Complimentary diffraction techniques at Necsa

UNIVERSITY OF JOHANNESBURG
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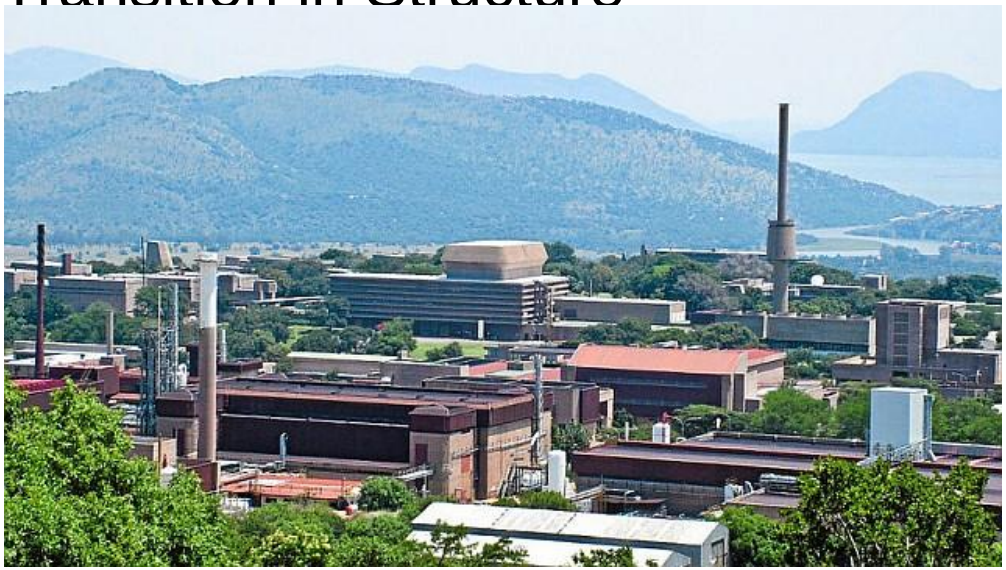
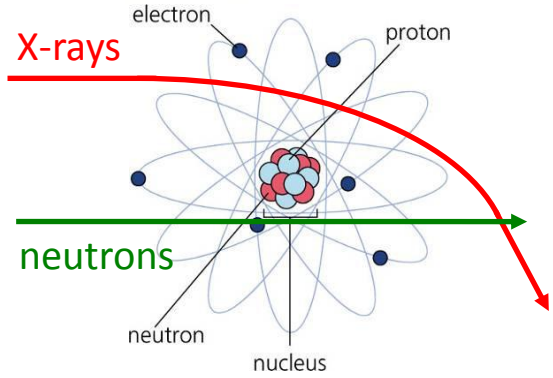
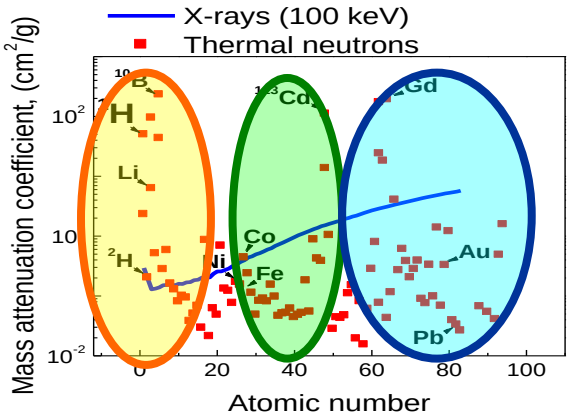
Outline

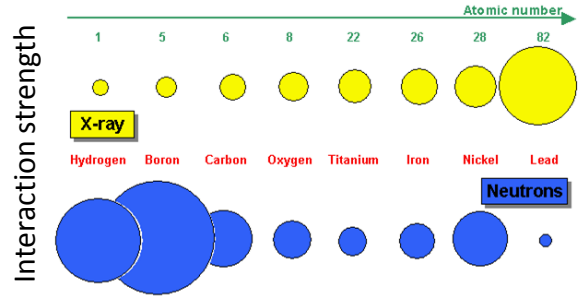
- X-ray and Neutron interactions with matter
- SAFARI-1 at Necsa
- Diffraction instruments at Necsa
  - Materials Probe for Internal Strain Investigations (MPISI)
  - Powder Instrument for Transition in Structure Investigations (PITSI)
  - Bruker D8 Advance
  - Bruker D8 Discover
- Project examples
  - Residual stress
  - Powder diffraction
- Beam time application



X-ray and Neutron interactions with matter







X-ray interact with electrons, scattering depends on atomic number (Z)

Neutrons interact with nucleus, scattering depends on isotopes

Atom size is ~1Å (10^{-10} m), nucleus size is ~10 fm (10^{-14}m)
According to neutrons ~99.9999999% of matter is "empty space"

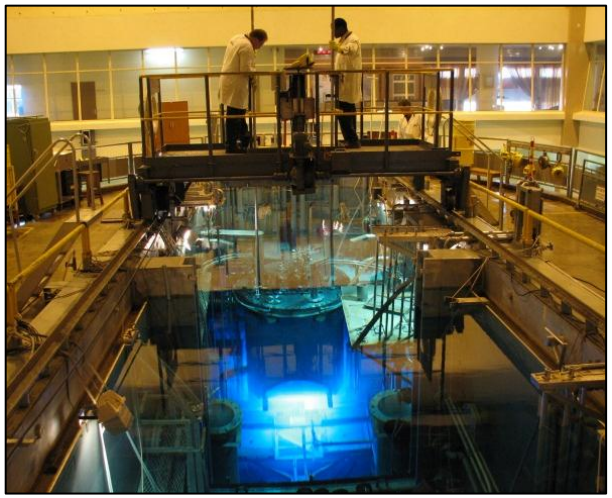
Attenuation of radiation: $I = I_0 e^{-\mu \cdot \ell}$ and $d_{½} = ln2 / \mu$ with:

- μ - linear attenuation coefficient
- ℓ - neutron flight path in material
- $d_{½}$ - half-attenuation length

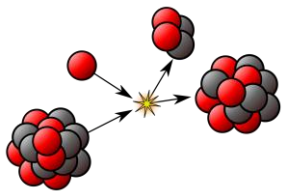
	X-ray (Cu-K_α) λ=1.54Å	Synchrotron 100keV λ=0.124Å	Neutrons λ=1.0 Å
Al	53 µm	22 mm	70.5 mm
Fe	15 µm	3.5 mm	30 mm
Cu	15 µm	2 mm	8.1 mm
Ti	11 µm	6 mm	15.8 mm
Pb	3 µm	0.3 mm	18.8 mm

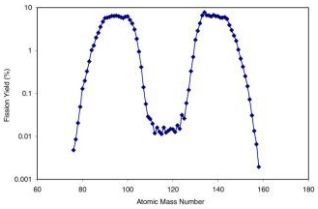
Atom	Z	25 meV neutrons [cm]	8 keV X-rays
H	1	0.288	28.388 cm
C	6	1.580	97 µm
N	7	2.140	106 µm
O	8	5.520	60 µm
Al	13	9.480	73.588 µm
ti	22	1.740	10.888 µm
V	23	1.350	7.382 µm
Pd	46	1.290	4.125 µm
Cd	48	0.008	5.129 µm
Au	79	0.159	2.498 µm

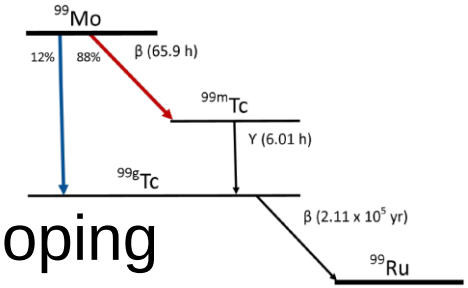
SAFARI-1 at Necsa



- South African Fundamental Atomic Research Installation
- 20MW tank in pool
- Commissioned in 1965 (54 years)
- ~300 days availability p/a
- Irradiation services
- In core Neutron Activation Analysis
- Medical isotopes







- Neutron transmutation silicon doping
  - $^{30}Si(n,\gamma)^{31}Si \rightarrow ^{31}P + \beta^-$ ($T_{1/2} = 2.62h$)

