Newsletter MRF

MATERIALS RESEARCH FACILITY

UK Atomic Energy Authority

January 2019

MRF supporting fission and fusion research at Culham

The Materials Research Facility (MRF) at Culham has been established to analyse material properties in support of both fission and fusion research and particle accelerators.

It is part of the National Nuclear User Facility (NNUF) initiative, launched by the Government and funded by Engineering & Physical Sciences Research Council (EPSRC), to set up a multi-site facility giving academia and industry access to internationally-leading experimental equipment; other equipment is available at the Dalton Cumbrian Facility and the National Nuclear Laboratory.

The MRF is also part of the Henry Royce Institute for Advanced Materials, which has been investing in the facility from 2016-2019. The MRF is an important component of its nuclear materials research theme.

The facility will very soon be able to offer hot cells and shielded instruments for processing and analysis of neutron-irradiated material. Meanwhile, the following are available for use on lightly active samples:

- Microstructural characterisation
- Mechanical and thermophysical testing
- Sample preparation for a range of instruments.

IN THIS NEWSLETTER:

New appointment

David Knowles will be appointed to the position of CEO of the Henry Royce Institute from February 2019.

David held a position as Director and Fellow at Atkins, within its Energy business, and is currently Professor of Nuclear Engineering at the University of Bristol.

In the latter role, he has collaborated with UKAEA on fusion materials.

The Henry Royce Institute has provided £5 million of investment funding for MRF.

Introduction

By Monica Jong

I am Monica Jong, the Head of MRF Operations. This is the first issue of the dedicated MRF newsletter, and I hope that you will gain from it a feel for what we can already do and the sort of research that has benefited from using the MRF.

My entire career has been dedicated to nuclear materials testing and it has been a great pleasure to use this experience to help establish a new, flexible, state-of-the-art facility for processing and testing radioactive material. Over £15M has already been invested and a similar level of investment is planned for the next few years.

Please get in touch if you think we can help you (contact details are on the back page).

Fuel retention in the JET ITER-like wall

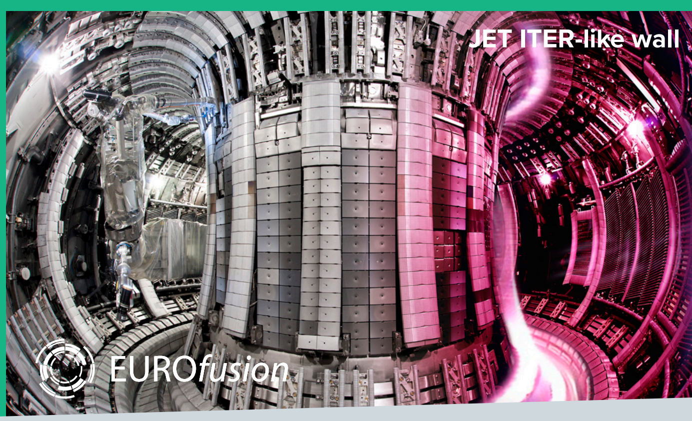
Due to tritium safety requirements in ITER, one area of ongoing research at JET is the investigation of fuel retention in Plasma Facing Components (PFCs). In 2011 JET started operations with the ITER-like wall configuration, with beryllium PFCs in the main chamber and tungsten PFCs in the divertor. To date, JET has run three operational campaigns lasting approximately 18 months and after each operating period a selection of PFCs have been recovered for analysis. From these studies the effect of plasma operations on the PFCs can be determined.

The handling and analysis of beryllium PFCs with tritium contamination is challenging. However, Carmen Makepeace, a fourth year PhD student of The University of Oxford (Department of Materials) and Culham Centre for Fusion Energy, Erosion Deposition Group, is making use of the unique facilities available at the new Materials Research Facility (MRF) to do just that. The aim of the project is to investigate the mechanisms of hydrogen isotope retention in beryllium PFCs using microstructural analysis techniques and temperature controlled fuel desorption studies.

MRF provides a facility where small samples may be prepared from larger samples which cannot be easily handled due to toxicity and radioactivity in many laboratories. In this case Focused Ion Beam (FIB) milling is used to prepare samples (10 µm x 30 µm x 1 µm) which are transported to Oxford Materials for analysis using Transmission Electron Microscopy (TEM), Electron Energy Loss Spectroscopy (EELS), Atom Probe, and Nano Secondary Ion Mass Spectroscopy (NanoSIMS). The results provide information of chemical bonding at the surface (OH bonds), supersaturation of the beryllium lattice with hydrogen located at interstitial sites, hydrogen atoms at point defects forming vacancy-hydrogen complexes and bubbles of hydrogen that have coalesced.

