

# **MODIFICATION OF STEEL SURFACES EXPOSED BY HYDROGEN/HELIUM PLASMAS STREAMS SIMULATING FUSION REACTOR CONDITIONS.**

a part of the IAEA's Coordinated Research Project "F43022",  
"Plasma–Wall Interaction with Reduced Activation Steel Surfaces  
in Fusion Devices"

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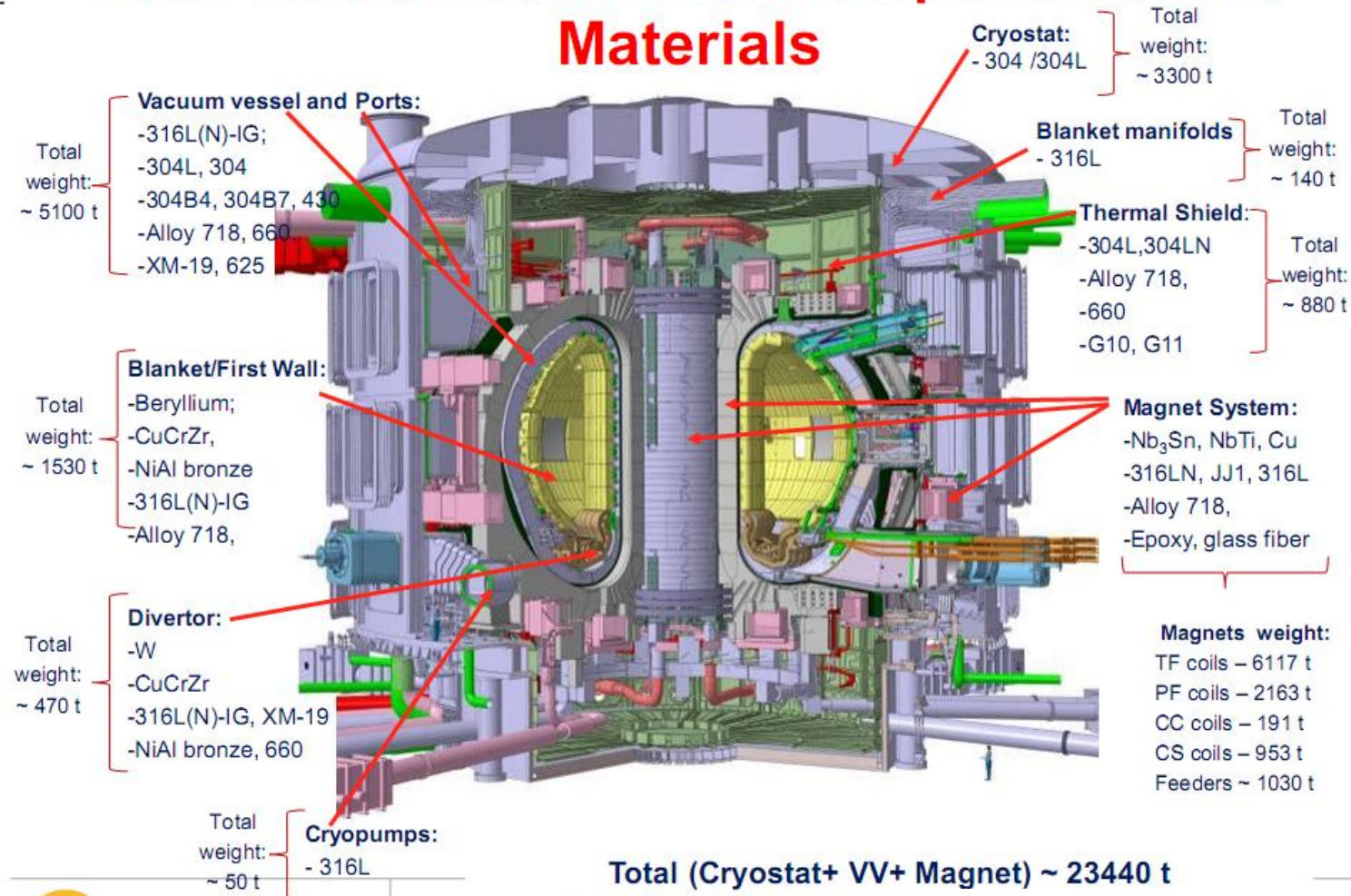
## *Outline*

- Introduction
- Experimental facility
  - QSPA Kh-50
  - MPC
  - Prosvet
- Feature of plasma-surface interaction with powerful plasma streams
- Damage/modification of surfaces exposed by plasma streams
- Future plans as a part of project activity

# Introduction

## Overview of Main ITER Components and Materials

**Materials issues are key factors for ITER success**



ICRFM-17, Aachen, Germany, 11-16 October 2015  
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B. Bigot, Key note lecture during ICRFM-17

## Introduction

The detailed experimental studies of threshold values for the damaging processes:

- ❖ roughening,
- ❖ crack formation,
- ❖ melting of the Plasma Facing Components,
- ❖ dust generation

under ITER or DEMO relevant loading scenarios are required for evaluation of the materials performance under short transient events. Numerical and experimental simulations with large number of repetitive impacts and variation of plasma energy density are required

# Introduction and Objectives

## DISRUPTION:

- peaked heat load profile
- $Q = (10-100) \text{ MJ/m}^2$
- pulse duration (1-10) ms

## Type I ELM:

- $\nu = (1-100) \text{ Hz}$
- $Q = (1-3) \text{ MJ/m}^2$
- $t = (0.1-1) \text{ ms}$

✓ Transient plasma loads expected for ITER disruptions and ELMs can not be achieved in existing tokamaks

✓ Material response to multiple exposures:

E-beams, QSPA, linear devices (PSI-2, MAGNUM-PSI), pulsed plasma guns, are used

✓ Quasi-steady-state plasma accelerator (QSPA) is attractive facility for :

- Simulation of heat loads typical for disruptions and ELMs
- Investigation of plasma/surface interaction:
  - Shielding,
  - melting,
  - evaporation,
  - erosion mechanisms
- Measurements of data for validation of numerical models

## Quasi Steady State ?

Duration ( $\tau$ ) of the process (discharge) essentially exceeds the time ( $t$ ) of flight of the plasma particles in the accelerating channel i.e.  $\tau/t > 1$  Time of flight:  $t = L/v_m$ ;  $L$  - length of accelerator channel  $v_m$  – maximal velocity

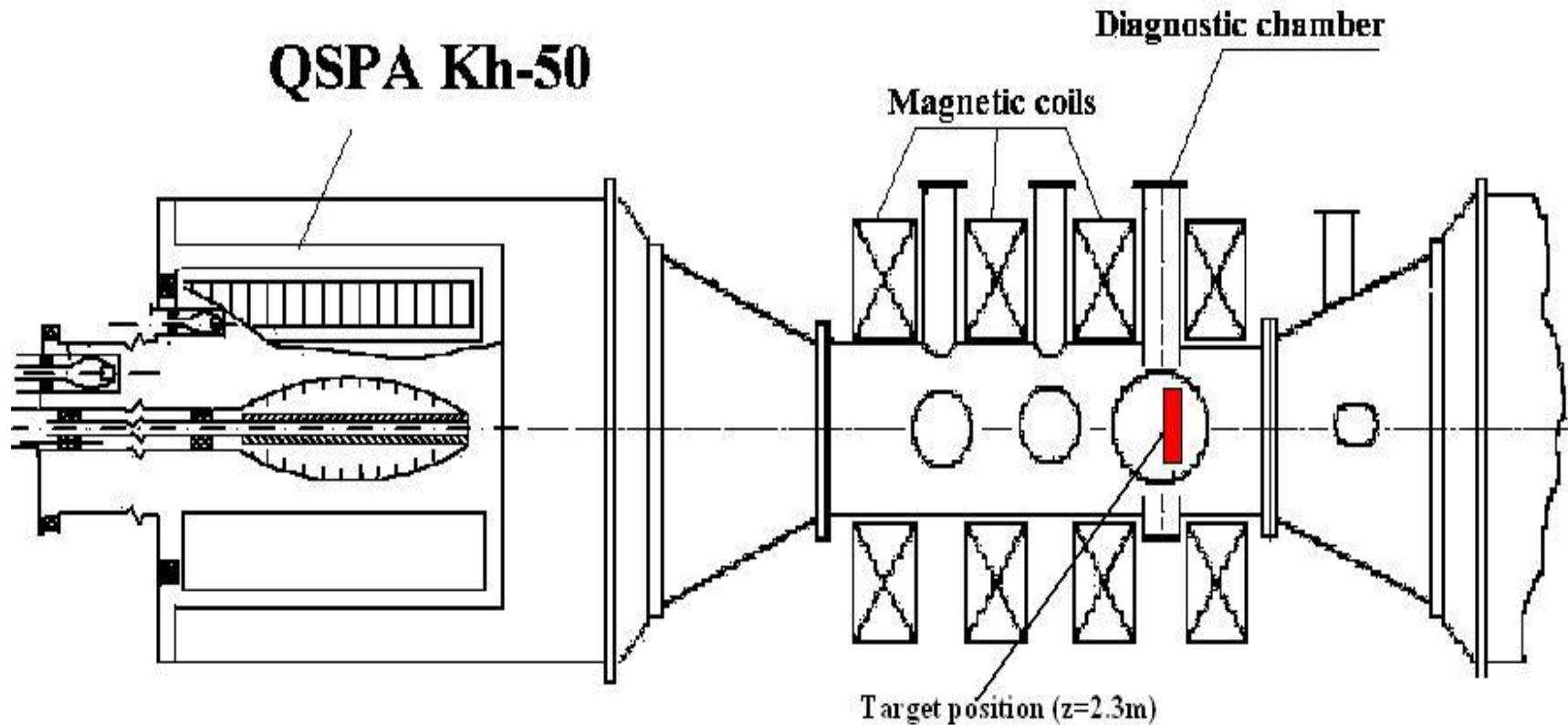


## QSPA Kh-50 Device



*The QSPA Kh-50 is the largest and most powerful device of QSPAs*

## QSPA Kh-50 Device



Energy density  $\rho_w = (0.5...30) \text{ MJ/m}^2$ ,

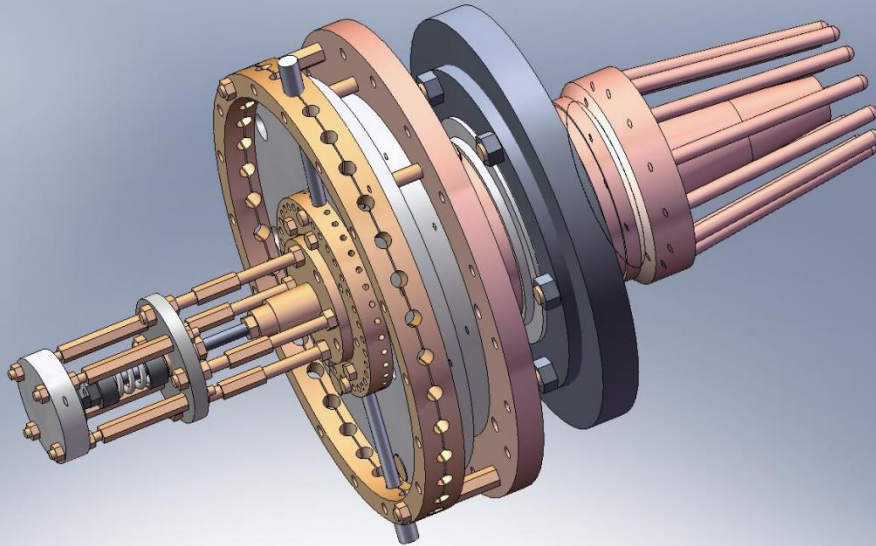
Plasma pulse duration  $\tau \approx 0.25 \text{ ms}$ ;

