 (2014) 462–471
Contents lists available at ScienceDirect
Journal of Alloys and Compounds
journal homepage: www.elsevier.com/locate/jalcom

Precipitate microstructure evolution in exposed IN738LC superalloy

Pavel Strunz [a,*], Martin Petrenec [b,1], Urs Gasser [c], Jiří Tobiáš [b], Jaroslav Polák [b], Jan Šaroun [a]
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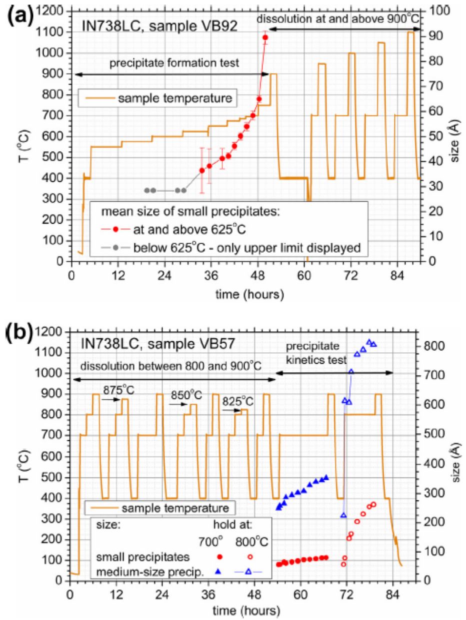
[b] Institute of Physics of Materials of the AS CR, Brno, Czech Republic
[c] Laboratory for Neutron Scattering, PSI, CH-5232 Villigen, Switzerland



APPLIED PHYSICS LETTERS **106**, 061911 (2015)

Early stages of spinodal decomposition in Fe–Cr resolved by *in-situ* small-angle neutron scattering

M. Hörnqvist,[1,a)] M. Thuvander,[1] A. Steuwer,[2,3] S. King,[4] J. Odqvist,[5] and P. Hedström[5]
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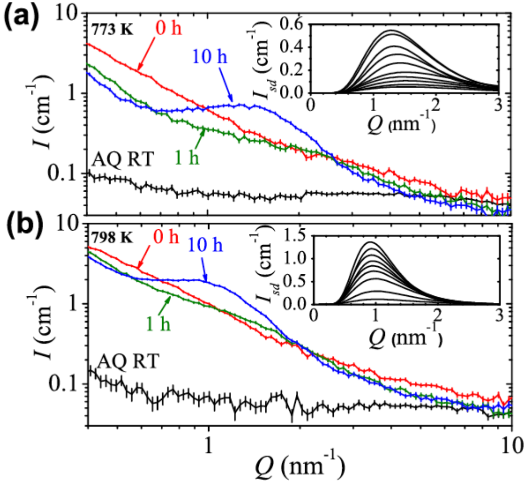
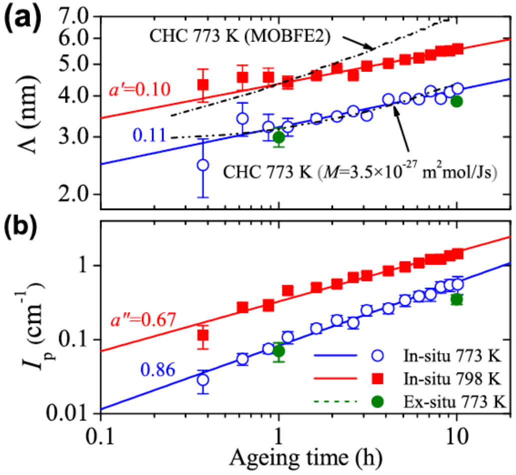
[5]*Materials Science and Engineering, KTH Royal Institute of Technology, Brinellvägen 23, S-100 44 Stockholm, Sweden*



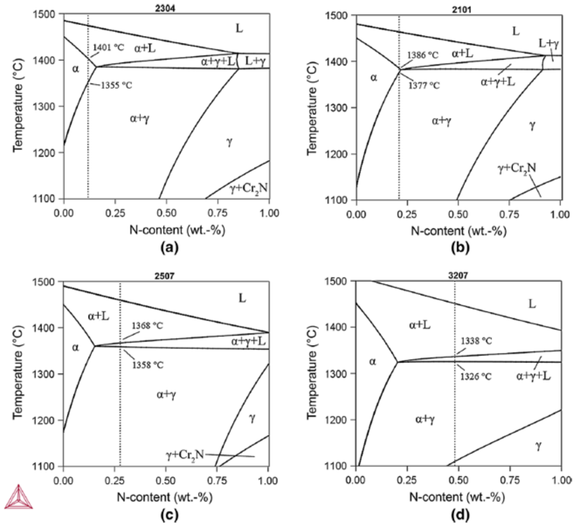


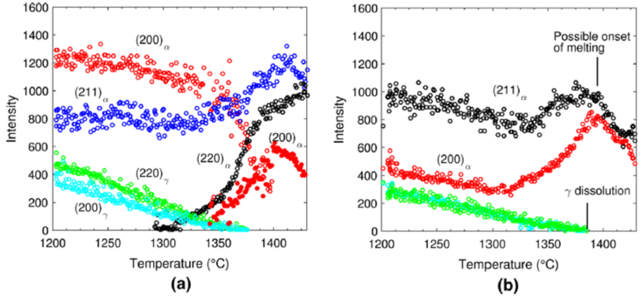
$$S(Q,t)=\frac{RT}{f''+2\kappa Q^2}+\left[S(Q,t=0)-\frac{RT}{f''+2\kappa Q^2}\right]\exp\left\{-2M\left(f''+2\kappa Q^2\right)Q^2t\right\}$$

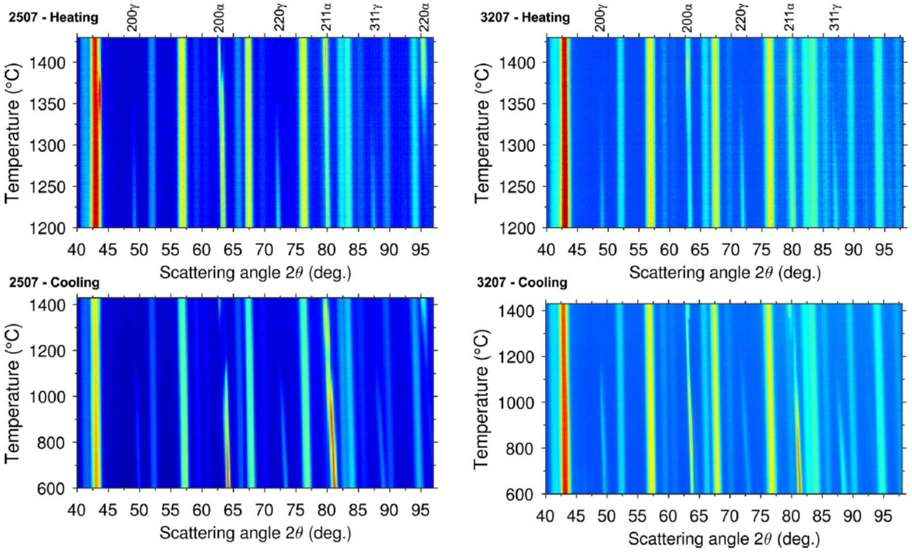
METALLURGICAL AND MATERIALS TRANSACTIONS A 1562—VOLUME 48A, APRIL 2017

High-Temperature Phase Equilibria of Duplex Stainless Steels Assessed with a Novel *In-Situ* Neutron Scattering Approach

NIKLAS PETTERSSON, STEN WESSMAN, STAFFAN HERTZMAN, and ANDREW STUDER







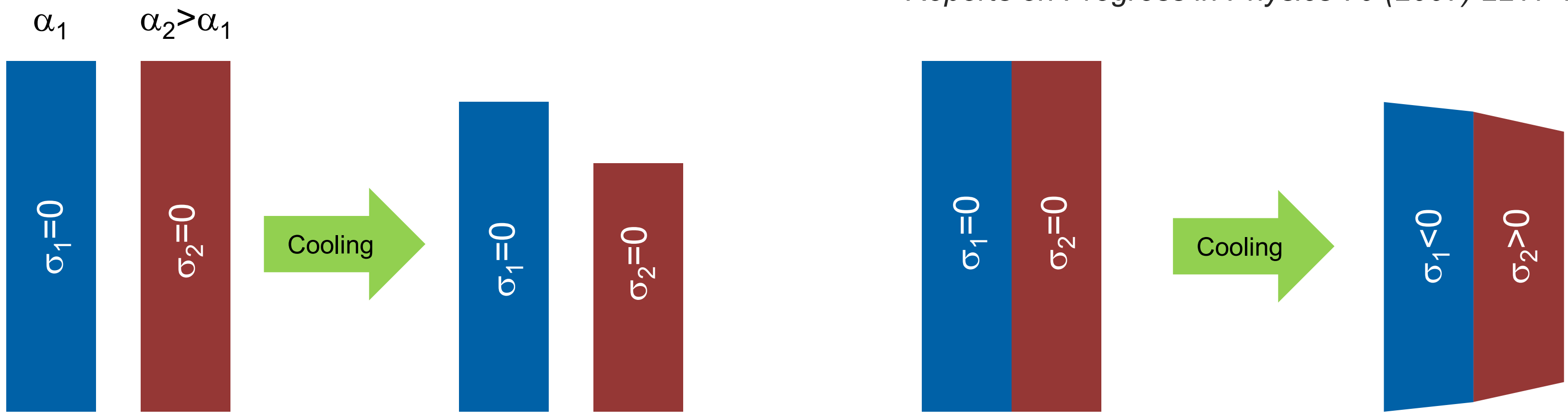
Residual stress

What are they, where do they come from, and why do we care so much?

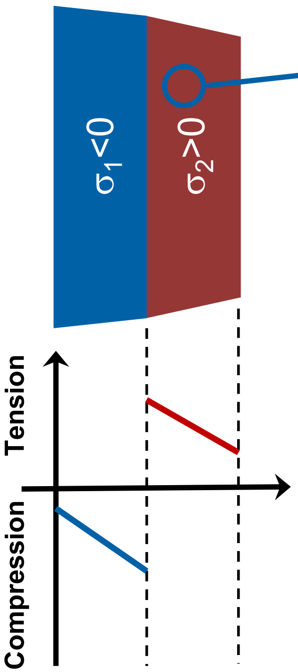
Residual stresses

"Residual stresses are those stresses which are retained within a body when no external forces are acting. Residual stresses arise because of misfits (incompatibilities) between different regions of the material, component or assembly"

P. J. Withers: Residual stress and its role in failure Reports on Progress in Physics 70 (2007) 2211–2264.

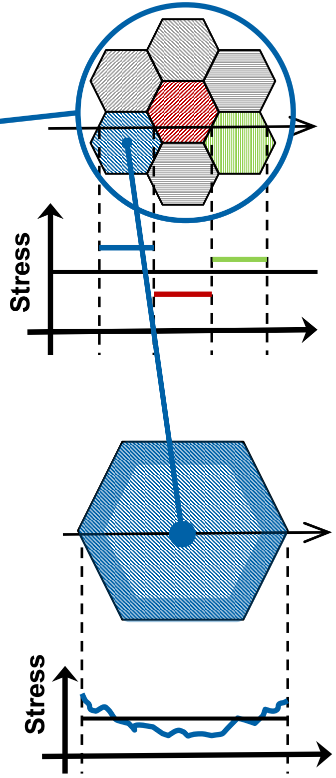


Types of residual stresses



Macroscopic (Type I)

- Balance over macroscopic scales (comparable to the component scale).
- Neglecting microstructure.
- Well described by continuum mechanics.
- Typical origin: processing, heat treatment, welding, deformation, shot peening, …



Microscopic (Type II, intragranular)

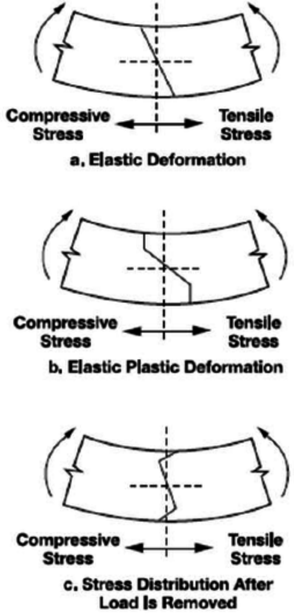
- Local deviations from the average Type I stress on scale of the microstructure (grain scale)
- In single phase materials it depends on e.g. anisotropic elastic or plastic response
- In multiphase materials depends on properties of individual phases as well.

