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A candidate fusion engineering material, WC-FeCr

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ABSTRACT

A new candidate fusion engineering material, WC-FeCr, has been irradiated with He ions at 25 and 500 °C. Ions were injected at 6 keV to a dose of ~15 dpa and 50 at. % He, simulating direct helium injection from the plasma. The microstructural evolution was continuously characterised in situ using transmission electron microscopy. In the FeCr phase, a coarse array of 3–6 nm bubbles formed. In the WC, bubbles were less prominent and smaller (~2 nm). Spherical-cap bubbles formed at hetero-phase interfaces of tertiary precipitates, indicating that enhanced processing routes to minimise precipitation could further improve irradiation tolerance.

The leading candidates for plasma facing materials (PFMs) in tokamak fusion power plants are tungsten and its alloys. The inherent brittleness of metallic tungsten [1] precludes its use in many structural applications, which has sparked research into tungsten-based composites with enhanced ductility. Recent approaches include fibre-reinforced tungsten [2], tungsten heavy alloys [3] and tungsten laminates [4]. One candidate class of materials that are widely employed in the extreme wear environments, but are as yet little explored as PFMs, is WC-composites. These materials possess excellent neutronics [5] and mechanical [6] properties, while they can be fabricated and shaped inexpensively. A particularly promising binder is the FeCr system, as it is low-activation and resistant to dry oxidation and irradiation.

Like all candidate PFMs a major concern with WC-FeCr composites is how their properties degrade under irradiation, in particular under helium bombardment. Helium will accumulate in PFMs via two mechanisms: firstly via (n, α) transmutation reactions and secondly from direct injection of helium ash from the fusion plasma, particularly in the near-surface region. Both processes can lead to formation of helium bubbles and associated defect structures (interstitial-vacancy pairs, dislocation loops, etc.). While these processes are well understood in many structural nuclear materials [7], they are as yet unstudied for WC-composites.

The general understanding of ion-irradiation in WC-composites is restricted mostly to surface hardening produced by N^{x+} ions on WC-Co (i.e. non fusion-compatible) materials [8]. Whilst the extent to which these studies pertain to helium irradiation or WC-FeCr is limited, we nevertheless discuss the general observations here. Most studies, typically employing 50–100 keV ions, report a two-stage microstructure-property evolution with dose: an initial increase in defect content, with a corresponding hardening, peaking at a dose of ~10^{17} ions/cm^2. Hardening is attributed to nitride particles forming within the binder [9] and high densities of dislocations and planar defects within WC particles [10]. Defect density is strongly dependent on particle orientation relative to the ion-beam [11]. This hardening is followed by softening due to amorphization of WC. Since the nitrogen is soluble (unlike helium), bubbles are never observed.

Irradiation of WC-composites with He^{x+}, on the other hand, is relatively poorly understood. Available information is limited to mechanical property evolution and in WC-Co only. For example, when irradiated with 32 MeV ions to ~1 × 10^{17} ions/cm^2, the surface hardness rises monotonically up to a 30% increase [12, 13]. These surface measurements were not on the helium accumulated region of the specimen (~100 μm below) and no microstructural observations were made. While some speculation about the performance of WC-FeCr can be made from previous studies of its constituents (e.g. monolithic Fe-Cr alloys [14, 15]) such predictions are limited, since there is currently little understanding reported on the response of metal-ceramic interfaces, which – as we will show here – can dominate microstructural evolution.

In what follows, we report He ion irradiation with in-situ transmission electron microscopy (TEM) on a WC-FeCr composite. Irradiations were performed at room temperature and 500 °C (a local maximum

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Table 1
Maxima in displacement damage and injected helium, as predicted from SRIM (ions), compared to FISPACT-II simulations (neutrons).

Phase	Neutron dpa	Ion dpa	Neutron He (at. %)	Ion He (at. %)
WC	10	15	0.020	49
FeCr	16	16	0.019	48

for swelling in ion-irradiated FeCr alloys [15]). Bubble size is reported as a function of irradiation dose and temperature. Large bubbles are observed at the interface of impurity phases, which could significantly embrittle the material. Our observations enable processing recommendations for removing these phases and thus enhancing the material's irradiation tolerance.

Tungsten carbide composite plate was obtained from Sandvik Hyperion Ltd. It was manufactured via a conventional liquid phase sintering process, details of which can be found elsewhere [16]. The material contained a nominal weight fraction of 0.9 WC particles and 0.1 Fe-Cr binder, which itself had respective weight fractions of 0.92 Fe and 0.08 Cr. A Cr fraction of 0.08 was selected as an intermediary value between 0.05 and 0.09 – which are known local minima in FeCr alloys for void swelling [15] and ductile-brittle transition temperature [17], respectively.