$P_{\text{max}} = (3-18) \text{ bar}$ ,  $n = (0.2-5) \cdot 10^{16} \text{ cm}^{-3}$ ;  $B_0 = 0.54 \text{ T}$  ( $\beta \approx 0.3...0.4$ );

Diameter of plasma stream- **15 cm**

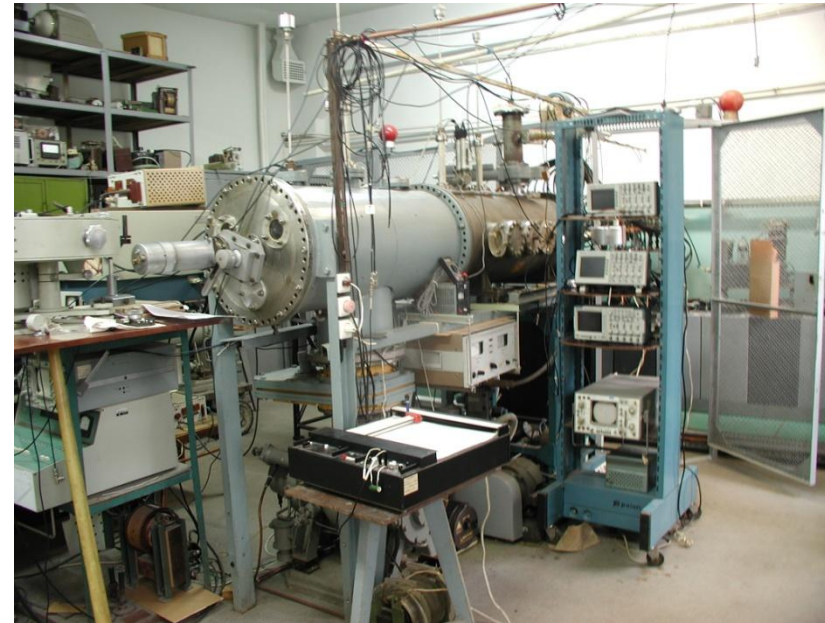


## Experimental facility MPC



Electrode system of MPC

$\varnothing$  cathode (outer electrode) = 6 cm, 3 cm  
 $\varnothing$  anode (inner electrode) = 12 cm, 8 cm  
 Copper rods diameter of 1 cm and of 14.7 cm in length

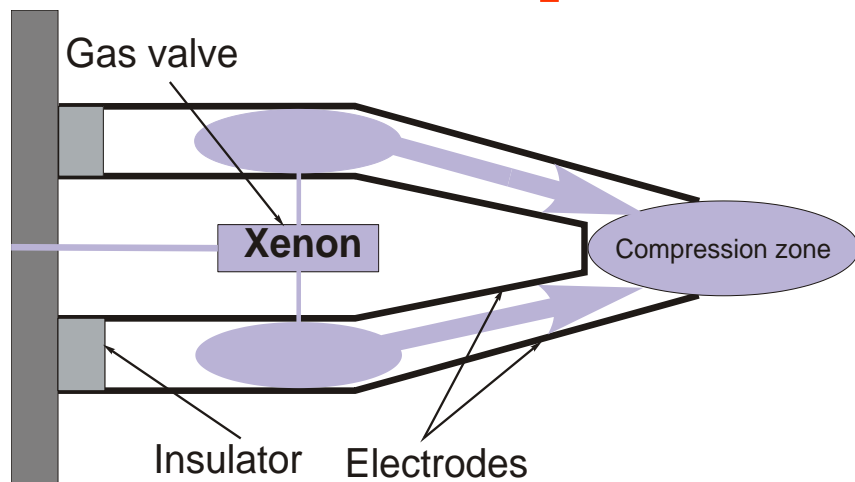


General view of MPC with diagnostics

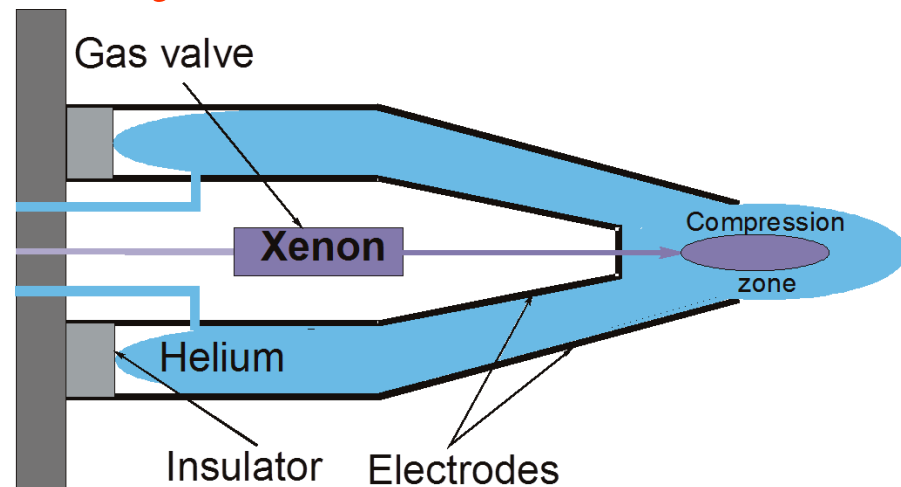
$C_c = 90 \text{ } \mu\text{F}$        $U_c = 20 \div 30 \text{ kV}$   
 $C_v = 700 \text{ } \mu\text{F}$        $U_v = 3 \div 5 \text{ kV}$   
 $I_d = 500 \text{ kA}$        $T_d = 15 \div 20 \text{ } \mu\text{s}$   
**Working gas – xenon, helium,  
 Xe+He**



## Operation modes of MPC



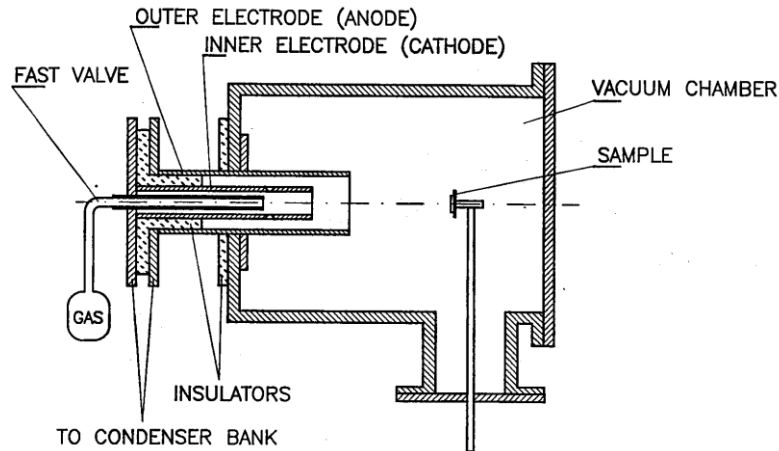
pure xenon discharges with pulsed gas supply



discharges in helium under different residual pressures with additional pulsed injection of Xe directly into the compression zone

The working regimes could be varied by choosing the quantity of gas, supplying the MPC, changing the time delay of the discharge ignition in respect to the gas supply pulse and also by selected value of the discharge voltage.

## Surface modification



$$U_c = 25-30 \text{ kV}$$

$$E = 2 \text{ keV}$$

$$\tau = 2-5 \text{ } \mu\text{s}$$

$$\varnothing_c = 5 \text{ cm}$$

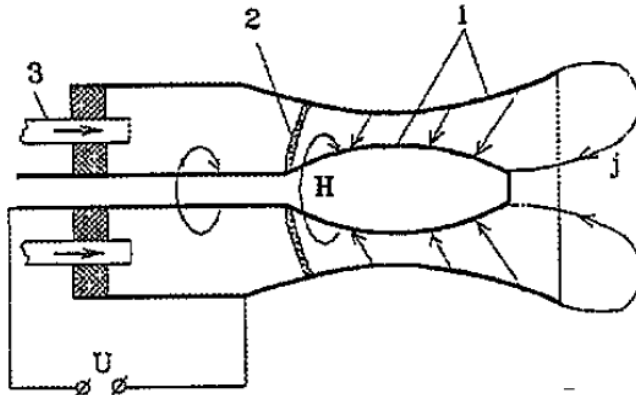
$$\varnothing_a = 14 \text{ cm}$$

hydrogen, helium, nitrogen,  
argon, ....xenon

## Pulsed Plasma Source (PPA)



Advantage: simple and robust design



**Scheme of accelerating channel:**  
1 - electrodes; 2 – front ionization;  
3 – gas injection

## Plasma stream parameters

Voltage on capacitor batter  
of accelerating channel

$U_{оч}$

mass flow of working gas

External magnetic field.

quantity of gas filled

Time delay ( $t_m$ ) between switching on the gas valve and  
start of discharge in main accelerating channel  $U_m$

### Diagnostic methods:

Magnetic probes; Local calorimeters (both at plasma stream and at the target surface);