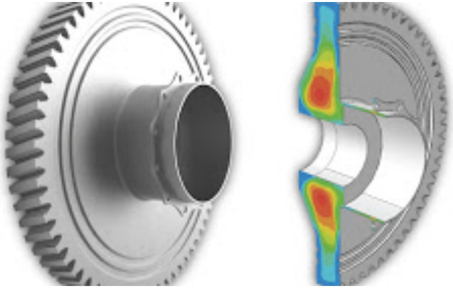
Microscopic (Type III)

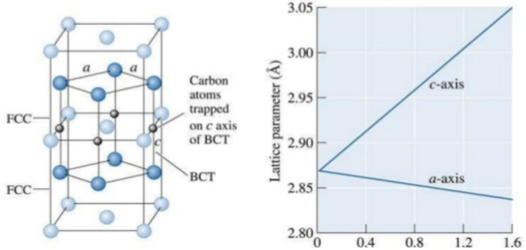
- Local deviations from the Type II stresses
- Usually caused by gradients in dislocation density or point defects

Origin of residual stresses

- **Plastic deformation**
  - Plastic working
  - Metal cutting
  - Shot peening
- **Thermal origins**
  - Rapid cooling
  - Thermal coefficient mismatch
- **Phase transformations**
  - Solidification
  - Surface treatment
  - Martensite formation
  - Oxidation

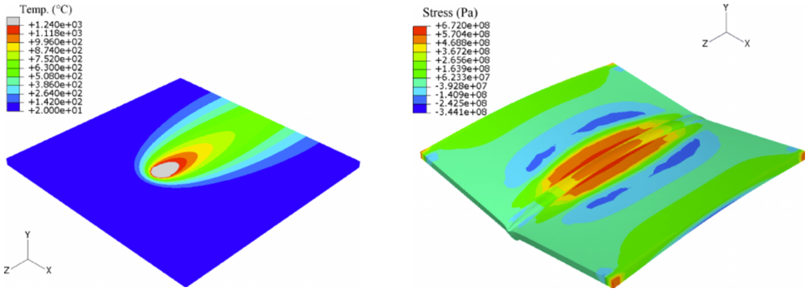






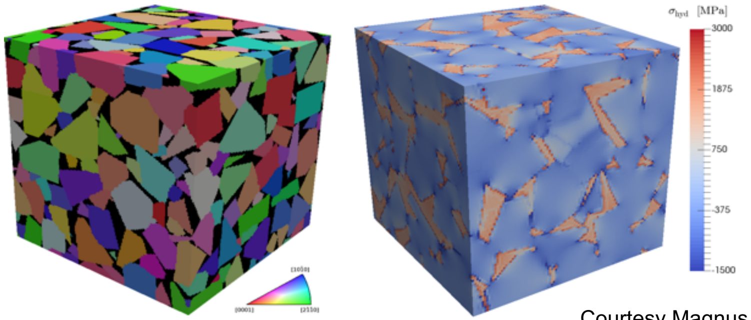
Origin of residual stresses

- **Welding or localized heat treatments**
  - Thermal gradients
  - Phase transformations



T. Hyde et al.: J. Multiscale Modell.. 1 (2011).

- **Composites/multiphase materials or systems**
  - Reinforcement/matrix compatibility
  - Surface coatings



Courtesy Magnus Ekh, Chalmers

Effect of residual stresses

- **Detrimental effects**
  - Large effect on fatigue life! Tensile residual stresses increases the risk for crack initiation, accelerated crack propagation rates and fracture. This need to be accounted for!
  - A particularly detrimental case is the interaction between mechanical loads and environment, e.g. stress corrosion cracking.
  - Additionally, the relaxation of residual stresses from previous process steps during e.g. machining could lead to geometrical changes outside allowed tolerances.

- **Beneficial effects**
  - Compressive residual stresses at surfaces can significantly reduce the risk for e.g. crack initiation. This is utilized by applying e.g. shot peening to fatigue sensitive surfaces. Only works as long as the stresses can be retained during service!

- Typically intentionally **or unintentionally** relieved by thermal treatments (and mechanical loads)
- Important to know and control the residual stresses!

Residual stress measurements

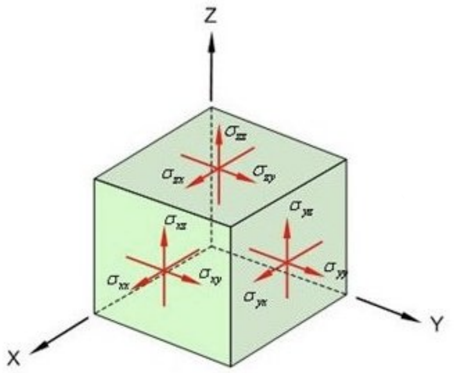
How can we find the residual stresses in engineering materials and components, and how do neutrons come into the picture?

Measuring residual stresses

Or rather measuring residual **strains**

For a general stress state **6 independent strain measurements** must be performed to obtain all stress components

$$\sigma_{ij} = \begin{pmatrix} \sigma_{xx} & \sigma_{xy} & \sigma_{xz} \\ \sigma_{yx} & \sigma_{yy} & \sigma_{yz} \\ \sigma_{zx} & \sigma_{zy} & \sigma_{zz} \end{pmatrix} \quad \epsilon_{ij} = \begin{pmatrix} \epsilon_{xx} & \epsilon_{xy} & \epsilon_{xz} \\ \epsilon_{yx} & \epsilon_{yy} & \epsilon_{yz} \\ \epsilon_{zx} & \epsilon_{zy} & \epsilon_{zz} \end{pmatrix}$$



If the principal stress directions can be inferred from geometry or modelling, the number of necessary measurements reduces to **three.** Further reductions for e.g. plane stress/strain or uniaxial states.

$$\sigma_1 = \frac{E}{(1+\nu)(1-2\nu)} \{(1-\nu)\epsilon_1 + \nu(\epsilon_2 + \epsilon_3)\}$$

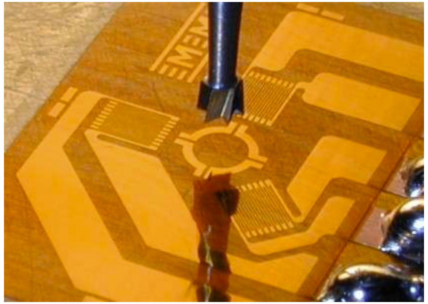
$$\sigma_2 = \frac{E}{(1+\nu)(1-2\nu)} \{(1-\nu)\epsilon_2 + \nu(\epsilon_1 + \epsilon_3)\}$$

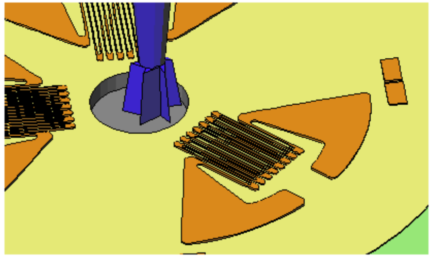
$$\sigma_3 = \frac{E}{(1+\nu)(1-2\nu)} \{(1-\nu)\epsilon_3 + \nu(\epsilon_1 + \epsilon_2)\}$$

Methods

Mechanical methods

- Curvature
- Sectioning
- **Hole drilling**
- Contour method

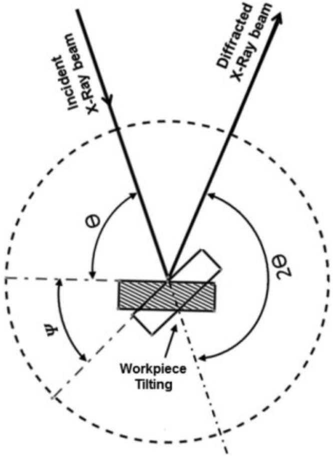




www.stresscraft.co.uk

Methods

http://ast.stresstechgroup.com/



F. Jafarian et al.: Measurement 63 (2015) 1.

Diffraction methods

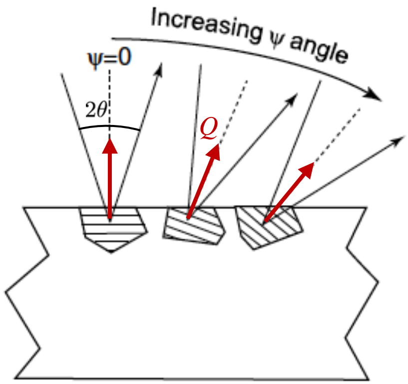
- **Lab X-rays**
- High-energy synchrotron X-rays
  - θ/2θ method
  - Angle dispersive diffraction
  - Energy dispersive diffraction
- Neutrons
  - Angle dispersive diffraction
  - Energy dispersive diffraction

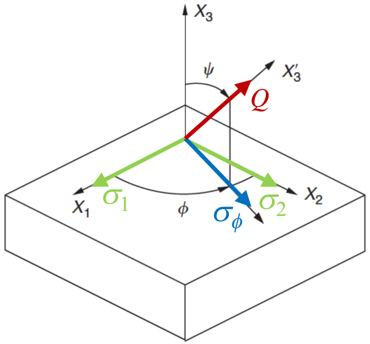
Diffraction methods – Lab X-rays

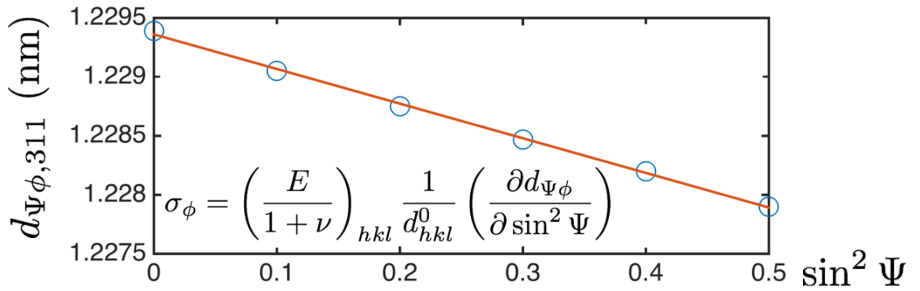
Applying elastic strain to a crystal will strech it, thus changing the interplanar spacing. By determining the changes in lattice spacing from the strain-free state, the lattice itself can be used as a strain gauge:

$$\epsilon_{hkl} = \frac{d_{hkl} - d^0_{hkl}}{d^0_{hkl}} = \{2d_{hkl}\sin\theta_{hkl} = \lambda\} = -\Delta\theta_{hkl}\cot\theta^0_{hkl}$$

$$\frac{d_{\Psi\phi} - d^0}{d^0} = \frac{1+\nu_{hkl}}{E_{hkl}}\sigma_\phi \sin^2\Psi - \frac{\nu_{hkl}}{E_{hkl}}(\sigma_1 + \sigma_2)$$







Limitations of lab X-rays

- Limited penetration depth – surface measurements only
- Bi-axial assumption – general case 3D and complicated
- Material removal and corrections necessary for depth profiling
- Geometrically constrained
- hkl specific elastic constants not always accurately known

Table 12.1 Neutron and X-ray scattering parameters for four elements which comprise the majority component of many common engineering alloys

Element	*Al*	*Ti*	*Fe*	*Ni*
Atomic number	13	22	26	28
Neutron scattering length (fm)	3.45	−3.44	9.45	10.3
Neutron attenuation length (mm)	100	20	8.3	5.6
1.5 Å X-ray attn. length (mm)	0.083	0.012	0.004	0.003
0.3 Å X-ray attn. length (mm)	6.71	1.06	0.38	0.27
0.15 Å X-ray attn. length (mm)	52.6	8.47	3.03	2.19

Note
The attenuation length L is the thickness of material required to attenuate a beam by the factor 1/e. It is the reciprocal of the linear attenuation coefficient μ, so the intensity I after attenuation of a beam of incident intensity I_0 through a thickness x is $I = I_0\,\mathrm{e}^{-x/L} = I_0\,\mathrm{e}^{-\mu x}$.

Introduction to the characterization of residual stress by neutron diffraction (2003), M.T. Hutchings et al. (Eds.)

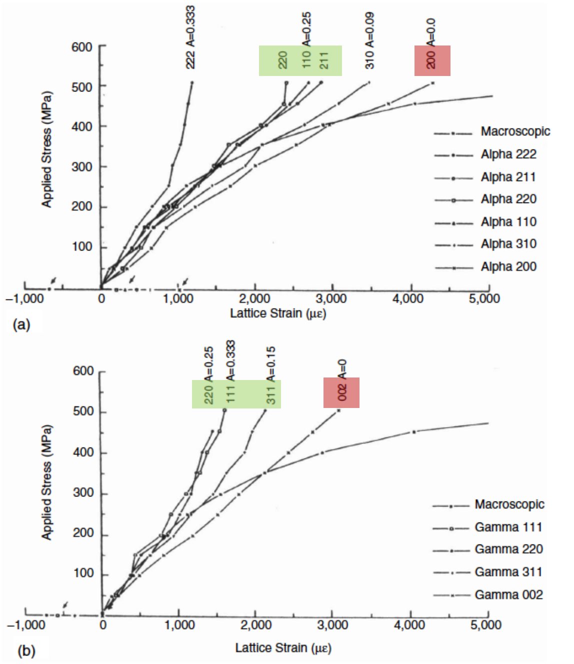
Limitations of lab X-rays

- Peak shift might not be linear with strain (only certain peaks suitable).
- Shifts are due to a combination of Type I and Type II stresses, not easily resolvable for single peaks (Type III mainly causes peak broadening)

Lattice Planes Weakly and Strongly Affected by Intergranular Strains

Material	Recommended Planes (Weakly Affected by Intergranular Strains)	Problematic Planes (Strongly Affected by Intergranular Strains)
fcc (Ni [43], Fe [44], Cu [45])	(111), (311), (422)	(200)
fcc (Al [46], [47])	(311), (422), (220)	(200)
bcc (Fe [47])	(110), (211)	(200)
hcp Ti [31])	Pyramidal (10$\bar{1}$2), (10$\bar{1}$3)	Basal (0002) and prism (10$\bar{1}$0), (1$\bar{2}$10)
hcp (Be [49])	Second-order pyramidal (20$\bar{2}$1), (11$\bar{2}$2)	Basal, prism, and first-order pyramidal (10$\bar{1}$2), (10$\bar{1}$3)

Analysis of residual stresses by diffraction using neutron and synchrotron radiation (2005). M.E. Fitzpatrick, A. Lodini (Eds.)



