SEM and EBSD Studies of Ion Implanted Stainless Steel

J. Davis[1]*, K. Short[1], R. Wuhrer[2], M. R. Phillips[2], G. R. Lumpkin[1] and K. R. Whittle[1]
[1]Institute of Materials Engineering, Australian Nuclear Science and Technology Organization.
[2]Microstructural Analysis Unit, Faculty of Science, University of Technology, Sydney.
*Corresponding author: Email joel.davis@ansto.gov.au

Abstract: Research is currently in progress to determine the effect ion irradiation at varying energy and fluence has on the microstructure of several grades of stainless steel. Plasma Immersion Ion Implantation (PI^3) and the Small Tandem for Applied Research (STAR) accelerator have been employed in this research to simulate some of the effects from a neutron irradiation environment using helium ions. The overall aim of the research is to eventually investigate other unique materials that have applications in both GenIV and fusion reactor programs and the Advanced Fuel Cycle Initiative (AFCI), e.g., inert matrix fuels, reactor core liners, and structural materials within the reactor (ODS materials) [2]. The main focus of this work will be to study the microstructure and understand grain-boundary interactions, and how they relate to the tolerance of materials to damage.

Introduction: PI^3 is a method designed to implant large areas of samples simultaneously, to very high fluences. The process involves forming an ionised plasma at a controlled temperature with radio frequency (RF) excitation. Ion implantation occurs when the positive ions from the plasma are accelerated towards all exposed surfaces by applying a high negative voltage to the sample. It has previously been used for surface hardening and improving corrosion properties of metals [1]. Fully commissioned in 2004 STAR is a modern, compact and computerised facility housing a 2 MeV tandem accelerator. It has been designed specifically with state-of-the-art capability for dual functionality, providing both ion beam analysis (IBA) and high-throughput and high-precision accelerator mass spectrometry (AMS) radiocarbon analysis. Using both PI^3 and STAR we can bombard a material with ions in both a low energy high flux and a high energy low flux situation to simulate the effects from a neutron irradiation environment.

Small pieces of stainless steel of varying grades have been prepared for EBSD analysis using the Struers method for polishing Low Carbon Steels (Method No. 1879). The samples were then mapped using a Zeiss Ultra Plus SEM with an Oxford Instruments HKL NordlysS EBSD detector system. The map was generated using the HKL Channel 5 software. Ion implantation utilising helium gas at 40 kV accelerating voltage and to a fluence of ~ 1 x 10^{17} ions cm^{-2} was used in the PI^3, portions of the materials were shielded using both a silicon wafer and a TEM grid. Post irradiation EBSD was used to analyse both the shielded and non-shielded surfaces. The STAR accelerator was operated at 5 MeV and to a fluence of ~ 2 x 10^{17} ions cm^{-2}, post irradiation EBSD was used to analyse the sample surface. Cross sections where made to study the damage and depth of penetration.

PI^3 using helium ions significantly degraded the backscattered electron images and EBSD patterns on the stainless steel samples [3]. Irradiation induced damage affected the crystallinity of the surface. Physical damage from the irradiation to the surface preparation for EBSD has removed the coherent scattering and effectively given rise to diffuse scattering from the surface. These results have also been confirmed using AFM and nano-indentation.

Current research and future research investigation of various grades of stainless steel will be discussed. A method for preparing cross sections of the irradiated steel has been developed; edge rounding in the cross sections has been kept to less than 100 nm. Fig. 2 shows an example of this method of sample preparation being used to visually determine with orientation imaging the penetration depth of the damage in the cross section. EBSD has also been used to determine whether patterns are obtainable from the damage layer in the cross section. Further experiments will be focused on a range of doses in combination with nano-indentation, to study the effect of radiation on the recovery from stress in materials, and the role it plays in recovery.

