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Experimental Studies on Initial Formation of Fiber-Form Nanostructure grown on Refractory Metal Surfaces

高融点金属表面に成長する繊維状ナノ構造形成初期段階に関する実験的考察

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The initial stage of fiber-form nanostructure growth on tungsten and molybdenum surfaces with noble gas plasma irradiation is investigated with FE-SEM images by a technique that allows time evolution of nanostructure growth converted to spatial variation. It is concluded that loop-like nano-scale structures are thought to be precursors of fiber-form tendrils. Loop raptures and branching out are considered as possible candidates of the nano-fiber growth. Each schematic sketch for the above initial stages is given.

1. Introduction

Since the discovery of fiber-form nanostructures on tungsten (W) surfaces irradiated by helium plasmas [1, 2], a lot of attentions have been paid from many kinds of aspects [3]. Among these view points, the reason why such structures may be formed has been interested experimentally [4], theoretically [5, 6] and with numerical simulations [7, 8] over the world, not only from the view point of plasma-wall interactions in fusion devices [9] but also from those of fundamental solid-state physics and industrial applications.

In the case of W, the experimental conditions for formation of nanostructure are as follows: incident helium ion energy must be larger than ~ 25 eV and the W surface temperature would be 1000 ~ 2000 K. However, the uppermost temperature may change under a competition of formation and annihilation of nanostructure so that it depends on ion flux density.

Similar to W, molybdenum (Mo) needs the temperature range, 800 K ~ 1050 K for similar nanostructure formation [10]. The thickness of Mo fibers is twice as thick as that of W. On the other hand, tantalum (Ta) having the same bcc crystal structure as W and Mo does not show any fiber-form structure in the temperature range of (0.2 ~ 0.4)T_m [11] where T_m is the melting point, 3290 K, moreover even lower temperature range although the bubble and hole are formed at relatively high temperature.

When considering physical mechanism for nanostructure formation, a lowering of metal viscocity with helium nano-bubbles or clusters may have an important role through visco-elastic deformation concerning the origin of nanostructure growth. The fact that an high temperature annealing of nanostructure may recover an almost flat original surface may support the above-mentioned visco-elastic theory [12]. However, Elastic Recoil Detection (ERD) using oxygen ion beam reveals helium atomic content imbedded in W nano-fibers and found that the fraction is small compared with the expectation from visco-elastic model [13].

At present we do not have any conclusive model from both theory and numerical simulations [14]. It promotes again detail examination of formation mechanism experimentally, especially focusing on its initial stage.

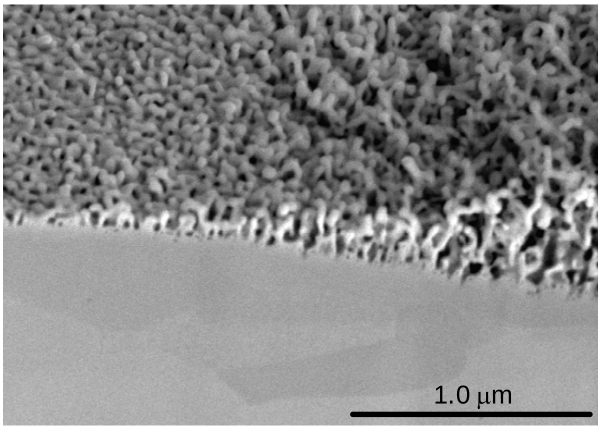

Fig.1. Oblique view of boundary zone sliced with CP (Cross-section Polishing) method which shows many loops.

2. Experimental Results

We have developed a technique on SEM images that allows time evolutions of nanostructure growth to be converted to spatial variation [15]. Examples are shown in Fig. 1 where the left-hand side has a smaller helium-ion fluence and it increases moving to the right-hand side. Many loop- or arch-like structures were observed in the low fluence region. It seems some loops stand almost perpendicular to the original surface, but others are at different angles, and the orientation of the loop surface is random. However, the size of loops is somewhat coherent. In the low-fluence region, the outer diameter of the loop is ~70 nm and the inner one is ~ 30 nm, while in the high-fluence region, the diameters becomes about twice, respectively.

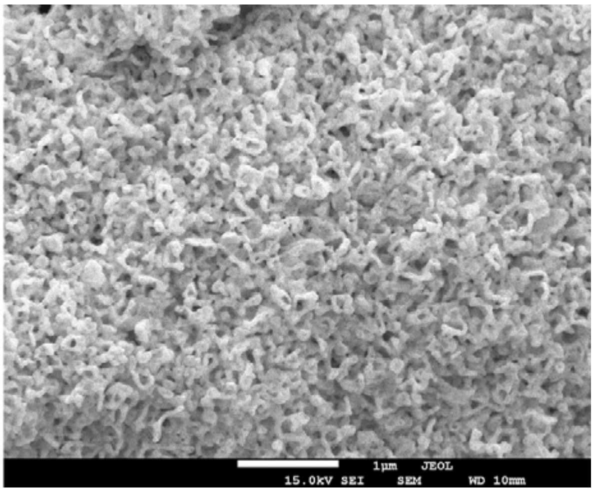

Fig.2. SEM micrograph of W sample with helium ion bombarding energy of 56.5 eV, a small ion flux of $1.0{\times}10^{20}$ $m^{-2}\cdot s^{-1}$, for a fairly long duration of 6 h 45 min at the temperature of 1000 K [16].

Figure 2 shows another example obtained in a magnetron sputtering device, where we can also distinguish many loop-like structures [16]. Similar observations were obtained for molybdenum surfaces irradiated by helium plasmas.

3. Modeling

From the above observations, it is thought that loop-like structure is a unit of structure for the growth of nano-fibers. Then, our growth model is proposed as shown schematically in Fig.3 [17]: ①accumulation of helium nano-bubbles in W bulk, ② holes formation due to arrival of thermally moving nano-bubbles to the surface, ③increase in surface area occupied by holes, ④connection of holes makes net works or mesh works, ⑤mesh or net works develop to be a kind of nano-walls, ⑥ growth of nano-walls and hole formations penetrating through wall surfaces, ⑦⑧growth of loops or arches, ⑨loop rapture or branching out from loops are possible candidate of further growth of nano-fibers

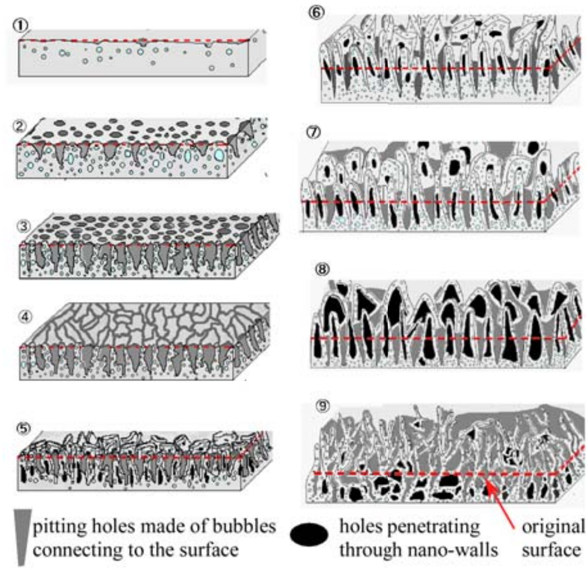

Fig.3. Modeling from bubble-made holes formation to the nano-fiber growth via loop-like structures.

Acknowledgement

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