Piezoelectric detector was applied for plasma pressure measurements

Stark broadening of  $H_\beta$  spectral line was applied for electron density measurements;

## Plasma Parameters in Different Working Regimes

Parameters	ELM 1 simulation no melting	ELM 2 simulation melting	ELM 3 simulation evaporation	Disruption
Plasma stream energy density [MJ/m <sup>2</sup> ]	0.9-1.0	1.2-1.5	2.4-2.5	24-30
Target Heat Load [MJ/m <sup>2</sup> ]	0.45	0.7-0.75	1-1.1	0.65-0.7
Plasma load duration [ms]	0.25	0.25	0.25	0.2-0.25
Maximal plasma pressure [MPa]	0.48	0.32	0.45	2
Average plasma density [10 <sup>16</sup> cm <sup>-3</sup> ]	1.5-2.5	0.5-0.7	0.2-0.3	4-8
Plasma stream diameter [cm]	12-14	18	16	14

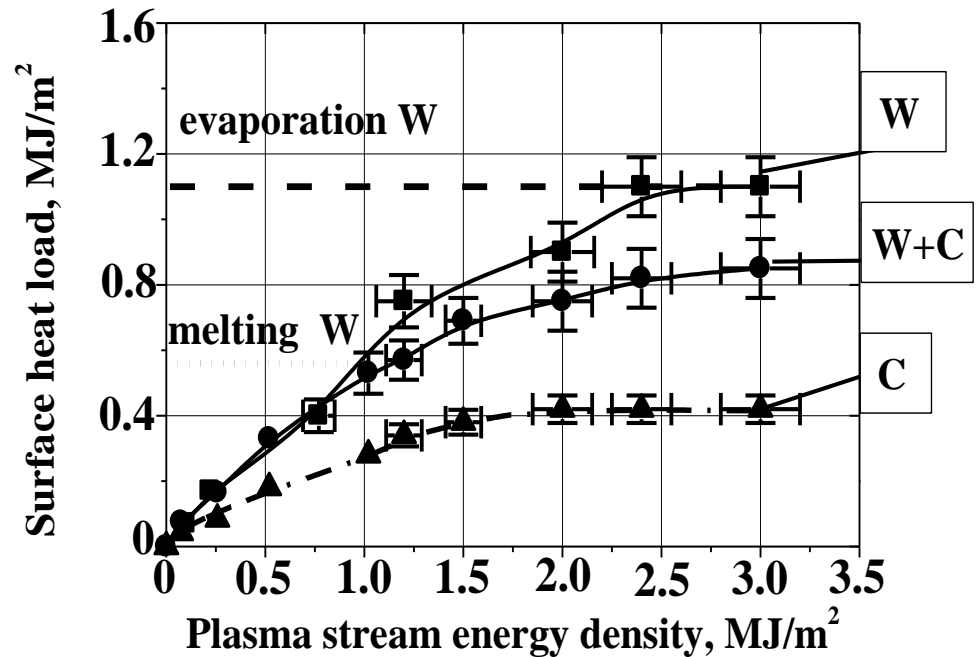
➤ Due to a vapor shield formation the exposed armour target will be protected from the high heat load and erosion by evaporation will be reduced in hundred times



# Features of plasma –surface interaction



Image of plasma stream interaction with the target for  $t= 30 \mu s$  after beginning of plasma-surface interaction.



Heat load to the target surfaces vs. the energy density of impacting plasma stream

Cracking threshold is  $\sim 0,3 MJ/m^2$

Melting threshold is  $0.6 MJ/m^2$

Vapor shield formation and its influence on plasma energy transfer to the surface became clearly seen when the surface heat load achieves

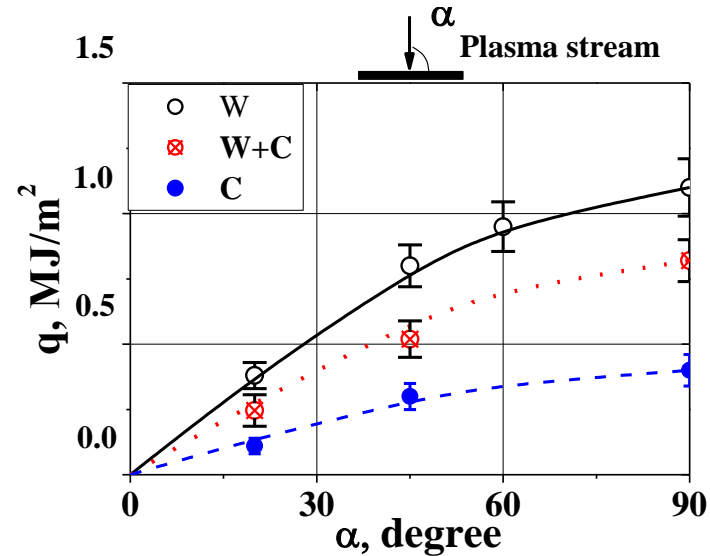
**1.1  $MJ/m^2$ .**

V.A. Makhilaj, et al. Acta Polytechnica 53(2) (2013)193–196; V.A. Makhilaj, et al // Prob. At Sc. Tech.. 2014. № 6. p.44

# Plasma-surface interaction inclined impact



Image of plasma stream interaction with the target for  $t= 30 \mu s$  after beginning of plasma-surface interaction (angle  $60^\circ$ ).

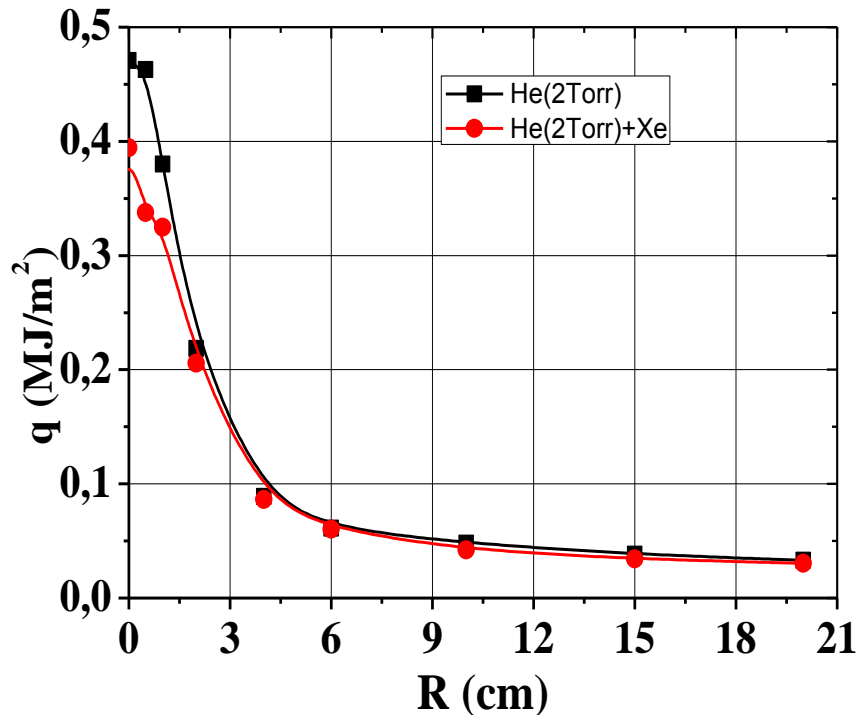


Heat load to target surfaces vs. incidence angle of impacting plasma stream. Plasma energy density is  $2.4 \text{ MJ/m}^2$

For inclined surface irradiation the radial distribution of energy density is non-symmetric. The thickness of the shielding layer (width of luminous area in front of targets) is the smallest at the upper edge of the sample (forwarded to the plasma stream). This layer of cold plasma is responsible for decreasing part of incident plasma energy which is delivered to the surface.

V.A. Makhraj et al. Acta Polytechnica 53(2) (2013)193–196; V.A. Makhraj, et al // Prob. At Sc. Tech.. 2014. № 6. p.44

## Energy density in plasma streams



The maximum energy density in axial region achieved (0.45 – 0.47) MJ/m<sup>2</sup> when operating with the residual gas.

Local injection of xenon into the compression zone led to decrease the energy density down to (0.35 – 0.4) MJ/m<sup>2</sup>.

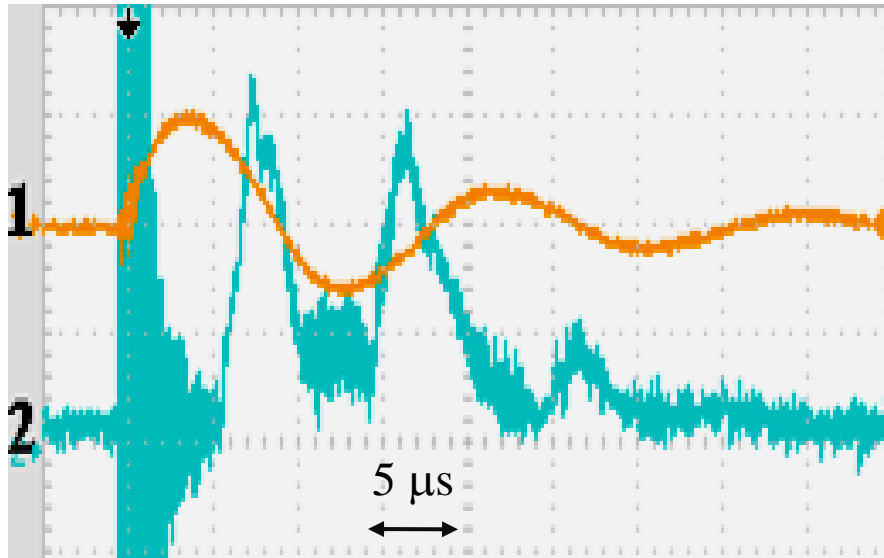
**Decreasing of total energy measured by colorimeter is caused by losses to ionization of heavy impurities (Xe).**

Maximal heat load to tungsten surface exposed to helium plasma achieved 0.39 MJ/m<sup>2</sup>.

