Lithium deposits from the T-10 tokamak after experiments with lithium capillary-porous system

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Abstract. The structure of lithium deposited specimens at the T-10 tokamak vessel after examination of lithium capillary porous system were analysed. Scanning electron microscopy have been used to analyse the deposits. Composites of lithium carbonate Li$_x$CO$_y$ have been found by analysis. Plasma irradiation of these lithium specimens have been carried out in the PLM plasma device with plasma parameters similar to the tokamak divertor plasma. Stationary plasma load up to 1 MW/m$^2$ during 200 minutes in the PLM provided the change of surface morphology revealed by post-mortem analysis.

1. Introduction

Liquid lithium components of the first wall like capillary porous systems (CPS) [1–4] are attractive for a fusion reactor. The CPS produces an improved recycling first wall in tokamak discharges, see [1, 2]. A liquid surface of the CPS has higher tolerance to neutron damage, it is not limited by erosion and can ‘self-heal’ from damage during transients like disruptions and ELMs. The efficiency of liquid lithium components has been demonstrated in experiments on tokamaks T-10 [5, 6], T-11M [7,8] etc. However, the reactivity of lithium (Li) leads to quickly reactions with impurities (oxygen, nitrogen etc.) in the plasma and on the in-vessel components. It can lead to the growth of solid deposits decreasing the advantageous effects of lithium and the CPS operation. In tokamaks the growth of deposits is observed due to the erosion of plasma facing components (PFCs) by plasma high heat flux [9-16]. The effects of the growth of deposits with a heterogeneous structure were detected in tokamaks [9-16]. These deposits have a universal tendency to form porous structures and surface with high specific area. Such deposits are the origin of fuel retention (D or T) and it is an important issue in future fusion reactors since tritium inventory has to be limited due to safety reasons. The roughness and irregularity of such plasma-facing surfaces can affect plasma-surface interaction influencing edge plasma parameters and plasma confinement. Therefore understanding the irregular and porous deposit formation on the tokamak vessel is an important objective. It is considered [15-22] that the long-term evolution of the plasma – facing component surface under the high heat plasma...
load in a tokamak is controlled by universal mechanism of surface growth known in the condensed matter physics due to evolution of the interface layers including plasma turbulence influence (see, e.g. [10-25]). Under the long-term plasma load it is expected the formation of a heterogeneous surface structure with a high surface area and porosity. Therefore, fusion materials should be tested with stationary plasma in order to predict their behavior in a thermonuclear reactor-tokamak. For such purpose lithium composite deposits from the T-10 tokamak were irradiated with stationary plasma in the PLM device [26,27] which is the simulator of divertor plasma. The results of such study are presented in this article.

2. Samples structure

Lithium samples collected from the vessel of the T-10 tokamak [5] after three campaigns in 2016–2018 were investigated. The T-10 tokamak operated with typical ohmic and ECR heating in these campaigns, mostly below 1 MW. In these experiments lithium from the heated CPS was deposited on PFCs. In experiments with lithium CPS carbon and oxygen concentration was considerably decreased in the plasma volume, which led to the reduction of $Z_{\text{eff}}$ from ~4 to 2.5. Details concerning particular experimental campaigns see in Ref. [5].

![Fig. 1. (a) SEM micrographs of the plasma-faced surface of film layer deposit, sample from the T-10 tokamak in experiments with the lithium CPS.](image)

The lithium samples were collected and cut from locations close to the lithium CPS and mushroom limiter downside in the plasma shadowed zones. The samples are of irregular shape like spherical balls of approximately 0.5 – 5 mm in diameters and thick layers of approximately 0.5–2 mm thickness. The samples surface morphology was investigated with the SEM scanning electron microscope. SEM examinations revealed that the surface of all examined samples is strictly irregular as evidenced by the images in Fig. 1. The surface shows a specific pattern in the form of stochastically agglomerated elongated elements of minimal size of approximately 200 nanometers, see Fig. 1a,b. It is worth noting that the similar hierarchical structure has been revealed on both the deposit layer surface faced to plasma and faced to the vessel [10-11]. Thus, it can be concluded that the structures are very likely to initiate at the plasma-surface interface by interaction of lithium with carbon and oxygen impurities in the plasma and on the vessel.

The ultimate gas pycnometer Micro-Ultrapyc 1200e was used for measuring the true density and volume of examined samples from the T-10. True density of the samples was evaluated of $1.9342 \pm 0.0008$ g/cm$^3$. Specific area of sample surface measured by the pycnometer was of $2.8$ m$^2$/g. Specific
volume of nanoscale pores was evaluated of 0.02 cm$^3$/g which is a relatively small value; this means that small-scale elements have a sufficiently high density without a large number of nanoscale pores. The X-ray photoemission spectroscopy (XPS) with the electron-ion spectroscopy unit based on Nanofab 25 (NT-MDT) platform [28] was used to analyze chemical composition of the samples was fixed. Standard XPS analysis of samples identified presence of chemical elements like C, O, Li for the lithium samples. Relative atomic concentrations estimated from the XPS spectra demonstrated a deviation of relative atomic concentrations from the reference high-purity powder Li$_2$CO$_3$, the reason is caused by their complex structure like Li$_y$CO$_x$.