Diffraction at Necsa: X-Rays

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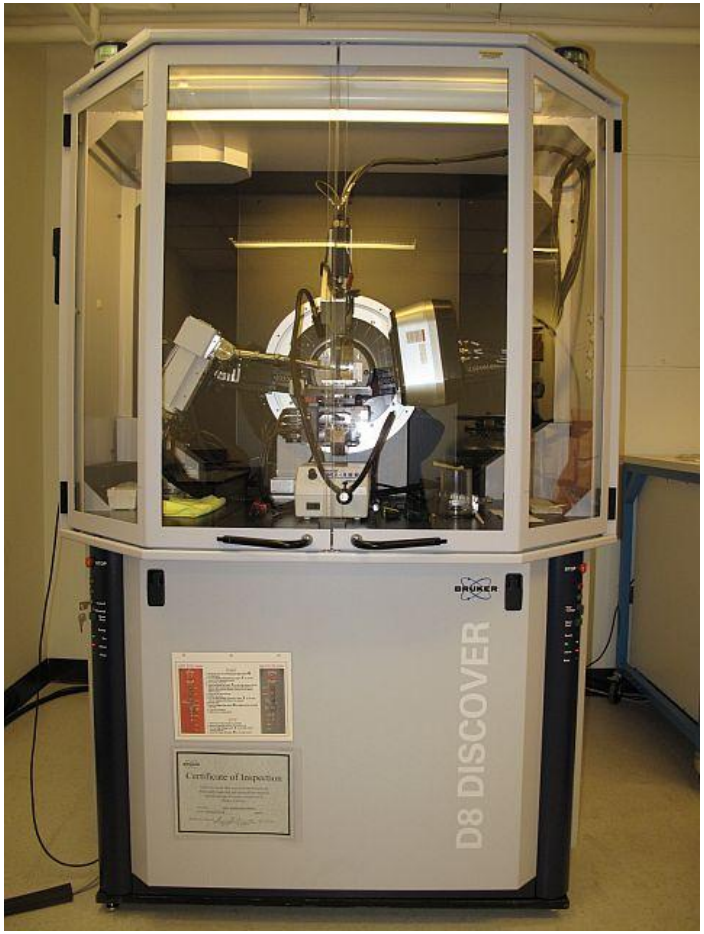
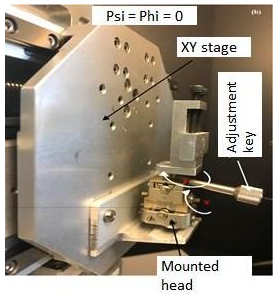
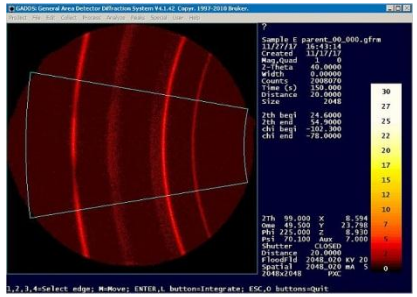
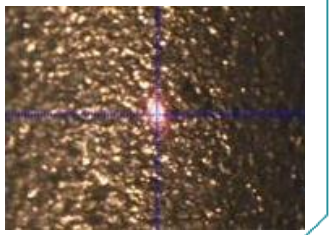
- Bruker D8 Discover with GADDS (strain scanning)



X-ray Tubes	Cu, Co, Cr
Beam conditioning	PG incident beam monochromator
Beam sizes	2.0, 0.8, 0.5, 0.3, 0.1, 0.05 mm
Sample stage	¼ cradle with integrated ϕ, χ, x, y, z
Sample setup	Video laser system
Detector	Vantec 500
Two-theta range	$10° \leq 2\theta \leq 108°$ (front) $10° \leq 2\theta \leq 158°$ (back)
Software	Stress: Leptos Texture: Multex Area 2







Diffraction at Necsa: X-Rays

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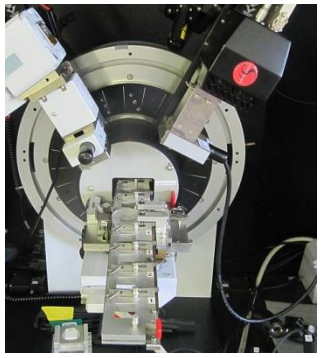
❑ Bruker D8 Advance (powder diffraction)

X-ray Tubes	Cu
Beam conditioning	Göble mirror Monocap
Beam sizes	0.1 x 15 mm^2; 1 mm collimator
Sample stage	7 position Flipstick, Large component XYZ stage, Capillary stage, Pseudo-SAXS stage, Hermitically sealed stages
Sample setup	Laser system
Detector	LynxEye
Software	ICDD, Topas, NanoFit (Pseudo SAXS)



Diffraction at Necsa: Neutrons

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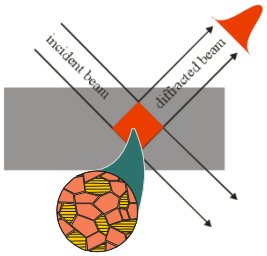
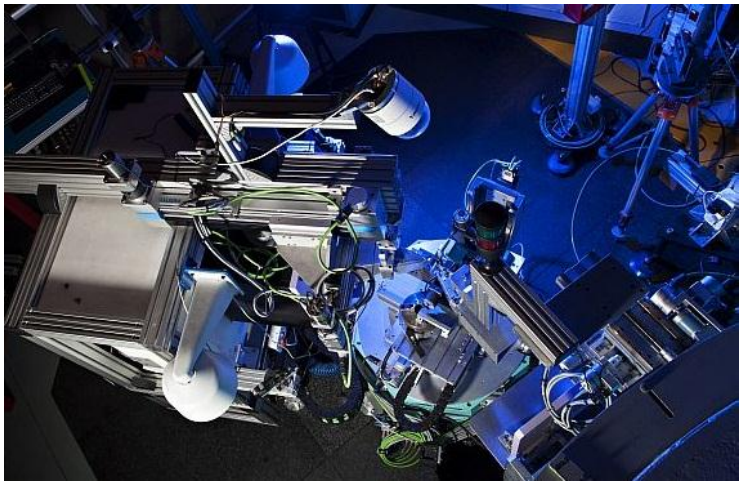
❑ MPISI (strain scanning)

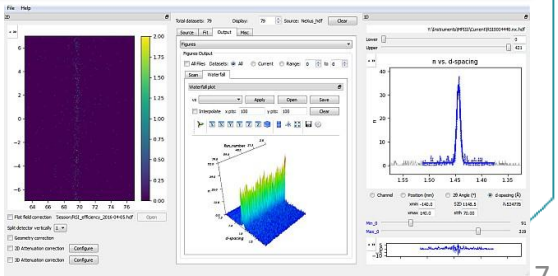
Materials Probe for Internal Strain Investigations



Spotted hyena in Zulu



Si Monochromator (331), (333), (011)	at $2\theta_M = 83.5°$ $\lambda = 1.67Å$, $\lambda = 1.49Å$, $\lambda = 5.11Å$
Beam size	Variable slits: Hor: 0.3 – 5 mm Ver: 0 – 20 mm
	Radial collimators 1, 2, 5, 10 mm
Sample stage	Huber integrated XYZ 250 kg and 250 mm travel ¼ cradle with integrated Φ
Sample setup aids	Telecentric camera, laser levels, theodolites
Detector	Denex 300 mm x 300 mm
	Two-theta range: $10° \leq 2\theta \leq 130°$
Resolution	$\Delta d/d = 3 \times 10^{-3}$



Diffraction at Necsa: Neutrons

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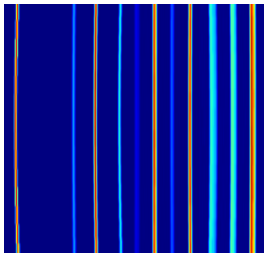
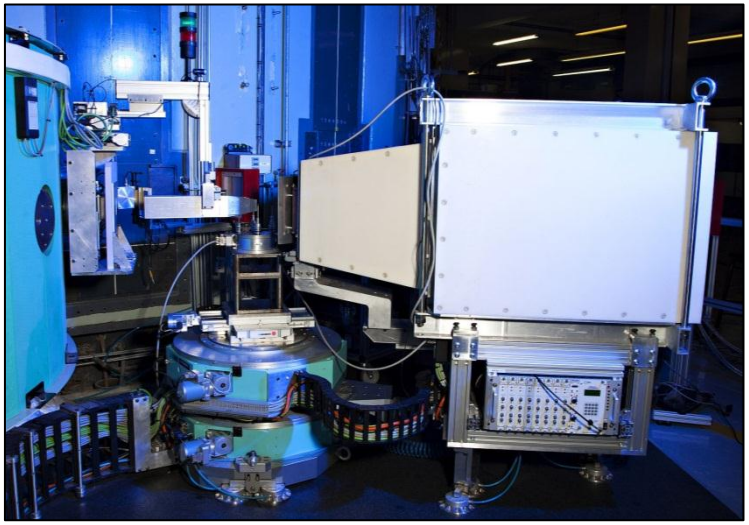
❑ PITSI (powder diffraction)

