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PROGRAMME & ABSTRACTS 9<sup>th</sup> International Conference on Mechanical Stress Evaluation by Neutron and Synchrotron Radiation

Hosted by the South African Nuclear Energy Corporation (Necsa) SOC Limited in cooperation with the International Atomic Energy Agency (IAEA)



South African Nuclear Energy Corporation SOC Limited













# MECA SENS 2017 Programme & Abstracts

MECA SENS 2017 Local Organising Committee

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## Committees

## Foreword

#### Dear colleagues

On behalf of the Organising Committee, it is a great privilege to welcome and receive you at the Nombolo Mdhluli Conference Centre in the Skukuza Rest Camp, capital of the UNESCO Biosphere Kruger National Park for the proceedings of MECA SENS 2017. This conference continues the tradition of providing a vibrant interactive forum for scientists, students and engineers interested in the most recent developments and capabilities of diffraction based techniques, complemented by mechanical and image-based methods towards rendering information on the prevailing stress conditions and material performance.

We thank all participants for submitting excellent contributions. This has enabled the assembly of an exciting scientific program for the next three days. The keen interest is underpinned by having 18 countries and 52 institutions represented. Special reference needs to be made to the large South African participation, substantially complemented by students from Africa. We thank the International Advisory Committee for having afforded us the privilege of hosting this event for the first time on the African continent. This presents a firm foundation to involve and build the local community. In addition special reference has to be made to the MECA SENS Spring School preceding the conference where 7 prominent international lecturers unselfishly availed their time.

With MECA SENS 2017 being the 9th event in this conference series since inception in 2000 following an initiative by Prof Alain Lodini, with this conference the prestigious Alain Lodini Plenary Lecture series is being initiated. To inaugurate this tradition, Prof. Philip Withers, having been one of the original participants and still very active in science, will pay homage with his talk titled "Insights into fracture mechanics: what can x-ray diffraction and imaging tell us ?".

Finally I want to take this opportunity to thank the Organising Committee and Inside Edge Conferencing for their hard work with organising the event, the International Advisory Committee for their confidence in us hosting the event, all the speakers, as well as our sponsors for contributing to make MECA SENS 2017 a success. The latter has made the participation by a large number of students, who constitute the future of our community, possible. We hope that you will have a truly stimulating time with vigorous discussion and networking.

Notwithstanding, afford yourself the time to enjoy the splendour of the Kruger National Park and the South African hospitality.

Sincerely yours

Andrew Venter Chairman: MECA SENS 2017

## Local Organising Committee

- Andrew Venter: Chairman. Section Leader Diffraction, Necsa SOC Limited
- Mihloti Baloyi: Secretary. Pel Labs for Accelerator Based Science, Necsa SOC Limited
- Tshepo Ntsoane: Senior Scientist, Necsa SOC Limited
- Deon Marais: Senior Engineer, Necsa SOC Limited
- Sheryl van den Berg: Conference organiser. Operations Manager, The Inside Edge

## Programme Advisory Committee

- Anthonie Cilliers: Advanced High Temperature Reactor R&D Lead and Program Manager Nuclear Engineering, School of Mechanical & Nuclear Engineering, North West University.
- Axel Steuwer: Director of Science Support Services at University of Malta, Spain, as well as Adjunct Professor in Materials Engineering, Nelson Mandela Metropolitan University, Port Elizabeth.
- Claudia Polese: School of Mechanical, Industrial and Aeronautical Engineering, University of the Witwatersrand.
- Daniel Francis: Expert: Pressure Vessels and Heat Exchangers Sasol Group Technology.
- Daniel Hattingh: Director eNtsa (Centre for Innovation through Engineering) and Professor in Mechanical Engineering, Nelson Mandela Metropolitan University, Port Elizabeth.
- Esther Akinlabi: Head of Department of Mechanical Engineering Science, Faculty of Engineering and the Built Environment, University of Johannesburg.
- Mark Newby: Senior Consultant, Eskom Research, Testing and Development, Sustainability Division.
- Philip Doubel: Corporate Specialist Materials and Welding, Research Test & Development Dept, Sustainability Group, Eskom Holdings SOC Ltd.
- Pieter Pistorius: Department of Materials Science and Metallurgical Engineering, University of Pretoria.
- Sisa Pityana: National Laser Centre, CSIR.
- Thorsten Becker: Dep. of Mechanical and Mechatronic Engineering, University of Stellenbosch.

### International Scientific Committee

- Alain Lodini (University of Reims, France).
- Andreas Schreyer (European Spallation Source, Sweden).
- Andrew Venter (Necsa SOC Ltd, South Africa).
- Axel Steuwer (University of Malta, Spain).
- Chedly Braham (PIMM, France).
- Cevdet Noyan (Columbia University, USA).
- Wan Chuck Woo (KAERI, South Korea).
- Daigo Setoyama (Toyota Central Research and Development Laboratories, Japan).
- Donald Brown (LANL, USA).
- Hahn Choo (University of Tennessee, USA).
- Hiroshi Suzuki (JAEA, Japan).
- Jens Gibmeier (KIT, Germany).
- Jette Oddershede (FYSIK, Denmark).
- Jon Almer (APS, USA).
- Ke An (ORNL, USA).
- Keisuke Tanaka (Meiji University, Japan).
- Krzystof Wierzbanowski (AGH University of Science and Technology, Poland).
- Lyndon Edwards (ANSTO, Australia).
- Mark Bourke (LANL, USA).
- Mark Daymond (Queen's University, Canada).
- Michael Fitzpatrick (Coventry University, UK).
- Ondrej Muransky (ANSTO, Australia).
- Petr Lukas (UJF, Czech Republic).
- Phil Withers (University of Manchester, UK).
- Ron Rogge (CNL, Canada).
- Shu Yan Zhang (ISIS, UK).
- Thilo Pirling (ILL, France).
- Thomas Buslaps (ESRF, France).
- Walter Reimers (TU-Berlin, Germany).
- Xun-Li Wang (The City University of Hong Kong, China).
- Yo TOMOTA (Ibaraki University, Japan).
- Yoshiaki Akiniwa (Yokohama National University, Japan)

## Sponsors



South African Nuclear Energy Corporation SOC Limited







Science & Technology Facilities Council







## DECTRIS®

## **Event Information**









Nombolo Mdhuli Conference Centre – Skukuza

All Plenary sessions will take place in the Main Venue. Oral presentations are scheduled to run as 2 parallel sessions respectively hosted in the Main Venue and the combined breakaway rooms Ndau and Nari. The poster session will be held in the foyer area.

### Registration

The registration desk is located in the Nombolo Mdhluli Conference Centre Foyer at the Skukuza Rest Camp and will be open during the following times:

- Sunday 17<sup>th</sup> September: 14:00 18:00
- Monday 18<sup>th</sup> September: 08:30 18:00
- Tuesday 19<sup>th</sup> to Thursday 21<sup>st</sup> September: 9:00 18:00

### Internet

As we are in the middle of the bush, connecting to the internet may be intermittent. No LTE signal is available at Skukuza, however coverage maps of Vodacom, MTN and Cell C claim 3G availability. From experience the use Vodacom is advised. SIM cards can be purchased at international airports (OR Tambo and Cape Town International) provided the delegate can show a valid passport and proof of accommodation.

The Skukuza Conference Centre provides limited WiFi access. In addition, the conference organisers have set up a dedicated WiFi hotspot in the foyer for the duration of the conference. WiFi is also available at the Cattle Barron restaurant. Remember, it is better to enjoy the interesting lectures and serenity of the Kruger than to be captured by gadgets.

## Catering

All meals and events are for conference delegates and registered accompanying persons only.

*Spring School*: We will be having an informal get-together braai for the School participants starting around 18:00 on Sunday the 17<sup>th</sup>. The exact area will be communicated during registration. "Braai" is the Afrikaans word for "barbecue" and is a popular social custom in South Africa. An important distinction between a braai and a barbecue is that wood or charcoal is used as heat source instead of gas. Breakfast on the 18<sup>th</sup> will be served in the Cattle Barron restaurant.

*Welcome function*: Please join us for an informal welcome function starting at 18:30 on Monday the  $18^{th}$  at the Skukuza Boma.

*Poster cocktail function*: The poster cocktail function on Tuesday the 19<sup>th</sup> will start at 18:00 and take place in the Conference Centre foyer.

*Gala dinner*: The conference gala dinner takes the form of a "bush braai" which promises to be an unforgettable experience. Vehicle loading for the bush drive to the off-site dinner venue will commence at 16:45 on Wednesday the  $20^{th}$  from the Conference Centre parking area.

*Conference lunches*: Lunch will be served in the Conference Foyer area for all delegates on the conference days. Accompanying persons are not allocated for.

*Breakfasts and dinners*: All breakfasts and dinners that have been booked will be at the Cattle Barron restaurant. The restaurant is open every day from 7:00 to 21:00. Breakfasts and dinners can also be taken on the spur of the moment at this venue. Payments can be made by cash (South African Rand) or bank cards.

### Presentations

Please refer to the Programme section for the detailed program. To accommodate delegate movement between parallel sessions, it is imperative that presenters strictly keep to the allocated time slots. To stimulate vibrant scientific exchange, 5 minutes questions / discussion time will be strictly reserved after each presentation in addition to the allocated times stipulated below.

- Alain Lodini Plenary lecture and the Keynote sessions: 40 minutes
- Invited speakers: 25 minutes
- Oral presentations: 15 minutes

A standard data projector (with VGA connector) and a Microsoft Windows laptop with Office and Adobe Reader are available for presentations. Presenters are requested to upload their PowerPoint presentations to the laptop (via USB) during the break preceding their talk. Should presenters wish to use their own equipment for presentation, it is their responsibility to provide the correct converters and cables to ensure compatibility.

All the keynote lectures will be in the Ndlopfu room, with parallel sessions to take place in the Ndlopfu and Ndau/Nari rooms.

### Poster session

Boards to accommodate A0 size, 841 mm x 1189 mm, portrait style posters will be located in the Conference Centre Foyer and delegates are requested to put up their posters during registration. Drawing pins / thumbtacks / Prestik / Blu Tack will be provided to secure posters to the boards. Posters must remain up for the full duration of the conference and presenters are required to be at their posters for discussion during the Poster Session starting at 18:00 on Tuesday the  $19^{th}$ 

## Certificate of attendance

Should certificates of attendance be required, please notify the registration desk during on-site registration. Certificates will be available at the conference desk on Thursday the 21<sup>st</sup>.

### Community outreach

Our focus is on locally manufactured corporate gifts of quality that do no harm, help clean up the environment, are recyclable and reusable, and enable companies to reduce their carbon footprint. We are passionate about supporting community crafters and local small businesses that create employment and up-skilling of disadvantaged people.

## Programme

## Spring School - Monday 18 Sept

	Room: Ndau	Foyer
08:15 - 9:30	Dr Tom Holden (Canada)	
	Fundamentals of neutron diffraction	
09:30 - 10:45	Dr Don Brown (LANL, USA)	
	Fundamentals of X-ray and synchrotron radiation and its	
	value addition in material science	
10:45 - 12:00	Dr Ondrej Muransky (ANSTO, Australia)	
	The role of numerical analysis in the assessment of weld	
	residual stresses	
12:00 - 12:45	Lunch	
12:45 - 14:00	Dr Saurabh Kabra (ISIS, UK)	
	Capabilities of in-situ loading investigations using	
	neutron diffraction/imaging towards elucidating	
	fundamental and industry related problems in material	
	science	
14:00 - 15:15	Dr Vladimir Luzin (ANSTO, Australia)	
	Neutron stress scanning with high spatial resolution for	
	surface engineering applications	c
15:15 - 15:30	Coffee	ck-ii
15:30 - 17:00	Dr Sven Vogel (LANL, USA)	chec
	Introduction to Texture Analysis and Microstructure	0 pr
	Characterization using Diffraction Data	n ar
17:00 - 17:50	Prof Phil Withers (University of Manchester, UK)	itioi
	Completing the picture: Correlative imaging and	stra
	diffraction	legi
17:50 - 18:05	Dr Andrew Venter (Necsa, SA)	£
	Diffraction based residual stress capabilities available to	
	South African researchers	
18:30 -	Conference Welcome Function	

## Day 1 – Tuesday 19 Sept

			Room: Ndlopfu			Room: Ndau/Nari
08:30 - 08:45	Registration					
08:45 - 08:50	an	Andrew Venter	Foreword			
08:50 - 09:10	γογ	Kelvin Kemm	Official Opening			
09:10 - 09:15	evl	Don Brown	Welcome			
09:15 - 10:00	Ŭ U		Alain Lodini Plenary:			
	haii	Phil Withers	Insights into fracture mechanics: what can x-			
	C		ray diffraction and imaging tell us?			
10:00 - 10:30			Coffee	e breal	(	
10:30 - 11:00			Application of in-situ experimental methods			
		Kristián Máthis	for revealing deformation mechanisms in		Supriyo Ganguly	Control of residual stress and microstructure
			advanced magnesium alloys			in large scale additive manufacturing
11:00 - 11:20			Stress partitioning and phase behavior in a			Investigating the residual stress distribution
	20	Braham C	pearlitic steel studied using high energy X-		*Anderson L S	in Selective Laser Melting produced Ti-6Al-
	r r	brunani e.	ray synchrotron radiation	≌ S	Anderson E.S.	4V using neutron diffraction
	odel Iwe			V ldir		
11:20 - 11:40	Mo		Internal Mechanics of Parallel Wire Cable	We uck		The influence of high welding speed on the
	n & el S	*Brügger A.	Strands	s ch	Bernard D.	residual stresses and microstructure in
	tion: Ax			sing		friction stir welded AA5182-H111
11:40 - 12:00	ma		In Situ Neutron Transmission Bragg Edge	ir: /		Development of Phase Stresses During
	ch Ch	Connolly M.J.	Measurement of Strain Fields Near Fatigue	Pro	Dahal J.	Additive Manufacture of 304L Stainless Steel
	ă		Cracks Grown in Hydrogen	- 0		
12:00 - 12:20			Stress Evaluation of Adhesively Bonded Lap		Cabeza Sánchez	Residual stress fields in additively
		*Khodja M.	Joints with Aluminium 2024-T3 Adherents		S.	manufactured Ni superalloy 718 as built and
			Using FEA			after release from baseplate
12:20 - 12:40			Micromechanical behavior in additively			Synchrotron XRD Evaluation of Residual
		An K.	manufactured textured Inconel 718		Newby M.	Stresses Introduced by Laser Shock Peening
10.10.10.50						for Steam Turbine Blade Applications
12:40 - 12:50			Conferei	nce pn	oto	
12.30 - 14.13			LU			
14:15 - 14:45						Internal stresses, strains and phase fractions
14:15 - 14:45		Chris Wensrich	Practical demonstration of Bragg-edge neutron transmission strain tomography		Petr Sittner	Internal stresses, strains and phase fractions surrounding martensite band front in superelastic NITi wire loaded in tension
14:15 - 14:45 14:45 - 15:05		Chris Wensrich	Practical demonstration of Bragg-edge neutron transmission strain tomography An update on the Engineering materials		Petr Sittner	Internal stresses, strains and phase fractions surrounding martensite band front in superelastic NiTi wire loaded in tension In situ x-ray diffraction of shock-driven
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10:20 - 10:40	iiques & Inst air: Jens Gib	Marais D.	Alignment and calibration procedures of the Necsa neutron strain scanner	cessing & W Chair: Ke A	Doubell P.	Neutron diffraction investigation of residual stresses in Nickel based austenitic weldments on creep resistant Cr-Mo-V material
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12:35 - 12:55	Surface Chair	*Ivanovic N.	Effectiveness of Laser Shock Peening in Post processing Additive Manufactured Ti-6Al-4V	Def Ch	Fatoba O.S.	Investigating stresses developed during mechanical forming of steel through Finite Element Analysis
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15:00 - 15:20	iracterisat uransky	Brown D.W.	Grain scale microstructure evolution characterization of ceramic nuclear fuels	and Coati ogel	Fatoba O.S.	Numerical modelling and performance effects of laser deposited Ti-Al-Sn coating on ASTM A29 steel
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#### Insights into fracture mechanics: what can x-ray diffraction and imaging tell us?

Withers P.J.<sup>1</sup>, Simpson C.<sup>1</sup>, Buslaps T., Lopez-Crespo P.

<sup>1</sup>School of Materials, Manchester University, M13 9PL, United Kingdom and Research Complex at Harwell, Didcot, OX11 0FA, UK <sup>2</sup>ESRF, 6 rue J Horowitz, 38000 Grenoble, France, <sup>3</sup>Department of Civil and Materials Engineering, University of Malaga, C/Dr Ortiz Ramos, s/n, 29071 Malaga, Spain

Traditionally fracture and fatigue cracks are followed in terms of global parameters such as crack length, sample geometry etc. based solely on surface observations alongside measurements such as potential drop and crack face compliance that provide average data. In most cases this is fine but increasingly novel strategies are being adopted exploiting intrinsic and extrinsic toughening mechanisms so that the stress at the crack tip is not well represented by global analyses. To better understand the relationship between the nucleation and propagation of microstructurally short cracks and the local stresses and phase changes that cause them, we need both imaging and stress mapping at the microscopic scale.

Here, I explore how this can be achieved by bringing together high spatial resolution X-ray diffraction and tomographic imaging. Conventionally, these are undertaken on separate instruments; however, instruments capable of both imaging and diffraction are beginning to emerge. I will explore the concept of threedimensional crack-tip X-ray microscopy, bringing them together to probe the crack-tip behaviour under realistic environmental and loading conditions and to extract quantitative fracture mechanics information about the local crack-tip environment. X-ray diffraction provides information about the crack-tip stress field, phase transformations, plastic zone and crack-face tractions and forces. Time-lapse CT, besides providing information about the three-dimensional nature of the crack and its local growth rate, can also provide information as to the activation of extrinsic toughening mechanisms such as crack deflection, crack-tip zone shielding, crack bridging and crack closure. Through a series of examples I will show how crack-tip microscopy allows a quantitative measure of the crack-tip driving force via the stress intensity factor or the crack-tip opening displacement both as a means improve the fracture toughness and fatigue life of components and as a means of testing FE model predictions. Finally, further opportunities for x-ray imaging and stress field mapping in fracture and fatigue will be explored.

Email corresponding author: p.j.withers@manchester.ac.uk

# Diffraction methods and scale transition model used to study evolution of the intergranular stress and micro-damage phenomenon during elasto-plastic deformation

Baczmański A.<sup>1</sup>, Wroński S.<sup>1</sup>, Wierzbanowski K.<sup>1</sup>, Gadalińska E.<sup>2</sup>, Zhao Y.<sup>3</sup>, Le Joncour L.<sup>3</sup>, Braham C.<sup>4</sup>, Buslaps T.<sup>5</sup>, Scheffzük C.<sup>6,7</sup>

<sup>1</sup>AGH- University of Science and Technology, WFiIS, al. Mickiewicza 30, 30-059 Krakow, Poland
<sup>2</sup>Institute of Aviation, al. Krakowska 110/114, 02-256 Warszawa, Poland
<sup>3</sup>ICD-LASMIS, Université de Technologie de Troyes (UTT), 12 rue Marie Curie, 10004 Troyes, France
<sup>4</sup>PIMM, Arts et Métiers ParisTech (ENSAM), 151 Bd de l'Hôpital, 75013 Paris, France
<sup>5</sup>ESRF, 6, rue J. Horowitz, 38500 Grenoble Cedex, France
<sup>6</sup>Frank Laboratory of Neutron Physics, Joint Institute for Nuclear Research, 141980 Dubna, Russia
<sup>7</sup>Karlsruhe Institute of Technology, Adenauerring 20b, 76131 Karlsruhe, Germany

A methodology combining diffraction experiments and self-consistent calculations was used to study the mechanical behaviour of groups of grains within single- or multi-phase polycrystalline materials. In this work, Al/SiC<sub>p</sub> composite, pearlitic steel, and duplex austenitic-ferritic steel are studied. Special attention is paid to the role of first- and second-order stresses on the yield stresses of the phases, as well as on the evolution of these stresses during the deformation process. Intergranular stress evolution was determined from lattice strain measured in situ during tensile tests and after sample unloading, using neutron diffraction (JINR, Dubna, Russia and ISIS, RAL, UK). The experimental results were used to study slip on crystallographic planes, localisation of stresses in polycrystalline grains, and the mechanical effects of damage occurring during plastic deformation. For this purpose a prediction made using the recently developed new version of the elasto-plastic self-consistent model was compared with the experimental data [1].

X-ray synchrotron radiation (ID15B, ESRF, Grenoble, France) was used to scan in-situ variation of interplanar spacings along the necking zone for samples of duplex steel subjected to tensile loading. The self-consistent model and FEM simulation were applied to interpretation of the experimental data. It was found that, for advanced necking, the phase lattice strains, especially those measured at some distance from the centre of the neck, show a large inversion of the loads localised in both phases compared to the undamaged state (the lattice strains in ferrite are smaller than in austenite; cf. Figure 1). This effect indicates stress relaxation in ferrite, which is connected with damage phenomenon [2].



Figure 1. (a) Scanning of lattice strain in the deformation neck using synchrotron radiation; (b) evolution of the lattice strains measured for different reflections in ferrite and austenite as a function of position in the neck

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Email corresponding author: Andrzej.Baczmanski@fis.agh.edu.pl

#### Saint Venant's Principle and Effect of Complex Geometries in X-ray and Neutron Strain Analysis

#### Noyan I.C.

#### Dept. of Applied Physics and Applied Mathematics, Columbia University, New York, NY 10027, USA

Most diffraction stress determination techniques assume simplified stress/strain states within the measurement volume from which diffraction data is acquired. Such assumptions enable saving precious measurement time, and/or or make any subsequent analysis more tractable. They are usually based on the macroscopic geometry of the sample. These assumptions are also incorporated in numerical models of the systems under examination. Despite widespread usage, such assumptions are not usually justified *a-posteriori*. Such omissions can lead to serious errors in the stress/strain analysis results.

In this presentation we will discuss the effect of local heterogeneities on the residual and applied elastic stress/strain states within macroscopically symmetric samples. Experimental data and modelling results from axial and radial mechanical loading will be presented and compared. We will demonstrate and discuss the length scales over which local stress components tend to far-field values, and show that the specification of St. Venant's "characteristic dimension" over which local effects tend to base values can be non-trivial in some cases.

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Email corresponding author: icn2@columbia.edu

#### Why Multiple Methods are Best

#### Bouchard P.J.<sup>1</sup>

#### <sup>1</sup>Materials Engineering, The Open University, Walton Hall, Milton Keynes, MK7 6AA, UK

Residual stress is a second rank tensorial quantity that is spatially distributed throughout engineered components and structures at multiple length-scales. Detailed quantification of the meaningful residual stress state in a body remains a challenge even with the development of increasingly powerful diffraction and strain relief measurement techniques [1] and sophisticated prediction methods. Instrument scientists at Central Facilities and strain relief measurement practitioners have deep understanding and skills in applying their specialist methods, such as neutron diffraction, synchrotron diffraction, incremental centre hole drilling and the contour method, but their knowledge of other techniques and modelling approaches can be sparse. Similarly, engineers in industry simulating fabrication processes such as forging, welding and heat treatment, are highly skilled in modelling but tend to have less knowledge of measurement technology. When the communities are brought together, experimentalists and modellers commonly express deep confidence in their own approach and work. Likewise there is a potential tension arising from the confidence of those practising diffraction methods.

This paper starts from the premise that application of multiple methods gives a more complete tensorial, spatial and length-scale data characterisation of residual stress in an engineered structure. The term 'multiple methods' encompasses any displacement, strain or stress measurement technique as well as modelling approaches including finite element analysis, eigenstrain approaches, analytical neural networks and measurement simulation (to inform or correct a measurement). Multiple application of the same measurement technique can give an estimate of stress measurement uncertainty but its rarely feasible or economic to do so outside of round robin exercises, such as those performed on the VAMAS shrink-fit ring and plug round robin sample [2] and the European NeT consortium on weldment benchmarks [3]. Multiple methods are of course applied when it is required to 'validate' a specific modelling approach and some examples, mainly associated with industrial applications, can be found in published literature [4]. It is argued here that greater attention and importance should be given to applying characterisation methods based upon diverse principles because this reduces the uncertainty in quantifying the meaningful stress state, as well as revealing the limitations of individual approaches and giving a more complete picture of the stress state in the component of interest. This paper demonstrates the benefits of applying multiple methods using published and new residual stress state characterisation examples.

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Email corresponding author: john.bouchard@open.ac.uk

#### In-Situ Phase Separation of U/Nb Alloys During Quenching

Brown D.W., Pokharel R., Losko A.S., Zhang J.

Material Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM, 87545

Alloying of uranium with niobium increases the corrosion resistance and ductility of the metal, but the microstructure is unstable. Specifically, the niobium which is stable in the high temperature cubic phase of uranium, is unstable at lower temperature phases and can phase separate during quenching unless quench rates are higher than 20°C/sec [1]. This, in turn, destroys the gains in corrosion resistance and ductility achieved through alloying. The metastable phase diagram of the uranium/niobium alloys system is rich and the room temperature structures as a function of niobium content and time at elevated temperature are well understood [2]. However, until recently, the pathways to the final structures including intermediate phase transformations were unobservable because of the high quench rate.



Coupled diffraction and SAXS data were collected with 0.3 second time resolution during quenching of U6Nb from 800C to 550C on 1-ID at the APS.

Recent advances in high energy (>80keV) synchrotron x-ray techniques have enabled first of their kind coupled small angle x-ray scattering (SAXS) and diffraction measurements during quenching and aging of uranium/niobium alloys. These techniques provide windows into phase behavior of U/Nb alloys during quenching on multiple length scales. The figure shows a composite of diffraction patterns and SAXS data collected during a quench. The streaks in the SAXS pattern are indicative of a plate like microstructure observed ex-situ, while the diffraction shows the evolution of the phases. The in-situ data reveals microstructural evolution in the alloys system at times scales not previously achievable.

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Email corresponding author: dbrown@lanl.gov

## The relationship between processing parameters and defect characteristics in a 3-D printed stainless steel alloy: A Synchrotron X-ray Tomography Study

Choo H.<sup>1</sup>, Ngo A.<sup>1</sup>, Xiao X.<sup>2</sup>, Ren Y.<sup>2</sup>, Matthews M.<sup>3</sup>, Garlea E.<sup>4</sup>

<sup>1</sup>Materials Science & Engineering, University of Tennessee, Knoxville, TN, USA <sup>2</sup>Advanced Photon Source, Argonne National Laboratory, Argonne, IL, USA <sup>3</sup>Lawrence Livermore National Laboratory, Livermore, CA, USA <sup>4</sup>Y-12 National Security Complex, Oak Ridge, TN, USA

One of the key issues related to the microstructure quality of 3-D printed structural alloys is the presence of defects at the micron-length scale; therefore, understanding the relationship between processing parameters and defect characteristics is of fundamental and technical importance. Moreover, the role of microscopic defects, such as pores and cracks, on the fatigue behavior of the 3-D printed engineering component is a critical issue.

The main objective of the current study is to understand the role of volumetric energy density (VED) in the development of microstructure and defect characteristics during the selective laser melting (SLM) 3-D printing process using 316L stainless steel as a model system.

First, the key processing parameters of SLM (i.e., laser-beam power and scan speed) were varied systematically to change the VED input. Specifically, the laser power was varied from 380 to 140 W with a fixed scan speed of 300 mm/s, thus varying the VED from 200 to 75 J/mm<sup>3</sup>, to understand the effect of the energy input on the formation of microscopic defects. Second, the volume fraction and size distribution of the pores and microcracks, as well as their spatial distribution in the as-printed alloy components, were measured as a function of the VED using the synchrotron x-ray microtomography. Third, the significance of temperature at a given VED on defect formations was investigated by systematically varying both the laser power and speed. For example, the 3-D printing was performed at a higher processing temperature with a given VED under high-power/high-speed conditions, while another specimen was prepared at a much lower temperature under low-power/low-speed conditions with the same VED input allowing us to probe the role of processing temperature in defect formation in addition to the VED input. Finally, the elastic moduli of 3-D printed components with varying defect concentrations were evaluated to offer a quantitative understanding of the key processing variables in the formation of defects and their influence on the basic mechanical properties.

Email corresponding author: hchoo@utk.edu

## Control of residual stress and microstructure in large scale additive manufacturing

Ganguly S., Honnige J.R., Eimer E., Williams S., Colegrove P., Martina F.

#### Welding Engineering and Laser Processing Centre, Cranfield University, Bedford, MK43 0AL, UK

Wire plus arc additive manufacture (WAAM) allows high degree of design freedom in component geometry and material selection when compared to conventional manufacturing processes [1]. The main business drivers for this advanced manufacturing technology are reduction of cost through material saving and logistical simplicity [2,3]. Research in the Welding Engineering and Laser Processing Centre (WELP) of Cranfield University are focussed on developing this technology for critical structural and engineering components in different sectors e.g. Aerospace, defence and Energy. WAAM employs a filler wire and arc as a power source to manufacture components with the aid of a robot for manipulation to manufacture of components of different shapes and geometries. However, by the very nature of the process, a molten metal pool is solidified which results in dendritic grain structure. The deposited layers also undergo a large number of thermal cycles which would vary depending upon the position of a layer with respect to the finished component and the deposition strategy. The large number of thermal cycles would result in formation of a variably distributed residual stress state and associated distortion. Therefore, in order to employ this technique for manufacture of safety and service critical components, it is important to understand the effect of thermal cycling from successive deposition passes and residual stress formation to develop a robust process control and mitigation technique for WAAM process.

Cold rolling as a means for local mechanical tensioning can plastically deform a component to redistribute the locked-in residual stress state resulting in significant reduction of the large tensile stress component in the principal strain directions and associated distortion. Inter-pass rolling also introduces strain energy inside a component which during successive deposition would result in recrystallization of the rolled microstructure with the formation of a strain free microstructure in place of the dendritic grain structure. Experiments carried out in some of the main structural alloys e.g. Ti64, aluminium 2xxx series of alloy and ferritic steel, showed excellent response of local mechanical processing in refining the microstructure and improving mechanical properties [4,5]. The presented research showed that the application of cold rolling or other mechanical straining techniques could be an important tool in overcoming the qualitative limitations of WAAM technology, making it a robust process for application in critical applications across different sectors.

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Email corresponding author: s.ganguly@cranfield.ac.uk

## Exploiting the features of energy-dispersive diffraction for depth-resolved residual stress analysis: From lab to synchrotron and back

Genzel C., Apel D., Meixner M., Klaus M.