TEM samples were prepared using an FEI Quanta dual beam Focused Ion Beam system employing Ga ions. Ion-irradiations were performed at the Microscope and Ion Accelerator for Materials Investigations (MIAMI) facility, details of which are given elsewhere [18]. Bright-field (BF) images were collected using a JEOL JEM-2000FX TEM, operated at 200 keV, using a slightly off zone-axis beam. The sample temperature was controlled during irradiation using a Gatan 652 double-tilt heating holder. High-angle annular dark-field (HAADF) TEM images of the as-received material were collected on a JEOL JEM-2100F microscope.

Samples were implanted with 6 keV He^+ ions to a dose of 2×10^{17} ions/cm^2. Ion stopping distributions were calculated (for low helium concentrations) using the software package SRIM [19]. The depth of the helium distribution's maximum was predicted to be 19 and 26 nm for WC and FeCr, i.e. well within the TEM foil thickness. The corresponding maxima in injected helium and displacements per atom (dpa) are reported in Table 1, alongside values for one year of neutron irradiation under a fusion relevant spectrum, as calculated using the FISPACT-II code [20]. The peak ion damage was approximately 15 dpa for both phases, which is comparable to one year of neutron damage (10–16 dpa). The maximum amount of helium introduced, if fully retained – i.e. not redistributed or lost from the foil edges – would correspond to ~50 at. %. Thus, the amounts of He introduced here are far in excess of the predicted annual He production from neutron irradiation alone (~0.02 at. %). Instead, they more closely resemble the direct injection of helium ash in the near surface region from the fusion plasma.

We first report the structure of the un-irradiated material as observed in the TEM. Fig. 1 contains a HAADF image in the top-left, showing heavier elements, i.e. WC particles, in bright contrast and lighter elements, i.e. FeCr binder, in dark. The WC particles are ~0.5–1 μm in diameter. Turning to the EDS maps in the smaller boxes, two other phases are distinguishable, in addition to the nominal constituents. Firstly, in the Cr-map, there are four regions of ~100 nm in diameter, delineated by dotted circles, where the Cr-content is high. Semi-quantitative chemical analysis estimates the Cr-enrichment to be ~10-fold higher than the binder. The significant carbon content, and negligible W or Fe in these particles suggest they are Cr-carbide,

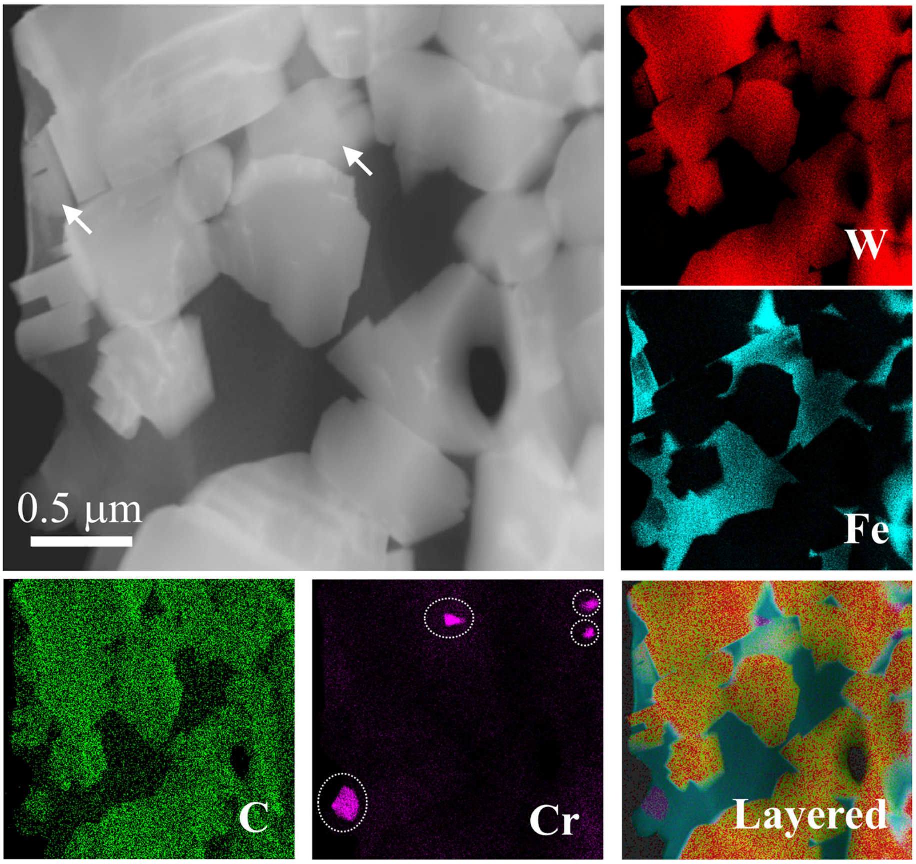

Fig. 1. Top left - HAADF image of the as-received material showing WC (light) and FeCr (dark). Smaller images - EDS dot maps showing W (red), Fe (blue), Cr (purple), C (green) and their sum (Layered). Cr-carbides are indicated by dotted circles and M_6C by arrows.