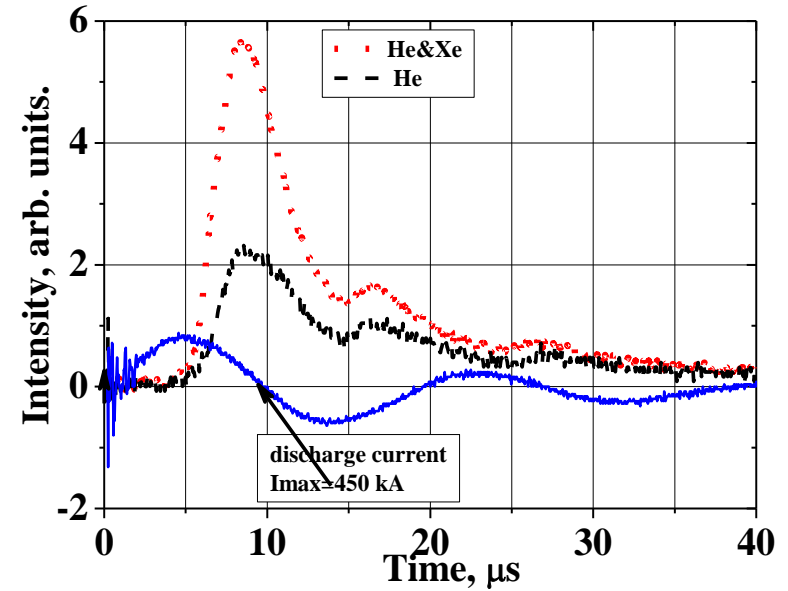
The heat load to tungsten surface exposed under additional injection of xenon is decreased to 0.33 MJ/m<sup>2</sup>.

# Measurements of plasma radiation

EUV



Visible



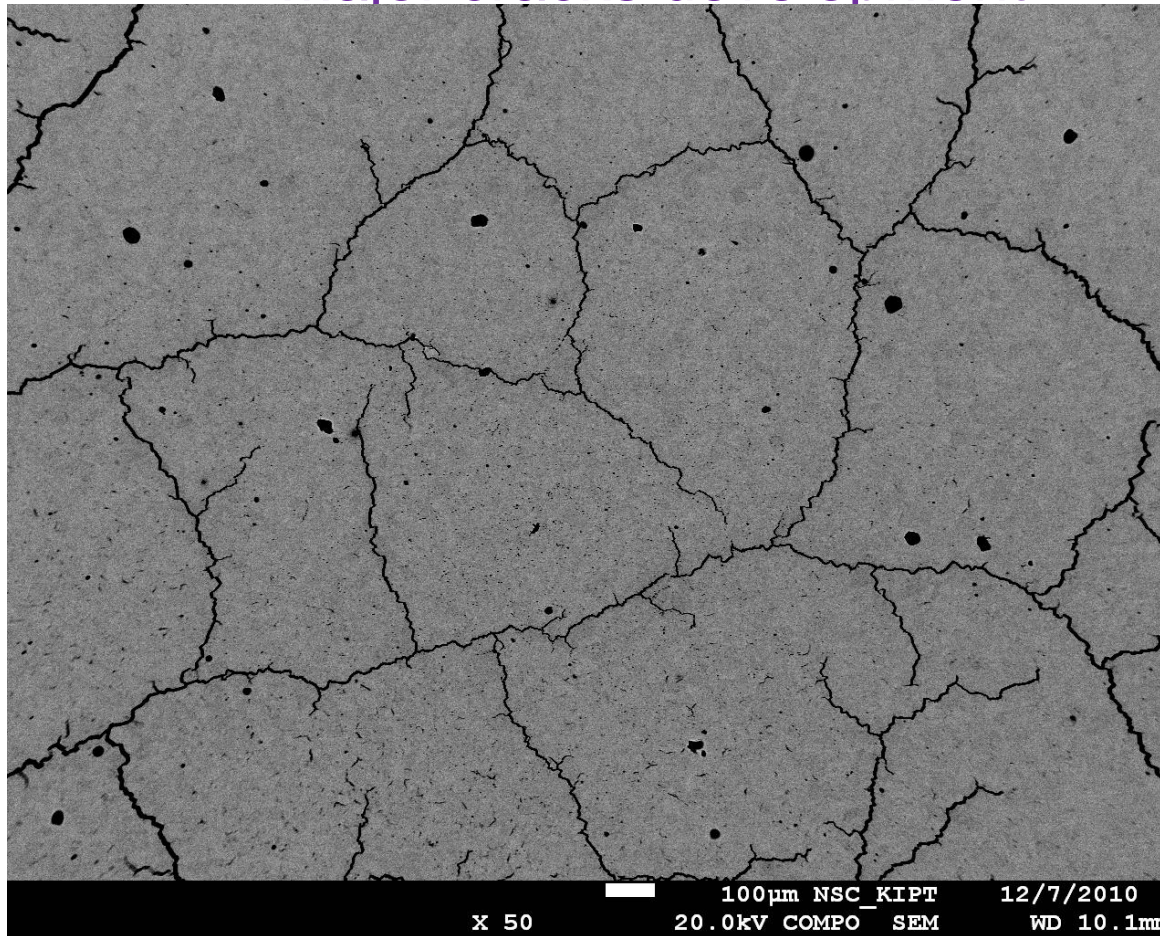
1- Typical waveforms of discharge current  
2- AXUV 20 signals for 12.2–15.8 nm waverange  
Maximal discharge current - 450 kA,  
pressure of He in working chamber 2 Torr.

Peaks on time dependences of radiation pressure and pressure correspond to the first, second and third half-periods of discharge current.

**Radiation energy about 50 mJ**



## Major cracks development

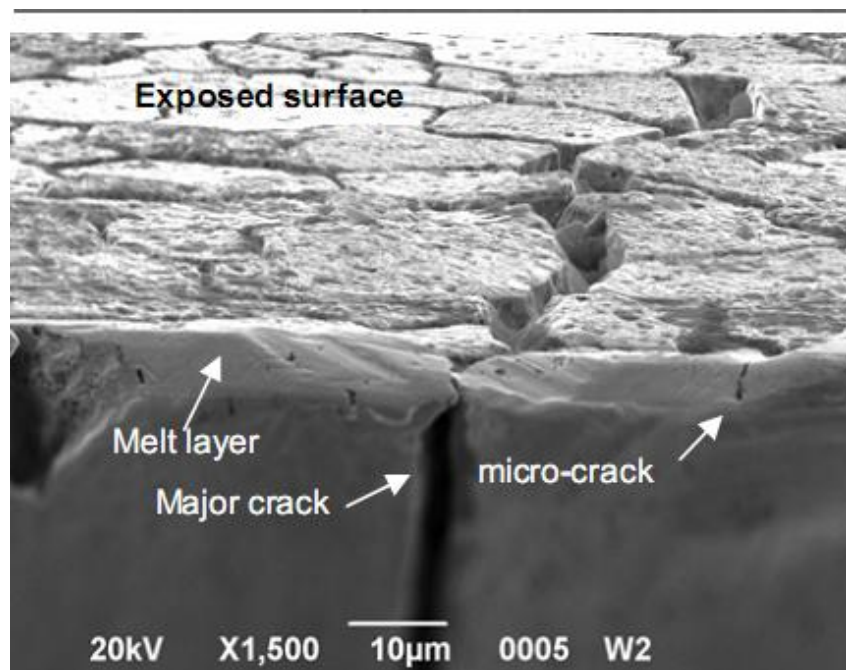


Heat load above cracking threshold below melting threshold

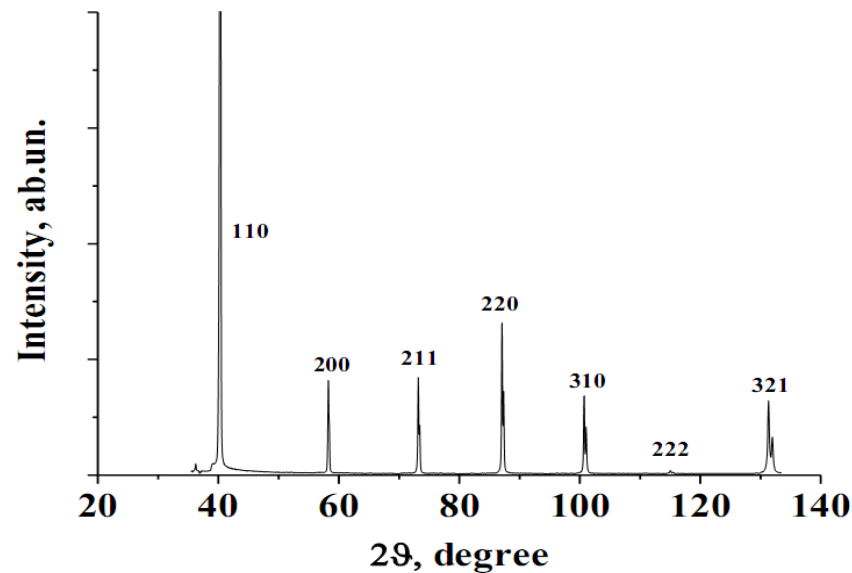
Average cell sizes of 0.8-1.3 mm. The width of cracks is about 0.5-1.5 μm