Introduction to the characterization of residual stress by neutron diffraction (2003), M.T. Hutchings et al. (Eds.)

Neutron and synchrotron diffraction

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	Element	*Al*	*Ti*	*Fe*	*Ni*
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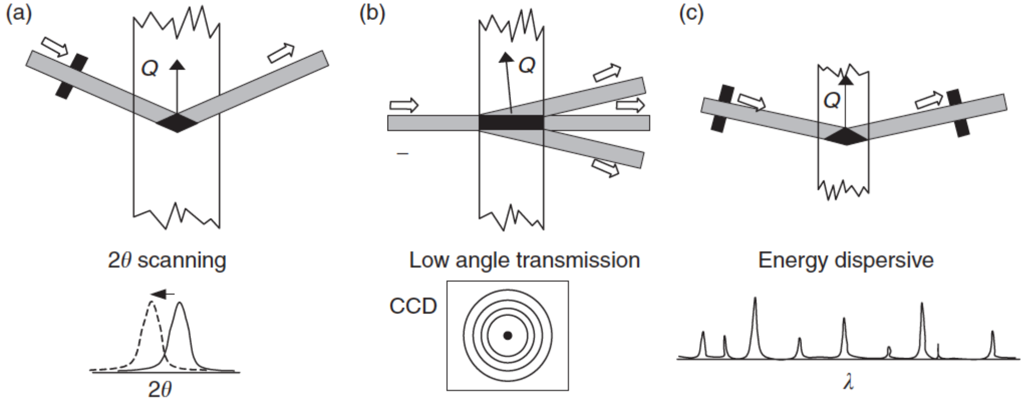
	Neutron scattering length (fm)	3.45	−3.44	9.45	10.3
	Neutron attenuation length (mm)	100	20	8.3	5.6
Lab X-rays 8.4 keV	1.5 Å X-ray attn. length (mm)	0.083	0.012	0.004	0.003
Synchrotron 41.3 keV	0.3 Å X-ray attn. length (mm)	6.71	1.06	0.38	0.27
Synchrotron 82.6 keV	0.15 Å X-ray attn. length (mm)	52.6	8.47	3.03	2.19

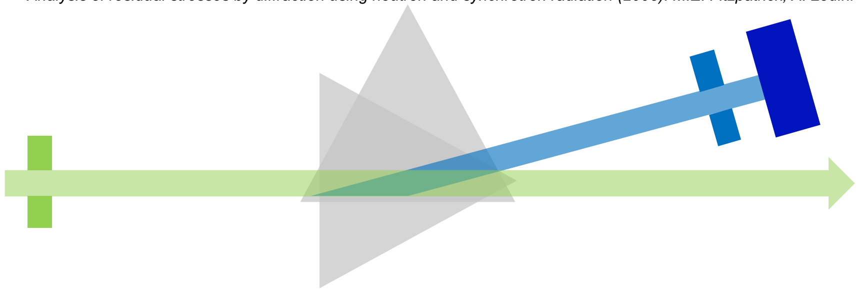
Note
The attenuation length L is the thickness of material required to attenuate a beam by the factor 1/e. It is the reciprocal of the linear attenuation coefficient μ, so the intensity I after attenuation of a beam of incident intensity I_0 through a thickness x is $I = I_0\,e^{-x/L} = I_0\,e^{-\mu x}$.

Introduction to the characterization of residual stress by neutron diffraction (2003), M.T. Hutchings et al. (Eds.)

Synchrotron diffraction



Analysis of residual stresses by diffraction using neutron and synchrotron radiation (2005). M.E. Fitzpatrick, A. Lodini (Eds.)



- High energies means large penetration (for HE white beam several cm depending on material), measurements typically done in transmission
- Small diffraction angles
- High flux gives short measurement times

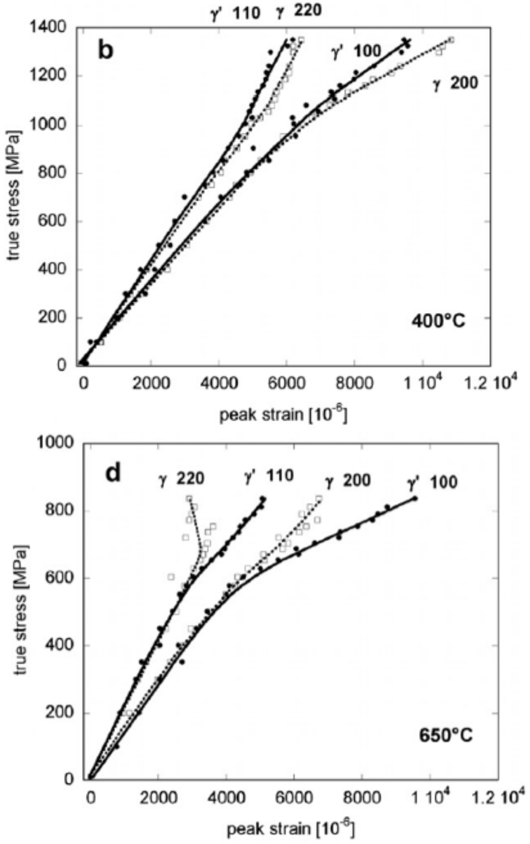
- High lateral spatial resolution, but through-thickness average (can be solved with conical slits etc) or very elongated gauge volumes, e.g. ~2.3 mm for 0.1 mm slits and 5° 2θ
- Difficult to maintain same diffracting volume when measuring strains in different directions

- For (b) and (c) it's possible to use **lattice parameter** instead of interplanar spacing through refinement of entire profile, which gives better average strain estimates and use of **macroscopic elastic constants**

In-situ mechanical testing

- The use of synchrotron or neutron scattering for determination of internal strains allows acquisition of data in-situ during application of an external load.
- Advanced sample environments facilitate such testing in combination with controlled temperatures (in the range from cryogenic to ~1500 °C) and aggressive environments
- During in-situ testing, it is possible to follow the development of (average) internal strain distribution between e.g.
    - different phases in a material
    - grains of the same phase with different orientations

  as a function of load, time, temperature etc.
- For some techniques texture evolution can be followed
- Specific synchrotron based techniques even allow extraction of data from single grains (and their environment) in polycrystalline materials

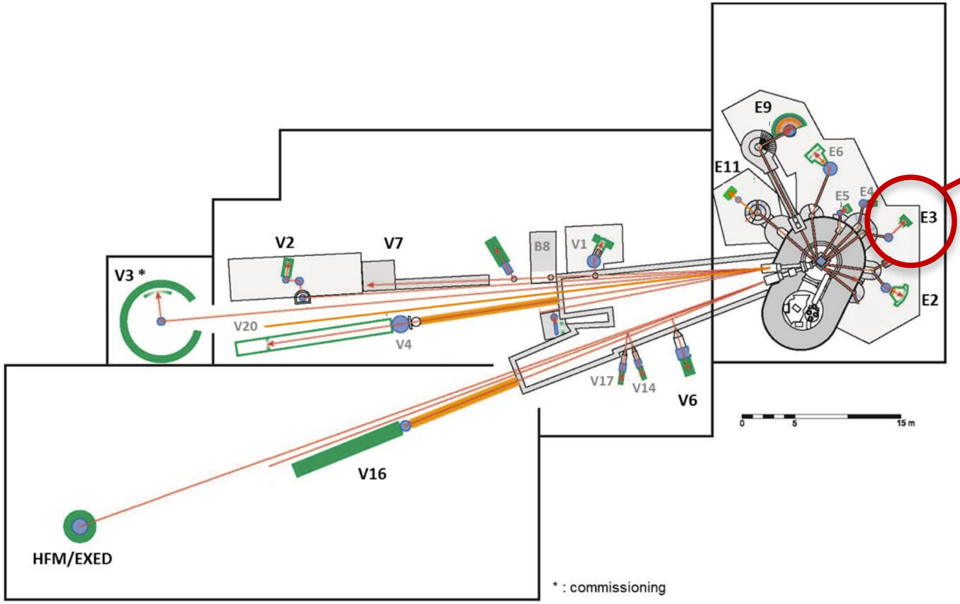


M. Daymond et al. Acta Mater 55 (2007) 3089

Neutron diffraction for strain mapping

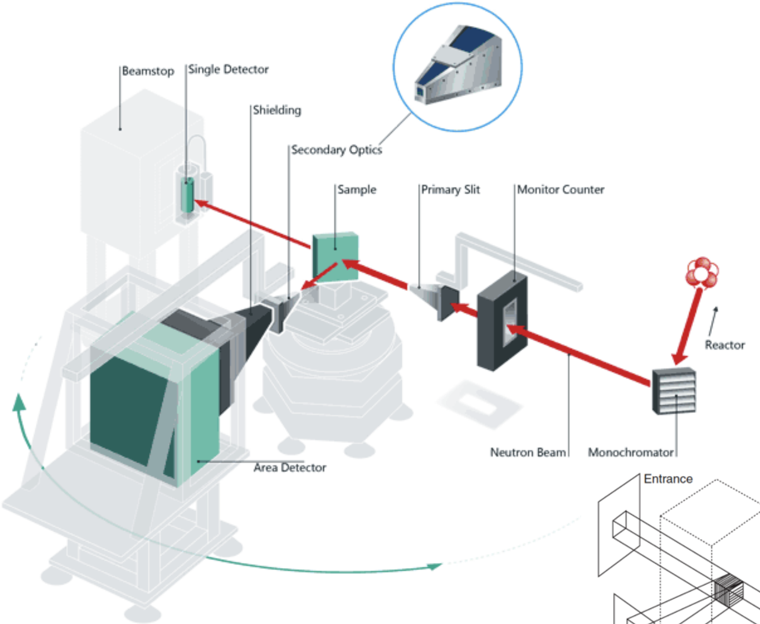
- Penetration depth of mm to several cm in engineering materials
- Angle dispersive (constant wavelength) techniques at continuous sources
- Energy dispersive (time-of-flight) techniques at pulsed sources
- Easy to obtain diffraction angles around 90°
  - (Nearly) cubic gauge volume
  - Measurements on complicated geometries
- Low flux compared to synchrotron radiation give long measurement times
- The ideal strain scanner includes possibility to adjust gauge volume, room for large specimens and flexible positioning and movement of specimen

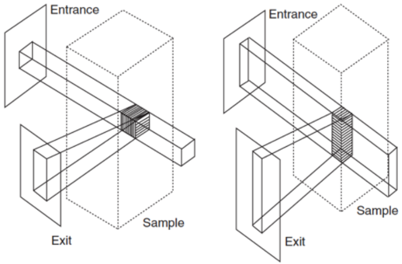
Constant wavelength sources (reactors)



E3 diffractometer BERII/HZB
https://www.helmholtz-berlin.de/

Angle dispersive (constant wavelength)





