Short term mobility report

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During my stay at the University of South Florida (USF) Prof. Saddow's group had to make several 3C-SiC/Si growth experiments for two ongoing projects:

1) Growth 3C-SiC films at least 25 μm thick on Si substrate for biological applications.
2) Growth of 3C-SiC to fulfil a contract for a well known semiconductor manufacturer.

I worked with Christopher Locke in the SiC chemical vapour deposition (CVD) lab, performing 3C-SiC epitaxial growths with him while learning about their high temperature deposition process in a hot wall reactor. One of the goals of my visit was to be able, with the aid of Prof. Saddow and Mr. Locke, to learn and transfer the hot wall design from USF to IMEM, to allow us to perform growth at higher temperatures to deposit monocrystalline 3C-SiC and process SiC bulk (6H and 4H polytypes) in order to obtain graphene.
Prof. Saddow has also interest in growing nanowires (NWs) for biomedical applications. In IMEM we have experience in the growth of SiC nanowires and the second goal of this visit was to discuss with the group how to grow NWs in USF and to perform some preliminary experiments.

The main part of the stay has been spent in the lab with Chris Locke. The process of 3C-SiC growth in USF has been discussed in details and several 3C-SiC/Si films were grown, in order to fully optimize the process to grow the thick SiC needed for the biological applications.
More than 20 test runs were performed on the reactor named MF2, aimed mainly at the minimization of interfacial voids at the SiC/Si interface and at the improvement of the surface morphology, with minimization of Si precipitates in the gas phase and the adjustment of the Si/C ratio.
Their reactor is home made, with a hot wall design: a pyrolytic graphite susceptor is placed inside graphite foam to insulate it from the outer quartz liner (Fig. 1)

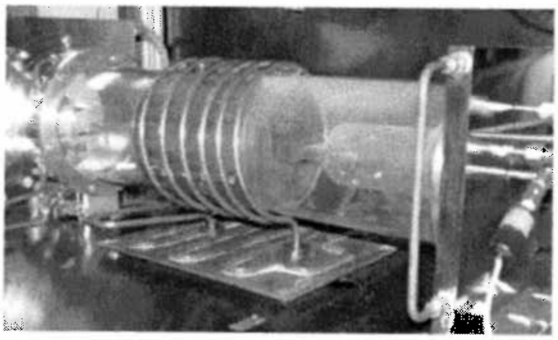

Fig. 1: MF2 hot wall reactor in USF photographed from the inlet (upstream) side.

Temperatures up to 2000 °C can be reached with this design and long deposition experiments (up to 4 hours for 3C-SiC) can be performed. In comparison, the reactor in IMEM can reach only 1400 °C but, due to the high thermal transfer to the quartz walls and the surroundings, these conditions can

