Journal of Nuclear Materials 486 (2017) 34–43

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

The effects of ion irradiation on the micromechanical fracture strength and hardness of a self-passivating tungsten alloy

Moritz T. Lessmann [a, b, *], Ivan Sudić [c], Stjepko Fazinić [c], Tonči Tadić [c], Aida Calvo [d], Christopher D. Hardie [b], Michael Porton [b], Carmen García-Rosales [d], Paul M. Mummery [a]

[a] *School of Mechanical Aerospace and Civil Engineering, The University of Manchester, Manchester, UK*
[b] *CCFE, Culham Science Centre, Abingdon, UK*
[c] *Rudjer Bošković Institute, Bijenička cesta 54, 10000 Zagreb, Croatia*
[d] *Ceit-IK4 and Tecnun (University of Navarra), San Sebastian, Spain*

ARTICLE INFO

Article history:
Received 18 September 2016
Received in revised form
14 December 2016
Accepted 26 December 2016
Available online 29 December 2016

Keywords:
Fracture strength
Tungsten
Self-passivating
Micromechanics
Irradiation effects
Ion implantation

ABSTRACT

An ultra-fine grained self-passivating tungsten alloy (W88-Cr10-Ti2 in wt.%) has been implanted with iodine ions to average doses of 0.7 and 7 dpa, as well as with helium ions to an average concentration of 650 appm. Pile-up corrected Berkovich nanoindentation reveals significant irradiation hardening, with a maximum hardening of 1.9 GPa (17.5%) observed. The brittle fracture strength of the material in all implantation conditions was measured through un-notched cantilever bending at the microscopic scale. All cantilever beams failed catastrophically in an intergranular fashion. A statistically confirmed small decrease in strength is observed after low dose implantation (−6%), whilst the high dose implantation results in a significant increase in fracture strength (+9%), further increased by additional helium implantation (+16%). The use of iodine ions as the implantation ion type is justified through a comparison of the hardening behaviour of pure tungsten under tungsten and iodine implantation.

1. Introduction

Self-passivating tungsten alloys are currently being investigated to respond to the safety concerns of pure tungsten as plasma facing first wall materials in a future fusion reactor. In an accident scenario encompassing loss of coolant and air ingress, tungsten components may remain at elevated temperatures and oxidise volatilely, leading to a release of radioactively activated tungsten oxides [1]. By alloying tungsten with oxide-forming elements this oxidation can be retarded by up to 2–3 orders of magnitude [2]. A number of systems have been studied, with chromium and titanium (i.e. W88-Cr10-Ti2 in wt.%) seen as promising alloying elements [1], even though recent work reveals that alloys of the W-Cr-Y system exhibit even higher oxidation resistance [3].

Similarly to pure tungsten, the W-Cr-Ti system has a bcc crystal structure and features a ductile to brittle transition temperature (DBTT). Recent bend tests place the DBTT of W-Cr10-Ti2 in the range of 900–950 °C [4], meaning a significant fraction of the material as part of a first wall component may operate in the non-ductile, brittle regime. The Weibull's weakest link theory, which is currently being considered as a possible tool for the design assessment of brittle materials, relies on linear-elastic fracture strength data [5,6]. For a full design lifetime assessment it is essential to have an understanding of the effects of neutron irradiation on the material's fracture strength.

The fusion neutron spectrum is defined by a unique 14 MeV peak and can result in an annual damage rate of up to 14.5 displacements per atom (dpa) per year in tungsten [7]. The combination of this high energy and fluence means that fusion relevant irradiation campaigns cannot be conducted in currently available test reactors or neutron irradiation facilities [8]. Recent focus has hence been placed on irradiating fusion relevant materials in ion irradiation facilities [9–12]. Ion irradiation enables the accumulation of high damage levels in very short time frames and is therefore a great tool for early irradiation effect studies. Besides questions surrounding its applicability to simulating neutron induced damage (transmutation effects, primary knock on atom

---

* Corresponding author. School of Mechanical Aerospace and Civil Engineering, The University of Manchester, Manchester, UK.
E-mail address: mor.lessmann@gmail.com (M.T. Lessmann).

http://dx.doi.org/10.1016/j.jnucmat.2016.12.030
0022-3115/

energy), one of its main experimental disadvantages is the associated small penetration depth. Accelerated ions in the range of 10s of MeV can only penetrate the material a maximum of several micrometres. Standard strength tests gauging thicknesses in the range of hundreds of microns to several millimetres are therefore not applicable.

It has recently been shown that through cantilever bend tests at the microscopic scale reliable and repeatable fracture strength measurements of W-Cr10-Ti2 are obtainable within the material's linear-elastic response window [13]. The cantilever test geometry adopted here queries a regular material cross-section of approximately 3.5 μm, with an additional 2 μm deep triangular undercut beneath. Tensile stresses in the cantilever beams are found solely within the first few microns of the regular cross-section. This means it is possible to perform repeatable tensile fracture strength tests within a homogeneously ion implanted damage layer.