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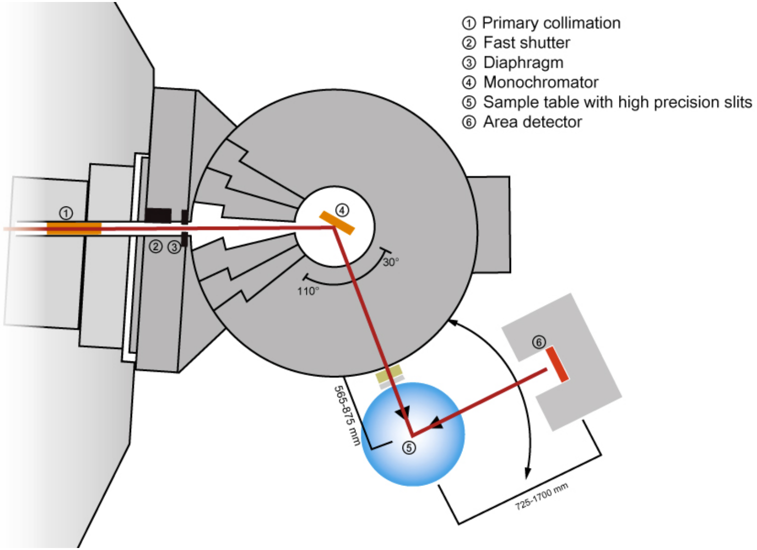
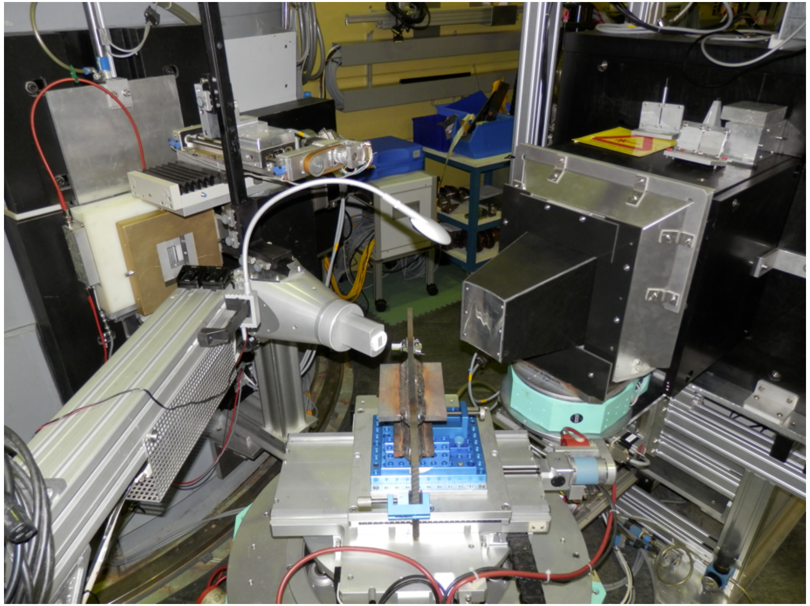
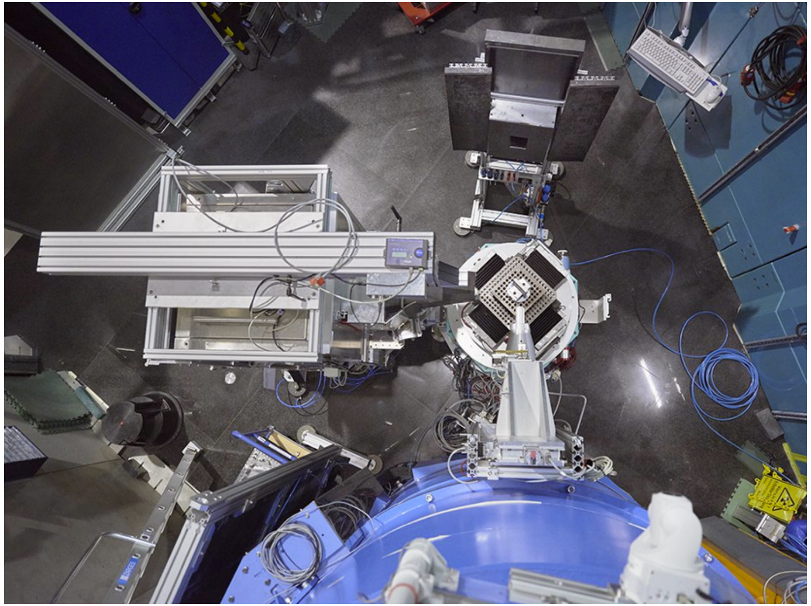


STRESS-SPEC FRMII
http://www.mlz-garching.de/stress-spec

Angle dispersive (constant wavelength)

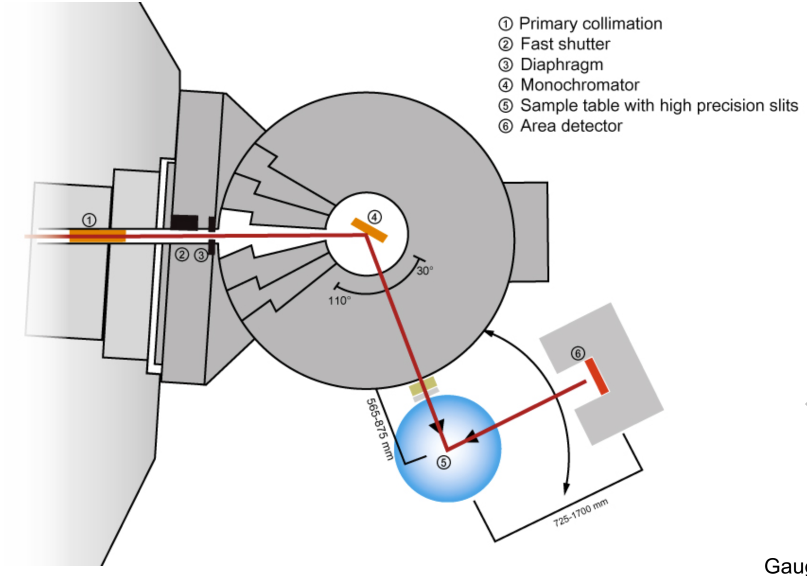


E3 diffractometer BERII/HZB
https://www.helmholtz-berlin.de/

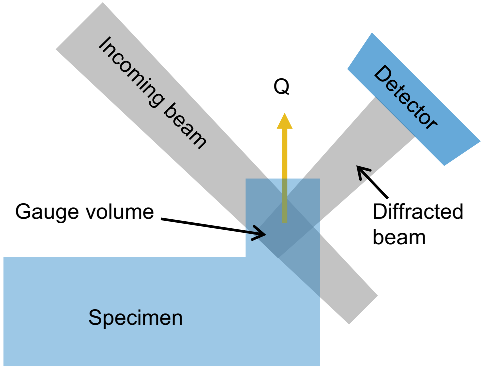


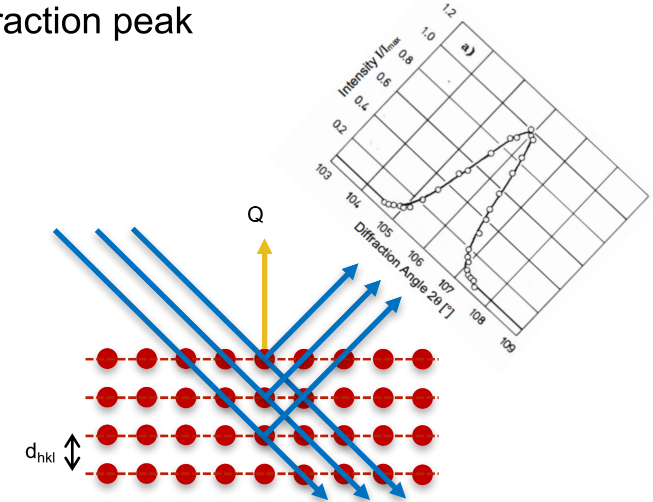
STRESS-SPEC FRMII
http://www.mlz-garching.de/stress-spec

Angle dispersive (constant wavelength)



- Suitable diffraction peak(s) for strain measurements chosen (not all are suitable!)
- Wavelength from monochromator selected to obtain scattering angles around 90° for selected diffraction peak





Strain scanning at constant wavelength sources (reactors)

Advantages

- Fast if single peaks are sufficient

Disadvantages

- May need several scans for multiphase materials
- Only one strain direction
- Does not measure intergranular strains
- Can be difficult for textured materials
- Multiple specimens for in-situ tests if several peaks are needed

Example of instruments

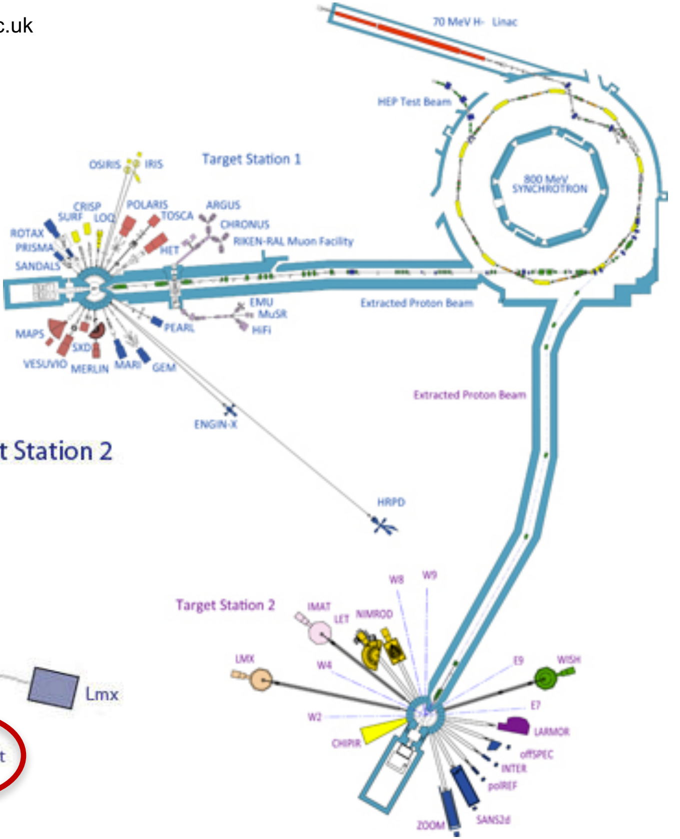
Europe
- SALSA at ILL (Grenoble)
- E3 at BERII (Berlin)
- STRESS-SPEC at FRMII (Munich)
- HK4 at LVR-15 (Prague)

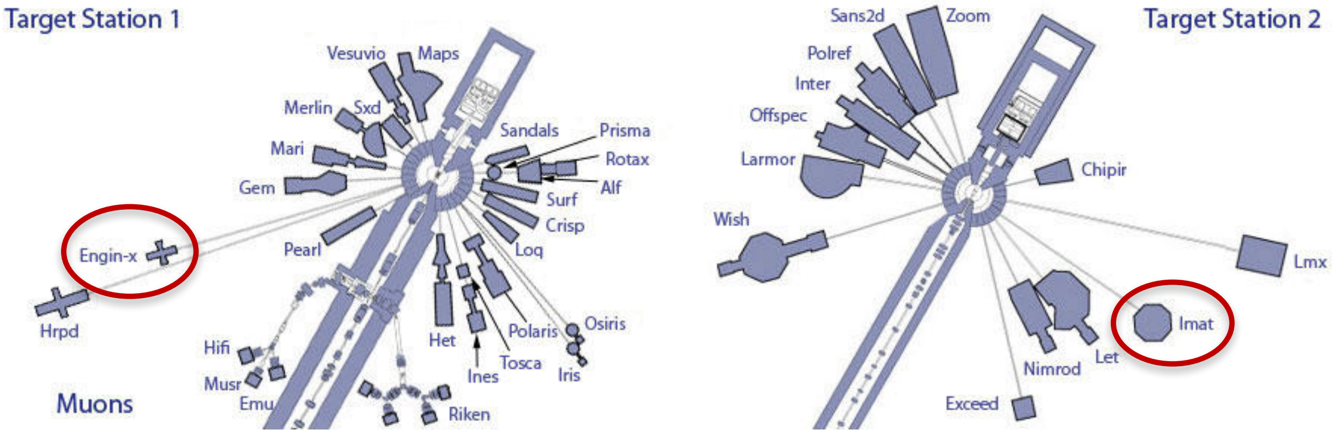
USA
- HB-2B NRSF2 at HFIR (Oak Ridge)
- BT-8 at NIST (Washington)

Australia
- KOWARI at OPAL (Sidney)

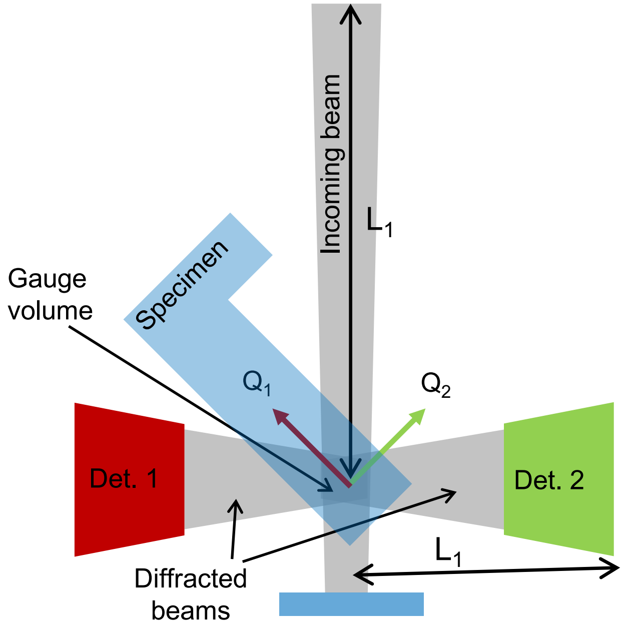
www.isis.stfc.ac.uk

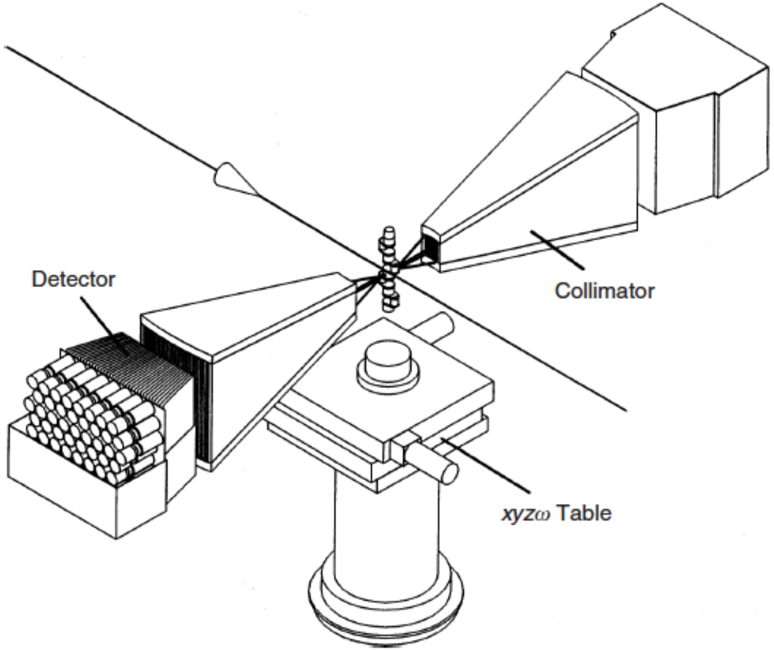
Time-of-flight (energy dispersive diffraction)



