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Investigation of radiation effect on tungsten and molybdenum materials

Abstract. This work studied damage and erosion of the tungsten and molybdenum after exposure to deuterium ions of a Plasma focus device. The main purpose and idea of this research work was an experimental investigation of surface changes on the tungsten and molybdenum materials at deuterium optimal gas pressure in the chamber of a PF device. Before and after irradiation the sample surfaces were analyzed using SEM, AFM and an optical microscope. The analysis showed that the change in surface relieves 0.0377 – 0,0125 µm, different types of damage on materials dependent on irradiation parameters. With increasing the 10-30 number of shots, the micro-cracks 74-172 nm, and radiation swelling for molybdenum, also the crater, bubbles, holes 64-453 nm and cavities on the tungsten sample surface were observed respectively. It is established that a noticeable erosion for tungsten up to Δm=-31 mg and molybdenum Δm=-43 mg samples as a result of irradiation with plasma flows simulating a stationary regime occurs only at relatively high sample surface temperatures.
Key words: radiation damage, irradiation surface, defects, optimal gas pressure, plasma focus device

Introduction

One of the most important tasks of plasma physics and physics of thermonuclear reactors [1] is to study the resistance of materials of the first wall, the divertor and other nodes of the thermonuclear reactor to stationary plasma-thermal effects with a capacity of up to 20 mW/m^2 and intense pulsed duration of 0.1–10 ms and a power of 1–10 GW/m^2 [1–3]. Many of the basic problems were investigated and solved but one of the main problems still not enough investigated is connected with material science and radiation nuclear physics [3, 4]. They are long-lasting irradiation and heat loads that are generated in the fusion devices that affect the construction materials and the appearance of defects on the surface materials. After long-lasting radiations, it leads to erosion of the protective coatings of the diverter and the first wall of the thermonuclear reactor [4-6].

According to experimental [5] and theoretical [6] studies, the final result of the interaction of intense plasma flows with matter is influenced by many factors. First of all, these are the parameters of the plasma stream itself: the plasma-forming substance, the velocity of the plasma stream, its density, duration and temperature. Physical properties of the materials: melting point, heat capacity and thermal conductivity. Moreover, the final result of such interaction is influenced by the initial relief of the surface materials. It should be noted that the parameters of the plasma stream afford to determine the type of defects and phenomena observed on the materials.

Nowadays, the study effect of nuclear-physical radiation on materials attracts the attention of many researchers [2-6]. It is afforded to study the fundamental laws under the thermal and radiating interaction of energetic neutrons and ions emanating from the hot intensive plasma with materials with different structures [7,8]. The studies of phenomena related to changes in the structure of the surface on the materials of the first wall and the divertor of thermonuclear installations under the influence of plasma beams have great importance [8]. Influence by plasma radiation, damage to the first wall materials, is characterized as sputtering, evaporation, mass loss, blistering, and other structural degradation [7-9].

Tungsten and molybdenum are the most perspective materials as plasma-facing materials [PFM] in future reactors with complex unique physical properties. Tungsten's physical properties are low physical sputtering coefficient, high melting point, and high thermal conductivity. Due to such properties, tungsten intends to be used as a first-wall PFM of ITER. [5, 9]. Also, molybdenum has a high melting point and high reflectivity retention after sputtering considered a candidate for the first mirror PFM [8, 9]. However, after long irradiation in nuclear fusion reactors, surface tungsten and molybdenum materials degrade their quality. Nowadays, experimentally investigating the modification and damage of these materials influenced by impulse plasma is very important. However, the study of these problems under fusion reactor conditions is a difficult task. Currently not available to researchers specific radiation environments as a first wall fusion reactor.

The Plasma Focus device (PF) is suitable for studying first-wall thermonuclear reactors and their related material. A plasma focus (PF)device is a source that can produce hot dense plasma, X-ray, high energy ions, electrons and neutrons (when operated on deuterium gas) [5, 8, 11]. Also, is widely applied to investigate materials irradiated to hot plasma and nanotechnology [8, 11].

Currently, a study combined the effects of thermal and plasma irradiation on materials successfully implemented on plasma focus devices [19, 20]. It was shown [21, 22] that simultaneously exposure to plasma and high thermal loads can increase the damage to the surface of the materials.

