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A Huber[1,3], G. Sergienko[1], M. Wirtz[1], I. Steudel[1], A. Arakcheev[2], S. Brezinsek[1], A. Burdakov[2], T. Dittmar[1], H. G. Esser[1], A. Kreter[1], J. Linke[1], Ch. Linsmeier[1], Ph. Mertens[1], S. Möller[1], V. Philipps[1], G. Pintsuk[1], M. Reinhart[1], B. Schweer[1], A. Terra[1], B. Unterberg[1]

[1]Forschungszentrum Jülich GmbH, Institut für Energie- und Klimaforschung - Plasmaphysik, 52425 Jülich, Germany,

[2]Budker Institute of Nuclear Physics (BINP), Novosibirsk 630090, Russia

E-mail: A.Huber@fz-juelich.de

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Abstract:

The impact of simultaneous exposure of tungsten to a steady-state plasma and transient cyclic heat loads on the deuterium retention has been studied in the linear PSI-2 facility with the main objective of qualifying tungsten (W) as plasma-facing material .The transient heat loads were applied by a high-energy laser, a Nd:YAG laser (λ=1064 nm) with an energy per pulse of up to 32J and a duration of 1ms.

A pronounced increase in the D retention by a factor of 13 has been observed during the simultaneous transient heat loads and plasma exposure. These data indicate that the hydrogen clustering is enhanced by the thermal shock exposures, as seen on the increased blister size due to mobilisation and thermal production of defects during transients. In addition, the significant increase of the D retention during the simultaneous loads could be explained by an increased diffusion of D atoms into the W material due to strong temperature gradients during the laser pulse exposure and to an increased mobility of D atoms along the shock-induced cracks. Only 24% of the retained deuterium is located inside the near-surface layer (d<4μm).

Enhanced blister formation has been observed under combined loading conditions at power densities close to the threshold for damaging. Blisters are not mainly responsible for the pronounced increase of the D retention.

Keywords: transient heat loads, deuterium retention, blistering, surface modifications, tungsten

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[3] Corresponding author: tel.: +44 1235 464809, e-mail: A.Huber@fz-juelich.de

1. Introduction

High power loads during intense transient events such as Edge Localised Modes (ELMs) could pose a severe threat causing surface modifications or even melting of plasma-facing components (PFC) in fusion plants like ITER and DEMO. In addition to thermal shock events, PFCs in future fusion devices will be exposed to a high flux low-energy steady-state plasma. Under combined loading conditions, synergistic effects might lead to increased material damage during transients as well as to significant impact on fuel retention in the PFCs.

Tungsten (W) has been considered to be one of the most important candidates for divertor material in ITER because of its excellent material properties such as a high threshold energy for sputtering, a high melting point and a low tritium inventory [1]. Therefore, a detailed study of the synergistic effects of these combined loading conditions on the damage behaviour of tungsten is necessary to qualify tungsten as PFM.

In this contribution, we report on the first experiments investigating the effect of the combined loading conditions on the surface modification of bulk tungsten as well as on the fuel retention.

2. Experimental set-up

The linear plasma device PSI-2 [2] (figure 1), which generates a deuterium plasma with flux densities up to 10^{23} m^{-2} s^{-1} using a LaB_6 heated cathode and a Mo hollow anode, has been used for successive as well as simultaneous ELM simulation experiments where the transient heat loads were applied by a high energy laser during the steady-state plasma operation. The plasma column has a hollow density and temperature profile with a diameter of 6 cm confined by a 0.1 T axial magnetic field. The electron density and temperature of the PSI-2 plasma were measured with a Langmuir probe.

Targets were mounted on a water cooled target stage and aligned properly with the maximum of the plasma profile. The temperature of the samples was measured by a chromel-alumel thermocouple that was connected to the rear part of the sample. In addition, a fast pyrometer was used to measure the surface temperature of the samples. PSI-2 is operated under steady-state conditions; therefore the required particle fluence was accumulated in a single plasma exposure. Ions are accelerated to the sample surface by biasing the sample to produce 60 eV ion energy at a flux of about 6×10^{21} m^{-2} s^{-1}.

