# ***Experiment report:*** MA-5393 "Kinetics of He bubbles growth in W(110): impact of W temperature during irradiation and He incident energy"


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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, 400 eV He bombardment under constant temperature conditions (RT, 500K, 900K and 1200K), have been performed on single crystalline W(110) samples. 400 eV incident energy of He is below He displacement energy threshold of W (538 eV) as it is the case in tokamaks. Vacancy mobility starts at 500K in W, hence temperatures were chosen to study the impact of the vacancies mobility on the He bubble growth process **Our main goal was to investigate the early steps of He bubbles formation, and the bubble size and shape evolution under experimental conditions close to tokamaks operation conditions.**

## Sample preparation

In CINaM laboratory, all W single crystalline samples were prepared following a multi cycling procedure that consists in an $O_2$ ($P \approx 10^{-6}$ Torr) annealing at 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 ESRF synchrotron.

## He bombardment

- **Au layer desorption**

Prior He bombardment, the protective Au layer is removed by heating the sample to 1100°C under UHV. Desorption of Au has been monitored by GIXD measuring the diffracted intensity along the in-plane radial scan 0k0 (see Figure 1 GIXD diffractogram along 0h0 in-plane direction of Au/W110 at 1100°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 k = 2. The peak at k=2.17 is due to the Au layer on the surface. By heating the sample to 1100°C, Au desorbs as the peak intensity at k = 2.17 decreases and finally vanishes.

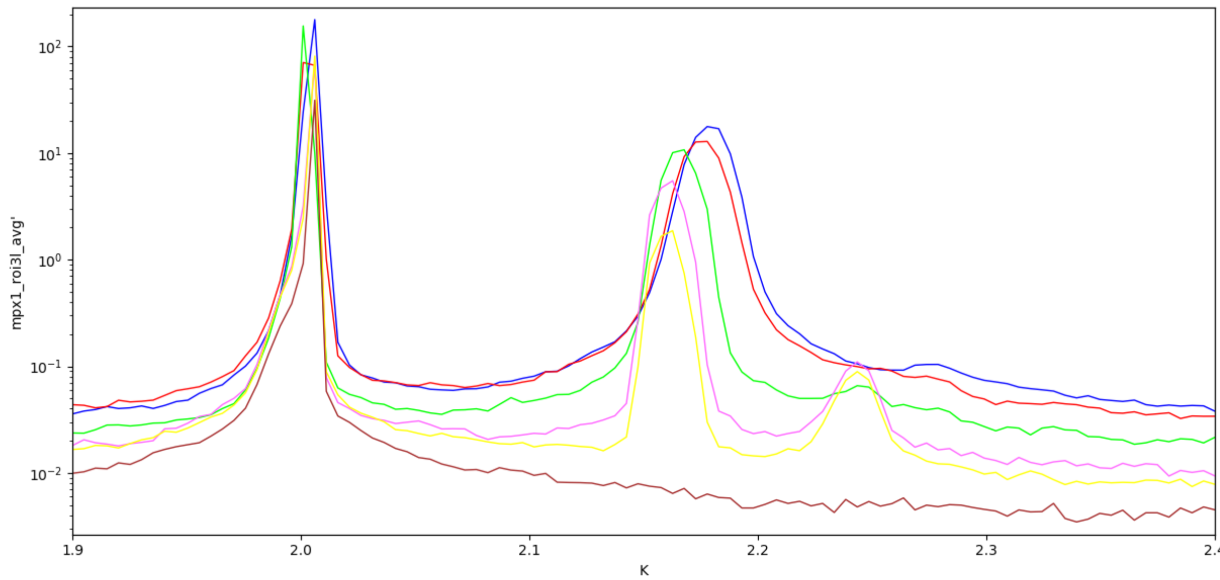



*Figure 1 GIXD diffractogram along 0h0 in-plane direction of Au/W110 at 1100°C. Black line is initial time. Chronological order is: black, red, green, purple, yellow and brown. Peak h = 2 corresponds to W and peak h = 2,17 corresponds to the Au layer.*

- **He bombardment (400 eV)**

*In*-situ GISAXS measurements during **400 V** He bombardments on **W (110) held** at **1000°C, 600°C, 250°C** and at **RT** have been performed. Bombardment current is $I_B$ ≈ **9 µA** leading to a flux of ≈ **1.05x10$^{17}$ He.m$^{-2}$.s$^{-1}$**. The total dose of He implanted is ≈ **4.30x10$^{21}$ He.m$^{-2}$**.

The real time GISAXS patterns during He bombardment were measured along crystallographic axes and at different incident angles. After implantation, 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 are mainly presented GISAXS patternd recorded at α = 0.2° on crystallographic axes.

