November 2019

Historical notes on the establishment of the EM laboratory for materials research at the Weizmann Institute of Science (WIS) (draft)

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Prior to my appointment at the Weizmann Institute, I had little experience with electron microscopy. Also, some work with scanning electron microcopy of semiconductor surfaces was done by Dr. Gary Hodes and Dr. David Cahen here in the late 1970's, based on instruments available at the Hebrew University of Jerusalem, both on the Givat Ram campus and the Faculty of Agriculture campuys across the raod from the WIS.

In the early 1980s, as a young investigator at the WIS, I was working on the modification of semiconductor surfaces and in particular CdSe, for application in photoelectrochemical solar cells. Observing that upon light-induced acid-etching the semiconductor surface becomes very opaque and highly responsive to light, I took it to a scanning electron microscopy (SEM) analysis. Since there was no EM facility for studying materials at the WIS, I made some preliminary studies on the SEM of the Faculty of Agriculture operated by Mrs. Neomy Bahat (under the supervision of Prof. Avraham Shahar z"l`). Unfortunately, this SEM did not have energy dispersive X-ray spectroscopy (EDS) detector and I was obliged to go elsewhere to perform chemical analysis of the CdSe-etched surfaces. I decided to try convincing the management of the institute to buy an SEM/EDS set-up. I talked frequently with my department head – Prof. Moshe Levy z"l and Prof. Joost Manassen z"l on this issue. But since I was a young investigator without tenure, my influence on the decision-making process was not high. After a few denials, the president of the Institute- Prof. Michael Sela graciously agreed to contribute money for this undertaking. Then one day in 1983 I was invited to the cramped (mini)office of Prof. Mordechai Avron z"l, the VP for Scientific Affairs (Rector) in the Ulman building. He granted me permission to buy an instrument and to hire a person to run this system, with the strict condition that it would be placed in the existing electron microscopy (EM) unit dedicated to research in life sciences. Subsequently, I went to the head of this unit, Dr. Victor Ben-Giat and while approaching his small office in the basement of the old Wolfson building, I saw a mouse watching me attentively- a most auspicious welcome to the EM unit, which was neighbored a mice breeding unit. At that time the EM unit possessed only a few old Philips TEM's. The most modern one there was, EM400 with scanning transmission electron microscopy (STEM) console which had been rarely used. It belonged to the Biological Services Department which was supervised by Dr. Yoram Solomon z"l who was very receptive to the idea of adding an SEM for materials research to this unit.

Victor located a small and dark room for the SEM in the EM unit. Since I was not an expert in the field, I was recommended by Lia Addadi to go to the TAMI Institute in Haifa Bay, where Dr. Eugenia Klein operated one of the first SEMs with EDS in the country and learn from her experience. Coming there, I met Eugenia who graduated from the Department of Structural Chemistry under the supervision of Prof. Mendel Cohen z"l. I was so impressed with her deep knowledge of SEM/EDS, that I told Victor that we must hire her for the job. Soon after, Eugenia and myself found ourselves examining together a few SEMs in various corners of the country. I recall a joint travel to the Degania B factory for cutting tools to study their set-up. We also visited the Jordan Valley company in Migdal Ha-Emek to see their line of SiLi drifted detectors for EDS. In the summer of 1983 I went to work in the CNRS in Meudon near Paris. Prior to going to Paris, I spent a few days with my family in Eindhoven, home of Philips, and it became apparent to me that the Philips SEM 505 (which was later upgraded to the 515 model) with EDS analyzer of another company-Tracor-Noran would be the most suitable system for us. The an EDS analyzer came with large (12 inch) red-color storage media and a computer of 32 or 64k. This SEM/EDS served us for 15 years if not longer with a few upgrades of the software and the interface electronics.