The transient heat loads were applied by a high-energy laser, a Nd:YAG laser (λ = 1064 nm) with an energy per pulse of up to 32 J and a duration of 1 ms (figure 1a). Laser pulses at the frequency of 0.5Hz were utilized in the current contribution. The laser was operated in the free generation mode (without Q-switching). The beam of the fibre-coupled Nd:YAG laser was introduced through a window in the vacuum chamber under angle of 38º to the target surface and imaged by two lenses on the spot of ~3.1 mm diameter (spot area around 7.5 mm^2). The power deposition profile in the irradiated area was nearly uniform (see figure 1c) to avoid the instrumental influence on the cracking behaviour. To keep the optical losses as small as possible, dielectric mirrors were used in the optical arrangement. The steering of the location of the laser spot was done by adjustment of the last mirror just in front of the vacuum window. In the presented experiments the samples have been exposed to ELM relevant power densities between 0.19 GW/m^2 and 0.90 GW/m^2. These values take into account the partial reflection of the laser energy from the sample. The laser pulse waveform was measured by a fast photodiode located behind the last dielectric mirror, as shown in figure 1b, demonstrating the nearly constant power over the pulse duration as well as the smooth temporal behaviour (no spikes which could lead to material ablation).

Samples used in these studies were cut by wire (electrical discharge machining − EDM) from tungsten blocks to the dimensions 12 × 12 × 5 mm^3. The material used was tungsten, which fulfil the ITER-grade specification and was manufactured by Plansee SE [3], with a purity of 99.5 wt%. It was produced by powder metallurgical routes (cold iso-static pressing of homogenised powder for the green compact (resulting pressed part), subsequent sintering at 2000 – 2500°C) and deformed by forging or rolling of the sintered blanks into rod-like geometries. The deformation processes induce preferential grain orientations. The grains of the tungsten specimens were oriented perpendicularly (transversal) to the loaded surface with a typical grain length around 110 µm and width of about 40 µm. Each sample is castellated (see Fig.1e), i.e. composed of four sections separated by narrow grooves (~ 0.3 mm). This castellation allows easy splitting of the sample into 4 pieces without increase of their temperatures, keeping the D-inventory unchanged. Each of the pieces contains only a single laser spot (or none of the laser spots if it is used as reference).

The experiment is carried out as follows. Firstly, only an area of piece 1 was exposed to laser pulses. After that, all four pieces were exposed to the plasma with simultaneous ELM-like transient heat loads by laser onto piece 2. The reference pieces 3 and 4 are located on the same plasma radius as pieces 2 and 1 and were correspondingly exposed to the same ion flux.

Before exposure, the surface of all samples was polished to a mirror finish with a roughness R_a of less than 0.1 µm. After laser irradiation, the samples were analysed by optical metallographic microscopy, scanning electron microscopy (SEM) and laser profilometry. In addition, the amount of D retained in the samples was measured by nuclear reaction analysis (NRA) with help of the $D(^3He,\alpha)p$ nuclear reaction on the laser spot locations and corresponding reference locations. The ^{3}He 3MeV beam of NRA has the diameter of about 1 mm and was positioned at the centre of the laser spot. Finally, the target was split in 4 pieces after which the deuterium content in each piece was measured with help of thermal desorption spectroscopy (TDS). Normal slow temperature ramps ~0.35 K/s were applied up to 1273 K. To compare local NRA measurements and total number of the desorbed D atoms from the pieces provided measured by TDS, the surface density of D atoms [C_D] inside the laser spot has been evaluated from the TDS data (applied to the laser exposed and the reference (only exposed to D-plasma) pieces) by the following equation:

$$C_D[at/m^2] = \left(D^{load} - (S_0 - S_{load}) \times \tfrac{D}{S_0}^{ref}\right)$$

where D^{load} and D^{ref} - the total amount of deuterium desorbed by TDS respectively from laser loaded and reference pieces, S_0 and S_{load} - the piece area exposed to the plasma flux as well as the area of the laser spot.

