

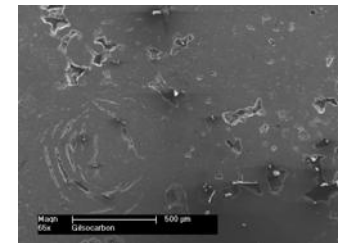
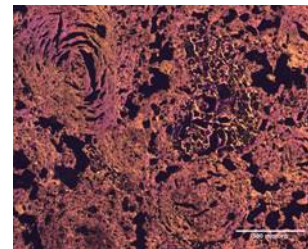
Microstructural Characterisation of Nuclear Grade Graphite using High Resolution Techniques

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Introduction

- Graphite has been used as a moderator and reflector material for neutrons in more than 100 nuclear power plants worldwide and in many research and plutonium-production reactors.
- It is used primarily as a neutron reflector or neutron moderator, although graphite is also used for other features of reactor cores, such as fuel sleeves, spacer rings, shield-wall graphite or other carbon-bearing material in certain cases.



Irradiation damage to nuclear graphite

The irradiation induced changes to the graphite crystal structure within nuclear graphite have been shown to lead to disruption of the bonding across the basal planes and closure of Mrozowski cracks giving similar graphitic structures to that seen before graphitisation.

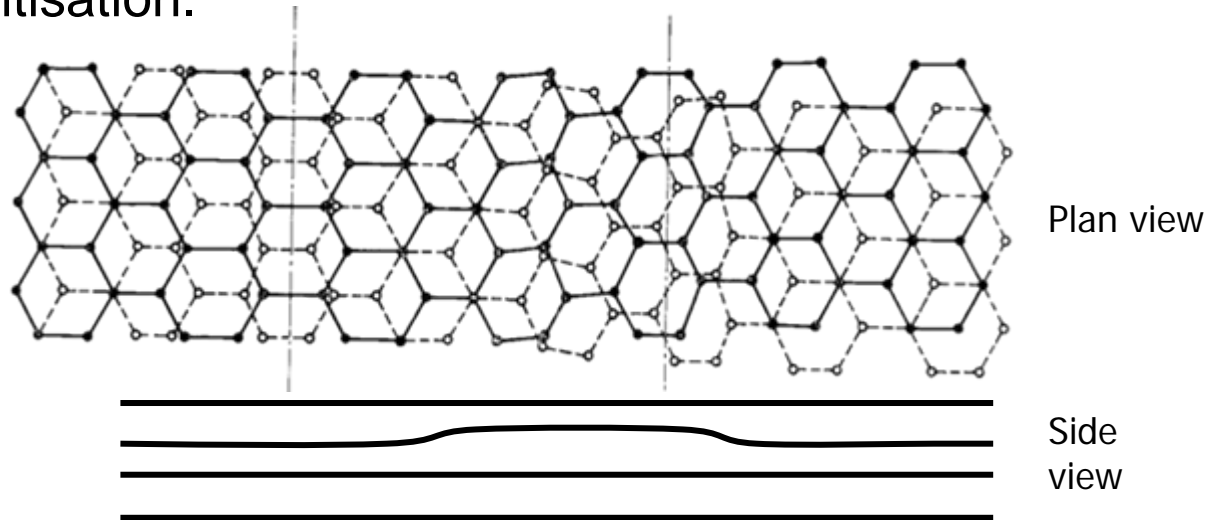


Figure 1: Basal dislocations observed in graphite after irradiation

[1] Ouseph P.J. *Phys. Stat. Sol. (a)* 169, 25 (1998)