Tungsten's damage effect was investigated by Saw et all [22] in a 2.2 kJ plasma focus (PF) device at different distances using a deuterium ion flux of 10^{28} $m^{-2}s^{-1}$. They reported that after irradiation, uniform cracks with a width of 300–500 µm and cavities up to 5 µm were observed. Dutta et al. [23] studied tungsten samples using a 2.6 kJ PF device at different angles in relation to the electrode and irradiated with helium ions in 10 shots. In the results, they observed surface modification and shift to high angles of tungsten crystal lattice. Erosion on tungsten samples using a low-energy plasma focus device was investigated by Bhuyan et al. [24]. They placed samples at different angles to the anode and irradiated by hydrogen 20 shots. After exposure cracks, blisters and melting were observed on the tungsten surface. Also, R. Niranjan et al. [25] investigated materials for the construction of fusion reactors (tungsten, nickel, stainless steels, molybdenum and copper) using a plasma focus device. The materials were exposed to fusion plasma without changing the distance between the anode and the targets. In results defined that narrow cracks were extended to deep and larger cracks with increasing exposed shots.

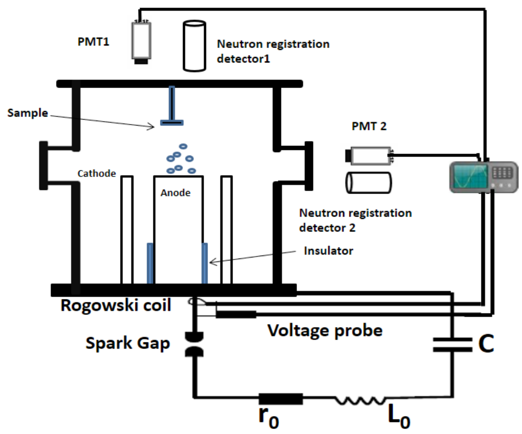
Based on these reviews, the main goal of this research work is to experimentally investigate the features of erosion and surface damage to tungsten and molybdenum samples at optimal gas pressure and under controlled conditions as the most suitable materials for a fusion reactor. The total irradiation dose is controlled by the number of irradiation exposures of the sample to the PF discharges.

Methods and Materials

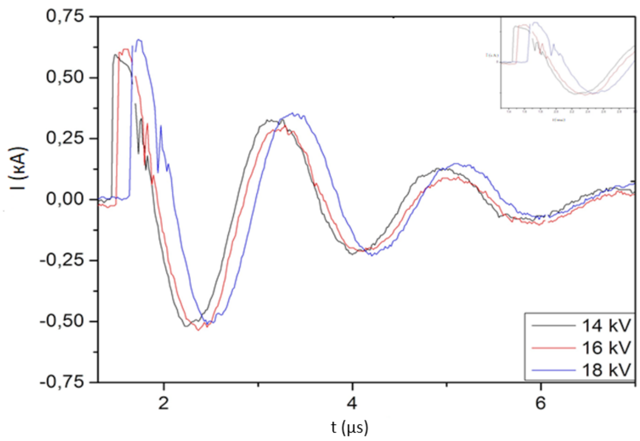
The tungsten and molybdenum samples were irradiated with deuterium plasma using a Mather-type plasma focus device PF-4 which is energetic 4 kJ, with a capacitor bank of 48 µF, maximum charge voltage to 20 kV, total inductance of 158 nH, and peak discharge current of 400 kA [11, 26]. A schematic diagram of experiments of the PF-4 is shown in Figure 1a.

The operating working condition and optimal deuterium gas pressure in the chamber were chosen, so that the strongest pinch plasma could be obtained for maximum neutron yield. The pinch time and pinch current are estimated due to analysis of the derivative current signal dip. The operating working conditions in PF-4 were determined for p=6 torr optimal deuterium gas pressure at U=18 kV. Neutron emission was registered by the silver activation detector used on a shot-to-shot basis. The neutron yield is measured at each filling pressure and it's the average value is $Y=1.35\cdot10^{8}$ n/shot. The ion fluence was numerically computed using Lee Model Code [27] and change in the range of (1.2 – 3.6) 10^{22} cm^{-2} for 10-30 shots operating charging voltage from 14 to 18 kV respectively. The characteristics of the current derivative, hard X-rays are measured using the Rogowski coil and scintillator-photomultiplier detector. The current derivative signals was measured by the Rogowski coil and signal form indicated in Figure 1b.

