#### Journal of Nuclear Materials 501 (2018) 319-328

Contents lists available at ScienceDirect

### Journal of Nuclear Materials

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

# The influence of low-energy helium plasma on bubble formation in micro-engineered tungsten



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#### ARTICLE INFO

Article history: Received 21 October 2017 Received in revised form 19 January 2018 Accepted 21 January 2018 Available online 31 January 2018

Keywords: Helium bubbles Tungsten Micro-engineered materials Plasma-material interaction Tungsten fuzz

#### ABSTRACT

Four different types of micro-engineered tungsten surfaces were exposed to low energy helium plasma, with a planar surface as control. These samples include two surfaces covered with uniform W-coated rhenium micro-pillars; one with cylindrical pillars 1  $\mu$ m in diameter and 25  $\mu$ m in height, and one with dendritic conical pillars  $4-10\,\mu\text{m}$  in diameter and  $20\,\mu\text{m}$  in height. Additionally, two samples with reticulated open-cell foam geometry, one at 45 pores per inch (PPI), and the other at 80 PPI were fabricated with Chemical Vapor Deposition (CVD). The samples were exposed to helium plasma at 30 -100 eV ion energy, 823-1123 K temperature, and  $5 \times 10^{25}$  -  $2 \times 10^{26}$  m<sup>-2</sup> ion fluence. It is shown that the formation of nanometer-scale tendrils (fuzz) on micro-engineered W surfaces is greatly reduced as compared to planar surfaces. This is attributed to more significant ion backscattering and the increased effective surface area that intercept incident ions in micro-engineered W. A 20% decrease in the average ion incident angle on pillar type surfaces leads to  $\sim$ 30% decrease in bubble size, down to 30 nm in diameter. W fuzz was found to be absent from pillar sides due to high ion backscattering rates from pillar sides. In foam samples, 28% higher PPI is observed to have 24.7%-36.7% taller fuzz, and 17.0%-25.0% larger subsurface bubbles. These are found to be an order of magnitude smaller than those found in planar surfaces of similar environment. The helium bubble density was found to increase with ion energy in pillars, roughly from 8.2% to 48.4%, and to increase with increasing PPI, from 36.4% to 116.2%, and with bubble concentrations up to  $9.1 \times 10^{21}$  m<sup>-3</sup>. Geometric shadowing effects in or near surface ligaments are observed in all foam samples, with near absence of helium bubbles or fuzz in deeper layers of the foam. © 2018 Elsevier B.V. All rights reserved.

#### 1. Introduction

Plasma-facing components (PFCs) in fusion energy devices will experience a huge flux of low energy helium ions emanating from the plasma edge. A significant fraction of the fusion reaction power is deposited by neutrons deep into the surrounding blanket and first wall structure, resulting in the production of helium gas from inelastic nuclear reactions. However, unlike helium gas production in the blanket and first wall structures, the helium flux to PFCs is orders of magnitude greater. Not only that, but the fact that helium ion energy is typically small (20–100 eV) does not allow helium ions to penetrate deep below the surface, and thus helium gas tends to agglomerate in large concentrations below the surface [1]. Thus, solid surface layers are full of gas, which leads to significant

\* Corresponding author. E-mail address: EdwardXiangGao@gmail.com (E. Gao). restructuring, potential material failure sites, and dust formation. This problem is very challenging, and there is the obvious need to develop mitigation strategies through understanding of the fundamental physics. The basic physics of deep helium ion pene-tration and the corresponding effects on material properties have been actively investigated several decades ago [2]. However, the interaction of low energy helium ions with metal surfaces has been more of a research focus in recent years. Since the acuteness of the problem stems from the near surface agglomeration of helium bubbles, one is left to wonder if there could be methods to manipulate surfaces so as to develop some degree of control over the existence of the severe gas/solid state in PFCs during plasma operation. The recent advent of many meta materials concepts through creative manufacturing provides the background for the present study.

Experimental studies on the interaction between energetic helium ions and various materials have been carried out in an effort to







map out the response of material surfaces to the implantation of helium ions. The effects of helium ions on tungsten (W) were especially characterized, because of the importance of W in the design of PFCs in ITER and DEMO [3,4]. A great deal of experimental information have emerged from the various studies at the NAGDIS-II plasma facility of Nagoya University, Japan [5-13], and the PISCES-A facility of the University of California, San Diego [4.12.14–16]. As a result, the general behavior of W when bombarded with low-energy helium ions is relatively well-established, and the dependence of the surface structure in W on ion energy, temperature, and fluence was mapped out [12]. The experimental studies showed that either small sub-surface bubbles form, or the surface itself is extruded into nano-fibrous structures containing small bubbles (referred to as W-fuzz). When the ion energy is greater than 20 eV and the temperature is in between 1000 and 2000 K, W nano-fibers form, otherwise nano-scale sub-surface bubbles precipitate. This picture is to be contrasted with the extensive surface restructuring observed under high energy helium bombardments (i.e energy in the keV range). More recent results have been obtained in the Plasma Physics Facility at the University of Wisconsin Madison [17,18]. The results of these studies show the formation of a high density of large, interconnected surface holes.

