May 2020

Scientific "failures"- how did I learn to cope with them and got beyond

Prelude: This document is intended for myself first, and then to draw some insights into future projects and perhaps to other people who can learn from my bitter failures or discontinued projects. During my long 50-years scientific career, I suffered many setbacks and faced many disappointing moments, some of them extremely bitter, others as a result of wrong timing and judgement or simply bad-luck. Some of the "failures" were partial, while others were total. Some of the failures were very costly, while others were essentially mental. When I was young, I thought I could pursue (almost) every research idea, simultaneously. Surely, I would not try to repeat this strategy now, but I was highly motivated to try them when I was younger and psychologically more daring or innocent. Some of these failures were the results of a lack of experience and poor judgement. Some other failures came as a result of my intellectual curiosity and self-belief that I can make it, although intellectually or technically I was not mature enough to face them. Eventually most failures did not disturb me to do what I wanted to do. Although the wounds have long healed, the self-accusations did not stop altogether. In any case, they did not stop me to do what I really wish to do, i.e. the discovery of inorganic nanotubes and fullerene-like nanoparticles in layered compounds (2D materials) and researching this field further-on.

PhD (1972-1976) and post-doc period (1976-1979): My very first failure was the day when I went to Prof. Gabor (Gabi) Stein- my MSc supervisor and the head of the Department of Physical Chemistry in the Hebrew University in Jerusalem and told him that I do not wish to continue my PhD with him, but rather pursue a thesis fully dedicated to theoretical chemistry. He sent me to talk to several potential supervisors, including Profs. Ruben Pauncz in the Technion and Prof. Shneior Lifson in the Weizmann Institute. Eventually, I chose to work on my thesis with Prof. Arieh Ben-Naim in the same department developing models for better understanding the fluid state via statistical mechanics, and hydrophobic interactions in particular. Admittedly and in retrospect, my mathematical skills were (and are) not sufficiently high and limited the scope and impact of my thesis work. However, I learned one important clue to success in science at this point- I felt that I know how to ask penetrating scientific questions, which was key to my later scientific achievements. I recall one day talking to a colleague and offering to do joint work on the "zero-separation theorem" in statistical physics. After few days of literature search, we found to our dismay that this theorem has been solved already for a hard sphere model. At no time, I suggested that we formulate this theorem to sticky hard-spheres, adding thereby attractive interaction to this theorem, which resulted in several joint publications.[1] The next great error was that, in the absence of a better choice, I decided to pursue my post-doc in Geneva – rather than go to the US. My work there turned into some nice publications. However, I did not have the courage at this point to stop this highly motivating, but frustrating intellectual trip and switch back to experimental work.[2] All in all, I spent seven years in an attempt to fulfill an intellectual dream which was doomed to fail from the very first day.

Then came the turning point, following my PhD in Jerusalem and post-doc stint in Geneva. Although I did a nice piece of work in both, I understood that my science is esoteric and as such is doomed to have low impact. Also, towards the end of my post-doc period, I was trying to get a position as a researcher in several Israeli academic institutions, but to my dismay my letters were not even acknowledged. At this point in time, I was heartbroken but I listened to the advice of my post-doc supervisor – Dr. Eric Bergmann who himself moved back from theory to experimental research. He recommended me to look into the work of the photoelectrochemistry group at the Weizmann Institute and join this group. This defining moment turned out to be a boon for my career and allowed me to follow my (scientific) dreams.

1. Distribution Functions at Zero Separation and an Equation of State for Hard-Core Particles with a Finite Interaction Tail, B. Barboy and R. Tenne, *Molec. Phys.* **31**, 1749-1964 (1976).
2. Scaled Particle Theory of Non**a**dditive Hard Spheres: Solutions for General Positive Nonadditivity, R. Tenne and E. Bergmann, *Phys. Rev. A* **17**, 2036-2045 (1978).

