

Investigation into Irradiation Effects in ODS Steels Using Ion Implantation and Micromechanical Testing.

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Abstract – Oxide Dispersion Strengthened steels are materials for the first wall of proposed nuclear fusion reactors including DEMO. They contain a nanoparticle dispersion of Y_2O_3 which restricts dislocation motion. The particles act as low energy sites capable of trapping helium. The experiments reported here focus on the mechanical effects of ion implantation on a model Fe-Cr alloy and an ODS equivalent. Samples were helium and iron ion implanted as a simulation of neutron damage and material transmutation. The main restriction of using ion implantation is the limited damage depth available in samples ($<3\mu m$), therefore nanoindentation was used to provide information on the surface hardness changes. Transmission Electron Microscopy was used to see how the dislocation structure within these test volumes changes with implantation.

Keywords – ODS, micromechanics, nanoindentation, ion implantation

I. INTRODUCTION

Nuclear fusion reactor materials have to be designed to withstand extreme environments, whilst remaining structurally and economically viable. The reactor first wall will be exposed to elevated temperatures ($>550^\circ C$), neutron damage ($\sim 20 dpa/year$) and helium transmutation ($\sim 10 appm/dpa$) [1].

Proposed materials for the first wall of fusion power plants, such as DEMO, include low activation steels based on iron-chromium alloys [2]. Oxide Dispersion Strengthened (ODS) Fe-Cr alloys are being considered for their superior properties under these conditions, compared with conventional ferritic-martensitic alloys. ODS steels can be produced from low activation elements and they have good mechanical, corrosion and thermal properties [3]. They also demonstrate good resistance to microstructural change in irradiation environments. They are BCC steels containing a dispersion of nanoscale particles of yttria which restrict dislocation motion, increasing yield strength and creep strength [4]. The usual processing route is mechanical alloying of steel and Y_2O_3 powders, followed by compression through hot (isostatic) pressing or extrusion. The processing is crucial for optimising the final material properties [5][6].

Ideally test materials would be subjected to reactor

conditions, however, neutron damage causes the materials to become radioactive and therefore all testing must be carried out in a controlled environment. This means that ion-implantation is often used as a simulation. Helium can be implanted to simulate the gas that would be introduced from the plasma, or transmuted from the wall material under reactor conditions, while self-ion implantation (i.e. Fe ions into an Fe substrate) is used to form damage cascades similar to those that would be induced by incident neutrons [7].

Ion-implantation, however, is limited to a thin, near-surface layer of the material ($\sim 1-10\mu m$ depending on the ion species and energies used) therefore it is necessary to perform all tests on the micro-scale. Nanoindentation makes it possible to accurately measure hardness changes in the surface of the implanted material.

II. MATERIALS AND METHOD

Tests were performed on two different materials:

- melt processed Fe-14wt%Cr – a model alloy provided by EFDA [8]
- extruded Fe ODS from CEA Saclay [9] ($\sim 14wt\%Cr + W, Mo, Ti, Y, Si, Ni \& Mn$) – an approximation to a commercial alloy.

Cut samples were ground with a final stage of 4000 grit silicon carbide paper and polished with 40nm colloidal silica – 10% hydrogen peroxide solution on a chemo-mechanical polishing cloth. This method has been shown to give a high quality surface finish, free from damage [10].

Implantations were planned using SRIM software [11] to predict the ion range in the materials, giving fairly uniform distribution to the required depth.

One set of samples was He ion implanted at the Surrey Ion Beam Centre at $300^\circ C \pm 10^\circ C$ to $\sim 2000 appm$ He in a flat profile to $\sim 3\mu m$ depth. This was done with ten successive implantations at 1.8, 1.6, 1.4, 1.2, 1.0, 0.8, 0.6, 0.4, 0.3 & 0.2 MeV with He^+ ions. Bulk samples were mounted on a stainless steel polishing plate and the temperature was recorded through a thermocouple mounted on the surface next to the samples. A second set was dual beam implanted

at JANNUS, CEA Saclay, with ~ 2000 appm He and Fe to cause 3dpa damage also to $\sim 3\mu\text{m}$ depth at 300°C . This used Fe^{8+} at 24MeV with spun aluminium degrader foils of 5, 3.8, 2.8, 1.6 & $0.8\mu\text{m}$ and an empty slot on one beam line. The second beam line used He^+ at 2MeV with degraders of 5, 4, 3, 2 & $0.8\mu\text{m}$ and an empty slot.

