Week 2 Report

The Effect of Dipping Duration on Perovskite Formation:

PbI_2 was coated onto cleaned glass in air (no glove box), then dipping times in CH_3NH_3I 2-Propanol solution was varied according to the following table:

Sample Number	Temp of Glass (˚C)	Dipping Duration (Sec)
F07	Room ~22	10
F08	Room ~22	20
F09	Room ~22	30
F10	65	10
F11	65	20
F12	65	30

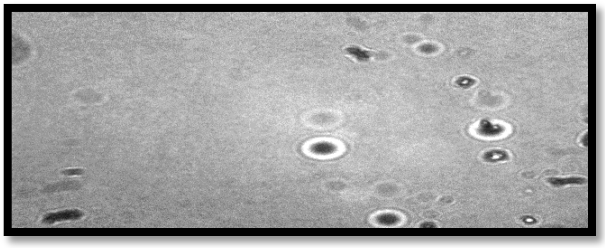
Glass temperature prior to dipping was also varied.

Observations:

F10-F12 were more opaque than F07-F09, in general. Longer dipping times led to darker sample, more perovskite. But this may be due to the thick PbI_2 layer.

The samples were studied under a microscope and revealed the following:

F07: (10 Sec)

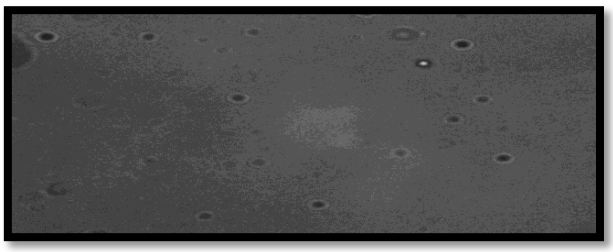


- Good, even coat
- Many black spots
- Red

F08: (20 Sec)

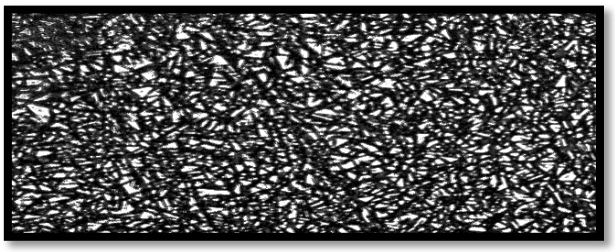
- Black spots
- Signs of cracking
- Very red

F09: (30 Sec)



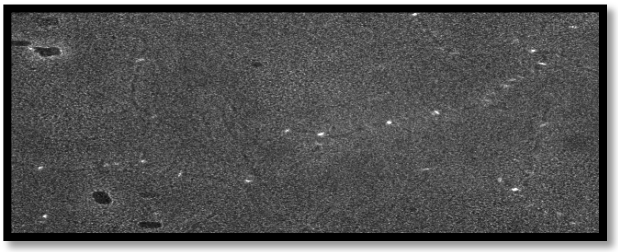
- Black spots
- No crack (maybe I was unable to see them)
- Very red

F10: (10 Sec)



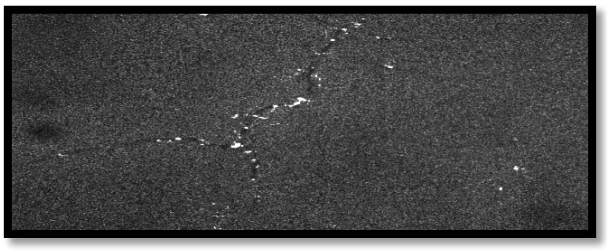
- ½ Pervoskite to hole ratio
- White (No red)

F11: (20 Sec)



- Few black spots
- Cracks

F12: (30 Sec)



- Few black spots
- Few holes
- Lots of cracks

Conclusion:

Longer dipping times yields darker samples, meaning higher perovskite density. The probability of cracks increases with time, and is proportional to the glass' temperature prior to dipping.

For the future we will stick with room temperature glass prior to dipping.