Tungsten and molybdenum samples were used on flat square plates of 10×10 mm. Pre-exposed deuterium plasma all samples were mechanically polished with commercially available abrasive paper. After polished all samples were observed by optical microscopy to eliminate various mechanical defects. The purpose irradiation tungsten and molybdenum samples were placed at 6 cm from the anode distance and were irradiated at 10, 20 and 30 shots under deuterium ions and neutrons. Irradiation of targets was carried out with a normal fall of the plasma flow to the surface. Morphology changes on tungsten samples at different numbers of shots were analysed by scanning electron microscopy (SEM) and optical microscopy.



a



b

Figure 1 – Experimental setup and diagnostics of plasma focus device
(a) Schematic arrangement of W and Mo samples for exposure
(b)Typical signals of the current derivative of the device at optimal gas pressure

It is known that plasma-irradiated samples can observe different types of surface damage like cracks, blisters and bubbles. These kinds of damage on the sample surface occur due to thermal expansion and thermal stresses during the heating-cooling cycles. During irradiation materials, increase the sample surface temperature which leads to generating defects, such as vaporization and ablation. Thus, the surface damage is proportional rise of surface temperature. Solving the 1D heat equation for homogeneous finite-duration heat loads on semi-infinite surfaces enables to calculation of the maximal rise of the material's surface temperature [28]. The sample surface temperature was calculated as follows:

$$\Delta T \approx 2 \cdot q \sqrt{\frac{\tau}{\pi \cdot \rho \cdot \lambda \cdot c}} \qquad (1)$$

where q – is power flux density, τ – is the influence time of the plasma flux, ρ – material's density, λ – the thermal conductivity and c – the specific heat capacity (at constant pressure).

The tungsten and molybdenum sample surface temperature was calculated during irradiation at optimal gas pressure. The total surface temperature was changed range from ~325 K to ~4725 K for tungsten samples and from ~339 K to ~5096 K for molybdenum respectively.

Results and Discussion

The tungsten and molybdenum sample's surface damage after irradiation in the plasma focus device at 10, 20 and 30 shots are shown in Figure 2. It can be seen that on the surface of materials, relief after irradiation at 10 shots. Surface temperature materials rise to T = 1575 K for tungsten and T = 1699 K for molybdenum respectively. The resulting relief consists of randomly arranged protrusions and depressions of various shapes. Besides relief on the surface of tungsten and molybdenum, also can observe some specific defects such as melting, bubbles, pores, and micro-cracks. Different types of damaged materials depend on plasma type, ion flow and surface temperature. The tungsten sample surface damage at 20 shots is shown in Figure 2b. The tungsten's sample surface temperature reached T=3150 K and appeared for bubbles formation at different sizes. Also, such an effect impulse for molybdenum's surface temperature at T=3397 K results in droplet formation with the simultaneous movement of the molten layer (Figure 2e). When the pulse acting number to 30 shots on the tungsten sample surface, bubbles, pores and droplets were observed. Compared to the bubbles formed at 20 pulses, the number of accumulated and burst bubbles increased. The radiation influence of Mo molybdenum material samples at 30 shots with deuterium ion particles, as a result, increases blisters and micro-cracks.

The radiation effect of plasma and fusion products on tungsten and molybdenum samples, including neutron irradiation, leads to form defects in the crystal lattice in the all volume of the material. Various crystalline defects formed such, as blisters, bubbles, holes, micro-cracks and voids, arise due to the exposure of Wand Mo by radiation ions from 10 to 30 shots. The increase in damage on the surface of the material samples can be explained by an increase in the deuterium ions flow. The appearance of such defects occurs due to high pulsed interaction. A similar result was obtained by Pimenov et al. [29] for vanadium material of the influence of high-pulse helium ions.

The results of the study of tungsten and molybdenum samples on an optical profilometer are shown in figure 3. It was determined that after irradiation with hydrogen plasma, the surface roughness changes depending on the irradiation temperature. The most increase in roughness R_a = 0.0377 µm is observed in samples irradiated at 10 shots surface temperature T = 1575K, which is associated with relief formation and melting on the surface layer. At the same time, in samples irradiated at 20 shots T = 3150 K, the surface of which is characterized by randomly arranged protrusions and depressions of various shapes. Tungsten samples irradiated at 30 shots and surface temperature attained to T = 4725 K surface samples became smooth. After radiation interactions, the arithmetic mean R_a deviation of the surface of the tungsten material samples was reduced by 3 times compared to the initial sample. The results obtained are in good agreement with the results of SEM analysis.