W (110) bombarded at 1000°C :

The kinetic of the bubble nucleation and growth is studied by *in-situ* GISAXS measurements. Figure 2 shows two GISAXS images taken with the X-ray beam perpendicular to the hh0 direction, before (a), and at the final stage of the He bombarmdent (b). The reference before bombardment (a) shows a narrow specular rod meaning that the surface morphology is composed of large flat areas. The final stage (b) shows a broader specular rod that means damage on surface are present. It is expected to be holes formed by bubble bursting and not due to irradiation dammage, because the implantation energy of He is below the displacement energy theresold of W by He. It is worth to notice that in january 2022 during our previous experimental run, where implantation were done with 2 keV energy (above the displacement thresold energy), the specular rod was even broader because of the generation of implantation damages on the surface. So, the modification of the surface morphology is presumably due to the generation of holes formed by bubble bursting. Also, the final stage (b) shows a lateral rod formed at an angle of 60° with respect to the specular rod. It is attributed to the (10-1) facet. The creation of (10-1) facets during He irradiation, is presumably due the facetted shape of He bubbles as observed during the previous run in january 2022.

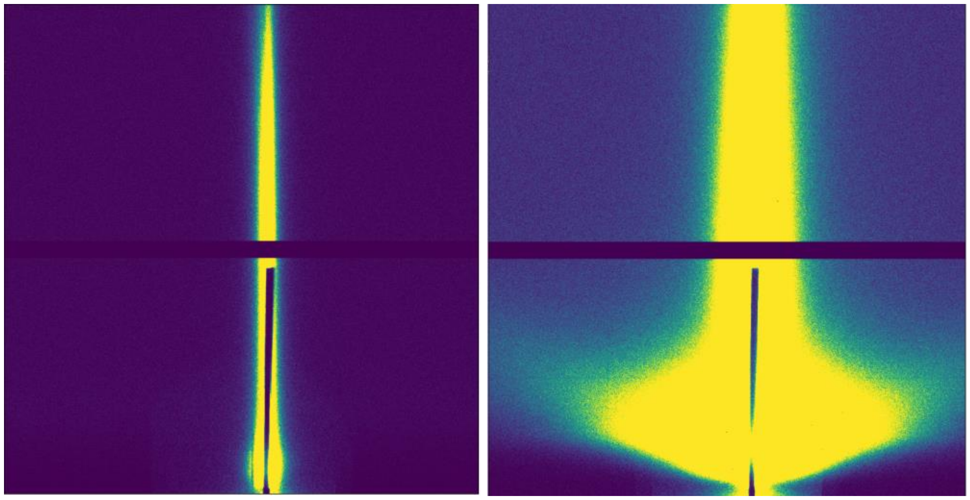
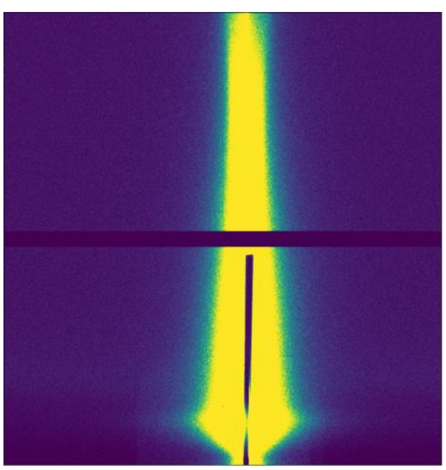
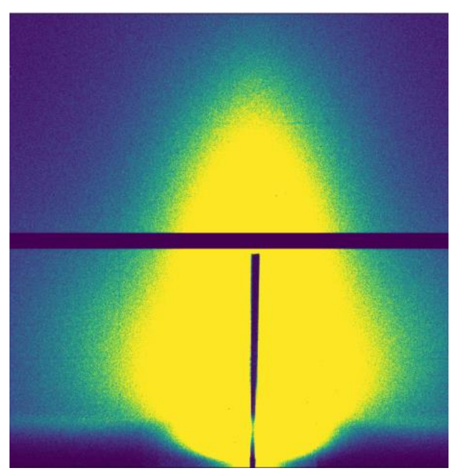


*Figure 2: In-situ GISAXS measurements at different stages of 400 eV He bombardment at 1000°C on W (110). The X-ray beam is perpendicular to the hh0 direction and the incident angle is α = 0.2° (critical angle). Narrow specular rod of reference (a) indicates a flat and well-defined surface. Broader specular rod and lateral rod scattering at the final stage (b) indicates the growth of large He bubbles and probably large morphology modifications of the surface.*

<u>Impact of the temperature:</u>

Implantations of 400 eV helium were performed at 1000°C, 600°C, 250°C and RT. Figure 3 shows the reference GISAXS pattern before implantation (left) and the final stage after implantation (right) for all temperatures. The well-defined flat surface seen on reference patterns (left) disappear during He implantation presumably due to bubble bursting. The loss of the flat surface is all the more important than the temperature is low. This could be explained by with the fact that the temperature is not high enough to allow W mobility to heal the surface. In addition , faceting of He bubble is clearly visible only in the case of 1000°C implantation. Hence, equilibrium facetted shape of He bubbles may not be available for low temperature implantation due to the lack of W mobility.

| | Before implantation | Final stage of implantation |
|---|---|---|
| RT |  |  |