Air Drying After 2-Propanol Rinse in Glove Box:

Dipping was attempted with the same methods previously used, but in a glove box. Unlike in air, letting the sample dry in the glove box did not result in any pervovskite dissolving. The use of an air jet is no longer needed when dipping is done in a humidity free environment.

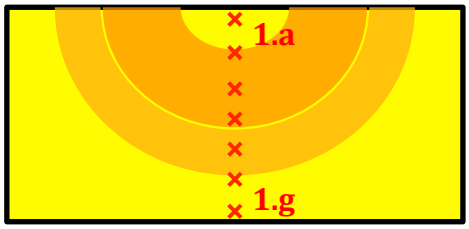
O_2 vs. O_3 Priming, & Heated vs. Unheated Glass Prior to Spin Coating:

Now we take a closer look at spin coating and its preceding steps to arrive at a thinner and more uniform initial coat of PbI_2. The sample were treated according to the following table:

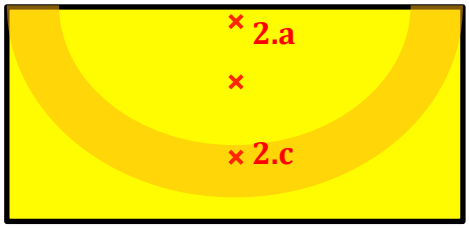
Sample	Primer	Glass Temp. (˚C)
F13	O_2	70
F14	O_2	70
F15	O_2	70
F16	O_2	Room ~20
F17	O_2	Room ~20
F18	O_2	Room ~20
F19	O_3	70
F20	O_3	70
F21	O_3	70
F22	O_3	Room ~20
F23	O_3	Room ~20
F24	O_3	Room ~20

Observation 1 (Heated vs. Unheated Glass):

Heated samples exhibited a multi color ring pattern, while unheated sample were uniformly colored. F19 & F23 were compared using a SEM, by taking pictures across the 'rings.' The following figures show where these pictures were taken:



F19: Images were taken starting from point 1.a, with 2mm spacing, and ending at point 1.g.



F23: Images were taken starting from point 2.a, with 5mm spacing, and ending at point 2.c.

The images revealed that the surface becomes more jagged as we move away from the center. Point 1.a is more even than 1.g, but exhibits more holes (Fig 1.a,f). Points 2.a, 2.b, 2.c were similar to each other, more jagged than 1.a, but more even than 1.g (Fig 2.a,c).