3. Result and Discussion

3.1 *Surface morphology of tungsten after combined deuterium plasma and transient heat load exposure*

Surface modifications were investigated with a scanning electron microscope (SEM) at the sample which coincides with the maximum of plasma flux density. Figure 2 shows SEM images taken after simultaneous exposure to transient heat loads and to D plasma (a,b) and after successive exposure of tungsten to transient heat loads and deuterium plasma (c) as well as after pure D loading (d) at PSI-2 at a fluence of 1.2×10^{25} m^{-2} (ion fluxes of 6.0×10^{21} m^{-2} s^{-1} for a time period of 2000s) and incident ion energy of 60 eV. Here the laser power density was 0.38 GW/m^2 at pulse duration of 1ms and the number of applied pulses amounted to 1000 at a repetition rate of 0.5Hz. The base temperature of the W samples before thermal shock exposure was around room temperature (RT), i.e. below the ductile-to-brittle transition temperature (DBTT) of ~ 500-700 K [4]. With the repetition rate used for these tests of 0.5 Hz and with an actively cooled target holder the base temperature of the test samples increased by 100 K at most.

The intrinsic defects in the material such as dislocations and vacancies could act as traps for deuterium and lead to bubble nucleation [5-7], so-called blisters. Enhanced blister formation has been observed in the experiment with simultaneous laser beam and deuterium loading. In this experiment the blister size amounts up to 2μm (see Fig. 2 a,b) whereas they are smaller (0.3 μm) (Fig.2c) at the successive exposure of tungsten to transient heat loads and to the deuterium plasma. Very little blister formation and much smaller sizes (Fig.2d), has been observed for samples which were exposure to the pure plasma loading. The blister formation has been verified by EDX measurements to exclude the deposition of material droplets on the surface and by FIB cross sections, which clearly showed the formation of small cavities below the surface [8].

The blisters are irregularly distributed over the sample surface with a tendency to form groups. The observed blisters with sizes much smaller than the width of the grain (40μm) appear with a large number density on certain grains whereas on others they are completely absent. This behaviour has been observed in the previous experiment [9] and this is probably related to the crystallographic orientation [10]. Blistering at tungsten surfaces may result in an increasing fuel inventory in the near-surface of plasma-facing components. Later we will show that only 24% of the retained deuterium is located inside the near-surface layer (d<4μm) and blisters are not manly responsible for the pronounced increase of the D retention. These results are fully consistent with the previous observation in [11] reporting that only 3-5% of the total D amount is retained in the blisters.

SEM images of a ITER-like tungsten exposed each to 1000 laser pulses for a power density range between 0.19 GW/m^2 and 0.86 GW/m^2 are shown in Fig. 3a,b,c. Previous investigations show that the ITER-grade W sample exhibits a damage threshold between 0.38 GW/m^2 and 0.76 GW/m^2 for 1000 heat pulses [12]. The sample exposure to power of 0.86 GW/m^2 exhibits the crack formation. These experiments have been performed under simultaneous laser beam and deuterium loading. Blisters of larger sizes (up to 2.0μm) have been observed during the laser exposure at powers around the damage threshold 0.38-0.76 GW/m^2. The exposures with lower and higher power densities show formation of the blisters with much smaller sizes as well with the reduced density numbers.

Blister formation has been observed during the simultaneous laser and plasma exposure of the longitudinal as well as the transversal W samples (see Fig3 d,e,f). Here the power density of the 0.38 GW/m^2 has been chosen. The longitudinal sample exhibits blister formation of the sizes around 2 μm. Despite the power density below the threshold for the damaging, thermal shock crack networks as well as surface roughening are formed on the blister surface (Fig.3f). Gas pressure inside of the blisters generates stresses in the material. Due to these additional stresses, the critical stresses for crack formation and propagation are lowered.

3.2 The impact of the transient heat cycling loads on the fuel retention in tungsten

Figure 4 summarized the results of the TDS and NRA analyses. In these experiments, the laser pulse length was 1 ms with a deposited energy density of 0.38 MJ m^{-2}. The number of applied pulses amounts to 1000 at a repetition rate of 0.5 Hz. The base temperature of the W samples before thermal shock as well as before plasma exposure was around RT. It increases continuously with loading exposure time and reaches the value of 130ºC after 1000 laser pulses**.** The samples here have been exposed to deuterium ion fluxes of 6.0×10^{21} m^{-2} s^{-1} for a time period of 2000s (a fluence of 1.2×10^{25} m^{-2}).

