ESRF	**Experiment title: Stacking faults in homoepitaxy on Ir(111): propagation, effect of impurities and stability**	**Experiment number**: SI 1534
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Report:

X-ray surface diffraction was used *in situ* to investigate the homoepitaxy on Ir(111), with special emphasis on the propagation and healing of stacking faults. A first goal was to achieve both qualitative and quantitative understandings through the measurement and analysis of crystal truncation rods (CTRs) [1], with a tunable surface sensitivity, during Ir deposition and through progressive subsequent annealing steps. Understanding the influence of CO and O_2 partial pressures on the stacking fault formation processes was a second main objective. With respect to our previous scanning tunneling microscopy (STM) investigations, which revealed surface manifestations of new atomic processes involved in Ir(111) homoepitaxy [2,3], complementary characterization was expected. Such a *comprehensive* characterization could not be achieved, due to difficulties in preparing the very demanding experimental ultra-high vacuum (UHV) setup which was, to a large extent, specifically designed for the purpose of the experiment. Consequently, a crucial lack of time was experienced and the pressure in the growth chamber could not reach the required very low 10^{-10} - 10^{-11} mbar range, which was mandatory to study the influence of CO and O_2 gases. However, we would like to point out that most experimental problems were at the end fixed, allowing to perform a single set of measurements, including the analysis of the growth of 70 Ir monolayers (ML), the subsequent progressive annealing of this layer, as well as its sputtering.

The growth was performed onto a bulk Ir sample. This was mounted on sample-holder especially designed for the requirements of the measurements, i.e. allowing (i) high voltage to be applied for electron-beam heating of the sample, (ii) thermal regulation thanks to permanent temperature measurement using a thermocouple, and (iii) fixation to the goniometer head for diffraction measurements. The sample first experienced severe treatment through melting of its backside with the Ta parts of the sample-holder, probably due to sudden increase of the thermal charge. Therefore the sample holder was then fully redesigned using W parts, and allowing fixation of the non-standard sample shape following the sample backside melting. Soon after both the ion gun and its power supply, which were mandatory for sample surface preparation, showed successively critical failures, causing a new venting of the UHV failure for replacement with a new ion gun. Finally, the bake-out of an isolated part of the UHV system, namely a spare Ir evaporator prepared in case the main one would experience problems, caused a general shut-down of the apparatus power supply, leading to an undesired venting of the UHV chamber. All these successive major issues required each time 2 full days UHV preparation, including bake-out. Taking into account the initial UHV chamber preparation, and keeping in mind that these issues occurred partly during beam-time, we were at the end left with the last 2 full days of

measurements. At this point, and before any Ir deposit, the base pressure in the chamber was as high as 3.10^{-10} mbar with the cooling trap filled with liquid nitrogen.

Ir deposition was carried out at T_s = 350 K, with a $2.6.10^{-10}$ mbar base pressure, using a home-made Ir evaporator consisting of a heated Ir wire carefully calibrated by measuring anti-Bragg oscillations. Measurements were performed using a 18 keV x-ray beam and a grazing incidence setup with incident angle, $\alpha_i = 0.273°$, i.e. the critical angle for total external reflection, yielding a ~ 6 nm penetration depth. Several ω-scans and scans along CTRs were systematically and continuously measured during deposit, providing qualitative information regarding the sample roughness, size, ordering and relative proportions of faulted and regularly stacked crystal regions. Additionally, full sets of CTRs, meeting the standard criterion for quantitative structural analysis [4], were measured at several key-stages of the Ir deposit (Θ), namely at Θ = 0, Θ = 0.76, and Θ = 70 ML.

Propagation of stacking faults and other defects

Figure 1(a) shows the [01L] and [11L] CTRs before and after the 70 ML Ir deposit. For Θ = 0 ML, the fully regular Ir stacking (ABCA...) yields the (012), (110) and (113) Bragg peaks. From Θ = 0 ML to 70 ML, one observes a dramatic decrease of the anti-Bragg [1 1 1.5] intensity, which underlines the roughness increase after Ir deposition. New peaks are also visible, namely peaks (1), (2), and (3), as an evidence of the occurrence of at least one new crystal stacking sequence. Peaks (1) and (2) can unambiguously be ascribed to the growth of stacking faults (SFs) (ABCB...), i.e. crystal twins along the [111] direction of the bulk Ir. In contrast, the origin of peaks (3) remains unclear.

Figure 1(b) shows a projection along the [00L] direction of a scan along the [H 0 3-H] CTR, after the 70 ML Ir deposit. Unfortunately, due to lack of time, systematic measurement of such scans was not possible while depositing Ir. Along the [H 0 3-H] CTR, one observes SFs signal at L = 3-H = 1 and 2, and additional peaks at L = 4/3 and 7/3. The latter peaks are ascribed to regions in the SF domains that would have experienced a second twin operation along a <111> direction non perpendicular to the sample surface. These domains will be referred as twin of twins (TTs) in the following. Our previous STM measurements could not put them in evidence, seemingly because they are not sensitive to them. To the best of our knowledge, such TTs were never reported in Ir homoepitaxy, and therefore would definitely be worth further experimental investigations. Though it is already clear that the TTs are created during Ir deposition, as evidenced by their vanishing upon a subsequent annealing-sputtering-annealing cycle, a model for their formation remains to be developed.

