New Nuclear Manufacturing Programme

Impact Report
2016
Manufacturing of nuclear components is undergoing a surge of technical development to meet the ongoing need for improvements in process cost and time, whilst maintaining the need for long-term performance and regulatory approval of the finished product. The UK nuclear supply chain is set to benefit from this as the nuclear ordering program in the UK gets underway.

The New Nuclear Manufacturing (NNUMAN) programme began work in October 2012 to develop existing and new manufacturing technologies in welding, machining, near net shape and advanced nuclear fuels. In this document we provide examples of where “real world” improvements are emerging from this work and also show how our research has enhanced the scientific basis and understanding of these technologies.

In all areas of our work we continue to benefit greatly from the support of UK and overseas colleagues in industry, in the Nuclear Advanced Manufacturing Research Centre (Nuclear AMRC) and other industry research bodies, and in academia. Their involvement in NNUMAN has helped to develop routes to exploitation and the growing technical insights that underpin our work.

The NNUMAN community is now well established and looks forward to working on the implementation of new manufacturing technologies into the reactors of the future (including the Small Modular Reactors now being considered for UK deployment) and further developing the material characterisation and modelling that provide the underpinning for the innovative and confident nuclear manufacturing of the future.
NNUUMAN and the UK nuclear landscape

Achievements and directions

**Welding**

* Narrow groove welding
  NNUUMAN’s work is providing a unique comparison of different narrow groove welding processes in 30mm and 130mm thicknesses of pedigree low alloy pressure vessel steel, including measurements of distortion, residual stress and material and microstructural properties across the weld region. This will inform welding choices in the fabrication of major nuclear components of the future.

**Machining**

* Advanced machining and surface properties of reactor structures
  The machining team in NNUUMAN has produced a coherent linkage between machining parameters and surface conditions for a range of nuclear materials, providing a means of optimising machining productivity for particular applications.

**HIP**

* Hot isostatic pressing
  In NNUUMAN, we have been able to link 304L and 316L powder oxygen content to the spatial density of non-metallic inclusions in the metal product and then link this to fracture properties. This has provided powder manufacturers with key evidence in setting lower powder oxygen levels.

**Fuel**

* Advanced nuclear fuel
  Research in NNUUMAN has identified a ceramic braze composition for joining Silicon Carbide composite fuel cladding that has been successfully tested under reactor coolant conditions. Such cladding could provide significant additional margin for fuel integrity under accident conditions.

Contact us
NNUMAN and the UK nuclear landscape

Since 2010 the UK Government has placed significant investment in new facilities to develop the country’s nuclear manufacturing. With a series of Gen III light water reactors on the horizon and with Hinkley Point C now given the go-ahead, the Government’s aim is to revitalise this significant and specialist UK manufacturing sector so that UK firms are well positioned to supply components and fuels for these new nuclear plants and can also support the UK’s role in international efforts to develop later generations of nuclear power plants. This investment has been centred on the Nuclear AMRC in Rotherham, operated by the University of Sheffield, and the complementary Manufacturing Technology Research Laboratory at The University of Manchester.

The NNUMAN research programme was launched in October 2012. Through a portfolio of activities and projects it delivers long-term research to develop innovative, high-productivity manufacturing techniques that will meet the future needs of the UK nuclear industry.

The programme is managed by The University of Manchester’s Dalton Nuclear Institute with support from the Nuclear AMRC. It was awarded £4 million in funding by the Engineering and Physical Sciences Research Council (EPSRC – EP/J021172), with the two universities committing an additional £4 million. Financial and in-kind support also comes from several industry partners: Rolls-Royce, National Nuclear Laboratory (NNL) and Amec Foster Wheeler.

Building foundations for new manufacturing

From the outset NNUMAN has worked closely with Nuclear AMRC to develop new manufacturing techniques; our research collaborations with NNL have focused on advanced fuels development. Our range of industrial partners continues to expand globally, for example through close working links with companies such as Electric Power Research Institute (EPRI) in the USA and AREVA in France.

Our research typically operates at TRL 1-4; these early stage studies complement mid-range TRL development at the Nuclear AMRC; NNL and other research bodies focus on industrial exploitation of new materials, components and manufacturing methodologies at higher TRL levels. NNUMAN’s role is essential because it feeds the pipeline for technological and manufacturing process development, both for reactor structures and fuel. We provide robust theoretical and empirical understanding of these manufacturing processes and how they affect long-term component and nuclear plant performance – knowledge that is vital in a high cost, long life and safety-critical industry.

Gen III/Gen III+ reactors for near-term deployment in the UK and elsewhere are already designed in detail, but our work remains relevant as manufacturing companies are always searching to lower their production costs while raising product quality and performance. The programme is also well positioned to contribute to manufacturing advances for Small Modular Reactors (SMRs). Initially derived from Gen III/III+ designs and using common materials, SMRs are attracting world-wide interest with the UK seen as a potential major market. Our research will help UK companies become major suppliers for SMRs as well.