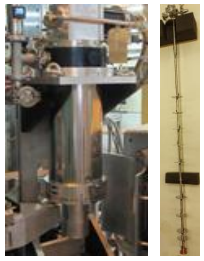
Powder Instrument for Transition in Structure Investigations



Zebra in Sotho

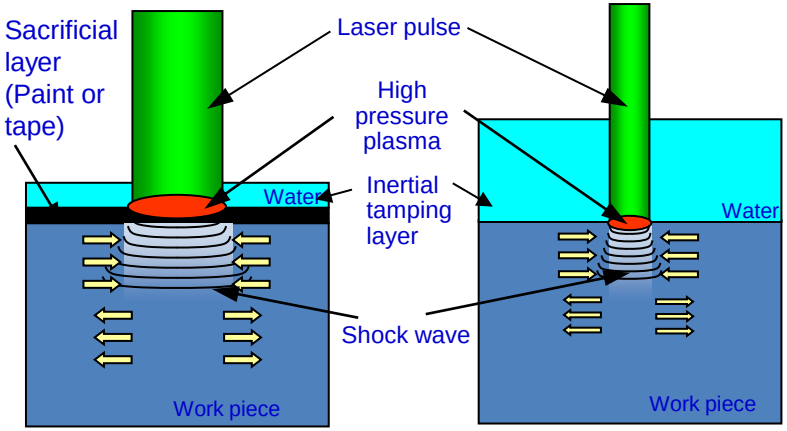


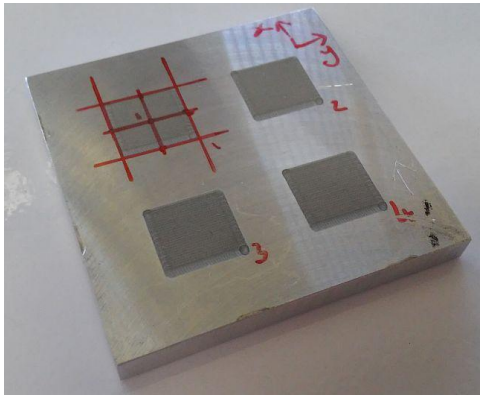
Monochr. Si (331) Si (551)	70° 90° 1.43Å 1.76 Å 0.87Å 1.07Å
Beam size	Variable: Hor: 1 – 20 mm, Ver: 1 – 50 mm
	Radial collimator
Sample stage	Huber integr. XYZ, 250 kg and 250 mm travel
Sample setup	Theodolites + Neutron camera
Detector	15 x Reuter Stokes tubes 660 mm (hor) x 375 mm (ver)
	Two-theta range: $10° \leq 2\theta \leq 130°$
Resolution	$\Delta d/d = 3 \times 10^{-3}$
Sample env.	Top-loader vacuum furnace: 400 K < T < 1800 K Bottom-loader cryostat: 4.5 K < T < 320 K Top-loader cryostat: 1.5 K ≤ T ≤ 800

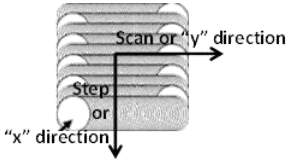


Project examples - Stress related

- Effect of Varying Laser Shock Peening Parameters on Aluminium Samples - Surface investigations

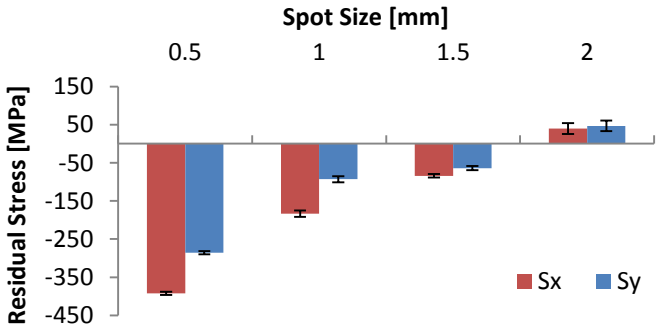




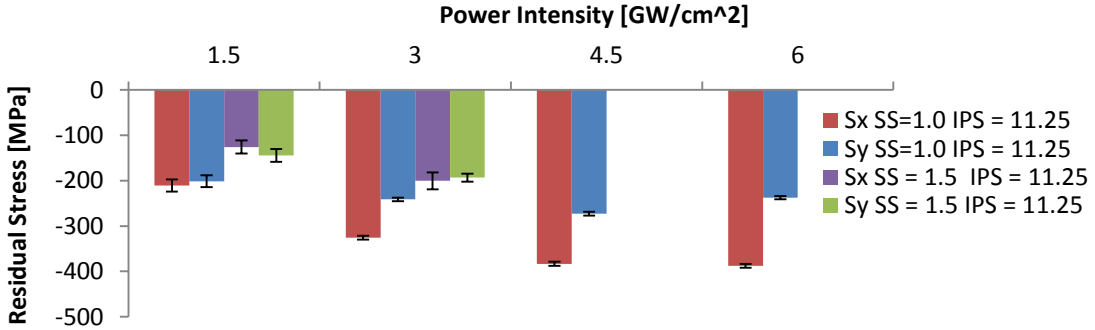


PI: Power intensity [GW/cm^2]
SS: Spot size diameter [mm]
IPS: Measure of overlap

Effect of Spot Size at PI=3 IPS = 11.25



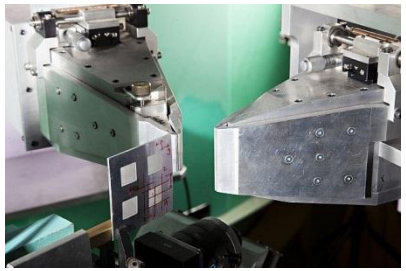
Effect of Power Intensity at IPS = 11.25 for different spot sizes

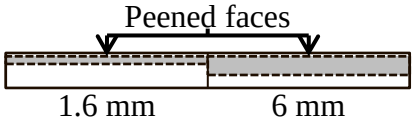


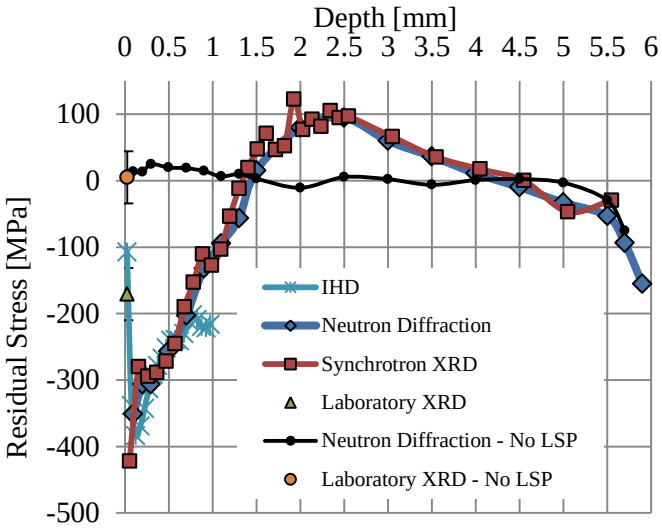
Mr. Sean van Staden, School of Mechanical, Industrial and Aeronautical Engineering, University of the Witwatersrand

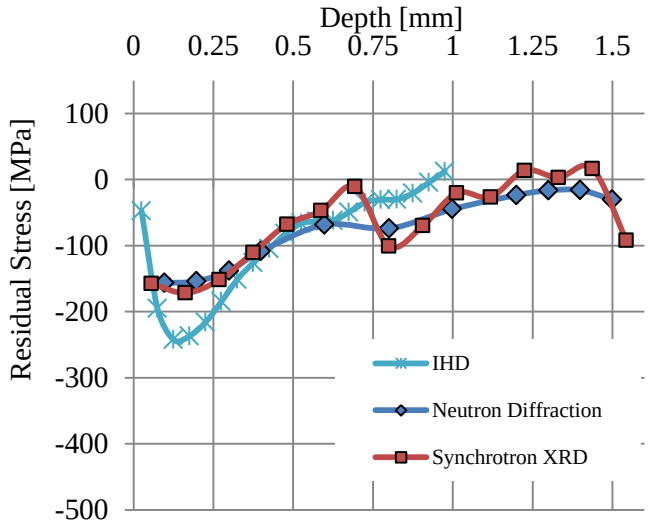
Project examples - Stress related

- Effect of Varying Laser Shock Peening Parameters on Aluminium Samples - Depth resolved study
  - Power intensity of 3 GW/cm^2, a spot diameter of 1.5 mm and a coverage of 500 spots/cm^2
  - Neutron gauge volume 0.3×10×0.3 mm^3