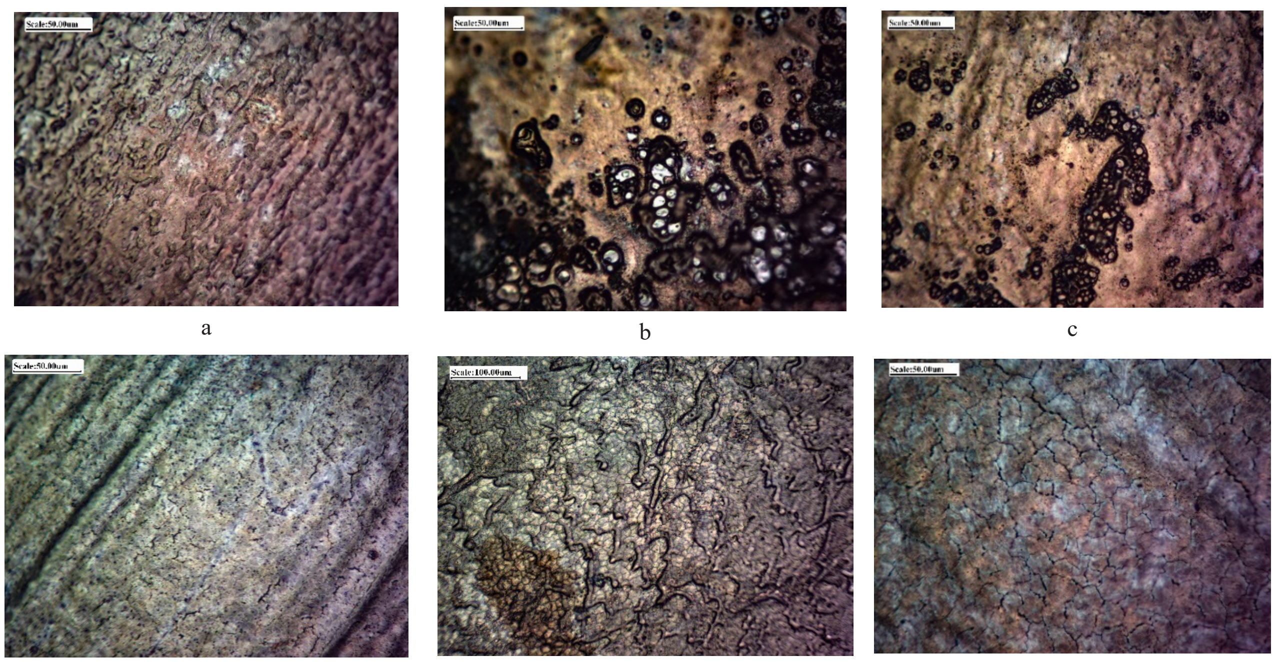


Figure 2 – Microstructure of tungsten surface (a-10 shots), (b-20 shots), (c-30 shots) and molybdenum surface (d-10 shots), (e-20 shots), (f-30 shots) after deuterium plasma irradiation