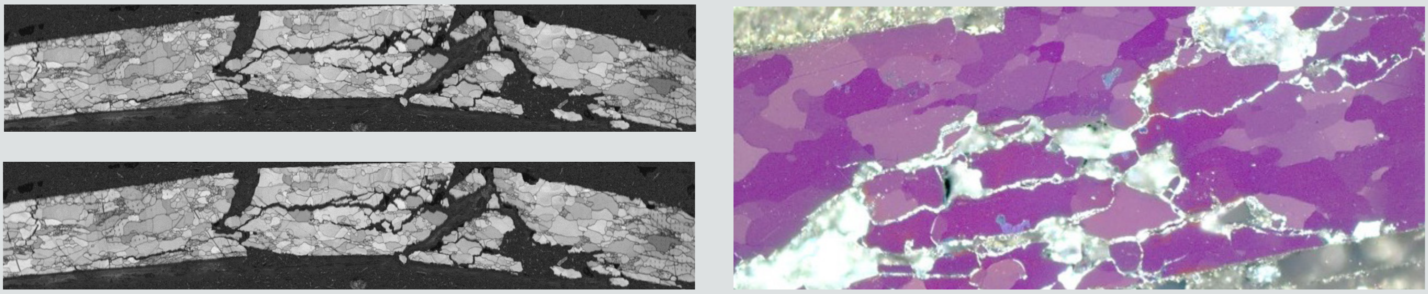
To accompany the microstructural studies, the desorption of hydrogen isotopes from larger beryllium PFC samples is investigated by Thermal Desorption Spectroscopy (TDS) based at the MRF. This method allows the quantification of desorbed hydrogen molecules using a quadrupole mass spectrometer with temperature (limited to 775°C for beryllium due to onset of evaporation, but up to 1000°C for other materials). With knowledge of the chemical bonding and spatial distribution of hydrogen in the samples from the microstructural studies and hydrogen desorption data a model of the trapping sites and release processes can be produced.

The project has been ongoing since 2015 and is planned to finish in 2019. The findings on fuel retention mechanisms will be useful for understanding tritium accounting and cleaning requirements of the ITER beryllium wall.



The effects of radiation damage in beryllium

A research project is currently being carried out at MRF on the effects of radiation damage in beryllium. The project involves the multi-scale characterisation of effects of extremely high energy proton irradiation on microstructure and mechanical properties of beryllium components, in a new generation of proton accelerator driven particle sources such as the Long Baseline Neutrino Facility (LBNF).



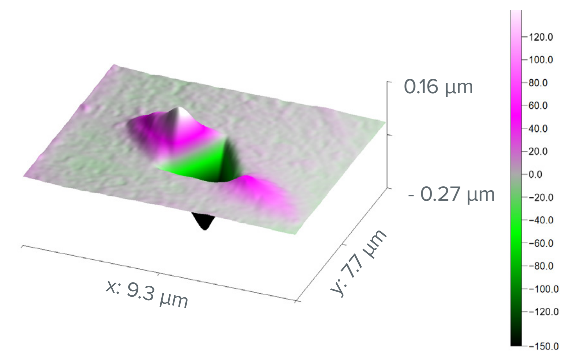
Cross-section of the fractured beryllium window after proton irradiation: secondary electron image (a) and EBSD (b), optical microscopy (c). Fracture accrues predominantly at grain boundaries.

The project sits within the RaDIATE collaboration (Radiation Damage In Accelerator Target Environments) and involves more than 15 participants from different countries. The main contributors to the "beryllium part" of the collaboration are from Fermi Lab (US), University of Oxford (UK), Rutherford Appleton Laboratory (UK) and UKAEA.

The future use of beryllium as target and beam window material in next generation particle accelerator facilities may be limited by higher dose and higher temperatures of operation. The existing database on beryllium response to radiation effects relevant to proton accelerator environments is very limited and this complicates material selection and lifetime predictions for beam windows and targets.

FIB, SEM, EBSD, EDX, TEM (done at Oxford University), Nanoindentation, Microcantilever fracture tests, proton irradiation and helium implantation experiments demonstrated extremely high radiation induced hardening and embrittlement at service temperatures relevant for current neutrino target facilities, and less evolution of properties for conditions relevant for the next generation targets. Potentially high impact lithium transmutant was highlighted for the next generation targets.

