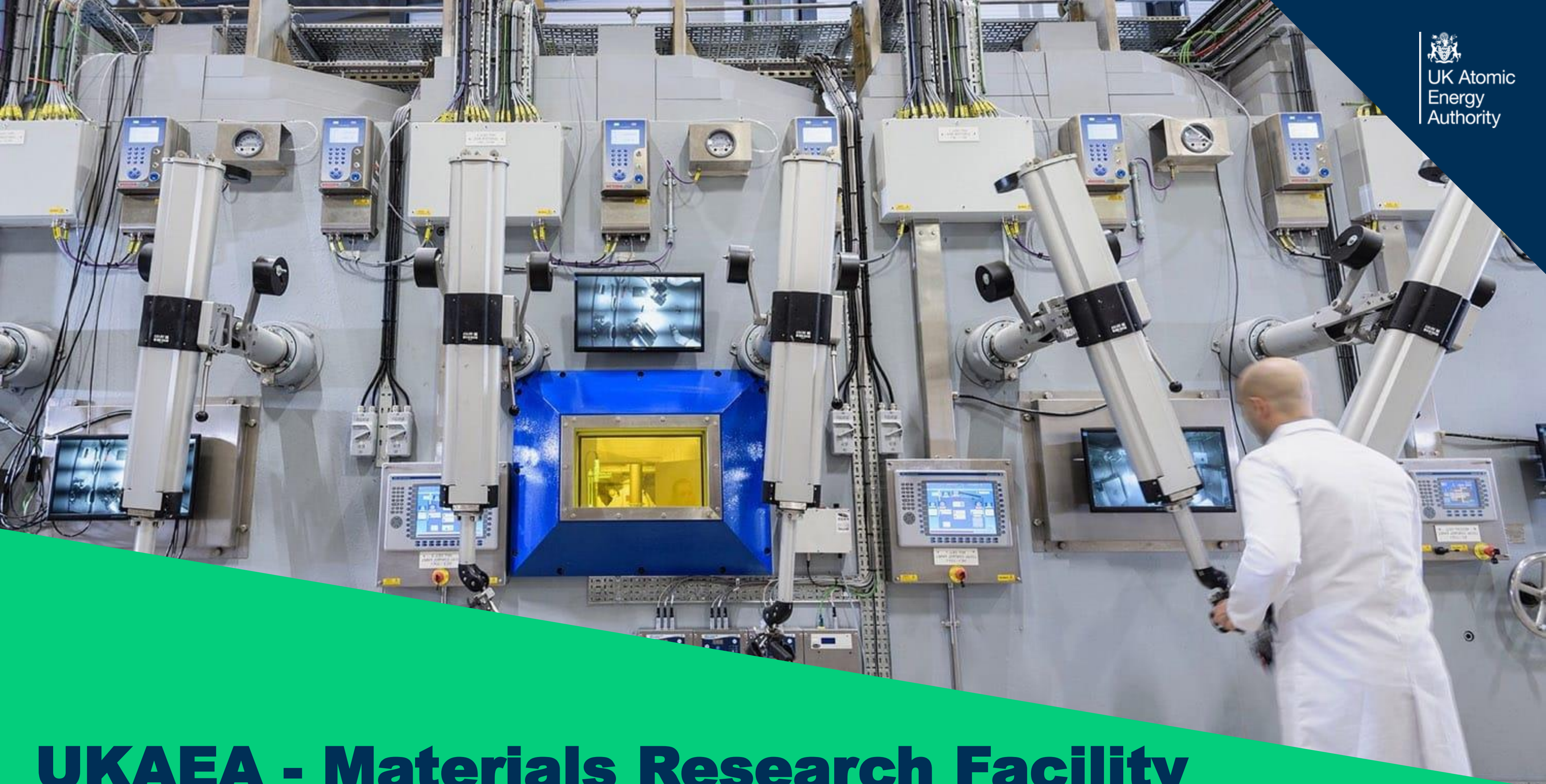

UK Atomic Energy Authority

UKAEA - Materials Research Facility PIE plans for the BeGrid2 samples

Slava Kuksenko

HENRY ROYCE INSTITUTE

NNUF

EPSRC Engineering and Physical Sciences Research Council

MRF is part of the National Nuclear Users Facility (NNUF) and the Sir Henry Royce Institute for Advanced Materials