#### Helmholtz-Zentrum Berlin für Materialien und Energie, Germany.

Energy-dispersive (ED) diffraction can look back on a more than 100 years tradition which started in 1912 with the first diffraction experiment at all carried out by LAUE, FRIEDRICH and KNIPPING. In those early days the single crystal itself served as 'energy selector' for the incoming white X-ray beam, leading to the well-known LAUE spots on a photographic plate. The reason why it lasted more than 50 years before GIESSEN & GORDON [1] and BURAS *et al.* [2] in 1968 independently of each other used the white bremsstrahlung of an X-ray tube to investigate polycrystalline materials by ED diffraction, was the lack of appropriate multi-channel detector systems at that time, which are able to decompose the X-ray spectrum diffracted by the sample in a predefined direction with respect to the photon energy.

The first attempts to apply the ED method to X-ray residual stress analysis (XSA) were restricted to transmission experiments, thus exploiting only the high photon energies being required to penetrate thick samples. It took a further quarter of a century before RUPPERSBERG used the different absorption of the photons contributing to the diffraction lines  $E^{hkl}$  and applied his 'Universal plot method' [3] to evaluate residual stress depth profiles  $\sigma_{11}(\tau)$  and  $\sigma_{22}(\tau)$  from strain data measured in the ED mode of diffraction [4]. This pioneering work can be seen as the birthday of modern ED-XSA, since it combined the traditional  $\sin^2 \psi$ -method with the features of ED diffraction to develop advanced techniques for the depth-resolved analysis of steep near-surface residual stress gradients. Based on this work other XSA methods such as the 'multiple wavelength method' and the 'scattering vector method' that were originally developed for stress gradient analysis by means of angle-dispersive diffraction were transferred to the ED mode [5].

In spite of the progress made in developing advanced methods for ED residual stress analysis, it needs to be noted that their wider application is strongly related to the availability of modern 3<sup>rd</sup> generation synchrotron sources providing high brilliant radiation within a broad energy range. With EDDI@BESSY II the first ED synchrotron beamline especially designed for near-surface XSA experiments was established, which has been extensively used in the past to further develop new ED-XSA methods and to extend the range for their application in different fields of materials sciences [6].

However, since it has turned out in the recent past that the increasing demand from users and industry for beamtime for ED-XSA cannot be covered by the few synchrotron beamlines suited for such experiments, there is a trend to downscale them on an enhanced performance level to laboratory conditions. Based on studies showing that ex-situ ED-XSA performed under synchrotron and lab conditions yield comparable results [7], we developed a new type of ED 8-circle diffractometer equipped with two detectors for simultaneous data acquisition. In the lecture it will be shown that its flexible setup opens up ways for new measuring and evaluation strategies for non-destructive depth-resolved ED-XSA. The corresponding concepts aim at almost fully exploiting the features of ED diffraction to provide tools that allow the investigation of larger sample series being required, for example, in material development and process control.

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Email corresponding author: genzel@helmholtz-berlin.de

## *In-Situ* Neutron Diffraction Study of the Effect of Biaxial Stress on the Deformation of Zircaloy-4

Gharghouri M.A.<sup>1</sup>, McDonald D.<sup>1</sup>, Ham-Su R.<sup>1</sup>

<sup>1</sup>Canadian Nuclear Laboratories, Chalk River, Ontario, Canada

Data from uniaxial *in-situ* neutron diffraction experiments, which allow the grain-level mechanical response to be investigated quantitatively, have been used extensively to understand how the limited number of slip systems influences the distribution of stresses between different grain orientations in hexagonal-close-packed zirconium alloys [1-3]. However, the primary stress state in many nuclear reactor components (fuel cladding, pressure tubes, and pressure vessels) is biaxial tension. Thus, in the present work, *in-situ* neutron diffraction measurements in tubular specimens of cold-worked and stress-relieved Zircaloy-4 simultaneously pulled in tension and pressurized internally are used to measure the crystal-level response under biaxial tension. The starting material was solid bar stock, in which the basal poles are preferentially oriented in the radial direction of the bar, while the prism poles are preferentially aligned with the longitudinal (axial) direction. Proportional loading paths were applied, in which the biaxial ratio, defined as  $\sigma_{hoop}/\sigma_{axial}$ , remained constant during loading.

Lattice strains measured parallel to the hoop direction for biaxial ratios of 0 (simple tension) and 1 (balanced biaxial tension) are shown in Figures 1(a) and (b), respectively. Data for several grain families are shown, identified by the plane normal parallel to the hoop direction. In simple tension (Figure 1(a)), different grain families respond very similarly up to an equivalent applied stress of 300-350 MPa, beyond which 1) the 1010 and 2110 grain families rapidly accumulate tensile strain; 2) the 1012 grain family also accumulates tensile strain, but more slowly than the 1010 and 2110 grain families; 3) the strain in the 1011 grain family decreases, reaching almost zero at the end of loading; and 4) the lattice strain in the 0002 grain family decreases steadily with increasing applied stress, becoming compressive at the end of loading. Thus, the responses of the various grain families diverge significantly in simple tension because of the redistribution of stress due to the grain-level anisotropy of plastic deformation of HCP zirconium. In contrast, Figure 1(b) shows that, in balanced biaxial tension, all of the grain families respond very similarly, accumulating tensile strain throughout the test, though the 1010 and 2110 families accumulate strain more rapidly at higher equivalent applied stress.



Figure 1. Lattice strain along the hoop direction for various grain families for biaxial ratios of (a) 0 and (b) 1

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Email corresponding author: Michael.Gharghouri@cnl.ca

## The role of residual stress measurements in US Air Force aircraft sustainment programs

Langer K.<sup>1</sup>, Garcia W.<sup>2</sup>, Spradlin T.J.<sup>1</sup>, Kobryn P.A.<sup>1</sup>

<sup>1</sup>AFRL/RQVS, Wright-Patterson Air Force Base, OH 45433 US <sup>2</sup>878 AESG/EN, Wright-Patterson Air Force Base, OH 45433 US

Residual stresses (RS) in metallic aircraft structural components, whether a consequence of manufacturing or introduced deliberately via surface treatment, have received increasing attention in sustainment of US Air Force (USAF) systems. With the average aircraft age surpassing 25 years, exploiting beneficial compressive stresses to extend service lives, lower maintenance costs, and improve reliability can be an attractive alternative to part replacement. Understanding and managing the residual tensile stresses throughout the service life of the structure is also a key area of interest.

For USAF airframes, three primary considerations drive "lifing credit" to formally incorporate RS into aircraft structural integrity assessments: (1) quality assurance of the RS processing, (2) a validated fatigue crack growth prediction capability, and (3) validated processes for non-destructive inspection. In all of these areas, RS measurements are play key roles, and are used in multiple ways for various purposes and with differing levels of required fidelity.

In this paper, we discuss (with examples) the types of measurements used, how they are used (or could be used), and suggest areas in which further research would be beneficial. Some of the key roles include:

- Exploratory investigations to better understand observed fracture phenomena, such as atypical crack growth;
- Initial conditions for modelling the effects of subsequent surface treatment and usage;
- Validation of stress predictions from surface treatment models and ICME (integrated computational materials engineering) model;
- Verification that RS processing yields anticipated results;
- Inputs for updating lifing models during in-service usage.

Email corresponding author: kristina.langer@us.af.mil

## Neutron through thickness stress measurements in two-phase coatings with high spatial resolution

#### Luzin V.<sup>1</sup>, Fraser D.<sup>2</sup>

#### <sup>1</sup>Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW, 2232 Australia <sup>2</sup>Commonwealth Scientific and Industrial Research Organisation, Clayton, Melbourne, VIC, 3168, Australia

Residual stress profiling with high spatial resolution in coatings sprayed by one or another technique is useful but a difficult task for many neutron diffractometer. The newer generation of neutron diffractometers with higher performance are capable of reaching 0.1-0.2 mm resolution, but normally has been used for single-phase coatings. Stress measurements in the two-phase or multi-phase coating are an even more formidable experimental task due to the necessity of measuring all phases with lower volume fractions then a pure coating and the necessity to resolve d0 problem in a more complex way than the for single-phase coating systems. When addressed properly, macro- and micro-stress can be separated providing micro-mechanical characterisation which is related to various aspects of coating formation mechanisms and spraying parameters, therefore, allowing to study these mechanisms.

The results of through-thickness residual stress profiling neutron diffraction experiments are reported on the selected two-phase systems, metal-metal and metal-ceramic composites, deposited by cold spraying technique. With both phases measured and the help of additional information provided by neutron diffraction phase analysis and mechanical characterisation, the full stress state was reconstructed allowing interpretation of the experimental data in terms of micro-mechanical modelling.

Email corresponding author: vladimir.luzin@ansto.gov.au
## Application of in-situ experimental methods for revealing deformation mechanisms in advanced magnesium alloys

Máthis K.<sup>1,2</sup>

#### <sup>1</sup>Charles University, Faculty of Mathematics and Physics, Department of Physics of Materials, Ke Karlovu 5, 121 16 Prague, Czech Republic <sup>2</sup>Nuclear Physics Institute of the CAS, Řež 130, 250 68 Řež, Czech Republic

Owing to their hexagonal close packed (HCP) structure and c/a ratio close to ideal value the deformation behavior of magnesium alloys differ from the other, e.g. fcc of bcc metals. The basal slip has the lowest value of critical resolved shear stress (CRSS)(0001)( $11\overline{2}0$ ), followed by prismatic slip and first-order pyramidal slip systems. All these slips provides deformation in (a) (i.e. ( $11\overline{2}0$ )) direction and their combination provides only 4 independent slip systems. Therefore, the von Mises criterion requiring five independent slip systems for homogenous deformation is not fulfilled and activation of second-order pyramidal system or mechanical twinning is necessary.

The experimental study of the deformation mechanisms includes both ex-situ (e.g. optical light, scanning or transmission electron microscopy) and in-situ techniques (e.g. diffraction methods, acoustic emission etc.). The main drawback of microscopy methods in studying twinning and dislocations is the relatively small observed volume in the specimen. On contrary, the irradiated volume in the diffraction experiments provides statistically representative data. The X-ray line profile analysis, pioneered by Ungár et al. [1], was successfully used for ex-situ analysis of the temperature dependence of the dislocation structure evolution during uniaxial tensile test of magnesium alloys [2]. The neutron diffraction (ND) technique was used first by Gharghouri et al. for study of twinning in Mg-Al alloy [3]. In this type of experiment, the overall twinned volume can be determined from the intensity variations of particular peaks, caused by the crystal lattice reorientation during twinning [4,5]. Furthermore, the activity of a particular slip system manifests as a deviation of the lattice strains from the ideally elastic response [5]. Muránsky [6] introduced acoustic emission (AE) as a useful complementary experimental technique to ND during in-situ testing of the wrought Mg alloy, ZM20. The main advantages of the method are the high time resolution and sensitivity to twin nucleation and collective dislocation motion [7]. A recent statistical method worked out by Pomponi & Vinogradov [8] was successfully applied for the determination of the dominant deformation mechanisms from the AE signal at the various stages of deformation.

In the talk the influence of various microstructural (composition, LSPO phase orientation) and experimental (loading direction, temperature) parameters on the deformation mechanisms are elucidated using combined usage of AE and ND.

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Email corresponding author: mathis@met.mff.cuni.cz

## Neutron Diffraction Evaluation of Residual Stress in Friction Welded Thick Walled Power Industry Components

<u>Newby M.</u><sup>1</sup>, Doubell P.<sup>1</sup>, Hattingh D.G.<sup>2</sup>, Steuwer A.<sup>2,3</sup>, James M.N.<sup>2,4</sup>

<sup>1</sup>Eskom Holdings SOC Ltd, Lower Germiston Road, Rosherville, Johannesburg, South Africa <sup>2</sup>Nelson Mandela Metropolitan University, Port Elizabeth, South Africa <sup>3</sup>University of Malta, Msida MSD 2080, Malta <sup>4</sup>Plymouth University, Drake Circus, Plymouth, UK

Management of high temperature / high pressure components in the power industry is critical for both safety and economic reasons. During the past ten years Eskom, the main electricity service provider in South Africa, together with the Nelson Mandela Metropolitan University and the University of Plymouth (UK) have developed sampling and friction welding repair techniques for thick walled high integrity components. The applications for these techniques have been reported in several papers [1-3]. The consequence of failure after repair from these techniques has required a rigorous validation process which included volumetric residual stress analysis. This resulted in several experiments at the Institut Laue-Langevin (ILL) on samples up to 38mm in thickness.

The present paper will discuss an overview of the neutron diffraction experiments that have been done in this area with a focus on the experimental difficulties of measuring residual stress profiles with high spatial resolution in thick friction welded components, covering aspects such as collimator selection, sample setup and dzero sample techniques.



Figure 1: As welded residual stress contour plot of a friction welded 38mm thick steel sample

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Email corresponding author: newbym@eskom.co.za

# Synchrotron X-ray techniques for understanding Zirconium fuel cladding degradation

Preuss M.<sup>1</sup>, Frankel P.<sup>1</sup>, Da Fonseca J.Q.<sup>1</sup>

### <sup>1</sup>The University of Manchester, School of Materials, Materials Performance Centre, Manchester, UK

Zirconium alloys are the material of choice for fuel assemblies in water-cooled reactor due to a combination of low neutron cross-section, excellent corrosion performance and good mechanical properties. However, fuel cladding performance, or our ability to predict its performance, remains the limiting factor in an effort to push for increased fuel burnup, i.e. the energy extracted from a fuel assembly before it is removed from the core. This is becoming a particularly important challenge as nuclear power will have to remain commercially competitive with renewable energy and there is a demand to minimise the production of new nuclear waste. Further, new light-water reactors are currently under construction or in planning, making research in the field of Zirconium-based fuel assemblies particularly timely.

During my presentation I will focus on progress we have made in understanding the effect of alloying elements on aqueous corrosion performance, hydrogen pick-up and irradiation damage in Zr-alloys while also highlighting the many remaining gaps in our understanding. I will present results of detailed studies using a multiscale characterisation approach by employing synchrotron-based diffraction and scattering techniques and compare those findings with novel electron microscopy studies. These techniques have been employed to investigate in detail the oxide grown during autoclave testing or during in-reactor service, to characterise irradiation damage formed during accelerated proton irradiation to compare with neutron irradiated material and investigate microstructure evolution during loss of coolant accident (LOCA) conditions. While state-of-the-art characterisation tools now allow us to make new observations and rethink previously proposed mechanisms, it is also clear that more modelling efforts are required in the future to fully explain the experimental observations.

Email corresponding author: michael.preuss@manchester.ac.uk

## Microstructural analysis of cold-reduced carbon steel by scanning 3DXRD microscopy and crystal plasticity finite element analysis

Setoyama D.<sup>1</sup>, Hayashi Y.<sup>1</sup>, Seno Y.<sup>1</sup>

<sup>1</sup>Toyota Central R&D Labs., Inc.

It is of increasing importance to conduct micromechanical as well as microstructural three-dimensional analysis, not only in academic society but also in commercial enterprises. Many of the researchers in such companies need to investigate the microscopic mechanical behaviour of commercial-grade polycrystalline materials which usually consist of fine crystals.

We have developed a scanning 3DXRD apparatus [1] at the BL33XU Toyota Beamline at SPring-8[2,3]. In the past MECASENS', authors have reported a scanning three-dimensional x-ray diffraction (3DXRD) microscopy, which has enabled non-destructive 3D orientation mapping of polycrystalline materials [4,5]. Authors also have demonstrated the corresponding crystal plasticity finite element analysis to discuss the inter-granular and intra-granular micromechanical interaction [6]. In this study, we have analysed microscopic mechanical distributions of cold-reduced carbon steel (JIS:G3141 or ISO:3574), by means of the scanning 3DXRD microscopy with finer spatial resolution than our past setup. The typical cold-reduced carbon steel has a mean grain size of about 20  $\mu$ m. We therefore applied a few  $\mu$ m focused synchrotron x-ray beam to overcome the overlap of diffraction spots as well as to obtain a few  $\mu$ m spatial resolution. The corresponding crystal plasticity finite element analysis was also performed to discuss the mechanical behaviour of such material from a microscopic point of view.

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Email corresponding author: daigo @mosk.tytlabs.co.jp

## Internal stresses, strains and phase fractions surrounding martensite band front in superelastic NiTi wire loaded in tension

Sittner P.<sup>1,2</sup>, Sedmak P.<sup>2,3</sup>, Wright J.<sup>4</sup>, Heller L.<sup>1,2</sup>, Sedlak P.<sup>1,5</sup>, Frost M.<sup>1,5</sup>

<sup>1</sup>Nuclear Physics of the CAS, Rez, Czech Republic <sup>2</sup>Institute of Physics of the CAS, Na Slovance 1992/2, 1822 Prague, Czech Republic <sup>3</sup>FNSPE CTU, Trojanova 13, 12000 Prague, Czech Republic. <sup>4</sup>European Synchrotron Radiation Facility, 71 Avenue des Martyrs, 38043 Grenoble, France. <sup>5</sup>Institute of Thermomechanics of the CAS, Dolejškova 1402/5, 18200 Prague, Czech Republic.

Macroscopic interfaces between the deformed and undeformed material appearing on the front of deformation bands propagating in solids (Luders bands) always attracted attention of mechanical engineers as well as theoreticians involved in modelling of material deformation. As the strain compatibility has to be maintained at these highly mobile interfaces extending over thousands of polycrystal grains, there have to be sharp gradients of stresses and strains. Experimental analysis is difficult since the macroscopic interface is a 3D object buried in the bulk which disappears on unloading, or at least the stress field around it dramatically changes, as for example in the case of the martensite band front propagating during superelastic tensile deformation of NiTi shape memory alloy wire [1,2]. The stress state in the material around the interface matters - the interface cannot exist without it. Looking from outside by the DIC method [3,4], the martensite band front in stretched NiTi wire appears to be rather broad and perpendicular to the wire axis. Since conventional microscopic methods could not be employed to investigate this buried macroscopic interface, its nature remained relatively unknown for decades.

In this talk, I will present the results of the detailed analysis of grain resolved internal strains, stresses and phase fractions around the martensite band front propagating in 0.1 mm thin NiTi wire we managed to obtain through the analysis of 3D x-ray diffraction /3D-XRD/ experiment at the ID11 beamline [4]. Particularly, we were able to determine full strain and stress tensors in ~15000 polycrystal grains of defined position, size and crystal lattice orientation (mean grain size 5.9  $\mu$ m) within the martensite band front stabilized by applied tensile stress of ~420 MPa [4]. To derive macroscopic internal stress state in the wire, we performed a scale transitions towards continuum. In this way, we were able to resolve macroscopic internal stress fields surrounding the front with 1 $\mu$ m spatial resolution and 20MPa stress resolution.

It was found that the martensite band front has a shape of a buried nose cone surrounded by internal stress field. The local stresses in grains ahead of the advancing front redistribute in such a way that the grains located at the interface experience ~200 MPa higher effective stresses compared to the grains located far from the interface. Consequently, those grains transform collectively, while very little is happening elsewhere in the wire. The superelastic deformation of NiTi wire was also simulated by a finite element implemented constitutive SMA model adapted for nonlocal effects [5]. The moving martensite band front was reconstructed [4] confirming its cone shape as well as the sharp internal stress gradient around it. For related videos and x-ray data for downloading see http://ofm.fzu.cz/.

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Email corresponding author: sittner@fzu.cz

## Diffraction plane dependence of micro residual strain by plastic deformation

Suzuki K.<sup>1</sup>, Shobu T.<sup>2</sup>

<sup>1</sup> Faculty of Education, Niigata University, Igarashi-2-no-cho, Nishi-ku, Niigata, 953-0035, Japan <sup>2</sup>Material Sciences Research Center, JAEA, Kouto, Sayo-cho, Sayo-gun, Hyogo, 679-5148, Japan

An austenitic stainless steel has an elastic anisotropy. To investigate the lattice plane dependence of the intergranular stress due to plastic deformation, the residual stresses were measured by four diffraction planes. We investigated the behaviour of the intergranular strains with the change in the azimuth angle  $\chi$ .

The test specimen used in this study was a plate with a thickness of 3 mm and a width of 15 mm. The material of the specimen was austenitic stainless steel (SUS316L). The uniaxial tensile deformation was applied to the specimen with a strain rate of  $6.67 \times 10^{-2}$  s<sup>-1</sup>. The measured plastic strain of the specimen was 2.8%.

The residual stresses of the specimen was measured using synchrotron X-rays with 66.40 keV at the beam line BL22XU in SPring-8. We used a

with 60.40 keV at the beam line BL22XO in SFIIng-8. We used a  $\cos^2 \chi$  method as the stress measurement method. The schematic drawing of the  $\cos^2 \chi$  method is shown in Figure 1 [1]. The diffraction is detected in transmission as shown in the figure. The stress value can be determined by a gradient of the  $2\theta - \cos^2 \chi$  diagram. The values of  $\cos^2 \chi$  were from 0 to 1. The used diffraction plane were shown in Figure 2.



Figure 1. Schematic drawing of  $\cos^2 \chi$  method.



Figure 2.  $2\theta$ -cos<sup>2</sup> $\chi$  diagrams.

While the azimuth  $\chi$  was tilted, the lattice spacing was measured using the 400, 331, 440 and 620 diffraction planes at the beam line BL02B1 in SPring-8. The X-ray energy was 72.312 keV, the dimensions of the divergent slit were 0.2×5 mm and the receiving slit



was a Soller slit.

The measured  $2\theta \cdot \cos^2 \chi$  diagrams are shown in Figure 2. Because the specimen is unloaded, the relation between  $2\theta$  and  $\cos^2 \chi$  must indicate a horizontal straight line, but it shows a

curve. For the lattice plane with a low atomic density, the diagram indicates an upward warp. For the lattice plane with a high density, the diagram indicates a downward warp. This is caused by intergranular strains. Figure 3 shows the changes in the intergranular stresses for 400, 331, 440 and 620 with the change in  $\chi$ -angle. In the direction of a principal stress, the lattice planes with a low atomic density have a tension, and those with a high atomic density have a compression. In the direction where shearing deformation is dominant such as  $\chi$ =45° or 135°, the above relation reverses. The behaviour of the intergranular stress is dependent on the interaction between the lattice planes. The intergranular strain influences the X-ray stress measurement, because it is based on the change of the strain with the change of a scattering vector.

Figure 3. Residual stresses.

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Email corresponding author: suzuki@ed.niigata-u.ac.jp

## Kinetics of twin boundary motion in ferromagnetic shape memory alloys

Pramanick A.<sup>1</sup>, <u>Wang X.-L.<sup>1</sup></u>

<sup>1</sup>Department of Physics and Materials Science, City University of Hong Kong 83 Tat Chee Ave., Kowloon, Hong Kong

In-situ neutron and synchrotron studies have played an important role in elucidating microscopic insights of deformation behaviors at multi length scales. We have recently applied the techniques to study ferromagnetic shape memory alloys (FSMAs), which have shown tremendous potential for applications of next-generation smart devices due to their exceptionally large magnetic-field-induced strains. Unlike in conventional shape memory alloys, in which a martensitic phase transformation occurs, in FSMA the shape memory effect is driven by twin boundary motion of the martensitic phase, leading to reorientation of the crystallographic twin variants. Using time-resolved neutron and synchrotron diffraction, we show that the twin reorientation under a magnetic field is described by thermally-activated creep motion over a distribution of energy barriers. The dynamical creep exponent  $\mu$  was found to be ~ 0.5, suggesting that the energy distribution is due to short-range disorders.

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Email corresponding author: xlwang@cityu.edu.hk

## Practical demonstration of Bragg-edge neutron transmission strain tomography

Wensrich C.M.<sup>1</sup>, Hendriks J.N.<sup>1</sup>, Gregg A.W.T.<sup>1</sup>, Shinohara T.<sup>2</sup>, Tremsin A.S.<sup>3</sup>, Meylan M.H.<sup>4</sup>, Kisi E.<sup>1</sup>, Luzin V.<sup>5</sup>, Kirstein O.<sup>6</sup>

<sup>1</sup>School of Engineering, University of Newcastle, Australia.
<sup>2</sup>Japan Proton Accelerator Research Complex (J-PARC), JAEA, Japan.
<sup>3</sup>Space Sciences Laboratory, University of California, USA.
<sup>4</sup>School of Mathematical and Physical Sciences, University of Newcastle, Australia.
<sup>5</sup>Australian Centre for Neutron Scattering, ANSTO, Australia.
<sup>6</sup>European Spallation Source, Sweden.

The rapid development of energy resolved imaging for neutrons has given prominence to Bragg-edge transmission geometry as a technique for strain imaging. Strain imaging raises the prospect of strain tomography and numerous studies have focused on solving the resulting inverse problem over the past decade. It is now accepted that the problem is not directly solvable; multiple distinct strain fields can project to the same set of strain images [1,2]. Fortunately, this does not eliminate the possibility of reconstruction if the mechanics of the system are considered; only one possible solution is physical. In a recent paper [3], the Authors have outlined a numerical approach for reconstructing strain fields that result from *in situ* loads (i.e. in the absence of residual strain). This algorithm relied upon the link between Bragg-edge strain measurements and the deformation of the boundary of the sample for this class of systems.

In this paper, the results of a recent physical experiment demonstrating this algorithm are presented (see Figure 1). This involved the reconstruction of a 2D strain field in a small C-shaped steel sample under a 7kN applied load from a total of 86 Bragg-edge strain projections made using the RADEN energy resolved imaging instrument (J-PARC) and a MicroChannel Plate detector. This experiment represents the first ever practical example of Bragg-edge neutron transmission strain tomography for a non-axisymmetric system.



Figure 1. The reconstructed strain field compared to an FEA simulation and results from DIC.

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Email corresponding author: Christopher.Wensrich@newcastle.edu.au

# Three-dimensional full-tensor mapping of a residual stress field produced by high-pressure rolling

Coules H.<sup>1</sup>, Horne G.<sup>1</sup>, Kabra S.<sup>2</sup>, Colegrove P.<sup>3</sup>

<sup>1</sup>Department of Mechanical Engineering, University of Bristol, Bristol, BS8 1TR, UK <sup>2</sup>ISIS Facility, STFC Rutherford Appleton Laboratory, Didcot, Oxfordshire, OX11 0QX, UK <sup>3</sup>School of Applied Sciences, Cranfield University, Cranfield, Bedfordshire, MK43 0AL, UK

Localised high-pressure rolling can be used to reduce the residual stresses which occur in welded joints [1]. Previous studies [2,3] have shown that when applied as a post-weld treatment, this technique can radically reduce the magnitude of residual stress. However, all experimental work so far has focussed on relatively thin welds, with a thickness of < 10 mm and it is unclear what depth of effect the rolling process can have in thick-section welds.

To investigate the depth effect of rolling, we performed extensive characterisation of a residual stress field introduced by rolling a simple unwelded thick plate of aluminium alloy 6082-T6 [4]. Neutron diffraction measurements were made using the ENGIN-X time-of-flight at ISIS Pulsed Neutron Source. Nine different scattering directions at each measurement location, which enabled calculation of the complete stress tensor at each point and analysis of the measurement uncertainty. The data were used in combination with FE analysis of the rolling process itself to show that during rolling, plastic deformation had occurred through the entire 25 mm thickness of the specimen. This indicates that it would be possible to use rolling for stress relief of much thicker welds than has previously been attempted.



Specimen Goniometer Figure 1. Measurement setup on the ENGIN-X beamline, showing Cybaman goniometer



Figure 2. Residual shear stress  $\tau_{yz}$  on size planes along the length of the rolled specimen.

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Email corresponding author: harry.coules@bristol.ac.uk

## In situ Diagnostics of Melting/Solidification and Segregation during Crystal Growth by Energy-resolved and Conventional Neutron Imaging

Vogel S.C.<sup>1</sup>, Tremsin A.S.<sup>2</sup>, Perrodin D.<sup>3</sup>, Losko A.S.<sup>1</sup>, Bizarri G.<sup>3</sup>, Bourret-Courchesne E.<sup>3</sup>

<sup>1</sup> Los Alamos National Laboratory, Los Alamos, NM, United States <sup>2</sup>University of California at Berkeley, Berkeley, CA, United States <sup>3</sup>Lawrence Berkeley National Laboratory, Berkeley, CA, United States

Multiple novel scintillation materials have been discovered in the past decade with only few of them available for production. The high cost, low yield and inconsistency of crystal growth are among the main obstacles for these materials to become widely available. In-situ diagnostics of crystal growth can be very helpful for the optimization of crystal growth procedures. Neutrons appear to be unique probes providing information on distribution of elements, location of liquid/solid interface, temperature and possibly internal stresses. The initial proof-of-principle experiments conducted both at pulsed and continuous neutron sources demonstrate the possibility to visualize in real time (in crystal growth terms) the dynamics of the liquid/solid interface and segregation of various elements, which have sufficient neutron attenuation cross section. The concentration of doping materials (such as Eu) can be mapped even for the compounds with only 0.1% doping. The thermal contraction of scintillator material during cooling stage can also be observed and possibly correlated to sample cracking. The unambiguous distribution of some elements within samples can be obtained through neutron resonance absorption. We have confirmed that it is the segregation of Eu which provided the contrast in the images acquired at a continuous source.

In our experiments we studied in-situ the distribution of the europium activator within a BaBrCl:Eu (0.1% to 5% nominal doping concentrations per mole) scintillator as well as several other materials during melting and solidification processes. The strong tendency of Eu dopant to segregate during the solidification process is observed in repeated cycles, with Eu forming clusters of multiple length scales (only for the clusters larger than ~50  $\mu$ m as limited by the resolution of our experiments). It is also demonstrated that the dopant concentration can be quantified even for very low concentration levels (~ 0.1%) in 10 mm-thick samples. The optimization of crystal growth procedures can lead to a substantial improvement of resulting crystal quality and yield. Crucial to this optimization is the possibility to obtain real-time characteristics of crystal growth processes, which energy resolved neutron imaging can provide.