Due to the toxicity of beryllium, sample preparation and destructive tests with this material are highly time consuming, or not currently possible. In order to overcome this challenge, considerable work was carried out on safety measures for beryllium sample preparation. A materials recovery facility was used for the FIB sample preparation.



Topography map of nanoindentation print in beryllium.

Moving forward

For the next step, the intention is that we will prepare detailed samples for Atom Probe Tomography (APT) and Transmission Electron Microscopy examinations at Oxford University after high temperature irradiation. Experiments with higher dose irradiations and comparison of micromechanical tests data with the standard size experiments are also planned.

The results of the project will aid design considerations for future neutrino target facilities with beryllium elements. The data is also useful for the fusion reactor community where beryllium elements may undergo neutron irradiation.

The University of Oxford's contribution finished this year, but the project is still ongoing. Among the businesses and organisations that could benefit from the research are STFC (UK), FNAL (USA) and KEK (Japan). Several manuscripts are being published, with more to follow.

Strength-ABLE project

The largest-to-date project undertaken by the MRF is now complete and has opened the door to both new models of data analysis and new academic partnerships.

The Strength-ABLE project (Strength Achieved By Length-scale Engineering) was part of a three-year €1.8M project funded by the European Association of National Metrology Institutes (EURAMET) and part of the EU Horizon 2020 research programme.

It was required because of the need for scientists to get a useful measure of the mechanical properties of materials subject to extreme environments such as those expected in a fusion reactor. Such material is inherently radioactive and therefore there is a desire to work with small samples to reduce the associated hazards.

Strength-ABLE focused on examining the interplay between the size of the test and the size of the features making up the materials microstructure when measuring mechanical properties. Nanoscale analysis carried out at MRF facilitated the production of a large experimental database.

The idea was then to develop a set of design rules to compensate for these changes in size, thereby upscaling the data set or results to provide information for mechanical properties on a macro-scale. In addition to the bespoke experimental techniques, several analytical and simulation techniques were developed for this task.

Helios 600i dual beam Focused Ion Beam (FIB) with scanning electron microscope (SEM).

MRF staff produced new data analysis tools with one of these being recognised widely and included on open source software package Gwyddion.

One aspect of this project was to identify what volume of material is physically probed during spherical indentation testing, which was one of the small-scale tests performed to measure mechanical properties.

The Strength-ABLE project brought forward new insights helping engineers to develop new methods and models to interpret data from small scale testing. It has also resulted in three follow-up PhD projects and new collaborations with universities.

Testing such as that carried out during Strength-ABLE, is of relevance to fusion because the smaller the radioactive sample needing to be examined, the less the safety risk. It is hoped that a scaling algorithm discovered during experiments will then allow tiny samples to be extracted from a future fusion reactor, or fission based test reactor, rather than using large samples or a full intervention to remove whole components.

Detector role for UKAEA in key nuclear industry project

UKAEA's other National Nuclear User Facility activity is ADRIANA (Advanced Digital Radiometric Instrumentation for Applied Nuclear Activities). ADRIANA provides instruments for use by industrial and university researchers at Culham and at the Universities of Lancaster and Liverpool. UKAEA have state-of-the-art high resolution gamma spectrometry systems with digital signal processing and analysis software. This equipment can be used to support power stations, nuclear security applications and decommissioning operations, as well as research projects.

Academics visiting UKAEA will get the chance to research fusion environments in greater detail following a new collaboration with UK universities funded by EPSRC.

The TORONE project, which stands for Total Characterisation for Remote Observation in Nuclear Environments, aims to provide detailed information about the nuclear environments associated with nuclear energy.

Future developments are dependent on the ability to characterise the various highly radioactive environments that occur in the nuclear industry for both efficient decontamination and decommissioning, as well as in the design of new nuclear fission reactors and fusion reactors.

One material used was copper-chromium-zirconium (CuCrZr) which is a primary candidate for structural material for future fusion devices such as ITER.

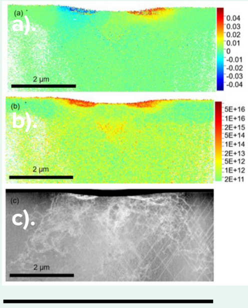
The Strength-ABLE project was led by the National Physical Laboratory, included EDF, AMEC, AWE and Tata Steel as well as European academic institutions. Strength-ABLE could benefit EU companies seeking to develop products with a longer life and lower energy use.

UKAEA’s Chris Hardie said: “In addition to the science, the real value of Strength-ABLE was the development and training of several future scientists and leaders in a growth area at UKAEA. Several of them have gone on to get jobs in international labs or continue to further study.”