MATERIALS RESEARCH FACILITY

UK atomic energy authority

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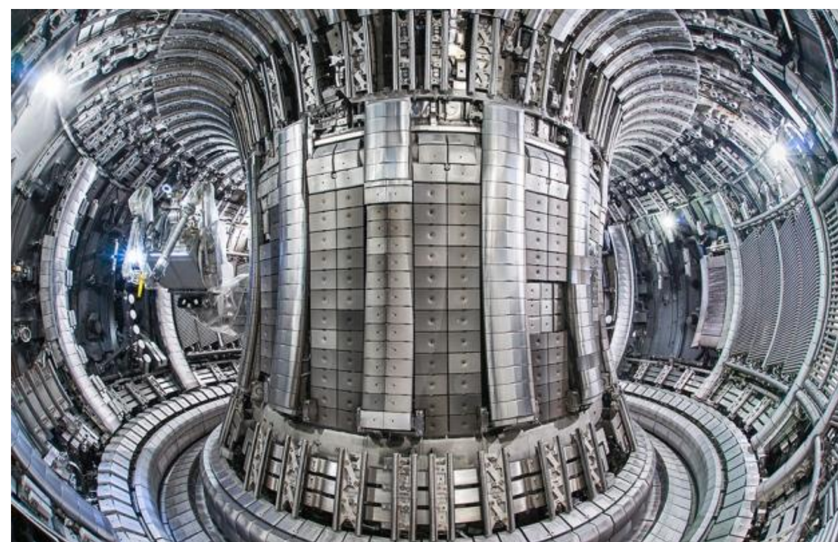


UKAEA - MRF

New facility for processing and analysis of neutron (and proton) irradiated materials

Open to Universities and Industry

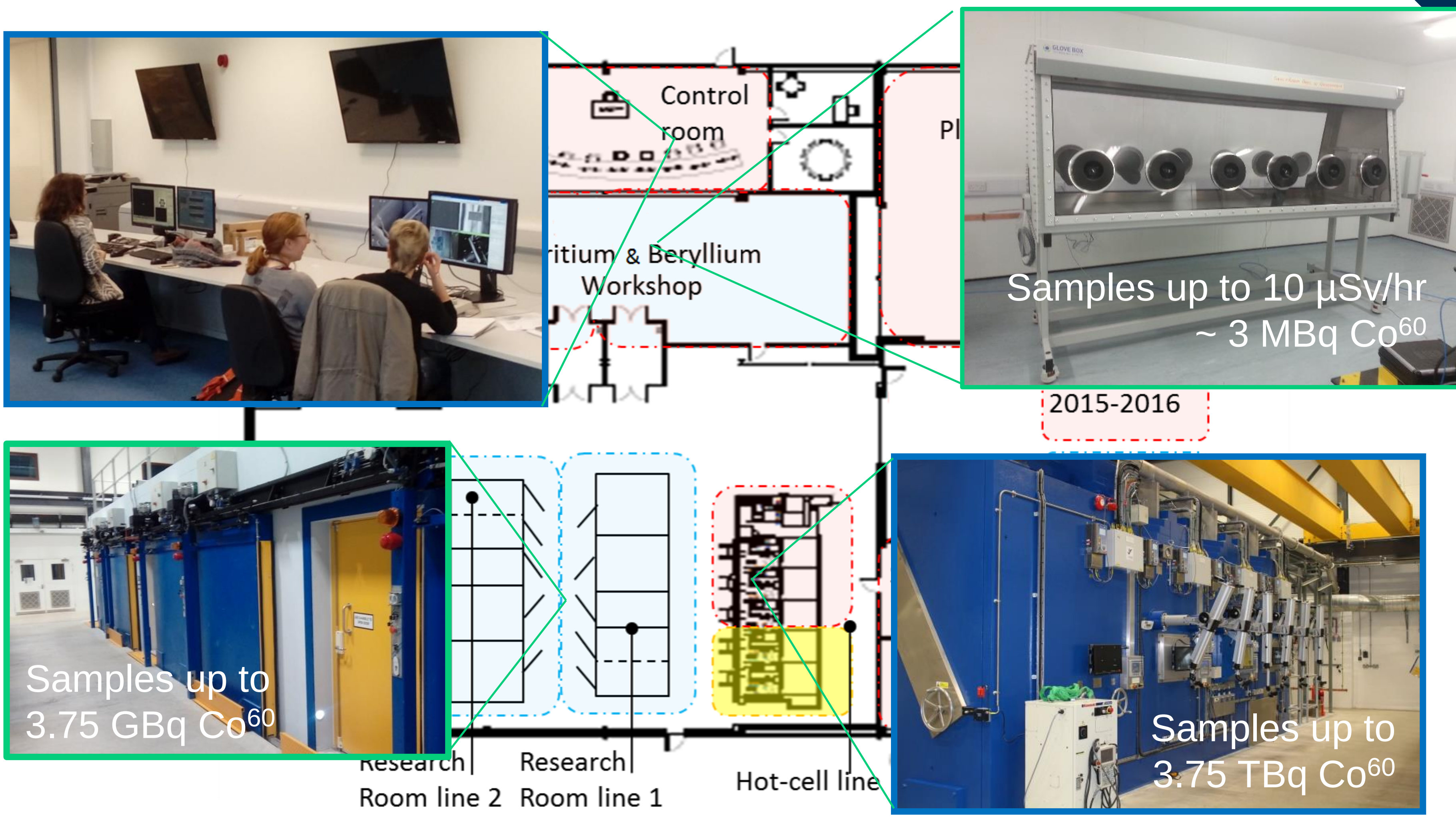
Opened May 2016, currently in commissioning