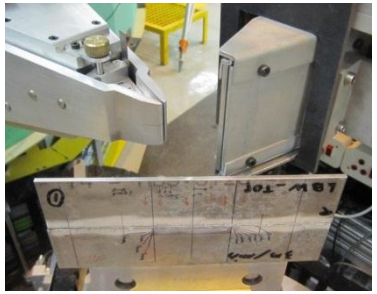


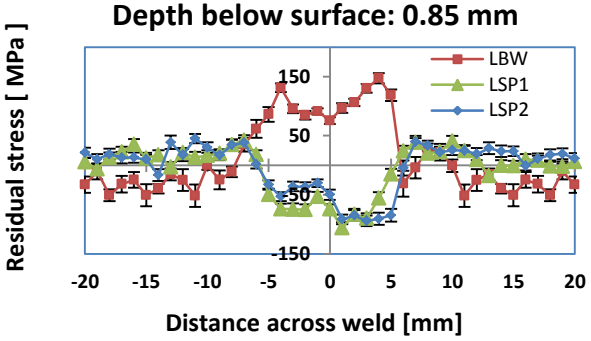
Depth-resolved residuals stress results in the x-direction obtained from multiple methods on the 6 mm and 1.6 mm thick samples

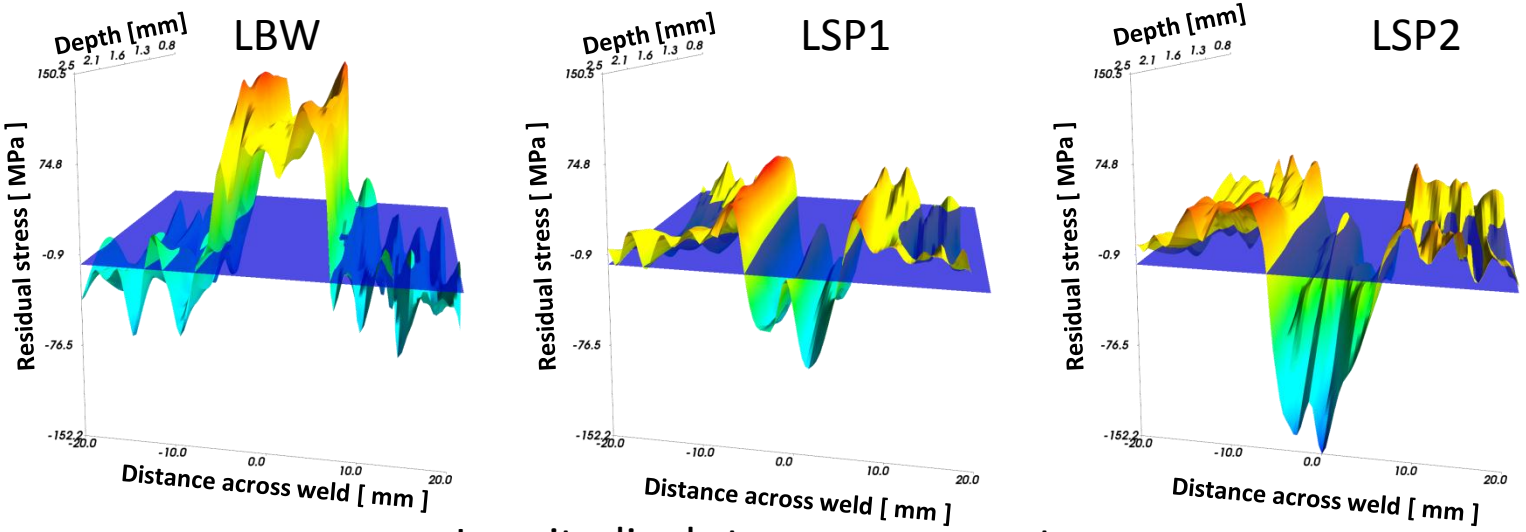
Mr. Sean van Staden, School of Mechanical, Industrial and Aeronautical Engineering, University of the Witwatersrand

Project examples - Stress related

- Laser Beam Welded aluminium plates: Comparison of as-welded and with LSP
  - 3.3 mm thick plate
  - LBW: Laser beam welded
  - LSP1: LSP treated 1 side
  - LSP2: LSP treated 2 sides





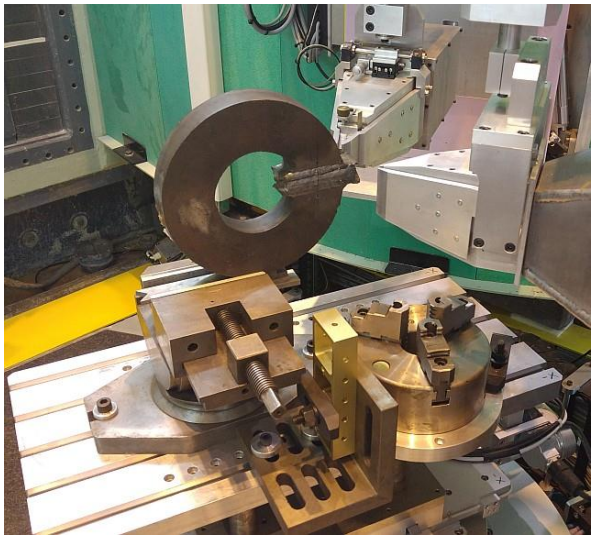


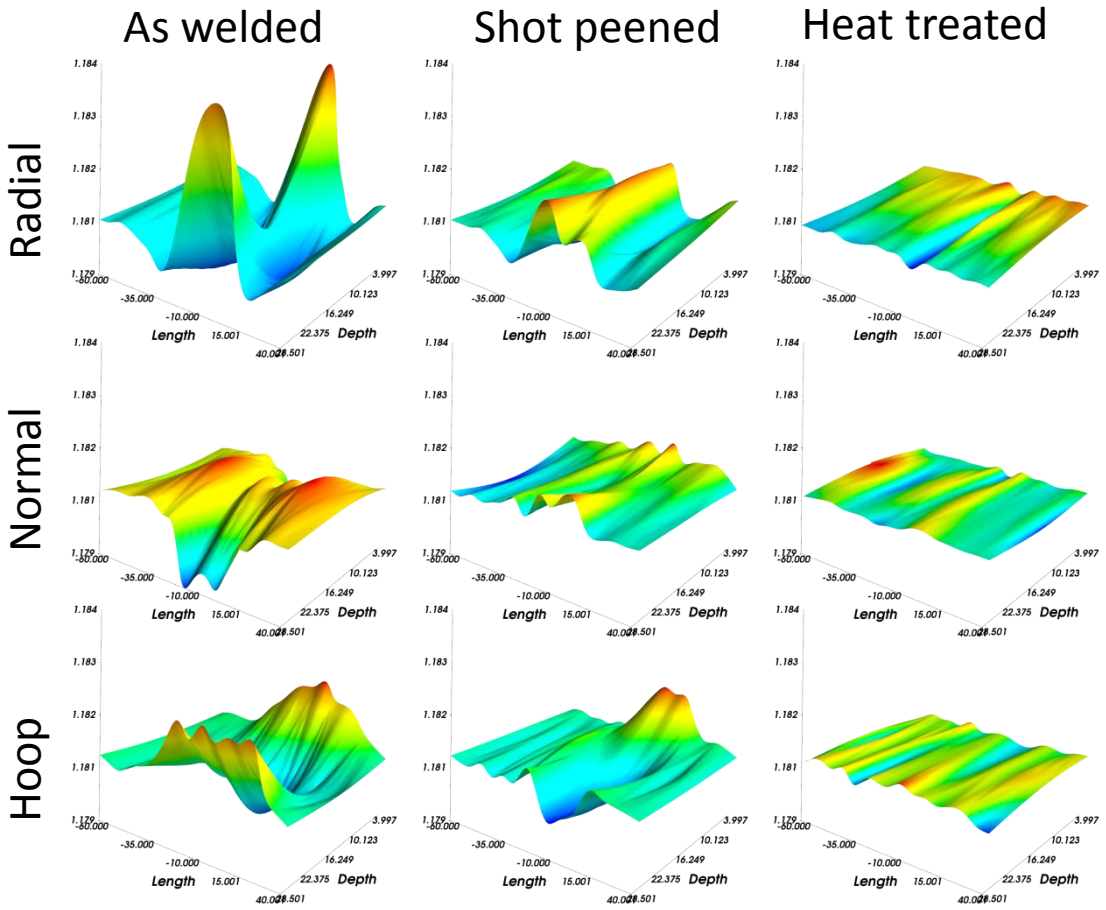
Longitudinal stress components

Dr. Daniel Glaser, CSIR National Laser Centre

Project examples - Stress related

- Large engineering samples: Welded, peened, heat treated rings

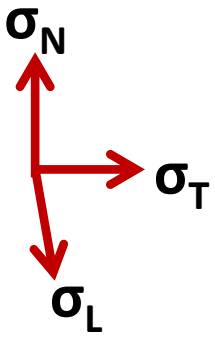




Prof. Gerrit van Rooyen, Department of Material Science and Metallurgical Engineering, University of Pretoria

