Evap-4 Technical Note

Deposition Rates and Flux Currents for Nickel, Tin, Chromium, and Titanium Using Mini E-beam Evaporator Evap-4

This technical note presents the results of deposition rate and flux current measurements for various metals deposited using the High Capacity Four Pocket Mini E-Beam Evaporator (Evap-4). The metals tested—Nickel, Tin, Chromium, and Titanium, each exhibit unique properties that influence their deposition behavior. Nickel (Ni) has a high melting point of 1455°C and a relatively low vapor pressure, requiring temperatures well above its melting point to achieve evaporation due to the significant energy needed to overcome its strong atomic bonds. Tin (Sn), on the other hand, has a relatively low melting point of 232°C; however, its low vapor pressure necessitates much higher temperatures, around 1000°C, to generate sufficient vapor pressure for evaporation. Chromium (Cr) has a high melting point of 1907°C but generates significant vapor pressure at temperatures below its melting point, enabling substantial deposition rates near 1300°C, where it sublimes. Titanium (Ti) has a high melting point of 1668°C, making it highly stable at elevated temperatures.

The data provided in this technote aims to enhance understanding of the deposition process for these metals using the Evap-4.

Experimental Conditions

The experimental setup for metal deposition experiments were conducted using the Evap-4, mounted on a 30deg angle port on a HV Box chamber Figure 1. Deposition rates were measured using an Inficon Cool Drawer Quartz Crystal Microbalance, (QCM) mounted on a linear shift mechanism, with source-to-QCM distances or throw distance of 100 mm and 200 mm. For all trials, the chamber base pressure was consistently maintained below 5×10^{-7} mbar to ensure stable deposition conditions. The same configuration was used for all metals, ensuring consistency in the deposition process.

Figure 1 Evap-4 mounted inside the box chamber with pocket 3 powered up.

1. For **Nickel (Ni)**, A 1000 mm³ tungsten crucible was loaded with approximately ¼ of Ni wire and fitted with a PbN liner. The crucible was placed in pocket 1 of the Evap-4 and the deposition power was limited to 125W.

2. For **Tin (Sn)**, A 1000 mm³ tantalum (Ta) crucible was approximately ¼ filled with Sn wire. The crucible was placed in pocket 2 of the Evap-4 and the deposition power was limited to 150W.

3. For **Chromium (Cr)**, A 1000 mm³ tungsten crucible was approximately ¼ filled with Cr pieces. The crucible was placed in pocket 3 of the Evap-4 and the deposition power was limited to 150W.

4. For **Titanium (Ti)**, A 1000 mm³ tungsten crucible was approximately ¼ filled with titanium slug. The crucible was placed in pocket 4 of the Evap-4 and the deposition power was limited to 160W.

Figure 2 Zoom-in of Flux Plates in a Mini E-beam Evaporator System

In all cases, the deposition rate was measured with the same QCM setup, ensuring consistency across the deposition experiments for each material. A QCM monitors the change in oscillation frequency of a

gold coated crystal as material lands on the crystal. The material properties of the evaporant are then used to convert this change in frequency into a deposited weight and thickness. For co-evaporation the QCM can only give an estimate of the measured thickness based on the average density and Z factor of the two materials. Also, QCM crystals operate over a finite frequency range and will fail when heavily coated. If this happens during a deposition run the flux current can provide a backup measure of the deposition rate. Flux plates are positioned above each pocket and measure the small ion current (few A) associated with the evaporation from that pocket. The ion flux is proportional to the deposition rate, so gives an independent measure of the deposition rate from an individual pocket, even under co-evaporation conditions. Through the dedicated Evap-4 software package the user can choose flux control to enable independent rate control for up to 4 different materials. In these experiments the EVAP-4 power supply was set to power control mode and the ion flux was monitored and later plotted against deposition rate.

Results

The results of metal deposition experiments using the Evap-4 showed distinct behaviors for each material, including deposition rates, ion flux stability, and issues related to coating and shorting.These behaviours are dependent on material properties, source-to-QCM distances, and power settings.