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Email corresponding author: sven@lanl.gov

## Stress partitioning and phase behavior in a pearlitic steel studied using high energy X-ray synchrotron radiation

Braham C.<sup>1</sup>, Baczmański A.<sup>2</sup>, Gonzalez G.<sup>3</sup>, Sidhom H.<sup>4</sup>, Gadalińska E.<sup>5</sup>, Wroński S.<sup>2</sup>, Buslaps T.<sup>6</sup>

 <sup>1</sup>Laboratoire Procédés et Ingénierie en Mécanique et Matériaux (PIMM, CNRS UMR 8006), Arts et Métiers-ParisTech, 151 Bd de l'Hôpital, 75013 Paris, France.
<sup>2</sup>AGH-University of Science and Technology, Faculty of Physics and Applied Computer Science, Al. Mickiewicza 30, 30-059 Kraków, Poland.
<sup>3</sup>Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, Circuito exterior S/N, Cd. Universitaria, A.P. 70-360, Coyoacán, C.P. 04510, Mexico.
<sup>4</sup>Mechanical, Materials and Processes Laboratory (LR99ES05), ESSTT, 5, Avenue Taha Hussein 1008, University of Tunis, Tunisia.
<sup>5</sup>Institute of Aviation, NET Institute, al. Krakowska 110/114, 02-256 Warszawa, Poland <sup>6</sup>ESRF, 6, rue J. Horowitz, 38500 Grenoble Cedex, France

The role of the interlamellar space in the mechanical behavior and fatigue resistance of pearlitic steels has been studied in a previous work [1]. In-situ diffraction technique during mechanical loading is a powerful method to investigate the mechanical behavior of the phases and the stress partitioning between the cementite and the ferrite [1-4].

In this study, the evolution of the phase stresses of a 0.68 wt%C pearlitic steel (specially designed and heat treaded for this study) is analyzed by synchrotron diffraction during uniaxial tensile loading, at room temperature. The diffraction measurements were done at ESRF beamline ID15B (Grenoble, France). Two microstructures, obtained by two different heat treatments and differing mainly by their inter-lamellar spaces, are compared. In the both cases, the microstructures correspond to full pearlitic steel with a cementite volume fraction of about 12%.

The results show a clear effect of elastic and plastic anisotropy in the both phases and higher stresses were observed in the case of the smaller interlamellar space. For the interpretation of the diffraction data, different models are compared. In elastic range and for small plastic deformation, the self-consistent model presents the best agreement with the experimental data. For large plastic deformation, this model does not predict correctly the stress partitioning between the phases as well as the macro behavior of the studied steel. Therefore a mixture model was used to better take into account the interaction between the phases. Using this model, the second order stresses were studied and their effect on the diffraction peak broadening was analyzed.

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Email corresponding author: chedly.braham@ensam.eu

## \*Internal Mechanics of Parallel Wire Cable Strands

Brügger A.<sup>1</sup>, Lee S.-Y.<sup>2</sup>, Betti R.<sup>1</sup>, Noyan I.C.<sup>2</sup>

<sup>1</sup>Columbia University, Dept. of Civil Engineering and Engineering Mechanics, 500 West 120<sup>th</sup> St., MC4709, 610 S.W. Mudd, New York, NY, 10027, USA <sup>2</sup>Columbia University, Dept. of Applied Physics and Applied Mathematics, 500 West 120<sup>th</sup> St., MC4701, 1120 S.W. Mudd, New York, NY, 10027, USA

The internal mechanics of suspension bridge cables have been our focus of study in recent years, most notably the quantification of residual strength of an aged, corroded cable. Over recent years, we have developed a methodology to accurately map the internal strains of reduced 7-wire parallel strands under various boundary and load conditions using neutron diffraction at both LANL SMARTS and ORNL VULCAN engineering materials diffractometers [1,2]. Particular attention was given to the accurate quantification of the development length, the distance over which a broken wire regains full service stress purely due to interaction with neighboring wires. The results from these studies have increased the fidelity of various numerical cable collapse models, resulting in more accurate assessments of the remaining strength of large-scale suspension bridges.

After quantifying successfully the development length in 7-wire specimens as a function of friction, mechanical interference, and most centrally the amount of confinement force inside a cable, it became readily apparent that the confinement force distribution itself is not well defined for parallel wire cables and merits detailed study. Due to a paucity of experimental data in this area, the structural engineering community indulges in broad assumptions regarding the internal stress distribution of parallel wire strands. For example, prescribed mean-field stress distributions initially developed to define the mechanics of elastic solids are often used to approximate the internal mechanics of parallel wire cables. Quite obviously, the amount of clamping on a wire strand and resultant confinement forces present in each wire is a major factor in the development length and therefore a bridge cable's ability to maintain its structural stability in the presence of wire breaks. We aim to shed light on this critical topic by reporting the first direct measurements of strains within individual wires of up-scaled parallel wire strands under various tensile loads and confinement conditions.

Neutron diffraction is used to measure the elastic strains along the three orthogonal axes across the strand cross-section underneath the clamp for all of the individual wires at various clamping loads while the strand is under tension. The results reveal that, while for all clamping loads, the confinement strains within individual wires are heterogeneously distributed; increasing the clamping force significantly decreases the strain heterogeneity. The experimental results are also compared to a companion finite element model and various simplifying assumptions tested. Our findings are in agreement with the hypothesis by Gjelsvik, which states that, within a parallel wire bridge cable, local variations in wire diameter due to manufacturing tolerances can lead to large wire-to-wire variations in clamping constraint [3].

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Email corresponding author: brugger@civil.columbia.edu

## In Situ Neutron Transmission Bragg Edge Measurement of Strain Fields Near Fatigue Cracks Grown in Hydrogen

Connolly M.J.<sup>1</sup>, Bradley P.E.<sup>1</sup>, O'Connor D.T.<sup>2</sup>, Slifka A.J.<sup>1</sup>, Drexler E.S.<sup>1</sup>

<sup>1</sup>National Institute of Standards and Technology, 325 Broadway, Boulder CO 80305 <sup>2</sup>Cold Regions Research and Engineering Laboratory, 72 Lyme Road, Hanover, NH 03755

Hydrogen is desirable for energy storage as it is cleaner burning and can store a larger amount of energy than an equal mass of gasoline. One problem related to the development of a hydrogen economy is to find or develop materials that ensure the safe, reliable, and cost-effective flow of energy from the source to the user. It is expected high-strength steels will be needed to serve this function. However, the existing network of natural gas pipeline, for example, is constructed of ferrous materials which are susceptible to embrittlement and subsequent increased fatigue crack growth rates (FCGR) during exposure to hydrogen [1]. Proposed mechanisms of hydrogen embrittlement include hydrogen-enhanced decohesion (HEDE) and the hydrogenenhanced localized plasticity (HELP) mechanisms. In the HEDE mechanism, accumulation of hydrogen at locations of high triaxial stresses lead to the weakening of Fe-Fe bonds once the hydrogen gas creates areas of extended dislocations in the Fe lattice and enhances dislocation mobility in the steel framework.

Quantifying stress and strain fields from fatigue cracking is crucial to the determination of the underlying mechanism behind hydrogen embrittlement and to the study of hydrogen embrittlement and hydrogen-assisted FCGR. Because of the fast diffusion of H2 from the steels, the effect of hydrogen embrittlement presents only during exposure, and steel specimens will exhibit in-air fatigue crack growth rates shortly after being removed from a hydrogen environment (~ 45 min.). Thus, any measurements designed to study hydrogen embrittlement and hydrogen-enhanced fatigue crack growth must be performed in situ.

We performed neutron transmission Bragg-edge measurements to characterize the strain fields near cracks that were grown both in air and in hydrogen. Through the use of a novel chamber uniquely compatible with neutron scattering, the measured cracks were grown *in situ* during the neutron scattering measurements [2]. An enhancement in the magnitude and spatial extent of the strain field near the crack grown in hydrogen compared to near the crack grown in air was observed. We compare the strain fields to predictions from finite element, and discuss the differences between the measured in-air and in-H<sub>2</sub> crack-tip strain fields in the context of the HELP and HEDE mechanisms.



Figure 1: Measured strain fields for the cracks grown in air (left) and in H2 (right). The strain direction measured is in the direction of the throughthickness of the CT specimen. An enhancement in the magnitude of the strain field near the fatigue crack grown in hydrogen of  $\sim$ 1.7x the strain field near the crack grown in air was observed. This is consistent with the HEDE mechanism.

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Email corresponding author: matthew.connolly@nist.gov

## \*Stress Evaluation of Adhesively Bonded Lap Joints with Aluminium 2024-T3 Adherents Using FEA

Khodja M.<sup>1,2</sup>, Ahmed A.<sup>1</sup>, Hamida F.<sup>1</sup>, Corderley G.<sup>2</sup>, Govender S.<sup>2</sup>

<sup>1</sup> LMPM, Mechanics and physics of materials Laboratory, Mechanical Engineering Department, University of Djillali Liabes, Sidi Bel Abbes, BP 98 Cité ben M'Hidi Sidi Bel Abbes 22000, Algeria <sup>2</sup>CSIR Materials Science and Manufacturing, Meiring Naude Road, Pretoria, 0184, South Africa

Several factors need to be considered when designing adhesive joints, notably the peak stresses at the ends of the overlap area and the stresses due to bending moments. It is necessary to be able to determine the state of stress within the joint during the design process in order to achieve the correct joint strength. This need necessitated the study of the stress state in a simple adhesive lap joint based on the analytical theories of Volkersen [1] and Goland & Riesener [2]. In the case of more complex geometrical configurations, it is impossible to analytically describe the stress field within the adhesive and it is then necessary to use a Finite Element Analysis (FEA). After the analytical results were compared with FEA results using Abaqus [3] and validated, the general results show that the analytical and FEA results were in good agreement and proved suitable for single lap joints [4]. The study presented in this covers the behaviour of adhesivel ybonded joints as predicted by FEA. Abaqus has been utilised to investigate the stress distribution along the adhesive layer of three different joint types (simple-lap joint, single-step-lap joint and single-step-lap joint with release at the edges of the bond area as shown in Figure 1. (b)), while under static tensile loading.

The objective of this study is to develop a numerical approach which will lead to a method to aid in the design of bonded assemblies. The AA2024-T3 aluminium alloy was used as adherent for this study with Adekit-140 as the adhesive. The overlapping surfaces of the adherent and the adhesive were modelled with 3D models that were based on surface-to-surface contact elements. Analyses were performed where the length and the thickness of overlap were fixed, keeping the bonding area the same for all geometries. The analysis focused on the central stress of the adhesive joint along line [A-B] in the lap joints modelled as shown in Figure.1 (a).

It was noted that there is symmetry in the stress distribution about the middle of adhesive joint layer according to the length of overlap region. The maximum stresses were at the edge of the bond. Observations have been made on peel and shear stresses in the adhesive layer.



Figure 1. (a)The Equivalent stress Von Mises distribution in the adhesive layer, (b) The stress concentration in the region of geometric discontinuity in different lap joints.

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Email corresponding author: khodja.malika7@gmail.com, MKhodja@csir.co.za.

## Micromechanical behavior in additively manufactured textured Inconel 718

Xie Q., Stoica A., Gorti S., Radhakrishnan B., Song G., Kirka M., Dehoff R., Bilheux J., Bilheux H., An K.

Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA

Additively manufacturing process induces solidification texture, which impacts structural integrity in terms of deformation anisotropy, residual stress, fatigue endurance and reliability etc. Using time-of-flight (TOF) insitu neutron diffractions, we evaluated the micromechanics behavior of an Inconel 718 alloy with strong columnar grain structure and cube texture prepared via electron beam additive manufacturing, and the hkl specific lattice strain behaviors are measured with crystallographic planes normal either parallel or perpendicular to the loading direction. Considering the initial texture, crystal elastic-plastic finite element modeling (EPFEM) framework was used to simulate the lattice strain and grain reorientation evolution. Here we tested the isotropic and the anisotropic work hardening models and implemented them using the Voce law to understand their predictions and interpretations of the plastic anisotropic behavior. Different kinds of dislocation interactions were taken into account for the anisotropic work hardening. The micromechanics behavior of the texture Inconel 718 and the quality of the predictions of lattice strain behaviors and the impact of initial texture will be discussed in detail. The correlation between the identified coefficients of the work hardening law and the micromechanical behavior will be illustrated.

Email corresponding author: kean@ornl.gov

## \*Calibration of Numerical Model for Shot Peening Simulation: Strain hardening measurement approach

Edward A.B.<sup>1</sup>, Heyns P.S.<sup>2</sup>, Pietra F.<sup>3</sup>

<sup>1,2</sup>Centre for Asset Integrity Management, Mechanical & Aeronautical Engineering Department, University of Pretoria.

<sup>3</sup>Merenco Helicopter, South Africa.

Shot peening is an impact cold working technique for fatigue lifting in critical load bearing components and for forming of thin shaped curved surfaces. Specifically, compressive residual stresses (CRS) via shot peening has been beneficial in its application to turbomachinery blade roots as a means of mitigating blade failures that emanate from fatigue, stress corrosion cracking and corrosion fatigue [1]. Modelling the response of these materials to shot peening for the optimization of its process parameters had been a long time engagement by the research community involved in the design, manufacturing and operation of turbines. The use of a recently developed DEM-FEM model [2] by the authors in this report, for a simplified and realistic approach to enhancing shot peening optimization will pave way for a greater confidence in its application. This work is therefore an improvement on the developed DEM-FEM model via:

- i. Strain hardening measurement of the top surface cold worked layer due to shot peening, using electron backscatter diffraction techniques (EBSD)
- ii. Calibration of the developed model with the EBSD measured data.
- iii. Validating results obtained from simulations making use of the calibrated model in single and double sided shot peening (figure 1) of turbine blade materials (12% martensitic steel and AISI 310 Stainless steel), with standard experimental results.

Our numerical results clarifies the correlation of the compressed depth with peening parameters which gives a quantitative understanding of the peening effects and measured results.



Figure 1. Shot peening model showing double sided shooting of sample

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Email corresponding author: britedy2@yahoo.com

## Force blocking by a shear defect in a 2-D granular system

Kisi E.<sup>1</sup>, Wensrich C.M.<sup>1</sup>, Luzin V.<sup>2</sup>, Kirstein O.<sup>3</sup>

<sup>1</sup>School of Engineering, the University of Newcastle, Callaghan NSW 2308 AUSTRALIA <sup>2</sup>Australian Centre for Neutron Scattering, ANSTO, Sydney AUSTRALIA <sup>3</sup>European Spallation Source, Lund SWEDEN

Recently, neutron strain scanning was adapted to fine granular systems to investigate the stress distribution in powder compacts within dies under in situ loads. Granular materials such as iron, copper or quartz powders were studied in various shaped dies (e.g. [1]). The behaviour of all macroscopically continuous systems is due to the sum of the behaviour of a multitude of individual entities, in this case particles. In monodisperse systems, it has been widely observed using low load photo-elastic experiments that the load is carried by relatively few particles in well-defined *force chain*. In an extension of our continuum work, neutron tomography was combined with neutron strain scanning to prove that force chains persist at much higher loads in a 3-dimensional assembly of 3mm steel spheres [2]. By reducing the system to two dimensions, it has now been possible to explore the force chains at a number of loads and ball configurations and those results are summarised in the present work. Force chains were created by loading 576 3mm steel balls into a slot die 50 mm wide x 120mm high and compressing to loads of 2.5, 5, 10, 20 and 30 kN. At each load, neutron radiography established the positions of the balls. Neutron diffraction was recorded from the iron *211* peak. Using 3mm slits to isolate each ball, strains were measured in axial, normal and transverse directions as well as at 45° to the large face of the die from which the average stress tensor within each ball was evaluated.

Figure 1 shows the stress distribution within the ball ensemble at 5 and 30kN. Note that a large shear or twinlike defect runs diagonally from upper right to lower left. The remarkable ability of the defect to trap the load in the upper region of the ball assembly can be clearly seen and is reinforced by digital image correlation analysis of the corresponding radiographs (Figure 1c). Analysis of stress distribution at the different loads and also in a ball assembly with no defect gives insight into the role and eventual breakdown of force chains in the system. A multifractal analysis has highlighted the effect of increasing load in expanding the load-sharing region around each ball; the end point of which would be a uniform continuous stress distribution.



Figure 1. Stress distribution and force chains in a 2-D ball array at a) 2.5kN and b) 20kN load; and c) total displacement at 20kN. The stress scale for (a) and (b) is shown at the right of (b).

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Email corresponding author: erich.kisi@newcastle.edu.au

## Investigating stresses developed during mechanical forming of steel through Finite Element Analysis

Akinlabi S.A.<sup>1</sup>, Fatoba O.S.<sup>2</sup>, Akinlabi E.T.<sup>2</sup>

<sup>1</sup>Department of Mechanical and Industrial Engineering Technology, University of Johannesburg, South Africa.

<sup>2</sup>Department of Mechanical Engineering Science, University of Johannesburg, South Africa.

Stresses majorly affect the mechanical properties of materials. However, structural failures are often caused by the combined effect of residual stresses and applied stresses. It is practically impossible for a manufactured component to be entirely free of residual stresses because these stresses developed during the manufacturing process and certain amount remain in the component even after the process is completed. This study reports the findings of the investigation into the developed stresses during mechanical forming of the steel sheet. The result revealed that the Von Mises stresses developed and increases during the neutral axis as the punch strokes increases. Lastly, it was observed that the locked in stresses in the material after the process were tensile in nature and such are not beneficiary to the structural integrity of the manufactured component even though an average value of 0.057540 MPa was recorded for this study at the bend radius along the neutral plane.

Email corresponding author: stephenakinlabi@gmail.com

## \*In-situ neutron diffraction deformation behavior of CrMnFeCoNi high-entropy alloy at low temperature

Naeem M.<sup>1</sup>, Wang B.<sup>1</sup>, Harjo S.<sup>2</sup>, Lan S.<sup>1</sup>, He H.Y.<sup>1</sup>, Wu Z.D.<sup>1</sup>, Lu Z.P.<sup>3</sup>, Wang X.-L.<sup>1,\*</sup>

<sup>1</sup>Department of Physics and Materials Science, City University of Hong Kong, Kowloon, Hong Kong <sup>2</sup>J-PARC Center, Japan Atomic Energy Agency, Tokai, Ibaraki, Japan <sup>3</sup>State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, China

High-entropy alloys (HEAs), which consist of five or more alloying elements in equal molar ratios, are a new class of structural materials. Despite the complex chemistry, HEAs can form a single phase solid solution with an incredibly simple lattice, FCC or BCC structure. Equi-atomic CrMnFeCoNi is a model high-entropy alloy with single phase FCC structure depicting good strength and ductility and has been widely explored for its mechanical behavior at room temperature, and at elevated and cryogenic temperatures. It has been shown that at room temperature, this HEA behaves in an anisotropic way similar to that of FCC metals and plastic deformation occurs by the operation of mixed dislocations [1]. With lowering temperature, enhancement in yield and tensile strength is observed. However, surprisingly, its ductility also increased thus making it a superior material for future cryogenic applications [2]. Twinning has been proposed as an additional deformation mode, similar to twinning induced plasticity in steels for this unusual behavior [2-4]. Through an in-situ study at low temperature, we explored the deformation behavior of CrMnFeCoNi HEA under tensile loading by neutron diffraction. Evolutions of lattice strain and orientation will be presented and their relations to different deformation mechanisms will be discussed, which are responsible for outstanding mechanical behavior of this alloy.

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\*E-mail of corresponding author: xlwang@cityu.edu.hk

## \*Qualitative Mapping of Axial Plastic Strain for a Roller Bearing undergoing Overloads using Bragg Edge Parameter Fitting

Reid A.<sup>1</sup>, Kabra S.<sup>2</sup>, Marshall, M.<sup>1</sup>, Martinez, I.<sup>1</sup>, Mostafavi, M.<sup>3</sup>

<sup>1</sup>Mechanical Engineering Department, University of Sheffield, Sheffield, S10 2TN <sup>2</sup> Rutherford Appleton Laboratory, ISIS Facility, Chilton, OX11 0QX, UK <sup>3</sup> Mechanical Engineering Department, University of Bristol, BS8 1TH

X-ray and neutron diffraction techniques are established methods for non-destructively measuring elastic strain within bulk materials. Recent developments have allowed for analysis of time-of-flight transmission spectra collected by a microchannel plate (MCP) detector demonstrating sudden increases of intensity, referred to as 'Bragg edges'. As no diffraction occurs once wavelengths exceed a threshold value, equivalent to 29 equalling 180°, these Bragg edges allow for a direct measurement of the average elastic strain in the direction of the beam [1]. Whilst Bragg edges may allow for the measurement of elastic strain directly, determining plastic strains using this technique is currently underdeveloped.

A three-parameter analytical function has demonstrated high levels of accuracy in ascertaining dspacing, with a sample dependent Gaussian broadening parameter ( $\sigma$ ) behaving analogously to the peak width in conventional diffraction experiments [1]. A major contributing factor to peak width, and hence Bragg edge broadening, is plastic deformation of the bulk material. Plasticity in the subsurface of wind turbine gearbox bearing raceways is of great concern to industry, as transient wind conditions and inertial effects contribute towards premature failure [2]. The aim of this experiment is to overload a bearing 'in situ' to produce a 2D map of the Gaussian broadening parameter, comparing results with FE simulations of yielding in the same region (Figure 1). The experiment is due to be completed on the ENGIN-X instrument at the end of March 2017.

A loading rig has been manufactured to apply overloads to a disassembled bearing using a tensometer. The FE model suggests that plasticity in the subsurface of the raceway begins at a load of 10 kN, with this region increasing in size until reaching the surface at 34 kN. The results will provide novel information about the effect of plasticity on Bragg edge broadening and further develop understanding of how overloads accelerate bearing damage. Future work involves 'in situ' loading of overloaded bearings, whilst collecting dynamic strain using stroboscopic neutron diffraction.



Figure 2. Finite element modelling (a) Raceway, (b) Estimated Plastic Zone at increasing overloads.

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Email corresponding author: agpreid1@sheffield.ac.uk

## Dislocation Density of Oxygen Free Copper with Compressive Strain applied at High Temperature

Sano M.<sup>1</sup>, Takahashi S.<sup>1</sup>, Watanabe A.<sup>1</sup>, Shiro A.<sup>2</sup>, Shobu T.<sup>3</sup>

<sup>1</sup>JASRI SPring-8, 1-1-1 Kouto Sayo-cho, Sayo-gun, Hyogo 679-5198, Japan <sup>2</sup>QST Kansai Photon Science Institute, 1-1-1 Kouto Sayo-cho, Sayo-gun, Hyogo 679-5148, Japan <sup>3</sup>JAEA Materials Sciences Research Center, 1-1-1 Kouto Sayo-cho, Sayo-gun, Hyogo 679-5148, Japan

Dislocation densities of oxygen free copper (OFC) were evaluated by X-ray line profile analyses with synchrotron radiation. OFC is known as one of the most popular materials as a heat-absorbing body in many accelerator facilities. At the SPring-8 front-end section, OFC has been applied to high heat load components such as photon absorbers and masks in all bending magnet and some undulator beamlines. Recently, we investigated the fatigue phenomenon of OFC so that these components could be used under safer conditions [1]. To extend the fatigue investigation, residual strain of the OFC samples were measured using synchrotron radiation [2]. In this study, the OFC samples with compressive strain applied at a high temperature were prepared simulating the actual operating conditions. The diffraction experiments were performed at beamline BL02B1 of SPring-8 with a monochromatic beam of 72 keV and a 2D detector of Pilatus3 X CdTe 300K. The thicknesses of OFC samples were 2 mm. In this measurement, a tilting oscillation was employed to decrease the problem of course grains with the OFC sample. In order to evaluate the dislocation density, we applied the modified Williamson-Hall and modified Warren-Averbach method based on the FWHM value and the Fourier coefficient of some diffraction profiles [3, 4]. The dislocation density and the character of dislocation could be obtained from the analysis. The details will be discussed in the conference.

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Email corresponding author: musano@spring8.or.jp

## \*Residual stress measurements in leached polycrystalline diamond using X-ray diffraction and Raman spectroscopy techniques

Vhareta M.<sup>1.2</sup>, Erasmus R.M.<sup>1.2</sup>, Comins J.D.<sup>1.2</sup>

<sup>1</sup>DST-NRF Centre of Excellence in Strong Materials, University of the Witwatersrand, Johannesburg, WITS 2050, South Africa <sup>2</sup>Materials Physics Research Institute, School of Physics, University of the Witwatersrand, Johannesburg,

WITS 2050, South Africa

Since the advent of sintered polycrystalline diamond materials (PCD) for use in earth boring and cutting tool applications there has been increasing efforts to improve their chipping and fracture resistance. Since PCD is virtually a two phase material composed of a cobalt alloy and diamond, some of the research efforts have been aimed at improving the wear resistance by focusing on the effects of leaching out the metallic phase in the PCD material. For the cobalt alloy removal, the PCD is leached in a pressure vessel using a mixture of different acids and water in a specific ratio maintained at a predetermined temperature [1]. It is believed that this would result in more thermally resistant material and a generally improved wear resistance. In this study leached PCD disc samples of 16 mm in diameter and 2 mm thickness were subjected to constant amplitude fatigue loading measurements at room temperature using a ball on three ball fatigue rig. A maximum load of 2.9 kN and a frequency of 4 Hz were employed for the fatigue measurements conducted in sets of 50 000 cycles, each followed by a systematic investigation and evaluation of the average in-plane residual stress fields on the surface of the leached PCD samples using two complementary non-destructive techniques, namely X-ray diffraction and Raman spectroscopy. The results are compared to previously published results for unleached PCD samples subjected to similar fatigue measurements, where it was shown that a general residual surface compressive stress for the lower numbers of fatigue cycles deteriorated to a high proportion of tensile regions with increasing number of fatigue cycles [2]. Whereas a general compressive stress is desirable in the PCD layer as it inhibits the propagation of cracks, on the contrary tensile stresses facilitate the formation of cracks ultimately leading to catastrophic failure of the tool-bits.

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Email corresponding author: mvhareta@gmail.com

## Analysis of multiaxial near-surface residual stress fields by energy- and angledispersive X-ray diffraction: Semi- versus nondestructive techniques

Apel D.<sup>1</sup>, Meixner M.<sup>1</sup>, Zinn W.<sup>2</sup>, Liehr A.<sup>2</sup>, Klaus M.<sup>1</sup>, Genzel C.<sup>1</sup>, Scholtes B.<sup>2</sup>

<sup>1</sup>Helmholtz-Zentrum Berlin für Materialien und Energie, Germany <sup>2</sup>Universität Kassel, Germany

Under laboratory conditions the information depth of X-ray stress analysis (XSA) experiments in steel is usually restricted to less than 10 µm. Access to residual stresses induced in deeper regions by mechanical and/or chemical surface treatment such as grinding or shot-peening requires either the application of the layer removal method or the analysis with highly penetrating X-rays and synchrotron radiation, respectively. Layer removal yields the actual residual stress depth profiles  $\sigma_{ij}(z)$  up to any depth below the surface, but at the price of being time-consuming and semi-destructive. High energy diffraction performed in the energydispersive (ED) mode avoids these drawbacks, but it provides only the LAPLACE stresses  $\sigma_{ij}(\tau)$  which have to be transformed back into the z-space.

With the example of uniaxial ground steel we show that the layer removal and the ED diffraction method yield comparable results for the in-plane stress components  $\sigma_{11}$  and  $\sigma_{22}$ . However, significant differences are observed concerning the out-of-plane stresses, which are due to the boundary conditions the  $\sigma_{i3}$  components must satisfy at the free surface. We demonstrate that stress redistribution at the newly generated surfaces after layer removal leads to an underestimation of the shear stresses  $\sigma_{13}$  (see Figure 1). The analysis of the normal stress component  $\sigma_{33}$  is shown to require nondestructive measurements.

The ED diffraction experiments were performed under synchrotron as well as laboratory conditions. The latter were realized on a new type of 8-circle diffractometer, which was developed for advanced residual stress analysis in the ED diffraction mode. It is equipped with two Si(Li) detectors which allow for simultaneous strain analysis in different sample orientations.

Email corresponding author: daniel.apel@helmholtz-berlin.de



Figure 1. Near surface residual shear stress depth profiles in an uniaxial ground ferritic steel sample obtained by different non- and semidestructive XSA methods performed in the angle-dispersive (AD) and the energy-dispersive mode, respectively.

## \*Evaluation of Residual Stresses Introduced by Laser Shock Peening using Different Measurement Techniques

<u>Glaser D.</u><sup>1,2</sup>, Newby M.<sup>3</sup>, Polese C.<sup>2</sup>, Berthe L.<sup>4</sup>, Venter A.M.<sup>5</sup>, Marais D.<sup>5</sup>, Nobre J.P.<sup>2</sup>, Styger G.<sup>6</sup>, Paddea S.<sup>7</sup>, Van Staden S.N.<sup>2</sup>

<sup>1</sup>CSIR National Laser Centre, Brummeria, Pretoria, South Africa
<sup>2</sup>University of the Witwatersrand, Johannesburg, South Africa
<sup>3</sup>Eskom Holdings SOC Ltd, Lower Germiston Road, Rosherville, Johannesburg, South Africa
<sup>4</sup>PIMM Laboratory (ENSAM-CNRS-CNAM), 151 Bd de l'Hôpital 75013 Paris, France
<sup>5</sup>Necsa SOC Limited, Pretoria, South Africa
<sup>6</sup>University of Johannesburg, Auckland Park, Johannesburg, South Africa
<sup>7</sup>The Open University, Milton Keynes, United Kingdom

Laser Shock Peening (LSP) is a residual stress engineering technology specifically used to introduce beneficial compressive residual stresses into critical components to enhance fatigue and/or Stress Corrosion Cracking performance [1]. The CSIR National Laser Centre is currently developing LSP capabilities in order to implement the process to enhance Low Pressure (LP) steam turbine blades used in the power generation sector. One of the benefits of the technology is the precise control of laser parameters, and hence the potential to introduce a beneficial compressive stress field to the desired level. A critical aspect of the process development for a given application is therefore the quantification of the residual stresses introduced.

There are a number of measurement methods that can be employed, whereby a combination of complimentary techniques is often desirable. The present paper discusses a comparison of results of the stress field introduced by LSP on 12CrNiMoV steel used in LP steam turbine blades. The samples for this investigation are  $20 \times 20 \times 15$  mm, with LSP treatment applied as a  $10 \times 10$  mm patch on the square surface as depicted in Figure 1. The most interesting features of the residual stresses introduced by the LSP process will be the near surface stress state, as well as the depth of the compressive residual stress.

The comparative study will consider the advantages and disadvantages of several methods. Non-destructive techniques include Synchrotron X-Ray Diffraction data from an experiment on ID15A beamline at the ESRF, and neutron diffraction to be performed at NECSA. The destructive measurement techniques include the incremental hole drilling technique with a SINT instrument, laboratory XRD combined with the electropolishing technique, and the contour measurement method. The results from a preliminary study conducted comparing XRD with electropolishing and the incremental hole drilling method for LSP are shown in Figure 1, which also depicts a photograph of the LSP process and the typical test coupon.