Although this particular EU Horizon 2020 project has finished, MRF engineers have a range of additional deliverables to carry out. These are waiting on the full commissioning of the MRF.

Spherical indentations were made in pure copper with a cross-section cut out from the middle using the Focused Ion Beam (FIB).

Images a) and b) are the result of scanning the sample in a scanning electron microscope (SEM) and measuring the lattice rotations caused by the deformation during indentation. These can then be used to calculate elastic strain:



Scale: 7μm

a. geometrically necessary dislocation (GND)

b. density using a piece of software called CrossCourt

c. this was taken in a Transmission Electron Microscope (TEM).

In this particular image, contrast is due to defects in the atomic arrangement.

UKAEA’s MRF is developing capabilities to reduce the sample sizes required to produce valuable mechanical property data.

Alex Cackett, a PhD student from the Queen Mary University of London, is focusing her three-year research project on size effect by measuring stress-strain properties from small volumes of material.

Using equipment within the MRF she can gain a greater understanding of how mechanical behaviour varies with length-scale.

Alex is focusing on testing copper-chromium-zirconium (CuCrZr). This material is used as part of the JET fusion experiment in components that are exposed to the hot plasma fuel. It will also be used within the divertor exhaust system of the ITER experiment and is envisaged for the DEMO prototype fusion reactor.

The overall aim of this research is to be able to take data from small-scale tests, apply a scaling algorithm that takes size effect into account, and predict what the large-scale mechanical properties are.

“It could then be possible to monitor the condition of components in fusion and fission power stations at MRF by extracting just a tiny sample,” said Alex. “This would avoid shutting the plant down to remove whole components. Engineers could probe the structural integrity of reactor components more regularly, over a wider range of materials and locations throughout the reactor.”

Academics from Manchester and Lancaster universities, including robotics expert Prof. Barry Lennox, nuclear instrumentation expert Dr Michael Aspinall and associated researchers, toured JET, MAST Upgrade, and MRF as part of an information-gathering mission. Lee Packer, Nuclear Technology Group Leader at UKAEA, will coordinate academic access and testing of radiation detectors as part of Culham’s input to the project.

“One of the case studies academics wanted to pursue, in addition to trials planned for nuclear fission environments at Sellafield and Fukushima in Japan, was to test sensors in fusion environments,” said Lee. “Fusion brings various challenging environments including the combination of high radiation fields, temperature and magnetic fields.”

TORONE project partners at UKAEA’s MRF (L to R): Ioannis Tsitsimpelis and Michael Aspinall (Lancaster University), Andrew West and Barry Lennox (Manchester University), Martin O’Brien and Lee Packer (UKAEA).

Henry Royce Institute funds new equipment for MRF

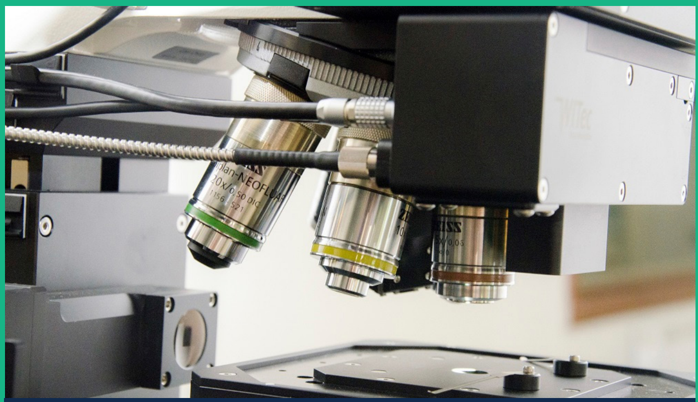
The Materials Research Facility (MRF) is set to bring even more detailed insights into nuclear reactor materials, thanks to new equipment funded by the Henry Royce Institute.

Scientists want to know more about the degradation of material properties due to neutron irradiation.

With a range of high-level technical equipment now in place at the MRF, scientists will begin to add to their knowledge of the behaviour of materials under a range of different circumstances.

One of the most interesting items delivered as part of the funding tranche was the Confocal Laser Scanning Microscope with Raman spectroscopy. The key difference to a conventional microscope is that it uses laser light in an optical configuration that has an extremely narrow depth of focus – allowing it to offer high-resolution optical imaging with the ability to do optical sectioning. This means it is possible to view visual sections of tiny structures which would normally be too difficult to see.

The added Raman spectroscopy element of the microscope offers the chance to discover further detail on the local chemical composition of the sample. This is gleaned by the fact the material absorbs the laser, allowing the material to consume energy and in return emit what is known as the "Raman shift."