In 1991, at the initiative of the then president of the Institute- Prof. Haim Harari, the EM unit was moved to a dedicated new facility in the de-Leonesco building. Around that time, the EM unit was transferred to the administrative responsibility of the Chemical Research Support Department, previously called Chemical Services, which was a blessing for the EM unit. I recall very well the inauguration ceremony with Mrs. De-Leonesco z"l standing happily beside Prof. Haim Harari, and our delight to have been moved from the "slums" in the basement of the old Wolfson building with permanent mice visits and the odor of their excrement to a modern air-conditioned building. Sometime before (1984), I started my journey into 2D materials, after receiving a large bright crystal of WSe_2 from Prof. A. Wold z"l of Brown University, who visited here at the invitation of Prof. David Cahen. In a short time I learned how to passivate the material surface, obtaining exceedingly good photoresponse and reporting record solar-to-electrical conversion efficiencies (> 14%) for solar cells based on 2D materials. Using the Philips 505 and EDAX analyzer, I could clearly see the beautiful hexagonal etch pits on the WSe_2 surface, which emerged from screw dislocations on the semiconductor surface.

In 1989 Prof. David Cahen and myself were contacted by a newcomer from the Moscow Institute of Steel and Alloys- Dr. Lev Margulis z"l, who was an expert in TEM and electron diffraction (ED) of silicon wafers. In the first interview he told us his story. He learned basic Hebrew in a clandestine site in Moscow and consequently was a Refusnik for several years and lost his job as a researcher. He suffered what appeared to us terrible anti-Semitic discrimination and hardships, but he never gave up on his Zionistic dreams, and his perserverence eventually won out. We described to him our science where silicon had no role whatsoever. It took probably two more meetings to see that we had no common interests, and the three of us were heartbroken. On the one hand both David and I did not have any interest in silicon dislocations, but we felt that we have an expert in TEM of materials, which we cannot afford to turn down. We developed a great empathy to Lev, which paid off handsomely in the years to come. We decided to take him for a trial period of a few years. Our decision was greatly facilitated by granting him initially the Shapira Fellowship for immigrants and subsequently the Gilaedi Fellowship from the Ministry of Absorption. Already in 1987, a student of David, David Soltz, following a visit to Germany, built an early version of an EBIC set-up, the first of its kind in the country, which permitted characterization of semiconductor junctions *in-situ* in the SEM. Later on using this set-up, David's post-doc Dr. Abraham Jakubovicz, who afterwards went to IBM Zurich, my student Diana Mahalu, and Lev Margulis studied the charge transfer across WSe_2-gold interfaces and published a few remarkable works in *Phys. Rev. B* on this topic.

Sometime around that period, another immigrant from the former Soviet Union, Dr. Konstantin Gartsman started his career in David's group further upgrading the EBIC set-up. Together with a slew of excellent students- Leonid Chernyak (now a professor at Florida State U.), Igor Lubomirsky (currently a professor in our department at the WIS), they were able to obtain with the EBIC/SEM a series of groundbreaking results on ion migration in semiconducting lattices under the influence of electric field and light.

When I came back from my sabbatical in the laboratory of Prof. C. Levi-Clement in the CNRS Merudon (near Paris) in 1989, it became apparent to me that I wanted to stop etching semiconducting surfaces and instead start growing new materials. Working with 2D materials already for 4 years, I decided to give a try on photoelectrochemical cells made of thin film MoS_2 and WS_2, which were prepared by chemical bath and electrochemical deposition techniques. While spending a few months in the laboratory of Prof. A. Fujishima in the University of Tokyo in the summer of 1991, I read the news of the discovery of carbon nanotubes by Dr. S. Iijima. I started quwstioning myself (and later on Prof. Gary Hodes) if it would be possible to prepare fullerenes and nanotubes from other 2D materials, like WS_2 and MoS_2. Independently, during this summer (1991) Lev Margulis was using the EM400 TEM to study the structure of the WS_2 and MoS_2 films prepared by my post-doc Menachem Genut. He found strange nanostructures, which he designated as "red blood cells", because they appeared like donuts, i.e. circular nanoparticles with low contrast in the middle. Without going into too much detail, after a few months of pondering these results, I concluded that these nanoparticles (designated as inorganic fullerene-like- IF and inorganic nanotubes- INT) are the multiwall

analogues of carbon fullerenes and carbon nanotubes. After having published a few joint papers in *Nature* and *Science* on these nanoparticles, Lev Margulis passed away untimely in the summer of 1995. Earlier, Talmon Arad z"l, a TEM expert, who collaborated with Ada Yonath while being at EMBL, Heidelberg, joined the Weizmann Institute. At his request, the Institute bought a new cryo-TEM the CM12, which had low contrast and no electron diffraction, but better resolution than the EM400. Talmon started working with Lev, too, and they went along very well until the tragic death of Lev.