Objectives

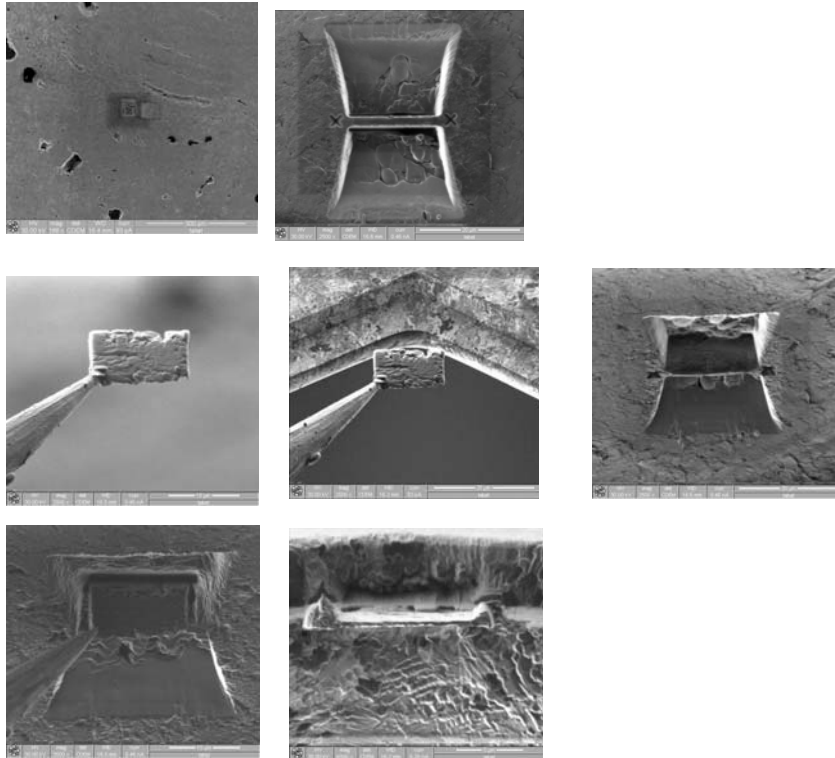
- The objective is to obtain microstructural thermal strain measurements by image correlation, with which to validate irradiation models for structure and property relationships.
- Sample preparation methods have been compared using both traditional mechanical polishing and state of the art Focused Ion Beam FEG-SEM.
- May lead to the development of improved graphite products for the nuclear industry.

Analytical Methods for Microstructural Characterisation

- FIB SEM – for TEM sample preparation
 - » *In situ* technique using electron and ion beam
 - » Produces electron transparent region of interest (<100 nm) with the entire specimen <3mm
 - » Rugged and Stable
 - » Free from specimen preparation debris
 - » Conducting
 - » Can produce HRTEM images with sub-angstrom resolution
- TEM Analysis
 - » Thermal experiments – CTE correlations using Image Correlation
 - » Irradiation studies: TEM, SEM, FIB -SEM
 - » Irradiated Tomography with TEM

Technique of FIB for TEM Preparation

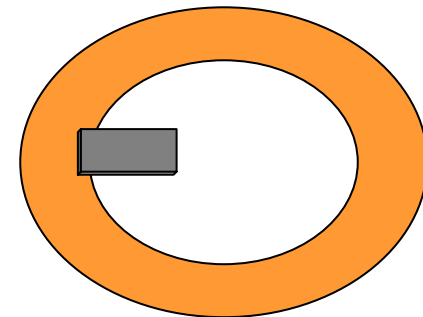
The objective of using a FIB sample is to produce a image which is faithful to the physical structure of the specimen being looked at.



Ion Beam

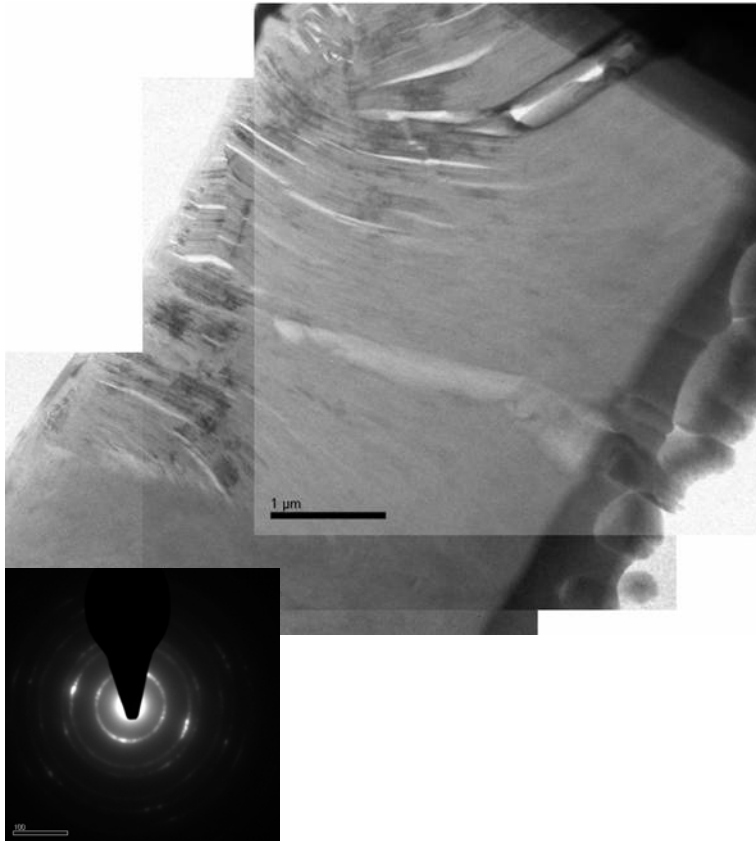
Staircase shaped cuts are milled on either side of the region of interest

Region of interest can be thinned further and cut free and from the bulk material.