Figure 1. Preliminary results comparing principle stresses from laboratory XRD used with electro-polishing and the incremental hole drilling method (left), and a photograph of the LSP process and resulting coupon (right).

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Email corresponding author: DGlaser@csir.co.za

## Residual stress distributions via high heat-input, different thermal expansion, and low transformation temperature weld cases

Woo W.<sup>1</sup>, Wang H.<sup>1,2</sup>, Kim D.-K.<sup>1</sup>, Yeol Lee S.<sup>2</sup>, Baek An G.<sup>3</sup>

<sup>1</sup>Neutron Science Division, Korea Atomic Energy Research Institute, Daejeon, 34057, Korea <sup>2</sup>Department of Materials Science and Engineering, Chungnam National University, Daejeon 34134, Korea <sup>3</sup>Department of Naval Architecture & Ocean Engineering, Chosun University, 61452, Korea

Contour method, deep hole drilling, and neutron diffraction measurements were performed to determine residual stress distributions through the thickness of the 70 mm thick welded plates. First, a weld specimen was prepared using flux-cored arc welding (FCAW) technique, which creates a conventional multi-pass weld with the heat input of 1.7 kJ/mm. Secondly, a high heat-input (56 kJ/mm) one-pass weld specimen was prepared using electro-gas welding (EGW). Thirdly, a dissimilar weld (DW) specimen was prepared joining between fcc austenite steel and bcc ferritic steel using FCAW. Finally, a low temperature phase transformation (LTT) weld specimen was prepared. The LTT uses the specially designed welding consumable composed of 0.04C-0.37Si-11Ni-12Cr, which decreases the martensite phase transformation temperature to near ambient temperature during cooling. The results show that significant tensile stresses (about 90% of yield strength) occur along the weld centerline near the top surface (within 10% of the depth) due to the heat accumulations of the multi-pass conventional FCAW specimen. Meanwhile, the high heat-input weld (EGW) shows that the maximum tensile stress moved towards the heat-affected zone at a depth of about 40% of the thickness, which can be attributed to the reduction of the yield strength of the weld by large heat-inputs. In the DW, significant tensile stresses (about 300 MPa) occur near the interface between the welding consumable (fcc) and the base metal (bcc). Such a high tension in the localized region is caused by the difference in the thermal expansion coefficient (CTE) between the weld and base materials. Finally, it is clear to provide the compression via the volume expansion of the martensite phase transition during cooling in the LTT weld. Two dimensional distributions of residual stresses will be compared for the conventional, high heat-input, dissimilar, and phase transformation welds.

Email corresponding author: chuckwoo@kaeri.re.kr

### \*Study of twinning-detwinning effect in magnesium-aluminium binary alloys by the neutron diffraction

Čapek J.<sup>1,2</sup>, Máthis K.<sup>1</sup>

<sup>1</sup>Department of Physics of Materials, Faculty of Mathematics and Physics, Charles University, Ke Karlovu 5, 121 16 Prague, Czech Republic

<sup>2</sup>Nuclear Physics Institute of the Academy of Sciences of the Czech Republic, Řež, 250 68, Czech Republic

Twinning belongs to the most important deformation mechanisms during straining of magnesium alloys. The  $\{10\overline{1}2\}\langle 10\overline{1}1\rangle$  twinning is associated with extension along the c-axis and reorientation of the lattice by 86.3°. During a cyclic process, twin nucleation takes place during loading, whereas detwinning is observed during unloading. The latter process results in anelastic behaviour [1].

Three different alloys, Mg pure, Mg 2 wt.% Al and Mg 9 wt.% Al were tested. In-situ neutron diffraction (ND) measurements were carried out at the SMARTS engineering instrument at the Lujan Neutron Scattering Center [2]. Compression testing were carried out using a horizontal 250 kN capacity load frame at a strain rate of 1 x 10-3 s-1 in strain control mode. In order to collect ND data with good enough statistics, the tests were stopped at predefined strain levels (0.1, 0.5, 1, 2, 3, 6 %) for approx. 60 min, subsequently unloaded to 0 MPa and loaded to next strain level.

The aluminium content strongly influences twinned volume and detwinning activity [3]. This work is focused on the examination of the lattice strains evolution for 10.0-00.2 diffraction peaks, which characterize the twinning. It is well known that during compressive loading 0002 reflection shows anomalous behaviour in transverse direction when the lattice strain values become negative owing to the twinning activity (Figure. 1 – solid symbols) [4]. We observed that aluminium content influence not only the critical resolved shear stress for the twinning activation, but also the residual stress after the unloading (Figure.1 – empty symbols): in pure Mg the absolute value of lattice strain decreases. In contrast, that for the Mg9Al increases.



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Email corresponding author: jan.capek89@gmail.com

### Influence of laser power and traverse speed on weld characteristics of Laser beam Welded Ti-6Al-4V sheet

Mashinini P.M.<sup>1</sup>, Hattingh D.G.<sup>2</sup>

 <sup>1</sup> University of Johannesburg, Mechanical and Industrial Engineering Technology Department, Johannesburg, South Africa
<sup>2</sup> Nelson Mandela Metropolitan University, Mechanical Engineering Department, Port Elizabeth, South Africa

In this paper laser beam welding was used for joining 3 mm Ti-6Al-4V alloy sheets in a full penetration buttweld configuration. Laser beam power and traverse speed were the only parameters varied in an attempt to characterize the influence on weld integrity with specific reference to residual stress and microstructural modifications. The iXRD residual stress data showed a definite influence of traverse speed on residual stresses, with low traverse speeds resulting in an increased tensile residual stresses in the longitudinal direction of the weld whilst in the transverse direction residual stress revealed a more compressive stress state. The residual stress data for this experiment compared favourably with published residual stresses data done by synchrotron X-ray diffraction. Weld joint integrity was further analyzed by evaluating the microstructure transformation in the weld nugget. These results revealed a degree of grain growth and the presences of fine acicular  $\alpha$  (needlelike  $\alpha$ ) in prior  $\beta$  grain boundaries with increased traverse speed. Grain growth was predominantly influenced by the cooling rate which is associated with traverse speed. Additionally, the  $\alpha$ -phase and  $\beta$ -phase were characterized in the various weld zones by electron backscatter diffraction (EBSD).

Email corresponding author: mmashinini@uj.ac.za

## On the measurement of bulk dislocation density using electron backscatter and synchrotron diffraction technique

Muránsky O.<sup>1</sup>, Balogh L.<sup>2</sup>, Tran M.<sup>3</sup>, Daymond M.R.<sup>2</sup>

<sup>1</sup> Australian Nuclear Science and Technology Organization (ANSTO), Lucas Heights, New South Wales, Australia <sup>2</sup> Queen's University, Kingston, Ontario, Canada <sup>3</sup> University of California, Davis, California, USA

The strain hardening of a polycrystalline aggregate is governed by the ability of the material to store dislocations that obstruct the motion of other moving dislocations. Plastic deformation is thus accompanied by the storage of ever-increasing amounts of dislocations when strain hardening is present. Two dislocation types are identified: (i) statistically-stored dislocations (SSDs), which are stored by trapping one another in a random way; and (ii) geometrically-necessary dislocations (GNDs), which are stored by the microstructure to maintain continuum deformation.

Recent developments in Electron Back-Scatter Diffraction (EBSD) analysis and high-resolution diffraction line profile analysis (LPA) techniques provide an opportunity for studying the way in which a material stores different types of dislocations. In general, the diffraction LPA techniques are sensitive to the strain field caused by the presence of all types of dislocations, while the EBSD technique is sensitive to a variation in the lattice curvature, which is attributed to the presence of GNDs. The rest of the dislocations have no geometrical consequence at the length scale studied, and are thus undetected by the EBSD lattice curvature analysis, and can be considered as SSDs.

In the present study, high-resolution synchrotron diffraction and EBSD techniques have been used to study the development of both the total (SSD + GND) dislocation density and the GND density, respectively, during the uniaxial loading of Ni201 and austenitic 316 steel. It is shown that the strain-hardening of both materials is governed by the collaborative effect of both SSDs and GNDs.

Email corresponding author: omz@ansto.gov.au

### Grain scale microstructure evolution characterization of ceramic nuclear fuels

Pokharel R.<sup>1</sup>, <u>Brown D.W.</u><sup>1</sup>, Clausen B.<sup>1</sup>, Reiche M.<sup>1</sup>, Byler D.D.<sup>1</sup>, McClellan K.J.<sup>1</sup>, Hunter J.F.<sup>1</sup>, Ickes T.L.<sup>1</sup>, Sharma H.<sup>2</sup>, Park J.-S.<sup>2</sup>, Kenesei P.<sup>2</sup>, Rollet A.D.<sup>3</sup>

<sup>1</sup>Los Alamos National Lab, Los Alamos, NM 87544, USA <sup>2</sup>Argonne National Lab, Lemont, IL 60439, USA <sup>3</sup>Carnegie Mellon University, Pittsburgh, PA 15213, USA

In this work, we study the effect of fabrication conditions on as-sintered microstructures of various stoichiometric ratios,  $UO_{2+x}$ , with the aim of enhancing the understanding of fabrication process and developing and validating predictive microstructure based model for fuel performance. We demonstrate the ability of novel, non-destructive methods such as high-energy X-ray diffraction microscopy (HEDM) and micro-computed tomography ( $\mu$ -CT) to probe bulk samples of high-Z materials by non-destructively characterizing three materials: UO<sub>2.00</sub>, UO<sub>2.11</sub>, and UO<sub>2.16</sub>, which were sintered at 1450°C for 4 hours. The HEDM and  $\mu$ -CT measurements revealed three-dimensional (3D) volumetric orientation and density information on the bulk samples at unprecedented resolution (~5  $\mu$ m, 0.1 deg) [1]. The non-destructive nature of HEDM and  $\mu$ -CT measurements enabled a second measurement of the microstructures following the annealing. After the initial state measurements, the three as-sintered materials were annealed ex-situ at 1950°C in an induction furnace for 2.6 hours in an ultra-high-purity argon gas environment. Microstructure and micro-mechanical parameters such as orientations, grain size distributions, and grain scale elastic strain and corresponding stress tensors were characterized. The before and after measured 3D microstructures revealed that grain size, grain stress, and bulk porosity were influenced by chemistry as well as processing conditions.

In addition, as a proof-of-principle study of grain growth in ceramic nuclear fuels, microstructure evolution in a depleted stoichiometric  $UO_2$  in its as sintered (1350°C for 12 hours) and annealed (2200°C for 2.6 hrs) states were measured (Figure 1). The initial measured microstructure was then used as an input to the Monte Carlo grain growth simulation based on the Potts model. Model parameters were calibrated using the measured microstructure obtained after heat-treatment. Microstructure evolution, grain size distribution, and texture change were quantified and predictions were compared with observation where possible.



*Figure 1. Before and after 3D views displaying microstructure evolution in UO*<sub>2</sub>. *Colors correspond to crystallographic orientations represented in Rodrigues vector mapped to RGB colors [2].* 

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Email corresponding author: reeju@lanl.gov

## Residual stress and diffraction line-broadening analysis of Al<sub>2</sub>O<sub>3</sub>/Y-TZP ceramic composites by neutron diffraction

Fan K.<sup>1</sup>, <u>Ruiz-Hervias J.<sup>2</sup></u>, Gurauskis J.<sup>3</sup>, Baudin C.<sup>3</sup>

<sup>1</sup>State Key Laboratory of Powder Metallurgy, Central South University, Changsha 410083, China <sup>2</sup>Materials Science Department, Universidad Politécnica de Madrid, E.T.S.I. Caminos, Canales y Puertos, Profesor Aranguren 3, E-28040 Madrid, Spain <sup>3</sup>Instituto de Cerámica y Vidrio, CSIC, Kelsen 5, E-28049 Madrid, Spain

Alumina-zirconia ceramics have received considerable attention in both engineering and academic fields due to their improved mechanical properties compared with pure alumina ceramics [1, 2]. Being regarded as a strong candidate for structural and biomedical applications, great interest is put on the possible reinforcing mechanisms as well as the influential factors. It has been proposed that interphase residual stresses generated by the thermal and elastic deformation mismatches between alumina and zirconia could contribute to toughening and enhance the structural performance of these ceramic composites [3]. Most of the previous research works [4] are focused on the uniform residual stresses between phases, without paying attention to the non-uniform microstrains at the subgrain scale in each phase. Such microstrains can be due to the existence of crystal defects (e.g., dislocations and crystal vacancies), which influence the mechanical properties of materials. In addition to that, non-uniform microstrains might provide an additional increment of tensile strain which would result in crack initiation and propagation. As a consequence, an accurate knowledge of residual stresses at different scales (uniform and non-uniform) would provide valuable data to optimize the performance and reliability of ceramic composites.

The objective of this research is the non-destructive determination of the uniform and non-uniform residual stresses in a series of  $Al_2O_3/Y$ -TZP (alumina/tetragonal ZrO<sub>2</sub> stabilized with 3 mol.% Y<sub>2</sub>O<sub>3</sub>) ceramic composites. Different zirconia contents (5 vol.% and 40 vol.%) and green processing routes (tape casting and conventional slip casting) were investigated.

Time-of-flight neutron diffraction (ENGIN-X [5], in ISIS, UK) has been used for reliable through-thickness residual strain measurement in bulk samples. Residual stresses, multi-phase qualitative and quantitative analyses, as well as crystal structure determination were performed by Rietveld refinement. Line broadening analysis was carried out, using the "Double-Voigt" profile modelling, to obtain the microstructural information (domain size and crystal microstrain). The effects of the addition of second phase zirconia particulates and the green processing technique employed were discussed in terms of the obtained residual stress fields, microstrains and domain sizes.

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Email corresponding author: jesus.ruiz@upm.es

## In-situ Investigation of Microstructure Evolution during Annealing in Ti-6Al4V Alloy Produced by Additive Manufacturing

Takajo S.<sup>1,2</sup>, Vogel S.C.<sup>2</sup>, Pesach A.<sup>3</sup>, Yeheskel O.<sup>3,4</sup>, Caspi E.<sup>3</sup>, Tiferet E.<sup>3,4</sup>

<sup>1</sup>Los Alamos National Laboratory, Los Alamos, NM, U.S.A. <sup>2</sup>JFE Steel Corporation, Kurashiki, Japan. <sup>3</sup>Nuclear Research Center Negev, Israel. <sup>4</sup>Rotem Ind. Ltd, Mishor Yamin, Israel

Electron beam melting (EBM) and selective laser melting (SLM) are additive manufacturing (AM) processes, in which three dimensional metallic objects are fabricated by melting the ingredient powder materials layer by layer on a platform pre-heated bed. Due to high solidification rates of small melt volumes, AM products may result in off-equilibrium microstructures, in which macro-strain, micro-strain and directional growth are present. It was reported on AM TiAl6V4 that the AM process leads to formation of  $\beta$  phase grains which can change the mechanical properties of the material substantially [1]. Furthermore, the type of AM process, such as the EBM and SLM processes studied here, influences the final microstructure and residual stresses [2].

In this study, to clarify the dependency of the AM methods on microstructure, three different AM TiAl6V4 samples were prepared: one produced by EBM with the bed temperature of 973 K (EBM1); the second, produced by SLM with the bed temperature of 473 K (SLM2); the third, also produced by SLM with the different laser machine and re-heated at 1113 K for 2 hours for stress release (SLM3).

Rietveld analysis of our preliminary measurements (carried out at HIPPO, LANL) on AM TiAl6V4 samples showed a dependency between the AM process and the content of  $\beta$ -phase and the strength of  $\alpha$ -phase texture in post processed samples. It was found that the weight percentage of the  $\beta$ -phase of one SLM without reheating process was ~10 times higher than in similar samples that were produced with EBM or SLM with different machine. As shown in Figure 1, it was also found that the micro-structure of the EBM sample was free of preferential orientation, whereas in the SLM samples, especially after re-heating process, significant preference appeared towards the hexagonal basal plane. We expand on the ambient condition measurements and we also report our findings of the microstructure evolution during annealing process at temperatures up to 1323 K.



Figure 1.  $\alpha$  texture for (0002) pole figures measured at room temperature.

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Email corresponding author: stakajo@lanl.gov

## \*Investigating the residual stress distribution in Selective Laser Melting produced Ti-6Al-4V using neutron diffraction

Anderson L.S.<sup>1</sup>, Venter A.M.<sup>2</sup>, Vranken B.<sup>3</sup>, Marais D.<sup>2</sup>, Van Humbeeck J.<sup>3</sup>, Becker T.H.<sup>1</sup>

<sup>1</sup>Department of Mechanical and Mechatronic Engineering, Stellenbosch University <sup>2</sup>Research and Development Division, South African Nuclear Energy Corporation <sup>3</sup>Department of Materials Engineering, University of Leuven,

The Selective Laser Melting (SLM) process makes rapid manufacture of both prototype and structural components possible for a variety of metals. However, high residual stresses are inherent to the building process itself and can pose a number of problems. These stresses are typically tensile in nature at the exterior of the part and can lead to part distortion and crack initiation, while reducing the components mechanical strength and fatigue life [1]. Various methods of measuring residual stress, such as the contour method, hole-drilling method and beam deflection method, have been employed to understand the stress distribution in SLM components. However, these methods do not provide stress distribution through the full volume of the part. The neutron diffraction technique is able to penetrate deeply into most engineering materials and can be used to calculate the stress distribution through the volume of a sample from measured lattice strains [2].

20 x 20 x 10 mm<sup>3</sup> rectangular Ti-6Al-4V samples were produced to investigate the influence of layer thickness and laser scan strategy on the residual stress. All samples were built on KU Leuven's in-house developed SLM machine using a laser power of 250W, scan speed of 1200 mm/s and a hatch spacing of 75  $\mu$ m and were grown in the z-direction, with the build layers deposited in the xy plane depicted in Figure 1. Neutron diffraction scans were performed at the NECSA neutron scan instrument MPISI. Measurements were taken, non-destructively, in each sample along cross sectional planes as shown in Figure 1.



Figure 1: Measurement planes through sample volume

The results show that there are distinct regions of stress, with the outer surfaces (top/bottom and sides) experiencing a tensile stress and the central volume being under compressive stress of the same magnitude. The results also show a trend that an increase in layer thickness reduces the stress gradients between the tensile and compressive regions in the part. There is also evidence to show that by alternating the scan pattern direction, layer by layer, stress is prevented from developing along a preferential axis and, instead, becomes more isotropic.

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Email corresponding author: landerson@sun.ac.za

## The influence of high welding speed on the residual stresses and microstructure in friction stir welded AA5182-H111

Bernard D.<sup>1</sup>, Hattingh D.G.<sup>1</sup>, Steuwer A.<sup>1,2</sup>, Goosen W.E.<sup>3</sup>

<sup>1</sup>Department of Mechanical Engineering, Nelson Mandela University, Port Elizabeth, South Africa <sup>2</sup>University of Malta, Malta, Europe

<sup>3</sup>Centre for High Resolution Transmission Electron Microscopy, Nelson Mandela University, Port Elizabeth, South Africa

Friction stir welding of 5xxx-series aluminium plate is currently performed at welding speeds below 500 mm/min. Compared to conventional welding processes this is considered slow and limits the commercial acceptability of FSW in the Aluminium fabrication sector. This was addressed by developing the process to successfully friction stir weld 5mm thick AA5182-H111-(T500) at a travel speed of 1500 mm/min.

In this manuscript, the residual stress distribution, determined through Synchrotron Radiation measurements, and weld microstructure for a slow and fast friction stir weld are compared to evaluate the influence of the different process thermal cycle introduced by the tool/material interaction for two weld speeds. AA5182-H111 with a thickness of 5 mm was welded at a feed rate of 200 mm/min (slow), and is compared to a feed rate of 1500 mm/min (fast). The low thermal cycles (Figure 1) and high process forces

associated with the fast weld resulted in high tensile residual stresses within the processing zone, while the slow weld displayed lower peak tensile residual stresses, with a wider profile as shown in Figure 2.

Through electron backscatter diffraction it was found that the high welding speed not only resulted in a highly refined, dynamically recrystallized stir zone, but the adjacent thermo-mechanically affected zone experienced significant plastic strain.

#### Distance from Centre (mm) 20 (a) 0 100 -20 150 -20 0 10 30 -10 20 40 50 60 250 Time from Tool Centre (s) 350 Distance from 450 Centre (mm) 20 550 0 (b) -20 -20 -10 0 10 20 30 40 50 60 Time from Tool Centre (s)

Figure 1. Process thermal cycles in terms of time to the tool centre for (a) the Slow weld and (b) the Fast weld.



Figure 2. Transverse residual stress distribution of the Slow and Fast weld.

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Email corresponding author: dreyer.bernard@nmmu.ac.za

### Development of Phase Stresses During Additive Manufacture of 304L Stainless Steel

Dahal J.<sup>1</sup>, Brown D.W.<sup>2</sup>, Carpenter J.<sup>2</sup>, Clausen B.<sup>2</sup>, Cooley J.<sup>2</sup>, Losko A.S.<sup>2</sup>, Strantza M.<sup>2</sup>

<sup>1</sup>Colorado School of Mines, 1500 Illinois St, Golden, CO 80401 <sup>2</sup>Los Alamos National Lab, Los Alamos, NM 87545

The last decade has seen tremendous advances in the ability of X-rays and neutrons at large scale facilities to probe microstructure at unprecedented length and time scales under unique environments that simulate manufacturing conditions. Concurrently, manufacturing is undergoing a revolution as investments are made in advanced manufacturing techniques, such as additive manufacture. It is natural that advanced manufacturing techniques in order to accelerate the process of qualification of products for critical applications.



The evolution of the lattice parameters of austenite and ferrite following deposition of 308L stainless steel. The symbols represent observed data, the lines the known thermal expansion of each phase. The difference is due to the mismatch of thermal expansion.

Email corresponding author: Dbrown@lanl.gov

As an example of studying the effect of processing on microstructure, high energy x-ray diffraction has been used to monitor microstructural evolution in-situ during additive manufacture of 304L stainless steel with sub-second time resolution and sub 0.1mm spatial resolution. The metal was deposited by Wire Arc Additive Manufacture (WAAM) as this process is inherently relevant for structural repair using additive manufacture. A ~3mm bead of 308L steel was deposited by a Cold Metal Transfer system onto the rim of a rotating 304L stainless steel disk. High energy x-rays passed through the deposited bead probing the microstructure of the material following deposition and during rapid cooling to room Specifically, in this talk we will present the temperature. evolution of stresses in the austenite and ferrite phases. Both the strong temperature gradient and thermal mismatch of the constituent phases contribute to the phases stresses. For example, the figure shows the evolution of the lattice strain during cooling relative to the known thermal expansion of each phase. The difference observed in the ferrite is due to large compressive stresses that develop due to the difference in thermal expansion. A finite element model has been developed including both of these effects in an attempt to better understand the observations gleaned from the diffraction data.

## Residual stress fields in additively manufactured Ni superalloy 718 as built and after release from baseplate

Cabeza Sánchez S., Thiede T., Mishurova T., Kromm A., Bruno G.

## Bundesanstalt für Materialforschung und –prüfung (BAM; Federal Institute for Materials Research and Testing), Unter den Eichen 87, D-12205 Berlin, Germany

The residual stress distribution of Nickel superalloy 718 parts produced by Selective Laser Melting (SLM) technique was studied by means of neutron diffraction.

Two deposition hatching lengths were considered in the fabrication. Both lateral (building direction) and top (finishing) near-surface regions were characterized. Measurements on samples in as-built condition and after release from the plate proved the presence of stress gradients both in-plane and along the building direction around  $\pm 300$ MPa.

As-built samples presented in top region a longitudinal stress relief for large hatching, whereas small one showed tensile stresses in the middle, evolving towards compression at the tip of the sample. Towards the lateral edge, longitudinal stresses shifted also to compression. The transverse stresses for large hatching were relief in the middle, whereas for small hatching shifted to compression at the edge. As for the normal component, this was more homogenous: stress-relief was proved for large hatching and, in contrast, was in compression for small hatching. In the building direction (lateral region from base plate to top) of the sample with large hatching all stress components showed tensile values near the base plate, decreasing towards compression to the top, where they were almost released.

After release, in the top region, the longitudinal stress component for small hatching showed high compressive stresses in the central part. In contrast, for large hatching a stress relief was found. In the transversal direction, this behaviour was inverted: a small hatching released stresses more effectively, while a large hatching presented high tensile stresses. As for the normal component (i.e., building direction), the sample with small hatching was found in compression, while that with large hatching was stress-released or slightly in tension. In the lateral surface region, all components showed similar behaviour: a small hatching promoted high compressive stresses along the building direction, whereas a large hatching showed small tensile values at the bottom, which balance towards the top region. There is an overall shift of stresses in 3 directions towards tension when compared with as-built condition for top region. In contrast, the lateral region is stress-relief or shifted towards compression after cutting from baseplate.

In conclusion, hatching length parameter strongly influenced the 3D distribution of residual stress in SLM produced parts.

Email corresponding author: <a href="mailto:s.cabezasanchez@gmail.com">s.cabezasanchez@gmail.com</a>

## Synchrotron XRD Evaluation of Residual Stresses Introduced by Laser Shock Peening for Steam Turbine Blade Applications

Newby M.<sup>1</sup>, Steuwer A.<sup>2</sup>, Glaser D.<sup>3,4</sup>, Polese C.<sup>4</sup>, Hattingh D.G.<sup>5</sup>, Gorny C.<sup>6</sup>

<sup>1</sup>Eskom Holdings SOC Ltd, Lower Germiston Road, Rosherville, Johannesburg, South Africa
<sup>2</sup>University of Malta, Msida MSD 2080, Malta
<sup>3</sup>CSIR National Laser Centre, Brummeria, Pretoria, South Africa
<sup>4</sup>University of the Witwatersrand, 1 Jan Smuts Avenue, Johannesburg, South Africa
<sup>5</sup>Nelson Mandela Metropolitan University, Port Elizabeth, South Africa
<sup>6</sup>Laboratoire PIMM (ENSAM, CNRS, CNAM, Hesam Université), 151 Bd de l'Hôpital 75013 Paris, France

Thermal energy is the most commonly used source for electricity generation globally, which is often extracted by the use of large steam turbines. The corrosion resistant steel blades of a typical Low Pressure (LP) rotor operate in a wet steam environment, whilst rotating at speeds in the range of 3000 rpm. The approximately 1 m long LP blades therefore see high centrifugal loading, which presents challenges of stress corrosion cracking or corrosion fatigue [1]. The highly stressed fir tree attachment root, as illustrated in Fig. 1, is conventionally Shot Peened (SP) in order to introduce beneficial compressive residual stresses to mitigate crack initiation. A catastrophic failure of one of these blades at a South African power station in 2003 resulted in over  $\notin$ 100 million damage, and raised concerns to the effectiveness of the conventional SP treatment for the achievement of uniform coverage over the complex geometry of the fir tree section.

Laser Shock Peening (LSP) has been identified as an attractive technology for this application in order to potentially enhance the lifespan of the critical LP turbine blades. The CSIR National Laser Centre and Eskom are currently conducting research into the application of LSP for turbine blade refurbishment in close collaboration with SA universities. Initial coupon level results seem promising, as shown in Fig. 1. The stress profile measured using laboratory XRD and electro-polishing reveals deeper levels of compressive residual stress achieved for the LSP treatment, with an improved surface finish compared to conventional SP.



Figure 1. Preliminary results comparing residual stress and surface topography of LSP and SP (left), and the typical LP steam turbine blade (right).

The present paper discusses the results of a Synchrotron X-Ray Diffraction experiment at the ESRF ID15A beamline on sample set devised to understand the influence of different LSP parameters. Increasing power intensity and overlap is expected to drive deeper compressive residual stresses, whilst the spot size affects the maximum depth of plasticity achievable. Since the LSP processing sequence is known to produce residual stress anisotropy, strains in the longitudinal and transverse directions will be examined with attention provided to location of the compensatory tensile residual stress.

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Email corresponding author: NewbyM@eskom.co.za
# Neutron diffraction investigation of residual stresses in Nickel based austenitic weldments on creep resistant Cr-Mo-V material

Doubell P.<sup>1</sup>, Newby M.<sup>1</sup>, Hattingh D.G.<sup>2</sup>, Steuwer A.<sup>2,3</sup>, James M.N.<sup>2,4</sup>

<sup>1</sup>Eskom Holdings SOC Ltd, Lower Germiston Road, Rosherville, Johannesburg, South Africa <sup>2</sup>Nelson Mandela Metropolitan University Port Elizabeth, South Africa <sup>3</sup>University of Malta, Msida MSD 2080, Malta <sup>4</sup>Plymouth University, Drake Circus, Plymouth, UK

Residual creep ductility of service aged Cr-Mo-V creep resistant material is considerably lower than that of new material which have a profound effect on the long-term creep life performance of the components manufactured from this type of alloy as the creep rate under these exhausted conditions is considerably higher than for new materials. This study focused on determining the effects of repair welds with nickel-based consumables on creep exhausted material with respect to residual stresses on the base material and the effect of post weld heat treatment on the remaining life of the components. Residual stresses attributed to phase transformation when ferrite transforms to austenite and back involves a sudden volume change of the welding material. This can have an adverse effect on aged material with low creep ductility and which exhibits notch sensitivity such as for a <sup>1</sup>/<sub>2</sub>Cr-<sup>1</sup>/<sub>2</sub>Mo-<sup>1</sup>/<sub>4</sub>V alloy, rendering this alloy prone to the reheat cracking phenomenon.



Reheat cracking damage in weld heat affected zone of ½Cr-½Mo-¼V alloy



SALSA neatron beam facility (ILL in Grenoble)

Coupons prepared from creep damaged Cr-Mo-V pipes (323 mm outside diameter and 36 mm thick) were joined with the tungsten inert gas (TIG) and manual metal arc (MMA) welding processes simulating the original construction joints. Standard welding procedures were used with and without the addition of stress relief and temper post weld heat treatment.

Samples were subsequently prepared for the experimental procedure for triaxial stress measurements on the SALSA neutron diffraction beam line (ILL Grenoble). One coupon contained a butt weld performed with Nibased austenitic consumables while a second was welded using conventional ferritic weld consumables as a comparison. For calibration purposes combs of sectioned weld coupons provided a simulation of the unstrained lattice parameter.

The results will be applied to the structural integrity of weld repaired Cr-Mo-V creep resistant materials used for sensitivity analysis when performing FE modelling of plant system stresses to establish the influence of these weld repairs on existing residual life prediction models.