1. For **Nickel (Ni),** the deposition rate and flux current for Nickel are plotted in Figure 3 for a source-to-QCM distance of 100 mm, with power settings ranging from 100 W to 125 W. At 125 W, the deposition rate was measured to be low, at 0.1 Å/s, and the flux current was also modest at 12.5 nA. Due to the low deposition rate and flux, the data exhibited significant noise. However, the correlation between flux and deposition rate remained consistent, and the flux current was stable throughout this At 125 W, the PbN liner in the crucible was displaced, as shown in Figure 4, which resulted in the premature termination of the measurements. This problem can be avoided in future by reducing the power ramp on the pocket to and by checking the tolerance on the PBN liner and crucible, to allow space for thermal expansion.

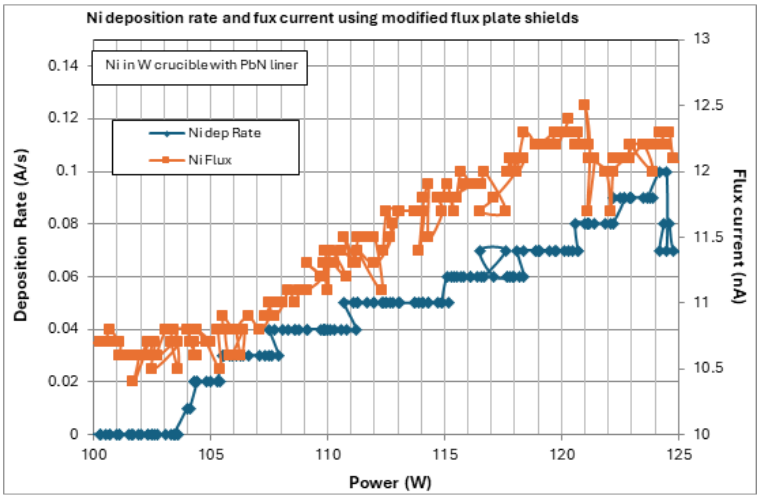

Figure 3 Deposition rate and Flux current for Ni in W crucible with PbN liner for a working distance of 100mm

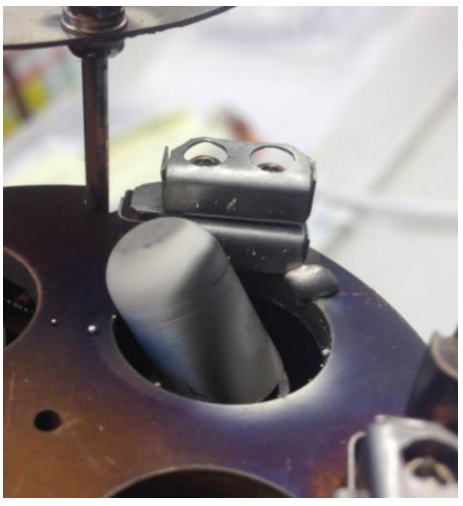

Figure 4 PbN liner popped out of W crucible at 125W

2. For **Tin (Sn),** the deposition rates are shown in Figure 5, with measurements taken at both 100 mm and 200 mm source-to-QCM distances. At 150 W and a 100 mm source-to-QCM

distance, the deposition rate was relatively high at 8.6 Å/s. For the 200 mm distance, the deposition rate dropped to 2.3 Å/s, which follows the expected $1/r^2$ dependence on throw distance. The flux current for the 100 mm distance remained stable up to 130 W, but deviated from steady correlation with the rate beyond this point. Upon inspection, it was observed that the flux plate had shorted to the lid due to a thick layer of deposited. To avoid this issue in future the use of a PBN liner to reduce material creep and regular checks that there are no flakes on the flux plates are recommended.