In general heavy ion implantations are conducted with self-ions [9–12]. However, due to the low stripping efficiency of tungsten ions it is not possible to achieve a combination of the required penetration depths and desirable doses in manageable time frames. We therefore present here an irradiation campaign conducted with iodine ions, which are able to produce doses of several dpa to depths of 3–4 μm within a timeframe of hours. El-Atwani et al. [14] have previously compared the effects of different ion types on the defect morphology of tungsten. Whilst differences in defect density and coalescence were observed, it was found that the heaviest ion type, copper, produced similar defect cluster sizes. It was therefore assumed that the even heavier iodine ions present an acceptable alternative to tungsten ions; whilst they likely do not result in identical damage morphology, they enable a first approximation of possible irradiation effects on the material's fracture strength. This is further supported by area corrected nanoindentation hardness measurements on tungsten and iodine implanted pure tungsten, which, as will be shown here, results in similar irradiation hardening behaviours.

2. Experimental methods

2.1. Self-passivating tungsten alloy

All tests were conducted on a mechanically polished W88-Cr10-Ti2 alloy (in wt.%) produced at Ceit-IK4 via a powder metallurgy route [4]. The material consists of two bcc phases, (αW,Cr) and (αCr,W) with a vol% of 88 and 11, respectively, and a minority Ti-containing phase, which are homogeneously distributed within the size-scale of cantilever beams [13,4]. The microstructure of the material is ultra-fine, with an average grain size of the majority (αW,Cr) phase of 209 ± 5 nm as measured by quantitative metallography (according to ASTM E112). The individual grain size ranges from 50 to 500 nm. The material has a relative density of 99.0%. The material has some large-scale heterogeneity in the form of chromium and titanium flakes spanning several tens of microns in size. All work was performed in areas well away from and clear of any observable heterogeneity within the matrix of the material as discussed in greater detail in Ref. [13].

2.2. Irradiation procedure

All ion implantations were conducted at room temperature in the ERDA chamber using ion beams accelerated by a 6 MV Tandem accelerator at the Ruđer Bošković Institute. The ion beam was rastered for increased uniformity before entering the target chamber through a collimator of approximately 2 × 3 mm size. The maximum implantation area was limited by this collimator size. The ion beams generally showed a degree of intensity variation as visualised on exposed Kapton sheet (Fig. 1(a–c)). Implantations

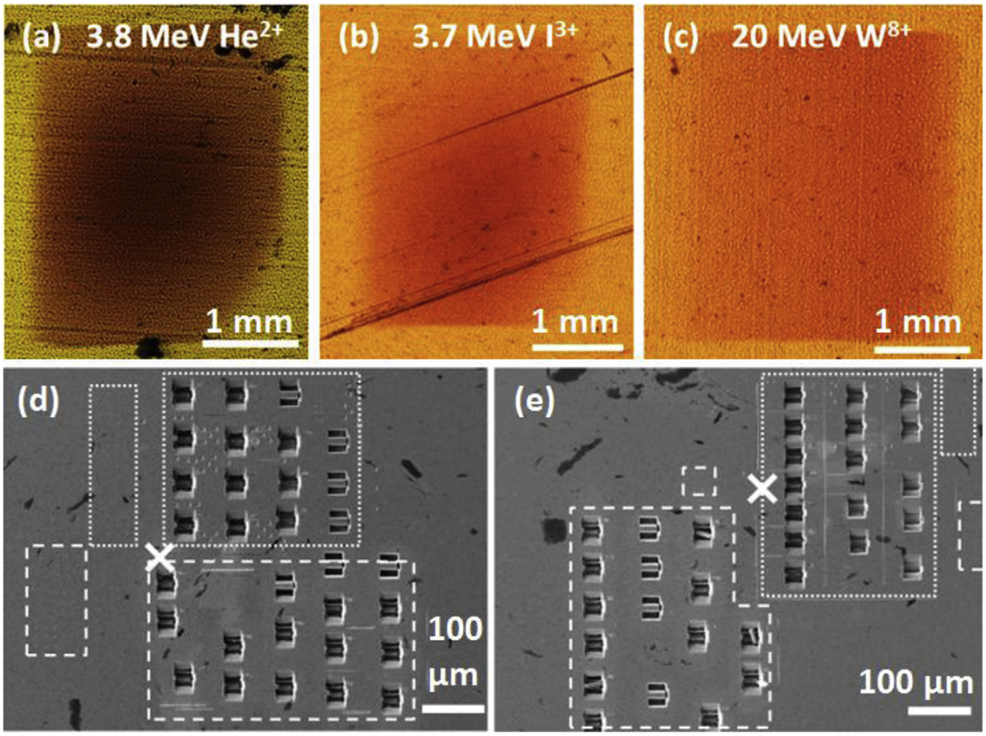

Fig. 1. (a–c) Kapton sheet exposed to various ion beams. A degree of intensity variation is observed. (d–e) Portion of implanted surface of sample Z02 (0.7 dpa) and Z03 (7 dpa), with its centre marked by a 'X'. Cantilever tests and indent arrays are highlighted by fine (heavy ion implantation) and coarse (heavy + He ion implantation) dashed boxes. Dark contrast flakes are chromium heterogeneities. All work was performed well away from these flakes.