TEM Schematic with FIB sample

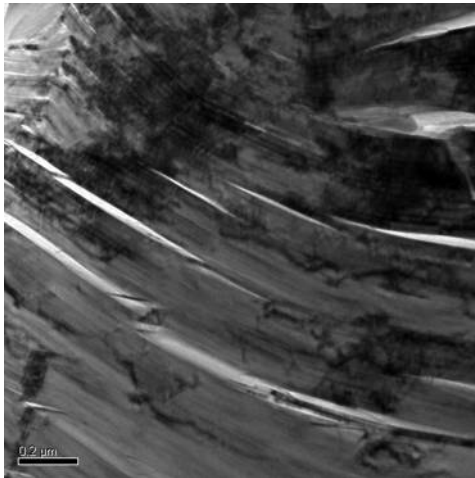
Microstructural characterisation: TEM



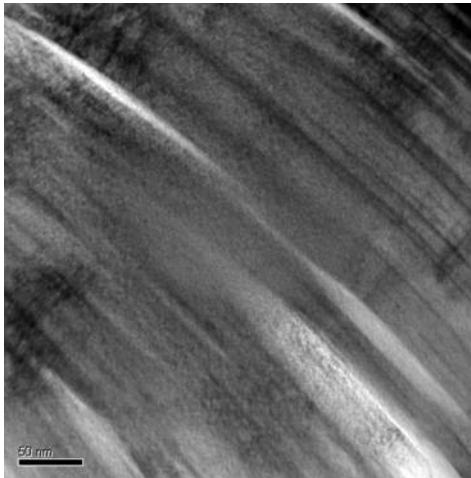
- Composite TEM Images of Gilsocarbon FIB sample.
- Scale bar is 1 µm
- HRTEM shows of crystalline graphite indicated from the SAD image.
- The cracks between the graphitic planes may be identified as Mrozowski cracks that form by anisotropic contraction of the materials on cooling from graphitization temperatures.

Microstructural characterisation: HRTEM

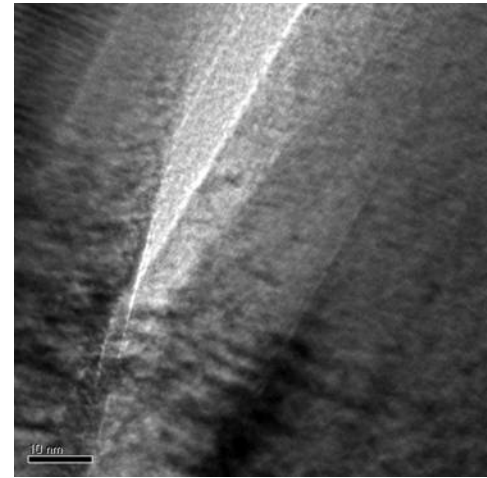
Location of Mrozowski cracks



• Scale bar is 0.2 μm



• Scale bar is 50 nm



• Scale bar is 10 nm

Mrozowski cracks range from 10 nm in width and over 1 μm in length

Conclusions

- New FIB technique has been demonstrated to successfully prepare electron transparent TEM foils which suitable for thermal expansion measurements.
- Foils are free from specimen preparation debris.
- The crystalline structures of unirradiated nuclear graphites have been evaluated using HRTEM.
- Gilsocarbon sample exhibits regions of high crystallinity and Mrozowski cracking within the graphite.

Future work

- Planned Thermal HRTEM experiments to observe the closure of Mrozowski cracks.
- Image correlation analysis of data and relationship to thermal expansion co-efficient.
- Use thermal –TEM experiment with irradiated (neutron / ion) graphite material to transfer the technique.
- Develop a TEM sample preparation technique for irradiated graphites using either FIB or the Tripod polisher.
- Analysis of further non irradiated graphite materials using the FIB as a TEM sample preparation method.

Acknowledgements

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