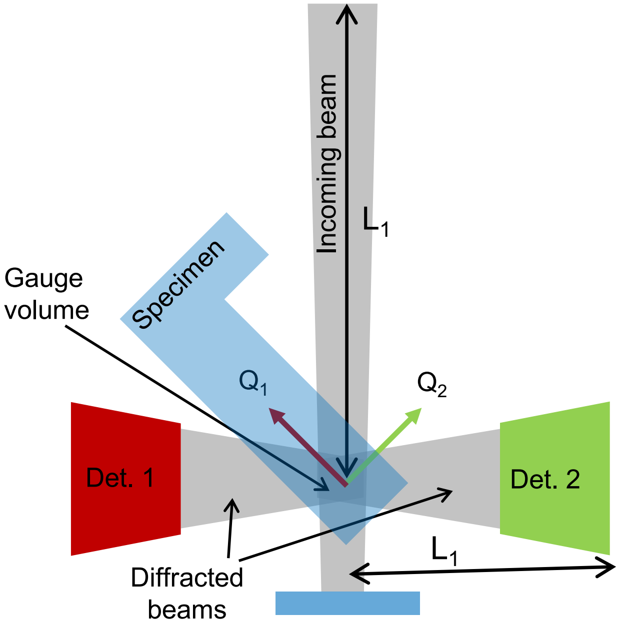


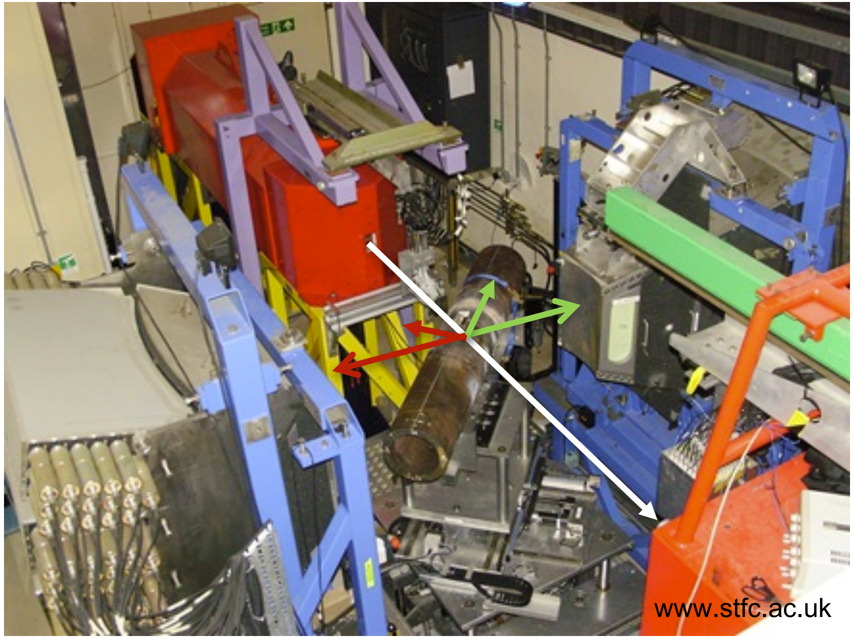
TOF engineering diffractometer



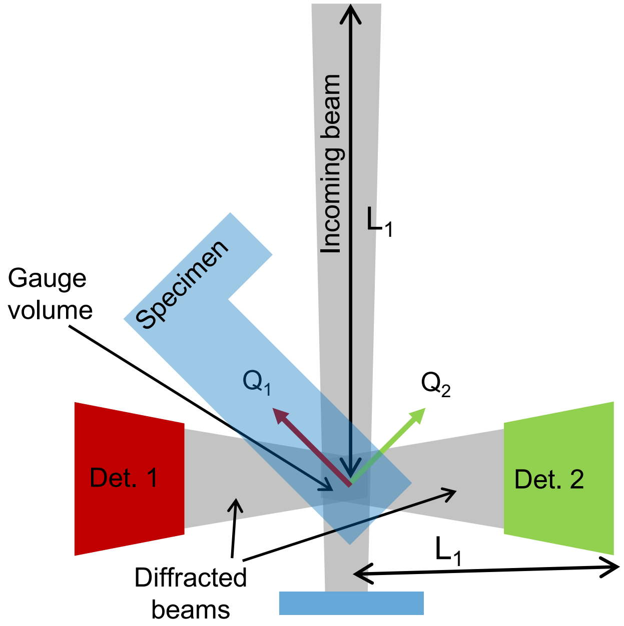


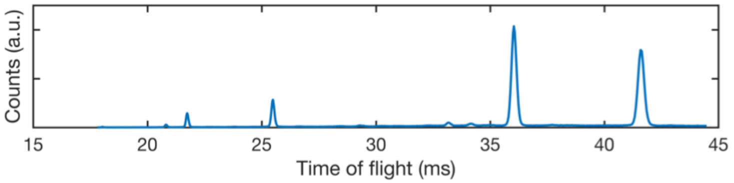
TOF engineering diffractometer





TOF engineering diffractometer

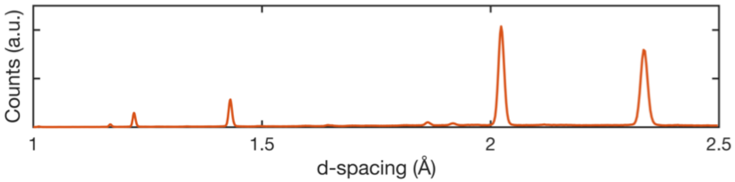




$$t = \frac{L}{v} = \left\{ v = \frac{p}{m} = \frac{h}{\lambda m} \right\} = \lambda \frac{mL}{h} = \lambda \frac{m(L_1 + L_2)}{h}$$

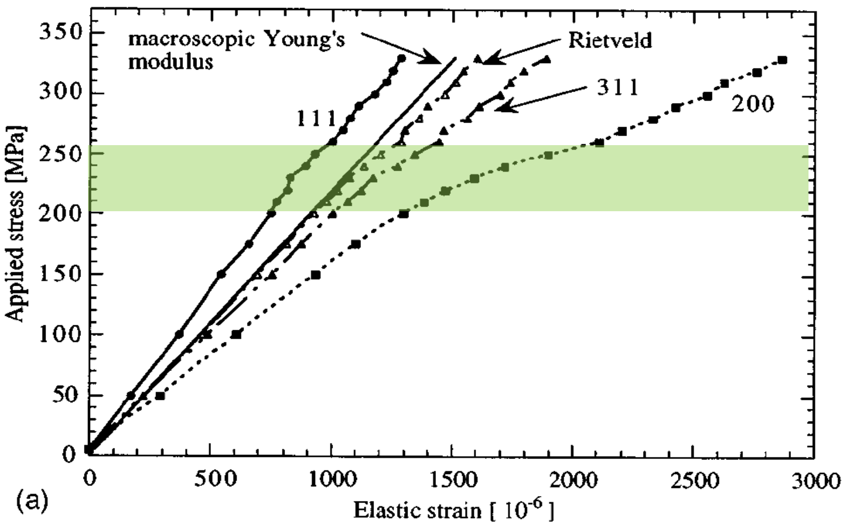
$$2d \sin\theta = \lambda$$

$$d = \frac{h}{2m \sin\theta (L_1 + L_2)} t$$



$$\epsilon_{hkl} = \frac{d_{hkl} - d^0_{hkl}}{d^0_{hkl}} = \frac{t_{hkl} - t^0_{hkl}}{t^0_{hkl}}$$

Intergranular stresses



M.R. Daymon et al.: J. Appl. Phys. 82 (1997) 1554.

- Complete diffraction pattern for each position (in both directions)
- Information about all present (measureable) phases
- Possible to use lattice parameter from refinements instead of interplanar spacing
- Resolve overlapping peaks
- Information about texture
- In-situ measurements on single specimens
- Possible to resolve intragranular (type II) stresses

Engineering diffractometers at pulsed neutron sources

Advantages

- Full diffraction patterns
- Faster for multiple peaks
- Good for textured materials
- Two orthogonal directions
- Intergranular strains
- Single specimen for in-situ tests

Disadvantages

- Lower time-averaged flux
- Limited availability (fewer sources and less ”up-time” compared to reactors)

Example of instruments

Europe

- POLDI at SINQ (Villagen)
- ENGIN-X at ISIS (Didcot)
- *BEER at ESS (Lund)*

USA

- VULCAN at SNS (Oak Ridge)
- *SMARTS at LANSCE (Los Alamos)*

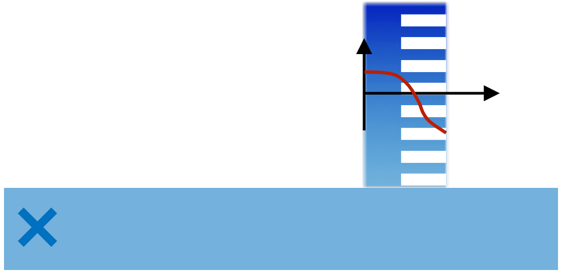
Asia

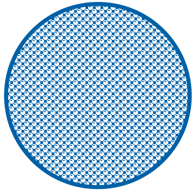
- TAKUMI at JPARC (Tokai)
- *CSNS*

Strain free lattice parameter, a_0, or interplanar spacing, d_0

For neutron (and synchrotron) measurements, separate determination of the strain free lattice parameter is required in order to obtain quantitative strain values. **Correct measurement of a_0 or d_0 is critical for the accuracy of the strain measurements.** Note that the lattice parameter is a function of **local chemistry** and **temperature**. There are a number of common ways to measure a_0/d_0:

- Far-field measurements (if representative)
- Powders and standards (if representative)
- Slices, cubes or **combs**
- Stress balance (if possible)





Other methods possible as way, see e.g. P.J.Withers et al. J. Appl. Cryst. 40 (2007) 891.

Examples?

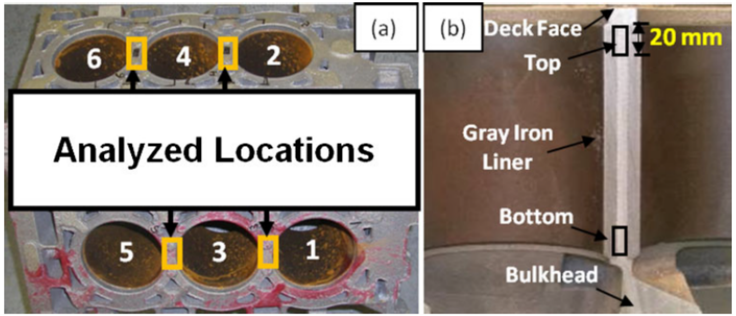
Some published examples connected to industrial use of neutron scattering for residual stress measurements

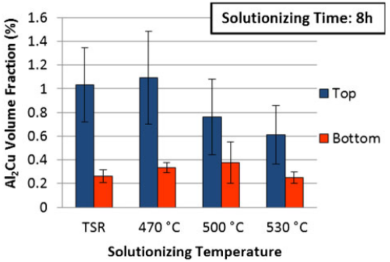
Note! Only based on publically available results!