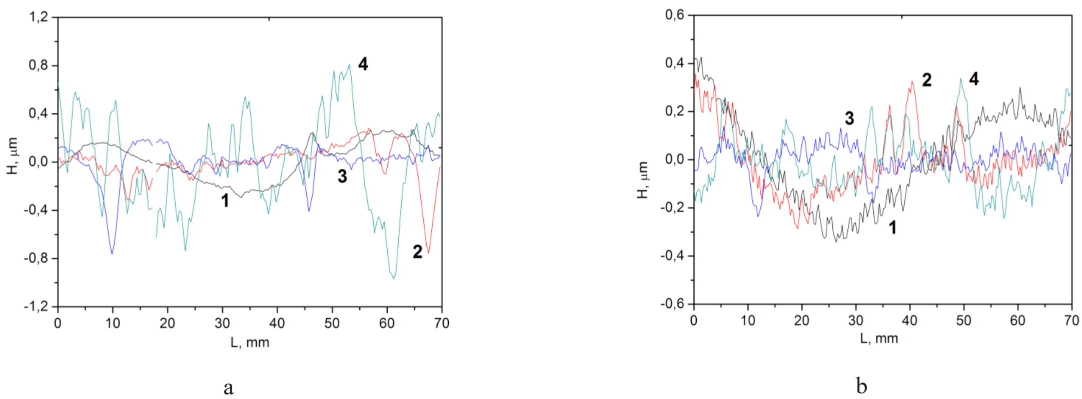


Figure 3 – Profile of surface roughness of samples W (a) and Mo (b).
30 shots (1), 20 shots (2), 10 shots (3), before radiation (4)

Thus, it can be approved that when irradiating tungsten and molybdenum with deuterium plasma, the main relief-forming mechanism is surface sputtering, characterized by thermal influences of the surface.

The erosion of tungsten and molybdenum samples was estimated by weighing the samples before and after irradiation. The dependence of the mass loss of tungsten on the mode of irradiation with deuterium plasma at optimal gas pressure is shown in Table 1. It rather corresponds to the regularity of erosion materials. Thus, it is established that the mechanism of erosion depends on the nature of the solid and the irradiation conditions.

Table 1 – Tungsten and Molybdenum changes in the masses of the material sample after radiation.

Number of shots	W, Samples weight		Δm (mg)
	Before radiating (±0.001), g	After radiating (±0.001), g	
10 20 30	1.649 1.645 1.681	1.634 1.622 1.650	-15 -23 -31
	Mo, Samples weight		
	Before radiating (±0.001), g	After radiating (±0.001), g	
10 20 30	15,061 14,774 15,171	15,05 14,735 15,128	-11 -39 -43

The obtained results showed that with an increase in the target surface temperature and shots, surface erosion increases. The results of the effect of plasma flow on the material are determined mainly by the specific power of the incident flow.

Due to the thermophysical properties of tungsten and molybdenum samples, as a result of plasma stream irradiation noticeable erosion. In addition, under severe exposure, surface cracking is observed due to the occurrence of significant thermal stresses due to the temperature gradient along the thickness of the target.

Experimental studies of the effect of deuterium plasma irradiation on the microstructure of tungsten and molybdenum were carried out. The structure of tungsten and molybdenum samples after irradiation with deuterium plasma from 10 to 30 shots was studied using scanning electron microscopy (SEM) (Figure 4). It was found that both materials' sample surfaces eroded when irradiated with 10 shots of deuterium plasma. At the same time, the molybdenum sample surface appears melting with pores and micro-cracks (figure 4d). Also, the tungsten's sample surface is formed ranging in size from 74 to 172 nm (figure 4a). It occurs the accelerated ions with an energy exceeding the sputtering energy interact with materials, and this moment on the surface materials is due to elastic and inelastic processes. On the surface materials nuclear and chemically react which as a result changes the properties of materials and causes modification and damage processes on tungsten materials. Due to the elastic and inelastic interactions of the deuterium ions with an energy exceeding the sputtering energy, occurs to sputtering atoms and micro and macroscopic erosion of the target.

In particular, irradiated at 20 shots, on the tungsten sample surface many small pores ranging in size from 64 to 453 nm and micro-cracks are formed (Figure 4b). The molybdenum sample surface develops radiation swelling and system cracks (Figure 4e). The system of cracks and pores creates on the materials an opportunity for deeper penetration of ions into the volume of the materials. The causes of these structural disorders appear to be mechanical stresses in the tungsten lattice caused by implanted deuterium.