Shared knowledge

From the outset NNUMAN has worked closely with a number of partners from academia and industry including the University of Birmingham on powder metallurgy, Nuclear AMRC on new machining techniques and the National Nuclear Laboratory (NNL) on advanced fuels development. Our range of industrial partners continues to expand both within the UK and globally, for example through close working links with companies such as EPRI in the USA, ANSTO in Australia and AREVA in France.

Looking further ahead, NNUMAN will likely contribute to the successor programmes for Gen IV reactors and to the potential fusion reactors (DEMO) of the future. These longer term developments involve large-scale international cooperation, to which we already contribute through strong links with many organisations, especially in Europe, Japan and, more recently, China. There are also opportunities for the scientific knowledge we have gained to inform manufacturing processes for radioactive waste management, not least the techniques and materials used to produce the many thousand containers required for long-term waste storage.

Our key expertise

The key to NNUMAN’s success has been the ability of our researchers and industry and academic partners to provide realistic test pieces, manufactured with well characterised process parameters and within strictly controlled conditions. Our scientists measure the mechanical properties of these materials and products and link their performance to underlying microstructure; together these measurements help to describe how manufacturing processes are likely to affect the long-term performance of new materials.

Our researchers also build models to estimate and predict component properties and performance, for example residual stresses and fracture in welds.

Our work reveals how the choice and control of a manufacturing process can dramatically alter the performance of nuclear components over their design life and thereby provides key knowledge for the UK supply chain to compete in the global nuclear industry.
**Achievements and directions:**

**Welding**

**Narrow groove welding**

Narrow groove weldments have established benefits in reduced heat input and process time, but which welding process should be used? What difference is there between gas tungsten arc (GTAW), submerged-arc (SAW), electron beam (EB) or laser beam welding systems? The through-life integrity of major nuclear reactor components, for example those in the primary circuit of a light water reactor (LWR), are critical to the safe, economic and long-term operation of new nuclear plants. We are working to provide comprehensive benchmark cases for each welding process, producing data that will allow direct comparison of weld integrity and performance in nuclear-grade low alloy ferritic steel.

Complementary modelling and simulation studies are already highlighting the physical processes, during the welding or once the component is in situ that affect the evolution of microstructure, mechanical properties and residual stress.

**Key issues addressed in welding**

1. **How does the performance of narrow groove weldments compare between different existing and new welding processes?**

Using pedigree SA508 Grade 3 Class 1 low alloy steel (30mm and 130mm thicknesses), NNUMAN researchers have produced butt weldments using current GTAW, SAW, EB and laser beam welding processes. The two arc welding systems are already employed in a narrow groove weld configuration; EB is a candidate welding process for future build programmes while laser beam welding is still in development for sections thicker than 30mm. All EB welding was carried out at TWI Cambridge. Our research team has measured temperature, strain, restraint loading and distortion alongside a range of metallurgical and material properties of the weldment. After observing significant transverse shrinkage during early trials, the team added 80mm specimens in SA533B steel for SAW and GTAW to set groove profile dimensions and check welding procedures. Results from these samples have been added to the data set.

**Routes to exploitation**

- A unique performance data set of thick section weldments produced using beam and arc-based welding processes for use by industry and incorporation into regulatory codes and standards
- A large collection of industry standard, well-characterised legacy test pieces and data for future researchers
- A comprehensive testing methodology for weld trials under controlled restraint
- Guidance on groove geometry and the control of distortion for narrow gap weldments
- Contribution of expertise and involvement in the development of codes and standards for specific welding issues

**The way forward**

- Strong industrial links with major nuclear manufacturing companies (e.g. Rolls-Royce, AREVA and BAES) and across the oil and gas industry via the BP-ICAM programme of welding trials on similar materials
- Dissemination of findings to the global research community through organisations such as EPRI (which has also provided additional weld test pieces to extend the weld process comparison study)
- On-going development of welding processes through Nuclear AMRC projects within their generic research and capability development portfolios on EB, GTAW and SAW welding including post weld heat treatment (PWHT)
2. How can we improve models of how residual stresses develop in thick-section, narrow groove weldments for both as-welded and post-weld heat-treated conditions?

Our researchers have measured residual stress in electron beam weldments using hole drilling, the contour method and neutron diffraction. These readings compared extremely well with the values for electron beam welds calculated using one of our models. In collaboration with the Australian Nuclear Science and Technology Organisation (ANSTO), researchers found that phase transformation has a significant effect. Using advanced SEM techniques, they were able to estimate phase distribution ratios in parts of the 30mm EB welds to confirm modelling assumptions, now further developed using dilatometry. This modelling work is currently focusing on welds that involve the deposition of filler material, looking at weld metal properties, dilution and the effects of re-austenisation and tempering in multi-pass welds and comparing calculations with measured values. There are significant differences in residual stress distribution between the weld types (Figure 1).