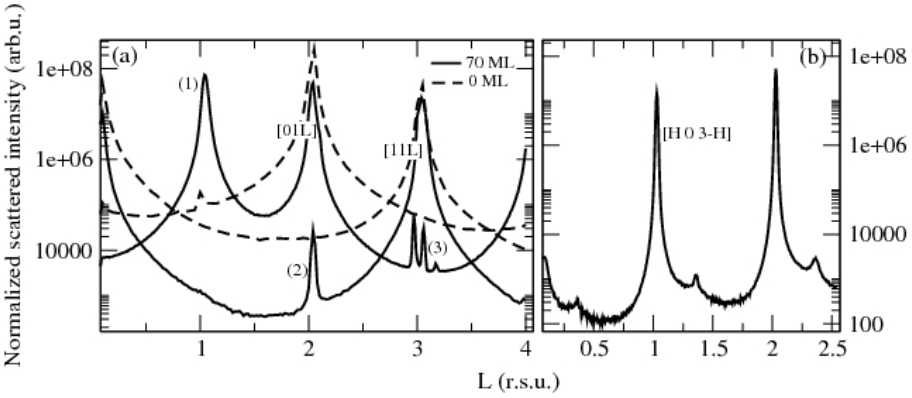

Fig. 1: *Normalized scattered intensity measured for $\alpha_i = 0.273°$, as a function of the L reciprocal space coordinate, for bulk Ir and after a 70 ML Ir deposit, (a) along [01L] and [11L] directions, and (b) along [H 0 3-H] direction.*

Between Θ = 0 ML and 70 ML, L-scans and ω-scans were systematically measured close to the (011), (012) and (1 1 1.5) points, in order to monitor: (a) the deposited Ir thickness, (b) the relative proportion of faulted and regular Ir stacking, and (c) the evolution of the sample roughness. Figure 2 summarises the corresponding results, putting in evidence the layer by layer growth up to, to some extent, 10 Ir ML [fig. 2(a)], and showing a progressive increase of the *total* SF amount in the Ir layer [fig. 2(b)]. Further analysis will require a quantitative calibration of the data shown in fig. 2(b). We plan to achieve such a calibration by refining the structure of the 0.76 ML Ir deposit, which is possible through fitting the extensive set of CTRs [2] measured for this deposit. The results will be compared to our previous STM analysis of the *surface fraction* of SFs [3].

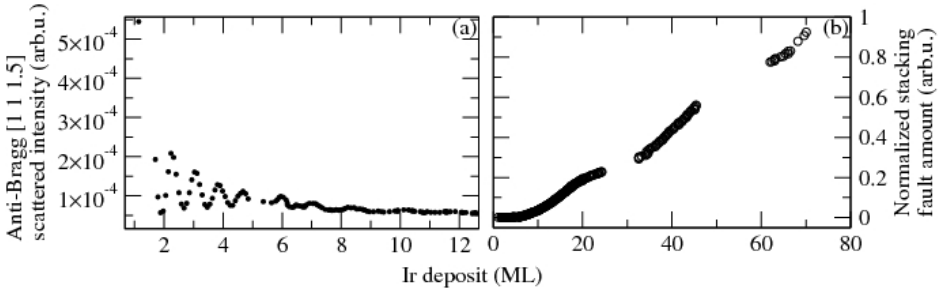

Fig. 2: *(a) Anti-Bragg scattered intensity showing layer by layer growth oscillations. (b) Qualitative evolution of the total amount of stacking faults as a function of the Ir deposit.*

Thermal stability of stacking faults and other defects

Following the Ir growth step, a single progressive set of annealing stages was performed, with the view of assessing the thermal stability of stacking faults and other kinds of defects. Accordingly, ω-scans and scans along CTRs were systematically measured in the vicinity of the corresponding reciprocal space points, at T_s = 350 K, after 20 s annealing at various temperatures ranging from 350 to 1600K. Figure 3(a) shows the area below ω-scans measured in anti-Bragg (1 1 1.5) position, revealing a strong smoothening of the surface around T_s = 900 K. This suggests a reorganization of the Ir layer formerly grown. This can also be inferred from fig. 3(b), which depicts the full width at half maximum (FWHM) of L-scans close to the (011) and (012) reciprocal space positions, i.e. quantities decreasing when the domain boundaries of SF and regular stacking, respectively, organize.

The evolution of the amount of SFs and other defects was also monitored through appropriate ω-scans. These results seem promising but yet insufficient in the view of thorough understanding and modeling of the defects stability. Indeed, the initial amount of SFs in the crystal, which depends on the Ir layer thickness, is a key-parameter which could not be tunned due to lack of time. Also, the influence of adsorbates, such as CO and O_2,over the SFs thermal stability, could not be studied due to both lack of time and non suffisiently low bas pressure.

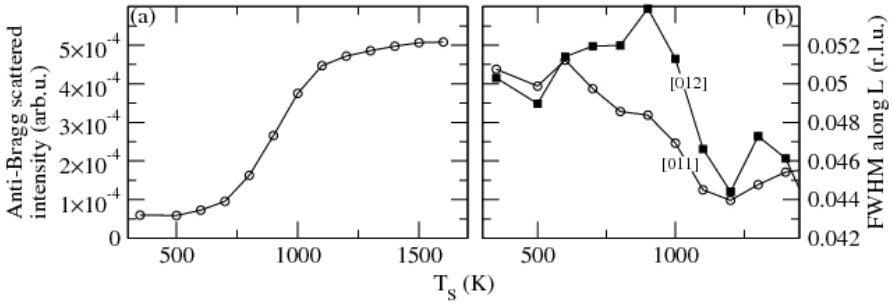

Fig. 3: *(a) Anti-Bragg scattered intensity. (b) FWHM of L-scans close to the [011] (SF) and [012] (regular stacking) reciprocal space points.*

These results are expected to be part of a publication submitted in the next months. However, it should be underlined that they would definitely deserve to be complemented, regarding the strong preparation effort (3 full weeks), the obvious evidence for the feasibility of the experiment, and the short amount of measuring time (2 days) available at the end, which was not sufficient to address all our initial objectives or to investigate the unexpected effects put in evidence during the measurements, e.g. the occurrence of TTs.

References

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