Nuclear experience (e.g. JET) on a non-licensed site: total inventory up to 3.75 TBq (Co^{60})

Universities — ~50 MBq (e.g. Oxford) — UKAEA - MRF: Medium activity, structural — 3.75 TBq (Co^{60}) — NNL: Most active, fuel cycle

Building layout

UK Atomic Energy Authority



MATERIALS RESEARCH FACILITY

MRF Hot-cell line

The hot-cell line

- Maximum activity up to 3.75 TBq (Co^{60})
- Sample reduction and fabrication
  - Slow cut saw
  - Shear cutter
  - Embedding
  - Grinding
  - Polishing
  - Cleaning
  - Gamma spectrometry
  - Optical microscopy
  - weighing

MATERIALS RESEARCH FACILITY

In-cell processes

Currently available

To be delivered

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✓ Cutting
- Slow cut saw
- *EDM wire (straight cuts only)*
- *EDM die sink*

✓ Grinding & polishing
- Conventional
- *Electropolishing*

✓ Mounting
- Hot-resin mounting
- Cold resin mounting
- Crystal bond mounting

✓ Sample evaluation
- Weighting
- Optical microscopy 20-220x
- Optical microscopy 200-470x

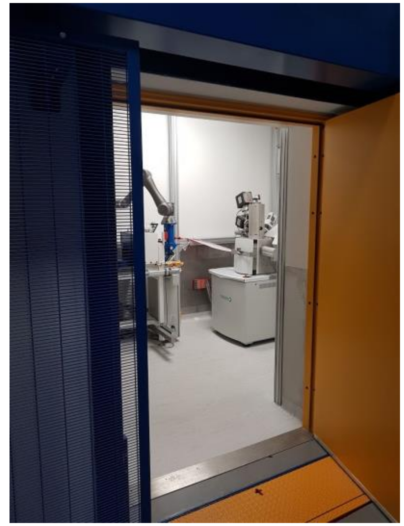
✓ Sample storage
- In air
- *Inert / vacuum*

✓ Gamma spectrometry
- Incoming materials
- Waste finger printing

✓ Waste processing
- Sorting ILW/LLW
- Drying out water based waste
- Solidifying liquid waste
- Separating resin from samples

MATERIALS RESEARCH FACILITY

MRF Research Rooms



- 10 fully independent shielded rooms
- Maximum activity up to 3.75 GBq (Co^{60})
- Scientific analysis and testing
- Manned access (no access if sample is exposed)
- Remote control from research rooms (ops) and from MRF control room (scientific evaluation)
- Containment connected to nuclear ventilation around experiment if required

MRF Research Rooms

Working with active samples in RR

- Equipment is configured, calibrated, prepared for specific test
- Active sample is brought into RR with shielded trolley
- Active sample is remotely transferred into shielded castle
- Shielded trolley can be removed from RR
- Active sample is remotely mounted into equipment
- Control of equipment is handed over to control room
- Tests are conducted
- Handover back to MRF technician
- Sample is transferred back to shielded castle
- Shielded trolley brought back into RR
- Active sample is remotely transferred into shielded trolley
- Active sample is removed from RR

Scientific capability

To be commissioned for use on active materials:

Glovebox
Research room (3.75GBq)
Hot-cell (3.75TBq)

- Overview scientific equipment

	2013/14	2015/16	2016/17	2017/18	2018/19
Microstructural analysis	FEGSEM, FIB	AFM, PMI XRF		CSLM Raman, WDS	XRD
Mechanical testing	Nanoindenter	Static load frame	Dynamic load frame	In-situ SEM frame, instr. indenter, DIC, EPD, IET	Small punch die, ultrasonic fatigue, high T testing for load frames
Thermo-physical characterisation	TDS	Gas Impregnation Setup	Dilatometer, LFA, TGA/DSC	Eddy current probe	Gas pycnometry
Advanced sample preparation		Electro-polishing, glovebox	Lab scale EDM, PIPS II, twinjet polisher, tube furnace	Sputter coater, dimple grinder, low activity prep	Diamond wire cutting, Lab scale EDM (hot-cell), electro-polishing, add. furnaces