Routes to exploitation

- Industrial use of heat source modelling approach (Rolls-Royce patent application)
- Validated models of residual stress distributions in EB welds as functions of heat treatment and thickness
- Guidance on when phase transformation effects are significant in the calculation of residual stress distributions
- Improved accuracy and validity range for the applications of TG4 for austenitics as part of the European NET programme
- Improved measurement techniques for residual stresses in nickel-based alloys (TG6) in collaboration with EDF as part of the European NET program
- Contribution to the EDF R6 procedure for narrow gap weldments
- Adjustment of assumed residual stress levels in post-weld treated condition in R6 procedure and BS7910 standard

The way forward

- Potential guidance on the use of residual stress estimation in structural integrity assessments within the ATLAS+ H2020 programme
- Modelling of HIP and weld materials within the Design by Science project funded by EPSRC (starting January 2017)
- EPSRC Manufacturing Fellowship awarded to Professor Mike Smith

Achievements and directions: Welding

Figure 1: Cross-process comparison of measured (neutron diffraction and contour method) residual stress profiles for 30mm thick welds. Processes compared: (1) NG-SAW, (2) NG-GTAW, (3) RPEB, in both as-welded and post-weld heat treated states.
3. Can laser welding work well for thicker section (≥20mm) ferritic steels?

Laser welding has directional flexibility with limited, highly directed heat input, but these advantages are offset by current difficulties in maintaining tight dimensional control, fit-up and cover gas management to achieve consistent weld quality.

We have fabricated multi-pass cold wire welding specimens in 30mm thick SA508 Grade 3 Class 1 steel that meet the requirements of Section IX of the ASME Boiler and Pressure Vessel Code (BPVC). This promising development supports the application of laser beam welding for thicker ferritic steels, although subsequent progress to achieve quality welds at higher thicknesses has been slow. Further work includes successful autogenous, single pass welding of P91 steel at 11mm to support the DEMO fusion program and some early trials on dissimilar metal welds at 40mm. We are also investigating the potential of autogenous, low vacuum laser welding in collaboration with RWTH Aachen University.

Routes to exploitation

- A range of new and improved techniques, equipment and know-how for future laser welding applications, including wire feed, cover gas control, laser spot shaping, distortion control and weld pool monitoring by infrared camera

The way forward

- Development of low vacuum laser welding that, unlike EB, does not require demagnetisation and can operate in rough vacuums with significant productivity gains; collaboration with laser welding developers, such as Cranfield University, TWI and RWTH Aachen
- Continued development of the autogenous laser welding of P91 working with the UKAEA DEMO team

CASE STUDY

Improving the physical basis of modelling residual stress in weldments

Residual stresses have significant effects on the formation and growth of weld defects; these internal forces can drive fracture, fatigue and creep and are independent of any external loading. Modelling to estimate these stresses is essential for both designers and plant operators to optimise operations whilst assuring safety. However, accuracy is essential; over-estimates of possible defects can restrict plant operation, while under-estimates have safety implications.

In the past, calculational models achieved reasonable estimates for austenitic steels; the best practice output has now been codified in assessment procedures such as EDF R6. However, low alloy ferritic steels are much more complex for modelling because any estimation of residual stresses and welding-induced distortion requires preliminary prediction of solid state phase transformation (SSPT), and hence microstructure development. In addition, ferritic steels are typically heat treated after welding. This process helps to recover their fracture toughness and reduce residual stresses, but the practice is largely empirically based and detailed knowledge of the distribution of residual stress after heat treatment is limited.

Our researchers have improved the modelling of this behaviour by focusing on the complex metallurgical and mechanical processes, including the effects of SSPT which occur in the near-weld region as the weld is formed. The model and validation are based on the pedigree SA508 Grade 3 Class 1 steel, a candidate material for the main vessels of a modern nuclear reactor primary circuit. Our research compared estimates of residual stresses generated by models with physical measurements from our range of test weldments.

