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Deliverable D2.1

Evaluation and implementation of suitable rate control

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1.- Introduction

The aim of this deliverable was the rapid identification and evaluation of a suitable technique for in-situ metallic precursor (copper, tin and zinc) deposition rate to enable independent monitoring and control. The control would be manual adjustment in the first instance.

Sputter deposition is a reliable technique for reproducibly depositing material. Using set deposition power and time parameters is results in film thicknesses that do not generally vary significantly. However, under circumstances where there are unforeseen minor problems or variations in the deposition parameters the properties of the deposited films may be significantly different from expected. In addition, the different metals within the deposited thin film precursors exhibit varying diffusion rates. This can result in individual component metals moving within the depositing/deposited film to a degree that is significantly different from the arrival rate of the materials at the depositing surface. In the case of composition sensitive kesterite absorber layers such variations may lead to significant performance variations.

A technique that enables the independent monitoring of precursor deposition rate would be a valuable tool to distinguish between the deposition parameter variations and the final thin film parameters.

Several possible techniques were available for monitoring of the deposition flux within the KESTCELLS activity at Northumbria University. These included the following:

- Mass spectrometry
- Optical spectrometry
- Quartz crystal thin film monitoring

2.- Preliminary Trials

Mass and optical spectrometry trials were completed. The geometry of the deposition chamber, the location of the mass spectrometer attachment to the chamber, the rotation of the substrate holder and the need to access the chamber by opening the top plate, made access for sampling the deposition flux more problematic than anticipated. This technique may be worth pursuing at a later stage, however, it was rejected for the purposes of the in-situ rate monitoring identified for D2.1.

Optical spectrometry techniques were installed to monitor depositing material. The initial trials investigated the ability to monitor emission and absorption spectra. Emission spectra obtained indicated a possible presence of zinc and tin, however, the measured spectra were dominated by the argon emission lines, as expected, and the low intensity of the signals indicated that this technique would require significant further optimisation before it would be possible to evaluate

it as an in-situ rate monitoring and control option. Geometrical sensitivity and coating of the detector head were identified as problems that were unlikely to be overcome within the time frame allowed for this activity (within KESTCELLS).

The use of quartz crystal monitoring (QXM) is a common approach in thermal evaporation deposition processes. The value of using this technique for sputtering is dependent on the sputtering technique used, for instance, in RF sputtering there can be increased uncertainty due to interference between the RF and the crystal oscillator frequency. Sputtering is typically carried out at lower source-substrate distances that are typical for thermal evaporation. There is a potential problem due to resputtering from the crystal when the head is in close proximity to the plasma that would reduce the reliability of deposition rate values. If the head is not carefully located they can interfere with the uniformity of deposited films or result in contamination due to sputtering of material from the housing.

Initial trials at NU focussed on positioning and calibration of the QXM heads to monitor individual sources. The heads used the existing thin film monitoring capability within the deposition system that was installed to monitor thermal evaporation. This was successful, the technique was used to monitor a tin target, the sensors were calibrated using the standard technique and use of a DekTak profilometer to measure the deposited thickness. Following initial calibration the monitor was tested (QXM thickness reading of 630 nm corresponded to a measured thickness of 660 nm). Further calibration is planned but as a rate monitor, a 5 % difference in thickness was considered acceptable for implementation trials.

3.- Evaluation and Implementation

Three monitor heads were installed and positioned in different locations with respect to the three precursor sources. The black round headed lines in figure 1, represent the locations of the sensor heads that were tested.

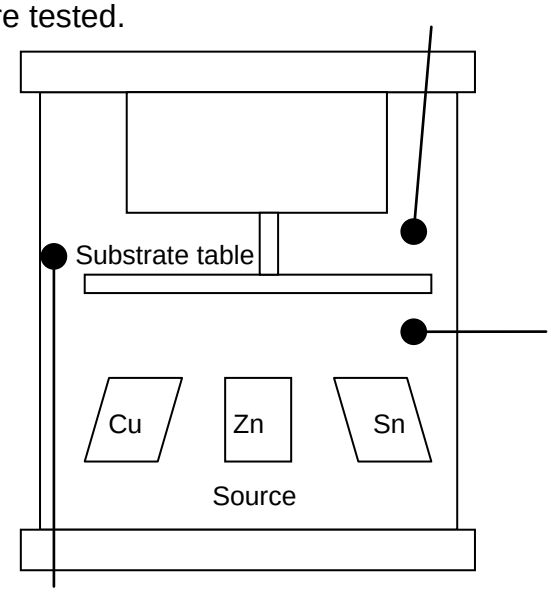

Figure 1. Schematic diagram of deposition system with location of QXM heads indicated by round headed lines.

The investigation resulted in locating all sensors above and to the side of the substrate table to maximise crystal life whilst providing reproducible measurements of the film deposition rate. Further refinement of location is anticipated as the technique is optimised.

3.2. Implementation for Multilayer deposition

The technique was successfully used to monitor the deposition rates for the three individual precursor sources. To implement the technique for monitoring during precursor deposition a series of trials was undertaken. These involved the deposition of thick stacked and the multilayer precursors. The multilayer precursor were considered to be susceptible to crosstalk between sensors due to all the sources operating at the same time.