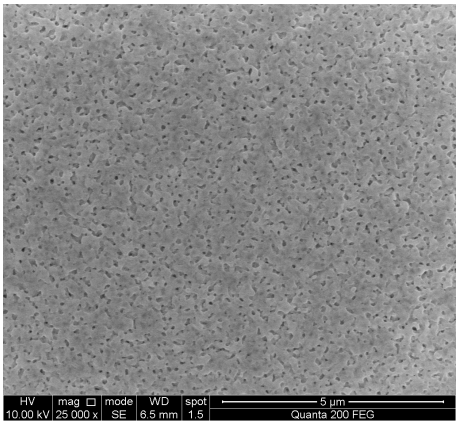


Fig. 1.a

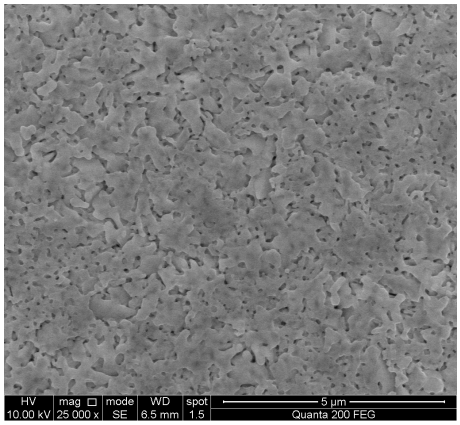


Fig. 1.f

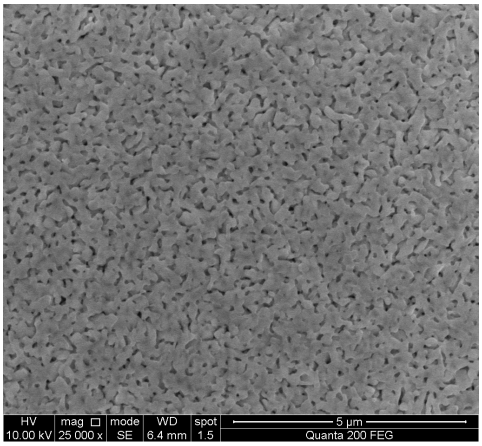


Fig. 2.a

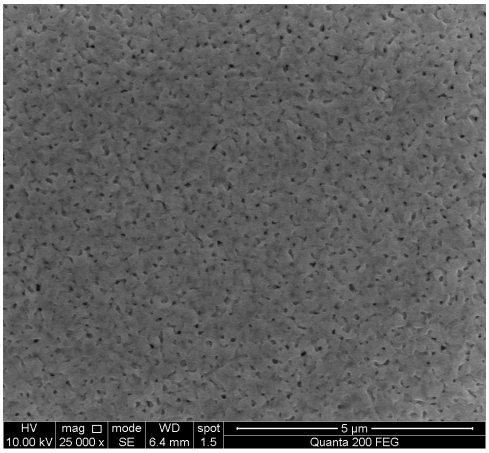


Fig. 2.c

Conclusion 1:

It seems that the best results are attained at the center of the heated glass, but become much worse than unheated glass at the edges. We must either use unheated glass or find a variation to the procedure remedies the jagged edges in heated glass. Makhsud proposed applying DMF to the heated glass prior to adding the PbI_2 solution during spin coating. Trying this resulted in no apparent rings, but no SEM images were taken yet. This variation to the procedure will be studied further.

Observation 2 (CH_3NH_3I Layer After Rinsing in 2-Propanol):

The rest of the samples aside from F19 & F23 were dipped in CH_3NH_3I 2-Propanol solution. While drying a white layer seemed to appear on the last surfaces to dry. It is speculated that this layer is CH_3NH_3I. This is troublesome since it will act as an insulator and dramatically decrease the efficiency of the solar cell.

Conclusion 2:

A method to remove access CH_3NH_3I that appears after dipping must be found. This will be studied further, and several methods such as double dipping, decreased solution concentration, and others will be tested.

Observation 3 (O_2 vs. O_3 as a Primer):

Room temperature glass prior to spin coating was revealed to be more prone to micro-cracks than heated glass, when studied under a microscope. O_3 treated glass also seemed to be more prone to micro-cracks than O_3 glass (Fig.3.a,b).

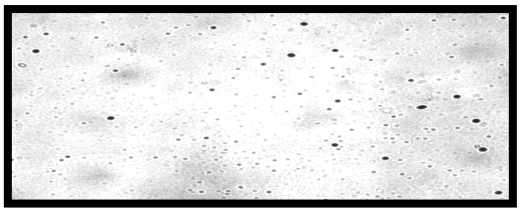


Fig. 3.a Sample F16

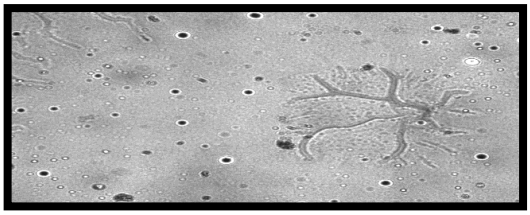


Fig. 3.a Sample F24

Conclusion 3:

O_2 should be used, and glass should be heated prior to spin coating to avoid cracks.

Next Week:

• Longer dipping times and variations in solution temperature will be more carefully tested with properly spin coated samples (spin coat in glove box).

• Applying DMF prior to the PbI_2 solution during spin coating, on heated glass, will be tested further.

• A method must be formulated and tested to remove the insulating CH_3NH_3I layer that results after dipping.