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Email corresponding author: philip.doubell@eskom.co.za

# Fast temporal and spatial resolved stress analysis at laser surface line hardening of steel AISI 4140

Kiefer D.<sup>1</sup>, Gibmeier J.<sup>1</sup>, Beckmann F.<sup>2</sup>

<sup>1</sup>Karlsruher Institute of Technology (KIT), Institute for Applied Materials (IAM-WK), Engelbert-Arnold-Str. 4, 76131 Karlsruhe, Germany <sup>2</sup>Helmholtz Zentrum Geesthacht (HZG), Institute of Materials Research, Max-Planck-Str. 1, 21502 Geesthacht, Germany

Steel components are often subjected to high mechanical (static or alternating) loads. Regarding the dimensioning of technical components a special importance is attached to its outer layers since degradation like e.g. corrosion or crack initiation generally starts at the surface. Here, often the highest loading conditions occur in service. An advantageous method to improve wear resistance and fatigue strength can be achieved reliably by laser surface hardening [1, 2]. A near surface hardness increase and the formation of favourable compressive residual stresses can be accomplished by a short time laser heat treatment followed by self-quenching of the workpiece. In comparison to conventional hardening processes laser surface hardening has, inter alia, the following advantages: (i) induction of localized heat input, (ii) minimal distortion of the workpiece, (iii) extremely short processing times. Moreover, fibre coupled laser optics allow a highly flexible local processing.

Current process analysis and improvement is only based on ex-situ investigations of hardened samples. For a complete understanding, about the effect of variable process parameters, information about kinetics and thermodynamics of the phase transformations need to be obtained. Hence, it is indispensable to take a closer look in the ongoing process. Furthermore, the results of such in-situ analyses can be used to optimize and validate numerical simulations that allow proper process predictions. In this regard we applied the experimental setup for real time monitoring of phase transformations and stresses during laser surface hardening by means of synchrotron X-ray diffraction described in [3]. The set-up was upgraded by a new laser optics with an inline one-color pyrometer for a fully temperature controlled process variation. Additionally an improved linear actuator allows higher (and hence technical relevant) feed rates of the laser beam. Using the upgraded measuring and evaluation approach an unprecedented data basis for validation of time resolved simulation of laser line surface hardening can be provided to investigate the influence of different laser feed rates on the hardening result.

Samples made of AISI 4140 were laser line hardened using a 4 kW high power diode laser with pyrometric temperature control and constant feed rate. In-situ X-ray diffraction measurements during temperature controlled laser surface line hardening experiments have been carried out at the synchrotron beamline P05@PETRAIII at the German electron synchrotron (DESY) in Hamburg. The local phase transformation kinetics and stress evolution is recorded by use of synchrotron X-radiation at a measuring rate of 20 Hz with a spatial resolution of 1 mm<sup>2</sup>. In the course of this, different measurement positions with respect to the laser beam axis were investigated. The in-situ analyses were complemented by high spatial resolved ex-situ residual stress analyses in accordance to the well-known sin<sup>2</sup> $\psi$  method [4].

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Email corresponding author: Dominik.Kiefer@kit.edu

# Effect of residual stress relaxation during sample preparation on the detectability of hot crack networks in LTT welds by means of µCT

Vollert F.<sup>1</sup>, Dixneit J.<sup>2</sup>, Gibmeier J.<sup>1</sup>

<sup>1</sup> Karlsruhe Institute of Technology (KIT), Institute of Applied Materials (IAM), Kaiserstr. 12, 76131 Karlsruhe, Germany

<sup>2</sup> BAM Federal Institute for Materials Research and Testing, Unter den Eichen 87, 12205 Berlin, Germany

Hot crack assessments in welds are of major concern for the safety of welded components. The surface cracks can be investigated using the externally loaded Modified Varestraint Transvarestraint (MVT) hot cracking test [1]. Here, the hot cracking resistance is determined by light microscopy adding up all visible crack lengths directly at the specimen surface. The shortcoming of this approach is that the information from the very near surface inhibits access to the characteristic of the hot crack network in the bulk. Hence, the restriction to the total crack length at the very surface might result in incorrect assumptions with regard to the assessment of the crack network. Here, we report about an alternative approach to monitor the entire 3D hot crack network after welding by means of microfocus X-ray computer tomography ( $\mu$ CT). However, to provide sufficient spatial resolution small samples must be sectioned from the welded components. The sampling is accompanied by local relaxation of the residual stress distributions that are induced by welding and this can have an impact on the crack shapes prior to the sampling.

The studies are carried out within the scope of developing novel Cr/Ni low transformation temperature (LTT) weld filler materials that mitigate detrimental welding residual stresses (RS) by means of a late martensite transformation [2]. Compared to conventional weld filler materials this can result in compressive RS in the weld line without cost intensive post weld treatments. Because of their dendrite structure and segregation behaviour LTT-alloys show a high hot cracking susceptibility. To provide a sufficient high resolution for  $\mu$ CT analysis it is necessary to cut the investigated specimen into smaller cuboids, with maximum edge length of 1 cm (maximum photon energy of 225 keV). As high compression stresses up to 800 MPa in the area of the crack networks were determined by means of the contour method, stress relaxation during the cutting process affects the detectability of the cracks by  $\mu$ CT. To investigate this effect, the specimens with hot cracks were subjected to a load test with known stress states. The results clearly show that stress relaxations have an strong impact on the volume images reconstructed from tomography analysis and that this effect must be considered during hot crack assessment on basis of  $\mu$ CT data.

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Email corresponding author: Florian.Vollert@kit.edu

## In-situ and quasi in-situ investigation of microstructure evolution of single and multiple additively manufactured SS 308 layers

Strantza M.<sup>1</sup>, Clausen B.<sup>1</sup>, Brown D.W.<sup>1</sup>

<sup>1</sup>Los Alamos National Laboratory, Los Alamos, NM 87545, USA

Additive manufacturing is a novel manufacturing technology that enables a layer-by-layer building methodology in order to build free-form and light-weight structures. Undoubtedly, the promising free-form capabilities are advancing the demands of the industry. However, parameters such as microstructure and residual stresses need to be well-understood in order to control their impact on the mechanical response of the material. Currently, there are studies that have focused on the microstructural [1] and residual stress [2] characterization of the metallic additively manufactured (AM) components. Yet, none of them has focused on the *in-situ* characterization of AM components. This investigation is using high-energy X-ray diffraction in order to profile microstructural variations within a single layer and within successive multiple layers of AM stainless steel 308. The main objective of the current investigation is to capture these variations *in-situ* –during the deposition of the layers- and quasi in-situ -after the solidification and before the deposition of the subsequent layers. Figure 1 shows the weight fraction of the bcc (ferrite) phase of a single layer and after deposition of a second layer. The ferrite phase fraction altered considerably, especially near the interface, from the state of the single layer (Figure 1a.) and after the deposition of the second layer (Figure 1b.). Similar behavior was also observed on the residual strain evolution of the subsequent layers during the *in-situ* and the quasi in-situ characterization. During the in-situ measurements, we obtained the best possible spatial (0.05 mm) and temporal (0.1 sec) resolution given the high-energy incident beam necessary to penetrate the deposited layer and the desire to collect data usable for the determination of lattice strain. It is concluded that it is possible to capture the significant microstructural redistribution of the AM metals by means of in-situ characterization. These data are promising and of high importance for the development of theoretical models of the additive manufacturing process.



*Figure 1. Ferrite-weight fraction evolution of the quasi in-situ investigation of: a. one layer and b. after the deposition of the second layer.* 

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Email corresponding author: maria.strantza@lanl.gov

# Synchrotron X-ray 2D Residual Stress Map in different Laser Shock Peened Edges

Coratella S.<sup>1</sup>, Langer K.<sup>2</sup>

## <sup>1</sup>University of Dayton Research Institute, 1700 South Patterson Blvd, 45409, Dayton, OH - USA <sup>2</sup>Air Force Research Laboratory, ARFL/RQVS, Wright-Patterson AFB, OH 45433

Laser shock peening (LSP) has proved to be a reliable and repeatable technique to introduce deeper and higher compressive residual stresses than most of the comparable surface techniques. While LSP is viable for both thin and thick sections, research is needed to better understand how laser peening interacts with a specimen's edges which, most of the time, act as crack initiators.

The target of this research is to understand the residual stress distributions in three different types of edges: a sharp edge, a concave edge, and a convex edge. For non-flat surfaces, finite element modelling was used to demonstrate that using the same laser parameters on concave and convex geometries leads to different stress distributions [1]. At the same time, recent residual stress measurements demonstrated that the compressive residual stress tend to 0 MPa at the sharp edge of a sample previously laser peened [2].

Three sample geometries were used in this research, all of them made from AA7075, with the same length and section area. The laser peening was processed at the National Laser Centre in Pretoria, SA. The laser parameters were kept constant during the whole peening process and they consist of a power density of 2.6  $GW/cm^2$ , pulse width of 18 ns, and a total coverages density of 500 spots/cm<sup>2</sup>.

Different residual stress measurements were used to map the residual stress fields at the surface and within the thickness. Incremental hole drilling and laboratory X-ray diffraction were used for measurements within the first millimetres from the surface in the depth while neutron diffraction and synchrotron X-ray diffraction were used to map the residual stress t the thickness. The mapping of the residual stress was also achieved via contour method for one stress component only.

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Email corresponding author: Stefano.coratella@ud-research.org

## The influence of erosion wear on the residual stresses in WC-based alloy coatings

Venter A.M.<sup>1,2</sup>, Marais D.<sup>1</sup>, Luzin V.<sup>3</sup>, Sacks N.<sup>4</sup>

<sup>1</sup>Research and Development Division, Necsa SOC Ltd, Pretoria, South Africa <sup>2</sup>DST-NRF Centre of Excellence in Strong Materials, University of the Witwatersrand, Johannesburg, South Africa <sup>3</sup>Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW, 2232 Australia <sup>4</sup>School of Chemical & Metallurgical Engineering, University of the Witwatersrand, Johannesburg, South Africa

Tungsten carbide cermets are known for their high strength, toughness and hardness that in conjunction with superior abrasion resistance leads to their extensive use as cutting tools as they can retain a sharp edge for extended machining times. Research of WC-based hardmetal alloys feature prominently within the DST-NRF Centre of Excellence in Strong Materials. Amongst others, the interests encompass the replacement of WC by similar hard carbides such as VC that have been found to produce a material with improved hardness, comparable toughness and are lighter with potential use in weight limiting applications. Integral to the evaluation of such new alloys are their ability to retain compressive residual stresses and render good abrasion performance.

We report on the evaluation of HVOF (high-velocity-oxygen-fuel) coatings of WC-12wt%Co and WC-10wt%VC-12wt%Co deposited to thicknesses of about 200 microns on mild steel plate substrates using an industrial HVOF system. The wear performances were evaluated on a slurry jet impingement erosion rig in which a stream of silica sand/water is directed at the sample surface at variable impact angles. Results indicated that the mass losses for the 10wt% VC coatings were generally lower compared to that of the WC-12wt%Co coating, which translates to a better wear resistance. Residual stresses were investigated in the wear scar regions using the neutron strain scanner instruments MPISI and KOWARI. Two approaches were followed respectively referred to as the indirect and direct approaches. In the indirect approach that was employed on the MPISI instrument, the investigations focused on the stress behaviours in the substrates by employing a fine measurement through-thickness mesh strategy from which the coating stress could be determined by imposing stress balance in the coating/substrate system. This required accurate sample alignment and many through-thickness data points to compensate for the unfavourable thickness ratio between the coating (0.2 mm) and the substrate (e.g. 4 mm). In the second approach that was performed on the KOWARI instrument, the stresses were measured directly in the coatings. Notwithstanding the direct measurement capability, prolonged measurement times still had to be applied to accumulate adequate diffracted intensity from the thin coating for the calculation of stresses. Investigations were subsequently limited to a subset of samples. The neutron diffraction results indicate that the erosion impact angles have an influence on the residual stresses and the coating composition.

Email corresponding author: Andrew.venter@necsa.co.za

## \*Effectiveness of Laser Shock Peening in Post processing Additive Manufactured Ti-6Al-4V

Ivanovic N.<sup>1,2</sup>, Polese C.<sup>1,2</sup>, Yadroitsev I.<sup>3</sup>

 <sup>1</sup>School of Mechanical, Industrial and Aeronautical Engineering, University of the Witwatersrand, 1 Jan Smuts Avenue, Johannesburg, 2000, South Africa
<sup>2</sup>DST-NRF Centre of Excellence in Strong Materials, University of the Witwatersrand, 1 Jan Smuts Avenue, Johannesburg, 2000, South Africa
<sup>3</sup>Centre for Rapid Prototyping and Manufacturing, Central University of Technology, 20 President Brand Street, Bloemfontein, 9300, South Africa

While metal additive manufacturing (Direct Metal Laser Sintering) presents the benefit of complex geometrical design, it also results in components with high residual stresses. These need to be addressed with stress relief treatment. Components still contain surface tensile residual stresses which can be detrimental to the fatigue performance [1]. Laser shock peening induces deep fatigue-beneficial compressive residual stress in the material. It is important to see the effectiveness of LSP as a post processing treatment in additive manufactured parts due to their well-established susceptibility to fatigue failure attributed to their high surface roughness.

LSP without ablative coating is to be performed on samples of AM Ti-6Al-4V in order to determine its effectiveness in inducing beneficial compressive residual stress without compromising surface integrity. Residual stress measurement will be performed using Synchrotron XRD on thinner samples and incremental hole-drilling on thicker samples. The samples will be analysed under scanning electron microscope before and after laser shock peening. Preliminary tests have shown that LSP on titanium results in significant surface oxidation as the water inertial barrier dissociates into its elements. In the presence of this oxide layer it is important to develop a method of reliable surface preparation for mounting strain gauges in samples to be measured using incremental hole-drilling. Since XRD measures stresses to a much smaller depth than incremental hole-drilling, surface treatment will not be performed on these samples in order to preserve the characteristic residual stress induce by LSP. It is also important to consider the effect the oxide layer has on XRD measurement and attenuation depth.

The study will contribute towards a further understanding of LSP on AM titanium alloy, and the challenges presented by the residual stress measurement techniques involved. The aim of the study is to learn to reliably induce compressive residual stress in critically fatigued locations in complex AM parts.

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Email corresponding author: 363397@students.wits.ac.za

# Numerical modelling and performance effects of laser deposited Ti-Al-Sn coating on ASTM A29 steel

Fatoba O.S.<sup>1</sup>, Akinlabi S.A.<sup>2</sup>, Akinlabi E.T.<sup>1</sup>

<sup>1</sup>Department of Mechanical Engineering Science, Faculty of Engineering and the Built Environment, University of Johannesburg, South Africa. <sup>2</sup>Department of Mechanical and Industrial Engineering Technology, Faculty of Engineering and the Built Environment, University of Johannesburg, South Africa.

The conventional surface modification and coating cannot always fulfil the performance of material surface under extreme corrosion and wear environments. Corrosion and wear phenomenon lead to the gradual deterioration of components in industrial plants that can result in loss of plant efficiency, and even total shutdown with aggravated damage in industries such as petrochemical, oil and gas, marine and ship building. Hence, surface modification by incorporating chemical barrier coatings can be beneficial to this extent we report on investigation aimed at enhancing the surface properties of ASTM A29 steel by incorporating Ti-Al-Sn coatings deposited by laser deposition technique. For this purpose, a 3 kW continuous wave ytterbium laser system attached to a KUKA robot which controls the movement during the alloying process was utilized to deposit coatings with stoichiometry 50Ti-30Al-Sn and 60Ti-20Al-Sn. The alloyed surfaces were investigated in terms of its hardness and wear behaviour as function of the laser processing conditions. Hardness measurements were done using a vickers micro-hardness tester model FM700. Wear tests were performed on prepared steel substrate deposited sample using the reciprocating tribometer (CERT UMT-2) under dry reciprocating conditions with continual recording of the dynamic coefficient of friction (COF) values. The microstructures of the coated and uncoated samples were characterized by optical and scanning electron microscopy. In addition, X-ray diffraction was used to identify the phase's contents. The optimum performances were obtained for an alloy composition of 60Ti-20Al-Sn, at laser power of 750 W and coating speed of 0.8 m/min. Its performance enhancement compared to the unprotected substrate comprised a significant increase in hardness from 115 to 509 HV and reduced wear volume loss from 0.717 to 0.053. The enhanced performance is attributed to the formation of the intermetallic phases Ti<sub>6</sub>Sn<sub>5</sub>, AlSn<sub>2</sub>Ti<sub>5</sub>, Ti<sub>3</sub>Al, and TiAl. The experimental results directly correlate with a numerical grey relational model (GRM) that predicted 1.000 for an alloy composition of 60Ti-20Al-Sn. Basically, the larger the grey relational grade, the closer will be the product quality to the ideal value. Thus the larger grey relational grade is desired.

Email corresponding author: drfatobasameni@gmail.com

## \*Depth-resolved strain investigation of plasma sprayed hydroxyapatite coatings exposed to simulated body fluid

Ntsoane T.P.<sup>1,2</sup>, Topic M.<sup>3</sup>, Theron C.<sup>2</sup>, Härting M.<sup>4</sup>, Heimann R.<sup>5</sup>

<sup>1</sup>Department of Physics, University of Pretoria, Pretoria, South Africa <sup>2</sup> Research and Development Division, NECSA SOC Limited, Pretoria, South Africa <sup>3</sup> Materials Research Department, iThemba LABS/National Research Foundation, Cape Town, South Africa <sup>4</sup> Department of Physics, University of Cape Town, Cape Town, South Africa <sup>5</sup> Am Stadtpark 2A, D-02826 Görlitz, Germany

The influence exposure to simulated body fluid (SBF) has on plasma sprayed hydroxyapatite (HAp) coatings on medical grade Ti6Al4V samples has been investigated. Through-thickness residual strain investigation of HAp coatings deposited on flat substrate surface incubated for 7, 28 and 56 days were performed using high-energy synchrotron diffraction technique.

In the as-spayed condition, the results show the top half of the HAp coating to be under compression with the maximum around the near-surface region relaxing with depth below the surface reaching a strain- free point around the coating thickness midpoint. On the contrary the remainder of the coating is under tension increasing with further depth; the maximum tension is observed near the coating-substrate interface region.

Upon immersion in SBF, the as-sprayed strain profile was retained with both the strain gradient and nearsurface strain relaxing before saturating at 28 days, see Figure. 1; the highest change was observed within the first week of incubation. The observed immersion profile agrees with earlier work performed on HAp coating deposited on a substrate of different geometry [1]. A similar trend was observed for FWHM indicating possible relaxation of micro strains. Results of cross section SEM examination will also be presented.



Figure 1. Immersion profile of HAp coating: a) near-surface strain and b) strain gradient.

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Email corresponding author: Tshepo.ntsoane@necsa.co.za

## \*Effect of Varying Laser Shock Peening Parameters on Residual Stresses in Different Thicknesses of Aluminium Alloy Samples

Van Staden S.N.<sup>1,2</sup>, Polese C.<sup>1,2</sup>, Glaser D.<sup>3</sup>, Plaisier J.R.<sup>4</sup>

 <sup>1</sup>School of Mechanical, Industrial and Aeronautical Engineering, University of the Witwatersrand, 1 Jan Smuts Avenue, Johannesburg, 2000, South Africa
<sup>2</sup>DST-NRF Centre of Excellence in Strong Materials, University of the Witwatersrand, 1 Jan Smuts Avenue, Johannesburg, 2000, South Africa
<sup>3</sup>CSIR National Laser Centre, Meiring Naudé Road, Brummeria, Pretoria, 0184, South Africa
<sup>4</sup>Elettra – Sincrotrone Trieste S.C.p.A, Strada Statale 14 – 14km 165,5 in AREA Science Park, 34149 Basovizza, Trieste, Italy

Laser Shock Peening (LSP) is a surface treatment technique that has been shown to improve the fatigue life of various metal components by the introduction of compressive residual stresses [1]. LSP has a number of parameters than can be varied, such as power density, spot size and coverage. Researchers in the LSP field are interested in the effect that these parameters have on the residual stress distribution through the depth, such as the value at the surface, the maximum value of the residual stress and the depth to which the residual stress is compressive, shown in Figure 1. For this reason it is important to measure accurately the residual stress field in samples that have been treated by LSP and to characterise the effect of varying these parameters.



Figure 1. An LSP residual stress profile indicating the points of interest to LSP researchers

LSP has shown promise in the aeronautical industry so a study aimed at characterising residual stresses in different thicknesses of laser peened AA7075-T6 (a standard aeronautical grade alloy) is ongoing. A preliminary study was conducted to determine the robustness of incremental hole-drilling residual stress results in thick and thin samples. Synchrotron XRD results were obtained as a benchmark at Elettra, Italy. The study found that, for the same LSP parameters, the residual stress decreased with thickness and also that using hole-drilling to measure residual stresses in thin samples caused an underestimation of the results [2]. Residual stresses need, therefore, to be measured by various complementary techniques and hole-drilling on thin samples requires the use of special calibration coefficients which need to be experimentally validated.

This paper presents research on 10mm and 1.6mm thick AA7075-T6 samples treated with different LSP parameters (which will be varied according to a factorial Design of Experiments algorithm) and the residual stresses will be evaluated. These will be measured near the surface and through the depth using different complementary techniques such as Incremental Hole-Drilling, laboratory XRD, Neutron Diffraction and Synchrotron XRD. The results will be analysed within the modeFRONTIER multi-objective optimisation platform and the Synchrotron XRD will be used to validate the hole-drilling results for thin samples.

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Email corresponding author: sean.vanstaden2@students.wits.ac.za

## \*Preliminary Investigation of Part Refurbishment Using Laser Shock Peening

Eisensmith D.<sup>1</sup>, Palazotto A.<sup>1</sup>, Coratella S.<sup>2</sup>, Langer K.<sup>3</sup>, Spradlin T.J.<sup>3</sup>

<sup>1</sup>Air Force Institute of Technology, Wright-Patterson AFB, OH 45433 <sup>2</sup>University of Dayton Research Institute, 1700 S Patterson Blvd, Dayton, OH 45409 <sup>3</sup>Air Force Research Laboratory, ARFL/RQVS, Wright-Patterson AFB, OH 45433

More than half of aircraft structural component failure can be attributed to fatigue [1]. One option for mitigating fatigue damage is the introduction of compressive residual stress via surface treatments such as cold working, surface burnishing, and laser shock peening (LSP). Laboratory experiments and in-service experience have demonstrated significant fatigue life increases with these techniques, on the order of 300% or more, across a wide range of materials and components; however, the majority of these successes, in particular with respect to LSP, have been for components with little to no pre-existing usage and/or very small initial flaws.

In this paper, we discuss a preliminary exploration of LSP applied over an existing surface crack just under the flaw size inspection limit. The intent was to better understand the consequences of LSP as a preventative repair for fatigue-prone locations in aluminum airframe.

Two variations of LSP were applied to pre-cracked specimens and then constant amplitude fatigue testing was conducted. Marker banding was used to mark the start of the crack growth and to track progression. Four peened specimens were reserved for residual stress evaluation using both mechanical and diffraction techniques.

The stress measurements showed significant compressive stress for both LSP variations, which resulted in zero crack growth to fatigue runout. While the unpeened specimens failed between 43000-53000 cycles, the laser peened specimens were subjected to 1,000,000 cycles without failure. In the planned follow-on work, the effects of different initial crack lengths and LSP treatments will be considered.

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Email corresponding author: david.eisensmith@us.af.mil; Alt. Email: kristina.langer@us.af.mil

# An update on the Engineering materials programme at ISIS neutron source: current status and future plans

Kabra S.<sup>1</sup>, Kockelmann W.<sup>1</sup>, Kelleher J.<sup>1</sup>, Burca G.<sup>1</sup>, Minniti T.<sup>1</sup>, Lee T.L.<sup>1</sup>

<sup>1</sup>ISIS Neutron Source, STFC, Harwell campus, Didcot, OX11 7RU, UK

The recent developments within the Engineering materials programme at ISIS neutron source will be reported. The ISIS facility has two dedicated neutron instruments to study engineering materials. It is supported by a vibrant and active UK and European scientific user communities. The facility also serves a growing number of Industrial partners through the industrial collaborative scheme.

The ENGIN-X instrument has been the workhorse instrument of engineering materials programme for the last 15 years. It has been optimised for measurements on engineering scale samples and sample environment. The instrument which boasts one of the best resolutions of any neutron instrument is predominantly used for residual strains and in-situ loading measurements. IMAT is an imaging/diffraction instrument which has recently been commissioned. IMAT is a flexible instrument which can be used in various imaging and diffraction modes e.g. energy dispersive imaging with small FOV, energy selective imaging with large FOV, energy dispersive diffraction etc. It also offers the possibility of simultaneous imaging and diffraction and a sample space which allows for bigger and heavier industrial samples. In order to cater to ever increasing academic and industrial demand, we are proposing a third instrument, EMAP which will primarily be a high flux strain measurement instrument. Some of the initial design concepts will be presented.

We have continued to make investments in software developments in view of a user friendly experimental experience. The Mantid framework is now being trialled on ENGIN-X and IMAT. Several developments related to positioning and the SSCANSS software will also be reported. Further developments in sample environment and novel techniques (notably stroboscopic) will be discussed and a small number of case studies will be presented.

Email corresponding author: saurabh.kabra@stfc.ac.uk

## Improving the efficiency in energy-dispersive residual stress gradient analysis: How to gain maximum benefit with minimal expense?

Klaus M., Apel D., Meixner M., Genzel C.

## Helmholtz-Zentrum Berlin für Materialien und Energie, Germany.

X-ray residual stress analysis (XSA) is usually based on experimental techniques that require the measurement of lattice strains  $\varepsilon_{\varphi\psi}^{hkl}$  in many directions ( $\varphi, \psi$ ) with respect to the sample reference system. Depending on the evaluation strategy, these data are used to calculate either individual values or complete depth profiles for different stress components  $\sigma_{ij}$  within the irradiated near surface depth range. In particular if steep stress gradients are expected to be present within the information depth of the X-rays, a dense network of strain data needs to be measured under grazing diffraction conditions very close to the surface [1]. Therefore, XSA usually is much more time consuming than other methods in material analytics such as Raman spectroscopy or many methods for mechanical testing. Consequently, most of the XSA methods, especially if applied under low flux laboratory conditions, are not suited for the investigation of large sample series being required in process control and material development.

Driven by the demand from our industrial partners for XSA methods that combine the unique features of diffraction to be nondestructive, phase- and depth-selective with the feature to provide results in a short time, we made efforts in the past to develop some new approaches which exploit the features of energy-dispersive (ED) diffraction. The ED method applied in reflection geometry provides diffraction patterns in fixed but arbitrary scattering directions with a multitude of diffraction lines  $E^{hkl}$ , each of them originating from different average depths  $\langle \tau^{hkl} \rangle$  below the surface and reflecting the material's elastic anisotropy on different length scales. Keeping in mind these features our attempts at reducing the experimental expenditure had the focus on minimizing the number of measuring directions being required for a robust residual stress analysis.

In the lecture several strategies are introduced which exploit the information contained in the ED diffraction patterns in the way described above. Based on the data evaluation concept underlying the scattering vector method [2] we show that two ED spectra recorded (simultaneously) under different inclination angles  $\psi$  are sufficient for a depth-resolved analysis of the in-plane residual stress state in a polycrystalline sample. The approach is demonstrated to be expandable to XSA of very coarse grain and single crystalline materials by combining the scattering vector concept with the crystallite group method introduced by HAUK and coworkers [3]. On the other hand, assuming the residual stress state within the diffracting volume to be homogeneous, even one ED measurement performed under  $\psi = 0^{\circ}$  is shown to provide enough information for a self-consistent stress evaluation, if the effect of elastic anisotropy is taken into account.



Figure 1. Schematic view of the 8-circle diffractometer LEDDI for ED residual stress-, texture- and microstructure analysis.

In order to verify the concepts mentioned above as well as various other measuring and data evaluation strategies based on ED diffraction, we developed a new type of ED 8-circle diffractometer equipped with two detectors for simultaneous data acquisition (Fig. 1). Thus, it can be considered as a prototype for later 'slimmed-down' versions to meet the industrial demand for fast nondestructive XSA experiments.

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Email corresponding author: klaus@helmholtz-berlin.de

## Scanning X-ray Nanodiffraction – from strain mapping to in situ microscopy

Krywka C.<sup>1,2</sup>, Davydok A.<sup>1</sup>, Mueller M.<sup>1,2</sup>

<sup>1</sup>Helmholtz-Zentrum Geesthacht, Institute for Materials Research (outstation at DESY), Notkestraße 85, 22607 Hamburg, Germany <sup>2</sup>Christian-Albrechts University of Kiel, Institute for Experimental and Applied Physics, Leibnizstraße 19, 24118 Kiel, Germany

Scanning X-ray nanodiffraction is an excellent tool for materials science related in situ studies. It readily serves structural information with sub-µm spatial resolution from crystalline and semi-crystalline materials (metals, biomaterials, synthetic compounds). That way grain orientation, residual stress profiles, crystal structure or texture can be obtained in a non-destructive analysis. Because of the long focal distance focusing, the wide X-ray energy range and a flexible sample positioning system, high resolution nanodiffraction experiments can be performed even under demanding conditions e.g. on strongly absorbing metallic samples or in extended sample environments.

The Nanofocus Endstation of beamline P03 (PETRA III, Hamburg) is part of the German Engineering Materials Science Center (GEMS) and is operated jointly by Helmholtz-Zentrum Geesthacht and the University of Kiel. It is one of only few places in the world where the experimental conditions for scanning X-ray nanodiffraction are provided and it offers a sub-micron sized, hard X-ray beam. The strong focus on materials science at P03 is demonstrated by the wide range of experiments already performed with in situ sample environments: pressure, indentation force, tensile stress, fluid shear, magnetic fields - all of these parameters were successfully modified in situ and combined with the high spatial resolution provided by nanofocused beam.

This contribution will provide a comprehensive introduction to the experimental facilities at P03 and showcase a compilation of past experiments where in situ applications have been combined with X-ray nanodiffraction.

