Experiment report: 20210372 “Kinetics of He bubbles growth in W(110)”

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Introduction

Due to its high melting point, low sputtering yield and low retention of hydrogen-isotopes, tungsten (W) has been chosen as the material for the divertor of ITER. Helium (He) irradiation of tungsten produces sub-surface bubbles that will affect the tungsten properties. Hence, understanding the formation and evolution of Helium bubbles is crucial.

During this study, 2 keV He bombardment at 1000°C, followed by Ultra-High-Vacuum (UHV) annealing up to 1500°C have been performed on single crystalline W(110), W(100) and W(111) samples. 2 keV incident energy of He is higher than He displacement energy threshold of W (538 eV) and the 1000°C bombardment temperature allows the migration of bulk vacancies. Also, a 2keV He bombardment at RT followed by UHV annealing up to 1500°C has been performed on W (110).

Our main goal was to investigate the early steps of He bubbles formation, and the bubble size and shape evolution under annealing at high temperatures for different W crystallographic orientations.

Sample preparation

In CINaM laboratory, all samples were prepared following a multi cycling procedure that consists of an O_2 ($P \approx 10^{-6}$ Torr) annealing of about 1000°C to remove carbon impurities, and a high temperature UHV flash up to 2000°C to remove the tungsten oxide on the surface. The surface contamination was checked by Auger spectrometer analysis.

For each sample, a 5nm-thick Au layer has been deposited as a protective layer to avoid surface contamination during transportation to the synchrotron.

He bombardment

- **Au layer desorption**

Prior He bombardment, the protective Au layer is removed by heating the sample to 1000°C under UHV. Desorption of Au has been monitored by GIXD measuring the diffracted intensity along the in-plane radial scan h00 (see Figure 1 GIXD diffractogram along h00 in-plane direction of Au/W110 at 1000°C. Black line is initial time. Chronological order is: black, red, green, purple, yellow and brown. Peak h = 2 corresponds to W and peak h = 2,17 corresponds to the Au layer.). The Bragg peak of tungsten is visible at index h = 2. The peak at h=2.17 is due to the Au layer on the surface. By heating the sample to 1000°C, Au desorbs as the peak intensity at h = 2.17 decreases.

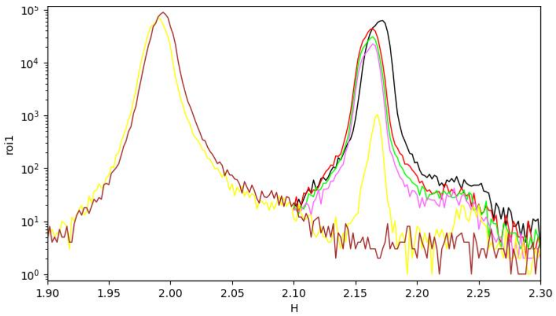


Figure 1 GIXD diffractogram along h00 in-plane direction of Au/W110 at 1000°C. Black line is initial time. Chronological order is: black, red, green, purple, yellow and brown. Peak h = 2 corresponds to W and peak h = 2,17 corresponds to the Au layer.

- **In**
- **In situ GISAXS under He bombardment (1000°C, 2 keV)**

In-situ GISAXS measurements during **2 keV** He bombardments on **W (110), W (100), W (111) held** at **1000°C and at RT (W (110))** have been performed. Bombardment current is I_B ≈ **20 µA** leading to a flux of ≈ **2,5x10^{17} He.m^{-2}.s^{-1}**. The total dose of He implanted is ≈ **1,0x10^{22} He.m^{-2}**.
The real time GISAXS patterns during He bombardment and annealing were measured along crystallographic axes and at different incident angles. After final implantation and/or temperature annealing GISAXS patterns where collected over a large range of azimuth (ω) and for different incident angles to discriminate between surface and bulk features.
In this report will be mainly presented GISAXS images taken at α = 0.4° on crystallographic axes.