## Major and micro cracks development

$$T_{in} = 200 \text{ }^{\circ}\text{C}$$



- ☐ Melt layer
- ☐ Major cracks
- ☐ Micro crack

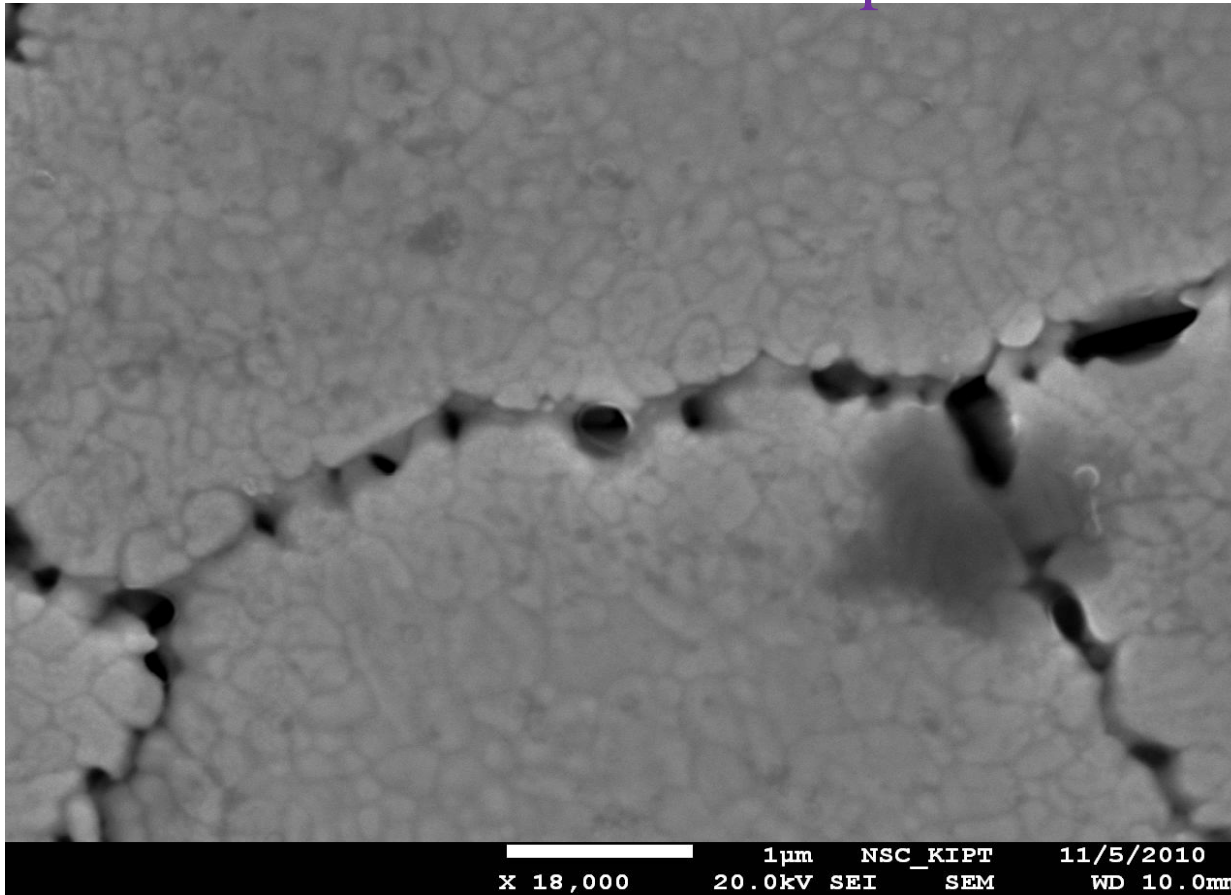


X-ray diffraction diagram of tungsten preheated to 200 °C after pulses of 0.75 MJ/m<sup>2</sup>; 2θ values (Cu K<sub>α</sub>).

No phases of impurities except of tungsten lines.

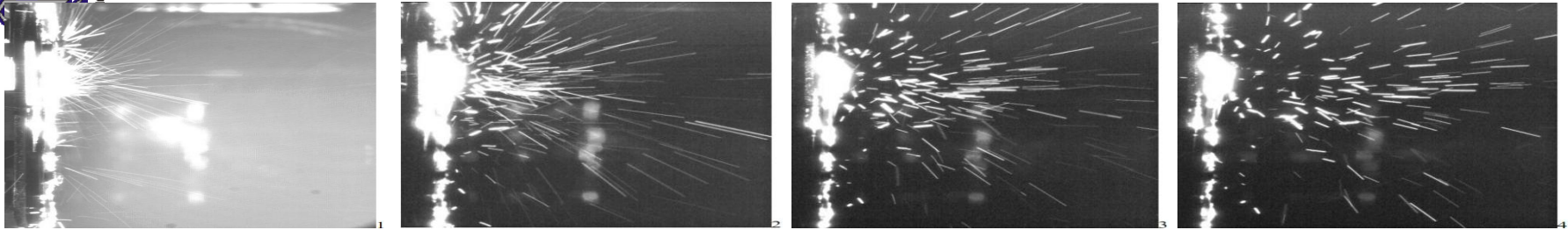
## Heat load above melting threshold

## Micro cracks development



Melting of surface and development of cracks along the grain boundaries are accompanied by formation of resolidified bridges through the fine cracks.





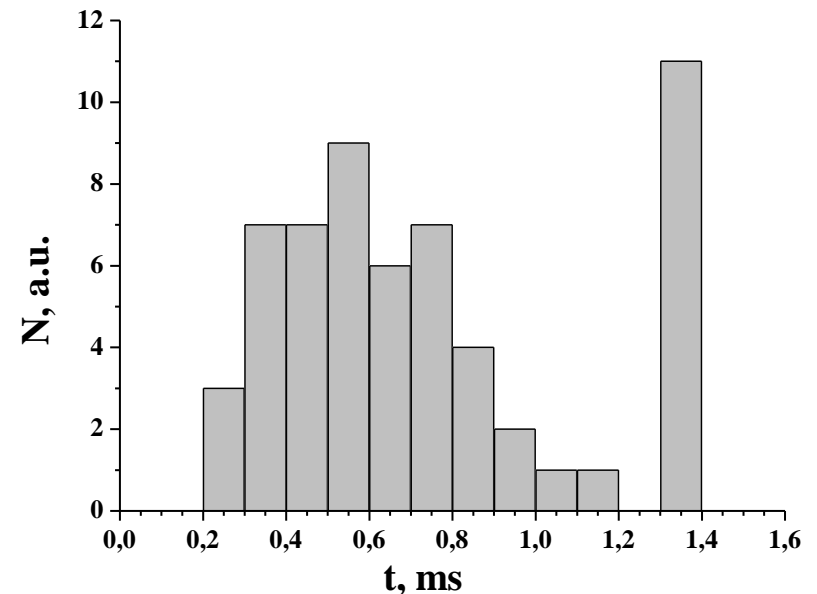
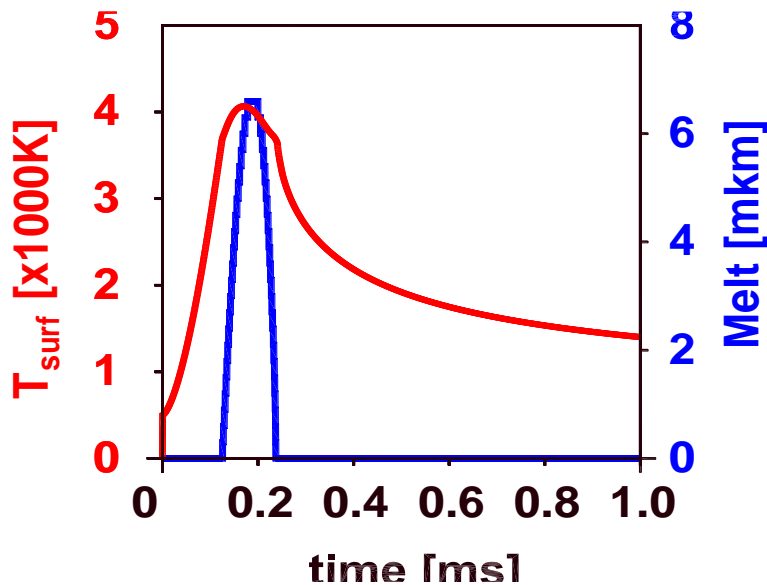
$t_1 = 1.2$  ms,

$t_2 = 3.6$  ms,

$t_3 = 6$  ms,

$t_4 = 8.4$  ms;  $t_{\text{exp}} = 1.2$  ms

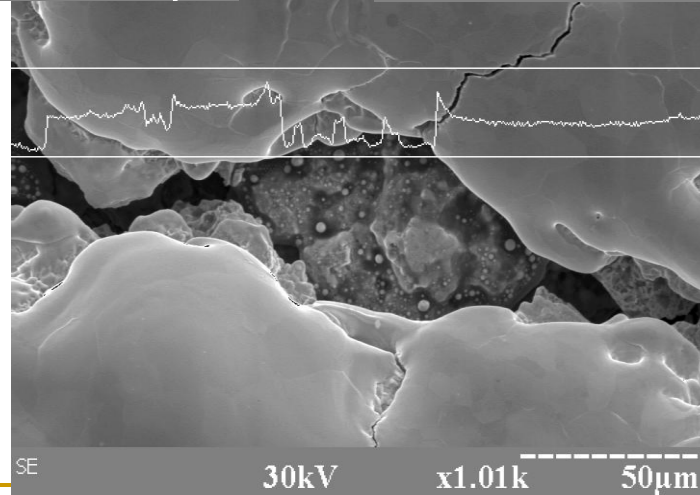
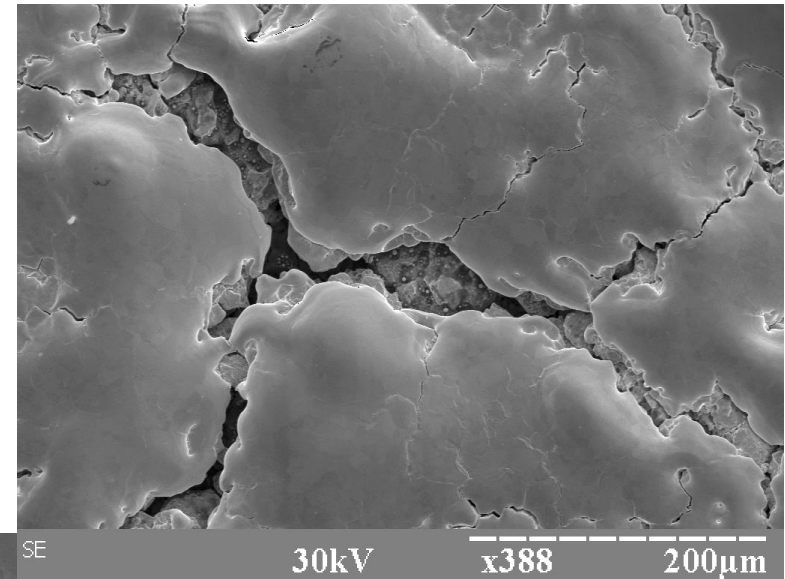
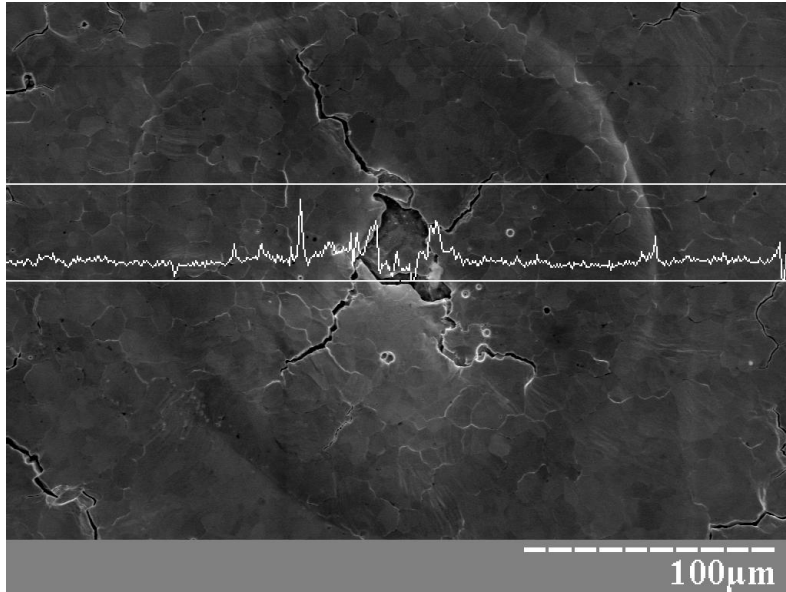
Plasma impacts with loads above the melting threshold cause the melting/dust particles splashing from the tungsten surface