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Email corresponding author: christina.krywka@hzg.de

## Precision and accuracy of stress measurement with a portable X-ray machine

Lee S.-Y.<sup>1</sup>, Ling J.<sup>2</sup>, Wang S.<sup>1</sup>, Ramirez-Rico J.<sup>2</sup>, Noyan I.C.<sup>1</sup>

<sup>1</sup>Applied Physics and Applied Mathematics, Columbia University, New York, NY, 10027, USA <sup>2</sup>Fisica de la Materia Condensada – ICMS, Universidad de Sevilla – CSIC, 41012 Sevilla, Spain

The use of portable X-ray stress analyzers, which utilize an area detector along with a newly adopted ' $\cos \alpha$ ' or full-ring fitting method, has recently gained interest. In laboratory conditions, these measurements are fast, convenient, and precise because they employ a single exposure technique that does not require sample rotation. In addition, the effects of grain size and orientation can be evaluated from the Debye ring recorded on the area detector prior to data analysis. Accuracy of the measured stress, however, has been questioned because in most cases just a single reflection is analyzed and sample-to-detector distances are relatively short. We present our recent article, a comprehensive analysis of the uncertainty associated with a state-of-art commercial portable X-ray device [1]. Annealed ferrite reference powders were used to quantify instrument precision, while accuracy of the stress measurement was tested by in-situ tensile loading on 1018 carbon steel and 6061 aluminium alloy bar samples. Results show that precision and accuracy are sensitive to the instrument (or sample) tilt angle ( $\psi_0$ ) as well as to the selected hkl reflection of the sample. The instrument, sample, and data analysis methods all affect the overall uncertainty and each contribution is described for this specific portable X-ray system. Finally, based on our conclusions, desirable measurement/analysis protocols for accurate stress assessments are also presented.



Figure 1. A schematic diagram of the portable X-ray machine used in this study (left) and a typical diffraction pattern of steel (211) peak measured by this device (right).

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Email corresponding author: SL3274@columbia.edu

## Alignment and calibration procedures of the Necsa neutron strain scanner

Marais D.<sup>1</sup>, Venter A.M.<sup>1</sup>, Luzin V.<sup>2</sup>

<sup>1</sup>Research and Development Division, Necsa SOC Limited, Pretoria, South Africa <sup>2</sup>Australian Centre for Neutron Scattering, ANSTO, Lucas Heights, NSW, Australia

The Materials Probe for Internal Strain Investigations (MPISI) neutron diffraction strain scanner is situated at the SAFARI-1 research reactor of the South African Nuclear Energy Corporation (Necsa). After recent major upgrade, a commissioning phase followed to meet the instrument positional/rotational accuracy and precision requirements. This presentation reports the alignment and calibration procedures established for the purpose of instrument optimisation and performance, which includes the following:

(*i*) Sample table centre-of-rotation (CoR). Automated determination of the CoR using either a digital dial gauge [1], or a telecentric camera system [2], in conjunction with an accurately machined calibration cell was developed. By minimising deviations upon cell rotation at three rotational positions  $45^{\circ}$  apart (procedure followed when limited sample rotation is possible), an error (cell deflection) of ~50 µm was achieved. This error can be reduced to ~10 µm by using four rotational positions 90° apart. Further alignment aids implemented include diode lasers and theodolites aligned to the CoR.

(*ii*) Doubly focused multi-wafer silicon single crystal monochromator. Accurate alignment is achieved through high-precision positioners having five degrees of freedom. Since the crystals have the Si[101] axis vertical and [110] axis horizontal, different reflections (providing a selection of wavelengths) can be accessed through the 83.5° chamber exit port by its horizontal rotation. Instrumental resolution is a function of (amongst others) the crystal horizontal focus and was optimized by maximising the figure-of-merit [3] performance for diffraction angles close to 90°. Alignment of the monochromator diffraction plane vertically to the centre of the detector was done by ensuring symmetry in low-angle Debye-Scherrer cones.

(*iii*) Detector offset. Using the Si(331) monochromator reflection in conjunction with an Al<sub>2</sub>O<sub>3</sub> NIST standard powder, diffraction patterns were measured as 7 separate detector frames covering the range  $28.05^{\circ} \le 2\theta \le 106.95^{\circ}$ . Corrected data, stitched using the in-house designed data reduction software ScanManipulator [4], were used to determine the detector zero-offset and neutron wavelength to be -1.78° and 1.646 Å using a Rietveld assessment. This indicates that the monochromator take-off angle was 82.7°, being within 1° of the intended angle of 83.5°.

(*iv*) *Gauge volume*. The vertical centre of the beam (without any apertures in place) was determined by stepscanning a mild steel bar vertically through the beam. The instrument lasers and theodolites were then aligned to the bar centre which were used in turn to vertically align the apertures. By positioning the calibration cell at the CoR, the required horizontal offsets of both the primary and secondary apertures is routinely evaluated to an accuracy of ~10  $\mu$ m through neutron beam step-scanning. The vertical and horizontal beam divergences as a function of primary slit distance from the CoR were also determined.

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Email corresponding author: Deon.Marais@necsa.co.za

# \*Neutron Transmission Strain Measurements on IMAT: Residual Strain Mapping in an AlSiC<sub>p</sub> Metal Matrix Composite

Ramadhan R.S.<sup>1</sup>, Kockelmann W.<sup>2</sup>, Tremsin A.S.<sup>3</sup>, Fitzpatrick M.E.<sup>1</sup>

## <sup>1</sup>Centre for Manufacturing and Materials Engineering, Coventry University, Coventry, CV1 2JH, UK <sup>2</sup>STFC, Rutherford Appleton Laboratory, ISIS Facility, Chilton, OX11 0QX, UK <sup>3</sup>Space Science Laboratory, Univ. of California at Berkeley, CA 94720 Berkeley, USA.

IMAT (Imaging and MATerial science) is a new imaging instrument at the ISIS pulsed neutron source, UK, with Bragg edge transmission analysis for strain mapping as one of its key features [1]. The instrument is designed to perform strain mapping with sub-mm spatial resolution and good accuracy. The purpose of this study is to conduct strain measurements for the very first time on IMAT, allowing the assessment of the instrument's performance. This study was designed to showcase the IMAT capabilities, as well as provide a benchmark for the further development of strain measurements via neutron imaging.

Strain measurements were performed on a quenched particulatereinforced aluminium/silicon-carbide (AlSiC) metal matrix composite. Owing to rapid cooling on the sample surface, a parabolic through-thickness in-plane strain variation is expected. This behaviour has been proven previously with neutron diffraction measurements on the sample [2]. In-plane strain was measured at IMAT, and Al and SiC powders were used for the strain-free reference  $d_0$  measurement. Transmission spectra were recorded using a microchannel plate (MCP) detector [3]. A comparison is made between residual strain values obtained from neutron transmission and previous literature values obtained using neutron diffraction. The strain maps of the aluminium matrix and silicon carbide reinforcement are reconstructed.



Figure 1. Comparison between diffraction and transmission result (top); strain map (bottom)

Figure 1 shows good agreement between in-plane residual strain values measured with neutron transmission at IMAT and the neutron diffraction results Figure 1 also shows the capability of IMAT in producing strain maps, even from a low-attenuation aluminium sample. Other findings such as the effect of texture on the Bragg edge signal and strain measurement, as well as the proposed ways to minimize (or analyse) these effects are further discussed in the study. This work produced some encouraging results. The preliminary data analysis shows that IMAT is capable to produce accurate strain maps from a challenging sample.

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Email corresponding author: ramadhar@uni.coventry.ac.uk

# In situ x-ray diffraction of shock-driven deformation and phase transformation in titanium

Bolme C.<sup>1</sup>, Lazicki A.<sup>2</sup>, Brown D.W.<sup>1</sup>, Gleason A.<sup>1</sup>, Cerreta E.<sup>1</sup>, Morrow B.<sup>1</sup>, Ali S.<sup>2</sup>, Swift D.<sup>2</sup>, Nagler B.<sup>3</sup>, Galtier E.<sup>3</sup>, Granados E.<sup>3</sup>, Milathianaki D.<sup>3</sup>

<sup>1</sup>Los Alamos National Laboratory, Los Alamos, NM, USA 87545 <sup>2</sup>Lawrence Livermore National Laboratory, Livermore, CA, USA 94550 <sup>2</sup>SLAC National Accelerator Laboratory, Menlo Park, CA, USA 94025

Titanium alloys are employed in demanding engineering applications due to their high strength-to-weight ratio and their resistance to corrosion. Pure titanium and titanium with high levels of oxygen impurities were studied under laser-driven shock compression at the Matter in Extreme Conditions endstation at the Linac Coherent Light Source. *In situ* x-ray diffraction data were acquired during compression and release, showing the lattice-level response of titanium as it underwent plastic deformation and phase transformation. The kinetics of these processes and the influence of oxygen impurities on the deformation behavior will be presented.

Email corresponding author: cbolme@lanl.gov

## \*Modelling pseudo-strain in inhomogeneous and anisotropic materials using Monte-Carlo neutron diffraction simulation

Brokx J.<sup>1</sup>, Moat R.<sup>1</sup>, Bouchard P.J.<sup>1</sup>, Nuttall W.J.<sup>1</sup>, Kelleher J.<sup>2</sup>

## <sup>1</sup>Materials Engineering, The Open University, Walton Hall, Milton Keynes, MK7 6AA, UK <sup>2</sup>Isis, Rutherford Appleton Laboratory, Didcot, Oxfordshire, OX11 0QX, UK

Neutron diffraction has been used by the nuclear industry for measuring residual stresses in structures for which integrity safety cases must be made. However measurement of stresses in materials containing large grains, or in anisotropic weld metal, or approaching air-metal interfaces, or metal-metal interfaces using neutron diffraction is particularly challenging. These types of measurements can give rise to errors (often termed "pseudo-strains") that can be significantly larger than the residual stress actually present. Such errors arise when the gauge volume is partially filled (e.g. air to metal interface for measuring near-surface stresses), or when the gauge volume composition is inhomogeneous (metal to metal interface), or the gauge volume material is anisotropic (welds with bundles of elongated grains). To mitigate such errors several approaches have been proposed [1,2,3] using numerical and analytical methods to calculate the magnitude of pseudo-strain. Whether these methods can be applied depends on the instrument used and the type of sample being measured. In this context a different approach based on the Monte-Carlo method, as embedded in the neutron ray tracing software package McStas [4], is proposed.

The aim is to validate by neutron measurements the ability of McStas to simulate pseudo strains associated with a gauge volume that is partially filled, or a gauge volume that samples inhomogeneous and anisotropic material. To understand and isolate the effects of traversing an interface made up of different materials a stepwise approach is taken. In the first experiment the gauge volume was traversed across the airmetal interface of an aluminium cube filled with iron powder using the ENGIN-X time of flight instrument located at ISIS (UK). A model of ENGIN-X and the powder cube was built in McStas to simulate the physical experiment. A good agreement between the physical measurement and the simulation was



Figure 1. Pseudo-strain powder cube scan

achieved (Figure 1). Further experiments are planned to validate simulated measurements where the gauge volume traverses a metal-metal interface. The sample in this case will be two different materials held together but not welded. A final experiment will simulate neutron measurements traversing a weld. The results will validate McStas neutron ray trace simulations aimed at understanding and quantifying the errors introduced when traversing a gauge volume across material-material boundaries. The findings will support planned best practice guidance for neutron diffraction measurements of dissimilar metal welds widely used in the nuclear industry and beyond.

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Email corresponding author: john.brokx@open.ac.uk

# Influence of hydrogenation on residual stresses in oxygen-implanted Ti-6Al-4V alloy

Nsengiyumva S.<sup>1</sup>, Ntsoane T.P.<sup>2</sup>, Topic M.<sup>3</sup>, Pichon L.<sup>4</sup>

<sup>1</sup>Rhodes University, 6140 Grahamstown, South Africa <sup>2</sup>The South African Nuclear Energy Corporation, Pretoria <sup>3</sup>iThemba LABS, National Research Foundation, P.O. Box 722, Somerset West 7129, South Africa <sup>4</sup>Institute Pprime, UPR 3346CNRS, Université de Poitiers, ISAE-ENSMA, France

We report the influence of hydrogenation on residual stresses in oxygen-implanted Ti-6Al-4V alloy. Prior to hydrogenation, oxygen ions were implanted in Ti-6Al-4V samples at fluence  $3 \times 10^{17}$  ions/cm<sup>2</sup> with energies 50 keV at room temperature and 550°C, 100 keV and 150 keV at 550°C. Hydrogenation was carried out on all samples at 550°C for two hours. Residual stresses were analysed by X-ray diffraction using the sin<sup>2</sup> $\psi$  in conjunction with LEPTOS v6 data reduction. Our results show stress relaxation in samples implanted with 50 keV and 100 keV with respect to unimplanted sample. However, samples implanted with 150 keV show tensile stress state. Subsequent to hydrogenation, a change in stress for samples implanted with 50 keV and 100 keV is observed whereas stress in samples implanted with 150 keV becomes more tensile.

Email: s.nsengiyumva@ru.ac.za

# Recent Progress on Structural Engineering Studies of Reinforced Concrete using Neutron Diffraction

Suzuki H.<sup>1</sup>, Kusunoki K.<sup>2</sup>, Kanematsu M.<sup>3</sup>, Mukai T.<sup>4</sup>

<sup>1</sup> Japan Atomic Energy Agency, Tokai, Naka, Ibaraki, Japan,
<sup>2</sup> The University of Tokyo, Bunkyo-ku, Tokyo, Japan
<sup>3</sup> Tokyo University of Science, Noda, Chiba, Japan
<sup>4</sup> Building Research Institute, Tsukuba, Ibaraki, Japan

The reinforced concrete, which is widely utilized for various architectural and civil engineering structures, is well known as a composite structure in which concrete with relatively low tensile strength and ductility is strengthened by reinforcements such as steel rods (rebars) with high tensile strength and/or ductility. The performance of the reinforced concrete is generally derived from the bond resistance between rebar and concrete, and it is necessary to guarantee the designed seismic performance of the reinforced concrete for expected huge earthquakes to minimize the "megarisk" from the urban earthquake hazard. In general, the bond resistance has been assessed by the axial stress distribution along the rebar measured using strain gauges. However, the waterproof treatment and wiring of the strain gauges around the rebar deteriorate the bond condition itself.

The neutron diffraction technique, which is a nondestructive and noncontact method, is applicable for measuring stress distribution along rebar in the reinforced concrete without any effects on the bond condition. In our previous studies, we have investigated on the potential of the neutron diffraction technique for the strain measurement of the rebar embedded in concrete as an alternative method to the conventional strain gauge. Our first relevant works were carried out using the engineering diffractometer RESA-1 in JRR-3 at JAEA, and we demonstrated that the neutron diffraction technique can be a novel stress measurement technique for rebar embedded in concrete [1]. After that, the three-dimensional deformation behaviour of the embedded rebar including the axial and transverse strains was successfully measured under pull-out loading using Time-of-Flight (TOF) neutron diffraction with TAKUMI in MLF of J-PARC [2, 3]. Additionally, bond deterioration around the cracks in concrete and that around corroded rebar were successfully observed by measuring a change in the stress distributions along rebar [4].

Recently, the neutron diffraction technique has been provided to some applications on the structural engineering studies of the reinforced concrete structure. For example, we applied it to evaluation of the bond condition of the post installed adhesive anchor. The result showed that the stress distributions exhibit different trend depending on the type of adhesive, and the difference in the bond stress calculated by the slope of the stress distribution in the anchorage region is correlated to the difference in their bond strengths. The other topic is evaluation of bending bond behaviour of the reinforced concrete beam reinforced by main rebar and some stirrups. The stress distributions along main rebar as well as stirrups embedded in concrete were successfully measured using neutron diffraction under bending moment, and bond degradation due to flexural cracking was captured by measuring a change in the bond stress distribution. These results suggest that neutron diffraction can provide insight into the real structural engineering issues on the reinforced concrete structures.

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Email corresponding author: suzuki.hiroshi07@jaea.go.jp

# [P01] Multiscale residual stress analysis on metallic wind mill components by combined neutron, X-ray and electron diffraction

Leemreize H.<sup>1,2</sup>, Christensen P. S.<sup>1</sup>, Zangenberg N.<sup>1</sup>, Poulsen H. F.<sup>2</sup>

<sup>1</sup>Danish Technological Institute, Kongsvang Allé 29, 8000 Aarhus C, Denmark <sup>2</sup>Department of Physics, Technical University of Denmark, Fysikvej, 2800 Kgs. Lyngby, Denmark

In metallic structures, an accumulation of lattice strain will, ultimately, result in failure of the entire structure. These lattice strains occur on several length scales varying from sub-grain variations through different strain levels in different adjacent grains to macroscopic strain variations. To properly understand the mechanical performance and predict failure of these metallic structures, a detailed insight into these strain variations on all length scales is hence essential.

The ambition of the project is to demonstrate the use of a combination of diffraction based techniques to probe the lattice strain variations on various length scales by performing analysis on selected components from the wind energy industry. The metallic ball bearings in, e.g., the hub are exposed to huge dynamic forces from the rotating wings. By improving the understanding of the strain and stress variations in the balls and integrating this knowledge into the quality control, the risk of failure can be significantly decreased. Another case component is the hub itself that is a singly casted component (3-5 m in each dimension) and is one of the most expensive components of the entire structure. Because of its complex geometry, the metal casting cooling process leaves significant stresses and strains that are not well understood [1]. From a better understanding of these strains, the material and casting process can be optimized, thus, reducing the cost of this component. Finally, the weldings in the towers and monopiles experience significant and continued stresses during the lifetime of the windmill. These can ultimately result in fatigue fractures. From a better understanding of the strains and hence failure processes, the large risk factors used to estimate the required material thickness could be significantly reduced, which significantly reduces the overall costs of the windmill.

To probe macroscopic strain variations and obtain proper grain statistics, scanning neutron diffraction will be used. Because of the large penetration depth of neutrons (several cm's), this can even be done in reflection mode on arbitrarily large objects. The spatial resolution of this technique is, however, limited to a few mm's. To gain resolution, X-ray diffraction will be used. This can routinely give spatial resolution down to a few µm's and is significantly faster than neutron diffraction. To additionally reduce the scanning time X-ray microscopy (3DXRD) will be used for these experiments. 3DXRD enables mapping of the orientation and strain value of each individual grain within a mm sized 3D sample volume [2,3]. As the sample sizes for 3DXRD are limited to a few mm's, the technique is complemented with the use of neutron scattering to study strain variations in industrial sized components. Using neutron microscopy, strain variations will be studied significantly faster compared to scanning neutron diffraction. To verify the small-scale strain variations, electron diffraction (EBSD) will be used [4]. This technique is more readily available but is limited to mm scans on surfaces giving very limited statistics.

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Email corresponding author: hle@teknologisk.dk

# [P02] Evolution of Internal Stresses during Cyclic Deformation in LPSO Type of Magnesium Alloy Monitored by *In-situ* Neutron Diffraction

Morooka S.<sup>1,2</sup>, Gong W.<sup>3,2</sup>, Aizawa K.<sup>2</sup>, Kawasaki T.<sup>2</sup>, Harjo S.<sup>2</sup>

<sup>1</sup>Materials Sciences Research Center, Japan Atomic Energy Agency, Tokai-mura, Naka-gun, Ibaraki, Japan <sup>2</sup>J-PARC Center, Japan Atomic Energy Agency, Tokai-mura, Naka-gun, Ibaraki, Japan <sup>3</sup>Elements Strategy Initiative for Structural Materials, Kyoto University, Kyoto, Japan

Recently, a series of magnesium alloys containing a 18R Long-Period Stacking Ordered (LPSO) structure, known as the 18R-LPSO structure phase, have attracted considerable attention because they exhibit excellent mechanical properties, including high strength and reasonable ductility [1]. To determine the plastic deformation behaviour of LPSO structure and the origin of the deformation mechanism are important for further materials development. In this study, deformation-kinking (quasi-twinning) behaviour of a Mg-Zn-Y alloy was investigated by *in-situ* neutron diffraction during dynamic cyclic deformation.

The nominal composition of the Mg–Zn–RE alloy used in the present study is  $Mg_{85}Zn_6Y_9$  (at.%). The initial microstructure is almost 18R-LPSO structure. A 3N pure Mg was also prepared for the comparison. The bar type testing specimens for *in-situ* experiments with a gauge length of 6 mm, diameter of  $\varphi$ 5 mm were prepared. *In-situ* TOF neutron diffraction experiments during tension-compression (TC) and compression-tension (CT) cyclic loading at room temperature were performed by TAKUMI (BL19) [2] at MLF of J-PARC. The diffracted neutron data was recorded continuously during the mechanical test in real time by an event mode of data acquisition system. The obtained neutron data was then extracted by changing data slicing time interval after measurement. The cyclic testing was performed using high-precision extensometer under fully-reversed cyclic loading conditions at constant maximum and minimum strain amplitudes of +0.5 % and -0.5 %. The cyclic strain increasing rate was  $2 \times 10^{-4}$  %/sec. TC and CT cyclic data were collected at N= 1, 5, 10, 30, 50, 100, 125, 150, 175 cycles. In a TC cycle, five points along the hysteresis loop were analysed, which are at zero-stress before tension (Pt 0), +0.5 % strain (Pt 1), zero-stress after unloading from +0.5 % strain (Pt 2), -0.5% strain (Pt 3) and zero-stress after unloading from -0.5% strain (Pt 4).

The applied stresses at the maximum tension (Pt 5) and the maximum compression (Pt 3) both increased gradually with increase of cycle up to the  $200^{\text{th}}$  cycle in TC case. Especially, at the  $200^{\text{th}}$  TC cycle, tension and compression peak stresses are approximately +97.4 and -176.7 MPa, respectively. The peak stress in the compression is larger than that of the tension, suggesting that kinking (quasi-twinning) accompanying with hardening was occurred in compression deformation. On the other hand, results of individual <hkl> lattice strains in the loading direction show that, the <60.0> family grains (prismatic plane) in the loading direction accommodated a greater portion of elastic strain than that of the <00.18> family grains in the loading direction start accommodated less portion. This indicated that the latter started to yield prior to that of the former, causing the former to carry more applied load, i.e., intergranular stress. The large intergranular stresses generated with the progress of deformation, were speculated to accelerate the backward plastic flow that resulting in an increase of the Bauschinger stress and Bauschinger strain.

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Email corresponding author: morooka.satoshi@jaea.go.jp

# [P03] Examination of deformation mechanisms of magnesium AZ31- in situ X ray diffraction and modeling

Wroński M., Wierzbanowski K., Baczmański A., Wroński S., Wojtaszek M., Łabaza A., Muzyka M.

AGH University of Science and Technology, Faculty of Physics and Applied Computer Science, 30-059 al. Mickiewicza 30, 30-059 Kraków, Poland

One observes a growing interest in magnesium and its alloys in the last decade (e.g., [1,2]). Magnesium thanks to its unique properties, finds many technological application. It has an advantageous ratio of the yield strength and mass density. The disadvantage of this material is a low ductility at room temperature, which severely limits its formability. Therefore, it is necessary to better understand the mechanisms of plastic deformation in magnesium, which may help to optimize the formability.

The samples of rolled commercial AZ31 magnesium were cut from as received sheet along rolling direction (RD) and prepared for X-ray and EBSD measurements. The initial microstructure, orientation maps and texture were determined using these techniques.

A more detailed insight into material structure can be done by internal stress measurements (e.g., [3,4]). The diffraction in-situ experiments were performed using Xray Cu radiation. The samples were stretched along RD and *in situ* X-ray measurements were performed. The macroscopic stress-strain curves were examined in order to find basic mechanical material parameters. Next, the lattice strains for different *hkl* reflections were determined in function of applied tensile stress



Fig.1. Example lattice strains vs. macroscopic load for 102, 103 and 106 reflections.

component. The obtained experimental results were compared with the predictions of elasto-plastic selfconsistent model – Fig.1. Basing on this data the activity and critical resolved shear stresses (CRSS) for slip and twinning systems were evaluated. Generally, it was found that mainly slip systems are responsible for the observed material deformation: the most active are the slip systems (listed according to increasing CRSS): basal, prismatic, pyramidal  $\pi <a>$ , pyramidal  $\pi_1 <c+a>$  and pyramidal  $\pi_2 <c+a>$ . The twinning systems (compressive and tensile) played only a secondary role.

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Email corresponding author: wierzbanowski@fis.agh.edu.pl

# [P04] Investigation of lattice strain in Mg-alloy and Al/SiC using in-situ TOF neutron diffraction

Wroński M.<sup>1</sup>, Baczmański A.<sup>1</sup>, Wierzbanowski K.<sup>1</sup>, Wroński S.<sup>1</sup>, Gadalińska E.<sup>2</sup>, Scheffzük C.<sup>3,4</sup>, Kot P.<sup>1</sup>

 <sup>1</sup>AGH University of Science and Technology, Faculty of Physics and Applied Computer Science, al. Mickiewicza 30, 30-059 Kraków, Poland
<sup>2</sup>Institute of Aviation, al. Krakowska 110/114, 02-256 Warszawa, Poland
<sup>3</sup>Karlsruhe Institute of Technology, Institute for Applied Geosciences, Kaiserstr. 12, 76131 Karlsruhe, Germany
<sup>4</sup>Frank Laboratory of Neutron Physics, JINR Dubna, 141980 Dubna, Russia

This work presents investigation of the mechanisms of plastic deformation and the evolution of lattice strain in polycrystalline magnesium AZ31 and in two phase composite Al/SiC (17% of SiC). Magnesium alloys belong to the group of lightweight materials and they have unique properties, such as high specific strength (the ratio of the yield stress to density), superior damping capacity and high thermal conductivity. These properties predetermine magnesium alloys for use in many structural applications, such as in the automotive and aerospace industries. The main restriction to a wider use of magnesium alloys is their limited ductility and poor formability at ambient temperature. Another materials investigated in present work are the metal matrix composites (e.g., Al/SiC) in which the ceramic reinforcement (SiC) improves significantly mechanical properties (stiffness and hardness) of Al and does not increase mass density of the final material. Applications of metal matrix composites are widespread in the aerospace and automotive industries where a reduction of weight of a vehicle will reduce its fuel consumption. The Al/SiC composite has high thermal conductivity and is also used in microelectronics.

Anisotropic elastic and plastic properties of magnesium alloy and Al/SiC composite were examined by performing loading tests and *in-situ* observations of lattice strains in different macroscopic. The neutron diffraction time of flight (TOF) method was applied during *in-situ* compression test. The lattice strains for groups of grains selected by different *(hkl)* reflections were measured. The aim of the study is determination of stress localisation and elastoplastic interactions between grains with different orientations of crystal lattice with respect to the applied load. Experiments were performed using EPSILON-MDS instrument (FLNP, Dubna). The advantage of this instrument is the possibility of *in-situ* measurements of lattice strains using nine detector blocks, positioned at  $2\theta$ =90°. Therefore, strains in nine directions for several *(hkl)* crystallographic planes in the range of d<sub>hkl</sub>: 0.8-2.7Å can be simultaneously detected. The results will be compared with elastoplastic model in order to find parameters determining plastic deformation of magnesium alloy and Al/SiC composite.

Email corresponding author: marcin.wronski@fis.agh.edu.pl

# [P05] Residual stress distribution and its influence on the strain evolution along the crack opening direction

Kim D.-K.<sup>1</sup>, Woo W.<sup>1</sup>, Wang H.<sup>1,2</sup>, Yeol Lee S.<sup>2</sup>, Baek An G.<sup>3</sup>

<sup>1</sup>Neutron Science Division, Korea Atomic Energy Research Institute, Daejeon, 34057, Korea <sup>2</sup>Department of Materials Science and Engineering, Chungnam National University, Daejeon 34134, Korea <sup>3</sup>Department of Naval Architecture & Ocean Engineering, Chosun University, 61452, Korea

Residual stress distributions were measured around the crack tip of compact tension (CT) specimens using neutron diffraction to examine the lattice strain evolution along the crack-opening direction under tensile loading conditions. Firstly, 6.35 mm thick CT specimens were pre-fatigue cracked to grow the crack length to 10 mm from the machined notch. Local out-of-plane compression (LOPC) testing scheme, which is a sidepunching onto both surfaces of the CT specimens, was employed to generate tensile or compressive residual stresses near the cracks. The CT specimens were pressed to 400 kN using a 30 mm diameter tool bar. One sample was pressed with the tool centre located on the crack edge causing a crack edge compression. The other was pressed with the tool edge located on the crack edge causing a crack edge tension. The residual stress distribution was confirmed by neutron diffraction measurements. As a result, three kinds of CT specimens were prepared; (i) standard state having only the pre-crack, (ii) pre-crack edge compression, and (iii) pre-crack edge tension. Secondly, in order to investigate the effects of residual stresses on the crack opening and propagation of the CT specimen, in situ neutron diffraction was applied for the measurements of the lattice strains along the crack-opening direction under loading. Tensile loads of 0, 5, 10, and 15 kN were imposed on the CT specimen along the crack-opening direction and the (211) diffraction peak were mapped around the crack tip, respectively. The results show that the edge compression prevents the evolution of crack opening, whilst the edge tension significantly promotes the crack opening and propagation. Details of the lattice strain evolution near the crack edge and possible residual stress effects on the crack propagation path and fracture toughness will be further discussed in the presentation.

Email corresponding author: chuckwoo@kaeri.re.kr

# [P06] Microstresses in thermally stable diamond composites made by high pressure infiltration technique

Luzin V.<sup>1</sup>, Voronin G.<sup>2</sup>

## <sup>1</sup>Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW, 2232 Australia <sup>2</sup> Smith MegaDiamond Inc, Schumberger Co, Provo, UT 84604, USA

Polycrystalline diamond (PCD) based composite materials are being increasingly used as cutting tools in the mining industry. While traditional PCD composites use Co as the binder phase, which restricts the operational temperature by approximately 800°C, the most modern type of thermally stable diamond composites (TSDC) do not have metal binder permitting much higher operational temperatures. Instead, TSDCs have SiC as a binder and such composites are usually produced by reactive sintering. Several methods differing by pressure and temperature conditions have been developed with time. One such technique, the high pressure infiltration, is characterized by high temperature (1800-2000°C) and high pressure (8-10 GPa) at which liquefied Si is delivered into diamond grit to initiate sintering.

Microstresses in both phases, diamond and SiC, in the TSDCs produced by high pressure infiltration technique were measured using neutron diffractometer KOWARI at OPAL research reactor. Microstresses are developed as result of quenching from high sintering temperature and their magnitude is determined by the thermo-mechanical properties of SiC matrix and diamond grit, pressure and temperature conditions as well as the exact TSDC phase composition. The experimental results were interpreted in terms the "matrix-inclusion" composite model that was used to evaluate the composite structural integrity.