Accurate modelling of SSPT requires detailed microstructural information about the melt zone and the heat-affected zone (HAZ). Our researchers have obtained a wealth of data using SEM at high magnification and dilatometry experiments. SEM imaging is used to measure austenite grain size and growth and to estimate the final proportions of key micro-constituents such as martensite, bainite, ferrite and austenite (Figure 2). Dilatometry on material exposed to thermal cycles equivalent to those in the welding process has allowed our researchers to measure directly the continuous cooling transformation behaviour of the steel. This empirical data has been compared with numerical models; the correlation between measured hardness and microstructure has been studied in detail. We are now incorporating the results of the SEM and dilatometry studies into improved descriptions of SSPT, concentrating on the descriptions of austenite formation and growth kinetics during the heating phase of a welding cycle.
Achievements and directions: **Welding**

Our modelling research has also investigated anisothermal metallurgical phase transformations in steel welds. These changes are also difficult to model because heating and cooling is rapid and numerous physical processes need to be considered in the models. Multi-pass welds with filler add further complexity: multiple materials, dilution, re-austenisation and tempering. We have tackled these challenges by examining EB welds. These undergo a single weld pass with no filler wire – a simplified scenario which has allowed our researchers to capture the fundamental metallurgical processes without the complications introduced by a second material and multiple thermo-mechanical cycles.

Working with ANSTO of Australia, we use a software module originally developed to model phase transformation in low alloy steels and first validated using the NeT Task Group 5 international benchmark. We compared the predicted stresses with diverse measurements made using neutron diffraction, the contour method and hole drilling. We found encouraging agreement between model estimates and measured values for both thicknesses of EB welds (30mm and 130mm), with a clearly defined “m” shaped stress distribution; residual stresses in the weld and HAZ are greatly reduced (or indeed compressive) as a result of low temperature SSPT during welding (see Figure 3). We have improved this software module and incorporated it into the ABAQUS-based residual stress modelling procedures deployed at The University of Manchester.

Our modelling work for multi-pass welds started with specially manufactured SAW and GTAW welds containing one, two and three passes. We have now successfully introduced a second material into the SSPT model, and developed techniques for modelling dilution and mixing between weld filler and fused parent material. The dilution model has been validated by direct measurements of chemical composition using EMPA, both in the NNUMAN low alloy steel welds and in the NeT Task Group 6 international benchmark (a three-pass nickel alloy weld).

Modelling of large, multi-pass welds imposes severe challenges for computational tractability. Here our primary objective is firstly to understand, then accurately model, the key SSPT variables in relatively simple geometries with limited numbers of passes. Once this greatly simplified model has been validated, we will identify appropriate analysis simplifications that will allow us to develop appropriate and industry-relevant models of large multi-pass welds.
Achievements and directions:

Machining

Advanced machining and surface properties of reactor structures

Unlike welding, which is highly codified in nuclear manufacturing, machining processes have traditionally been developed by manufacturers based on the recommendations of tool and equipment suppliers. The research evidence for common practices is limited or non-existent. This large knowledge gap gives scope for significant developments in this area to improve productivity without compromising on the surface conditions of finished components.

Key issues addressed in machining

1. How do machining parameters link directly to surface condition?

Traditionally manufacturers select machining parameters to preserve tool life and deliver products with acceptable surface conditions. However, this conservative approach is rarely compatible with industry’s demand for high productivity. When NNUMAN began in 2012, manufacturers had no data that coherently linked machining strategy, surface integrity, tool life and productivity for nuclear grade materials (304L and 316L stainless steel, SA 508 Grade 3 pressure vessel steel and Alloy 690). Using a rigorous Design of Experiments (DoE) approach, we have produced a strong body of evidence which indicates that far more ‘abusive’ machining strategies can be deployed with minimal impact on the surface condition of the machined component (as measured by near-surface residual stress, hardness and microstructure). The improvement in productivity far outweighs the slight reduction in tool life.

Routes to exploitation

- Significant body of robust evidence (based on a framework of rigorous multi-parameter testing) which indicates that less conservative machining parameters can improve productivity without sacrificing surface conditions in the main structural materials
- New machining parameter selection and optimisation is embedded in the Nuclear AMRC and applied in several research projects to significantly reduce machining time

The way forward

- Further development through follow-on projects (e.g. the European-funded MCSCAMP project and another currently under consideration) to investigate the impact of advanced cooling techniques and the use of larger test pieces for more representative geometry and machining methods

2. How can mobile machining centres improve their performance?

The large components found within a nuclear plant necessitate large machine tools; specialist equipment and heavy lifting gear are deployed to move and set up the machining operation. Robotic machine tools (Figure 4), represent an attractive alternative that could save manufacturers and plant operators significant time and effort. Unfortunately, robotic tools are generally too flexible and far less accurate than traditional machine tools, so they are normally unsuitable, especially for large metallic components.

Our researchers have conducted a full dynamic assessment of the Fanuc F200i hexapod. Using the robotic machining cell at the Nuclear AMRC, researchers have evaluated the robot throughout its working envelope. Mapping the robot’s dynamic behaviour, they have successfully optimised machining strategies to minimise vibration and maximise productivity.
This project also investigated the use of minimum quantity lubrication (MQL) to increase the productivity of machining operations (traditional ‘flood’ lubrication is not always practicable for robotic procedures). The work has proved the concept of using robots as machine tools; machined samples have good surface integrity and the robot’s metal removal rates are comparable to those of standard machine tools. If deployed, this robot could dramatically lower the capital investment required by companies in order to produce large machined components as well as reducing complicated lifting and moving operations.

