Erosion behavior of actively cooled tungsten under H/He high heat flux load

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Abstract

We devised a method to measure erosion in the micrometer range on actively cooled tungsten samples. The method is based on applying micrometer-sized markers onto the side faces of the samples using a focused ion beam. With this method we measured the erosion of tungsten samples exposed to a hydrogen beam with 6% helium content. At 10.5 MW/m² the samples were exposed to particle fluences between $2 \cdot 10^{25}$ m^{-2} and $7 \cdot 10^{25}$ m^{-2}. Up to 630 pulses with a pulse duration of 30 s were employed. The actively cooled samples reach an equilibrium surface temperature which was varied from about 600°C to 2000°C. We find an amount of erosion which clearly exceeds the value computed from physical sputtering yields by roughly a factor of two. This is possibly correlated with the formation of a complex nanometer-sized morphology, which was observed for all temperatures.

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1. Introduction

The formation of nano-sized structures on tungsten surfaces as a consequence of irradiation with helium or hydrogen/helium mixtures has been reported from a number of experimental devices [1, 2, 3]. The question of how this nano structure formation may influence the erosion of such tungsten surfaces has been raised and investigated by spectroscopic erosion yield measurements in linear plasma devices using Argon impurities [4] or intense laser pulses [5] as well as in a tokamak edge plasma by exposure of pre-treated tungsten surfaces in the TEXTOR device [6].

Modifications of the erosion behavior of tungsten may have an influence on the component lifetime in a fusion reactor. Therefore an assessment of the influence of the nano structure formation on the erosion behavior under DEMO divertor relevant conditions is required. We have performed erosion measurements of actively cooled tungsten samples in the high heat flux device GLADIS [7,8] using a hydrogen beam with 6% helium content at a power density of 10 MW/m².

Measurements of the yield of physical sputtering are usually performed by measuring the weight change of small samples, the thickness change of thin films by ion beam analysis techniques, or by spectroscopic detection of the sputtered atomic flux [9]. In the case of light atoms impinging on tungsten surfaces the weight change and thickness change methods can be regarded as direct methods to analyze the amount of eroded tungsten. In the case of large actively cooled samples, however, both are not trivial to apply, because the relative change in thickness or weight is insignificant. To measure a thickness change in the micrometer range on a large-scale sample requires a micrometer-precise reference point. The usual method is partial shielding of the surface by applying a protective coating or an aperture. Both of these can, however, not be applied at high surface temperatures and high power densities.

We have devised a method to analyze the erosion-induced thickness change of centimeter-sized actively water-cooled tungsten samples. The method is based on placing markers onto the side faces of the samples by using a focused ion beam (FIB) device in combination with scanning electron microscopy (SEM). We have used this method to investigate tungsten erosion in the GLADIS device in the fluence range $3\text{-}7\cdot10^{25}$ m^{-2} at surface temperatures ranging from 600°C to 2000°C.

2. Sample preparation and experimental conditions

2.1 Sample preparation

Commercially available tungsten from Plansee SE with a purity of 99.97% was employed to manufacture the samples. The surfaces were mechanically polished.

The samples were prepared using a focused ion beam (FIB) in a dual beam FEI HELIOS NanoLab 600 machine with a maximum acceleration voltage of 30 kV, which combines a focused ion beam with scanning electron microscopy.

The first preparation step was Ga ion beam cutting a recess into a side face of each sample, i.e. a surface perpendicular to the surface to be heat loaded. This recess was subsequently smoothed and trenches were cut into the smoothed wall at a number of distances to the surface. The trenches were placed roughly 5, 10, 20, and 40 µm from the surface. The initial width of all trenches was 2.0 µm. Depending on the amount of erosion this allowed for a number of distance measurements to the remaining surface after exposure. In the vicinity of the marker trenches the top surfaces were ion beam polished.

Finally the distance from the respective trench edge to the polished surface was carefully analyzed for each marker trench by scanning electron microscopy. With this method a distance measurement accuracy of about 100 nm can be achieved. Figure 1 shows an SEM image of a ready-for-use tungsten sample together with a schematic sketch.