Email corresponding author: vladimir.luzin@ansto.gov.au

# \*[P07] Residual Stress Investigation of the Orapa AK 1 and Letlhakane DK 1 mines, Red mudstones

Kgaodi O.<sup>1</sup>, Oladijo P.<sup>2</sup>, Seitshiro I.<sup>1</sup>, Keitumetse M.<sup>3</sup>, Ntsoane T.P.<sup>4</sup>

<sup>1</sup>Department of Mining and Geological, Botswana International University of Science and Technology, Palapye, Botswana <sup>2</sup>Department of Chemical, Material and Metallurgical Engineering, Botswana International University of Science and Technology, Palapye, Botswana <sup>3</sup>Debswana Diamond Company, Gaborone, Botswana <sup>4</sup>Research and Development Division, Necsa Limited, Pretoria, South Africa

Orapa AK 1 and Letlhakane DK 1 mines are one of the Debswana Orapa, Letlhakane and Damtshaa Mines (OLDM) diamond operations in Botswana. The AK1 and DKI mines are currently experienced several slope failures at different benches of the pit due to the slaking of the red mudstone. The scope of this project is to study the residual stresses present in the red mudstone (i.e. AK1 and DK1 rock) samples using X-ray diffraction (D8 discovery) in the raw as-discovered forms. It is thought that residual stress analyses could give clues on the rock strength of their formation.

Studies performed on the red mudstone rock include microstructure investigation using scanning electron microscopy equipped with EDS. Chemical phase identification by XRD. The tensile and uniaxial compression strength was also determined. The study reveals the red mudstone comprises predominantly quartz, calcite, clay minerals and other minor elements. The residual stress analysis reveals higher compressive stress on DK 1 than AK 1 stone. However, tensile stresses were found on some spot on AK 1 due to the impurities and origin of the rock. There was a correlation between the residual stresses measured and the tensile strength which were distinctly discussed.

Email corresponding author: seyiphilip@gmail.com

# [P08] The effect of nano particles on serration phenomena for 18Mn-0.6C twinning-induced plasticity steel

## Shin E., Woo W.

### Korea atomic Energy Research Institute, Daejeon, Korea

The nature of plastic deformation of high-manganese steel is mechanical twinning by lower levels of stacking fault energy, so it is referred to as twinning-induced plasticity (TWIP) steel. And another unique characteristic of TWIP steel is its serration phenomena upon strain, which is referred to as dynamic strain aging (DSA) based on the Portevin-LeChatelier effect. Such prominent serration affects various bulk mechanical properties in TWIP steels, for example, increases of the flow stress, tensile strength, and work-hardening rate, and decreases of the elongation and fracture toughness in the stress–strain curve. Pronounced DSA is generally explained in terms of the interaction between Mn–C pairs and dislocations. A number of studies have reported direct observations and analyses of cluster characteristics and serration behavior. However, there is very limited information about these particles and their evolution under strain in TWIP steels. Small-angle neutron scattering (SANS) has become a well-established method that can be used to measure nano-scale microstructural features statistically with the benefit of mm-scale deep penetration and volumetric sampling with a scattering beam into the specimen. The purpose of this work is to provide evidence of the presence of the Mn–C cluster and its evolution as a function of strain.

Fe-18Mn-0.6C fcc austenitic TWIP steel was prepared using a vacuum induction. Tensile test were performed at 25 °C and 100 °C and each deformed specimen was cooled down to room temperature in air. The engineering strains of each specimen corresponded to 0, 5, 15, 30, 45 and 50% at 25 °C and 0, 5, 30, and 50% at 100 °C. The middle part of the gauge length of the tensile specimens was cut for SANS measurements and microstructural analyses. SANS measurements were performed using the 18M-SANS instrument at the Korea Atomic Energy Research Institute (KAERI). The microstructure was analyzed using high-resolution TEM and the chemical composition was determined by EDXS.

Based on the different scattering contrasts in the SANS analysis, particles obviously exist in the initial and strained specimens with diameters of 2–14 nm. The particles analyzed by TEM and EDXS showed the higher Mn and C composition (32Mn–13C in wt.%) compared to the base material. As strain increases from 0 to 50% at 25 °C, the number of particles significantly increases from 0.3 to  $1.2 (\times 10^{14}/\text{cm3})$  with a similar size (mean diameter of 7.2 nm). It results in a higher volume fraction ( $11.9 \times 10^{-6}$ ) by 4.3 times compared to the base material ( $2.7 \times 10^{-6}$ ). When temperature increases to 100 °C, a large number of small particles ( $3.5 \times 10^{14}/\text{cm3}$  in ~3 nm size) were observed at 0% strain. As straining, the particles became coarse to ~7 nm and the total volume fraction increased from  $2.7 \times 10^{-6}$  to  $8.7 \times 10^{-6}$ . As straining, increased numbers and size of clusters are correlated to shorter interval and higher steps of the servation peak, respectively.

Email corresponding author: it-sej@kaeri.re.kr

# \*[P09] Effect of building direction on residual stresses in Ti6Al4V components produced by Direct Metal Laser Sintering

Chingowo S.<sup>1</sup>, Mendonidis P.<sup>2</sup>, Isaac A.<sup>3</sup>

## <sup>1</sup>Department of Metallurgical Engineering, Vaal University of Technology Vanderbijlpark, South Africa <sup>2</sup>Vaal University of Technology, Vanderbijlpark, South Africa

One of the additive manufacturing techniques is Direct Metal Laser Sintering (DMLS); this technique utilizes laser as power source to sinter powdered material into a solid structure by aiming the laser at points in space. The process involves melting of the powder and the incursion of heat during the process results in residual stresses due to thermal expansion and contraction of the material [1]. Residual stresses are stress fields remaining in some materials without application of external sources of stress. Evaluation of residual stresses is significant because their existence can introduce dimensional distortions and they have an essential role in fatigue behaviour and corrosion resistance of Ti6Al4V components. Several factors affect residual stresses however the scope of this work was to investigate how building direction affects the amount of stress fields remaining in Ti6Al4V components. Four tensile specimen were manufactured by EOSINT M 280 under argon atmosphere. Two of the specimen were built vertically (z direction) to the building platform and the other two horizontally (x direction) to the building platform. Residual stress values were measured using neuron diffraction method using a wavelength of 1.537Å [2].

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Email corresponding author: shylinec@vut.ac.za

# [P10] Residual stress state improvement of a 316L - ITER welded plate by machining Surface and bulk measurements versus FEM simulation

<u>Rebelo Kornmeier J.</u><sup>1</sup>, Hofmann M.<sup>1</sup>, Robin V.<sup>2</sup>, Valiorgue F.<sup>3</sup>, Gibmeier J.<sup>4</sup>, Šaroun J.<sup>5</sup>

<sup>1</sup>Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München, 85478 Garching, Germany
<sup>2</sup>AREVA NP, 10-12 rue Juliette Récamier,69456 Lyon Cedex 06, France
<sup>3</sup>LTDS\ENISE, 58 rue Jean Parot, 42023 Saint Etienne, France
<sup>4</sup>Karlsruhe Institute of Technology (KIT), Institute of Applied Materials (IAM), 76131 Karlsruhe, Germany
<sup>5</sup>Nuclear Physics Institute of the ASCR, v.v.i., 250 68 Rez, Czech Republic

Arc welding generally introduces unwanted local residual stress states on engineering components hindering a high quality performance in service. Most common procedures to reduce the undesired residual tensile stresses are post heat treatments or mechanical surface treatments as e.g. hammering or shot peeing. However, all of these post treatment processes are elaborate and rather cost-intensive. The question arises if successive surface machining can be applied to effectively mitigate detrimental near surface tensile residual stresses. Within the framework of the Task Group (TG4) of the NeT project (The European Network on Neutron Techniques Standardization for Structural Integrity) a three pass slot weld made from austenitic stainless steel 316L has been manufactured with the aim to undertake 3-dimensional analyses of these residual stresses by both experimental and numerical means [1]. In this presentation we report on the effects of successive surface machining on the residual stress in a welded TG4 austenitic steel plate [2]. The residual stress profile was determined experimentally using incremental deep hole drilling, X-ray and neutron diffraction measurements. The near surface neutron diffraction data were corrected for spurious strain effects using an analytical approach developed by us [3,4]. The experimental results are compared to numerical simulations using a dedicated "hybrid method", specifically set up to simulate finish milling, which has been subsequently applied to the welding simulation so as to predict the final state in the component and its interaction with previous operations.

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Email corresponding author: joana.kornmeier@frm2.tum.de

# \*[P11] Residual stress determination of ductile cast iron by means of neutron diffraction

Smith F.J.<sup>1</sup>, Markgraaff J.<sup>1</sup>, Marais D.<sup>2</sup>, Venter A.M.<sup>2</sup>

<sup>1</sup>School of Mechanical and Nuclear engineering, North-West University, South Africa <sup>2</sup>Research and Development Division, Necsa SOC Limited, Pretoria, South Africa

Ductile cast iron valves which are mechanical of nature, play a critical role in the daily running of plant operations, industrial applications, pipe fluid transport of liquids, slurries and gasses, mining applications and much more. This wide spread application brings about huge economic opportunities resulting in intense competition in the valve industry which is the result of optimisation with respect to casting. This is the cause of improved manufacturability, weight reduction with reduced material cost and enhanced reliability. Reducing material weight and cost, increases the risk of failure, which is further effected by the presence or absence of residual stress.

Casting deficiencies that lead to failure are reduced and/or eliminated by means of modern casting simulation software. MAGMASOFT aims to simulate all aspects of the casting process and also predicts the presence of residual stress [1]. Residual stress on the other hand can also be determined by numerous methods including hole drilling, X-ray diffraction and neutron diffraction [2].

In this report the results of residual stresses in a ductile iron casting, obtained using MAGMASOFT, is presented and compared with residual stresses measured by means of neutron diffraction.

A cast tree (Fig. 1), with different diameter branches, with the form of test samples was modelled and simulated. The residual stress values in the branches were then extracted.

Two identical trees were cast to replicate the simulated models. Three branches, A, B and C (Fig. 1), with the highest simulated residual stress values were cut from the trees. The residual strains in these samples were then measured at the MPISI neutron diffraction instrument at Necsa. The samples from the duplicated trees were then respectively heat treated and machined to uniform tensile test samples of the same diameter (8 mm). A similar neutron diffraction analysis was then carried out on them to determine the residual stress.



Figure 1. Ductile iron cast tree.

The maximum values of residual stress obtained through the simulation process in the samples A to C found to be about 60 MPa, 50 MPa and 35 MPa respectively. The simulation predicted a relief in stress of up to 40% and 70% respectively in the heat treated and machined tensile test samples.

The values obtained from the neutron diffraction analysis will be compared with these values and the results discussed with relevance to casting optimisation.

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Email corresponding author: swysnwu@yahoo.com

# [P12] Stress profiling in cold spray coatings by different experimental techniques: neutron diffraction, x-ray diffraction and slitting method

Luzin V.<sup>1</sup>, Spencer K.<sup>2</sup>, Hill M.R.<sup>3</sup>, Wei T.<sup>1</sup>, Gnäupel-Herold T.<sup>4</sup>

<sup>1</sup>Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW, 2232 Australia <sup>2</sup> School of Mechanical and Mining Engineering, The University of Queensland, QLD, 4072, Australia <sup>3</sup> Department of Mechanical and Aerospace Engineering, University of California, Davis, CA 95616, USA <sup>4</sup> NIST Center for Neutron Research, National Institute of Standards and Technology, Gaithersburg, MD 20899, USA

The residual stress profiles in Cu and Al coatings sprayed using kinetic metallization to thickness of  $\sim 2$  mm have been studied. Due to specific parameters of the cold spray process and particular combination of materials of coatings and substrates, the resultant residual stress distributions are characterised by low stress values, few tens of MPa. This poses challenges on accuracy and resolution of a technique when measuring through-thickness stress distribution. Three experimental techniques, neutron diffraction, x-ray diffraction and slitting method were used to measure through-thickness stress distribution in the coated systems. All three techniques demonstrated acceptable accuracy and resolution to analyse stress profiles. Advantages and disadvantages of each technique are discussed.

Email corresponding author: vladimir.luzin@ansto.gov.au

## [P13] Residual stress measurements at the powder diffraction beamline MCX at Elettra

Gigli L.<sup>1</sup>, Plaisier J.R.<sup>1</sup>

## <sup>1</sup>*Elettra-Sincrotrone Trieste, Basovizza, Trieste ITALY*

The beamline Material Characterization by X-ray diffraction (MCX) [1], is the general purpose powder diffraction beamline at the Elettra synchrotron in Trieste, one of currently four diffraction beamlines at Elettra. The beamline is designed to host a wide range of experiments, that cover many scientific fields with standard applications such as phase identification, structure determination, The experimental setup makes MCX also an excellent station for performing residual stress measurements.

MCX operates in the energy range between 8 and 20 keV. The photon flux on the sample is about 1011 photos/sec. The X-ray spot on the sample can be tuned from horizontal line focus (5mm x 1mm) to point focus (0.3mm x 0.3mm). The main experimental station houses is a four circle Huber diffractometer with an angular resolution in 2-theta of 0.0001°. The two-theta arm is equipped with a scintillator detector and a Si-111 analyzer crystal. A laser interferometer is present for the exact alignment of the samples. The large space available at the sample stage allows the installation of custom sample holders. A translation stage allows the fully automatic measurement of multiple points of interest using the beamline control software. This control program is written in python and allows scripting giving the opportunity to fully customize the measurement protocol ad data collection. Additional software is available at the beamline to quickly evaluate the result of residual stress measurements.

Recently MCX has been involved in projects dealing with the determination of residual stress induced by laser peening (LSP) in aluminum alloys [2]. Here, the beamline is presented and some results of these measurements are provided for illustration of MCX's capabilities in the field of measuring stress and strain.

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Email corresponding author: Lara.gigli@elettra.eu

## \*[P14] The design of a gas-reaction chamber for a vacuum furnace on the PITSI neutron powder diffraction instrument

Grobbelaar I.J.<sup>1</sup>, Cilliers J.<sup>1</sup>, Cilliers A.<sup>1</sup>, Marais D.<sup>2</sup>, Venter A.M.<sup>2</sup>

## <sup>1</sup> School of Mechanical and Nuclear Engineering, North-West University, Potchefstroom, South Africa <sup>2</sup>Reasearch and Development Division, South African Nuclear Energy Corporation (Necsa) SOC Limited, South Africa

The PITSI instrument (Powder Instrument for Transition in Structure Investigation) is a medium resolution neutron powder diffraction instrument located at the SAFARI-1 research reactor of the South African Nuclear Energy Corporation (Necsa) SOC Limited. PITSI is used to study crystallographic structures, atomic substitutions, chemical composition and phase transformation in solid and powdered polycrystalline materials. Neutron powder diffraction is complementary to other material characterization techniques which includes X-ray diffraction and electron microscopy [1,2].

This presentation reports on the design of a gas-reaction chamber to be used in conjunction with the current vacuum furnace that is available on PITSI for in-situ studies. This expanded capability attachment should be capable of delivering either of four gases (helium, nitrogen, carbon dioxide and low concentrations of hydrogen) to the sample at flow-rates of up to 1000 ml per minute and be able to withstand pressures of up to 200 Bar and function in temperatures up to 1000 °C. This will enable *in-situ* studies of chemical reactions at elevated temperatures. The gas-reaction chamber will be developed to replace the standard top-loaded sample stick which operates in the high vacuum environment of the furnace. This will require the consideration of an enclosed chamber that fits into the 70 mm diameter central shaft of the furnace, gas delivery lines that are temperature compensated to prevent condensation and maintain constant temperature and pressure conditions at the sample position, as well as thermometry and heaters that can function in this altered environment, and materials that can remain functional up to 1000 °C.

The design will be optimised with computer aided design software, especially FloXpress® from Solidworks® to perform gas flow and stress analysis through the furnace. The design was influenced by reviewing existing instruments such as the Australian Nuclear Science and Technology Organisation's (ANSTO) gas reaction chamber [3] and the XRK 900 reactor chamber from Anton-Paar [4].

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Email corresponding author: nwu23446226@gmail.com

# \*[P15] Considerations of a cost-effective solution for expanding the detector subtending angle of the PITSI instrument

Haasbroek A.<sup>1</sup>, Marais D.<sup>2</sup>, Van Heerden P.R.<sup>2</sup>, Venter A.M.<sup>2</sup>, Cilliers J.<sup>1</sup>, Cilliers A.<sup>1</sup>

<sup>1</sup>School of Mechanical and Nuclear Engineering, North-West University, Potchefstroom, South-Africa <sup>2</sup>Research and Development Division, South African Nuclear Energy Corporation (Necsa) SOC Limited, South Africa

The Powder Instrument for Transition in Structure Investigations (PITSI) instrument is located at the SAFARI-1 research reactor of the South African Nuclear Energy Corporation. Within a limited budget its detector was established as a pseudo area detector built-up from 15 oneinch, vertically stacked *Reuter-Stokes* 1-D position sensitive detectors (PSD) to have an active area of ~ 600 x 375 mm<sup>2</sup>. A photograph of this detector distance is variable between 1.2 to 1.6 m where the latter subtends a viewing angle of 20°. To attain a diffraction pattern, the detector bank is step-scanned in a number of frames and the data sets stitched during post-processing to produce a diffraction pattern covering 110° [1]. This scanning methodology is not only cumbersome, but also



Figure 1. Detector Bank at PITSI

insensitive to dynamic investigations where larger  $2\theta$  sections, or even full diffraction patterns are required from one detector setting in parametric studies, e.g. re-crystallization, or phase transformations as function of temperature.

This presentation reports on the consideration of mechanical solutions to enlarge the detector span to least  $110^{\circ}$  in 20 by incorporating 25 additional detectors and subsequently eliminating the required step-scanning approach. By considering the existing detector bank arrangement as point of departure, the concept design of Figure 2 is being considered. This staggered configuration effectively replicates the methodology applied with the existing detector array. The staggered pattern allows overlap between detector banks thus eliminating "dead" zones between banks [2]. Since the 110° are subtended in one instrument setting, the need for the  $\theta$  repositioning of the detector bank around the sample is not required. When reducing the sample-to-detector distance, the detector frames can be integrated to increase the number of detectors at each location. For this

purpose different clamping and suspension methods had to be considered to provide the PSDs with secure and integrateable latching. Data handling of this new solution can be incorporated with minimal modification to the existing data reduction software. The new geometry will be validated by comparing the detector performance per  $\theta$  position to the current detector setup.



This study could provide innovative cost-effective solutions to similar facilities towards improving measurement efficiencies in neutron diffraction applications.

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Email corresponding author: abramhaasbroek@gmail.com
### [P16] Multi-reflexion and multi-wavelength diffraction method used for residual stress determination in mechanically treated surface layer of Ti (grade2)

Marciszko M.<sup>1</sup>, Baczmański A.<sup>2</sup>, Genzel C.<sup>3</sup>, Klaus M.<sup>3</sup>, Wrobel M.<sup>4</sup>, Braham C.<sup>5</sup>, Sidhom H.<sup>6</sup>

<sup>1</sup>AGH-University of Science and Technology, ACMIN, al. Mickiewicza 30, 30-059 Kraków, Poland <sup>2</sup>AGH-University of Science and Technology, WFiIS, al. Mickiewicza 30, 30-059 Kraków, Poland <sup>3</sup>Institut für Angewandte Materialforschung, Helmholtz-Zentrum Berlin für Materialien und Energie, Albert-Einstein-Str. 15, Berlin 12489, Germany

 <sup>4</sup>AGH-University of Science and Technology, WIMiIP, al. Mickiewicza 30, 30-059 Kraków, Poland
<sup>5</sup>Arts et Métiers ParisTech (ENSAM), PIMM, 151, Bd de l'Hôpital 75013, Paris, France
<sup>6</sup>ALaboratoire de Mécanique, Matériaux et Procédés, LAB-STI-03 ESSTT, 5 Avenue Taha Husseïn BP 56, Bab Menara, 1008 Tunis, Tunisia

Methodology of stress determination using MGIXD method (multireflection grazing incidence diffraction) [1] was developed and applied to measure depth dependent stress profile in the surface layers of the mechanically treated Ti (grade 2) samples.

In this study the stress evolution vs. depth below the sample surface was determined from lattice strains measured for different incident angles and also using simultaneously different wavelengths ('multi-reflection' and 'multi-wavelength' method). In the presented methodology the experimental results obtained for given penetration depth were selected. The advantage of this approach is that more experimental data are available to calculate the stresses and also the information depth is significantly increased.

The approach is based on multireflection analysis applied for the energy dispersion method in which white beam containing radiation having different wavelengths was used. Measurements were performed on EDDI beamline (BESSY, Berlin, Germany) [2]. The results obtained from synchrotron measurements were compared with those from classical diffractometer (X-Pert Philips X-ray diffractometer, Cu K $\alpha$  radiation) equipped with a Göbel mirror in incidence beam optic. Summarizing the results obtained with synchrotron radiation it can be stated that using different wavelengths (energies) of radiation gives similar stress depth dependent profiles. In addition the determined values of  $a_0$  and c/a lattice parameters vs. depth do not vary significantly with depth. For the first time the multireflection method in which the data for the same penetration depth are selected was successfully used to analyze the EDDI data.

Perfect agreement was obtained between the measurements performed using synchrotron radiation as well as Cu K $\alpha$  radiation on laboratory diffractometer (for MGIXD and also for EDDI methods). Certainly, synchrotron radiation with higher energies allowed measurements for larger depths in comparison with laboratory X-rays.

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Email corresponding author: marciszk@agh.edu.pl

#### \*[P17] Improving Reliability of the Neutron Diffraction Technique for Residual Stress Characterization in Dissimilar Metal Welds

Rahman M.<sup>1,2</sup>, Moat R.<sup>1</sup>, Pirling T.<sup>2</sup>

<sup>1</sup>The Open University, Milton Keynes, UK <sup>2</sup>Institut Laue-Langevin, Grenoble, France

Neutron diffraction is a well-established technique for characterizing residual stress non-destructively in the bulk of engineering components. However, deviation from the powder diffraction characteristics can give rise to different experimental effects on the measurements. These deviations are often encountered in welded structures, and they challenge the reliability of the characterized stresses. This becomes far more challenging when the weld is fabricated from dissimilar metals. Among the various issues, step gradient in chemical composition across the bimetallic interface, texture and uneven distribution of large grains in the weld region pose considerable difficulties in measuring accurate lattice spacing of the material(s). Several researchers have reported their efforts in characterizing residual stresses in typical dissimilar metal welds (DMWs). However, these work often failed to acknowledge the possible inaccuracy in the measurements or remove certain measurements from the results quoting the limitations of the technique. The root cause of the inaccuracy could be the anomalous peak-shift(s) in the measurements when the gauge volume is submerged across the bimetallic interface. This introduces pseudo-strains due to the combined effect of geometric and attenuation shifts of the effective centroid of the sampled gauge volume. These pseudo-strains can yield in unrealistic stress values often within the locations of interest [1, 2]. In this work, we have simplified the DMW configuration in a systematic order, which has facilitated separation of the effects associated with an internal material-to-material interface. Precisely designed samples with known interface-characteristics (i.e. position relative to the traversing gauge volume, strain gradients) are employed to identify the peak-fitting constraints. It was found that the material-to-material profiles agree with the averaged profiles produced from material-toair measurements. These fitting constraints will enable development of a deconvolution method for isolating partially overlapping diffraction peaks leading to the possible correction of the pseudo-strains. These results will be employed to develop and validate analytical modelling efforts and McStas neutron ray trace simulations aimed at understanding the errors introduced when traversing a gauge volume across material-tomaterial interfaces. Finally, the findings will support the planned inclusion of a best practice guide for neutron diffraction measurements on DMWs in the UK nuclear industry, R6 structural integrity code.

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Email corresponding author: Mushfiqur.Rahman@open.ac.uk

#### [P18] Modelling of spatial resolution effects in neutron residual strain scanning

<u>Šaroun J.</u><sup>1</sup>, Rebelo Kornmeier J.<sup>2</sup>, Gibmeier J.<sup>3</sup>, Hofmann M.<sup>2</sup>

<sup>1</sup>Nuclear Physics Institute CAS, Řež, Czech Republic <sup>2</sup> TU Munchen, FRM II Garching, Germany <sup>3</sup> Karlsruhe Institute of Technology, Institute for Applied Materials, Germany

Spatial resolution is one of critical parameters in mapping of residual strains by neutron diffraction. It affects not only the level of smearing of the measured strain patterns, but it may also give rise to peak shifts (false strains) resulting from perturbations of the sampling volume by sample heterogeneities. The well-known surface effect is a trivial example, but false strains arise in general from any gradient in material properties causing significant variation of scattering intensity over a distance comparable with the size of the sampling volume. To tackle this problem, we propose the use of two complementary methods: (i) analytical description of the sampling volume and neutron beam distributions within Gaussian approximation and (ii) neutron ray tracing simulations.

The analytical approach [1] is limited to linear approximations of dispersion and neutron transport relations (paraxial optics), but permits fast calculations and data fitting including deconvolution of measured strain profiles and corrections for the false strains. The analytical model has been implemented in a set of Matlab<sup>(R)</sup> scripts which allow to calculate false strains for both monochromatic and time-of-flight diffractometers and to carry out indirect deconvolution of measured strain profiles. The deconvolution is performed by least-square fitting of a suitable model for the spatial dependence of stress tensor components expressed through damped polynomial or piecewise polynomial ansatz functions. The model requires a few instrumental parameters, which can be determined by fitting of peak intensities measured as a function of scan position. The parameter describing the coupling between sampling volume perturbation and peak shift can be determined either analytically using the instrument configuration data. Alternatively, the model parameters can be determined by analysis of scans simulated by neutron ray-tracing.

The ray-tracing simulation is thus a suitable complementary technique, which can provide required instrumental parameters for the analytical model, simulate realistically the sampling volume in complete phase space of the neutron beam as well as to generate synthetic data for validation of data analysis methods. The program SIMRES [2] has been equipped with several features particularly useful for simulations of strain scanning experiments in both time-of-flight and monochromatic modes. Apart of the possibility to simulate rather detailed instrument configurations, the key features are (i) an efficient particle generation algorithm and (ii) a polycrystalline sample model. The particle generation code employs adaptive sampling strategy and reverse tracing methods, which allow for fast simulations even with a very small gauge volume typical for strain scanning. The sample model permits to define multiphase samples with macro-strains, in addition to other material properties. The model is currently limited to isotropic materials. However, the ray-tracing simulation is in fact a Monte Carlo integration method, where integration over additional variables can be added almost for free in terms of computing time. Therefore, a new model is being developed which includes also averaging over grain orientations in textured materials provided that the orientation distribution function is available.

Treatment of the spatial resolution effects by combination of the analytical and Monte Carlo simulation methods will be demonstrated on the examples of strains developed during uniform bending test and strain profiles typical for surface hardening.

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Email corresponding author: saroun@ujf.cas.cz

# [P19] Rotating and revolving spiral slits system with area detector for in-situ synchrotron x-ray diffraction analysis of thermal strains in automotive power electronic module

Setoyama D.<sup>1</sup>, Kimura H.<sup>1</sup>, Yamaguchi S.<sup>1</sup>, Masanori U.<sup>1</sup>

#### <sup>1</sup>Toyota Central R&D Labs., Inc.

A rotating and revolving spiral slits system was used with an area detector to measure synchrotron x-ray diffraction from inside of an automotive power electronic module. The slit system is a unique tool to analyse local stress and strain distribution of arbitrary materials simultaneously. The slits can not only rotate around the collimator shaft to overcome the blind azimuth by the slits themselves but also revolve around the focal point of the slits system. Therefore the users can measure coarse-grained or highly textured materials as well as phase-transformed materials, even if the diffraction angles are higher than the radii of the slit disks [1]. Authors have developed and installed the slits system at the BL33XU, Toyota Beamline, of SPring-8[2, 3].

In this study, authors applied this technique to the strain distribution analysis in an automotive power electronic module often used for hybrid, plug-in hybrid and electric vehicles. The power electronic module is one of the most important components in such vehicles, and controls high power electricity for driving which will lead to Joule heating. The power electronic module usually has a layered structure composed of single crystal semiconductor, coarse grained lead-free solder as the joints, polycrystalline ceramic plate as the electric insulator, and polycrystalline metals as the electric wiring and cooler. The Joule heat and the misfit of the thermal expansion of the composing materials will give rise to thermal stresses, the understanding of which is important for developing advanced vehicles. To the best of authors' knowledge, the rotating and revolving spiral slit system is the only means to measure the stress distributions of the device with such a variety of component materials with the different microstructures.

Authors have prepared a model of the power electronic module. The strain distribution was measured by the developed slits system with an area detector. The synchrotron x-ray energy was set to 29 keV with the width of the incident beam and the slit aperture set to 100  $\mu$ m and 50  $\mu$ m respectively, to obtain the depth resolution less than the component thickness. The analysed results showed that the diffraction angles of all components of the module shifted lower by the Joule heat. The expansions were, however, different by the composing materials. The lead-free solder was considered to be under compression, whereas the silicon semiconductor and the aluminium cooler were exposed to tensile load.

The synchrotron radiation experiments were performed at the BL33XU (Toyota beamline) of SPring-8 with the approval of the Japan Synchrotron Radiation Research Institute (JASRI).

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Email corresponding author: daigo @mosk.tytlabs.co.jp

#### [P20] Deformation Analysis of Reinforced Concrete using Neutron Imaging Technique

Suzuki H.<sup>1</sup>, Sekine M.<sup>2</sup>, Koyama T.<sup>2</sup>, Kanematsu M.<sup>2</sup>, Iikura H.<sup>1</sup>, Matsumoto Y.<sup>3</sup>, Kai T.<sup>1</sup>, Shinohara T.<sup>1</sup>

<sup>1</sup> Japan Atomic Energy Agency, Tokai, Naka, Ibaraki, Japan,
<sup>2</sup> Tokyo University of Science, Noda, Chiba, Japan
<sup>3</sup> CROSS, Tokai, Naka, Ibaraki, Japan

The reinforced concrete, which is widely utilized for various architectural and civil engineering structures, is well known as a composite structure, in which concrete with relatively low tensile strength and ductility is strengthened by reinforcements such as steel rods (rebars) with high tensile strength and/or ductility. The performance of the reinforced concrete is generally derived from the bond resistance between rebar and concrete. In our previous studies, we demonstrated that the neutron diffraction technique can be an alternative method to the conventional strain gauge for evaluation of bond resistance by measuring stress distribution of the rebar embedded in concrete [1-3].