Routes to exploitation

• Successful demonstration and characterisation of Fanuc F200i’s machining capabilities, including comparable metal removal rates and industry standard component surface conditions
• Guidelines on optimal MQL lubrication

The way forward

• A Nuclear AMRC generic project is now developing an in-process metrology system to improve the geometric accuracy of the robot
• European funding for a follow on robotic machining project at the Nuclear AMRC, based on the underpinning knowledge developed by our researchers

3. How can the reliability of deep hole drilling be improved?

Deep hole drilling often creates excessive vibration in the workpiece; an operator must carefully set machine dampers based on the geometry of the workpiece and the specific machining parameters. This set-up typically requires a high level of skill and knowledge and is hard to replicate. The machining is, therefore, often inconsistent leading to large numbers of components failing quality control. Chips formed by the drilling process also tend to be long and continuous; they are difficult to remove and may clog the tool, which also causes product failure.

Our researchers have developed a bespoke dynamic test rig to enable consistent machining set-up (Figure 5). Data from the rig was used to optimise positioning for the dampers to guarantee a stable machining process and consistent product conformance. A novel ultrasonic system in the tool holder ‘excites’ the cutting tool which shatters the chips so that drilling remains smooth and controlled. Further work on the general application of assisted machining using cryogenic techniques is also planned.

Routes to exploitation

• New, analytically-based vibration management coupled with an ultrasonically assisted tooling component
• Improvements in process reliability, reproducibility and productivity for deep hole components in ductile steels

The way forward

• Nuclear AMRC can use this knowledge in the manufacture of SMR control rod drive mechanisms
• The proposed multi-partner MEACTOS EU NUGENIA project, involving Nuclear AMRC and The University of Manchester will further investigate the effect of assisted machining on stress corrosion and cracking (SCC) susceptibility
• A bid for Innovate UK funding has been submitted to support commercial development of ultrasonic-assisted machining

Figure 5: Dynamic vibration control of Nuclear AMRC's TBT LM700 deep hole drilling machine
Understanding the implications of machining on susceptibility to stress corrosion cracking

Stress Corrosion Cracking (SCC) is one of the most insidious forms of material degradation; it occurs when components are exposed to specific environmental conditions. Typically characterised by long incubation times, cracks in components in service can go undetected for many years, if not decades, before leading to fast crack propagation and the risk of catastrophic failure.

We have carried out a package of work designed to give alloys superior material performance, including resistance to SCC, through controlled machining processes. The work focused on austenitic alloys (304 and 316 stainless steels, Alloy 600 and 690) and investigated fundamental relationships between manufacturing processes, material structures, properties and long-term, in-plant product performance.

Given the long incubation time related to SCC, it is a challenge for researchers to test materials in ‘real time’. Instead, studies rely on methods that will forecast the performance of materials over very long periods. We study specimens which have experienced accelerated testing conditions, i.e. more severe irradiation or mechanical stresses.

Crevice beam bent (CBB) specimens were tested in an oxygenated environment to simulate off-spec water chemistry according to a methodology developed by Hitachi for testing in boiling water reactor (BWR), plant-relevant conditions. Other tests included the application of a slow dynamic deformation (slow strain rate test) and the use of tapered samples to investigate the effect of stress and strain, and predict SCC in real components that might not necessarily ever experience dynamic deformation (Figure 6).

Our researchers also used a specialist imaging autoclave to detect and monitor the evolution of embryonic microcracks in samples within a high temperature water environment.

We have complemented our SCC testing with correlative analytical techniques which help to characterise how advanced machining processes create microstructure and residual stresses in materials and their effect on structures. Preliminary results clearly indicate a different oxidation behaviour, indicating that second phases can have a detrimental role.

Specifically, the selective oxidation of martensite, which can be induced by heavy machining or material processing, may lead to subsurface oxide penetration. Conversely, we have also shown that delta ferrite, a chromium-rich second phase, can form a thinner oxide than the matrix; strain incompatibilities and cracking can develop at the delta ferrite/matrix interface because delta ferrite has a different crystal structure. Slow strain rate tensile testing of solution-annealed and machined stainless steel has also revealed that cracks develop only within the superficial region of abused materials; work is underway to investigate the transition from initiation into mature cracks on work-hardened materials (see Figures 7 & 8).
Figure 8: Cracks in the 20% cold rolled and machined samples after crevice beam bent test

Figure 7: Crack in the annealed and machined sample after slow strain rate tensile test

Figure 8: Cracks in the 20% cold rolled and machined samples after crevice beam bent test
Hot isostatic pressing (HIP) is a process in which a powder or metal component is subjected to high pressure, usually in a vacuum, and high temperature simultaneously. This is to allow the material to consolidate and become denser. HIP is particularly useful for making components that need to be very strong and of uniform density, such as those used in nuclear power plants. It is an alternative to conventional methods of manufacturing, such as casting or forging, and can be cheaper and more efficient. Components made by HIP are more uniform and have better properties than those made by other methods.
2. What is the relationship between oxygen content and fracture behaviour in HIP Type 300 steels?