After these preparation and analysis steps the samples were investigated by confocal laser scanning microscopy (OLYMPUS LEXT OLS4000). This method yields 3D topographies of the samples' surfaces. The initial surface roughness of the samples was on the order of 200 nm. This was determined by computing mean square deviations from the average height level from the confocal laser scanning data.

Finally, the samples were brazed onto a copper heat sink for the actively cooled exposure to the GLADIS beam.

2.2 High heat flux loading in GLADIS

We employed samples with heights of 5, 10, 15, and 20 mm. A photograph of the complete component is shown in figure 2. Under steady state conditions these sample geometries yield surface temperatures of about 600°C, 1000°C, 1500°C, and 2000°C, respectively, at a calorimetrically measured central power density of 10.5 MW/m². Such a set of samples can be simultaneously exposed to the GLADIS neutral beam to investigate the temperature dependence of the erosion process. Three sets of four such samples were initially placed on the heat sink. To investigate the fluence dependence of the erosion process these sets were removed after reaching a pre-defined number of pulses.

The ion source was operated with a gas mixture of hydrogen and helium containing 10 atomic percent of helium at an extraction voltage of 31 kV. Since the discharge generates a specific composition of charged hydrogen species which are extracted from the source, this leads to a helium atomic fraction of 6% reaching the target. The hydrogen species distribution in the beam was determined from Hα Doppler spectroscopy [10]. The hydrogen/helium composition was determined according to the extraction-current-based procedure described in ref. [11].

In the GLADIS facility the heat sink was placed centered in the beam. Since the GLADIS beam has a Gaussian profile, the exposure conditions varied locally depending on the distance of the respective sample from the beam center. The local conditions for each sample set are given in table 1. Up to a total of 630 pulses were applied for the highest fluence. The pulse duration in each case was 30 s. For 2000°C surface temperature the samples reached an approximate equilibrium after about 10 s, at 600°C the duration was about 5 s. The sum of the pulse durations for the highest fluence was 18800 s. This means that the sample surface of the 2000°C sample with the highest fluence of $6.9 \cdot 10^{25}$ m^{-2} spent approximately 6300 s at this temperature.

After reaching the respective intended total particle fluence at normal incidence the heat sink was removed from the GLADIS facility and the respective samples were removed for SEM analysis of the remaining distance between marker trenches and sample surface. The heat sink was subsequently re-inserted into GLADIS and the remaining samples were further high heat flux loaded.

2.3 Post-exposure analysis

After the respective sample sets had been removed from the heat sink, they were analyzed by scanning electron microscopy. For each remaining marker trench its distance to the sample surface was carefully measured after the erosion process. Again this can be done with an accuracy in the range of 100 nm. Two to three such measurements were averaged to get the final result. Together with the initial measurement this yields the erosion loss in micrometers. An example for a sample after exposure is shown in figure 3.

As was done before exposure, the samples were again investigated by confocal laser scanning microscopy to obtain a 3D topography of the exposed surface.

Figure 4 shows an SEM image of a sample after loading (2000°C, $6.9 \cdot 10^{25} m^{-2}$). As can be seen, there are individual grains which display height levels varying from grain to grain. As our actual distance measurement of the height level change due to erosion can only be done at the location of the marker trenches, the height variation of the individual grains cannot be taken into account in this measurement. Therefore we have used the 3D topographic data of each sample after exposure to compute a mean square deviation from the average height level. In figure 5 this mean square deviation was used as a measure for the additional error of this analysis step.

The error bars shown in figure 5 include errors from the SEM image analysis before and after exposure as well as the mean square deviations from the average height level deduced from 640×640 µm² sized topographic images.

3. Results and discussion

With our sample setup it was possible to investigate the erosion behavior and surface morphology change of tungsten exposed to a 31 kV hydrogen beam with 6 atomic percent

of helium as a function of temperature and fluence for 4 different surface temperatures and in a fluence range of $1.7 \cdot 10^{25}$ m^{-2} to $6.9 \cdot 10^{25}$ m^{-2}.