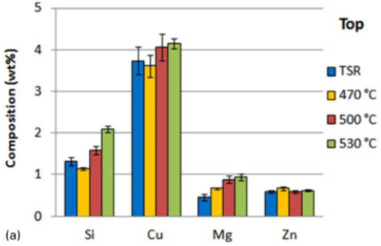
Canadian Metallurgical Quarterly 2015 VOL 54 NO 1

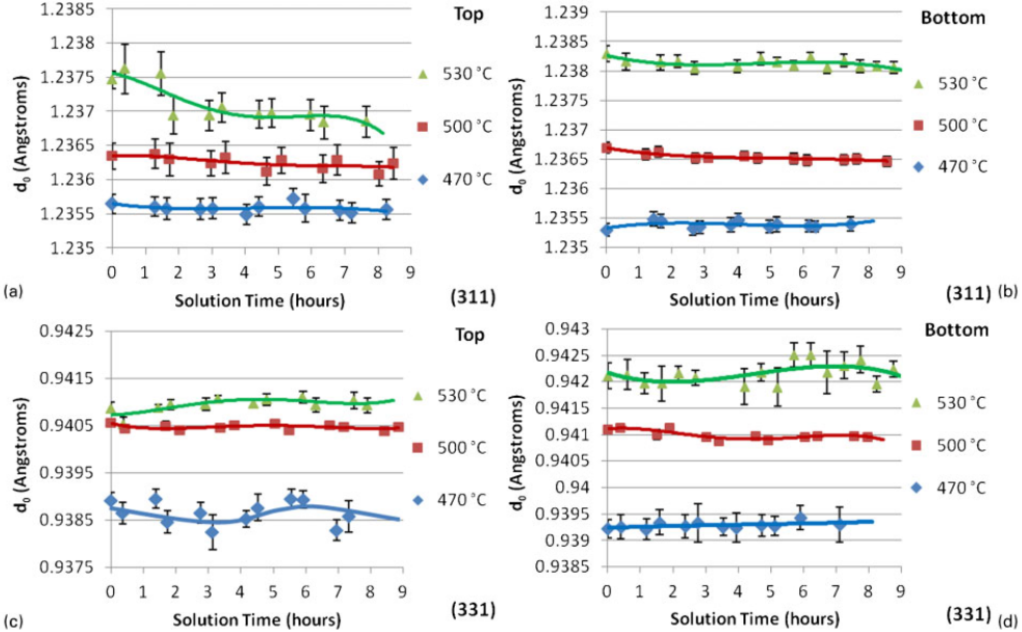
In situ neutron diffraction analysis of stress-free d-spacing during solution heat treatment of modified 319 Al alloy engine blocks

A. Lombardi*[1], D. Sediako[2], C. Ravindran[1] and R. MacKay[3]









Measurements at L3 diffractometer at CNBC

- Dissolution of secondary phases increases solute content, which changes the lattice parameter
- The effect is different at different locations
- The effect is different for different lattice planes

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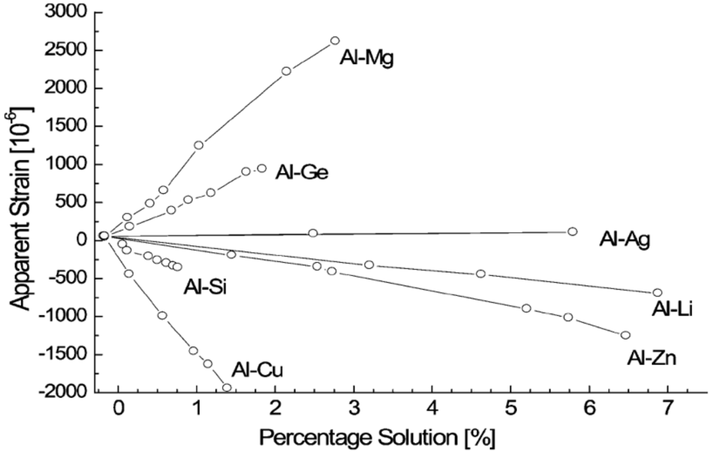
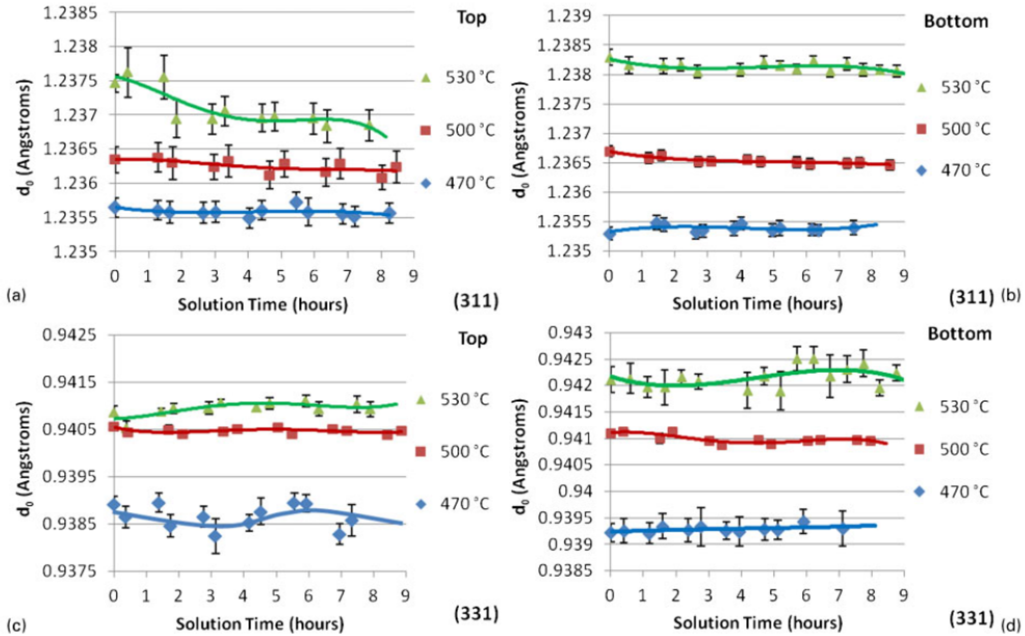


P.J.Withers et al. J. Appl. Cryst. 40 (2007) 891.



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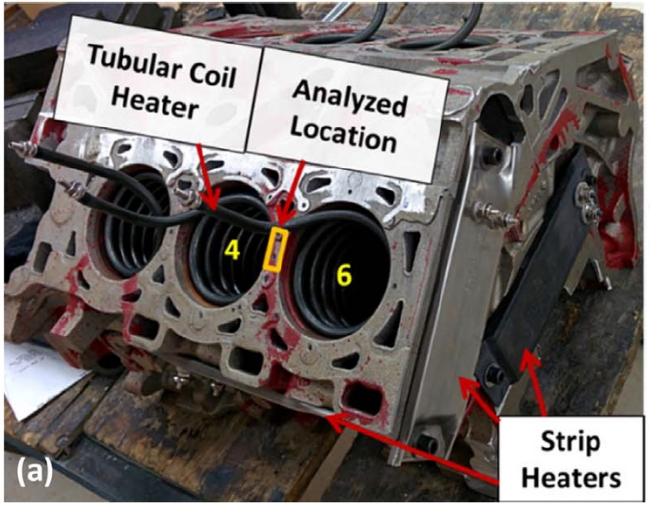
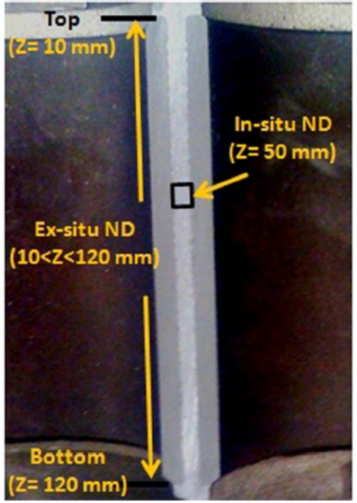
Residual stresses in Al alloy engine blocks

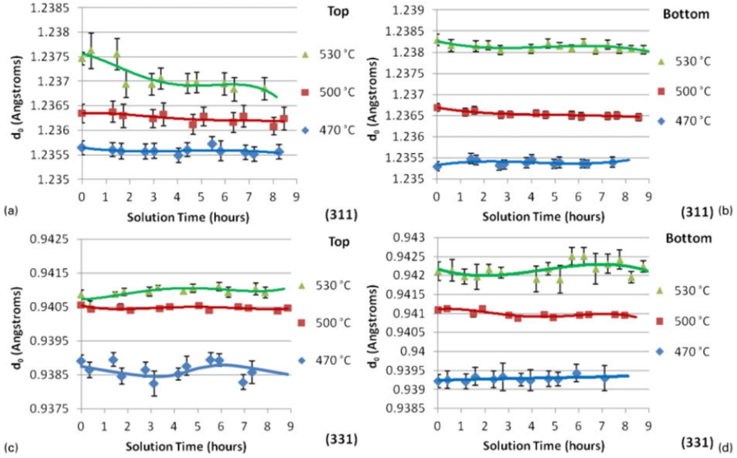
A. Lombardi et al.: Canad. Metall. Quart. 54 (2015) 30.

A. Lombardi et al.: Mater. Lett. 157 (2015) 50.

A. Lombardi et al.: Mater. Sci. Eng. A697 (2017) 238.

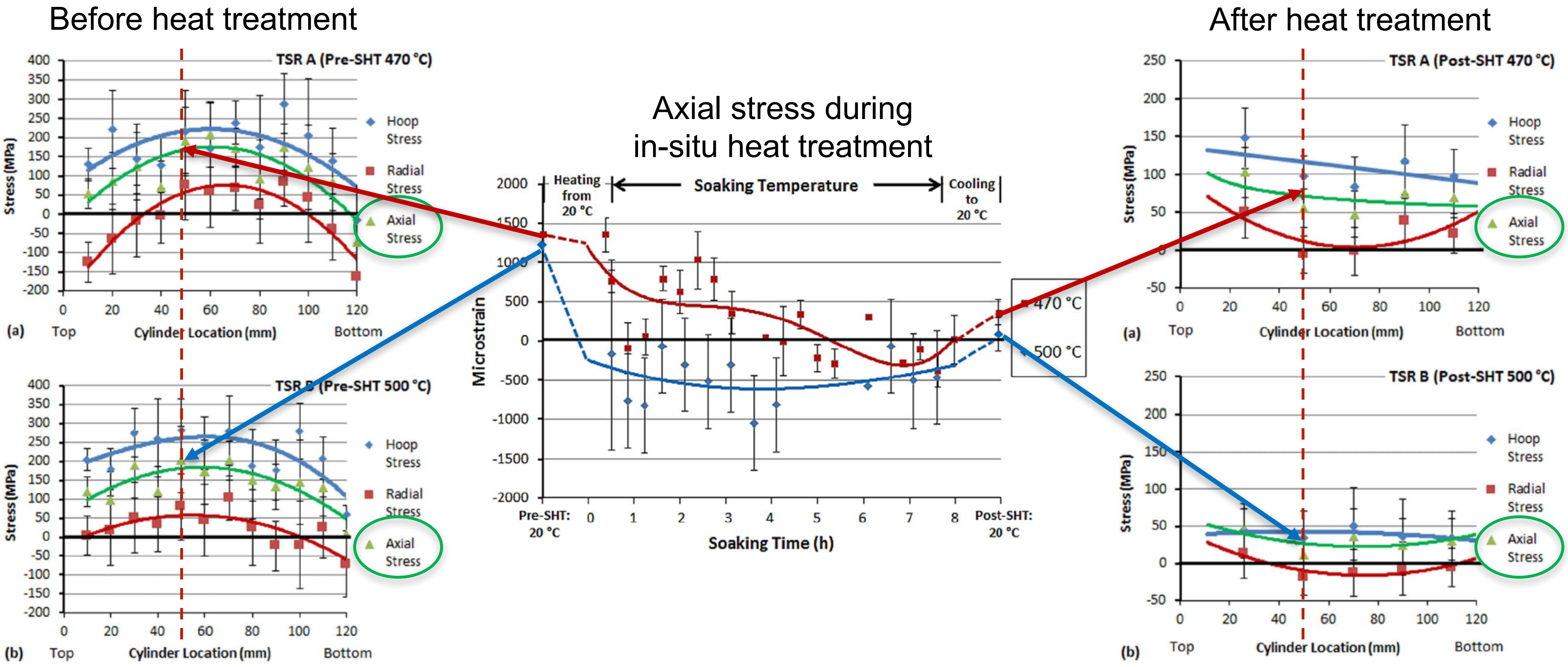




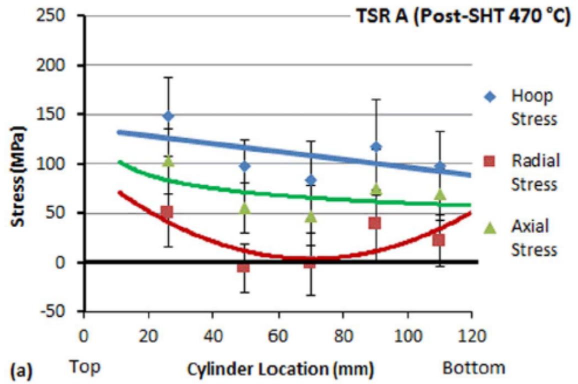


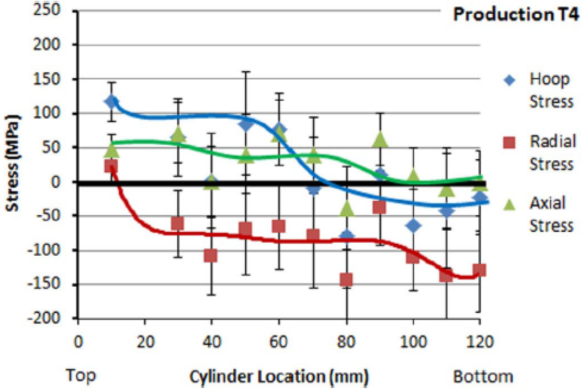
Measurements at L3 diffractometer at CNBC