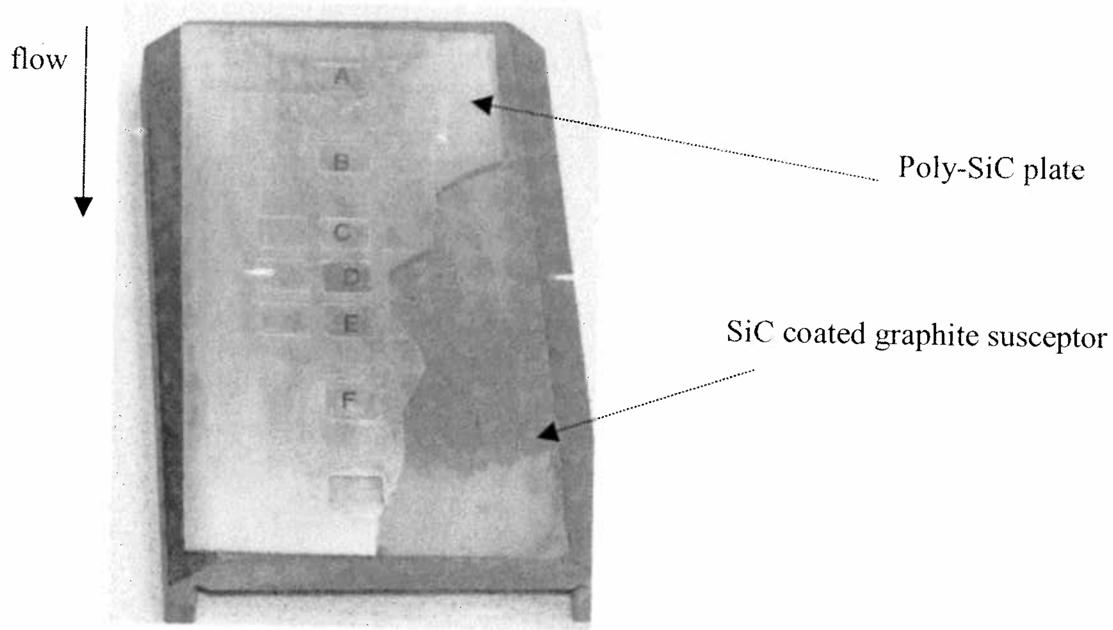

Fig. 3: Broken (but still used) poly-SiC plate used for the test runs showing sample position in the reactor. Machined recesses ensure sample position is fixed during processing.

I assisted Mr. Locke in each step of the deposition process, from the substrate cleaning to the final film inspection. We discussed the different approaches to crystal growth in Parma and USF, analyzing the capabilities and the design of our reactors and our procedures.

As an example, a typical test run procedure is reported hereafter:

Several Si samples (1x1 cm^2) are HF cleaned from the oxide and positioned on the SiC plate, then placed inside the reactor.
Reactor is purged at low P (<100 Torr) for at least 10 min in 10 slm Ar flow. This ensures evacuation of contaminants from the laboratory environment such as O_2 and moisture.
Carrier gas is H_2 (10 – 25 slm) depending on the particular process, growth pressure is between 100 and 400 Torr.
Pure propane is injected at room temperature in the reactor chamber (18-20 sccm) , with 10 slm H_2 as carrier gas.
RF generator is turned on with a soft-start
T is slowly increased up to 1135 °C (actual substrate temperature) with about a 20 minutes ramp. Ramp time is controlled by limiting the output power of the RF generator and letting the PID handle the RF power control.
Once the setpoint is reached, the carbonization lasts for 2 minutes.
After carbonization SiH_4 (10% in H_2) is immediately injected into the reactor with propane (C:Si=1).
H_2 carrier gas is increased in steps, while maintaining the same C and Si dilution.
The second temperature ramp, up to growth temperature, is about 10 minutes long.
When T=1380°C (actual substrate temperature), pressure is reduced to 100 Torr and the 15 minutes growth test starts.
Cooling down is in Ar flow and the samples are unloaded when the temperature is below 600 °C.

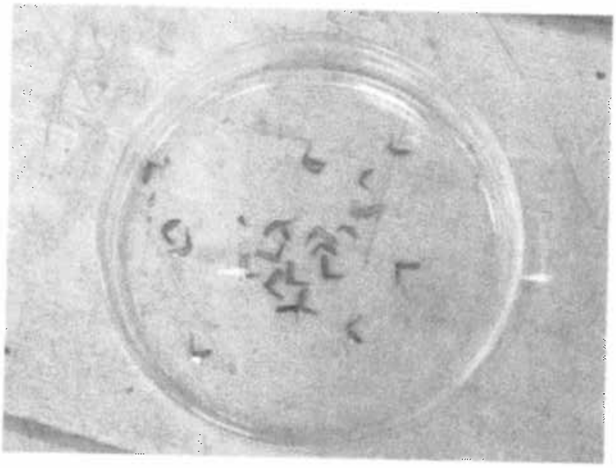

Fig. 5: Si micro-machined needles for in-vivo tests. Similar SiC needles will be realized from the film grown during my stay.

We also discussed the possibility to grow SiC/SOI or polycrystalline Si, in the framework of a possible collaboration with IMM-Bologna (CNR) and IMEM (with Dr. Antonella Poggi, whom Prof. Saddow has also known for over a decade). In order to obtain good SiC/SOI deposition, temperature is most important parameter, because SiO_2 layer must not be damaged by the thermal process. 1150°C should be optimal. Suggestion is to:

1) heat substrates at different temperatures
2) check substrate state with SEM
3) perform standard growth at the best temperature
4) adapt C/Si to minimize surface precipitates and to obtain good surface

Aim of SiC/SOI or poly-Si is to obtain MEMS microstructures for micro sensors. Some depositions on substrates provided by IMM-Bologna were planned but due to the tight schedule it was not possible to fulfil. We will perform these growth runs in Parma and ask for feedback to Chris Locke and Prof. Saddow in USF.