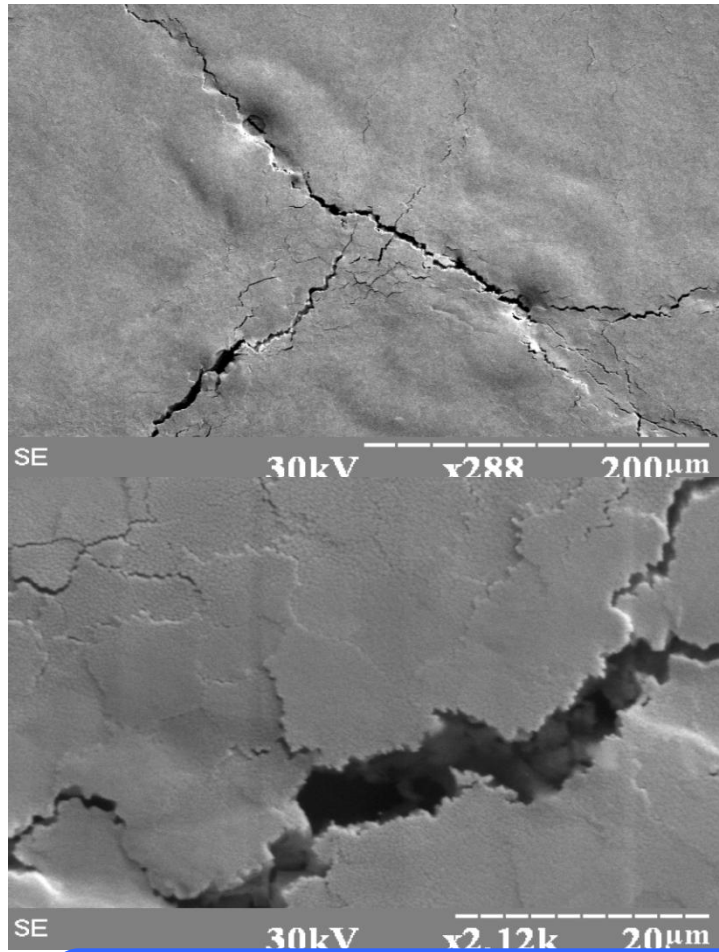


It was show that at least more than 95% of particles started from solid surface



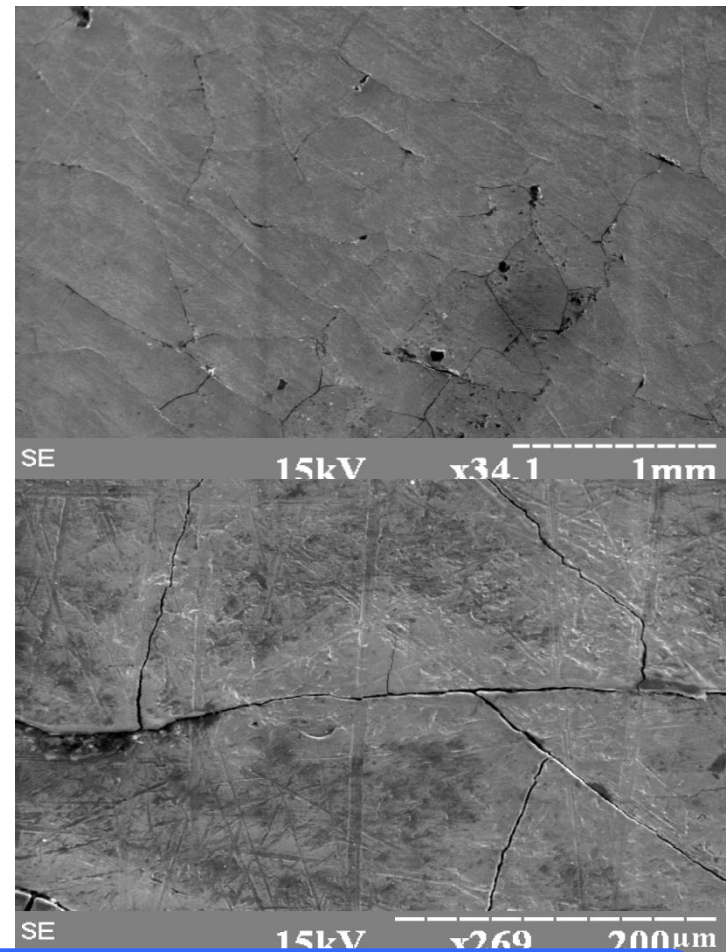
Blisters and bubbles of 100-300  $\mu\text{m}$  in size develop on exposed surface



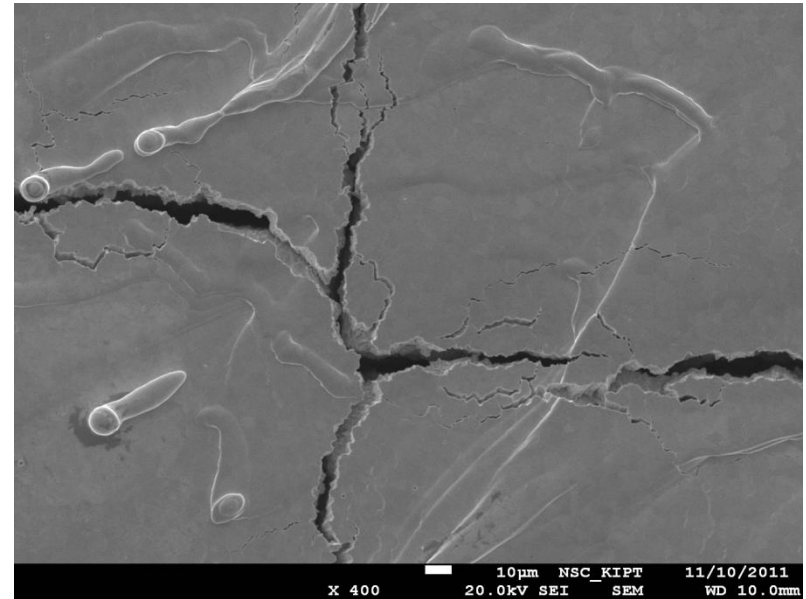
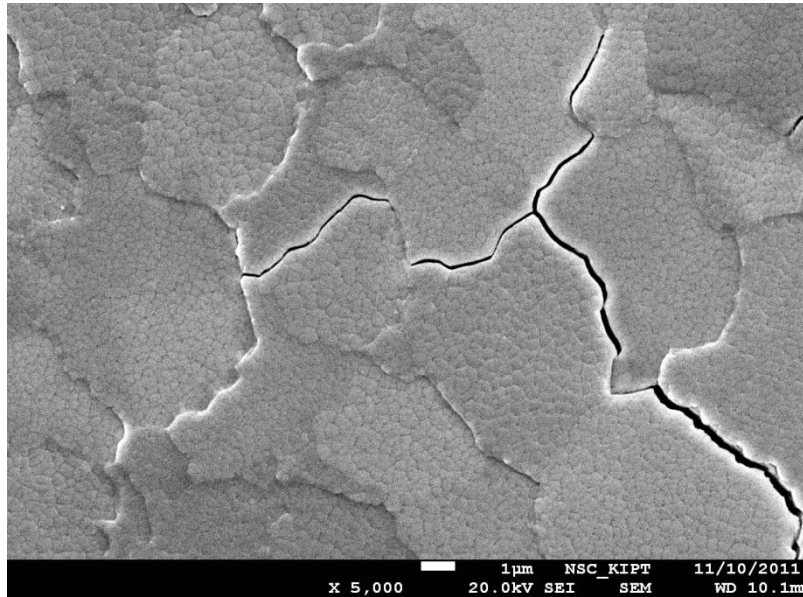


**Helium**

SEM views of tungsten surface after 10 PPA plasma pulses with heat load above melting threshold



**Hydrogen**

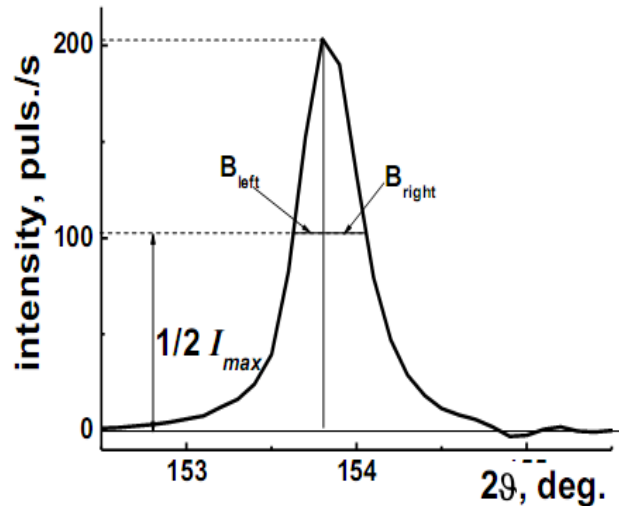


## Helium

**SEM views of tungsten surface after 10 MPC plasma pulses  
with heat load above melting threshold**



## XRD studies of exposed surfaces



The example of the definition of asymmetry parameter ( $\delta B = (B_{left} - B_{right}) / (B_{left} + B_{right})$ ) for diffraction line (400)

### Parameters of structure

#### Latticing parameter ( $a_0$ )

$a_0 < a_{ref}$  the surplus of vacancies;  $a_0 > a_{ref}$  the surplus of interstitial atoms ( $a_{ref} = 0.3165$  nm tungsten).