Confocal Laser Scanning Microscope with Raman spectroscopy.

Steven Van Boxel, Materials Scientist at UKAEA, said: "The confocal laser scanning microscope gives up to 10 times better magnification than a standard model. As it is confocal, it is able to track the height of the sample and interpret it. This then allows us to construct 3D pictures from this. The added Raman spectroscopy enables us to find out further chemical information about the type of bonding in the sample."

Equipment currently available at MRF

Even more detailed insights into nuclear reactor materials will now be possible with the new equipment that is now in place. The equipment includes:

Microstructural characterisation equipment

- Scanning Electron Microscope
- Dual-beam Focused Ion Beam Scanning Electron Microscope
- Confocal Laser Scanning Microscope with Raman spectroscopy
- Atomic Force Microscope
- Positive Material Identification XRF

Mechanical testing equipment

- Nanoindenter
- Instrumented indenter
- 10kN Universal testing maching
- 15kN Dynamic load frame
- 5kN in-situ load frame in SEM
- Impulse Excitation testing
- Digital Image Correlation setup
- Electrical Potential Drop system

Metallographic sample preparation facilities

- Metallographic sample preparation suite in hot-cell
- Metallographic sample preparation suite in glovebox
- Metallographic sample preparation suite for non-active samples
- Labscale Electron Discharge Machining
- Precision Ion Polisher
- Sputter Coater
- Dimple Grinder
- Diamond wire saw
- Electrolytical polishing equipment

Thermo-physical analysis equipment

- Thermal Desorption Spectroscopy
- Laser Flash Analyser
- Simultaneous Thermogravimetrical Analyser
- Dilatometer

Robotic arms allow for detailed study of ‘hot’ materials

The robotic arms, also called ‘Cobots’, are used to move around radioactive samples.

A set of ten hi-tech robotic arms is ready to handle radioactive facilities in the MRF.

The TM5 Techman devices are ‘collaborative’ robot arms, meaning they can easily be programmed by teaching them the desired movements which need to be carried out.

They will be instrumental as scientists at the MRF carry out various projects later this year. The first robotic arm has already been used to demonstrate the capabilities of the technology to visitors.

The radioactive material arrives at Culham with each piece sitting inside a container flask. These pieces are then transported into the receiving cell on the Hot Cell processing line — the first part of the sample preparation — and then placed into a transport trolley. The robotic arm moves the sample through an additional shielded vessel ready to be examined.

A variety of scientific instruments will be in the research rooms, dependent upon which task needs implementing. Due to the need for containment, a thick concrete exterior (550mm) provides another layer of safety.

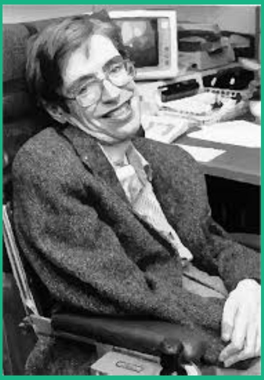
Paul Iwanczak, MRF Mechanical Engineer, said: “The first TM5 is ready to be operational for the FIB (Focused Ion Beam) which is installed in Research Room 1. We will soon be able to use the robotic arm to move around radioactive samples and load them in the FIB.

“Essentially, these arms are needed to pick up and place radioactive samples from a mobile trolley to a stage inside a scientific instrument. Work like this must be unmanned because of the radioactive nature of various pieces.

“Each robot arm sits on a plinth mounted inside the containment area inside the room.

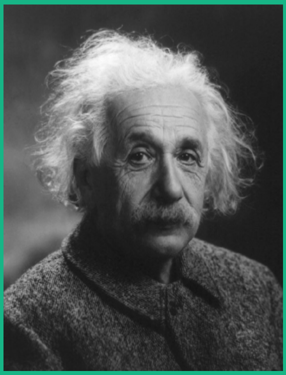
“The sole purpose of these pieces of kit is to pick up a hot piece and place it inside the instrument, and then to reverse that operation to get it out.” The first robotic arm arrived at MRF in 2018.

Science matters



Successful names in the world of science have been given to robots in the MRF unit.

Stephen Hawking, Albert Einstein, Sir Isaac Newton and Mary Somerville are among those immortalised in a bid to move away from the more traditional numerical convention of naming.



The UK Atomic Energy Authority's mission is to lead the commercial development of fusion power and related technology, and position the UK as a leader in sustainable nuclear energy

MATERIALS RESEARCH FACILITY

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