Researcher at the Weizmann Institute (1979-1992): Very early on in my scientific work at the WIS, I decided that being (materials) problem oriented and not a technique oriented, I will not try to focus my physical and intellectual resources to any specific scientific technique, but rather try to keep ask interesting questions which could open up new scientific paths. Notwithstanding the examples below, which seem to suggest the contrary, I remained committed to my philosophy and kept the flexibility to move from one materials field to the other. This is precisely the reason why I worked so hard to help establish the *Chemical Research Support Department* of the WIS and in particular the electron microscopy facility and the early version of the clean room in the basement of the Perlman Building. I always believed that such advanced facilities are generic to materials research and will allow moving from one materials-related problem to the other.

In the first ten years at the WIS, I studied photoelectrochemical cells for the conversion of solar energy into electricity and the optical properties of semiconductors. I investigated several families of semiconductors for that purpose and slowly developed the necessary knowhow and experience in materials science; electrochemistry and in optical and electronmicroscopy characterization of semiconductors.

Low temperature optical properties of semiconductor surfaces: After receiving tenure at the WIS in 1985, I took a semi-sabbatical in the Solid-State Institute of the Technion, where I learned of the great potential of low temperature photoluminescence measurements to study semiconductor surfaces. I also paid many visits to the CNRS lab in Meudon working with Dr. C. Levy-Clement. CNRS Meudon was an excellent center for materials growth and characterization with highly sophisticated laboratories for materials characterization including low temperature physics and optical measurements. After long deliberation, I decided to build a cryogenic optical lab of my own and engage myself in such measurements. Towards this idea, I submitted a proposal to the Institute management and to the Israel Science Foundation and received 250k dollars to build such a lab. Since I had no student to take care of this lab, so I built this lab and operated it basically on my own. I started some interesting measurements of electrochemically treated semiconductor surfaces.[1] In the first 5 years of the lab existence (1988-1993) this lab produced one significant result (not in low temperatures though)- selective gettering of iron impurities from CdSe.[2] This very interesting work went almost unnoticed and is poorly cited. The most interesting result obtained with this set-up came 10 years after this lab was established (1998), when the optical absorption spectra of fullerene-like nanoparticles were studied by Gitti Frey.[3] It was observed that, in contrast to the quantum size effect in semiconducting nanoparticles, the bandgap of IF nanoparticles shrinks when their size gets smaller. This unexpected result was confirmed via tight-binding DFT calculations, and is cited quite extensively. This lab was moved to Perlman building in 1998 but ever since did not produce any outstanding data and was finally dismantled in 2006. This bitter experience taught me again that I should focus on (scientific) questions and not on technique-oriented research.

1. Shallow Donor State Removal via Photoelectrochemical Etching of Cd(Se,Te), E. Galun, G. Hodes, M. Peisach, E. Muranevich, and R. Tenne, *J. Crystal Growth* **117**, 666-671 (1992).

2. Photostimulated Gettering of Deep Band-gap Impurities from Semiconductors by Resonance Excitation: Fe from $Cd_{0.98}Fe_{0.02}Se$, M. Homyonfer, H.-H. Strehblow, W. Giriat, and R. Tenne, *Phys. Rev. B* **47**, 1244-1248 (1993).

3. Optical Absorption Spectra of Inorganic Fullerene-Like MS_2 (M=Mo, W), G. L. Frey, S. Ilani, M. Homyonfer, Y. Feldman, and R. Tenne, *Phys. Rev. B* **57**, 6666-6671 (1998).