<u>W (100) bombarded at 1000°C :</u>

The kinetic of the bubble nucleation and growth is studied by *in-situ* GISAXS measurments. Figure 2 shows three GISAXS images taken with the X-ray beam perpendicular to the h00 direction, before (a), at initial stage (b) and at the final stage of the He bombarmdent (c). The reference before bombardment (a) shows a narrow specular rod meanning that surface morphology is composed of large flat areas. At the left of the specular rod, intensity bonce corresponds to impurity reconstruction of the surface and can be associate to the carbon reconstruction superstructure R(5x1) on W (100). The first stage of bombardment (b) shows more diffused intensity due to the creation of spherical He bubbles (isotropic diffusion in all the directions), the average distance between bubbles can be determined thanks to the intensity bounces from either part of the specular rod. The final stage (c) shows a broad specular rod due to formation of He bubbles and damages (roughness) on the surface. The rod formed at an angle of 44.7° with the pecular rod is attributed to the (10-1) facet. Two hypothesis could explain the creation of (10-1) facets during He irradiation (1) the spherical He bubbles change their shape into facetted-bubbles (10-1). (ii) The (10-1) plan is due to the surface roughness.

Before bombardment First stage of bombardment Final stage of bombardment

W (100)

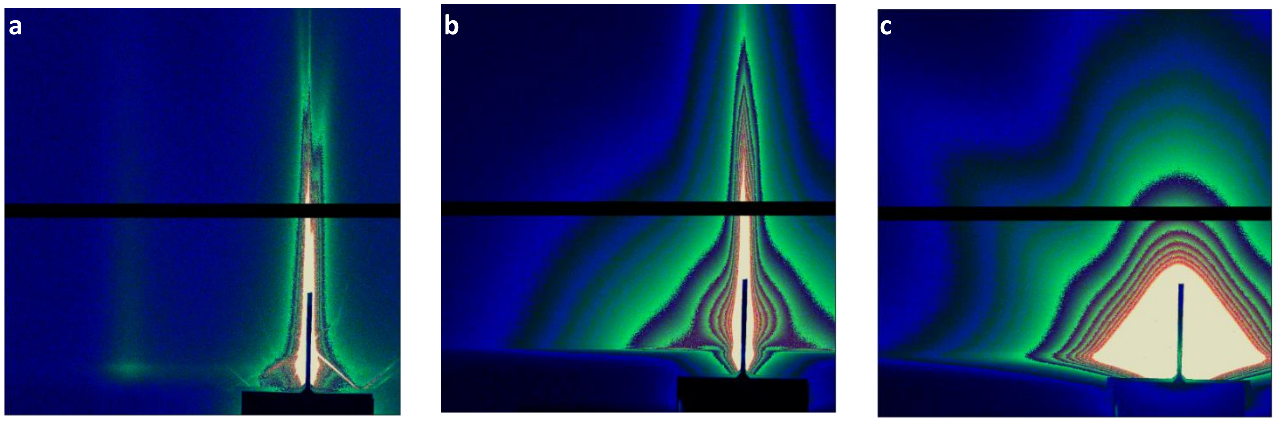


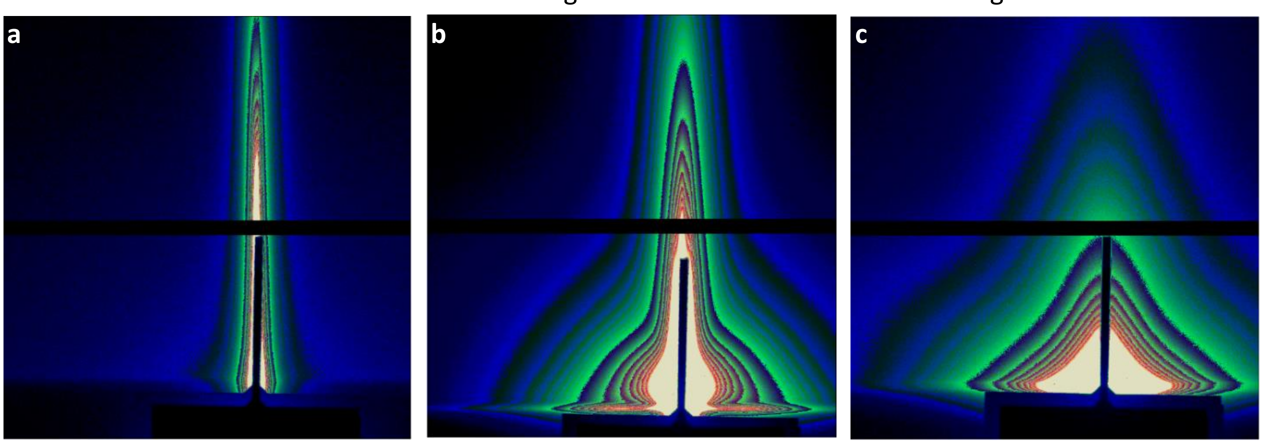
Figure 2: In-situ GISAXS measurements at different stages of 2 keV He bombardment at 1000°C on W (100). The X-ray beam is perpendicular to the h00 direction and the incident angle is α = 0.4° (critical angle). Narrow specular rod of reference (a) indicates a flat and well-defined surface. On first stage measurements (b), broad scattering indicates the creation of He bubbles. Broad and intense scattering at the final stage (c) indicates the growth of large He bubbles and probably large morphology modifications of the surface.