Project examples - Stress related

- Welded mild steel plate
  - Sample size: 300 × 300 × 17 mm³
  - Gauge volume: 3 × 3 × 3 mm³

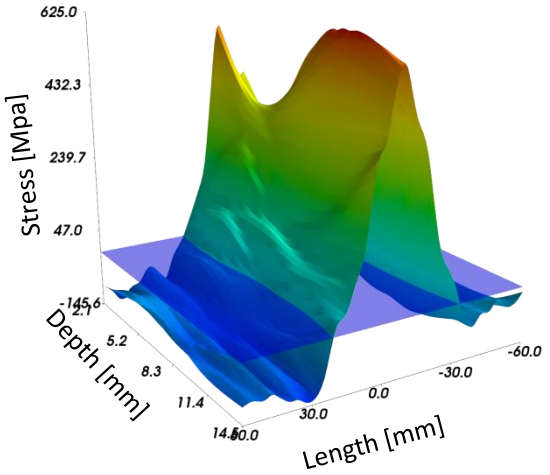


Sample orientations:
Normal component to longitudinal component

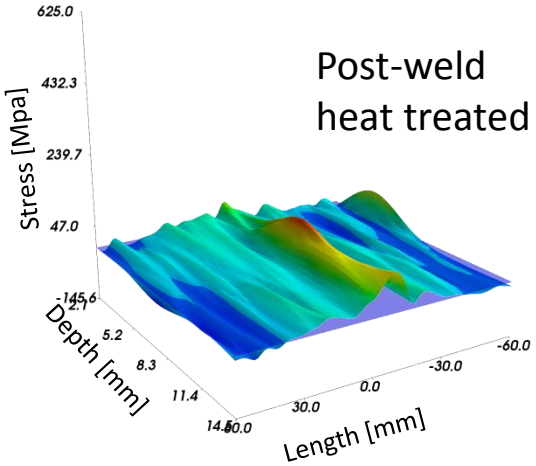
Mr. Deon de Beer, University of Pretoria

Longitudinal stress components

As welded



Post-weld heat treated



Project examples - Stress related

❑ Characterisation of Welding-Induced Residual Stress

- Gauge volume 2 × 2 × 2 mm^3 collimator

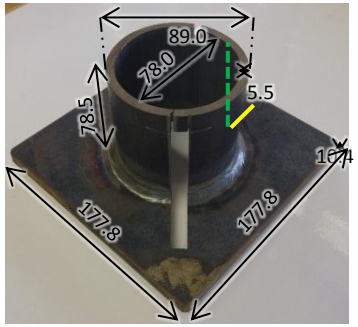
Sample parameters & Maximum stress results

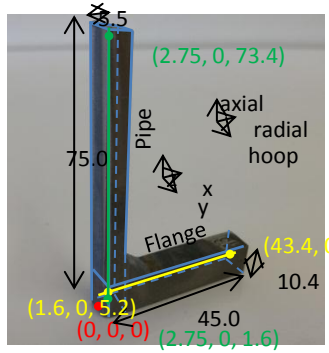
Sam	I	V	S	FR	Mode	Flange [MPa]			Pipe [MPa]		
	[A]	[V]	$\left[\frac{mm}{s}\right]$	$\left[\frac{mm}{s}\right]$		Hoop	Axial	Radial	Hoop	Axial	Radial
4	380	30	10	18.3	CA	371	-11	37	431	106	61
6	380	25	8	18.3	CA	402	52	78	423	85	58
7	360	25	8	18.3	CA	359	12	36	295	-74	-16
8	360	25	10	18.3	CA	292	64	82	357	150	1
9	380	25	10	18.3	CA	425	66	130	507	132	69
12	360	30	8	16.7	CW	407	51	80	409	41	53
13	360	30	8	18.3	CW	375	49	42	381	-11	-26
15	360	25	8	18.3	CW	355	27	65	558	203	148
16	360	25	8	16.7	CW	412	70	117	507	76	41

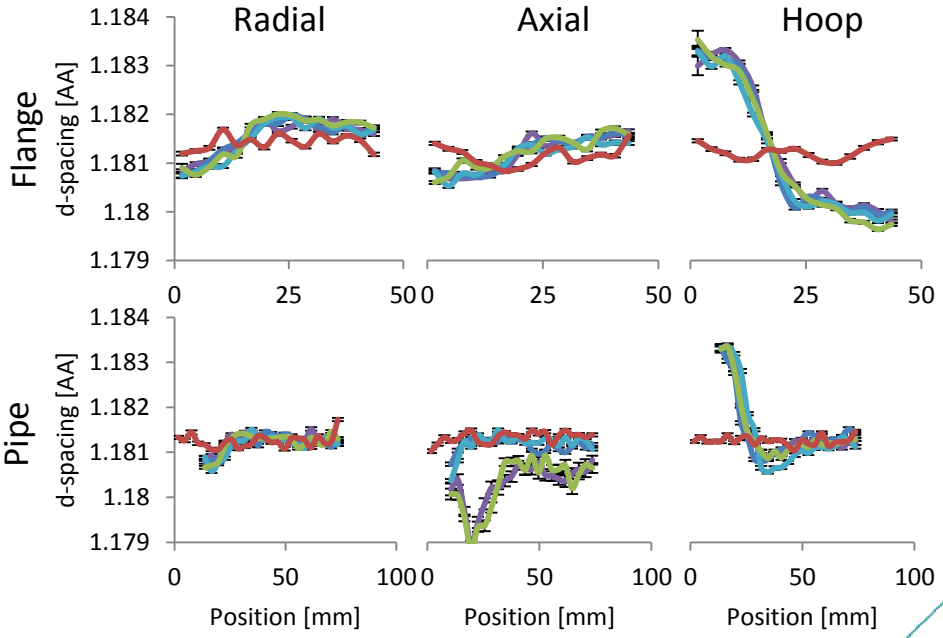
	Flange			Pipe		
Position	Hoop	Axial	Radial	Hoop	Axial	Radial
1	8	4	7	7	7	13
2	15	7	4	8	13	7
3	7	15	13	13	12	8

Result ranking, winner 360A, 25V and 18.3mm/s wire-feed rate.