The result of the erosion analysis explained in section 2 is shown in figure 5. It shows the difference of the measured distances of marker trenches from the surface before and after the GLADIS beam exposure. As explained before the data show averages of two to three such measured distances. These numbers give the erosion of the respective tungsten samples in µm. They are plotted for the four different surface temperatures versus the total particle fluence the respective sample has experienced.

As the beam composition and particle energies are known, the erosion due to physical sputtering can be computed from the published values of sputtering yields, see reference [9]. The fitted sputtering yield for helium at 31 keV given there is $3.5 \cdot 10^{-2}$. For the hydrogen species distribution an average energy of 17 keV per atom was calculated from the percentages given in reference [10] for a power density of 10 MW/m². For this hydrogen particle energy a fitted sputtering yield of $2.1 \cdot 10^{-3}$ was extracted from reference [9]. Since the fitted hydrogen erosion yield curve is rather flat in the relevant energy range, using individual yields for each energetic hydrogen species would affect this number only on the percent level. The erosion expected from this calculation is shown in figure 5 as a black solid line increasing linearly with fluence (see also table 1). From comparing our data with this prediction it can be concluded that the amount of erosion we observe exceeds the value computed from physical sputtering data significantly for temperatures of 1000°C and above.

As for a dependence of the observed erosion on the surface temperature we think that our data do show a trend indicating an increase of the erosion yield as function of temperature. This trend, however, does not exceed the measurement error and therefore cannot be regarded as confirmed. Again this is due to the relatively larger error bars.

Figure 4 shows an SEM image of the 2000°C sample after exposure to an erosion fluence of $6.9 \cdot 10^{25}$ m^{-2}. As can be seen the individual grains are of a size on the order of 100 µm. The image demonstrates qualitatively that each grain shows an individual height. This is possibly caused by the well-known crystal orientation dependent sputtering yield [12]. In our error estimates we have taken this into account by analyzing the 3D confocal laser scanning microscopy topographic images. An additional effect which may add some uncertainty to this error bar assignment procedure is the formation of a very complex surface morphology due to the particle bombardment. Figure 6 shows an example of a surface morphology of a sample after a fluence of $2.0 \cdot 10^{25}$ m^{-2} at a surface temperature of 2000°C. As can be seen the surface shows protrusions, the number and size of which is possibly also grain-orientation-dependent. It is not fully clear how much this affects the height level measurement by the confocal laser scanning microscope.

The formation of a complex surface structure containing µm-sized protrusion could be connected with an erosion loss of material in excess of the amount expected from physical sputtering. This could be simply due to fracture of protrusions by thermal shock due to the

large number of heat pulses. Other more complicated causes could be the angular dependence of the sputter yield or an influence of the impinging H/He flux on the W surface binding energy.

4. Summary

The measurement of micrometer-sized erosion patterns on actively cooled high heat flux samples represents a considerable challenge. We have developed and applied a method which allows such measurements. Our method is based on the application of a dual-beam machine consisting of a focused ion beam in combination with a scanning electron microscope. This machine is used to prepare and analyze marker trenches on the side faces of high heat flux tungsten samples prior to brazing them to the actively cooled heat sink.

We investigated the erosion behavior of tungsten exposed to a mixed hydrogen/helium beam in the high heat flux facility GLADIS. The investigated fluence range was $2\text{-}7 \cdot 10^{25}$ m^{-2} at a central power density of 10.5 MW/m². The samples were designed such that they reached peak surface temperatures in the range between about 600°C and 2000°C. For all samples we observed an erosion which exceeded the value computed from physical sputtering data. Due to the relatively larger error bars a clear temperature dependence of this effect could not be distinguished. The error estimate is based on a surface roughness analysis performed by confocal laser scanning microscopy. The roughness of the eroded sample surfaces exhibits a relatively high value due to orientation effects of individual grains. The formation of a nano-sized surface morphology on tungsten surfaces as a consequence of the irradiation with a mixed hydrogen/helium beam in the high heat flux facility GLADIS was described. This complex surface morphology could be connected with the observed excessive erosion.