Scientific Equipment

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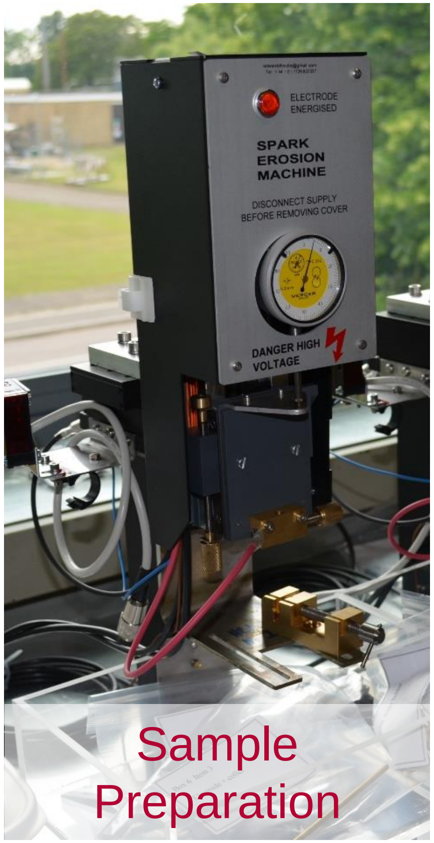
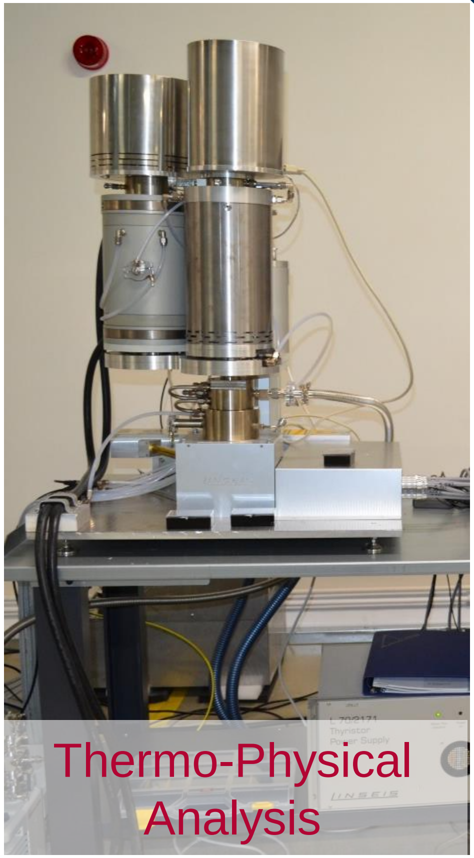
Microstructural Characterisation



Sample Preparation

Mechanical Testing



Thermo-Physical Analysis

MATERIALS RESEARCH FACILITY

Microstructural Characterisation

Currently av

Delivered in 2017/18

To be delivered 2018/19

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FEGSEM
- Tescan Mira XH
- EDX, EBSD, TKD, WDS

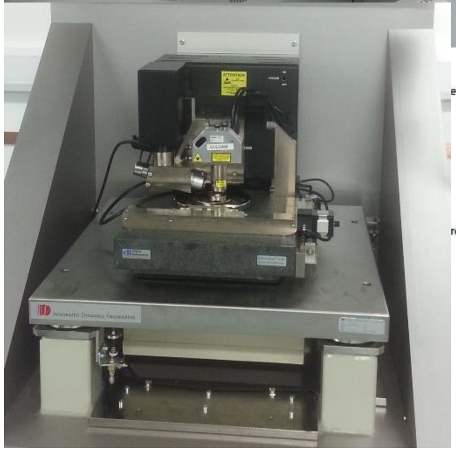
Dual beam FIB
- FEI Helios
- GIS: Pt, W, C
- Nano-manipulator for lift outs

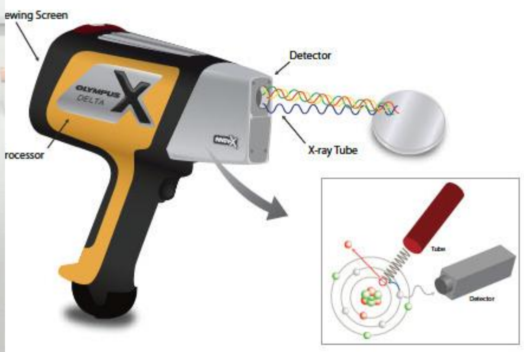
AFM
- Veeco D3100

PMI XRF
- Olympus delta premium

Laser Confocal Scanning Microscope – RAMAN spectrometry

XRD goniometer





MATERIALS RESEARCH FACILITY

Thermo-physical Analysis

Currently av

Delivered in 2017/18

To be delivered 2018/19

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Thermal Desorption Spectroscopy
- Hiden TPD workstation
- Up to 1000^{o}C

Gas Impregnation Technique
- Ar, N_2, Air, He, D_2, Tritium
- Ion energy < 500 eV, RT – 500^{o}C

Dilatometer
- Linseis L75V, dual pushrod
- -150^{o}C – 600^{o}C and RT – 2000^{o}C

Laser Flash Analyser
- Linseis LFA 1000
- -100^{o}C – 500^{o}C and RT – 2000^{o}C

TGA/DSC
- Linseis STA PT1600
- -150^{o}C – 500^{o}C and RT – 1600^{o}C

Gas pycnometry

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Mechanical Testing

Currently av

To be delivered 2018/19

Proposed for 2019/20

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Nanoindenter
- Agilent G200

Static load frame 10 kN
- Shimadzu AGS-x
- Environmental chamber for up to 800^{o}C