3. IRRADIATION OF SAMPLES WITH STATIONARY PLASMA

The plasma irradiation of lithium samples from the T-10 tokamak (Fig. 1) were performed on the plasma linear multicusp (PLM) plasma device [26, 27]. The PLM device is a linear plasma trap with a multi-cusp configuration of a magnetic field and a stationary plasma discharge that provides the powerful plasma-thermal load up to 5 MW/m$^2$ on test materials. The test samples were irradiated with helium plasma of discharge duration up to 200 min. Plasma parameters measured by Langmuir probe and spectroscopic diagnostics were as follows: plasma density was up to $3 \times 10^{18}$ m$^{-3}$, the electron temperature was up to 4 eV with a fraction of hot electrons of temperature up to 50 eV, the ion plasma flow onto the test sample was up to $3 \times 10^{21}$ m$^{-2}$ s$^{-1}$, discharge current reached the value of more than 15 Amps. Magnetic field was of 0.01 Tesla on the trap axis and up to 0.1 Tesla in the cusps. The target samples have no active cooling in these experiments, plasma heat load on test target samples was of 0.5 - 1 MW/m$^2$.

![SEM micrographs of lithium sample after helium plasma irradiation in the PLM divertor simulator during ~200 minutes.](image)

The lithium species from the T-10 tokamak were fixed in the stainless steel bad. Such target with lithium species at floating potential and biasing voltage from -20 Volts to -100 Volts was installed in the centrum of the PLM discharge and irradiated with plasma during 200 minutes. Depending on the biasing voltage applied to bath with specimens, the lithium specimens were heated from 200 °C to more than 700 °C under plasma load. Under stationary plasma load lithium, carbon, oxygen from sample enter to the plasma volume as was detected by optic spectroscopy. The radiation of lithium influxed to plasma was detected by spectroscopy diagnostics. Spectra of radiation have been measured detecting lithium lines at 460.6, 497.1, 610.3, 670.8, 816.6 nm. Post-mortem analysis of the specimens after plasma irradiation in the PLM have revealed a change of the structure comparing with initial one. The colour of the specimens has changed from white to black one after the treatments in the PLM. The SEM analysis of the samples surface (Fig. 2) has shown the
reforming of irregularity, cf. Fig.1. Hierarchical structure within scales from hundreds nanometers to tens of micrometers of the samples treated in the PLM was drastically changed. Hierarchical granularity of the structure in the range of scales 0.5 - 500 microns was observed, Fig.2. These structures are similar to the previously observed morphology of hydrocarbon composites deposited in tokamaks such as cauliflower-like and others hierarchical granularity structures [10-12].

4. Conclusion

The structure and the composition of deposited lithium layers and specimens at the vessel of the T-10 tokamak during campaigns in 2016 - 2018 with lithium capillary porous system were analysed. The samples were cut and collected from locations in the plasma shadowed zones. The reactivity of lithium has led to reactions with impurities in the plasma and on the vessel. The post-mortem analyses have evaluated materials mixing and evidence of plasma-induced structure. High resolution scanning electron microscopy and the X-ray photoemission spectroscopy have revealed lithium carbonate Li$_x$CO$_y$ composites with porous and roughen irregular surface. Experiments devoted to the interaction between hot plasma and this lithium composite have been carried out in the PLM plasma device which is the divertor simulator with plasma parameters similar to the tokamak far-SOL and divertor plasma. Such plasma irradiation with stationary plasma load of 0.5 - 1 MW/m$^2$ during ~200 minutes led to the change of surface morphology and elemental chemical composition revealed by post-mortem analysis. Under the stationary plasma load, new structures with a hierarchical granular structure with high surface area are formed. These structures are similar to the previously observed morphology of hydrocarbon composites deposited in tokamaks such as cauliflower-like and of others hierarchical granularity structures. Comparative tests of lithium materials from the T-10 and the reference industrial powder of lithium carbonate irradiated with stationary plasma in the PLM have shown an identical change in their structure indicating universal influence of stationary plasma on the structure of irradiated materials. For a fusion reactor, the use of lithium can lead to the growth of lithium chemical solid composites on in-vessel components. Such deposited films and layers can be formed due to the absorption of carbon and oxygen from the volume during the periods between discharges or during conditioning. These new phenomena have been evidenced about the issue of retention if Li walls will be implemented in a fusion reactor.

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