The multilayer precursors were deposited and the sensor rate values and deposition power values recorded. Figure 2 shows the rate readings from the QXM sensors plotted on the same graph as the measured deposition power values for the duration of the thin film deposition.

The dashed lines represent the measured power values for the copper and tin targets (the software is currently not configured to monitor the power supply that feeds the zinc target). The power supplies are 1.5 kW and their ability to control is stated as 2% of full scale. In figure 2 this can be seen as the step changes in the control as this resolution is approached. The deposition rates vary from each other due to location. The trials identified the need for the recalibration of the thickness and rate when the multilayer deposition is implemented. However, the ability to monitor the rate is demonstrated. This is clearly seen when considering the signal for the tin source. There is gradual decrease in deposition rate as the deposition progresses and the generator output also shows a drift within the specifications of the generator over the same period.

It may also be noted that there is a ripple in the signals for the rate values. This is due to the location of the sensors and the decision to leave one of the four substrate positions in the substrate table, unoccupied during the deposition, to provide a closer approximation to the rate than an individual substrate is exposed to, and b) to determine if the sensors would pick up the variation due to this unoccupied space. This will provide an understanding needed for further refinement of the technique.

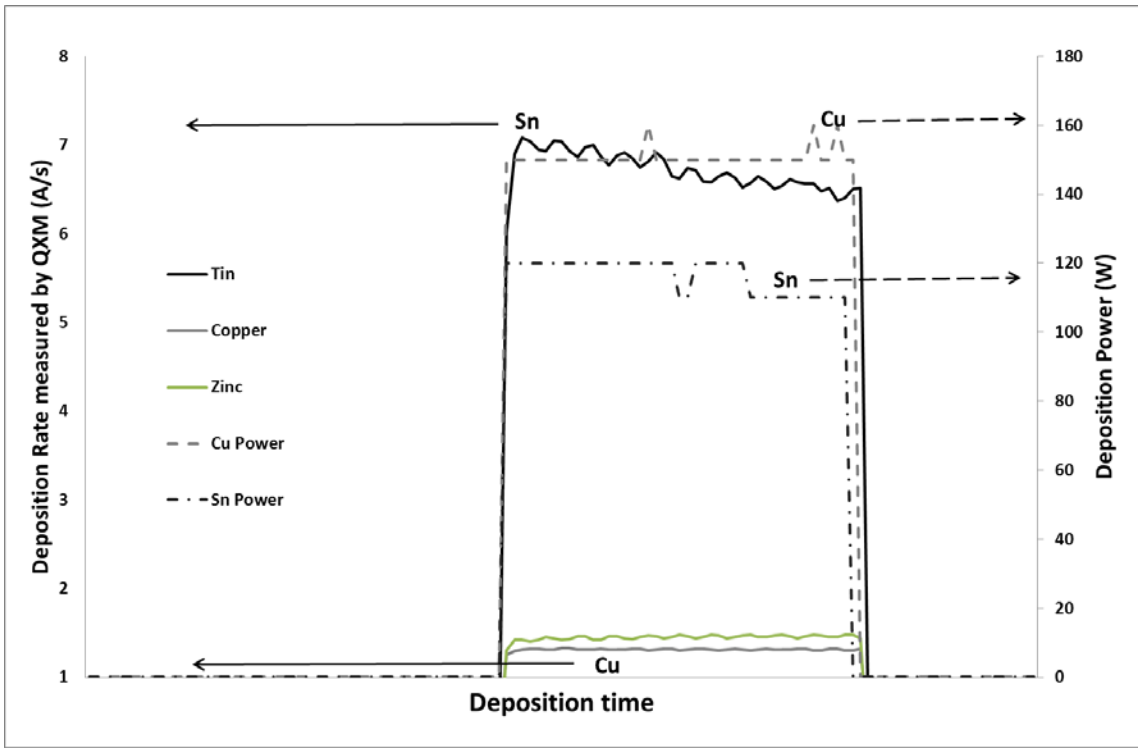

Figure 2: Plot demonstrating the correlation between the deposition rate as measured using the Quartz Crystal Monitor and the Deposition power applied to the Copper and Tin sources (the current deposition software does not record the zinc deposition power).

It is noted that although the power applied to the zinc target was not monitored the results from the copper and tin rate and applied power measurements together with the constant rate for the zinc monitored by the QXM for that source, provide confidence in this remaining constant throughout the deposition. There are some slight timing discrepancies between the two temporal measurements (of rate and power) that are attributed to the different software used for each being not completely synchronised. This is not considered a problem for the intended use at NU. Further multilayer samples were produced with deliberate variations in power level and these were clearly identified by the rate monitor for the particular source. This demonstrates that it is able to be used to manually control the rate of deposition.

The technique has been implemented and is currently in an optimisation phase that will involve further evaluation and investigation of the sensitivity of the deposited and converted layers to rate variations measured using the in-situ technique. The deposited multilayer precursors are currently being analysed for composition and uniformity with depth.

4 Conclusions

Three techniques for the in-situ deposition rate techniques were evaluated. Of these the most effective for the purpose of NU activity within Workpackage 2, was identified as the use of quartz crystal monitors. This technology is commonly used for thermal evaporation and is an established and reliable technology for measuring film thicknesses and deposition rates in-situ. The technique has been implemented at NU for monitoring and manual control of precursor deposition rate and will continue to be refined during the KESTCELLS project.