On the other hand, it is also important to evaluate deformation behaviours of concrete around the embedded rebar, in order to discuss the mechanisms of bond degradation between concrete and rebar for the reinforced concrete structure. However, the neutron diffraction technique is difficult to be applied to the strain measurement of concrete since diffraction from concrete is difficult to be observed due to high background noise from hydrogen involved in concrete. Alternatively, the image analysis techniques, i.e. a lattice method and a digital image correlation (DIC), are commonly utilized for evaluating deformation of concrete quantitatively. They can assess the deformation of concrete by analysing image contrast or marker displacement taken by high resolution camera. In this study, we develop a novel method to observe internal deformation of concrete by combining the image analysis technique with neutron transmission imaging.

In order to visualize the internal deformation around the embedded rebar, the reinforced concrete samples including cement paste markers containing 34 wt%  $Gd_2O_3$  powder (called "Gd marker") with about 1mm in diameter were prepared. The Gd markers were two-dimensionally dispersed around rebar embedded in the rectangular concrete brick (50 mm × 130 mm). This sample mounted on the loading device was set up on the sample table of BL22, RADEN [4], in MLF of J-PARC, and the transmission images were taken under applying pull-out loading to rebar. As shown in Figure 1, the Gd markers in concrete were successfully observed by transmission imaging with 0.05 mm in spatial resolution. They were confirmed to be moved by increasing applied loading, suggesting compressive deformation occurred in concrete.



In the presentation, we will show the concrete deformation analysed by Gd marker displacement, DIC and PIV (Particle Image Velocimetry) with neutron transmission image, and the possibility of the concrete deformation analysis with neutron imaging technique will be discussed.

Figure 1. Transmission image of reinforced concrete with Gd markers.

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Email corresponding author: suzuki.hiroshi07@jaea.go.jp

#### \*[P21] Design concepts for a replacement beam aperture for the MPISI (Materials Probe for Internal Strain Investigations) instrument

Van der Merwe J.C.<sup>1</sup>, Cilliers J.<sup>1</sup>, Cilliers A.<sup>1</sup>, Marais D.<sup>2</sup>, Venter A.M.<sup>2</sup>

<sup>1</sup>School of Mechanical and Nuclear Engineering, North-West University, South Africa <sup>2</sup>Research and Development Division, South African Nuclear Energy Corporation (Necsa) SOC Limited, South Africa

A prominent application in materials engineering is the non-destructive determination of internal strains in materials and components that originate from the manufacturing processes or in-service loads, using neutron diffraction techniques. In these applications the thermal neutron waves are directed onto the sample under investigation from where it scatters from the periodic arrangement of the atoms in the polycrystalline material.

To enable depth analysis the size of the illuminated volume inside the material, i.e. the gauge volume, it is strictly controlled with neutron absorbing apertures that transmit neutrons only through its window, with the remainder being neutron tight [1]. In addition these apertures should minimally infringe on the space available for the sample movements. Traditionally such apertures are tapered geometries with various ways enforced to control the window size, i.e. either by interchangeable fixed geometries, adjustable masks, or fully motorised 2D configurations.

To accurately define the projected gauge volume and taking cognisance of the natural beam divergences, the final defining horizontal and vertical aperture sizes should be at the same distance from the instrument centre. Specifically with small gauge volumes the window should be as close as practically achievable from the instrument centre otherwise the real gauge volume could be substantially larger than the intended nominal gauge volume. To ensure accurate definition of the gauge volume, the positions of the vertical and horizontal regulating points should be at the same position.

Figure 1 displays the current MPISI apertures. The aperture is vertically adjustable from 0.5 to 20 mm and horizontally from 0.1 to 20 mm with the use of micrometres. Notwithstanding the gauge sizes being accurate adjustable a limitation is that the regulating points are not coincident which does introduce inaccuracies in the exact gauge volume size.

This presentation considers various concept configurations for possible replacement apertures for MPISI that will render overall tighter tapering and sharper beam definition. The use of efficient neutron absorbing materials will also be discussed. The design will be created using SolidWorks and verified using the Monte Carlo Ray Trace McStas model of MPISI [2].



Figure 1. Photograph of the current apertures on the MPISI instrument.

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Email corresponding author: 23473207nwu@gmail.com

#### [P22] MPISI: The neutron strain scanner Materials Probe for Internal Strain Investigations at the SAFARI-1 research reactor

Venter A.M.<sup>1</sup>, Van Heerden P.R.<sup>1</sup>, Marais D.<sup>1</sup>, Raaths J.C.<sup>1</sup>

#### <sup>1</sup>Research and Development Division, South African Nuclear Energy Corporation (Necsa) SOC Limited, South Africa

A modern neutron strain scanner instrument with crystallographic texture measurement capability has been established at the SAFARI-1 research reactor. This instrument, MPISI shown in Fig. 1, has been benchmarked against the VAMAS Ring-and-Plug specimen, as well as in high spatial resolution mode using sub-millimeter beams [1]. It is equipped with a double focused Si-multiwafer monochromator, sturdy high-precision sample manipulation stages, adjustable primary and secondary beam apertures interchangeable with radial collimators (RCs) on the secondary beam side and a 300 x 300 mm<sup>2</sup> position sensitive neutron area detector. The RCs facilitate measurement of large specimens and interfaces. Variation in monochromator horizontal curvature allows optimization of the intensity (texture), resolution (peak broadening), or Figure-of-Merit (strain) with an achievable resolution of  $\Delta d/d \sim 3 \times 10^{-3}$ . An Android based Wi-Fi hand-held control module in conjunction with lasers and theodolites aid with sample positioning and alignment. Data acquisition and control has been standardised on the SINQ Instrument Control Software. In-house developed data reduction software [2] enables: flat field correction, geometric correction, vertical integration, data normalization, peak and entry-curve fitting, as well as multi-dimensional data visualisation. Acquiring data against statistical criteria rather than time improves the overall instrument utilisation efficiency [3].



Figure 1. Photographs of the MPISI instrument

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Email corresponding author: Andrew.venter@necsa.co.za

#### [P23] International Stress Engineering Centre - a proposed international facility for hybrid multi-scale stress measurements

Zhang S.Y.<sup>1,2</sup>, Bouchard P.J.<sup>3</sup>, McGreevy R.<sup>2</sup>

<sup>1</sup>Dongguan Centre of Excellence for Advanced Materials, Konggu Building, 1 Libin Road, Songshan Lake, Dongguan, Guangdong, China

<sup>2</sup>ISIS Neutron and Muon Source, Science and Technology Facilities Council, Rutherford Appleton Laboratory, Didcot,

OX11 0QX, UK

<sup>3</sup>The Open University, Milton Keynes, MK7 6AA, UK

Over the past two decades there has been a revolution in techniques for measuring the deformation, strain, stress and damage "state" of engineered structures from atomic to metre length-scales. An international centre of excellence for hybrid multi-scale stress measurements is proposed that will help industry to improve design, optimise manufacture, extend life, secure safety and reduce costs of man-made structures on land, at sea, in air and into space. The planned International Stress Engineering Centre (ISEC) will comprise twin facilities to be built, in the UK on the Harwell Campus, and in China at the China Spallation Neutron Source near Dongguan in Guangdong province. ISEC will educate, train and help industry measure stress in complex structures using innovative full field techniques and bring about a better understanding of material and structural behaviour under normal and extreme operating conditions. The new facilities will be designed to handle heavy-section complex structures of interest to industry and will provide access to specialist equipment for measuring stresses including neutron diffraction, X-ray diffraction, the contour method and hole drilling, as well DIC-based strain mapping, modelling and hybrid methods. ISEC will help drive both UK and China economies by partnering with industry to develop high performance structural materials, promoting competitive high value manufacturing in a global market and broadening the skills base in advanced techniques.

Email corresponding author: shuyan.zhang@ceamat.com

#### **Fundamentals of neutron diffraction**

#### Holden T.

This talk will introduce the properties of the neutron, its energy, momentum and wavelength. Bragg's law, the relationship between lattice spacing, wavelength and angle of scattering, is readily obtained by considering of the interference between the neutron wavelets scattered by the individual nuclei of the atoms in a solid. The introduction of the nuclear scattering cross sections (absorption, coherent and incoherent) leads to a calculation of the structure factor for coherent diffraction of neutrons in solids and therefore to the intensities of the neutron beams measured in experiments. The law governing the attenuation of the incident neutron beam as it passes through solids is easily calculated from the nuclear cross sections and is the basis for neutron imaging. In general, the attenuation of neutrons in solids is far less for neutrons than laboratory x-rays leading to the greater penetration of neutron beams. The idea of using Bragg's law to measure lattice strains at depth non-destructively with neutrons follows naturally. Various sources of systematic errors are identified. Finally, the two types of neutron source, reactors and spallation sources, are described as well as the instruments in use to measure lattice spacing, strains, diffraction line-widths and intensities

### Fundamentals of X-ray and synchrotron radiation and its value addition in material science

#### Brown D.W.

#### LANSCE and Materials Science & Technology Division, Los Alamos National Laboratory, Los Alamos, NM 87545, U.S.A

Engineering diffraction measurements have traditionally belonged in the realm of neutron diffraction, since laboratory based x-ray sources produce relatively low energy x-rays (<20keV) which penetrate most engineering materials to depths of 10  $\mu$ m or less. In comparison, neutrons penetrate cm's into most engineering measurements enabling bulk microstructure observations at depth. However, the advent of 3<sup>rd</sup> generation synchrotron x-ray sources and beamlines optimized to provide x-rays at much higher energies (60-130keV) has recently enabled x-ray diffraction measurements at macroscopic depths in engineering materials of e.g. cm's in Al, 5mm in Fe, 0.5mm in U. Moreover, the rapidly increasing brilliance of synchrotron x-ray beams has allowed diffraction data to be collected with very high spatial (~0.1mm) and temporal (<1sec) resolution in macroscopic components. However, due to the high incident x-ray energy (short wavelength) the diffraction angle becomes small (~5°). This change in diffraction geometry makes it difficult to measure strain components in 3-dimensions in bulk samples as necessary for stress calculations. Thus, neutron and x-ray engineering diffraction measurements remain complimentary, as they should be. Examples from various applications will be presented.

#### The role of numerical analysis in the assessment of weld residual stresses

#### Muránsky O.

#### Australian Nuclear Science and Technology Organization (ANSTO), Lucas Heights, New South Wales, Australia

There is currently a strong interest in quantifying the residual stresses in welds found in nuclear power stations, for both the existing and proposed next generation of nuclear reactors. In most cases, it is impractical to perform residual stress measurements for every weld in a welded structure. Additionally, the dimensions of real structural components are usually very large, such that non-destructive neutron and X-ray methods of residual stress analysis cannot be used due to path length restrictions. In these cases, validated numerical analysis may be the only method available for reliable determination of residual stresses. It is therefore important to establish the accuracy of modelling techniques for situations where critical comparisons can be made between simulation and experiment.

The aim of numerical weld simulation is to develop modelling techniques that are usable for the design and optimisation of welding procedures. This process subsequently allows us to estimate the appropriate mechanical performance of a welded component under its intended service conditions. Although numerical models cannot fully replace experimental methods, their benefit lies in better understanding the welding process. As a result, fewer validation experiments are necessary in evaluating and optimising different welding procedures for specific applications.

A series of measurements and simulations on gradually more complex geometries have been carried out under the auspices of the European Network on Neutron Techniques Standardisation for Structural Integrity (NeT). The present work is part of this effort, and is intended to predict and measure using diffraction techniques a three-dimensional residual stress field produced in two scenarios: (i) a three-pass austenitic steel slot weld; and (ii) an autogenous ferritic steel weld. In addition, a dissimilar metal weld extracted from a nuclear power station is analysed by the finite element technique and validated using deep hole drilling measurements.

#### Capabilities of in-situ loading investigations using neutron diffraction/imaging towards elucidating fundamental and industry related problems in material science

#### Kabra S.

#### ISIS Neutron source, Science Technology Facilities Council, Harwell, Oxfordshire, UK

In-situ experiments on energy dispersive neutron instruments are extremely useful to probe the fundamental properties of materials. With respect to engineering materials, this provides a unique opportunity to study strains, phases, twinning, plasticity, grain rotations etc. under a variety of thermo-mechanical conditions. High neutron penetration facilitates large sample environment and representative sample sizes. Recent advances in imaging techniques and instrumentation means that these studies can now be done with an unprecedented resolution.

I will be using a number of case studies in both in-situ diffraction and imaging, to highlight the variety of studies that can be undertaken on a multitude of engineering relevant materials. These will include both fundamental properties like twinning, creep, fatigue and highly applied studies which are of relevance to the industry.

### Neutron stress scanning with high spatial resolution for surface engineering applications

#### Luzin V.

#### Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW, 2232 Australia

A great variety of techniques is used nowadays and properties for the purpose of surface properties enhancement and/or changing its functionality. Depending on application and design, it can be surface treatments: thermal (e.g. laser melting/processing), mechanical (e.g. shot peening or roller burnishing) or both (e.g. laser shock peening). Also, entirely new material can be deposited on surfaces by means of thermal spray, cold spray, laser deposition or any other deposition method. Intentionally or non-intentionally, residual stresses are introduced due to the high-impact treatments. Since mechanical state and integrity of surface layers (or coatings) is critical for the most of applications, residual stress control and mitigation are very essential to prevent mechanical failures on surfaces and ultimately to improve performance and overall lifetime of engineering components.

Neutron diffraction stress scanning can provide an effective tool for non-destructive in-depth stress measurements in metals and ceramics with high spatial resolution of 0.1-0.2 mm. This technique is available through use of neutron diffractometer with constant wavelength based on the rector sources such as KOWARI (OPAL research reactor, ANSTO, Australia) or MPISI (SAFARI-1 reactor, Necsa, South Africa). The most recent results on neutron diffraction stress measurements made with high resolution will be presented for a wide range of surface treatment applications.

#### Introduction to Texture Analysis and Microstructure Characterization using Diffraction Data

#### Vogel S.C.

### LANSCE and Materials Science & Technology Division, Los Alamos National Laboratory, Los Alamos, NM 87545, U.S.A.

Several material properties, describing for instance elastic and plastic deformation or thermal and electric conductivity, are anisotropic when determined for single crystals. Such properties are mathematically described by tensors. In polycrystalline materials, the properties of the aggregate are then determined by the single crystal properties and the orientation distribution of the crystals in the polycrystalline aggregate, the so-called texture. Therefore, knowledge of the orientation distribution to understand and ultimately predict properties of both man-made and natural materials is a requirement in many branches of physical sciences such as materials science and engineering or geology. Diffraction methods, like neutron and X-ray diffraction, allow measurement of the orientation distribution of the crystals within a polycrystalline aggregate. Modern diffraction methods, using e.g. pulsed neutron sources or 2D X-ray detectors in the laboratory or at synchrotron sources, combine the texture analysis with microstructure analysis (e.g. volume fractions of different phases, dislocation densities) and crystallographic analysis (e.g. lattice parameters, atomic positions) and therefore provide powerful tools for materials research. This lecture will

- introduce the concept of the orientation distribution function, pole figures, inverse pole figures,
- introduce various representations of the orientation distribution function,
- provide examples why texture is an important parameter of the microstructure of a material,
- describe methods to measure texture,
- introduce software tools to derive the texture from diffraction data (MAUD, GSAS, GSAS-2)
- introduce software tools to interpret the texture, e.g. plotting, determination of volume fractions of texture components etc. (MTEX, JTEX)

#### **Completing the picture: Correlative imaging and diffraction**

Withers P.J.

#### School of Materials, Manchester University, M13 9PL, United Kingdom and Research Complex at Harwell, Didcot, OX11 0FA, UK.

A single technique can only provide limited information about a material. Increasingly multiple techniques are being brought to bear on the same region of interest to provide a fuller picture. This can include imaging, diffraction spectroscopies and even local materials property measurements. Sample information can be correlated across time, length scale and modalities. Such correlative approaches are finding significant application in the life sciences and in this talk I will introduce correlative workflows and explain the experimental challenges. In addition I will discuss certain data acquisition strategies as well as illustrate the utility of the approach through a series of examples. Examples will be taken from additive manufacturing, creep cavitation failure of nuclear plant, self healing materials, polymer composites and corrosion cracking.

# Diffraction based residual stress capabilities available to South African researchers

Venter A.M.

<sup>1</sup>Research and Development Division, Necsa SOC Ltd, Pretoria, South Africa

A steady grow in the use of diffraction based techniques for residual stress analysis is occurring in South Africa. A number of laboratory based X-ray diffraction instruments exists at various institutions, as well as a neutron diffraction instrument at the SAFARI-1 research reactor. In addition international facilities are accessible through User Programs based on scientific merit.

An overview will be given of the facilities existing in South Africa, as well as international accessibility to synchrotron and neutron facilities.

## List of Attendees

	First Name	Last Name	Company/Organisation	Country	Email Address
Dr	Ке	An	Oak Ridge National Laboratory	USA	kean@ornl.gov
Mr	Lucas	Anderson	University of Stellenbosch	SA	landerson@sun.ac.z a
Dr	Daniel	Apel	Helmholtz-Zentrum Berlin für Materialien und Energie	Germany	daniel.apel@helmh oltz-berlin.de
Prof	Andrzej	Baczmanski	AGH University of Science and Technology	Poland	andrzej.baczmanski @fis.agh.edu.pl
Ms	Nkele	Baloyi	Vaal University of Technology	SA	nkeleb@vut.ac.za
Dr	Dreyer	Bernard	Nelson Mandela University	SA	dreyer.bernard@n mmu.ac.za
Dr	Cindy	Bolme	Los Alamos National Laboratory	USA	cbolme@lanl.gov
Prof	Peter	Bouchard	The Open University	UK	john.bouchard@op en.ac.uk
Dr	Chedly	Braham	AMVALOR	France	chedly.braham@ens am.eu
Mr	John	Brokx	The Open University	UK	john.brokx@open.a c.uk
Dr	Donald	Brown	Los Alamos National Laboratory	USA	dbrown@lanl.gov
Dr	Adrian	Brügger	Columbia University	USA	brugger@civil.colu mbia.edu
Dr	Sandra	Cabeza Sánchez	Institut Laue–Langevin	France	cabeza@ill.fr
Mr	Jan	Čapek	Charles University	Czech Republic	jan.capek89@gmail. com
Ms	Shyline	Chingowo	Vaal University of Technology	SA	tafadzwawanyasha @gmail.com
Prof	Hahn	Choo	University of Tennessee	USA	hchoo@utk.edu
Dr	Matthew	Connolly	National Institute of Standards and Technology	USA	matthew.connolly@ nist.gov
Dr	Stefano	Coratella	University of Dayton Research Institute	USA	stefano.coratella@u d-research.org
Dr	Harry	Coules	University of Bristol	UK	harry.coules@bristo l.ac.uk
Mr	Jinesh	Dahal	Los Alamos National Laboratory	USA	jdahal@laln.gov
Mr	Philip	Doubell	Eskom Holdings SOC Ltd	SA	philip.doubell@esko m.co.za
Mr	Aghogho	Edward	University of Pretoria	SA	britedy2@yahoo.co m
Dr	Olawale	Fatoba	University of Johannesburg	SA	drfatobasameni@g mail.com
Prof	Michael	Fitzpatrick	Coventry University	UK	Michael.fitzpatrick @coventry.ac.uk

	First Name	Last Name	Company/Organisation	Country	Email Address
Dr	Supriyo	Ganguly	Cranfield University	UK	s.ganguly@cranfield .ac.uk
Prof	Christoph	Genzel	Helmholtz-Zentrum Berlin für Materialien und Energie	Germany	genzel@helmholtz- berlin.de
Dr	Michael	Gharghouri	Canadian Nuclear Laboratories	Canada	MIchael.Gharghouri @cnl.ca
Dr	Jens	Gibmeier	Karlsruhe Institute of Technology	Germany	jens.gibmeier@kit.e du
Dr	Lara	Gigli	Elettra-sincrotrone Trieste	Italy	lara.gigli@elettra.eu
Mr	Daniel	Glaser	Council for Scientific and Industrial Research	SA	DGlaser@csir.co.za
Mr	Izak	Grobbelaar	North-West University	SA	nwu23446226@gm ail.com
Mr	Abraham	Haasbroek	North-West University	SA	abramhaasbroek@g mail.com
Prof	Danie	Hattingh	Nelson Mandela University	SA	danie.hattingh@ma ndela.ac.za
Dr	Thomas	Holden	Northern Stress Technologies	Canada	holdent@magma.ca
Mr	Nikola	Ivanovic	University of the Witwatersrand	SA	nikolaivanovicnikola @gmail.com
Dr	Saurabh	Kabra	STFC	UK	saurabh.kabra@stfc .ac.uk
Dr	Kelvin	Kemm	Necsa SOC Limited	SA	exec@nuclearafrica. co.za
Ms	Oratile	Kgaodi	Botswana International University of Science and Technology	Botswana	obkgaodi@gmail.co m
Ms	Malika	Khodja	University of Djillali Liabes	Algeria	khodja.malika7@gm ail.com
Mr	Dominik	Kiefer	Karlsruhe Institute of Technology	Germany	Dominik.Kiefer@kit. edu
Prof	Erich	Kisi	The University of Newcastle	Australia	erich.kisi@newcastl e.edu.au
Dr	Manuela	Klaus	Helmholtz-Zentrum Berlin für Materialien und Energie	Germany	klaus@helmholtz- berlin.de
Dr	Christina	Krywka	Helmholtz Zentrum Geesthacht	Germany	christina.krywka@h zg.de
Dr	Kristina	Langer	Air Force Research Laboratory	USA	kristina.langer@us.a f.mil
Dr	Hanna	Leemreize	Danish Technological Institute	Denmark	hle@teknologisk.dk
Mr	Mitchell	Leering	University of the Witwatersrand	SA	mitchell.leering@wi ts.ac.za
Dr	Vladimir	Luzin	ANSTO	Australia	vladimir.luzin@anst o.gov.au
Dr	Deon	Marais	Necsa SOC Limited	SA	deon.marais@necsa .co.za
Dr	Madindwa	Mashinini	University of Johannesburg	SA	mmashinini@uj.ac.z a

	First Name	Last Name	Company/Organisation	Country	Email Address
Dr	Kristián	Máthis	Charles University	Czech	mathis@met.mff.cu
				Republic	ni.cz
Prof	Mike	Meylan	The University of Newcastle	Australia	mike.meylan@newc
					astle.edu.au
Dr	Satoshi	Morooka	Japan Atomic Energy Agency	Japan	morooka@post.j-
	0			A	parc.jp
Dr	Ondrej	Muransky	ANSTO	Australia	ondrej.muransky@a
NAr	Muhammad	Nacom	City University of Hong Kong	China	nsto.gov.au
1411	Wulldillindu	Naeem	City Oniversity of Hong Kong	China	rindeeniz-
Dr	Mark	Newby	Eskom Holdings SOC Ltd	SΔ	mark newby@esko
DI	Wark	Newby		5/1	m.co.za
Prof	Joao	Nobre	University of the	SA	ioao.nobre@wits.ac
_			Witwatersrand	-	.za
Prof	Ismail	Noyan	Columbia University	USA	icn2@columbia.edu
Dr	Schadrack	Nsengiyumva	Rhodes University	SA	s.nsengiyumva@ru.
		0,	·		ac.za
Mr	Tshepo	Ntsoane	Necsa SOC Limited	SA	tshepo.ntsoane@ne
					csa.co.za
Dr	Sanjoo	Paddea	The Open University	UK	sanjooram.paddea
					@open.ac.uk
Dr	Thilo	Pirling	Institut Laue–Langevin	France	pirling@ill.eu
Prof	Claudia	Polese	University of the	SA	claudia.polese@wits
			Witwatersrand		.ac.za
Prof	Michael	Preuss	The University of Manchester	UK	michael.preuss@ma
	D. Al	Daharaa		<b></b>	nchester.ac.uk
IVIr	Mushfigur	Kanman	Institut Laue–Langevin	France	ranman@III.fr
Mr	Ranggi	Pamadhan	Coventry University		ramadhar@uni.cove
1411	Naliggi	Namaunan	coventry oniversity	UK	ntry ac uk
Dr	Joana	Rebelo	FRM II. Technische Universität	Germany	ioana.kornmeier@fr
5.	Joana	Kornmeier	München	Cermany	m2.tum.de
Mr	Anthony	Reid	University of Sheffield	UK	agpreid1@sheffield.
					ac.uk
Prof	Jesus	Ruiz-Hervias	Universidad Politecnica de	Spain	jesus.ruiz@upm.es
			Madrid		
Prof	Natasha	Sacks	University of the	SA	Natasha.Sacks@wits
			Witwatersrand		.ac.za
Dr	Mutsumi	Sano	Japan Synchrotron Radiation	Japan	musano@spring8.or
		_	Research Institute		.jp
Dr	Jan	Saroun	Nuclear Physics Institute, CAS	Czech	saroun@ujf.cas.cz
Dø	Daiga	Satavama	Toyota Control DO Labor Labor	керирііс	daigo @maale + +lak -
Ur	Dalgo	Seloyama	Toyota Central R&D Labs., Inc.	Japan	uaigo@mosk.tytlabs
Dr	Dor	Sigaard	Danish Technological Institute	Denmark	nech@teknologisk.d
ы		Christensen		Dennark	k
Dr	Petr	Sittner	Institute of Physics of the CAS	Japan	sittner@fzu.cz
		5		Japan	

	First Name	Last Name	Company/Organisation	Country	Email Address
Mr	Francois	Smith	North-West University	SA	swysnwu@yahoo.co m
Dr	Axel	Steuwer	University of Malta	Spain	axel.steuwer@gmail .com
Dr	Maria	Strantza	Los Alamos National Laboratory	USA	maria.strantza@lanl .gov
Dr	Hiroshi	Suzuki	Japan Atomic Energy Agency	Japan	suzuki.hiroshi07@ja ea.go.jp
Dr	Kenji	Suzuki	Niigata University	Japan	suzuki@ed.niigata- u.ac.jp
Dr	lan	Swainson	IAEA	Austria	i.swainson@iaea.or g
Mr	Shigehiro	Takajo	Los Alamos National Laboratory	USA	s-takajo@jfe- steel.co.jp
Mr	Christiaan	Van Der Merwe	North-West University	SA	23473207nwu@gm ail.com
Mr	Sean	Van Staden	University of the Witwatersrand	SA	sean.vanstaden2@s tudents.wits.ac.za
Dr	Andrew	Venter	Necsa SOC Limited	SA	andrew.venter@nec sa.co.za
Mr	Maxwell	Vhareta	University of the Witwatersrand	SA	mvhareta@gmail.co m
Dr	Sven	Vogel	Los Alamos National Laboratory	USA	sven@lanl.gov
Mr	Florian	Vollert	Karlsruhe Institute of Technology	Germany	florian.vollert@kit.e du
Prof	Xun-Li	Wang	City University of Hong Kong	China	xlwang@cityu.edu.h k
Prof	Chris	Wensrich	The University of Newcastle	Australia	christopher.wensric h@newcastle.edu.a u
Prof	Krzysztof	Wierzbanowski	AGH University of Science and Technology	Poland	wierzbanowski@fis. agh.edu.pl
Prof	Philip	Withers	The University of Manchester	UK	philip.withers@man chester.ac.uk
Dr	Wanchuck	Woo	Korea Atomic Energy Research Institute	Korea	chuckwoo@kaeri.re. kr
Dr	Marcin	Wronski	AGH University of Science and Technology	Poland	mwronskii@gmail.c om
Prof	Shu Yan	Zhang	Centre of Excellence for Advanced Materials	China	shuyan.zhang@cea mat.com

Country	#	Country	#	Country	#	Country	#	Country	#
Algeria	1	Canada	2	France	4	Korea	1	UK	11
Australia	5	China	3	Germany	8	Poland	3	USA	13
Austria	1	Czech Republic	3	Italy	1	SA	27	Total	93
Botswana	1	Denmark	2	Japan	6	Spain	2		

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전 선 Muránsky O.	Woo W.		17:40
Mashinini P.M.	*Glaser D.	Mecl meth diffr	17:20
ructu terisa *Čapek J.	Apel D.	hanic Iods v actio	17:00
tion Don Brown	John Bouchard	al s. n	16:30
Chair: Hiroshi Suzuki	": Hahn Choo	Chair	
ffee	Cof		16:05
Suzuki H.	Noyan I.C.		15:45
Nsengiyumva S.	Krywka C.	Teo In:	15:25
Othe *Brokx J.	Klaus M.	:hniqı strum	15:05
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Petr Sittner	Chris Wensrich		14:15
Chair: Daigo Setoyama	Chedly Braham	Chair: (	
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& Bernard D.	*Brügger A.	n & N	11:20
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Supriyo Ganguly	Kristián Máthis	ling	10:30
Chair: Wanchuck Woo	Axel Steuwer	Chair:	
e break	Coffee		10:00
	Phil Withers		09:15
	Don Brown		09:10
	Kelvin Kemm		08:50
	Andrew Venter		08:45
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tration	Regist		08:00
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er			*Eisensmith D.	*Van Staden S.N.	*Ntsoane T.P.	Fatoba O.S.	Kristina Langer	ir: Sven Vogel		Fatoba O.S.	Kisi E.	*Edward A.B.	Xun-Li Wang	Nichael Fitzpatrick		Kiefer D.	Doubell P.	Mark Newby	hair: Ke An			ım: Ndau/Nari	tember

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18:30	17:50	17:00	15:30	15:15	14:00	12:45	12:00	10:45	09:30	08:15		Sp
Welcome Function	Dr Andrew Venter	Prof Phil Withers	Dr Sven Vogel	Coffee	Dr Vladimir Luzin	Dr Saurabh Kabra	Lunch	Dr Ondrej Muransky	Dr Don Brown	Dr Tom Holden	Room: Ndau	ring School: Monday 18 Septem
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SKUKUZA, SOUTH AFRICA, 19-21 SEPTEMBER 2017

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Lunch	Don Brown	Tom Holden	Chair:Andrew Venter	chai Vogel S.C.	3D/4E racter On Coules 	risati Daigo Setoyama	Chair: Christoph Genzel	Coffee b	Defc M *Reid A.	ormat odelli *Naeem M.	wichael Gharghouri	Chair: Shu Yan Zhang	Cev Noyan	Chair: Don Brown	Room: Ndlopfu	Conference Day 3: Thursday
ſ				Prov Strantza M.	veldir Vollert F	& Hahn Choo	Chair: Xun-Li Wang	reak	Fi P *Vhareta M.	atigue Creep lastici Sano S.	Kenji Suzuki	Chair: Thilo Pirling			Room: Ndau/Nari	/ 21 September