<u>W (110) bombarded at 1000°C</u>

As for the W (100) we have studied the W (110) under the same conditions: the reference GISAXS pattern before bombardment (a) shows a well-defined surface. (b) Spherical bubbles are generated during the first stages of bombardment. The final stage of bombardment (c) shows a the width increase of the specular rod which means the surface is rough due to bombardment damages. Also a diffuse scattering rod with an angle of 63.5° with respect to the surface normal can be associated to (011) facets: Either the He bubbles are facetted and/or the surface rearrangement exposes (011) planes.

Before bombardment — First stage of bombardment — Final stage of bombardment

W (110)



<u>W (111)</u>

Figure 3: In-situ GISAXS measurements on different stages of 2keV He bombardment at 1000°C on W (110) on hh0 direction with α = 0.4° Narrow specular rod of references (a) indicates well-defined crystallographic structure. On the first stage measurement (b), maximums of intensity from either side of the specular rod indicate the creation of a periodic distribution of bubbles Broad specular rod on the final stage (c) indicates the loss of well-defined crystallographic structure. The rod with an angle of 63.5° visible in the final stage is associate to (011) plane.

<u>bombarded at 1000°C</u>

Identical procedure has been done on W (111) (Figure :4 In-situ GISAXS measurements on different stages of 2keV He bombardment at 1000°C on W (111) on 0h0 direction4). The particularity of W (111) sample is that the sample presents initial facets that can be explain because of the instability of the (111) face of bcc structures.

Just before bombardment — First stage of bombardment — Final stage of bombardment

W (111)
bombarded at
1000°C

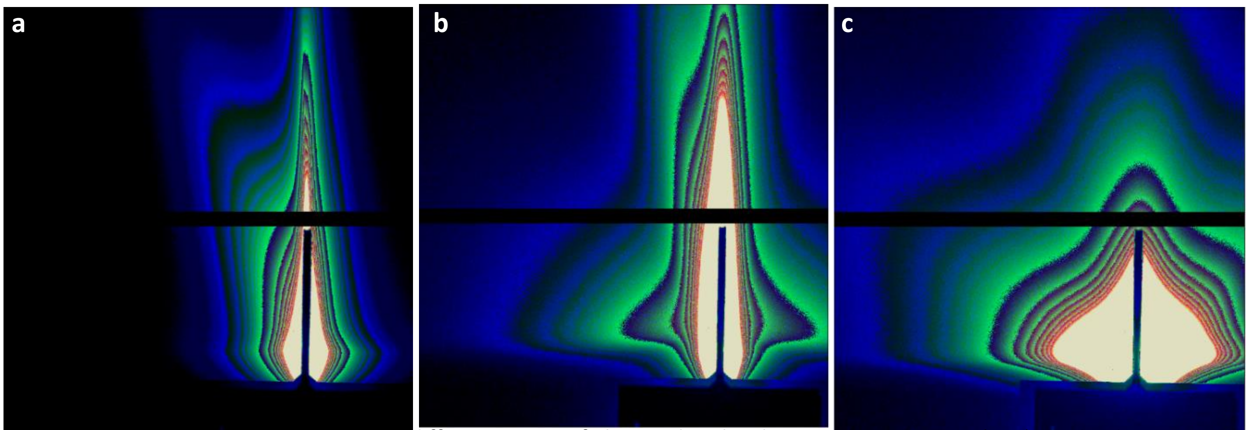


Figure :4 In-situ GISAXS measurements on different stages of 2keV He bombardment at 1000°C on W (111) on 0h0 direction.

UHV annealing

After He bombardment, the W(110), W(100) and W(111) single crystals were annealed up to 1500°C.

