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E-Beam Deposition of ITO at 250ºC and HCl Wet Etch

Objective: A customer wants us to deposit ITO film at a higher temperature with an E-beam evaporator and a sheet resistance of less than 100 Ω/square at the film thickness of 100 nm.

Sample: a double-side-polished 2" sapphire wafer as well as a small one-side-polished Si piece.

Experimental:

1)Sample Clean: a) sapphire wafer: acetone (2')+methanol (1') in ultrasonic bath, DI rinse and N_2 blow-dry; b) Si piece: acetone (2')+methanol (1') in ultrasonic bath, DI rinse, dipping in BHF for 1 minute, DI rinse again and N_2 blow dry.

2) ITO E-beam Evaporation:
a) Loading both sapphire wafer and Si piece onto the sample holder (the upper one) of E-beam#2;
b) Pumping down the chamber and setting the heater to 580ºC (the actual stage temperature is 250ºC);
c) Setting the nominal ITO thickness to 150 nm and the tooling factor to 69.9;
d) Waiting for the chamber vacuum reach to 1.94e-6 Torr before turning on the high voltage and filament current, then, setting the beam sweepings within the ITO material crucible along both longitudinal and lateral directions and raising the longitudinal and lateral sweep frequency to 46 and 27, individually;
e) Flowing 30sccm O_2 into the camber (must have both the voltage and current turning on; otherwise, they won't be on) (the chamber pressure is 3.51e-4 Torr);
f) After 5 minutes, opening the shutter and turning the current up so that the rate reaching to ~0.5 Å/s.
g) After reaching to the target thickness, turning the voltage and current off, turning the heater off (0ºC);
h) Waiting for 20 minutes before turning the O_2 flowing off, then, venting the chamber, getting the samples (be very careful: the sample stage is very hot!).

3) Measurements of the Film Properties:
a) Ellipsometer Measurement on the Si piece: using the Cauchy model with a native oxide layer (10 Å) between the film and substrate to fit the curves: d=1660 Å and n=1.970 at the wavelength of 632.8 nm (MSE=50.8);
b) 4-Probe Resistance Measurement on the 2" Sapphire Wafer (16 points) the average sheet resistance is 27.0543 Ω/sq.

4) Lithography to Pattern the Sapphire Wafer:
a) Gasonic oxygen treatment (Program#3) at 250^{o}C for 3 minutes;
b) Spin-on HMDS at 3 krpm for 30 s (waiting for 30 s before spinning; using the spinner close to Microscoper#3 and one of the 2" wafer chucks);
c) Baking at 105^{o}C for 90 s and let it cool down for 30 s;
d) Spin-on AZ 4110 resist at 4 krpm for 30 s;
e) Baking the resist at 95^{o}C for 60 s;
f) Exposing the resist with MJB-3 aligner for 12 s (making sure there is no filter in the UV path!)
g) Developing the pattern in AZ400:DI (1:4) for 60 s;
h) O_2 plasma descum: 300mT/100W for 60 s;
i) Baking the pattern at 123^{o}C for 3 minutes to harden the resist.

5) Wet-etch ITO Film using HCl:
a) Making the solution: HCl:DI (1:2.5/60ml:150 ml), putting onto 70^{o}C hot plate with a glass dish covering the top, letting it heat up for 30 minutes (the actual solution temperature: 52^{o}C);
b) Dipping the sample (ITO#3: a quarter 2" sample) into the HCl solution for 35 minutes (See Figure 1 below; the actual solution temperature is ~56^{o}C);
c) Measuring the etch depth using Dektak: the resist thickness before etch is ~ 1.066 μm, after the etching, the total thickness is ~ 1.202 μm.
d) Removal of PR: 1. in 1165 striper for 1hr50min in 80^{o}C hot-water bath; 2. in a fresh 1165 striper for 10 min in the same bath; 3. acetone (2')+methanol (1') in ultrasonic bath, DI rinse and N_2 blow dry; 4. O_2 descum: 300mT/100W for 2 minutes.
e) Dektak: 1650.9 Å (see pattern in Figure 2 below). **The etch rate is ~ 47 Å/min**).
f) The cross section of the ITO film is showing in Figure 3 below.