During the test runs a cleaning of the CVD reactor and a silicon melt test became necessary.
Quartzware was cleaned with isopropanol and then dipped in NH_4F_2 (weak solution) overnight. SiC-coated high density pyrolitic graphite susceptor is cleaned in a methanol and ultrasonic bath for about 10-15 minutes.
No thermal annealing is done after reactor reassembling. The Si melt test was performed immediately after reactor reassembling to recalibrate the pyrometer.
In order to perform the Si melt test another SiC plate was used, similar to the one shown in Fig. 3, with 4H-SiC substrates with a Si Si die placed on it.
Si melt test is performed with the same H_2 flow and at the same pressure of the growths, to ensure the same flow dynamical boundary conditions. The Si melt test is the most important tuning of the reactor, since it gives a way to calibrate the temperature reading from the graphite (from a pyrometer) with the Si melt temperature, which is 1410°C. It is performed every two-three months and after each reactor cleaning, upgrading or change in the hardware.
After the Si melt test it was apparent that the thermal profile of the growth chamber was changed, and the pyrometer setpoint have been subsequently adapted for the next runs.

During my stay I presented a seminar to Prof Saddow's students about NWs.
We had discussions about how to growth NWs at USF. The first idea was to use Au-Pd as the catalyst, because it was readily available. Instead of Au-Pd, at IMEM we use Ni as a catalyst so the

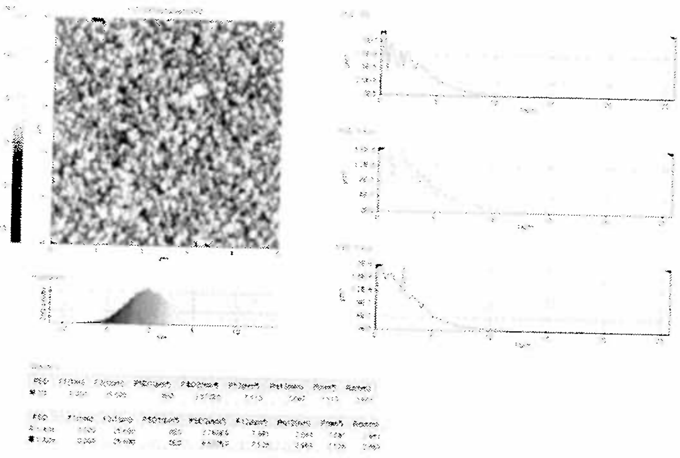

Fig. 6: AFM measurements on 3C-SiC on Si epitaxial layer obtained in USF on the samples grown in Parma during the visit of Prof. Saddow. Data provided by A. Oliveros, USF SiC group.

Conclusions

During the visit at USF the CVD reactor for 3C-SiC deposition was analyzed and several processes to grow epitaxial single crystalline SiC/Si were performed. The design of the hot-wall reactor at USF was examined and the details about the upgrade from the old cold-wall reactor to the 2'' hot-wall reactor and, finally, to the new MF2 4'' hot-wall reactor were discussed. This knowledge will be useful for future improvements of the process and on the hardware of the IMEM reactor.
The experience about NW growth in IMEM was shared with the SiC-group in USF. Even if it was not possible to perform any NW tests during my visit, we agreed to continue the collaboration and to provide feedback about the growths that will be done in USF.
Some AFM measurements on 3C-SiC previously grown in Parma during the visit of Prof. Saddow were obtained and several samples were left at USF for further MEMS processing, to study residual stress of our films. The results will be hopefully presented to a conference or to a peer-reviewed journal.

Acknowledgments

The two most important persons to acknowledge are Prof. Stephen E. Saddow and Dr. Salvatore Iannotta, who strongly supported my visit here at USF. I am extremely grateful to Steve and his wife for the great hospitality I have received. For me it was a pleasure to work in the lab and to discuss SiC growth with Christopher Locke, and to be part of his family during the weekend we spent together. I would also like to thank all the people and students who work in the SiC group, above all Alexandra for AFM measurements and Alexander for the great time spent together, but also Christopher Frewin, Justin, Joe, Jean and Shamima for the time spent together and the useful discussions we had.

Dr. Giancarlo Salviati
(proponente del programma)

Matteo Bosi
(fruitore del programma)