Our research shows that temperature and oxygen content clearly affect Charpy fracture toughness in 316L steel made from HIP powders of different oxygen levels. Materials formed at lower temperatures and higher oxygen contents are much less tough than forged material (Figure 9). Detailed microstructural analysis of specimens produced by industrial partners and by NNUMAN researchers have revealed oxygen-based inclusions of a spatial density which increased with the level of oxygen: HIP material has a much higher oxygen content than forged. Similar tests on Alloy 690 have helped us to eliminate martensitic transformation as a possible cause at lower temperatures. We have complemented extensive Charpy and J-R testing at a range of temperatures with finite element modelling of Charpy and J-R fracture processes.

Routes to exploitation

- NNUMAN researchers have already demonstrated to the HIP industry in Europe and the USA that oxygen in HIP powders affects the fracture toughness of HIP Type 304L and 316L austenitic steels and Alloy 690
- Deeper understanding of the mechanism of oxygen uptake into HIP material via inclusion formation
- Evidence that inclusions influence ductile tearing in fractures with a clear difference between HIP and equivalent forged material (Figures 10 & 11) across a wide temperature range

The way forward

- BEIS/Innovate UK programme on advanced nuclear materials and manufacturing
- The provision of high quality fracture toughness data (J-R, Charpy) obtained on these steels for inclusion in materials properties databases and nuclear codes and standards
- Further application of this approach to the fracture performance of HIP low alloy ferritic steels

![Figure 9: Charpy impact data for forged steel compared to HIP’d steel containing varying concentrations of oxygen and showing the effect of oxygen on impact toughness](image-url)
Figure 10: Typical microstructure of HIP’d stainless steel, showing the presence of voids which are the result of dislodged oxide inclusions, and which play a significant role in the ductile fracture mechanism.

Figure 11: J-R data for forged 304L and HIP 304L stainless steel with 120ppm oxygen at differing temperatures.
Grain boundary engineering studies in hot isostatic pressing (HIP) Type 316L steels

The development of HIP as a manufacturing process in the nuclear industry has, primarily, focused on the production of components with acceptable bulk mechanical properties and chemistry, near-theoretical density (typically above 99% of the density of forged materials) and consistent low grain size. However, little attention has been given to grain boundary control, even though the topology of the grain boundary network has a significant influence on many aspects of material performance. By controlling the grain boundary, our researchers hope to influence SCC, fatigue and ductile fracture by limiting crack formation and growth. A better understanding of HIP processes should lead to improvements in the in-service reactor performance of HIP components (Figure 12).

To deepen fundamental understanding of these phenomena, we conducted a suite of studies on the mechanisms that influence microstructure development during HIP production of 316L austenitic stainless steel. The research identified how microstructure can be engineered through process control to produce a desired network of grain boundaries.

Our researchers analysed the grain boundary character distributions (GBCD) of a forged 316L specimen and a fully HIP-manufactured and solution-annealed 316L specimen. They selected metrics based on recommendations from related technical studies: number and length fractions of special boundaries, triple junction distributions and twin-related domains. Their analysis revealed that forging and HIP both produced materials with a high proportion of annealing twins, whereas purchased 316L powder contained random high angle boundaries.

The team also ran experiments to interrupt the HIP process at different points in time during production. This series showed that, in 316L, the consolidation of the powder to a fully dense material promotes dynamic recrystallisation by twinning. When twin boundaries (first and higher order) are part of the grain boundary network, they disrupt the connectivity of random boundaries and enhance the material’s resistance to percolate phenomena such as intergranular SCC and fracture. However, in the time series observations, the twins formed within the grains and were, therefore, not part of the grain boundary network. Nevertheless, subsequent studies demonstrated how the formation of twins could be engineered by thermomechanical processing to tailor material properties.