The amount of D retained (figure 4a) in the samples was measured by nuclear reaction analysis on the laser spot locations at two ^{3}He incident energies limiting the measurement depth for the NRA analysis to about of 0.5μm and 4μm correspondingly. Figure 4a shows that, under simultaneous loading conditions, synergistic effects lead to a significant impact on fuel retention in the PFCs. In contrast to the pure plasma loading the combined plasma and laser exposure shows maximal D-content stored in 4 μm near-surface layer: a) 6×10^{16}at/cm² (spot2) with combined loading versus 1.74×10^{16}at/cm² (spot3) for the pure plasma loading. The reference spot 3 (4) is located on the same plasma radius as spot 2 (1) and, correspondingly, has the same ion flux.

The TDS confirm the pronounced increase of the D retention by a factor of 13 during the combined transient heat loads and plasma exposure. The D-atoms concentration during the successive exposure to transient heat loads and deuterium plasma is also increased and it is more than a factor of 4 larger than the pure plasma loading. The deviation of the TDS and the NRA results for successive and combined exposure loadings indicating a deeper penetration of the D atoms in the W material. In contrast to the TDS method, the measurement depth for the NRA analysis is limited in this experiment to maximum depth of about of 4μm.

Figure 4b shows the D-content measured by TDS analysis as function of the D-content registered by NRA method. The deuterium content measured by two methods demonstrates the linear dependence for successive and combined exposure loadings as well as for the pure plasma exposure experiments. Assuming that the entire D-inventory for the pure plasma loading is located inside 4μm near-surface layer, both analyses should show the same result. However, a systematic deviation by 30% (slope = 0.696) of the NRA and TDS results has been observed. Taking into account this systematic deviations we could evaluate the fraction of the deuterium stored in the 4μm near-surface layer ($1/(2.95/0.696) \approx 0.24$) which is about 24%.

These data indicate that the hydrogen clustering is enhanced by the thermal shock exposures, as seen on the increased blister size due to mobilisation and thermal production of defects during transients. In addition, the significant increase of the D retention during the simultaneous loads could be explained by an increased diffusion of D atoms into the W material due to strong temperature gradients during the laser pulse exposure ($T_{surf} \approx 1300$ K at 0.3 GW m^{-2}) and to an increased mobility of D atoms along the shock-induced cracks.

Conclusions

The impact of the repetitive ITER-like ELM heat pulses in combination with steady-state plasma exposure in the PSI-2 facility has been studied in recent experiments with the main objective of qualifying tungsten (W) as plasma-facing material. The observed results show that the damage behaviour as well as the deuterium retention strongly depend on the loading conditions and the sequence of the particle and heat flux exposure.

Pronounced blister formation with a size of ≈2μm has been observed in the experiment with simultaneous laser beam and D loading. In contrast to the combined loadings, the successive exposure of tungsten to transient heat loads and to deuterium plasma demonstrates blister with lower density as well as with smaller sizes (≈ 0.3 μm). Blisters are not the main responsible mechanism for the pronounced increase of the D retention.

The effect of surface modification (or plastic deformation) on the deuterium retention in tungsten exposed to a high-flux, low-energy plasma was studied by thermal desorption spectroscopy and nuclear reaction analysis. A pronounced increase in the D retention (more than a factor of 13) has been observed during the combined transient heat loads and plasma exposure. These data indicate that the hydrogen clustering is enhanced by the thermal shock exposures, as seen on the increased blister size due to mobilisation and thermal production of defects during transients. In addition, the significant increase of the D retention during the combined (plasma and laser exposure) loads could be explained by an increased diffusion of D-atoms into the W material due to strong temperature gradients during the laser pulse exposure ($T_{surf} \approx$ 1300K at 0.3 GW m^{-2}) and to an increased mobility of D atoms along the shock-induced cracks. Combined measurements by NRA and TDS of the amount of D retained in the samples shows that only 24% of D is stored in 4 μm near-surface layer in tungsten after transient heat load cycling.