Acknowledgement

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Figure Captions:

Figure 1: Top: Schematic representation of the respective orientations of the heat flux loading top surface, the recess in the front side and the marker trenches in the recess wall. Bottom: SEM image of a W sample after completion of preparation.

Figure 2: Photograph of the mock-up showing the copper heat sink with water connection pipes and the brazed tungsten samples on top.

Figure 3: SEM image of a sample after GLADIS exposure ($4.4 \cdot 10^{25}$ m^{-2}, 2000°C).

Figure 4: SEM image of the 2000°C sample after exposure ($6.9 \cdot 10^{25}$ m^{-2}).The typical grain size is on the order of 100 µm. The image qualitatively shows the height variation of individual grains.

Figure 5: Measured erosion in micrometers versus particle fluence for the four temperatures.

Figure 6: SEM image of a sample surface after exposure ($2.0 \cdot 10^{25}$ m^{-2}, 2000°C) showing the surface morphology with micrometer-sized protrusions.

Table 1: Local loading conditions of the samples showing the local power density, surface temperature, and particle flux, the three total exposure times as well as the local total fluence and the subsequently expected local tungsten erosion due to physical sputtering in micrometers. The number of pulses for the third fluence step was 630 with a pulse duration of 30 s.

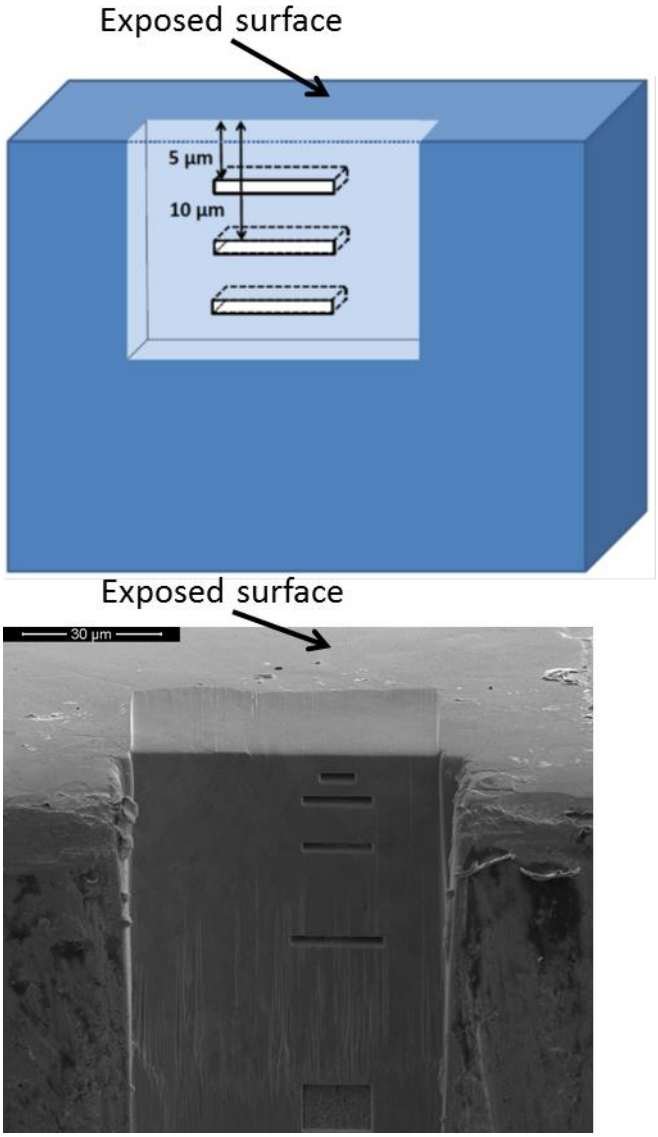

Figure 1

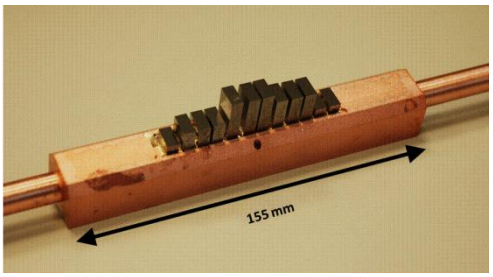

Figure 2

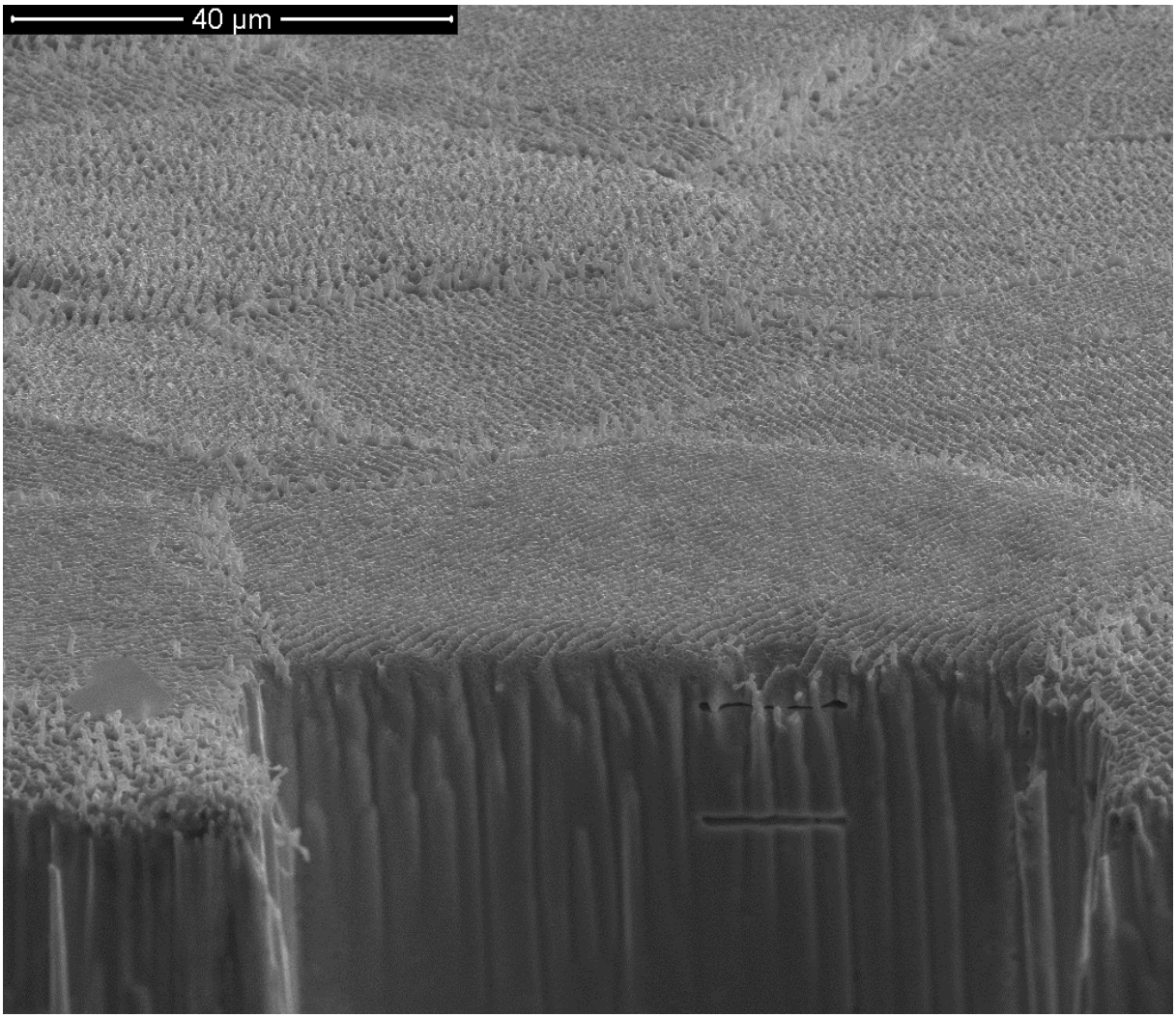

Figure 3

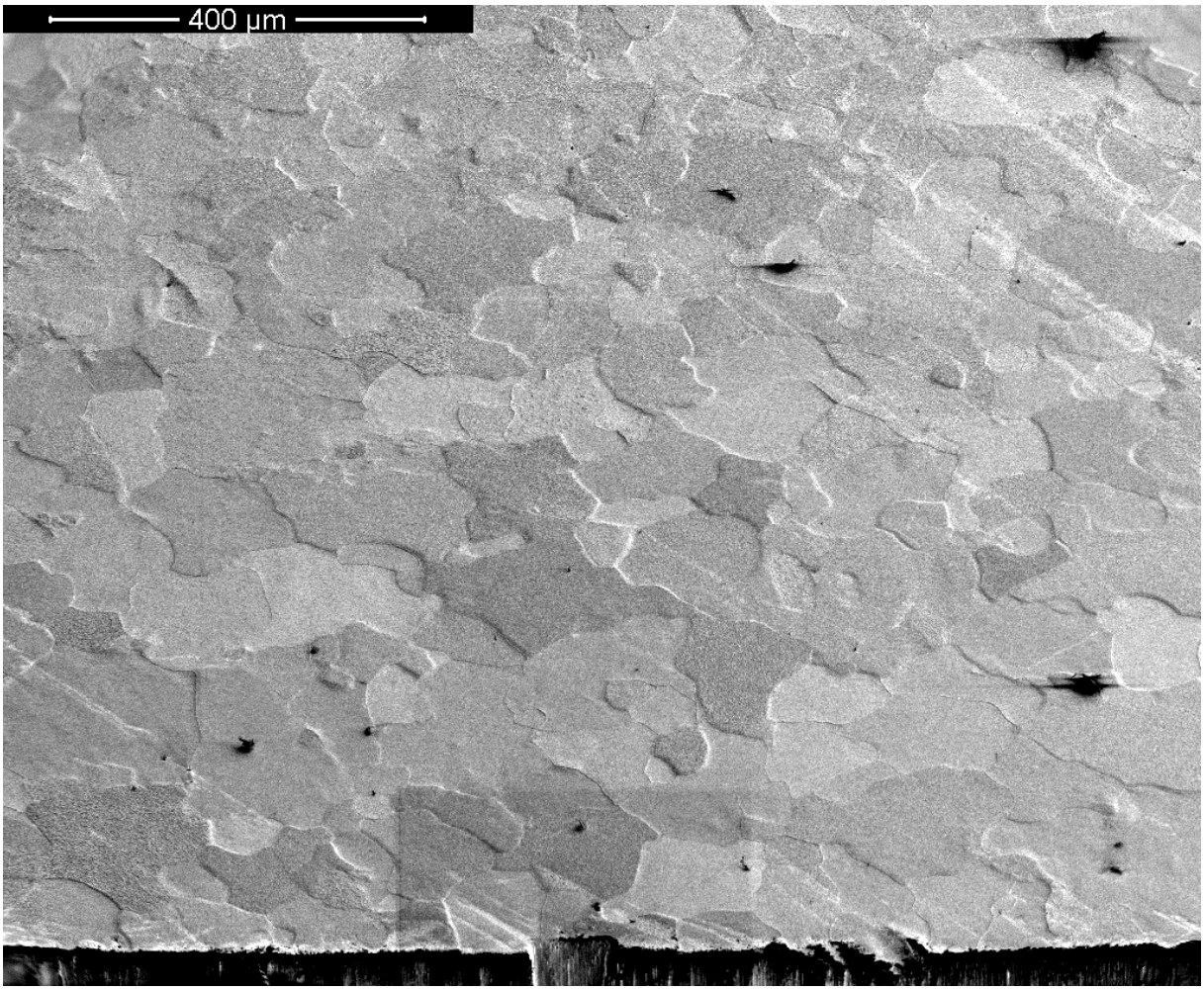

Figure 4

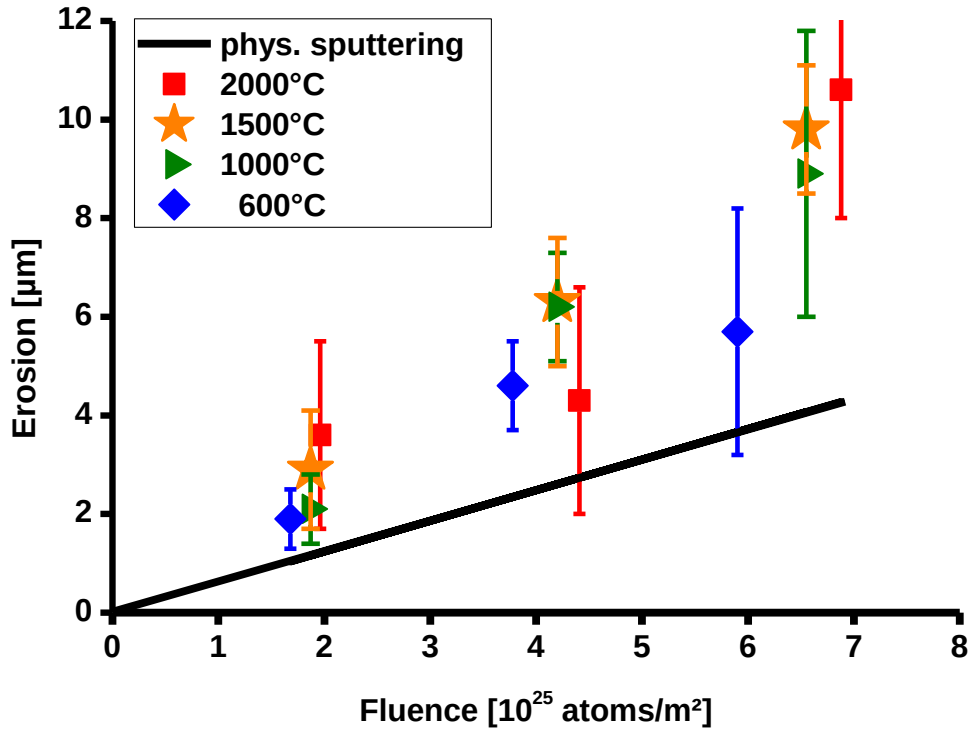
phys. sputtering
2000°C
1500°C
1000°C
600°C
Erosion [µm]
Fluence [10^25 atoms/m²]
0
2
4
6
8
10
12
0
1
2
3
4
5
6
7
8