<u>W (100) bombarded at 1000°C</u>

GISAXS measurements taken in <h00> direction are presented in Figure 5, the specular and the facet (10-1) rods are more and more narrow with the increasing of temperature. It can be interpreted as following:

1) The surface is healing and is recovering its well-defined crystallographic structure
2) Bubbles coalescence generates large facetted bubbles.

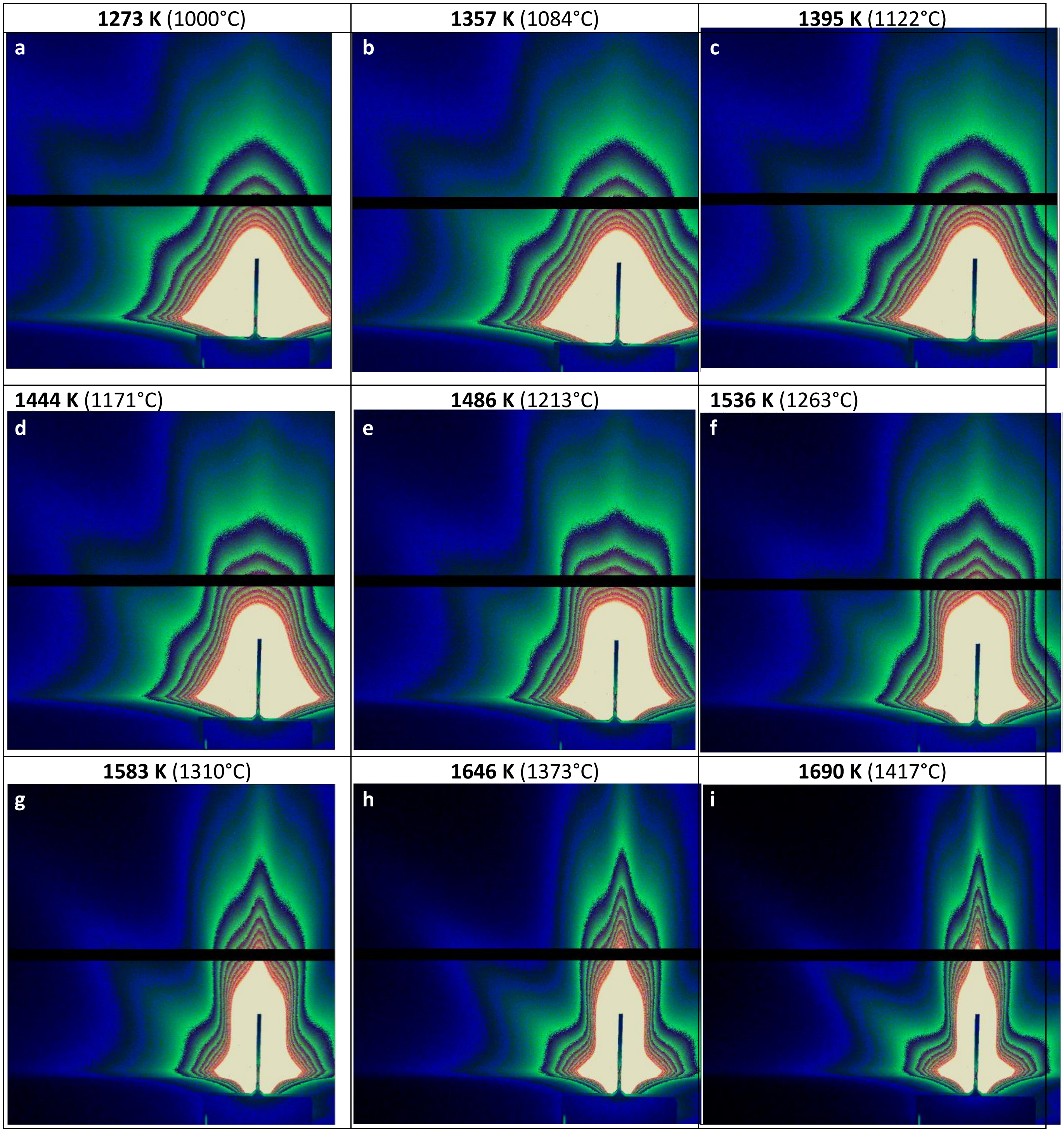


Figure 5: GISAXS measurements of W(100) in h00 direction with α = 0.4°, at 1000°C (a), 1084°C (b), 1122°C (c), 1171°C (d), 1213°C (e), 1263°C (f), 1310°C (g), 1373°C (h) and 1417°C (i). Surface healing and bubbles coalescence are occuring.