Prof. Enrique Gruenbaum z"l, who retired from Tel-Aviv university and made remarkable contributions to Lorentz microscopy of magnetic materials, joined us around 1995 (or 1996) as a consultant in my group. He helped Yaron Rosenfeld-Hacohen analyzing his newly discovered $NiCl_2$ nanotubes and fullerene-like nanoparticles. Enrique was travelling via boats and trains to Oxford University almost every summer (he did not dare to fly). There he worked with John Hutchison, the foremost TEM of materials expert of that university of the time using the high resolution JEOL 400 keV machine. John had several talented students with him- Jeremey Sloan (now a professor in Warwick University) and R. Dunin Borkowski (currently, a professor in RWTH Aachen University and the director of the Ernst Ruske Center in Juelich- one of the foremost electron microscopy facilities in the world). Due to their joint work, we also gained the first high resolution TEM images of WS_2 and MoS_2 nanotubes.

Following the tragic loss of our personnel in TEM, my research got a boost from a fortunate turn of events. Graciously, Prof. Meir Lahav, the department chair offered in 1997 to transfer Dr. Ronit Popovitz-Biro from his own group to replace Lev's vacancy. To our surprise, in one year or two Ronit became fully versed with electronmicroscopy and started producing splendid results with the CM12 and later on the CM120. At one point in 1998 we submitted our first paper on $NiCl_2$ nanotubes to *Nature*. After some time the report came from the journal. One of the referees argued justifiably that the data lacks an EDS (chemical) analysis of the nanotubes. Indeed none of the existing TEMs in the EM unit was earmarked for materials research and we had no access to EDS/TEM set-up. I was extremely disappointed, because, so I believed, our $NiCl_2$ tubes and fullerenes were not sufficiently stable against humidity and they were scarce. At the advice of Meir, I went directly to the vice president Prof. Yoram Groner to complain. Under his supervision, it took no longer that two months to install the wonderful CM120 which was a boon to our research. The CM120 operated smoothly until four years ago when it was retired due to the arrival of the new JEOL2100. In the meantime, Dr. Ana Albu Yaron (Angie) who just retired from her post in the Volcani Center and worked frequently with John Hutchison in Oxford, joined our group as a consultant. Angie was an extremely dedicated microscopist and I admired her tenacity and patience. Perhaps the culminating point of her work was the synthesis and analysis of IF-Cs_2O nanoparticles in 2005 where she had to build and install a dedicated drybox and attach it to the CM120 in order to transfer the inflammable nanoparticles free of any exposure to the ambient atmosphere. One reason these rare nanoparticles were incredibly difficult to find and analyze was the somewhat bizarre fact that the TEM column was contaminated with IF-WS_2 nanoparticles synthesized by my other students.

Around 2000, while Prof. David Cahen served as the academic head of the EM unit, discussions started of adding an NMR unit for brain research next to the EM unit. Following consultations with the EM manufacturer Philips, the risk of jeopardizing the resolution of the electron microscopes due to interference with the strong magnetic fields of the NMR sunk this plan. One day, the president of the Institute – Prof. Haim Harari contacted me and said that he is ready to offer the EM unit the two lower floors of the old Wolfson building. He asked me to go and visit the site and then call him back. I went there and other than smelling the nasty odor of the mice, noticed that the ceiling is too low to allow accommodation of a modern high resolution TEM. I called back Haim and told him that the level of science there will not be higher than the ceiling height itself. He immediately recognized the problem and said he will build an extra high ceiling room for the future high-resolution TEM (HRTEM). The next day he called me again and said that he decided to build a whole annex to the old Wolfson building with a room for six advanced microscopes. Obviously, I was enthusiastic and impressed by his far-sighted vision. We of course know now that even

the higher ceiling there are not adequate for the new generation of *Cs*-corrected TEM, but at the time it was the greatest present he could possibly offer to us. The new EM center was very well planned to allow cryo microscopy with 20% humidity for biological specimens and equipped with a very modern air-conditioning system, too. To verify that the acoustic noise would not exceed the specifications of the company for HRTEM, Prof. Mudi Sheves – the then dean of the faculty, hired a municipal garbage collecting car, which travelled back and forth along the road separating the EM center and the power center of the WIS, with no noticeable effect on the noise level, which would jeopardize the future TEM performance.