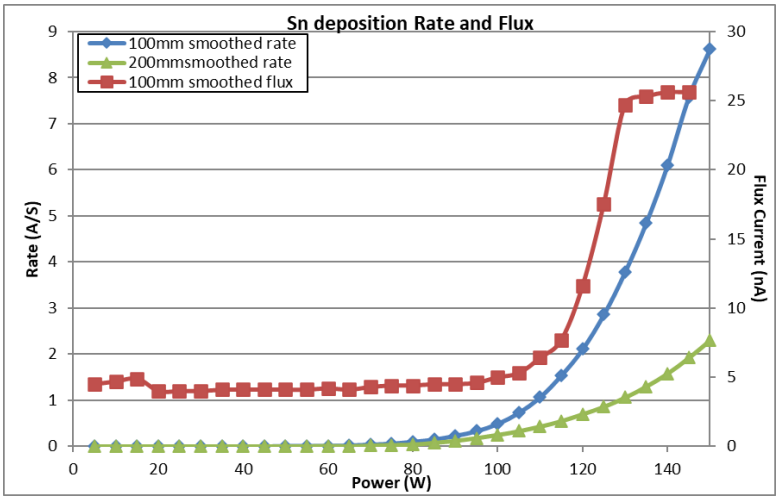

Figure 5 Deposition rate and flux current for Sn in a Ta crucible

3. For **Chromium (Cr),** the deposition rates and flux currents for Chromium are plotted in Figure 6 for both 100 mm and 200 mm source-to-QCM distances. At 150 W, a moderate deposition rate of 1.7 Å/s was recorded at 100 mm. For the 200 mm distance, the deposition rate dropped to 0.46 Å/s, in accordance with the $1/r^2$ relationship. The measured flux current was relatively low, at 33 nA at 150 W, and was independent of the source-to-QCM distance. The flux current remained stable throughout the 1 hr 20 min measurement period, indicating a consistent rate of material deposition. Figure 6 illustrates that the ion flux is independent of working distance, as expected. This demonstrates a repeatable measurement but is also a reminder that a tooling factor must be used when calibrating the actual deposited thickness for different working distances. some instability in the flux current was observed towards the end of the measurement cycle when the power was ramped down to 0 W. The flux current returned to its original curve once the power was reduced to low levels, indicating that the issue was non-permanent and likely caused by thermal effects.

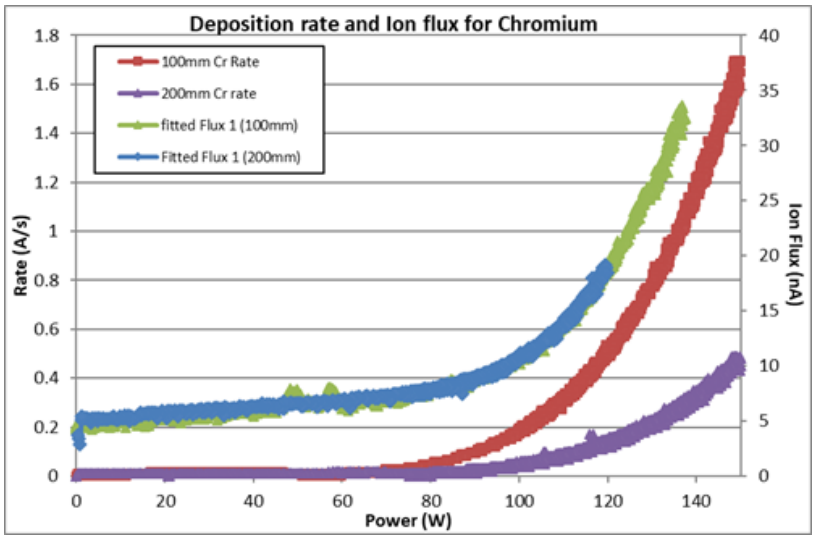

Figure 6 Deposition rate and Flux current for Cr in a W crucible

Figure 7 Cr in W crucible after deposition

4. For **Titanium (Ti)**, the deposition rate measured at a 100 mm source-to-QCM distance, and is shown in Figure 8. The deposition rate was very low, at 0.05 Å/s for 160 W. Despite the low deposition rate, the flux remained stable over the measurement period at 100 mm. Due to the very low rate, measurements were not taken at the 200 mm source-to-QCM distance. Upon removal of the Evap-4 from the vacuum system, inspection of the Ti crucible revealed that the Ti slug had only melted at the point of contact between the crucible and the slug. Higher deposition rates are expected for evaporation from a rod and therefore recommended for titanium.