6) Wet-etch ITO Film using ITO Etchant TE-100:
a) Pouring TE-100 into a glass cup, putting onto 70^{o}C hot plate with a glass dish covering the top, letting it heat up for 30 minutes (the actual solution temperature: 45^{o}C without the cover);
b) Dipping the sample (ITO#2B: a small sample piece) into the solution for 11 minutes (1+10 minutes; the actual solution temperature is ~45^{o}C);
c) Measuring the etch depth using Dektak: the resist thickness before etch is ~ 1.066 μm, after the etchings, the total thickness is ~ 1.102 μm (**The etch rate is ~ 33 Å/min**).

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Figure 1 Microscopic pictures show the pattern, with the PR etch mask still on the top and a clear undercut along the resist edge, after 35-minute HCl-solution etch.

Figure 2 Microscopic pictures show the pattern, with the PR etch mask being removed, after 35-minute HCl-solution etch.

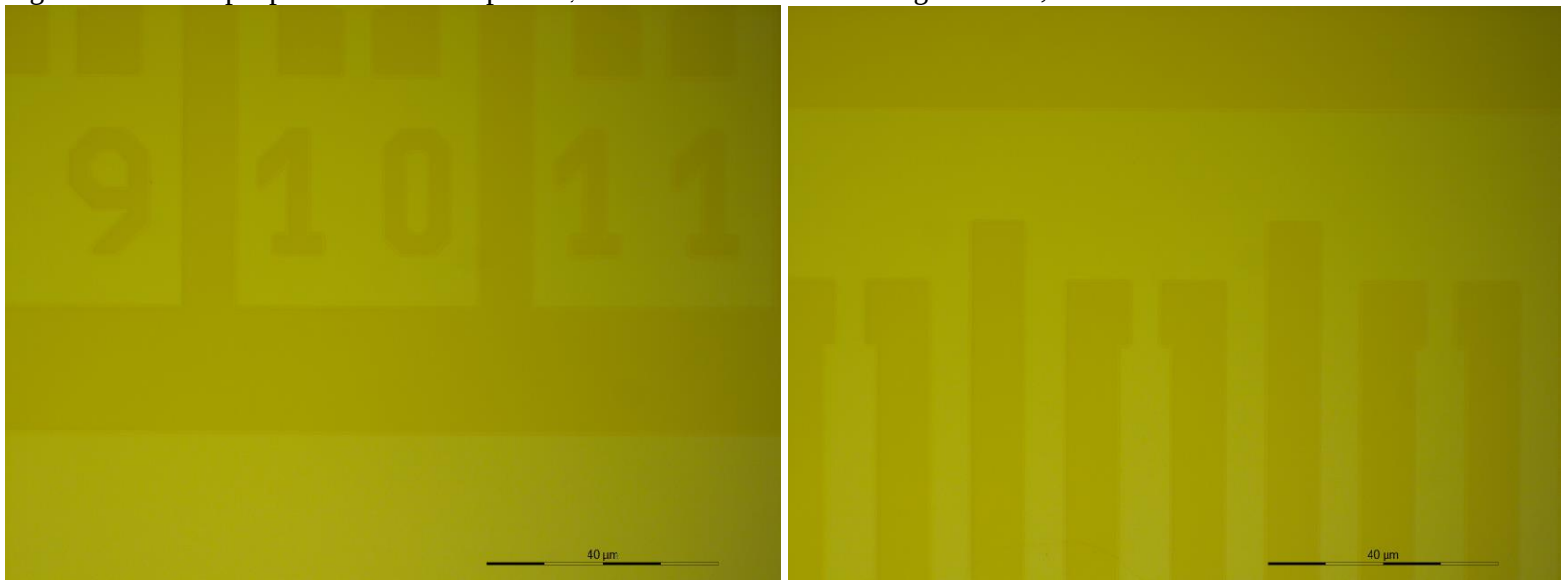

Figure 3 SEM pictures show the cross section of the ITO film.