Figure 12: A general overview of the HIP process illustrating how the grain boundary network of the 316L powder changes after HiPing. One of the key questions we are interested in answering is: “Can we, by applying small changes to the standard HIP cycle used by the industry, alter the grain boundary network so that the resulting microstructure has reduced susceptibility to intergranular degradation phenomena?”
The researchers discovered that partially consolidated specimens formed at 950°C had enough stored energy to undergo static recrystallisation if they were subjected to annealing for a short time (10 mins at 1100°C). Figure 13 illustrates how this simple heat treatment changes the grain boundary network: after HIP, the specimen contained a high proportion of sub-grain boundaries; heat treatment led to the formation of annealing twins as a result of static recrystallisation.

The annealed specimen is still in a partially consolidated state, but now it contains twin boundaries, albeit within the grains. Our researchers are currently investigating what happens when materials containing twin boundaries go through a second round of HIP and recrystallisation. Do the twin boundaries undergo multiple twinning during this dynamic recrystallisation and consequently form part of the grain boundary network? The specimens produced using the altered HIP cycle will undergo SCC and fracture tests to compare their performance against specimens produced using a standard HIP cycle. Microstructure stability (i.e. grain growth) will be monitored during ageing tests. The data from these tests will indicate whether rounds of annealing and HIP can produce materials with specific fracture resistance. Manufacturers may also be able to adapt the technique for other austenitic alloys such as Alloy 600 and 690, which also undergo profuse twinning.

Figure 13: Grain boundary misorientation maps of the 316L specimen following HIP at 950°C. The map of the as-HIP’d specimen is shown in (a) and the map of the same specimen (but different region) after annealing at 1100°C for 10 minutes is shown in (b). A dramatic change in the grain boundary structure can be seen after annealing where in the microstructure in (a) has a lot of subgrain boundaries and the microstructure in (b) has a lot of annealing twins.
**Achievements and directions:**

**Fuel**

**Advanced nuclear fuel and cladding**

While improvements in fuel cladding tend to occur in the light of recommendations from post-accident investigations, the design of advanced fuels tends to build on scientific discovery and new knowledge at a more fundamental level. We have addressed a number of issues in both of these areas as part of a broader portfolio of research within the UK’s Nuclear Fuel Centre of Excellence.

**Key issues addressed in nuclear fuel**

1. **How can manufacturers overcome the problem of joining silicon carbide?**

   Silicon carbide (SiC) composite cladding could offer significantly better performance and safety of fuels under accident conditions, but so far engineers have been unsuccessful in developing a suitable, robust joint to seal the cladding tube. Our researchers have now identified ceramic brazing as a promising material and method for reliable gas-tight joints. They have experimented using a novel laser-based technique and a range of potential ceramic brazes. They brazed joints on representative SiC material and then subjected them to extreme conditions inside an autoclave as well as a range of mechanical tests. These studies have identified a preferred braze composition. Early results from ion beam irradiations suggest that the braze material has good performance and maintains integrity under irradiation conditions. We are supporting patent protection for the braze composition and the manufacturing method.

**Routes to exploitation**

- We have assessed a wide range of potential braze materials, and have identified a composition that can be successfully manufactured using a laser-based technique, and that has demonstrated good resistance to corrosion and dissolution under representative autoclave testing
- Early evidence from proton beam irradiation suggests that the braze will be effective in withstanding the high levels of neutron fluence in the core
- We are working with industry partners to identify fuel materials that are particularly well suited to use with SiC cladding. At present, uranium silicide appears to offer several significant advantages, including a higher density and higher thermal conductivity than the current fuel material (uranium dioxide). Higher density offers the important advantage of allowing a reduced level of enrichment (or extended cycle lengths) and hence better economic performance. We are exploring manufacturing routes for uranium silicide and how these influence properties of the resulting fuel material

**The way forward**

- We are continuing to demonstrate the effective performance of the braze by extending the range of autoclave and, in particular, irradiation testing
- At the same time, we are working to optimise the braze manufacturing conditions, with particular emphasis on identifying a range of acceptable manufacturing parameters, whilst minimising the time needed to produce the joint (Figure 14)
- Westinghouse and Rolls-Royce have expressed an interest in patenting or licensing the new technique
2. How can the thermal performance of uranium dioxide (UO₂) fuel be improved?

Uranium dioxide is the fuel material used in all of the world’s civil nuclear power plants. It has demonstrated good performance, but suffers from one significant drawback: its poor thermal conductivity. This results in high operating temperatures and consequently a high core stored energy, as well as increasing the fraction of gaseous fission products released from the fuel.

A NNUMAN project has explored the potential of composite fuels to overcome this limitation. The research team identified SiC and molybdenum (Mo) as two potential additives to improving UO₂ thermal conductivity. Subsequent work (not funded by NNUMAN) has shown that SiC additions are likely to react with UO₂ during operation, resulting in undesirable SiO₂ and UC being formed (Figure 15). However, data from experiments with Mo additives appear more promising and further examination of this material is now underway.