Mr. M. Clyde Zondi, UKZN School of Engineering ,UKZN

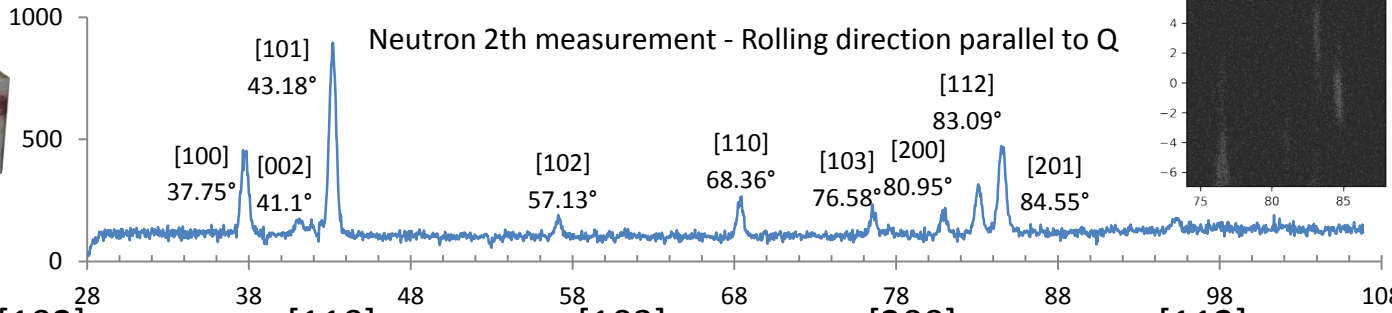


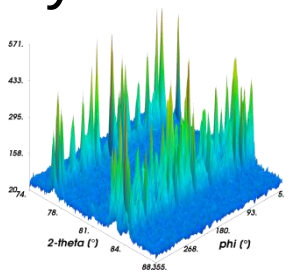


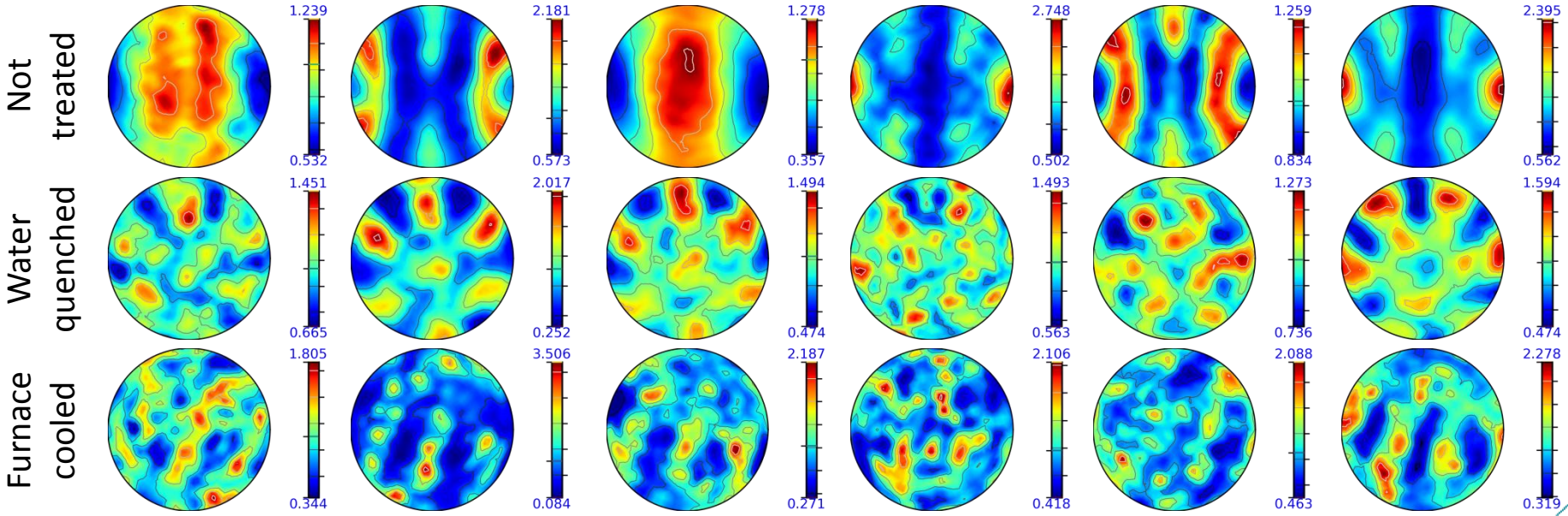


Project examples - Stress related

- Texture change due to heat treatment of Ti-6Al-4V alloy.



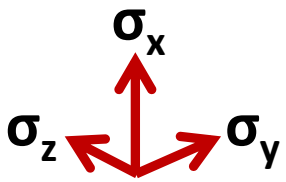


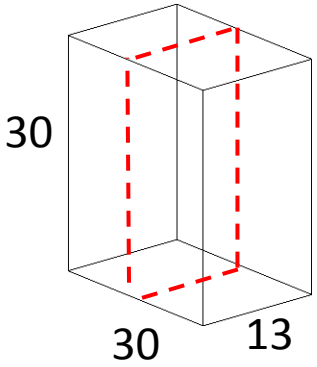


Mr. Stephen Masete, University of Pretoria, Department of Materials Science and Metallurgical engineering

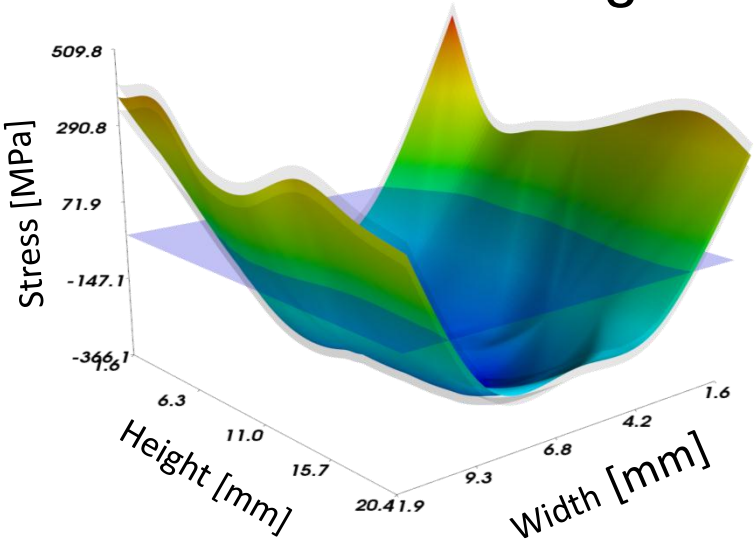
Project examples - Stress related

- Additive manufactured titanium by selective laser melting
  - Sample size 30 × 30 × 13 mm^3
  - Gauge volume 2 × 2 × 6 mm^3





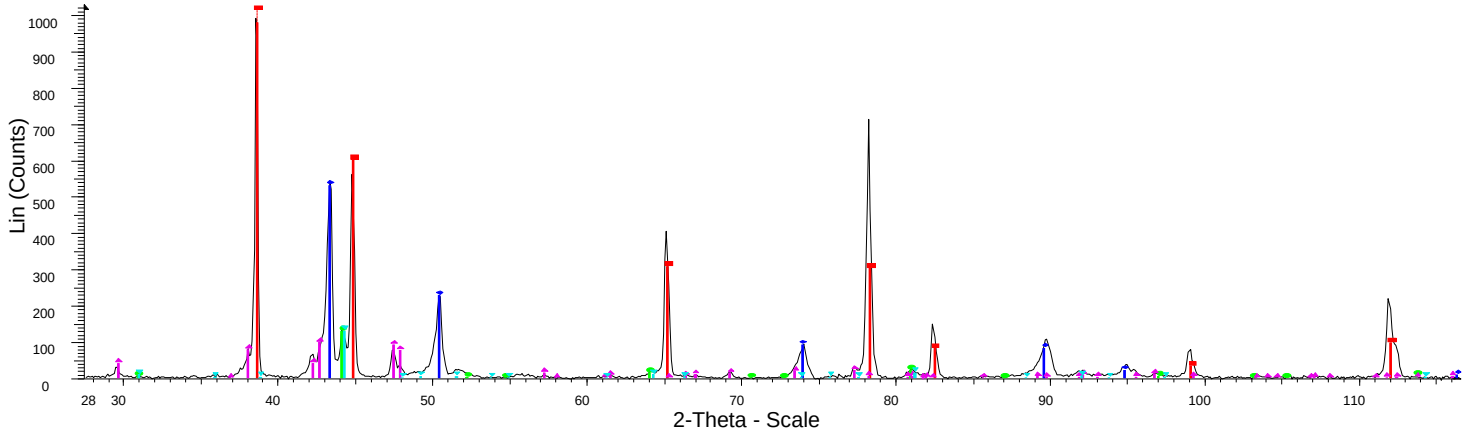
Map of residual stress σ_z on the cross-section indicated



Dr Victoria Cain, Department of Mechanical Engineering, Peninsula University of Technology

Project examples - Powder diffraction

- Micro Diffraction
  - Phase identification in Friction Spot Stir Weld



Y + 0.1 mm - File: FSSW 11 Al-Cu pt1_working range.out - Type: 2Th alone - Start: 27.400 ° - End: 116.600 ° - Step: 0.100 ° - Step time: 210. s - Anode: Cu - WL1: 1.54184 - WL2: n.a. - Generator kV: 20 kV - Generat

Operations: Enh. ackground 2.570,1.000 | Import

01-071-4622 (A) - Aluminum - Al - WL: 1.54184 - Cubic - a 4.05000 - b 4.05000 - c 4.05000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Fm-3m (225) - Face-centered

01-071-4611 (A) - Copper, syn - Cu - WL: 1.54184 - Cubic - a 3.62700 - b 3.62700 - c 3.62700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Fm-3m (225) - Face-centered