Acknowledgements

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Figure captions:

Figure 1 Schema of the laser irradiation setup with laser pulse waveform, spatial distribution and surface temperature evolution during the laser pulse.

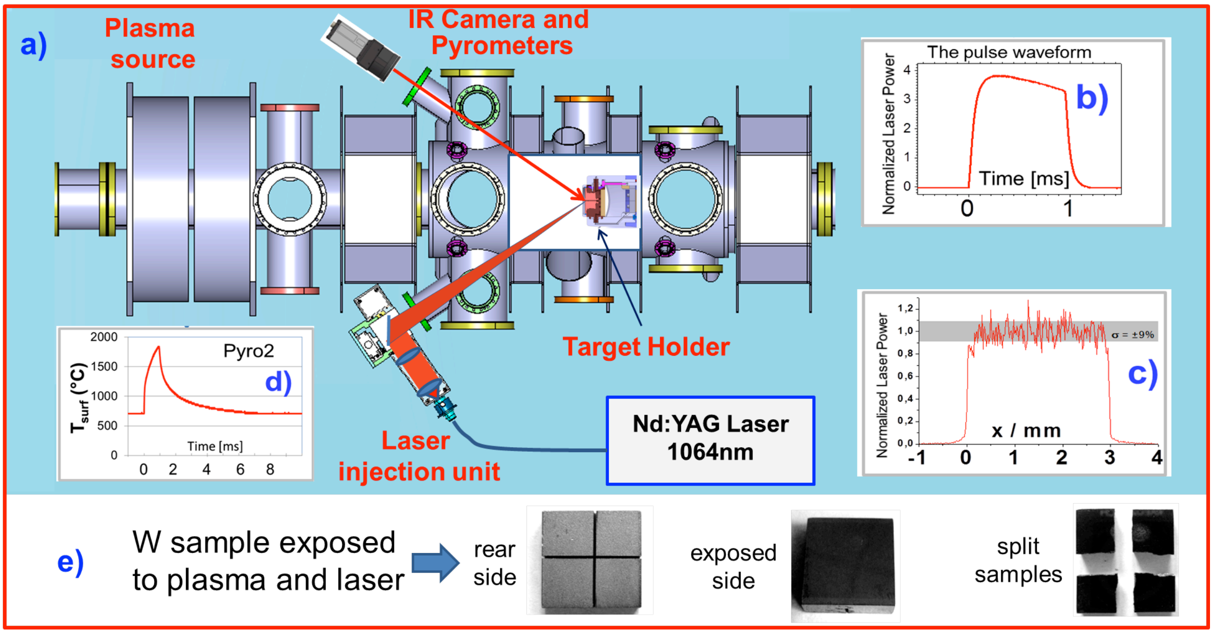
Figure 2 SEM images taken after simultaneous exposure to transient heat loads and to D plasma (a,b) and after successive exposure of tungsten to transient heat loads and deuterium plasma (c) as well as after pure D loading (d) at PSI-2 at a fluence of 1.2×10^{25} m^{-2} (ion fluxes of 6.0×10^{21} m^{-2} s^{-1} for a time period of 2000s) and incident ion energy of 60 eV. The absorbed laser power density is 0.387GW/m^2

Figure 3 SEM images of an ITER-like tungsten exposed each to 1000 laser pulses for a power density range between 0.19 GW/m^2 and 0.86 GW/m^2 (top row). SEM images of the induced surface damage of W samples with transversal and longitudinal grain orientation after exposure with ELM like events at absorbed power of 0.38 GW/m^2 (bottom row).

Figure 4 Nuclear reaction analyses on the laser spot locations at two ^{3}He incident energies (top row). Total D-inventory measured by TDS analysis as function of the D-content in the near-surface layer delivered by NRA method (bottom row).