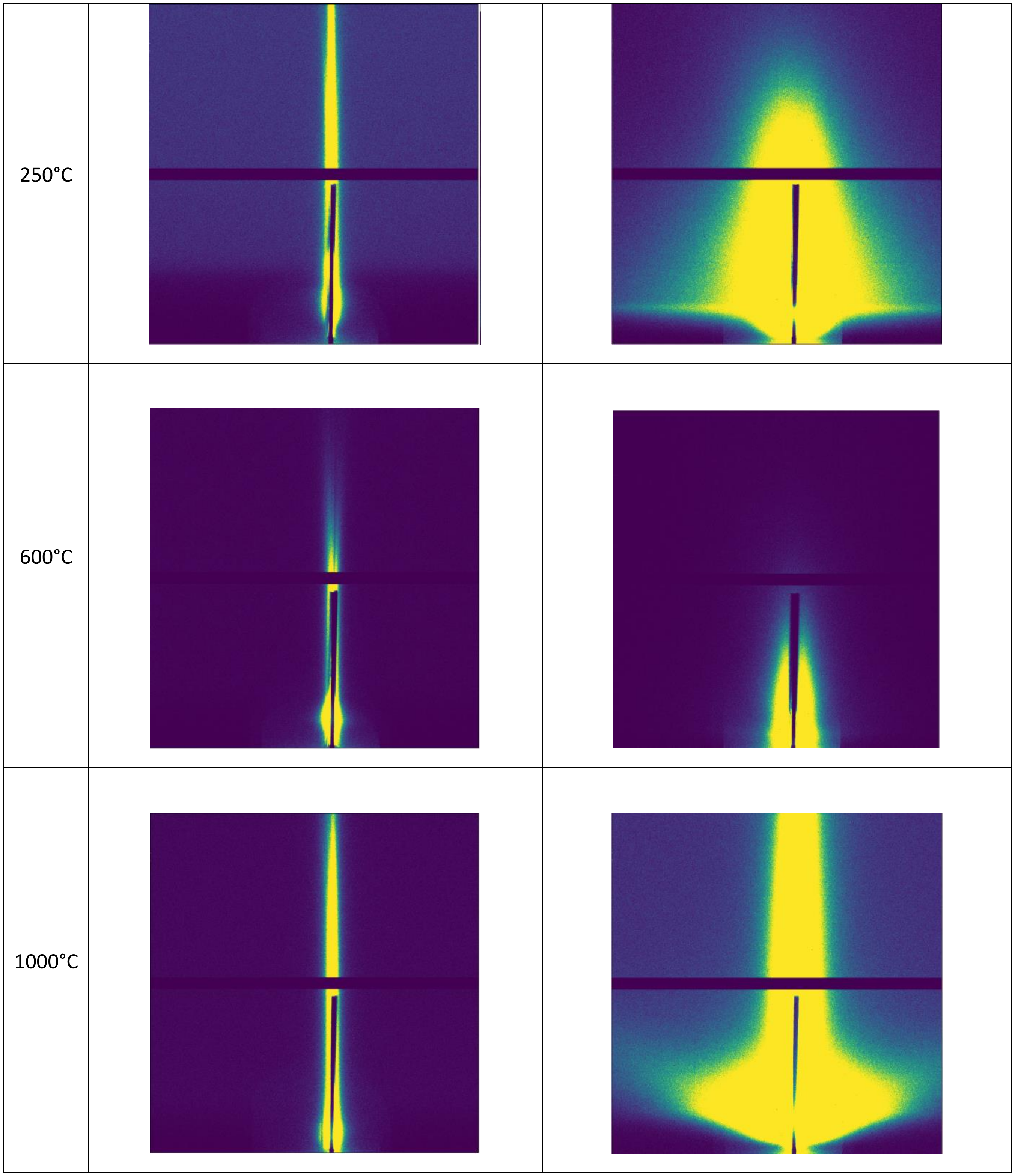



*Figure 3: In-situ GISAXS measurements on different stages of 400 eV He bombardment at different temperature on W (110) on hh0 direction with α = 0.2° Narrow specular rod of references (left) indicates well-defined flat surfaces. Broad specular rod on the final stage (right) indicates the loss of well-defined surfaces presumably due to bubble bursting. The rod with an angle of 60° visible in the final stage is associated to (10-1) plane on the surface of He bubbles.*

## 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 purpose, surface characterizations by SEM and AFM are planned. TEM analysis would confirm the shape of He bubble, facetted shape are expected for 1000°C implantation and spherical shape for 600°C, 250°C and RT implantations.

Finally, *IsGISAXS* simulations are planned to complete the kinetic description of the He bubbles formation, and their evolution regarding the temperature.

In conclusion during our second experiment on INS2 setup (BM32-ESRF) in September 2022, we have measured, by GISAXS, the size and shape evolution of He bubbles in W single crystals held at 1000°C, 600°C, 250°C and RT during continuous implantation of 400 eV He ions. Preliminary data analysis shows the major impact of temperature on the bubble growth, facetted He bubbles were formed for 1000°C and spherical bubbles were formed for lower temperatures. **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 experiments closer to operating conditions of tokamaks considering oxidized W samples are achievable.**