01-073-2762 (I) - Copper Aluminum - Cu3Al - WL: 1.54184 - Cubic - a 5.82000 - b 5.82000 - c 5.82000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Fm-3m (225) - Face-centered

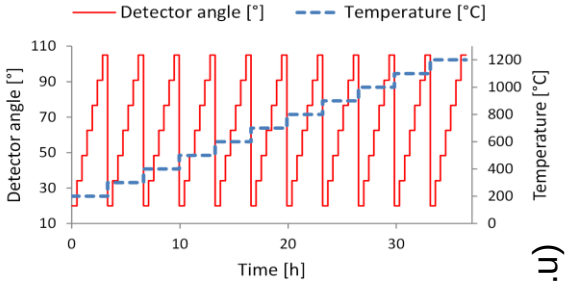
01-071-5027 (*) - Khatyrkite - CuAl2 - WL: 1.54184 - Tetragonal - a 6.07000 - b 6.07000 - c 4.89000 - alpha 90.000 - beta 90.000 - gamma 90.000 - I4/mcm (140) - Body-centered

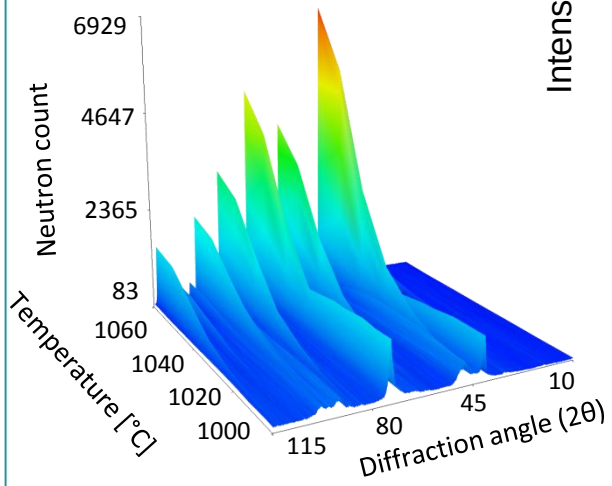
00-024-0003 (C) - Aluminum Copper - Cu9Al4 - WL: 1.54184 - Cubic - a 8.70270 - b 8.70270 - c 8.70270 - alpha 90.000 - beta 90.000 - gamma 90.000 - P-43m (215) - Primitive

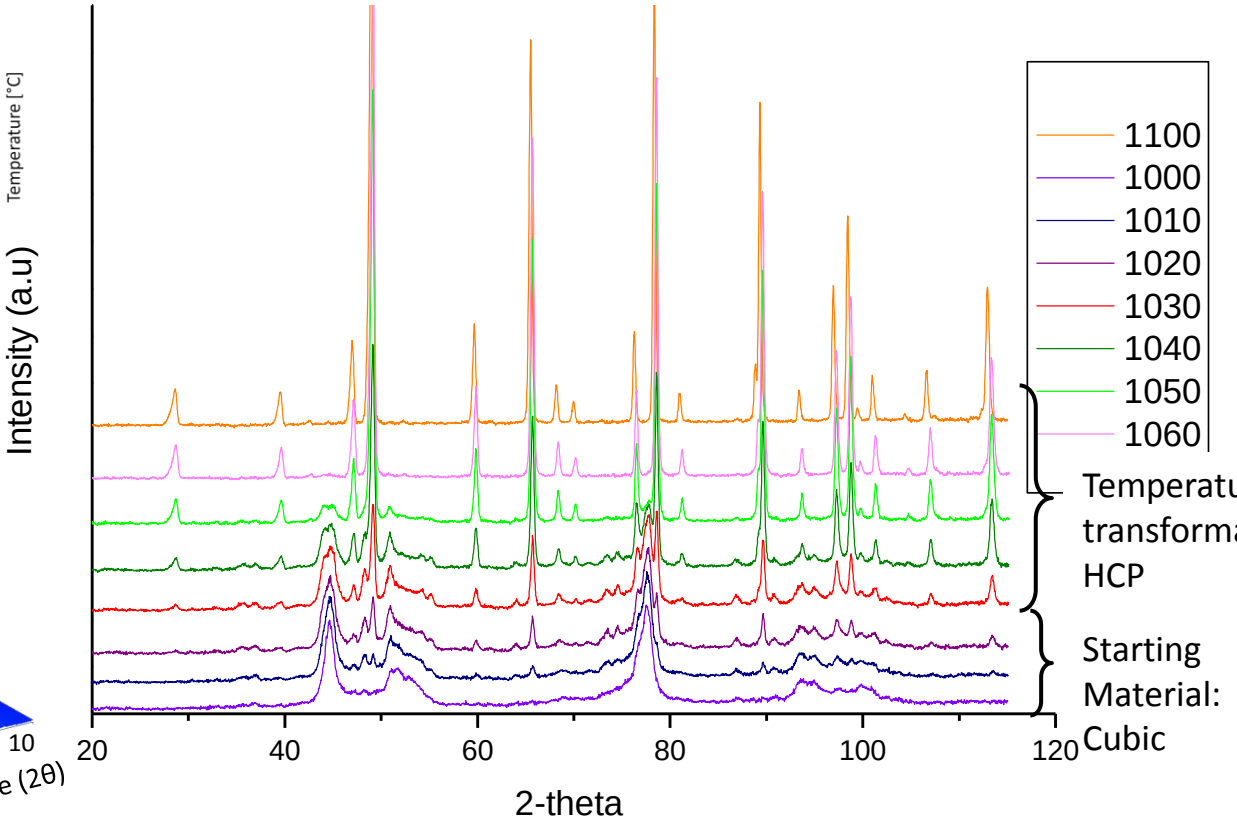
Mr P. Mubiayi, University of Johannesburg

Project examples - Powder diffraction

- In-situ phase transformation study of Al_2O_3 :Nuclear waste storage starter material



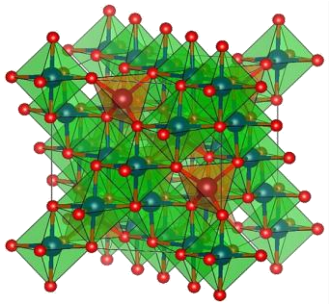


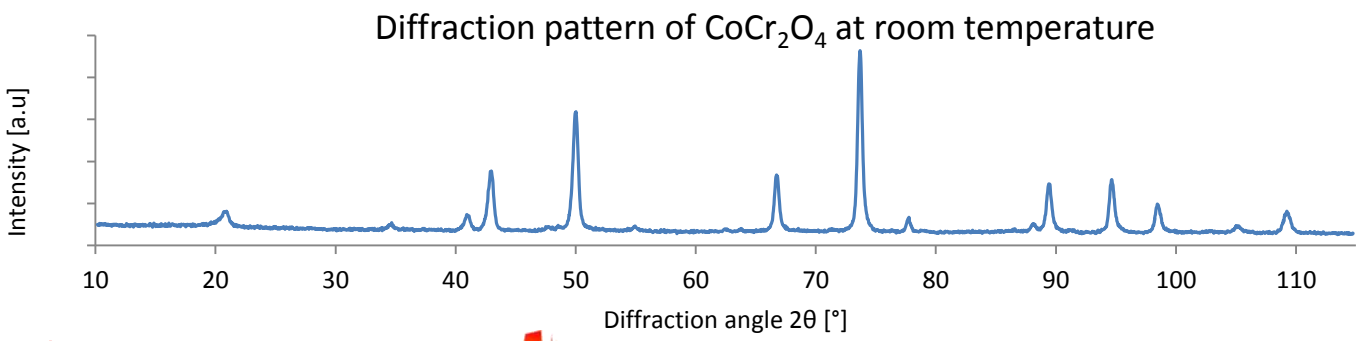


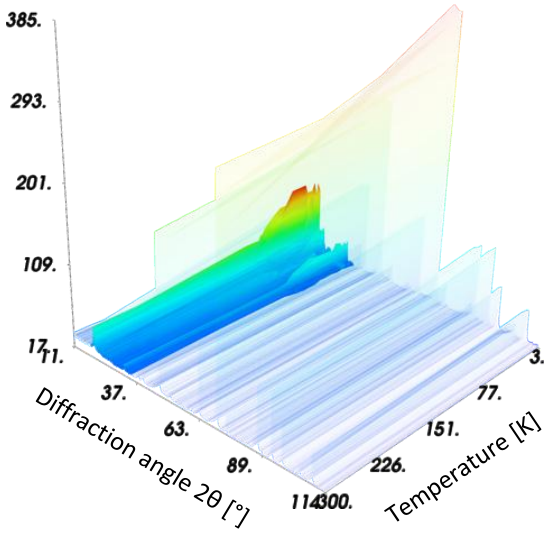
Ms. Mualusi Nelwamondo, Nuclear Waste Group, Necsa