Routes to exploitation

• Higher thermal conductivity of UO₂ fuel would reduce operational risks by reducing the core stored energy (and hence reducing the demand on emergency core cooling) and improving the performance of reactors by reducing the fraction of fission gases released from the fuel

• Advanced fuel materials such as uranium silicide could offer significant improvements in performance, but also provide a significant licensing challenge, and are likely to require extensive testing and validation. By comparison, a modest change to the existing fuel material is likely to present a lower licensing barrier and shorter times to commercialisation

• The addition of Mo to UO₂ cannot easily be explored by our partners at NNL because they do not have an established uranium recovery route. The smaller quantities used by Manchester allow us to explore this option

The way forward

• We are working with NNL, Westinghouse and Rolls-Royce as our industry partners

• In conjunction with our work on composite fuel materials, we are also investigating the potential of a novel fabrication method, Spark Plasma Sintering, to reduce fuel manufacturing times and hence improve economic performance

Figure 14: Ceramic brazing using a 1 kW fibre laser

Figure 15: UO₂ containing SiC particles showing the induced micro-fracturing
Characterising the mechanical performance of small irradiated material

Nuclear engineers need to know how radiation affects the mechanical response of materials; this information is vital so they can predict the evolution of components once they are placed in service. The selection of materials with robust in-service performance profiles contributes to safe and efficient plant designs and operation.

Unfortunately, samples of ex-service and test reactor material are rare and usually too expensive for many researchers to source. Furthermore, irradiated samples often come from legacy reactor designs, but researchers want to test new materials, manufacturing methods or components for the next generation of reactors.

Scientists have developed methods that irradiate and damage specimens much faster than would occur in an actual reactor core. These techniques can quickly generate alloy samples with damage comparable to many years of irradiation. Protons are used as a surrogate for neutrons because they produce defects in materials more efficiently than neutrons, but have limited residual activity. However, the charge of protons drastically limits their ability to penetrate into a sample surface, so accelerated proton irradiation produces thin irradiated layers rather than bulk irradiated specimens. This makes it difficult to generate standard mechanical test data that is suitable for reactor design.

We expect our research to establish a new method for obtaining stress-strain curves using a novel combination of well-established techniques at a relatively low cost. The method uses laboratory X-ray diffraction (XRD) stress analysis ($\sin^2 \theta$) and digital image correlation (DIC) to record stress and strain in a proton-irradiated surface during mechanical loading (Figures 16 & 17). So far, the XRD-DIC method has been applied to non-irradiated material and validated against standard mechanical tests. Verification of the technique applied to irradiated samples will use small grain-sized, micro-machined samples using the unique plasma-focused ion beam (PFIB) facility at The University of Manchester (Figure 18).

XRD and DIC are standard laboratory methods that are readily available in institutions so the combination technique should experience rapid adoption for stress-strain analysis in a wide variety of disciplines and studies.

Figure 16: In situ measurement of applied stress in near surface region using XRD
Figure 17: Flow curves of bulk (broken line) and surface (points) error bars represent the gradient error of the sin$^2$Ω plot, proof stress is 641 MPa and 652 MPa respectively; (b) Elastic range of bulk and surface stresses are 188.7 GPa and 188.8 GPa respectively; (c) Log-log plot of flow curves for bulk and surface, with strain hardening exponent 0.122 and 0.126 respectively. These data were all obtained on low alloy ferritic steel SA508 Grade 4N.

Figure 18: Flow curves of bulk (unbroken line) and small sample (open circles). Proof stress, Young’s modulus and initial strain hardening parameter are in good agreement; (b) Specimens mounted and tested in situ to failure in Zeiss Sigma SEM.
Our industrial links

NNUMAN has built effective working relationships with its industrial partners through a bi-annual Technical Advisory Board. If you would like to attend, to contribute or learn from our research output, please contact the team (see page 23).

Amec Foster Wheeler
Ansaldo Nucleare
AREVA
BAE Systems
Bodycote
Carpenter Powder Products
Culham Centre for Fusion Energy (CCFE)
Doosan Babcock
EDF Energy
Electric Power Research Institute (EPRI)
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Nuclear AMRC
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SPX ClydeUnion Pumps
Starrag UK Ltd
Tata Steel
TWI
UK Research Centre for NDE (RCNDE)
US Department of Energy
Westinghouse / Springfields Fuels Ltd

Useful links

Key websites
The NNUMAN website
www.dalton.manchester.ac.uk/NNUMAN
SharePoint
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NNUMAN publications
The full list of publications of NNUMAN's work is available at:
www.dalton.manchester.ac.uk/NNUMAN

Annual reviews
2012-13 Annual Review
www.man.ac.uk/wVa3PT

2013-14 Annual Review
www.man.ac.uk/9opHnw

2014-15 Annual Review
www.man.ac.uk/xdJiw0
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