Rutherford backscattering in the van de Graaf (vdG) accelerator (1987-1995): One day I get a telephone call that a researcher from South Africa- Dr. Max Peisach, who is an expert in chemical analysis using ion accelerators, came for a summer visit in the Institute and is looking for an interesting project to engage in. I met Max and discussed with him possible collaborations, but it was clear that the van de Graaff (vdG) accelerator of the WIS is poorly prepared for studies of the kind we have imagined. We started to work together. Yigal Shachar built a dedicated line for our measurements and we built a vacuum chamber (30 k$). In fact it was built in several stretches when Max came to visit the Institute again and again each summer. Max was the chief scientist of the accelerator complex in Pretoria and had an amazing knowhow in surface analysis of materials using Rutherford backscattering, nuclear activation analysis and other nuclear techniques. In contrast to other surface analytical techniques, here the analytical technique for a given studied surface is very specific. Accordingly, the instrumentation is very specific to a given surface reaction that we have studied. Over the years, after retirement Max immigrated to Israel and settled in Beer-Sheva close to his daughter's family. We worked on several projects, among them the etching mechanism of WSe_2 under illumination was the most enticing one.[1,2] For this project, Moshe the old veteran of the isotope separation project gave me a substantial amount of the precious heavy oxygen (O^{18}) water. The heavy water was left over after the project has been essentially terminated and the facility was shut down and eventually dismantled. With this heavy water we studied the Photoelectrochemical oxidation of WSe_2 electrode. We could discriminate between the tungsten oxide film formed in the presence and absence of gaseous oxygen in the water. This study provided a first-hand evidence for the synergy between the oxygen dissolved in the solution and the water oxygen in the formation of the WO_3 film on the WSe_2 surface. Following 3-4 years of productive work and several joint publications, I felt that I exhausted my imagination in the field and I abandoned this activity all together. I remained in contact with Max for many years and consulted with him from time to time.

1. Controlled Photocorrosion of Tungsten Diselenide: Influence of Molecular Oxygen, D. Mahalu, M. Peisach, A. Wold, and R. Tenne, *J. Phys. Chem*. **94**, 8012-8013 (1990).

2. WSe_2/Tungsten-Oxide Interface. Structure and Photoluminescence, R. Tenne, K. Eherman, K. Mahalu, M. Peisach, W. Kautek, A. Wold, R. Matson, and D.H. Waldeck , *Ber. Bunsenges. Phys. Chem*. **97**, 702-709 (1993).

Evaporation set-up for thing-film photovoltaic cells (1998-2002): In the year 1998 Mr. Palle von-Huth joined my lab as a Ph.D. student. He insisted to do research on solar cells. I offered him to build a set-up to deposit CdTe films on *p*-type (B-doped) diamond in order to fabricate an inverted solar cell where the front (high bandgap material) would be a *p*-type (diamond) material. The *p*-type diamond films were obtained from Jim Butler from the Naval Research Lab in Washington. Palle was certainly my (scientific opposite), i.e. he liked to build new set-ups and this proposition perfectly suited his skills and motivation. In a matter of two years, he built a physical vapor deposition (PVD) set-up and fabricated the *n*-type CdTe/*p*-type diamond solar cells, which were absolutely new (see **Fig. 1** for schematic rendering of the evaporator). However, the performance of these solar cells was rather disappointing.[1] He published two papers and upon completion of his PhD, his 100 k$ PVD set-up was abandoned and later cannibalized. As in the precedent story, the dedicated set-up was built and used for the preparation of one PhD thesis and was not used again.

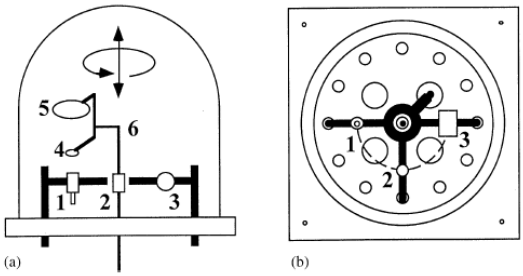

Fig. 2. (a) Schematic side view drawing of the high vacuum reactor setup. 1. CdTe/In evaporation source. 2. $CdCl_2$ evaporation source. 3. Annealing oven. 4. Sample holder. 5. Sample heating lamp. 6. Linear/Rotary sample manipulator. (b) Top view of the vacuum reactor.