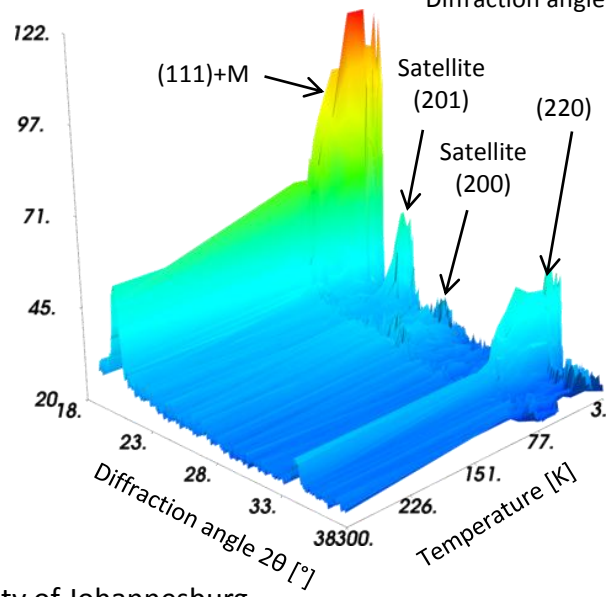
Project examples - Powder diffraction

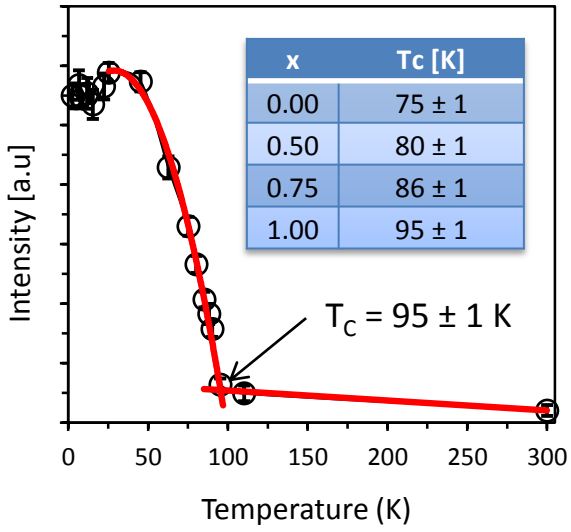
- Structure and magnetic phase transitions in $(Ni_{1-x}Co_x)Cr_2O_4$ spinel nanoparticles











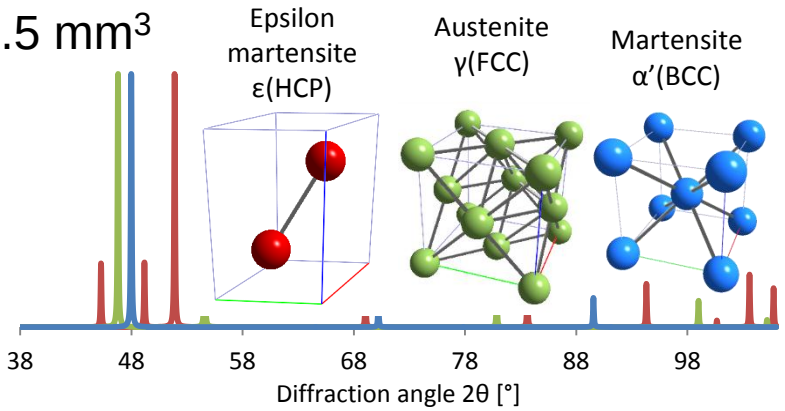
x	Tc [K]
0.00	75 ± 1
0.50	80 ± 1
0.75	86 ± 1
1.00	95 ± 1

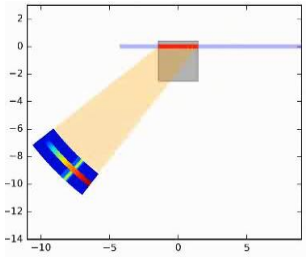
Dr P. Mohanty, Faculty of Science, University of Johannesburg

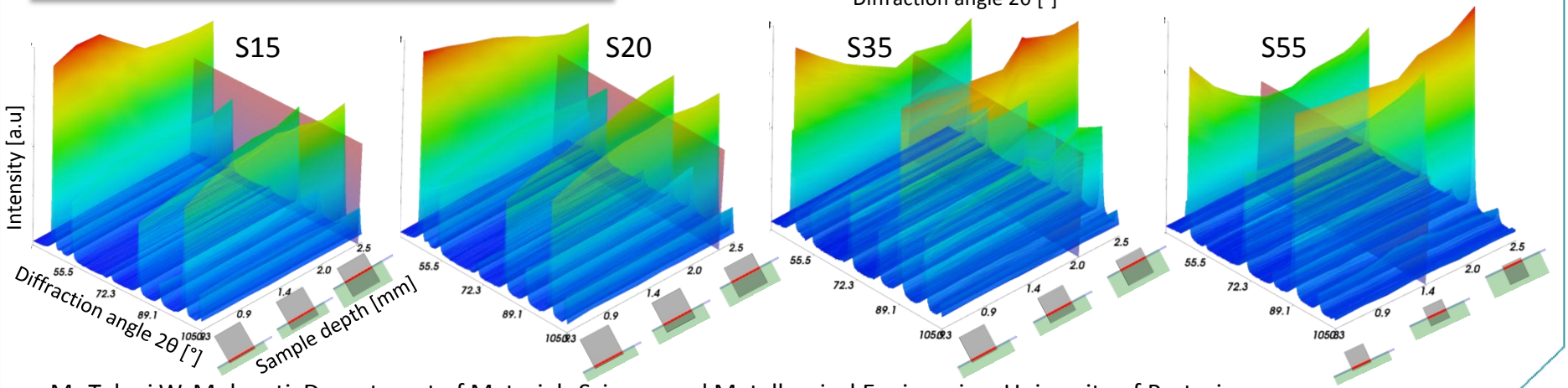
Project examples - Powder diffraction

- **Depth resolved powder diffraction:** Investigation of volume fractions of retained austenite, strain-induced martensite and epsilon martensite in cold worked AISI 301 stainless steel
  - Gauge volume 0.3 x 20 x 8.5 mm^3

Sample name	Reduction [%]	Width [mm]	Depth [mm]	Length [mm]
S15	15	5	5	75
S20	20	4.9	4.7	75
S35	35	4.1	4.1	75
S55	55	2.9	2.9	75



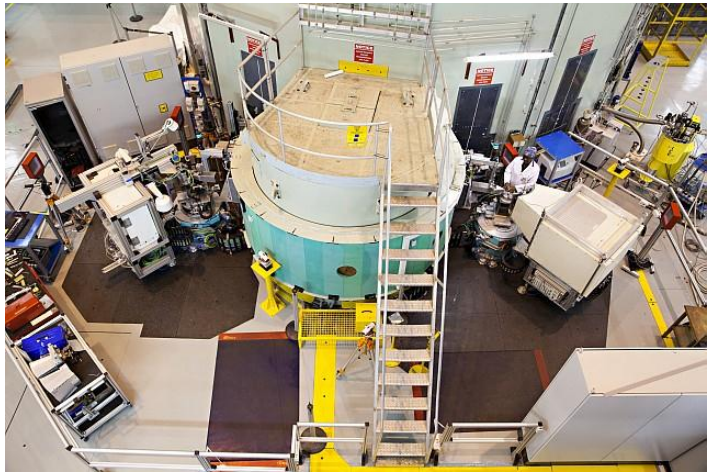




Mr Tulani W. Mukarati, Department of Materials Science and Metallurgical Engineering, University of Pretoria

Beam time application

- Active user program
- Scientific merit
- Beam time application form available from www.necsa.co.za/mpisi or www.necsa.co.za/pitsi
  - Scientific background and significance
  - Preliminary research work / simulated results
  - Sample description
  - Proposed experiment
  - Measurement methodology/planning
  - References



- Instrument scientist contacts
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  - Neutron strain scanning: Dr. Deon Marais (deon.marais@necsa.co.za)
  - Neutron powder diffraction: Ms. Zeldah Sentsho (zeldah.sentsho@necsa.co.za)
  - X-Ray instruments: Mr. Tshepo Ntsoane (tshepo.ntsoane@necsa.co.za)

Questions?