Figures:

Figure 1



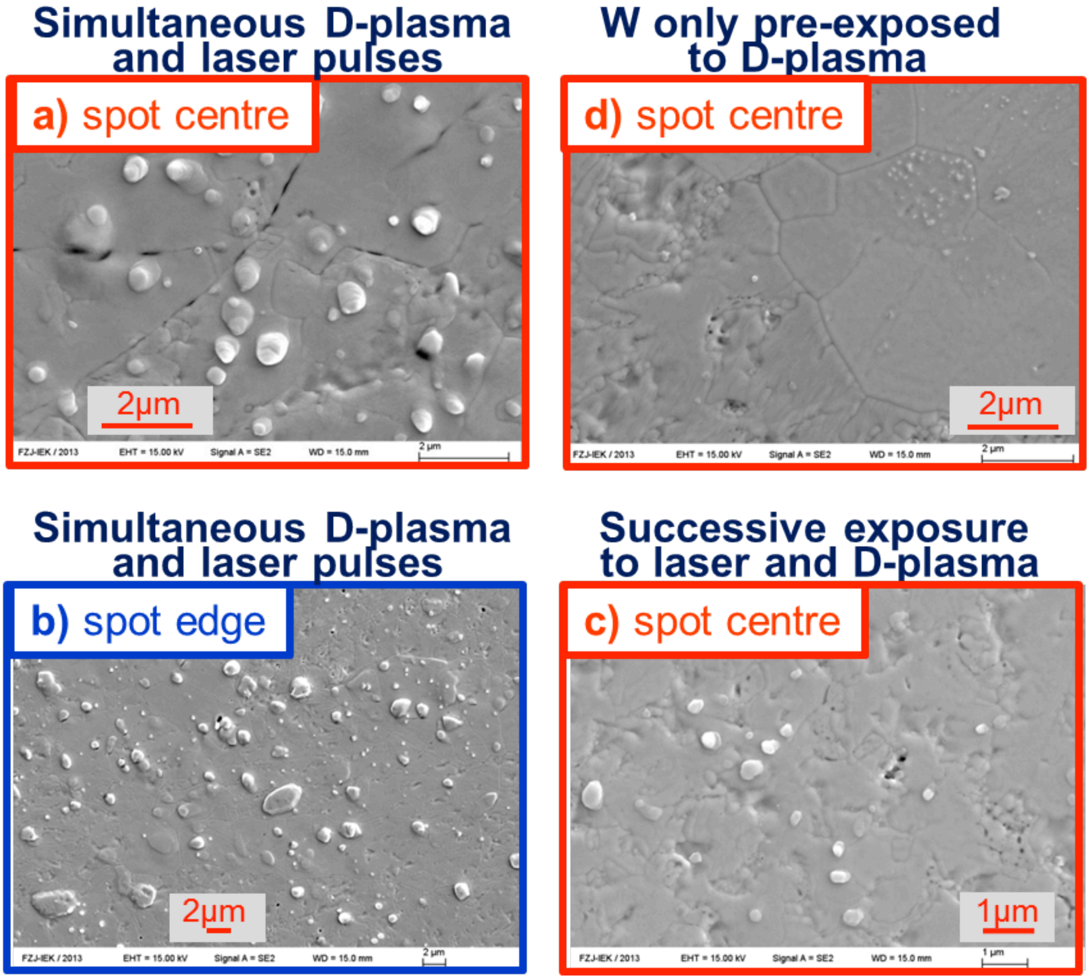

Simultaneous D-plasma and laser pulses
a) spot centre
2µm
FZJ-IEK / 2013 EHT = 15.00 kV Signal A = SE2 WD = 15.0 mm 2 µm
W only pre-exposed to D-plasma
d) spot centre
2µm
FZJ-IEK / 2013 EHT = 15.00 kV Signal A = SE2 WD = 15.0 mm 2 µm
Simultaneous D-plasma and laser pulses
b) spot edge
2µm
FZJ-IEK / 2013 EHT = 15.00 kV Signal A = SE2 WD = 15.0 mm 2 µm
Successive exposure to laser and D-plasma
c) spot centre
1µm
FZJ-IEK / 2013 EHT = 15.00 kV Signal A = SE2 WD = 15.0 mm 1 µm