Fig. 1. Evaporator for thin film solar cells built by P. von-Huth

1. The Inverted *p*-Diamond/*n*-CdTe Heterojunction Solar Cell, P. von Huth, J.E. Butler, W. Jaegermann, and R. Tenne, J. Electrochem. Soc. 149, G55-G62 (2002).

Hall set-up: Already in 1996, in the absence of electronic characterization tools in the Faculty of Chemistry here, I thought of having a Hall set-up of my own to determine the mobility and carrier concentration of semiconductors. Towards this goal, I bought an old and abandoned magnet from a Physics lab here. My then PhD student Udi Galun helped me installing the magnet in the -1 floor lab in Levine building. Dr. Michael Rapoport from the *Physical Services* built a liquid N_2 cryostat for the purpose of making low temperature Hall measurements. However, the set-up has never took off. After two years, we moved the magnet to the first floor in Perlman building where my lab was set. I think that other than a failed experiment to orient nanotubes of WS_2 film, we have not used this 50-100 k$ set-up anymore. The set-up was finally cannibalized around 2006, too.

Induction oven: One of my preeminent fiascos is the construction of the induction oven, which was the subject of the PhD thesis of Inna Wiesel (2008-2013). I wanted to go beyond the traditional syntheses processes used in my lab, and go to temperatures of up to 1600 °C. She built this relatively sophisticated set-up with the help of an outside expert with a total cost of about 100 k$. For reasons beyond me, she was unable to concentrate on this project and it took her more than three years to turn the keys and start synthesizing nanotubes and fullerene-like materials with this unique set-up (see **Fig. 2** for a shematic drawing of the induction furnace). Up to her graduation, we have published one single paper based on this exceptional set-up.[1] Later on a few more experiments by other students proved to be futile and we did not make any further use of this set-up. When I emptied my lab in the -1 floor (basement) of Perlman some 5 years ago, the induction oven was stored somewhere for several years and then simply cannibalized and thrown to the garbage, not long time ago. I was deeply dismayed when I heard that, because I am confident that this synthetic tool could be used by an active research group. Indeed, this set-up was not simple to operate and use, but it was unique in many aspects.

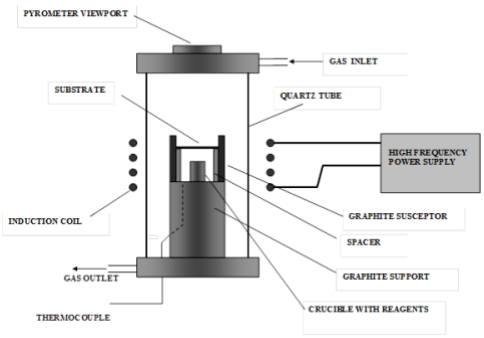

Fig. 2. Schematic drawing of the induction furnace built by I. Wiesel

1. Encapsulation of Mo_2C in MoS_2 Inorganic Fullerene-Like Nanoparticles and Nanotubes, I. Wiesel, R. Popovitz-Biro and R. Tenne, *Nanoscale* **5**, 1499-1502 (2013).

Evaporation set-up for metal-IF films: Another bitter failure, which eventually turned to be useful for another research group, is the thin-film evaporation set-up (100 k$). The idea behind this was to construct an evaporation set-up which could evaporate metal like Ti or TiN and IF nanoparticles at the same time, so as to obtain a self-lubricating and hard metal-IF composite film. Such films could have numerous applications. Actually, two research groups in Europe built dedicated set-ups of this kind before us, but we had several original ideas in our hand. For lack of adequate resources, we bought a set-up with diffusion pump, rather than the desirable turbo-pump. The vacuum provided by this pump was not satisfactory and hence the titanium films were partially oxidized. Also, in the absence of sufficient resources we did not have ion gun for the metal evaporation, and instead we used thermal evaporator which resulted in many compromises and complex maintenance (see **Fig. 3** for the schematic drawing of the evaporator). Nevertheless, both Ohad Goldbart[1] who build the original version of this set-up and Olga Elianov[2] who perfected this set-up were able to come up with significant results and published high-quality papers based on their work.