Dynamic load frame 15 kN
- TA Electroforce 3500
- Environmental chamber for up to 800^{o}C
- Vacuum chamber with induction heating

In-situ SEM load frame 5 kN
- DEBEN MTEST
- Clamp heating up to 600^{o}C

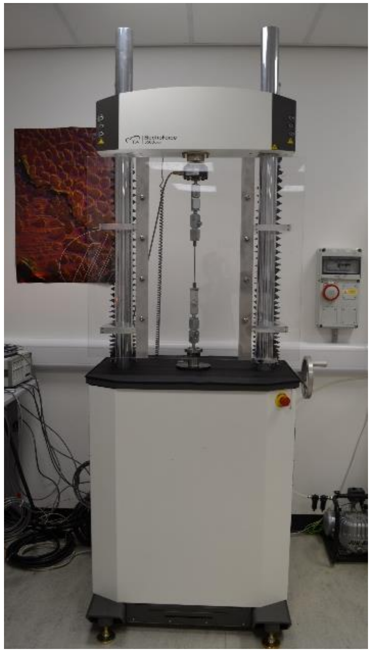
Instrumented indenter

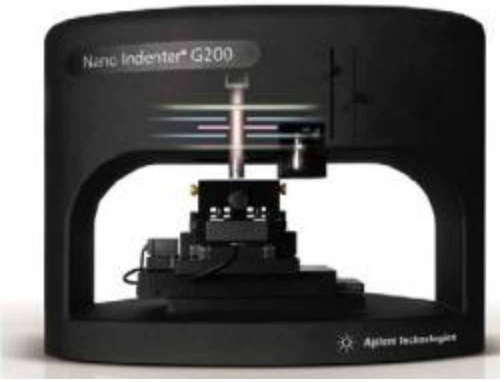
High Cycle Fatigue Setup

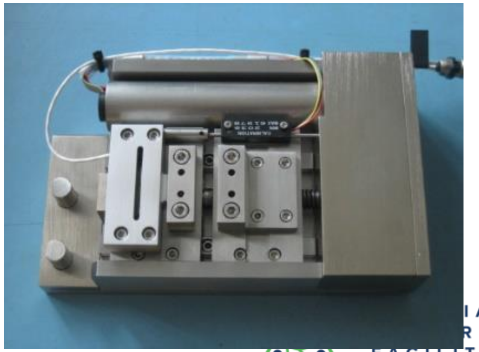
DIC strain measuring system

Small-punch testing

Impulse excitation testing





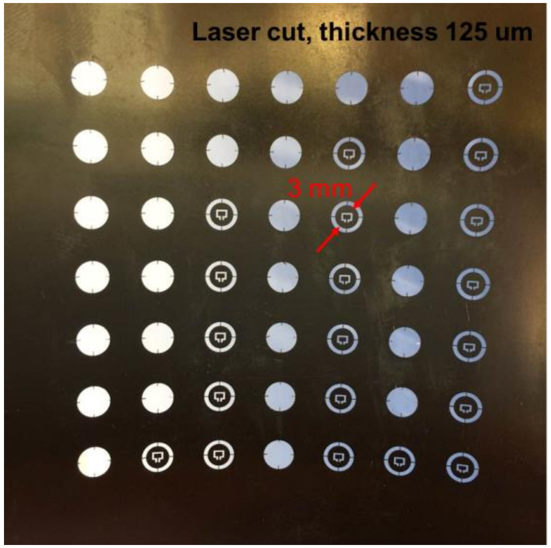


IALS
RCH
FACILITY

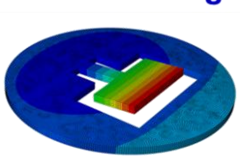
Ultrasonic Fatigue Rig

The meso-fatigue rig installation in the MRF (2018).

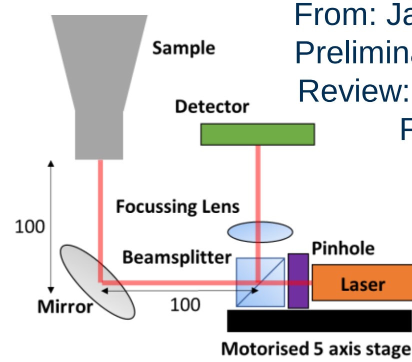
Ultrasonic resonant VHCF tests in the University of Oxford



FE Modelling



From: Jason Hess. Preliminary Design Review: Ultrasonic Fatigue Rig



- Existing setup at Oxford (Wilkinson, Gong)
- Laser cut 3mm discs, with 200 µm cantilevers
- To be integrated in active environment (Research Room)
- Current setup for RT is being replicated at MRF
- 20 kHz = 10^8 cycles in 1.5 h

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MATERIALS RESEARCH FACILITY

Sample Prep

Currently av

Delivered in 2017/18

To be delivered

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Non active Sample prep lab
- Cutting, grinding, polishing
- Electro-polishing
- Optical microscopy
- Non active sputter coater