At low temperature in W (< 1000 K), trapped helium ions form small gas bubbles that diffuse very slowly with little coalescence into larger bubbles or surface holes. No substantial surface damage was observed in W when exposed to these conditions, and only surface ripples were detected [8,13]. At intermediate temperature, however, (1000 K <T < 2000 K), nano-fibrous structures (fuzz) are readily observed. Helium gas bubbles diffuse much quicker and coalesce into larger ones in the proximity of the surface. Additional Frenkel pairs are created from a trap-mutation mechanism, developed by Wilson et al. [19], assisting bubble growth. As bubbles migrate towards the surface, they extrude fuzz-like nano-fibers. Careful studies of this phenomenon on single crystal tungsten (SC-W) with (100) and (110) crystal orientations, as well as two doped tungsten samples, W-2at%Fe and W-2at%Cr were conducted by Kajita et al. [9]. Their findings suggest that there are no significant orientation difference in the surface morphology, nor a difference in between doped and undoped W. Similarly, Ueda et al. [12] discovered in their studies that helium bubbles are embedded within the tungsten nano fibers, and that the structures grow in height with increasing ion fluence. Nishijima et al. [5] studied the temperature dependence of this phenomenon. Their results on powder-metallurgy tungsten (PM-W) with increasing temperature and steady helium ion energy and fluence show a significant increase in bubble size with temperature. At 1300 K, only newly nucleated bubbles can be found, with a size of roughly 5 nm in diameter. Comparatively, at 1650 K, the bubbles were observed to coalesce to 200 nm in diameter; and at 1950 K, bubbles sizes up to 500 nm were observed. Although these temperatures are still within the fuzz formation regime, only bubbles and holes were observed at the higher temperatures, suggesting a helium ion energy threshold for fuzz formation near 20 eV - 25 eV. At high temperatures (T  $\geq$  2000 K), helium bubbles diffuse and coalesce to significant size before breaching the surface, eventually forming interconnected pin hole structures. This effect is observed by De Temmerman et al. [16].

Based on information from current literature, an overall defect "phase" diagram can be constructed to delineate three distinct surface morphologies ("phases"). Fig. 1 is a compilation of some of the data on W, indicating the presence of three morphologies: (1) ripples, (2) nano-fibers (fuzz), and (3) pin holes. The simplest boundary for fuzz formation within this "phase" diagram is temperature, where only wave like structures form on the surface below 1000 K and only large interconnected surface holes appear



**Fig. 1.** "Phase" diagram of fuzz formation region during helium plasma irradiation with ion fluence vs. temperature. Here the red triangles ( $\blacktriangle$ ) represent the study from Ref. [14]; the blue triangles ( $\blacktriangledown$ ) represent the study from Ref. [15]; the purple triangles ( $\blacktriangleright$ ) represent the study from Ref. [8]; the green triangles ( $\blacktriangleleft$ ) represent the study from Ref. [9]; black circles ( $\bullet$ ) represent the study from Ref. [20]; the red squares ( $\blacksquare$ ) represent the study from Ref. [13]; and the blue diamonds ( $\blacklozenge$ ) represent the study from Ref. [5]. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

above 2000 K. In addition, it appears that there also exist an ion fluence threshold around  $1.5\times10^{24}~m^{-2}$  for fuzz formation below which only surface waves are observed.

So far, the influence of the initial surface structure on the formation of surface ripples, nano fibers, and pin holes has not been systematically explored. Changing the local surface curvature intercepting helium ions, creating more surface area, and the presence of surface "tunnels" are all possible avenues to changing how helium ions interact with the surface. The main objective of the present study is to present the first systematic investigation of how such surface characteristics influence helium-induced morphologies explained above, and summarized in Fig. 1. To determine the effects of surface curvature, effective surface area, surface tunneling, and shadowing, two micro-engineered surfaces have been fabricated. The first type is in the form of massive surface micro pillars that have near conical shapes, thus affecting ion impact angle and effective surface area. The second type is a "foam" structure that looks like a 3D micro-truss, where ions can easily tunnel through open spaces, and where top foam ligaments shadow deeper ligaments. A set of low-energy helium plasma exposures are conducted on each architecture in the UCSD's PISCES-A facility. The results will be interpreted in terms of the initial surface geometry. In section 2, we discuss sample fabrication and preparation for plasma exposure experiments. This is followed by a description of the experimental procedure in section 3 and results obtained for these two types of W surface architecture in section 4. Finally, the discussions of discoveries are given in section 5 and conclusions are given in section 6.

#### 2. Micro-engineered sample preparation

Two different surface architectures have been fabricated for testing in a helium plasma environment similar to that expected for PFCs in fusion energy devices, such as DEMO. The fabrication process is primarily based on Chemical Vapor Deposition (CVD), and is described elsewhere [21]. The first type of micro-engineered surface is one covered with a uniformly dense layer of micro-pillars bonded firmly to the substrate. The second type is an open cell 3-dimensional foam structure, where each cell is composed of 12–14 ligaments, and the overall structure looks like a micro-truss. We discuss the salient aspects of each type here.

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