Figure 2

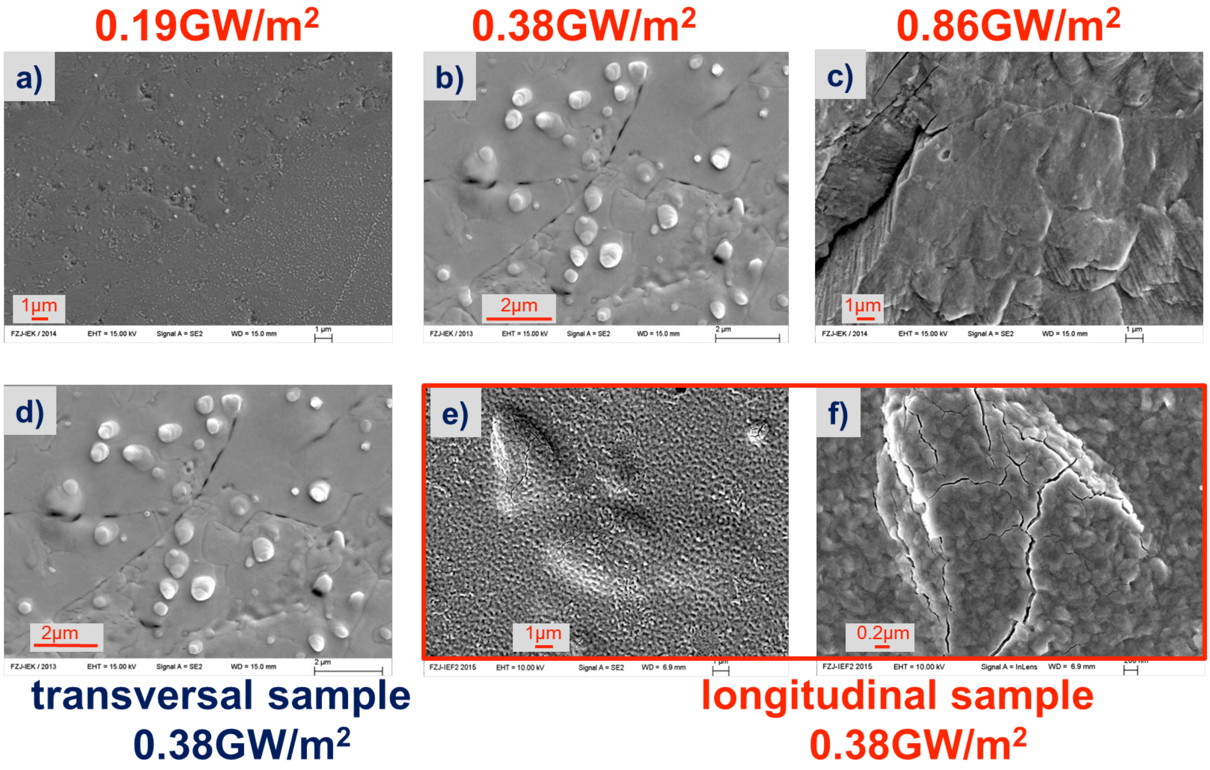

0.19GW/m²
0.38GW/m²
0.86GW/m²
a)
1µm
b)
2µm
c)
1µm
d)
2µm
e)
1µm
f)
0.2µm
transversal sample
0.38GW/m²
longitudinal sample
0.38GW/m²