PIPS-II ion polisher

Lab scale EDM

3mm disc punch

3 zone tube furnace 1200^{o}C

Dimple grinder

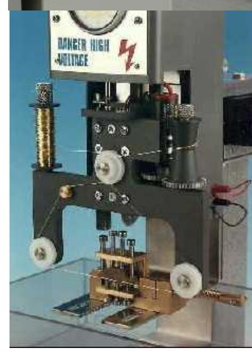
Glovebox sputter coater

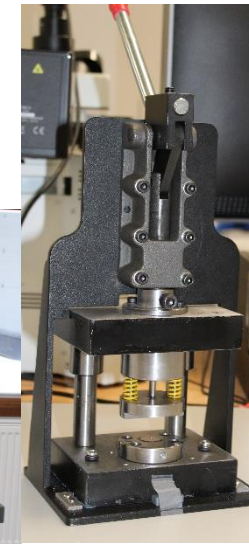
Glovebox cutting, grinding, polishing

Hot-cell labscale EDM

Hot-cell electro-polishing

Additional furnaces (vacuum)





MATERIALS RESEARCH FACILITY

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Investment Roadmap - 2016/17 to 2020/21

	2016/17 (£2M)	2017/18 (£2M)	2018/19 (£5M)	2019/20 (£5M)	2020/21 (£3M)
HOT CELLS (£3M)			NNUF	NNUF	NNUF
MICROSTRUCTURAL CHARACTERISATION (£3M)		Royce	Royce	NNUF	
SECOND FIB (£2M)			NNUF		
SHIELDING & OTHER ADAPTATIONS (£2.5M)	UKAEA/Royce		Royce *3rd shielded rooms*		
MECHANICAL TESTING (£2M)	Royce	Royce	Royce	NNUF	
THERMO-PHYSICAL TESTING (£0.5M)	Royce	Royce		NNUF	
SAMPLE PREPARATION (£1M)	Royce	Royce	Royce	NNUF	
ADAPT & EXTEND BUILDING (£5M)				BEIS	
ONGOING SUPPORTING ACTIVITIES	*Recruitment, business development, safety, sample archive, health physics, waste management, etc.* →				

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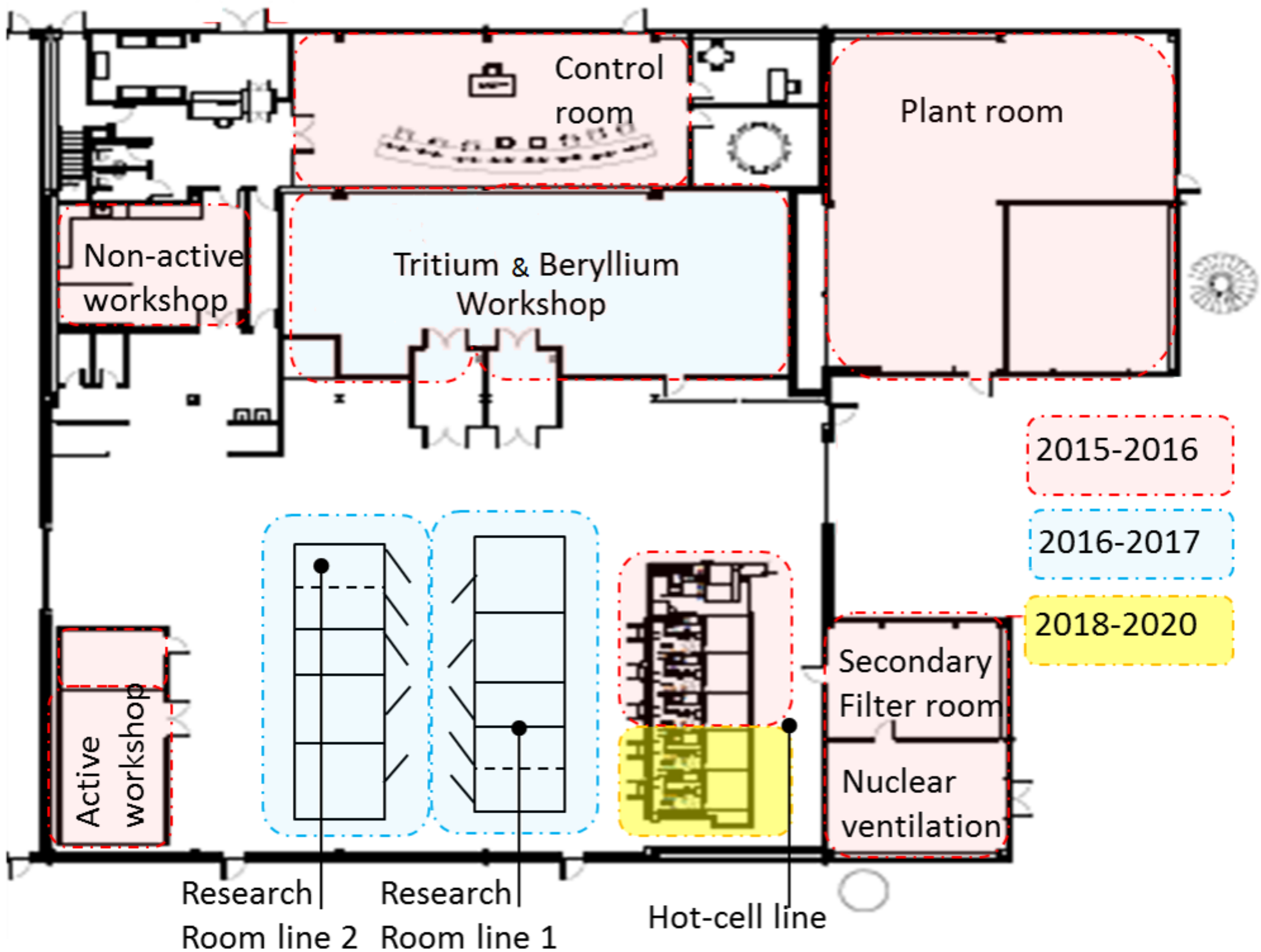
Investment: building extensions