<u>W (111), W (110) implanted at 1000°C and W (110) implanted at RT</u>

Surface healing is also observed on W (111) and (110) implanted either at 1000°C or at RT

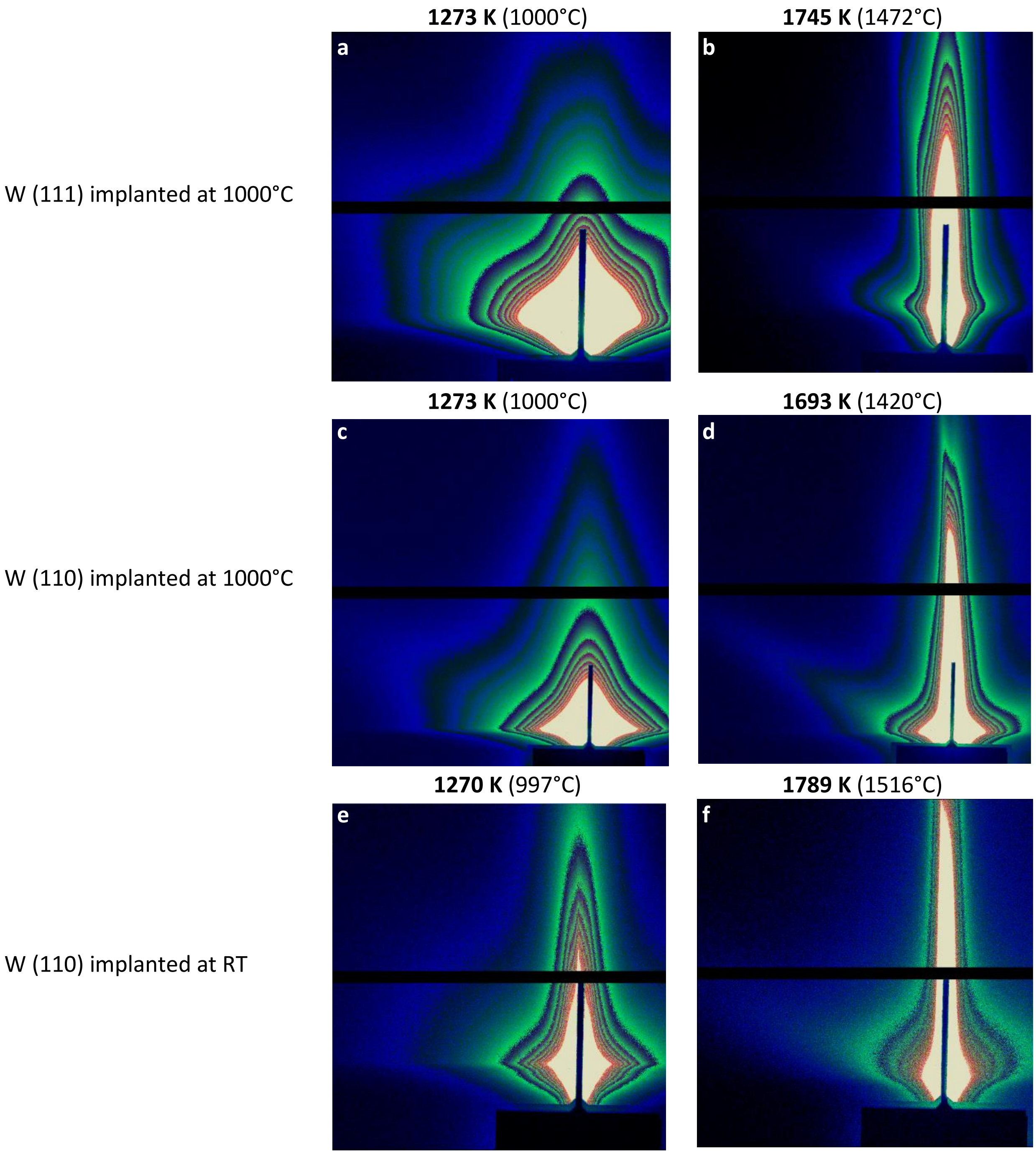


Figure 6 GISAXS measurements of UHV annealing of W(111) in 0h0 direction at 1000°C (a) and 1072°C (b) ; W (110) implanted at 1000°C in hh0 direction at 1000°C (c) and 1420°C (d) and W(110) implanted at RT in hh0 direction at 997°C (e) and 1516°C (f) with α = 0.4. Surface healing and bubbles coalescence are making the specular rods and the facet signals narrower with higher temperature.