Figure 5

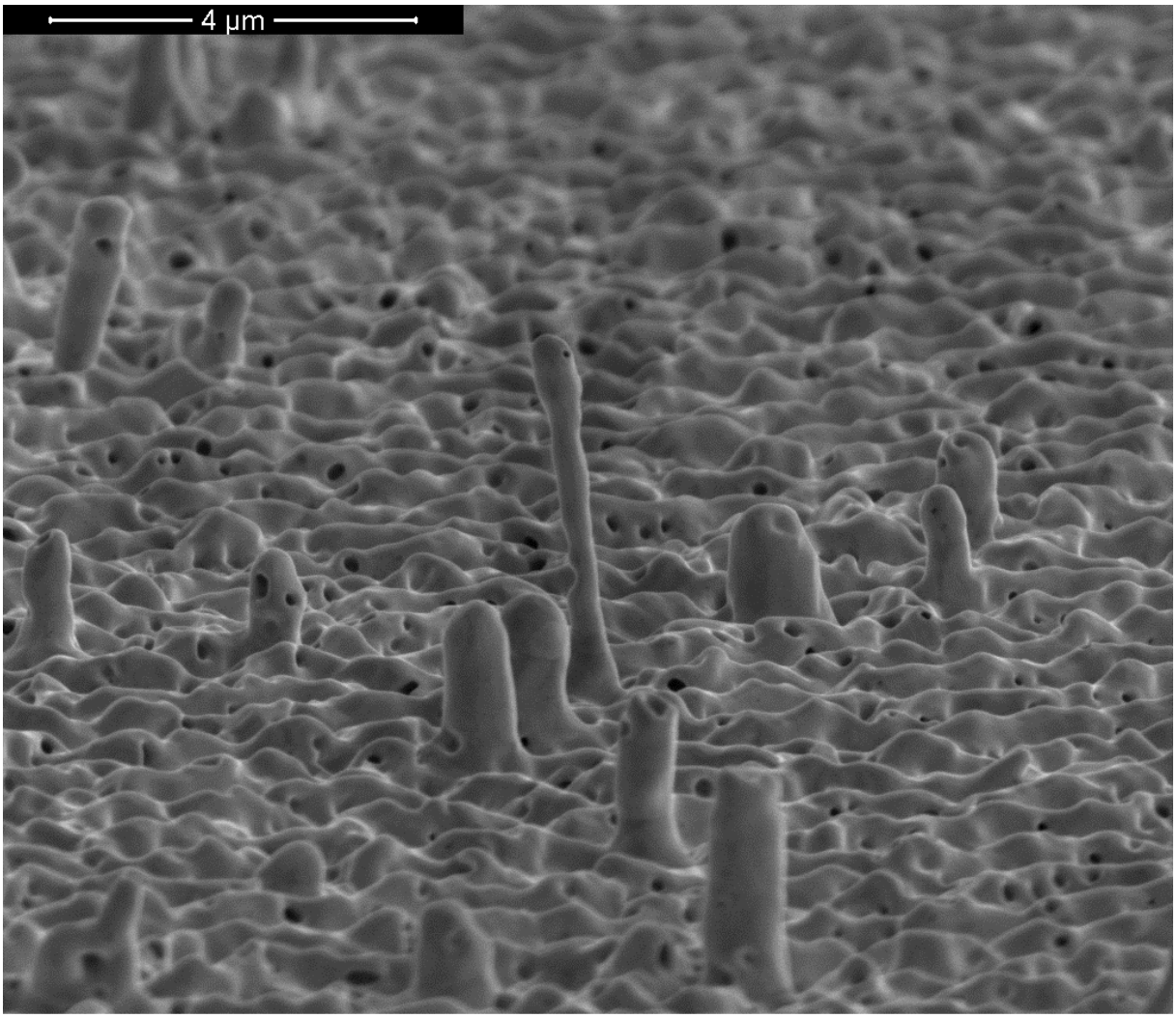

Figure 6

Table 1:

sample		**q'**	**T_{surf}**	**flux, calc.**	**exposure**	**fluence, F**	**Δs $W_{calc.}$**
		MW/m²	°C	$10^{21} m^{-2} s^{-1}$	s	$10^{25} m^{-2}$	µm
20 mm	1. fluence	10.5	2050	3.7	5.4E+03	2.0	1.2
	2. fluence	10.5	2050	3.7	1.2E+04	4.4	2.7
	3. fluence	10.5	2050	3.7	1.9E+04	6.9	4.3
15 mm	1. fluence	10	1470	3.5	5.4E+03	1.9	1.2
	2. fluence	10	1470	3.5	1.2E+04	4.2	2.6
	3. fluence	10	1470	3.5	1.9E+04	6.6	4.1
10 mm	1. fluence	10	1000	3.5	5.4E+03	1.9	1.2
	2. fluence	10	1000	3.5	1.2E+04	4.2	2.6
	3. fluence	10	1000	3.5	1.9E+04	6.6	4.1
5 mm	1. fluence	9	580	3.1	5.4E+03	1.7	1.0
	2. fluence	9	580	3.1	1.2E+04	3.8	2.4
	3. fluence	9	580	3.1	1.9E+04	5.9	3.7