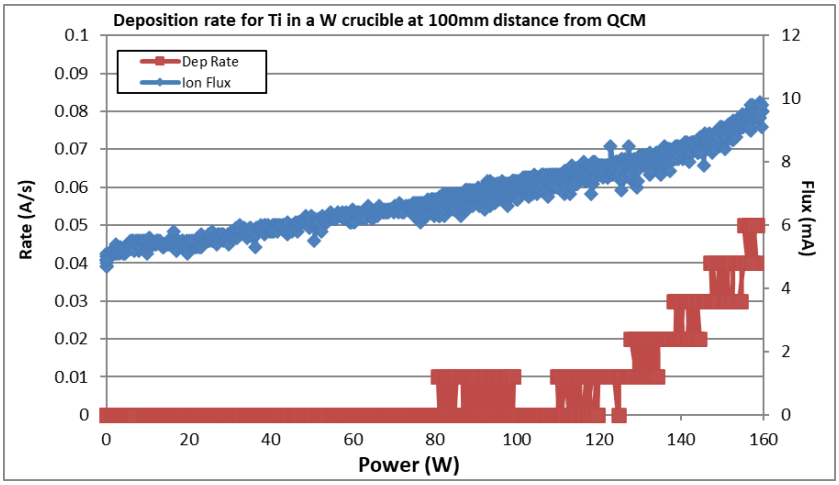

Figure 8 Deposition rate and Flux current for Ti in a W crucible

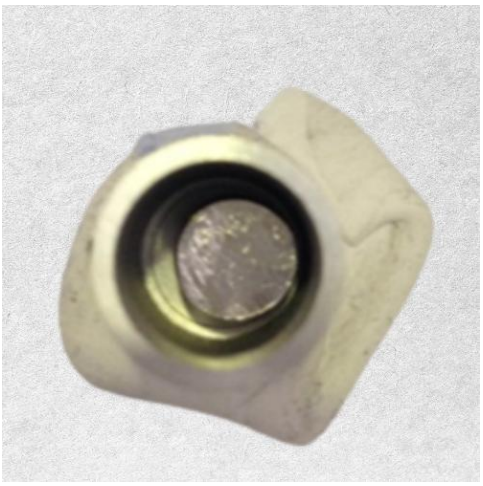

Figure 9 Ti in W crucible after deposition at 160W

Conclusion

The deposition experiments for Nickel, Tin, Chromium, and Titanium using the Evap-4 demonstrated controlled evaporation and stable flux currents with each material exhibiting distinct and favorable characteristics under controlled deposition conditions.

1. The **Nickel (Ni)** deposition process demonstrated stable flux currents and good correlation with deposition rates. Although some challenges with thermal expansion mismatch were observed with the tungsten crucible and PbN liner. An alternative approach is evaporation from Ni rods with longer-style filaments , offering a promising approach for applications requiring thin Ni films in the 5-10 nm range.
2. The **Tin (Sn)** deposition experiment showed a high deposition rate and stable flux current up to a certain point, with valuable insights gained on the effects of crucible liners and flux plate shields. The use of a PBN crucible liner is a viable solution for reducing shorting issues, providing a promising path forward for Sn deposition in high-rate processes.
3. The deposition of **Chromium (Cr)** exhibited stable flux current and consistent deposition rates, confirming the effectiveness of the current experimental setup at 150 W. The flux current measurement showed stable and repeatable results for different working distances.
4. The **Titanium (Ti)** deposition process showed stable performance with low deposition rates, confirming the suitability of the current experimental configuration for Ti deposition. The Ti rods is expected to increase deposition rates, further improving efficiency.