Post mortem analysis

The major concern of the study is to be able to decorrelate bubbles and surface signals in the GISAXS patterns. In this aim, surface characterizations by SEM and AFM have been performed (Figure 7 and 8). They show rough surfaces and many holes with a depth around 10 nm.

TEM analysis are planned to confirm the average bubble size and bring out a size distribution and bubble density.

Finally, *IsGISAXS* simulations are planned to complete the description of the early stages of He bubbles formation, and their evolutions regarding the temperature and the crystallographic orientation of the sample.

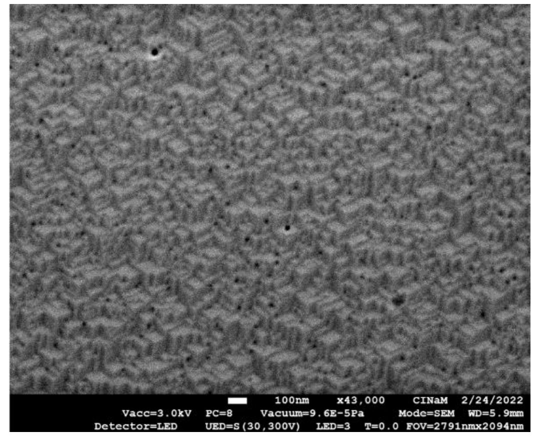


Figure 7 SEM analysis of W (111). Holes and reconstruction patterns are visible on the surface.

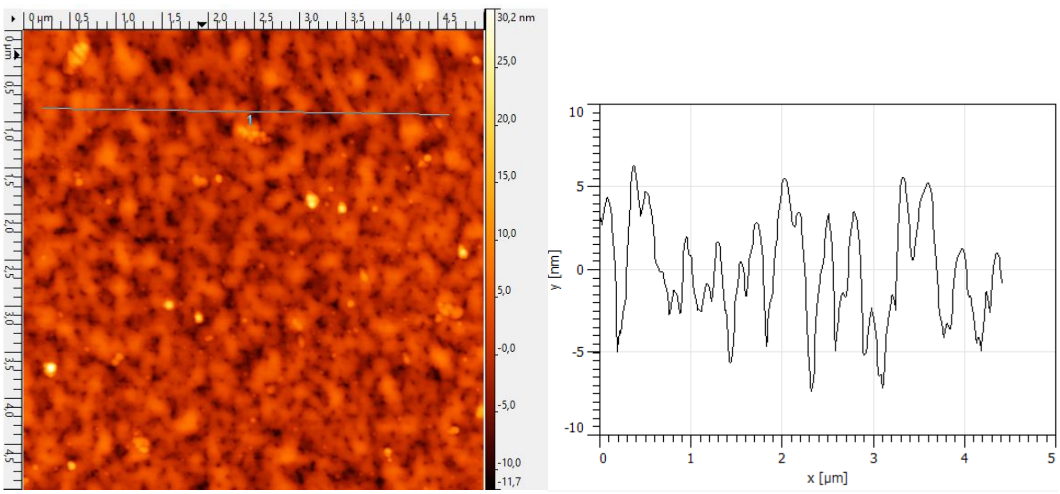


Figure 8 At left: AFM Analysis of W(111) the blue line is the depth profile shown in the right part. 10nm holes are observed.

In conclusion during our first experiment on INS2 setup (BM32-ESRF) in January 2022 (N°: 32-03-752), we have measured by GISAXS the size and shape evolution of He bubbles in W single crystals held at 1000°C during continuous implantation of 2 keV He ions and for different surface orientations, *i.e.* W(110), W(100) and W(111). Preliminary data analysis shows the growth of facetted He bubbles. **The set of data obtained clearly establishes the potential of the experimental setup to study He bubble formation in a wide range of temperatures (RT to 1400°C), He fluence (10^{19} – 10^{22} He ions/m^2) and He ion energy (0.3 keV – 5 keV).**

It is clear now that experiment closer to operating conditions of tokamaks: i.e. irradiation temperature (300°C-500°C) and Helium implantation energy below the energy threshold (500 eV) for damage are achievable.