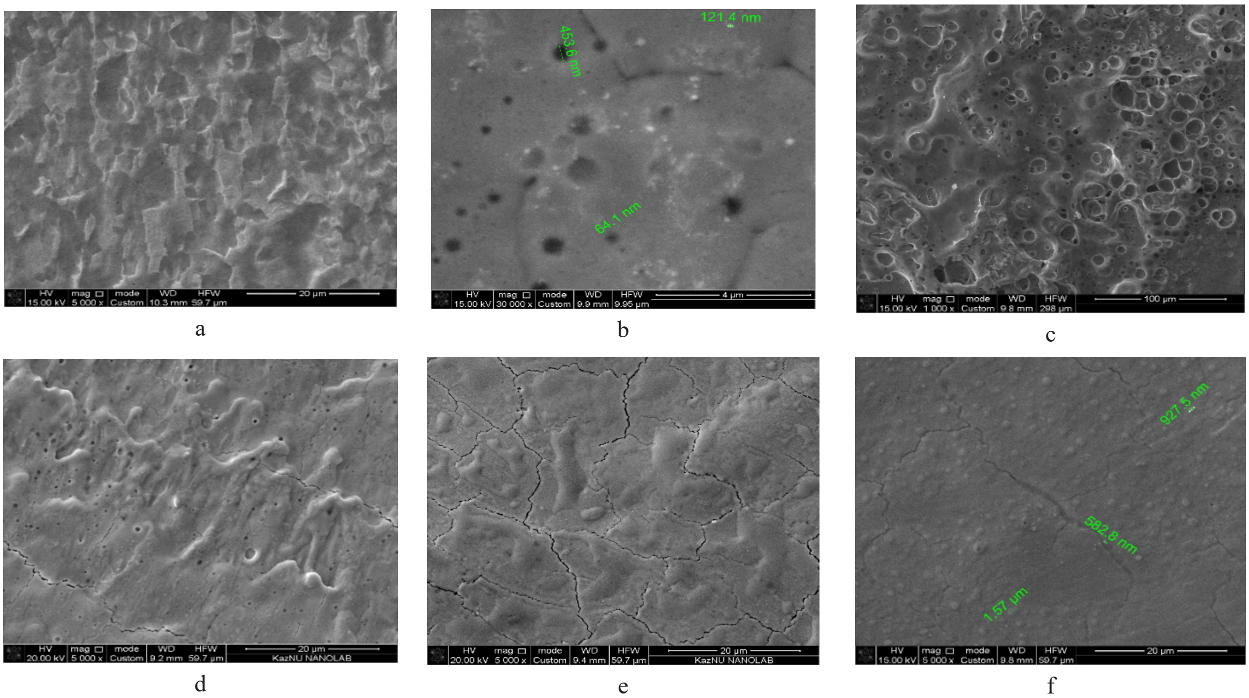


Figure 4 – Microstructure of tungsten surface (a-10 shots), (b-20 shots), (c-30 shots) and molybdenum surface (d-10 shots), (e-20 shots), (f-30 shots) after deuterium plasma irradiation

An optimal gas pressure, irradiation at 30 shots on the surface of tungsten and molybdenum targets was observed as burn bubbles, cracks, and blisters (from 157 nm to 927 nm) and some parts of the tungsten's sample surface became smooth. It may have connected with an increased influence of intensive pulse plasma from 10 shots to 30 shots leading to a decrease in the sputtering process and an increase in the implementations of ions. At optimal deuterium 6 torr gas pressure in the chamber due to more exposure shots deep penetration of ions begins to form defects in material samples.

Conclusion

In this study, the tungsten and molybdenum sample surface was irradiated to optimal gas pressure by deuterium ions and neutrons produced in a plasma focus device. A plasma focus device is used to study damage on materials relevant to fusion reactors. The analysis of experimental results showed that the change in the relief and structure of the surface layer of irradiated samples depends on the type of material and irradiation parameters.

The analysis showed that irradiated tungsten and molybdenum samples at optimal gas pressure lead to the damage of materials. Damage to the surface samples appears in the form of melting, evaporation, bubbles etc. Erosion of tungsten and molybdenum was studied by a large number of repetitive pulses. The repetitive pulse in the plasma focus device can affect the change in surface temperature. Increasing surface temperature leads to begin the evaporation process on the samples and results in enhanced mass losses of tungsten and molybdenum. During irradiation of tungsten and molybdenum sample surface temperatures below, the melting threshold on samples appears melting which is mainly caused by thermal damage. Also, the melting process leads to increased surface roughness. Change in the level of relief and structure surface layer of irradiated samples depends on the type of material and irradiation parameters. On optimal deuterium gas pressure increasing plasma shots, surface material samples became smoothing.

Information on the macroscopic erosion of materials will be used in the future to build computational models that allow predicting the erosion rate, as well as the amount and composition of erosion products in a tokamak with reactor parameters.

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