MATERIALS RESEARCH FACILITY

Conclusion

Overall status on MRF facility

- Hot-cell line close of going active
- Research Rooms nearing completion
- Sample handling systems for RR are currently being developed
- Sample prep in glovebox planned for this year

Overall status on MRF scientific equipment

- The past 1.5 year MRF expanded from 4 instruments to > 15 instruments, additional techniques will follow in coming years
- Large majority of equipment is currently available for non-active and low active work
- Implementation for higher activity work will be a staged process

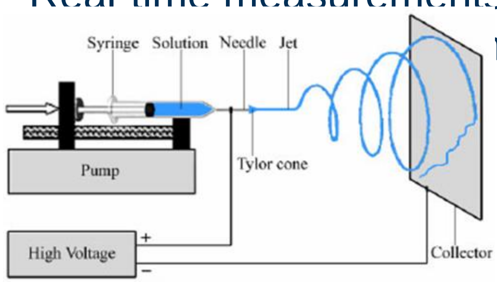
HRMT43

In-beam thermal shock test completed in Oct. 2018

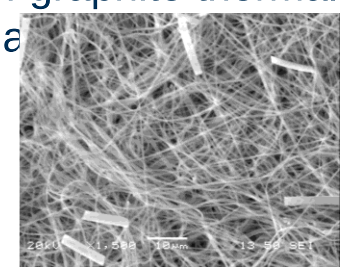
- Experiment carried out at CERN's HiRadMat facility
- Comparison of thermal shock response between non-irradiated and previously irradiated materials from BNL BLIP (Be, C, Ti, Si, SiC-coated C)
  - **First/unique test with activated materials at HiRadMat**

- Explore novel materials such as metal foams (C, SiC) and electrospun fiber mats (Al_2O_3, ZrO_2)

- Real-time measurements of graphite thermal



Electrospinning concept



SEM as-spun Al_2O_3

BeGRID2 - Upcoming PIE work

UK Atomic Energy

ARRAY	High-dose Irradiated Specimens (box 1.1)											
1	PF-60	S-65F	S-200F	S-200FH	low dose S-200FH	low dose PF-60	POCO	low dose IG-430	-	-		

Low-dose Non-irradiated Specimens (box 1.2)																			Slug
PF-60 (1)	PF-60 (2)	S-65F (1)	S-65F (2)	S-200F (1)	S-200F (2)	S-200FH (1)	S-200FH (2)	GC20 (1)	GC20 (2)	ZXF-5Q (1)	ZXF-5Q (2)	IG-430 (1)	IG-430 (2)	RVC (1)	RVC (2)	SiGraFlex	SiGraFlex		ZXF-5Q

ARRAY	High-dose Irradiated Specimens (box 2.1)											
2	PF-60	S-65F	S-200F	S-200FH	low dose S-200FH	low dose PF-60	low dose POCO	low dose IG-430				

Low-dose Non-irradiated Specimens (box 2.2)																			Slug
PF-60 (1)	PF-60 (2)	S-65F (1)	S-65F (2)	S-200F (1)	S-200F (2)	S-200FH (1)	S-200FH (2)	GC20 (1)	GC20 (2)	ZXF-5Q (1)	ZXF-5Q (2)	IG-430 (1)	IG-430 (2)	RVC (1)	RVC (2)	SiGraFlex	SiGraFlex		ZXF-5Q

ARRAY	High-dose Irradiated Specimens (box 3.1)											
3	Ti alloy	Ti alloy	SiC-C (no.4)	Silicon	Silicon							

