brought about by the formation of a surface layer consisting of a conglomerate of amorphous nanorod like structures that contain nanometre scale bubbles or voids [11]. The increased surface area, and fragility of these nanostructured surfaces, raises new concerns for the use of W as a fusion reactor plasma-facing material. Should these structures not be limited to the near surface, surface-thermal properties, fuel retention and the formation of tungsten nanodust will be issues that require further scientific investigation. As a first step towards understanding the formation of these nanostructured surfaces under fusion relevant operation, this paper focuses on the growth kinetics of the nanostructured layer and its composition.

2. Experimental procedures

W-target disc samples of 25 mm diameter and 1 mm thickness, were cut from a sheet of rolled PLANSEE 99.9999 weight % tungsten. Discs were polished to a mirror finish of average surface roughness below 50 nm and then exposed to pure He plasma in the Piscos-B linear-divertor-plasma simulator [12]. Target temperature regimes of 1120 and 1320 K, and plasma exposure times spanning (3-2.2 \times 10^3) s were explored. In each plasma exposure the local-target plasma conditions were kept fixed at a typical density of \( n_e \approx 4 \times 10^{18} \text{m}^{-3} \) and temperature \( T_e \approx 6-8 \text{eV} \) as measured by a reciprocating Langmuir probe. During plasma exposure, targets were biased to \(-75 \text{V}\) to facilitate energetic-ion bombardment of typical flux \( n_{\text{He}^+} \approx 5 \times 10^{12} \text{ions m}^{-2} \text{s}^{-1} \). The energy of the impinging ions is estimated from the target bias, \( \langle E_{\text{ions}} \rangle \approx V_{\text{bias}} - 2kT_e \) to be \(~60 \text{eV}\) under the present conditions, below the threshold \(~100 \text{eV}\) for physical sputtering. Subsequent to plasma exposure, each target is cross-sectionally cleaved and the sectioned surface observed, with no further preparation, in a JEM-6360 scanning electron microscope (SEM). Compositional information was obtained using an INCA energy dispersive x-ray microanalysis (EDX) system from Oxford Instruments.

3. Results

Figure 1 shows a cross-sectional SEM micrograph of a W target that was exposed at 1120 K to pure He plasma for \(4.5 \times 10^3\) s. The action of the He plasma is found to induce a dramatic change in the surface morphology. The once polished surface is transformed into a layer \( \approx 2 \mu\text{m} \) in thickness, of interconnected nanoscopic structures. Visually, these structures appear as a tangled mass of disordered nanorods that can be up to a micrometre in length, with widths of the order of only 10-50 nm. Interestingly, the nanorods appear to be consistently thicker in width near the unaltered underlying tungsten compared with those near the plasma-facing surface, perhaps indicative of a branching effect as individual structures grow.[2] In contrast to other studies [7] where bubble formation in the W bulk and along grain boundaries is noted, the image of figure 1 shows only a mild dimpling of the W material immediately below the nanostructured layer.[7]

Additional preparation steps, such as mounting, section polishing and conductive film deposition (C or Au), are destructive to the appearance of surface nanostructures that are under examination.

Figure 2. EDX spectra taken from the wide area regions of nanorods marked 'A' and 'B' in figure 1. The nanostructured layer is almost pure W. Small amounts of C and Mo are likely due to impurity deposition during plasma exposure. Measured O fractions are likely due to surface oxide passivation on contact with atmosphere when targets are removed from Piscos-B after cooling to ambient temperature.

Figure 2. EDX spectra taken from the wide area regions of nanorods marked 'A' and 'B', reveal that the structures are almost pure W. Near the plasma-exposed surface, small
amounts of C, O and Mo are present. Beneath the plasma-exposed surface the elements Mo and C are not present, but a small amount of O can be detected. The presence of near surface Mo and C is therefore likely due to sputtered impurity deposition from the Pasco-B plasma (the sample holder is made of Mo, and the intrinsic ionized C impurity fraction is known to be ~0.01% n_C). Based on this, it is thus interesting to note that once the nanostructured layer is sufficiently thick, the base (bulk W interface) of the growing nanomaterial is not exposed to the effects of the plasma. This is suggested by the absence of plasma impurities deeper within the nanostructure layer and points to the fact that the W nanostructure growth is supplied He from the plasma interacting surface but yet grows away from this surface into the bulk. Further, this W target, within uncertainty, showed no measurable weight loss after plasma exposure. Not only is this consistent with lower than sputter threshold ion bombardment but it also implies that the formation of nanostructures is not related to a sputtering/redeposition process.

The nature of the growth of nanostructured layers is further revealed by the SEM images of figure 3. In this selection of images, the cross-sections of five polished W targets are displayed in order according to increasing length of He plasma exposure time. These cross-sections clearly display nanostructured layer growth that is dependent on plasma exposure time. For the shortest exposure time of 300 s, as shown in figure 3(a), there is almost no indication of a modified surface, but as the exposure time is increased, the nanostructured layer grows in thickness to more than 5 μm for the exposure time of 2.2 × 10^4 s, shown in figure 3(e). The growth kinetics of the nanostructured layer is explored in figure 4. Here layer thicknesses, measured from SEM cross-sections, as in figure 3, are plotted against the square root of the plasma exposure time. The uncertainties reflect variations in defining the substrate and surface edges. Layer thickness data are shown for exposure temperature regimes of 1120 and 1320 K. For both exposure temperatures, the data are well characterized by a t^1/2 dependence, as indicated by straight line fits. The good agreement between the data and fits suggests that the nanostructured growth process is dominated by diffusion, at least in the range of plasma exposure times explored. If the simple one-dimensional growth law, d = (2Dn) t^1/2, arising from Fick’s law is assumed, these straight lines correspond to a diffusional growth process characterized by effective diffusion coefficients of D_{1120K} = 6.6 ± 0.4 × 10^{-12} cm^2 s^{-1} and D_{1320K} = 2.0 ± 0.5 × 10^{-11} cm^2 s^{-1}, and a thermal activation energy of 0.71 eV.