Figure 3

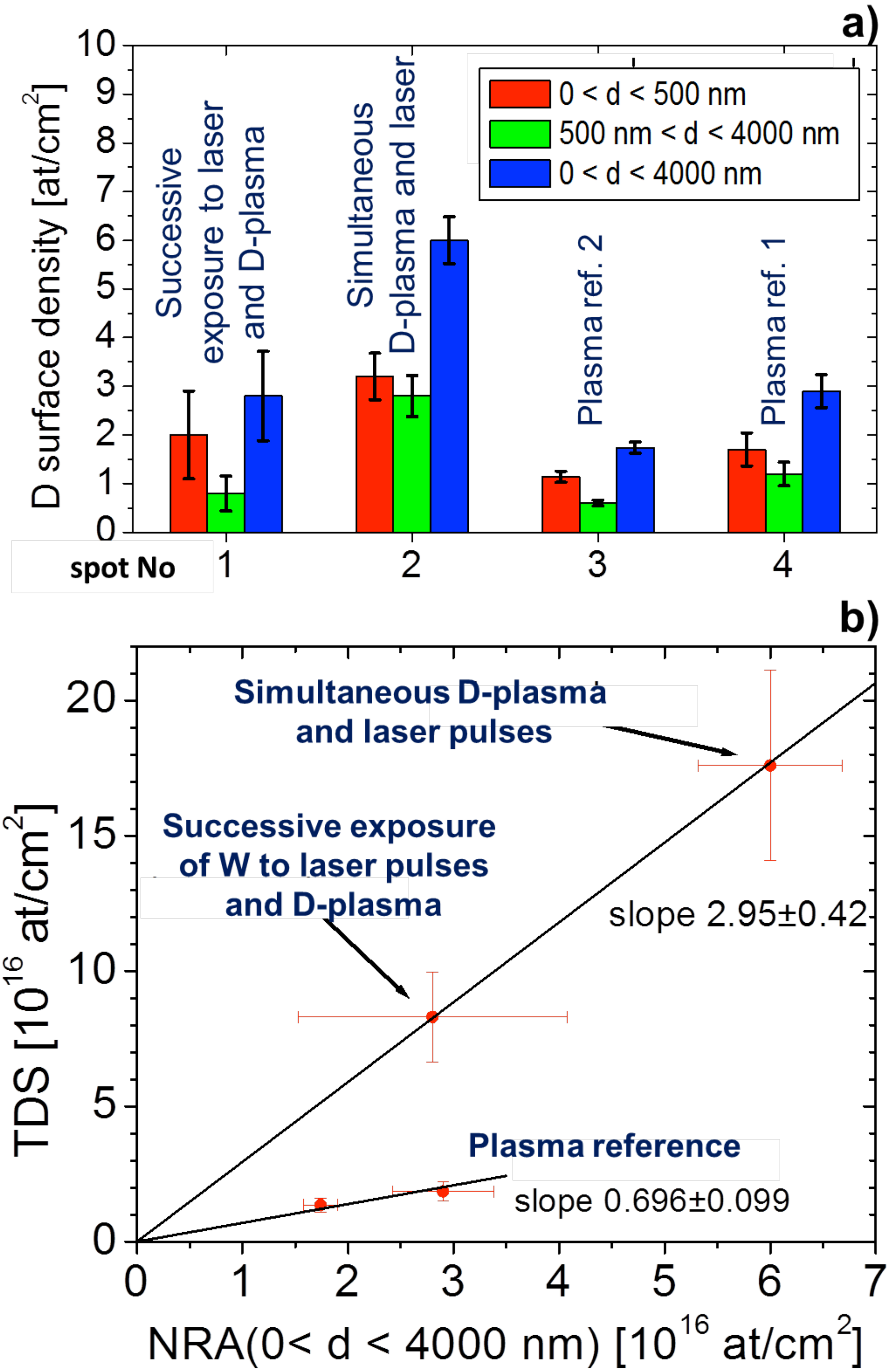

a)
D surface density [at/cm^2]
10
9
8
7
6
5
4
3
2
1
0
spot No
1
2
3
4
0 < d < 500 nm
500 nm < d < 4000 nm
0 < d < 4000 nm
Successive exposure to laser and D-plasma
Simultaneous D-plasma and laser
Plasma ref. 2
Plasma ref. 1
b)
TDS [10^{16} at/cm^2]
20
15
10
5
0
NRA(0< d < 4000 nm) [10^{16} at/cm^2]
0
1
2
3
4
5
6
7
Simultaneous D-plasma and laser pulses
Successive exposure of W to laser pulses and D-plasma
slope 2.95±0.42
Plasma reference
slope 0.696±0.099

Figure 4