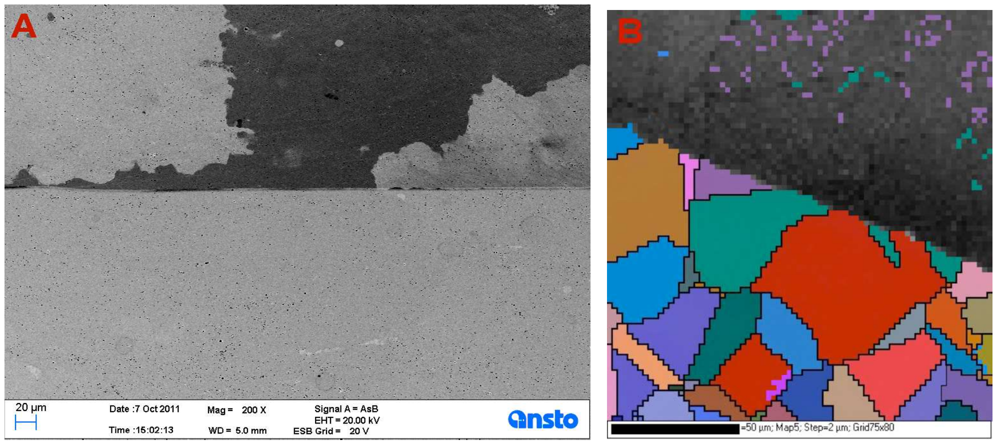

Fig 1 a) Backscattered electron image showing the PI^3 irradiated and shielded regions in ODS material and b) EBSD map showing the loss of EBSD pattern in the PI^3 irradiated region (top of map) in 316 stainless steel.

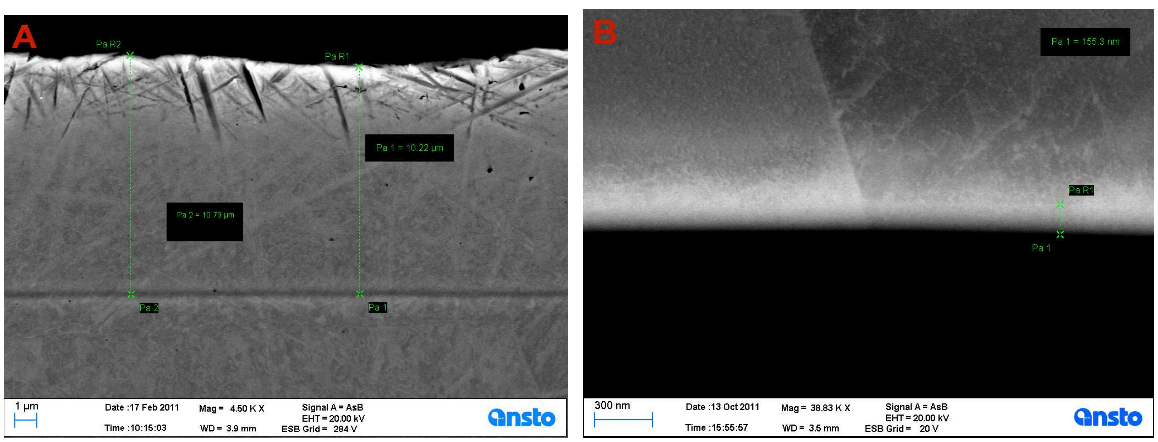

Fig 2 a) Angle selective backscatter image of the STAR irradiated stainless steel showing the helium ion damage layer in the cross section and b) Angle selective backscatter image of the PI^3 irradiated stainless steel showing a loss of crystallinity in the sub 200 nm region under the surface of the stainless steel.

Acknowledgements

We would like to acknowledge the help of the following people from the Institute of Materials Engineering, ANSTO. Robert Aughterson for cross section sample preparation, Tim Palmer and Clint Jennison for EBSD sample preparation. We would also like to thank Mihail Ionescu from the Institute for Environmental Research, ANSTO for the STAR irradiations.

References

[1]. M.J. Kenny, L.S. Wielunski, J. Tendys, G.A. Collins, A comparison of plasma immersion ion implantation with conventional ion implantation, Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, Volumes 80-81, Part 1, 3 June 1993, Pages 262-266

[2]. Steven J. Zinkle, Jeremy T. Busby, Structural materials for fission and fusion energy, Materials Today, Volume 12, Issue 11, November 2009, Pages 12-19

[3]. J. Davis, K. Short, R. Wuhrer, M. R. Phillips and K. Whittle, Plasma Immersion Ion Implantation of Stainless Steel 316, Microsc. Microanal. 17 (Suppl 2), 2011, © Microscopy Society of America 2011