Nanoindentation was performed using an MTS Nanoindenter XP with a Berkovich tip and continuous stiffness measurement, allowing hardness as a function of depth to be measured. Each line on the graphs is an averaged hardness over 20 indents, with the error bars showing one standard deviation. Each material was tested and calibrated in a set covering all implantation conditions. The indentations for hardness testing were made to a depth of 3000nm at a loading rate of 5nm s^{-1} . The indentations for Transmission Electron Microscopy (TEM) imaging were made to 100nm depth.

Samples for TEM imaging were cut from the material, thinned using a Zeiss Auriga FIB-SEM and then imaged on a JEOL JEM-2010 at 200kV.

III. RESULTS AND DISCUSSION

The graphs 1a and 1b show that the ODS material has a higher baseline hardness than its non-ODS equivalent.

Implantation with 2000appm He causes a significant increase in hardness – this change is more pronounced in the non-ODS material. Also visible on the graphs (most noticeable in the Fe-14wt%Cr) is a shoulder in the hardness around the 500nm depth mark on the 2000appm He line.

All the curves converge to their respective baseline hardnesses at around 2000nm depth. Although the implantations are to a depth of $\sim 3000\text{nm}$, the plastic zone size of the indent at depths below 2000nm will make the effects of the implanted ions negligible [12].

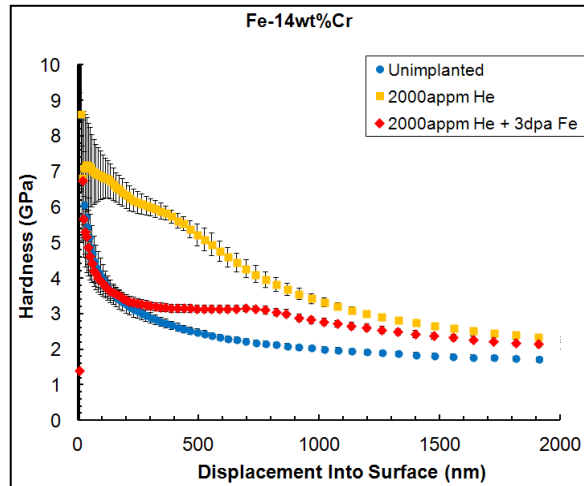


Fig. 1a Hardness vs. displacement in Fe-14wt%Cr

A. 2000 appm He

The hardness changes in materials are due to the He forming defects in the lattice, so restricting dislocation motion. As He is implanted, it is most likely to initially fill vacancies (generated by ion collisions) in the ferritic matrix. Above a critical concentration, He starts to collect into bubbles throughout the matrix, which causes significant hardening. In Fe alloys, the hardness results indicate that this concentration is >100 appm He [13]. At 2000appm He, where significant hardening occurs, a bubble population has been seen in TEM images take of these materials [14].

In ODS materials, the nanoparticles also provide low energy locations for the helium to collect and form fine, well dispersed, bubbles. This reduces bubble population and therefore the hardening effects in the material. It is also expected to reduce the amount of helium migrating to the grain boundaries so reducing the grain boundary embrittlement.

B. 2000 appm He + 3 dpa Fe

It is interesting to observe, however, that in the dual-beam implanted samples the hardening effect of the He is lessened compared to the He only implantation, and in the case of the ODS material, apparently reversed – i.e. the hardness is lower after implantation.

As the Fe ions are implanted they cause damage cascades in the lattice, simulating the primary knock-on atom of an incident neutron. The “collision cascade” thus produced, results in residual point defects and prismatic dislocation loops [15]. A possible explanation for the decreased hardening in the dual beam experiment is that the implanted He is attracted to these defects and so there is less spread throughout the lattice to cause the hardening that was seen in the materials implanted with He alone. The reason for the apparent softening in the ODS material is unclear.