Low-dose Solid, foam and novel Materials (box 3.2)																			Slug
SiC solid (1)	SiC solid (2)	SiC foam (1)	SiC foam (2)	Al2O3 solid (1)	Al2O3 solid (2)	ZrO2 solid (1)	ZrO2 solid (2)	SiO2 spun (1)	ZrO2 spun (1)	ZrO2 spun (2)	Ti alloy	Ti alloy	SiC-C (no.3)	Silicon	Silicon				ZXF-5Q

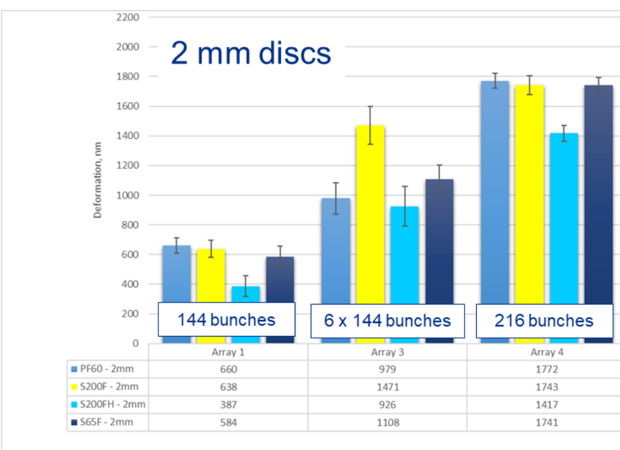
ARRAY	Low-dose Novel Materials (box 4.1)											
4	SiO2/ZrO2 spun (1)	ZrO2 spun (1)	ZrO2 spun (2)									

Low-dose Solid & foam Materials (box 4.2)																			Slug
SiC solid (1)	SiC solid (2)	SiC foam (1)	SiC foam (2)	Al2O3 solid (1)	Al2O3 solid (2)	ZrO2 solid (1)	ZrO2 solid (2)	Be solid 1	Be solid 2										ZXF-5Q

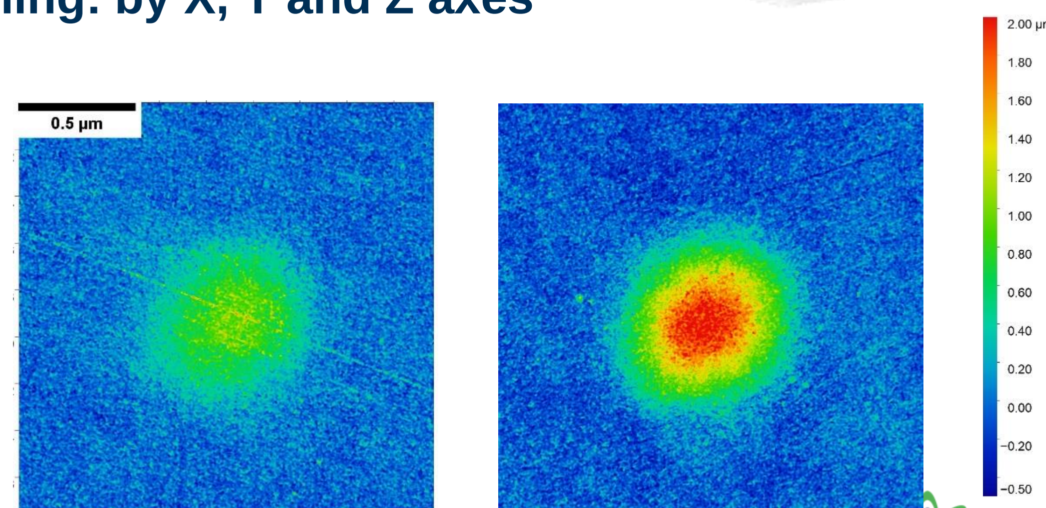
Upcoming PIE work

alpha300 R – Confocal Raman Imaging

- Topographic Raman Imaging ("TrueSurface Microscopy")
- Confocal microscopy in reflected light
- Fluorescent and Raman spectroscopy
- Large area mapping: up to 25 × 25 mm2
- 3D layer mapping
- Automated sample positioning: by X, Y and Z axes



	Array 1	Array 3	Array 4
PF60 - 2mm	660	979	1772
S200F - 2mm	638	1471	1743
S200FH - 2mm	387	926	1417
S65F - 2mm	584	1108	1741



BeGrid1, beryllium

Profilometry

- The out-of-plane deformation of the samples will be measured using Confocal Laser Raman Microscopy
- Topography of areas of the HiRadMat facility beam exposure will be collected.
- Profilometry will be performed on the samples as exposed at CERN, without removing them from the frames

SCANNING ELECTRON MICROSCOPY

- Exposed samples will be investigated to detect consequences of the HiRadMat facility beam exposure.
- Selected samples will be removed from the frames and put on the SEM pin-stubs. Carbon tape or silver DAG will be used for samples fixture.
- Microscopy images of the exposed areas will be collected. Localised deformation and surface fracture images (if exists and detectable) will be collected.

THANK YOU