Dr. Sharon Wolf, currently the director of the EM unit, joined the EM unit in 1998 and slowly geared her efforts towards electron tomography. Around 2004 it became clear that a new generation of microscopes is needed for both life sciences and materials research. In particular, the lack of HRTEM became a stumbling block for us and we have desperately pleaded with the management to buy one for us. Luckily, the life scientists and Sharon Wolf also needed a new HRTEM for their electron topography experiments. Under the leadership of our dean, Prof. Mudi Sheves we slowly crystallized and established a deal with FEI to buy the Cryo F20 for the life sciences and the F30 HRTEM for materials research. Unfortunately, we lacked the expertise and did not buy the STEM unit and consequently HAADF experiments could not be done on this microscope. However, the quality of the HRTEM run by Dr. Ronit Popovitz-Biro was so high that at one point Dr. Lothar Houben, who later joined the EM unit to head the Titan Themis project, remarked that with Ronit and F30 you have 95% the capacity of a *Cs*-corrected Titan, which although not completely accurate, was a great complement to her.

Some 18 years ago two excellent students joined my group, Ifat Kaplan-Ashiri and Maya Bar-Sadan. While Ifat made the first in-situ SEM measurements of the mechanical properties of WS_2 nanotubes, Maya became interested in the synthesis of MoS_2 nanooctahedra, which are considered to be the smallest hollow cage structures of that compound. Following a post-doctoral spell in Austin Texas, Ifat joined the EM lab and became the head of the SEM section. Maya, at my encouragement, started to look for a place where she could carry out detailed TEM analysis of the MoS_2 nanooctahedra. Finally, she ended up in the laboratory of Knut Urban, who headed the Ernst Ruske Center in Juelich, one of the largest TEM facilities in the world if not the largest one. Under his supervision, the first aberration (*Cs*) -corrected TEM was developed in 1998 demonstrating the first sub-Angström resolution. In fact, our first work using this revolutionary TEM technique (*PNAS*, 2008) was published as a result of this collaboration. Over the years, I collaborated with various laboratories, mostly in Europe, to gain access and analyze our nanotubes with the latest technology in *Cs*-corrected TEM. Sometime around 2013, I started to nag my department chair Prof. Leeor Kronik and the new dean (Prof. Gilad Haran) that *Cs*-corrected TEM is a must on our campus. At their encouragement I sent a detailed letter to the president (Prof. Daniel Zajfman). Coincidentally, Nobel Laureate professor Ada Yonath understood that the future of protein structural elucidation is in cryo-TEM. Unexpectedly, and to my delight Maya became a life-partner with Dr. Lothar Houben who is one of the leading experts in *Cs*-corrected TEM. Upon returning to Israel in 2011 and joining the faculty of Ben-Gurion University, she convinced Lothar to join her in Rehovot in 2015 and he received an invitation to join the EM center and establish the would be *Cs*-corrected TEM unit here. It takes a vision and courage to make a major decision, like the one taken by our president Prof. Daniel Zajfman to upgrade the EM unit to its present status. In fact after 40 years that I preach for electron microscopy in our campus, this is the first time that the WIS runs an electron microscopy facility which is state of the art. This is of-course not achieved through purchase of upgraded SEMs and TEMs, alone. Hiring experts to run the EM center is no less important and I wish to congratulate the new EM facility and especially the staff and wish them to go from strength to strength.

I would like to conclude my personal account of the history of the EM-materials efforts by saying how grateful I am to the Weizmann Institute which spared no effort or resources to make the entire EM unit a remarkable success story and thank all the current and past researchers and staff of the EM unit for their dedication. I wish to emphasize that this piece reflects my personal perspective of the historical development of the EM center, which is naturally focused on hard matter. I skipped important aspects of

the EM center, which was mostly dedicated to soft-matter and biology or combination of soft and hard matter and was pursued by other researchers in the WIS campus. I apologize if I missed any important aspect and people in my personal account of the EM facility.