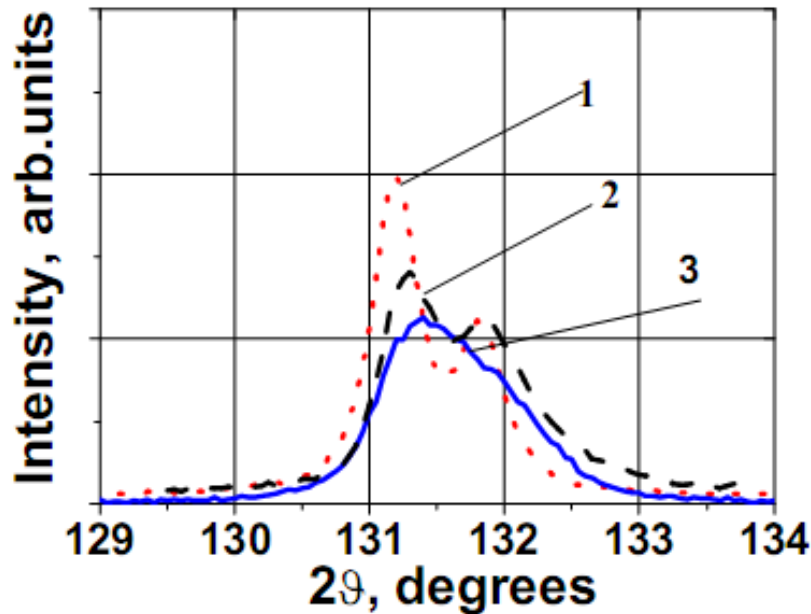
**B - the width of the profile** is proportional to the number of line defects (dislocations) in the structure.

**The asymmetry ( $\delta B$ )** is attributed by the presence of complexes of point defects. The sign of  $\delta B$  is caused by the type of defects: vacancies ( $\delta B > 0$ ) or interstitial atoms ( $\delta B < 0$ ).

#### Residual Stresses

Shift of profile position





*Profiles of diffraction maximum (321): initial state (1); exposed areas after 10 PPA pulses of 0.4 MJ/m<sup>2</sup> hydrogen (2) and helium (3) plasmas*

First plasma pulses lead to defects creation and structure degradation. The diffraction peak width of the targets is increased after 5 plasma helium pulses of 0.4 MJ/m<sup>2</sup>. Some slow diminution of peak width is observed when irradiation dose was increased twice). There is the small change of diffraction profile as a result of hydrogen plasma exposures of the same heat load. The repetitive heat load below melting threshold affects diffraction peak profile not strongly. This is due to creation of lower number of line defects.

# High power plasma streams is unique tool for surface modification

Combination of physical mechanisms:

ion bombardment,

heat load (melting, but no evaporation, thermal quenching),

shock waves,

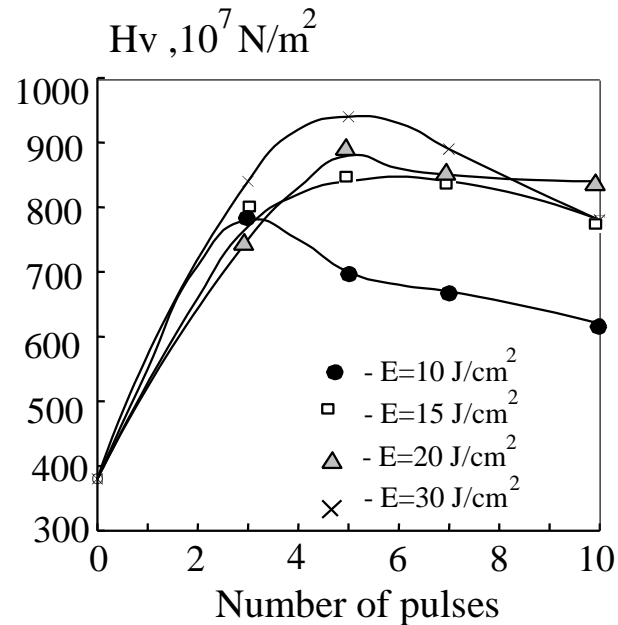
material alloying with plasma species,

mixing in molten stage.....

## Grades of industrial steels used for pulsed plasma processing

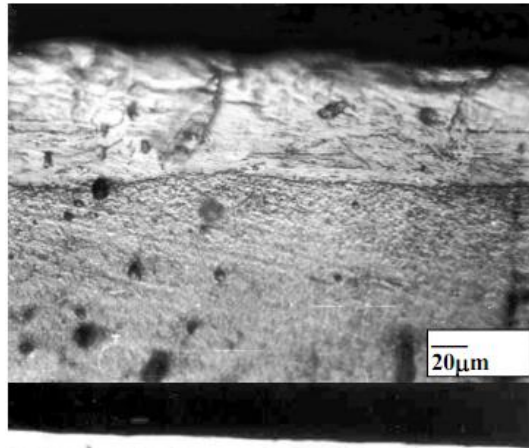
40H	Fe, 0.37-0.45%C, 0.17-0.37%Si, 0.5-0.8%Mn, <0.035%S, <0.035%P, <b>0.8-1.1%Cr</b> , 0.25%Ni, <0.2%Cu
12HN3A	Fe, 0.12-0.16%C, 0.37-0.56%Mn, <b>0.71%Cr, 3.3%Ni</b>
H12	Fe, 0.07-0.12%C, <b>11,0-13,0%Cr</b> , <0.6%Si, <0.6 %Mn, <0.025%S, <0.03%P
steel 45	Fe, <b>0.4-0.48%C</b> , 0.17-0.37%Si, 0.5-0.8%Mn
steel 10	Fe, <b>0.07-0.13%C</b> , 0.17-0.37%Si
ShH15	Fe, 1%C, 1.5%Cr, 0.17-0.37%Si, 0.5-0.8%Mn
65G	Fe, 0.62-0.7%C, 0.17-0.37%Si, 0.9-1.2%Mn

## Microhardness changes induced by pulsed plasma processing

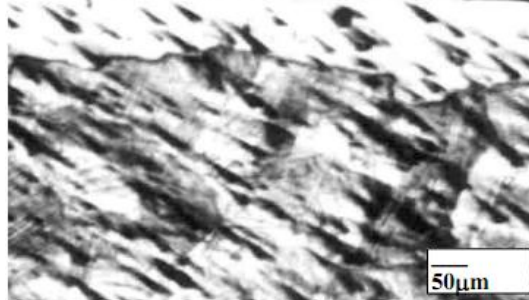


material	$H_v$ , initial	$H_v$ , proc		material	$H_v$ , initial	$H_v$ , proc
steel 10	200	510		65G	350	560
steel 45 quenched	370 400	796 870		12HN3A quenched	236 387	630 715
40H quenched	252 386	751 794		H12 quenched	312 553	510 593
37CrS4	352	742		ShH15	360	770

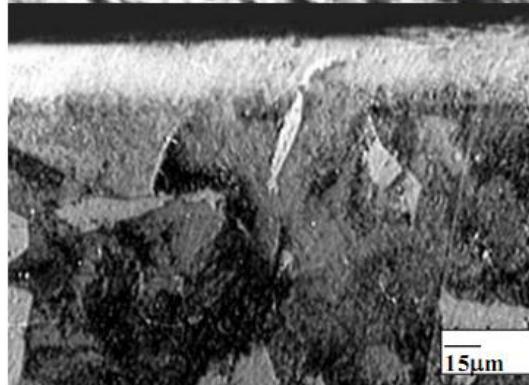
Microhardness and wear resistance were increased as result of plasma treatment