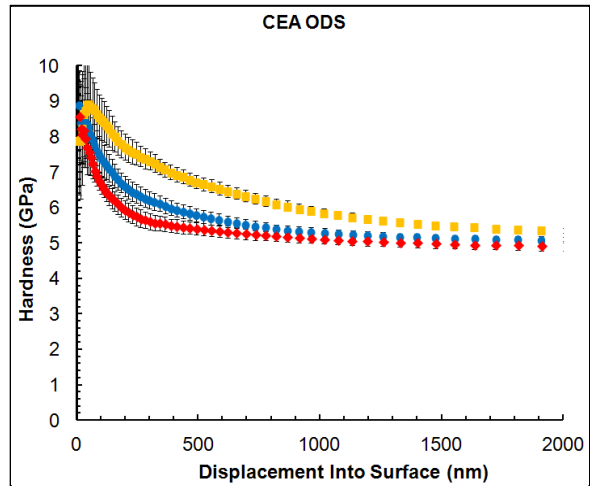


Fig. 1b Hardness vs. displacement in CEA ODS

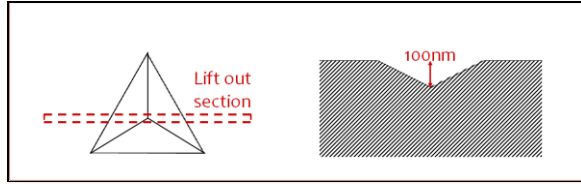


Fig. 2 Diagram of lift out geometry

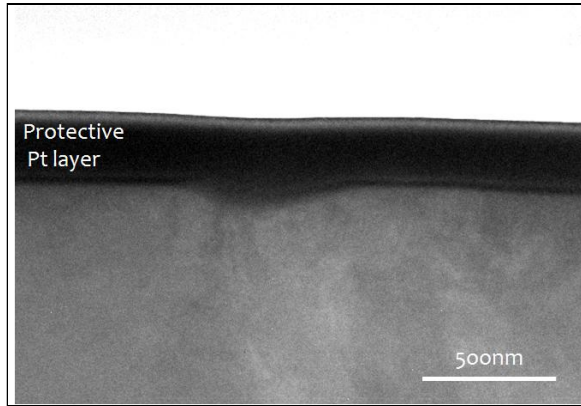


Fig. 3a TEM of indentation cross section in Fe-14wt%Cr + 2000appm He

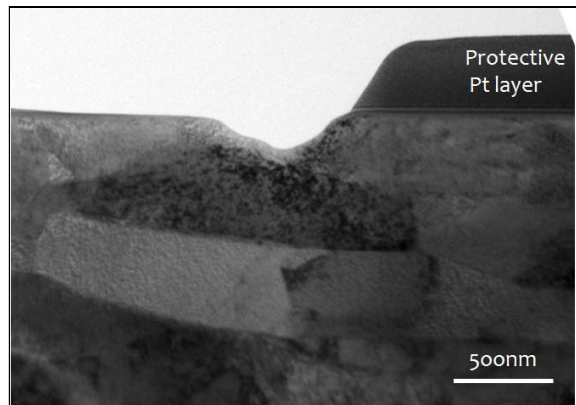


Fig. 3b TEM of indentation cross section in CEA ODS + 2000appm He

C. TEM of Indentation Lift-outs

In order to understand the mechanical property changes, FIB-cut lift-outs were made across shallow nanoindentations (Figure 2).

Preliminary images 3a and 3b show higher density dislocation structures beneath the indentations in the ODS materials (3b) than the non-ODS (3a). In 3a slight darker bands can be made out spreading from the indentation showing the location of the damaged material. In 3b the dislocation structures are much more visible as dark spots below the indentation. This is possibly due to the dislocation interactions with the ODS particles, but further lift out samples with varying TEM diffraction conditions are required to draw any more definite conclusions.

IV. CONCLUSION

Implantation of high concentrations of helium into iron based alloys has a hardening effect. In ODS variants of these alloys the hardening effect is observed to be less. This is possibly due to the interaction of the ODS particles with the implanted helium.

When the samples are dual implanted with helium and iron ions the hardening effect is significantly changed, possibly due to helium/iron damage interactions.

In order to fully investigate the effects of the implantations and the reason for the differences in hardness changes, further experiments are in progress.

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