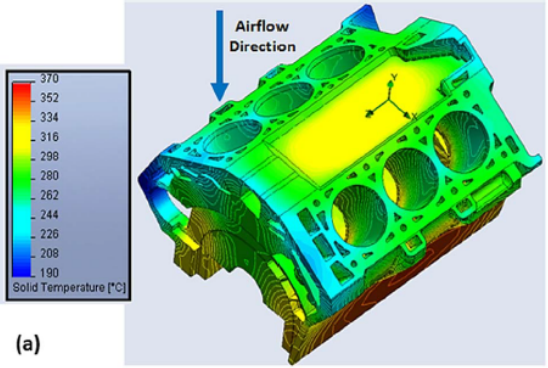
Residual stresses in Al alloy engine blocks

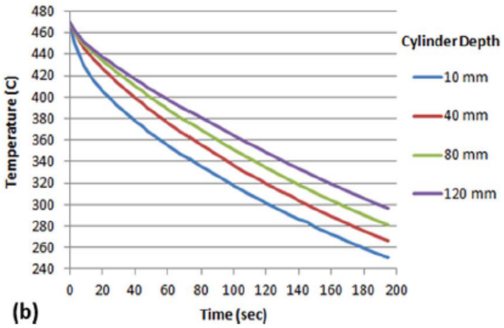


Residual stresses in Al alloy engine blocks









- Possible to follow the relaxation of residual stresses in-situ at high temperatures
- Possible to determine local residual stresses internally in large complex engine blocks
- Allows verification of industrial processes and models
- This in turns allows optimization of processes and modelling approaches

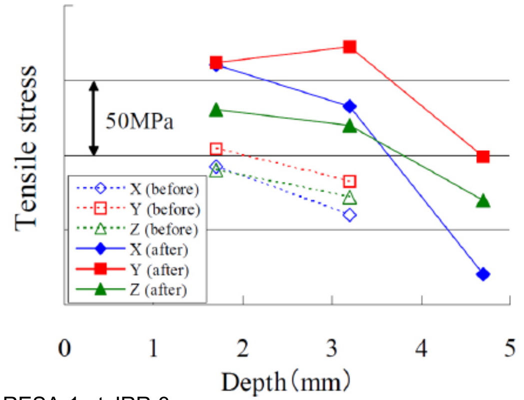
Residual stresses in combustion engine cylinder head before and after durability test

Internal Residual Stress Measurement of Aluminum Alloy Castings Using Neutron Diffraction

Yumi Kubota, Jun Kubo, Keitaro Ishida, Akinori Okada and Minoru Yoshida
Nissan Motor Company Ltd.

Toru Saito and Hiroshi Suzuki
Japan Atomic Energy Agency

SAE Int. J. Engines 5(2):2012, doi:10.4271/2012-01-0549.



Measurements at RESA-1 at JRR-3

Neutrons peer into a running engine

July 26, 2017

In a first-of-a-kind experiment, researchers used neutrons to investigate the performance of a new aluminum alloy in a gasoline-powered engine—while the engine was running.

A team from the Department of Energy's Oak Ridge National Laboratory worked with industry partners to perform the test, which looked at whether a high-performance alloy that is promising for automotive applications held up under the heat and stress of an internal combustion engine.

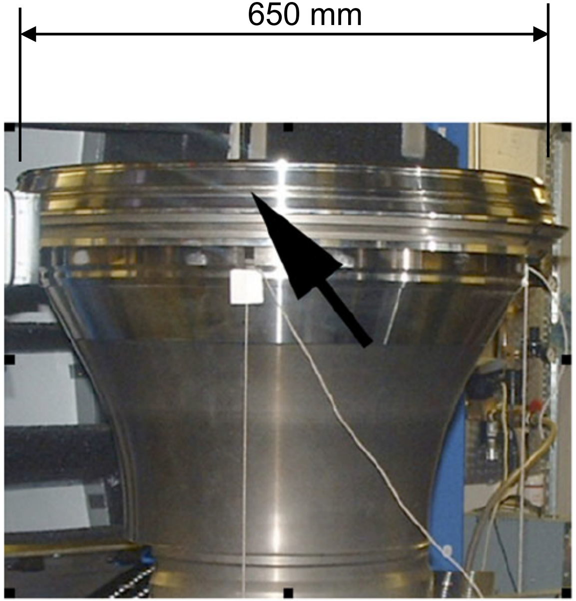
The feat was a first for the Spallation Neutron Source, said Ke An, lead instrument scientist for the facility's VULCAN instrument. "This was the first time an internal combustion engine has been run on our diffractometer, and, as far as we know, on any other," he stated.

Measurements at VULCAN (SNS)

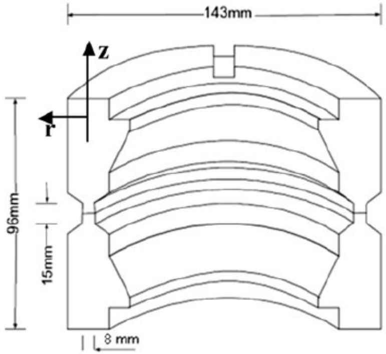
Thermal relaxation of residual stresses in Nickel-based superalloy inertia friction welds

M. Karagde et al.:. Metall. Mater. Trans. 42A (2011) 2301.

Full-scale aero engine high pressure compressor drum



Sub-scale inertia welded rings



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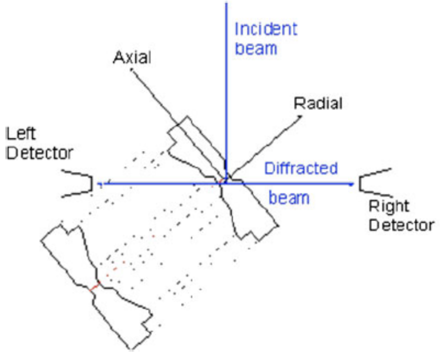
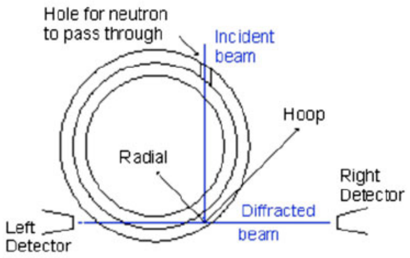
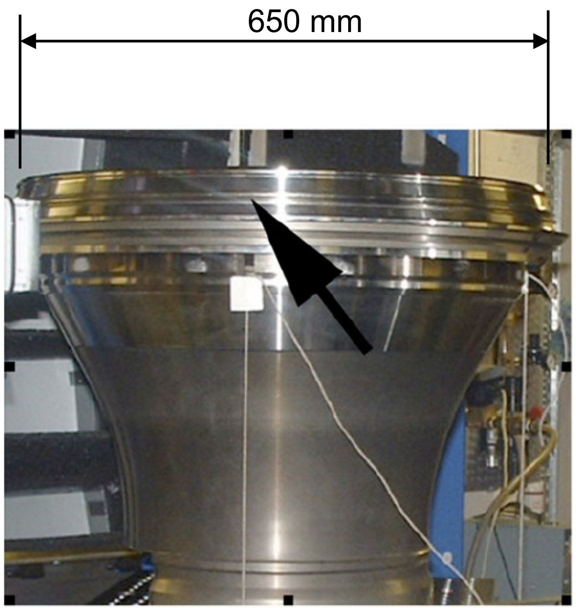




Measurements at ENGIN-X at ISIS and SALS at ILL

Full-scale aero engine high pressure compressor drum



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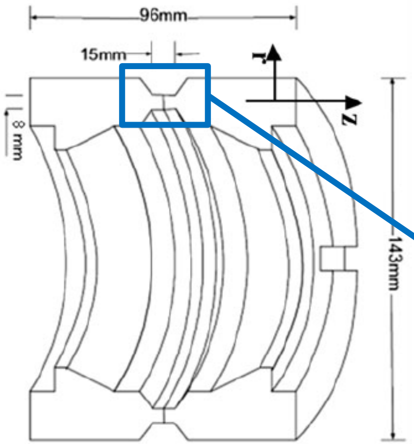

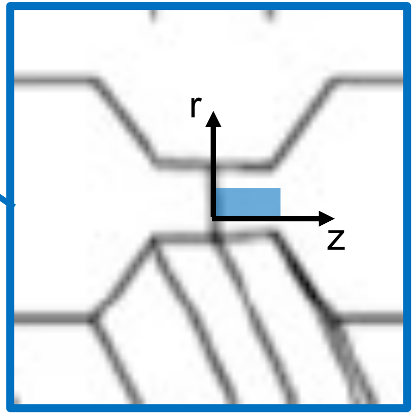

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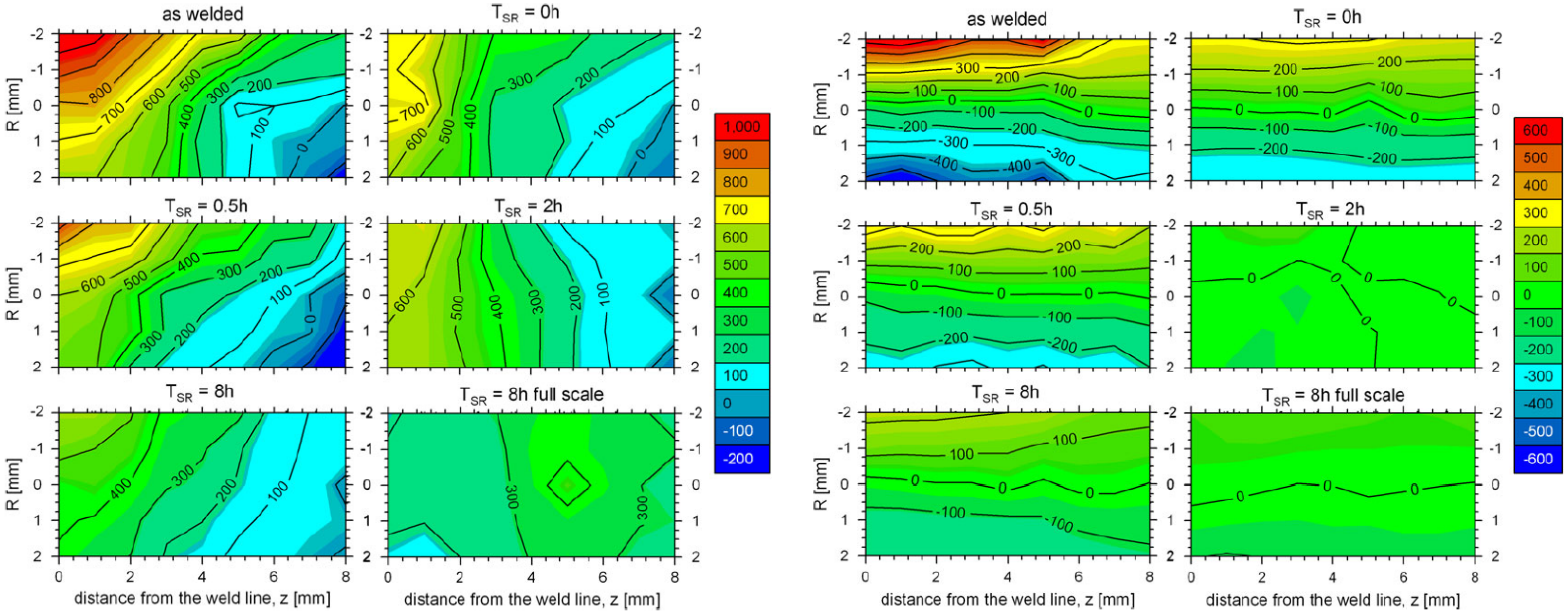
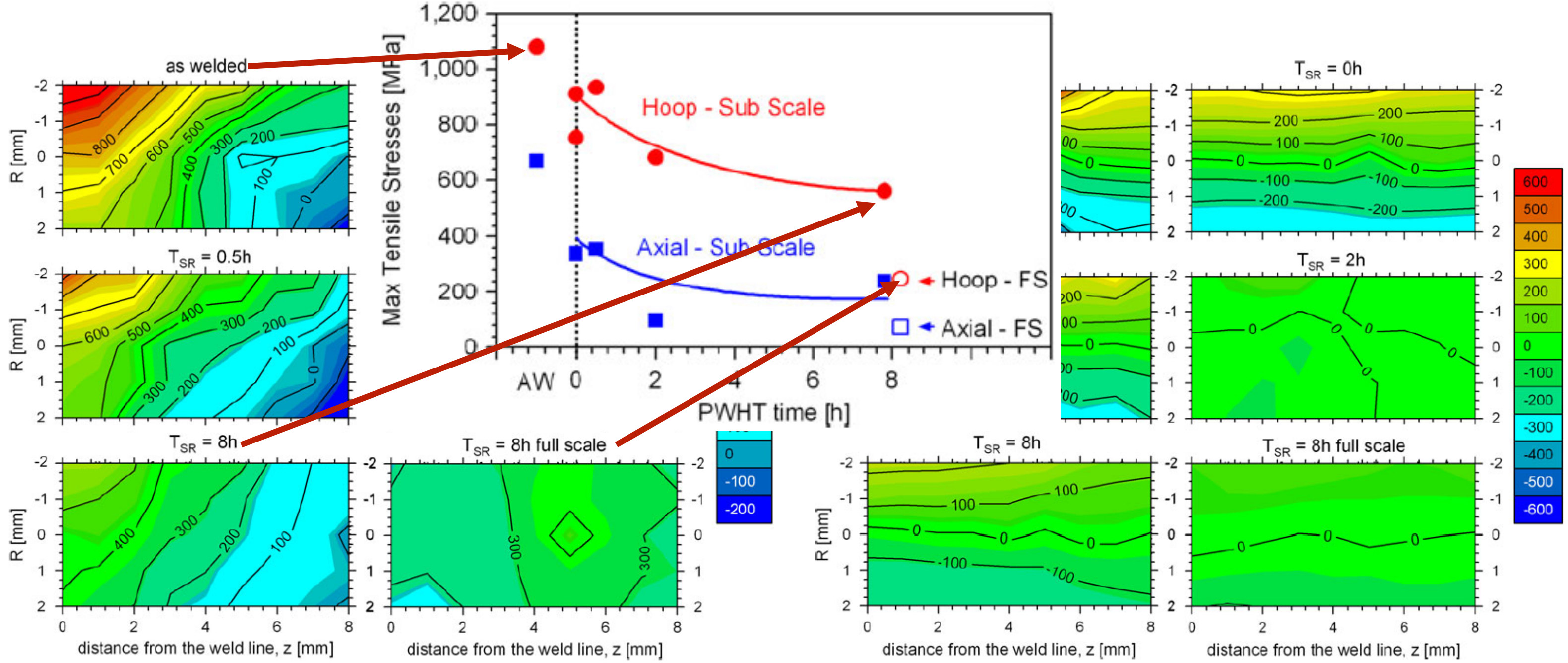