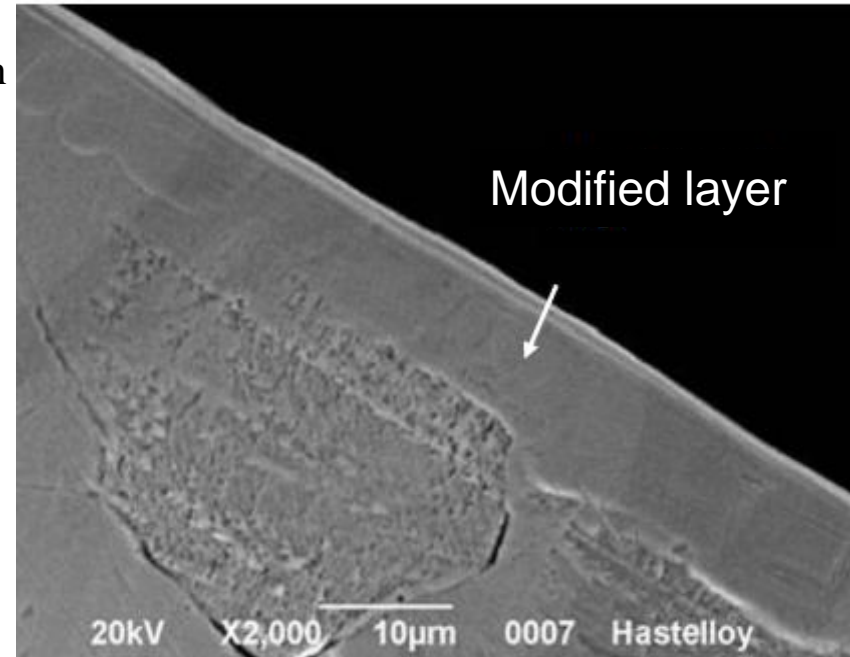
Al exposed by Oxygen



BT22 exposed by Helium



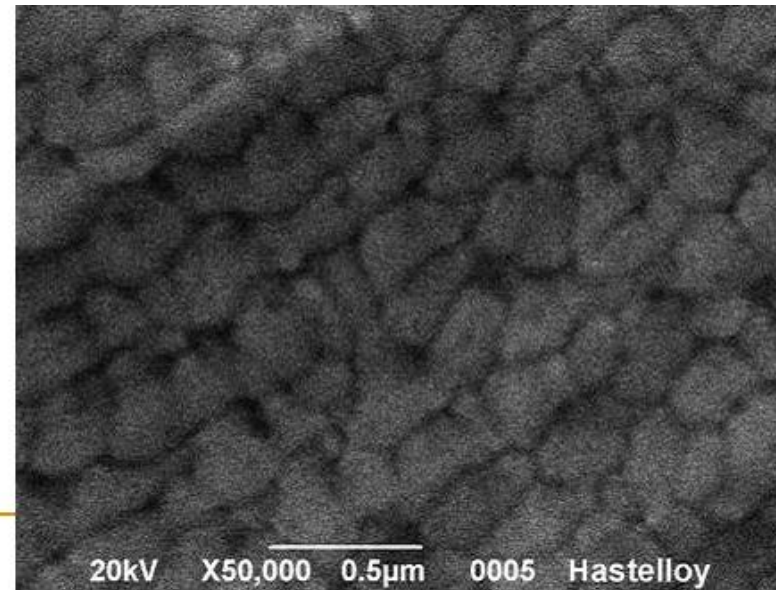
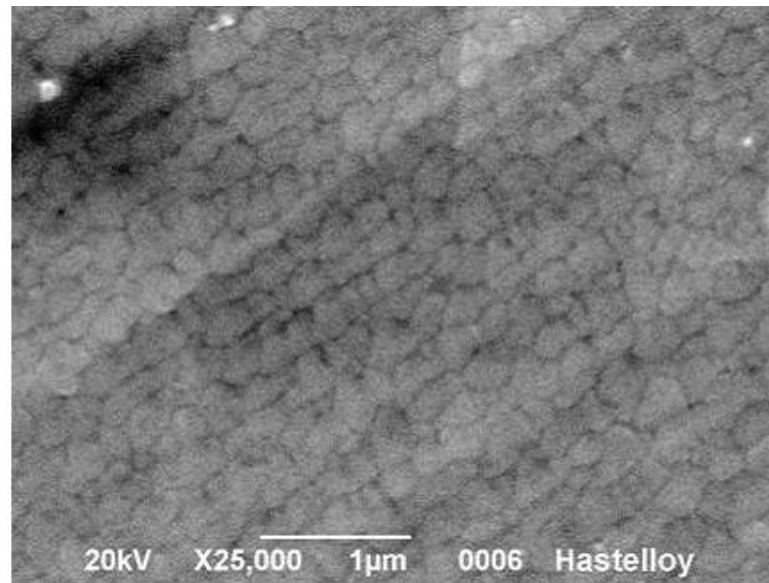
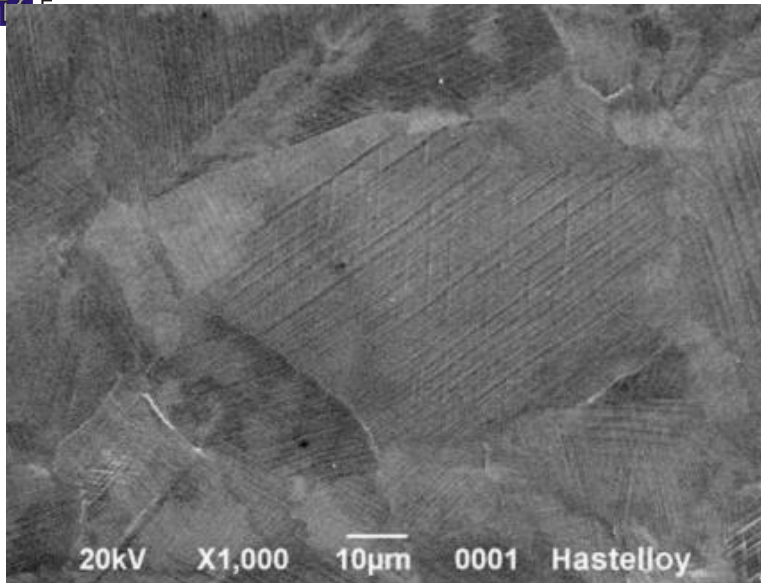
Steel 40X exposed by Nitrogen



Modified layer is not a coating, it is just modified substrate material (no problem with adhesion)

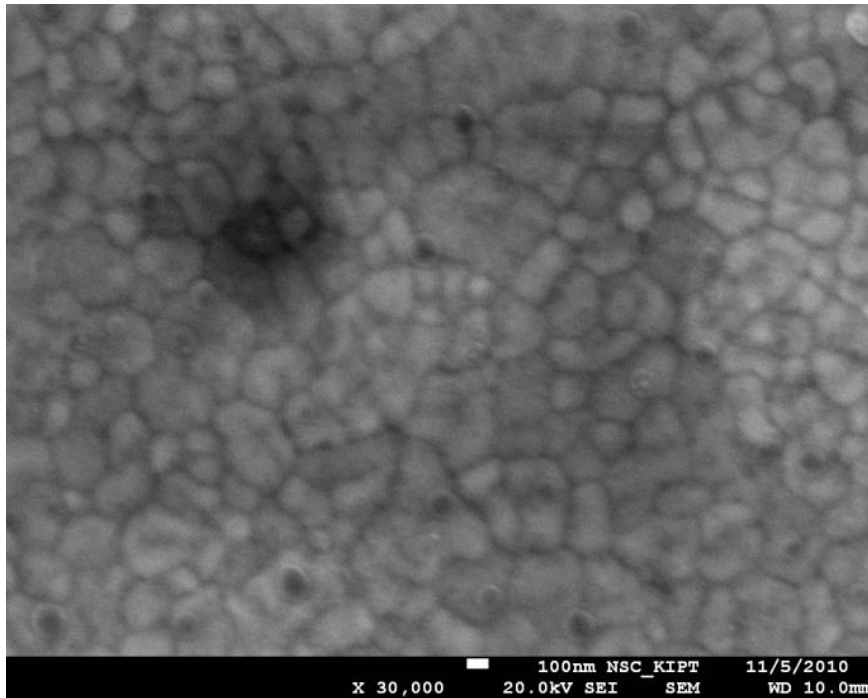


## Examples of ordered nanostructures Hastelloy Ni is base material

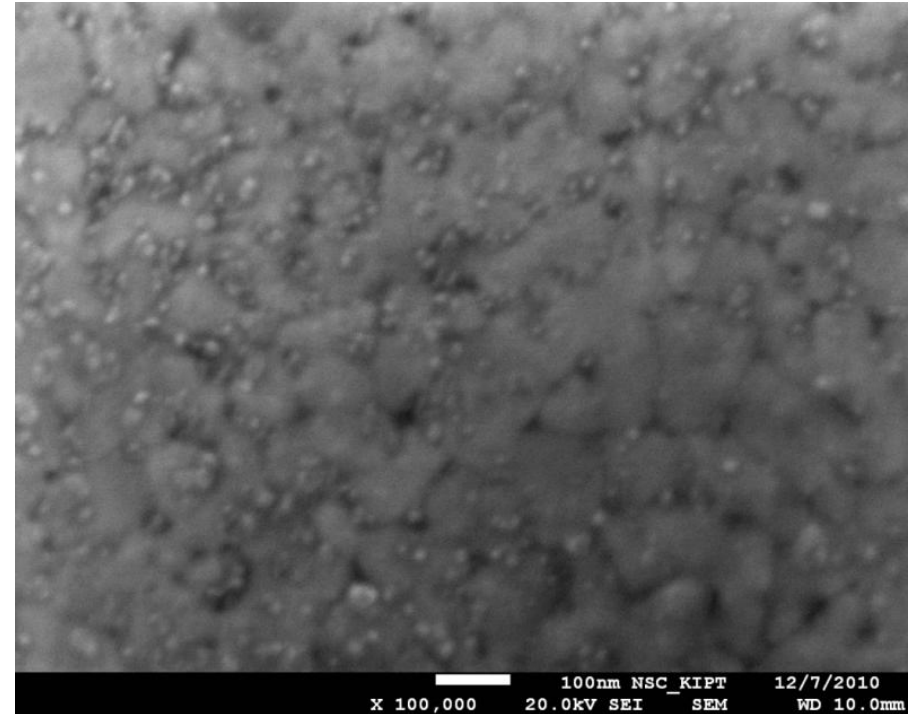


## Surface modification

$Q = 0.75 \text{ MJ/m}^2$ , 20 pulses



$Q = 0.45 \text{ MJ/m}^2$ , 100 pulses



Development of ordered submicron and nano cellular structures

## Alloying of surface layers of reactor steel EP-823 under pulsed plasma treatment

element	initial	№1 0.5 $\mu\text{m}$ Mo + 10 pulses He	№2 0.5 $\mu\text{m}$ Mo+ 5 pulses He+ 0.5 $\mu\text{m}$ Mo+ 5 pulses He	№3 5 pulses He	№4 1-1.5 $\mu\text{m}$ Mo + 5 pulses He
C	0,167	1,73	2,51	0,44	0,42
Si	1,3	1,25	2,5	2,3	1,65
Mn	0,82	0,064	0,37	1,4	1,48
Cr	12,1	1,15	6,24	18,15	30,25
Ni	1,7	0,14	0,6	1,75	1,6
<b>Mo</b>	<b>0,46</b>	<b>23,8</b>	<b>25,8</b>	<b>0,45</b>	<b>15,9</b>
W	0,62	0,58	0,66	0,68	0,56
V	0,34	0,024	0,16	0,6	0,6
Nb	0,24	0,25	0,22	0,2	0,2
N <sub>2</sub>	0,026	0,14	0,66	0,054	0,075
O	0,155	0,93	2,48	0,62	0,53
Ti	0,0046	0,4	0,15	0,005	1,38
Co	0,055	0,01	0,002	0,023	0,022

**Possibility of the modified surface layer creation alloyed with Mo or W for subsequent deposition of thick protective coatings.  
Aim: resistance to corrosion in salts and liquid lead.**

	initial	5 pulses- $9 \times 10^{18}$ ion/cm <sup>2</sup>	10 pulses - $1,8 \times 10^{19}$ ion/cm <sup>2</sup>
<b>EP-823 + Mo</b>	<b>400 kg/mm<sup>2</sup></b>	<b>450 kg/mm<sup>2</sup></b>	<b>480 kg/mm<sup>2</sup></b>
<b>EP-823</b>	<b>400 kg/mm<sup>2</sup></b>	<b>400 kg/mm<sup>2</sup></b>	<b>385 kg/mm<sup>2</sup></b>

This proposal focuses on 3 most critical issues relevant to plasma-facing materials of fusion reactor:

- (1) Characterization of various steel grades with respect to their response to intense plasma pulses and dust production under high flux plasma loads;
- (2) Modification and alloying of steels under pulsed plasma treatment aimed to improvement it working characteristics.
- (3) Comprehensive studies of hydrogen/helium retention in reduced activation steels modified by pulsed plasma streams in comparison with virgin materials, i.e. without plasma treatment.