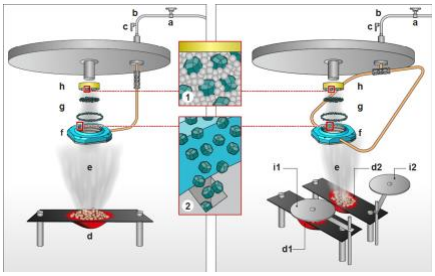

Fig. 3. Schematic rendering of the deposition set-up of the composite metal-IF film constructed by O. Goldbart, O. Elianov with the technical support of S. Garusi.

Lucky enough this set-up was found to be very suitable to evaporate lead films and the set-up was transferred to another group for other kinds of studies.

1. New Deposition Technique for Metal Films Containing Fullerene-Like (IF) Nanoparticles, O. Goldbart, A. Yoffe, S.R. Cohen, R. Rosentsveig, L. Rapoport and R. Tenne, *Chem.Phys.Chem*. **14**, 2125-2131(2013).

2. Deposition of Metal Coatings Containing Fullerene-Like MoS_2 Nanoparticles with Reduced Friction and Wear, O. Elianov, S. Garusi, R. Rosentsveig, S.R. Cohen, Y. Feldman, I. Pinkas, T. Bendikov, I. Kaplan-Ashiri, A. Moshkovich, V. Perfilyev, L. Rapoport, J. Moshonov, R. Tenne* and B. Shay, *Surf. & Coating Tech*. **353**, 116-125 (2018).

Vacuum annealing set-up: Frieda Kopnov joined my group as a PhD student around 2003. Frieda was an immigrant from Russia with basic training in physics. One of her early projects involved measuring the transport properties of pelletized IF-WS_2 nanoparticles and compare it to that of a powder of 2H-WS_2 microcrystallites. The easy part in this project was the preparation of the pellets, their electrical contacts and the measurements, which were done jointly with Dr. Gregory Leitus from the Chemical Research Support Department. The hard part of the project turned out to be the annealing of the samples. Towards this goal, jointly with Alex Yoffe, we built a new vacuum annealing device (20 k$) shown schematically below (**Fig. 4**), which was unfortunately cannibalized as soon as the project was over.

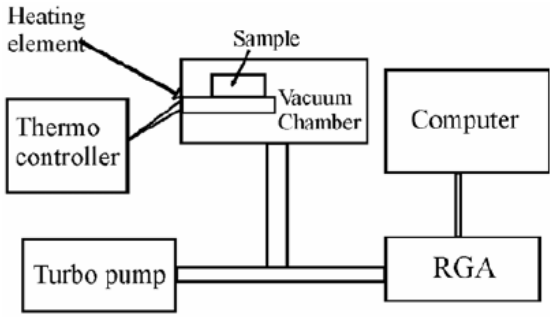

Fig. 4. Vacuum set-up for annealing IF nanoparticles constructed by F. Kopnov and A. Yoffe

The main problem we have faced was the forced diffusion of the heated IF nanoparticles under the high vacuum (10^{-7} Torr) conditions. Once heated under vacuum these nanoparticles would diffuse along the pressure gradient and shorted the electrical contacts of the set-up. These measurements were summarized in a single publication[1] and the set-up has not been used any further and cannibalized shortly afterwards with most of the components used by other groups for different purposes.

1. Transport Properties of Fullerene-Like WS_2 Nanoparticles, F. Kopnov, A. Yoffe, G. Leitus, and R. Tenne, Phys. Stat. Solidi B 243, 1229-1240 (2006).

Transfer chamber to the SEM for humidity sensitive nanoparticles: This project (10 k$) was initiated jointly with Dr. Eugenia Klein around 2008. After completion, we tested this set-up several times, but we have not used it any further.