Hoop stresses

Axial stresses

Thermal relaxation of residual stresses in Nickel-based superalloy inertia friction welds

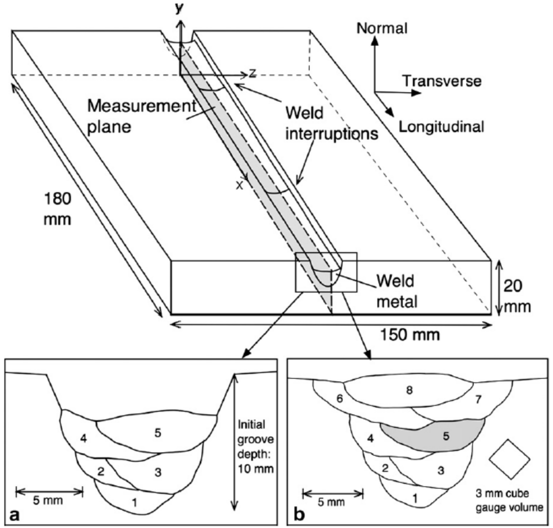


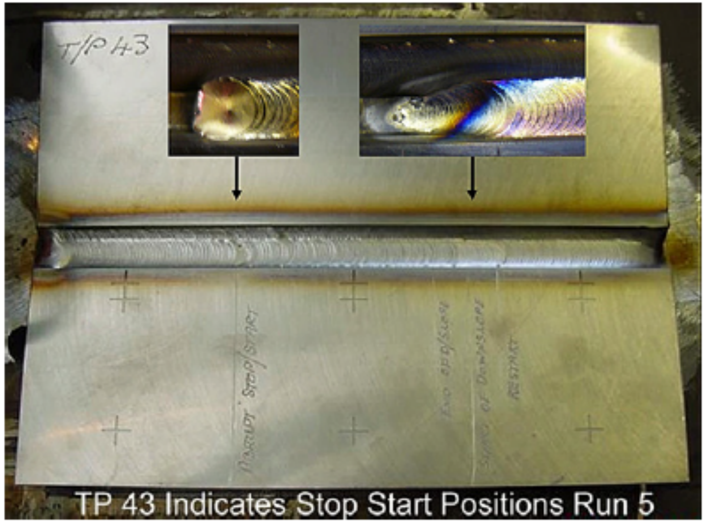
Hoop stresses

Axial stresses

Effects of stop–start features on residual stresses in a multipass austenitic stainless steel weld

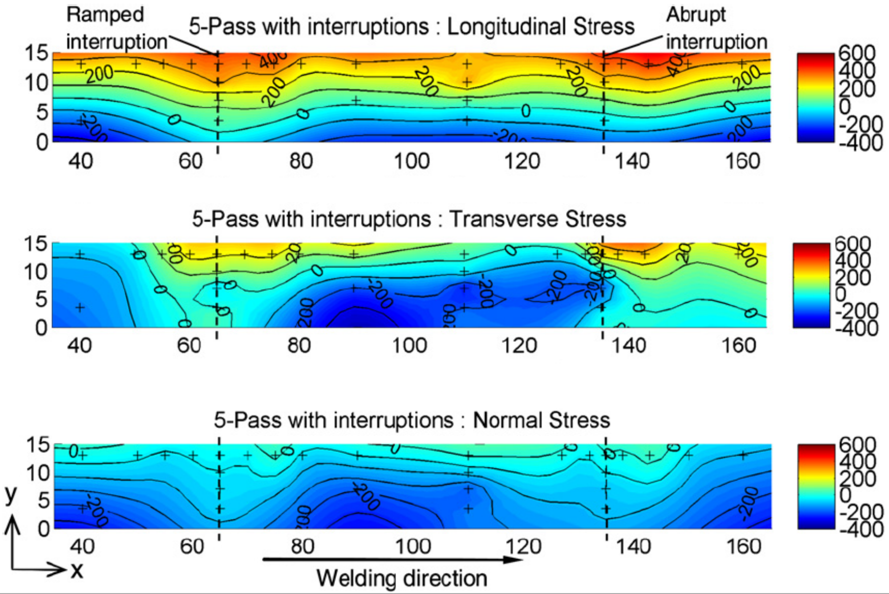
M. Turski, et al.: Int. J. Press. Vess. Piping. 89 (2012) 9.

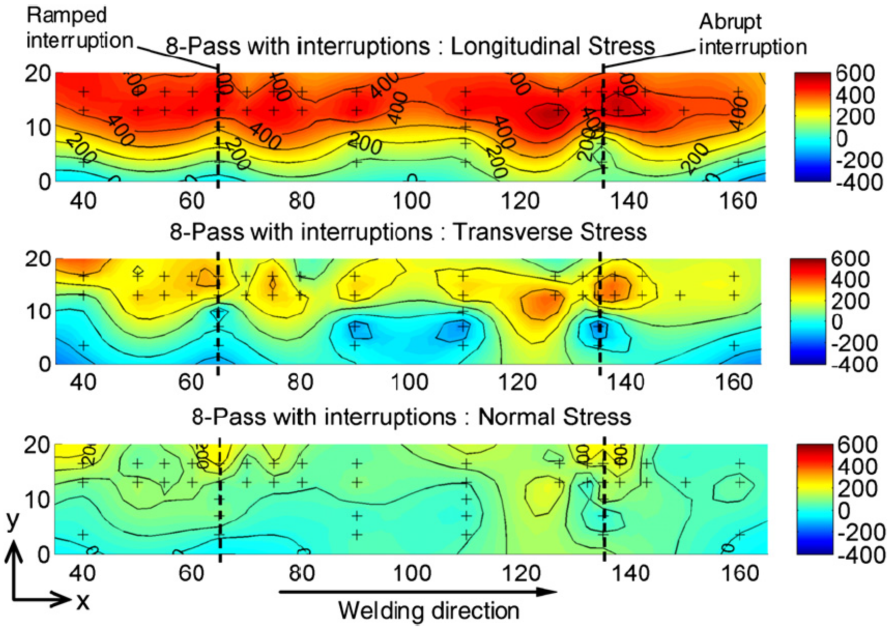




Effects of stop–start features on residual stresses in a multipass austenitic stainless steel weld

- Start–stop features result in local significant increases in tensile residual stresses, in particular if the interruption is abrupt (less for ramped interruptions).
- In the case when over-lay weld passes are applied, the local stress increase persists in the case of abrupt interruptions.
- These effects are important for the planning and execution of welding operations, in particular repair welds when grind-outs are necessary.

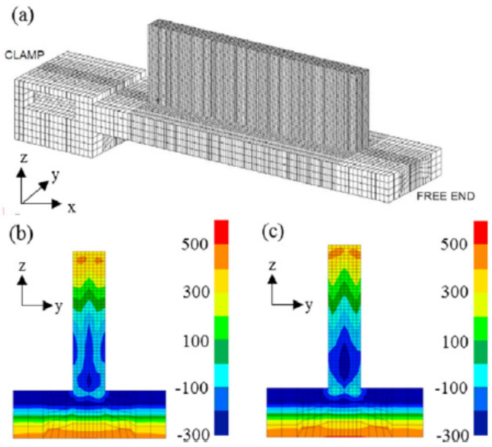


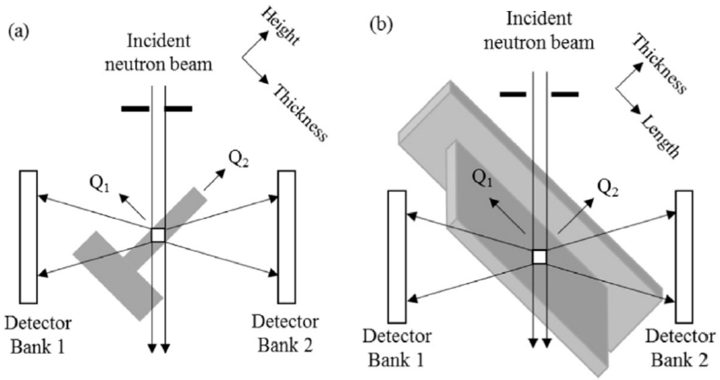


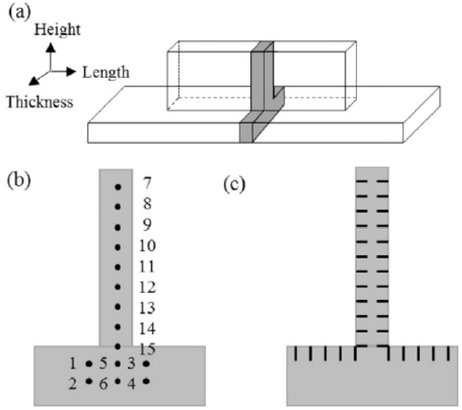
Measurements at POLDI (SINQ at PIS) and STRESS-SPEC (FRM-II)

Residual stress mapping in Inconel 625 fabricated through additive maufacturing: Method for neutron measurements to validate thermomechanical model predictions

Z. Wang, et al.: Mater. Design 113 (2017) 169







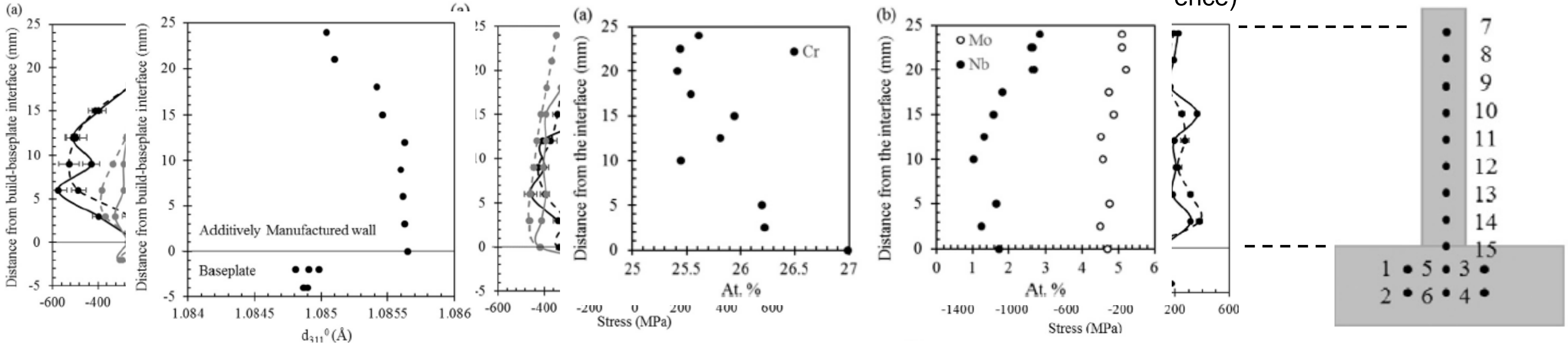
Measurements at VULCAN (SNS)

Residual stress mapping in Inconel 625 fabricated through additive maufacturing: Method for neutron measurements to validate thermomechanical model predictions

Stress in length direction

Stress in height direction

Stress in height direction
(stress relieved reference)



- Good agreements between experiments and simulations
- Stress relieving reference specimens can lead to erroneous results due to microstructural changes.
- Remember: Differences in strain free lattice parameter depends on local chemical variations

Outlook: neutrons for engineering

- In-situ measurements during (simulated) processing and service. This is aided by increased neutron flux and development of advanced sample environments.
- Additive manufacturing will likely increase the interest for neutron scattering strain measurements (in particular for TOF diffraction since texture is usually pronounced in AM processes)
- More focus on model development/calibration/validation, not least with respect to AM processes

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