Chapter 18

Diamond Dreams

It all started as an argument over thermal conductivity.

Dr. Jack Gilman, my boss at the Allied Chemical Material Research Center in Morristown New Jersey, was directing a discussion with his managers on the best material that they might fabricate for maximum thermal conductivity and that could still be useful as a host for optical lasers. This led to my suggesting diamonds, which he dismissed as being almost impossible to grow and totally impossible to modify with other atoms.

I had been supervising a program at RCA Labs a few years before, where I had potential optical laser crystals grown by flame fusion. Flame fusion works by spraying atoms of the desired host material on a hot substrate of the same material. Under the right conditions, the lattice of the substrate forces the new material to grow as a continuous crystal. I thought diamonds might possibly be grown in the same way.

Dr. Gilman hesitated and considered my argument. Jack was a short man and worked at a “standup” desk. He also had no problem in considering and evaluating an idea for several minutes before responding. He normally did this in total silence. It was always interesting in watching a new meeting attendee being unable to sit in silence for such a time and finally speak out, interrupting Dr. Gilman’s thinking. Usually he/she interrupted this way only once. Jack said that he did not believe that diamond could be grown under low pressure. He said that he had read of some Russian work on CVD growth, but that the growth was in filaments. In any event, no work was done on diamond growth at Allied.

Allied Chemical had constructed a New Materials Research Center in Morristown, New Jersey under the direction of Dr. Jack Gilman. The center was set up as an advanced academic site with individual offices and research laboratories immediately adjacent to the offices. The offices were all identical and Dr. Gilman designed the entire building. The offices had modular wall-hung furniture and were designed to place an emphasis on neatness and concentration on the project at hand. This research center design won Jack an award.

Allied (underJack’s leadership) concentrated its material research resources on amorphous metals. These were difficult, but not impossible to grow. One form of making them was so-called “splat cooling” where a blob of liquid material was impacted on a cold wall at high speed. These

sample amorphous metals had some extremely valuable properties, but could be only fabricated by this extremely rapid quenching of a liquid. One very exciting amorphous material was a low loss magnetic composition, which could be formed into a wire by squirting the liquid rapidly through a cold aperture. The resultant wire was ejected at several feet per second. The magnetic properties were great, but producing the material in a useable thin strip some six inches or so wide was a significant engineering project and not really in the charter of the Allied Chemical materials research laboratory.

At Allied at this time, I was a consultant without a group or a project. I convinced Jack that it would be worth our while to try and find a partner that would fund the development of this strip material. I contacted EPRI, which is the Electric Power Research Institute at Palo Alto California. EPRI was funded by the entire electric power industry of the United States and its goal was to develop new and better electrical power systems and material for electric motors and transformers. EPRI agreed on the value of this work and funded the development of equipment to manufacture wide strips of this amorphous metal, leading to very low loss power transformers and very efficient electric motors. The nozzle was replaced by a slit and a large water-cooled drum did the quenching. This 6 inch wide strip came out at some tens of miles per hour and had to be captured and wound on a cylinder. It was exciting early on when the “catcher” did not

work. This amorphous program was ultimately indeed a success and led to the “Metglass” product line for Allied Chemical. I am sure that I recogned that this would be a good project for Allied and that Jack Gilman received a fair amount of the credit for Metgass. This later helped convince Jack to both fund my startup company (XMR) with a laser project and then later convince Amoco that they should invest in and ultimately buy XMR.

At this time I was living in Ringwood NJ and commuting to Morristown New Jersey, which was exactly 49 miles south over narrow roads. After nine months of consulting and commuting, I was offered a full-time job at Allied. Allied was, however, a large and rigid Corporation and did not pay relocation expenses unless the move was over 50 miles. Despite Dr. Gilman's request, the Corporation would not vary from its policy As a result, the family and I accepted a job with GTE Sylvania in Mountain View California. Interestingly, if the commute had been 1 mile longer or if Allied had been slightly flexible, we might have remained in New Jersey.

My ongoing fascination with diamonds had been increased by my earlier work at “Holobeam” which made a laser-drilling tool used for eliminating dark spots in the interior of diamond gemstones. The laser did not actually eliminate the dark imperfections, but just drilled a very small diameter hole from the surface to the imperfection. The imperfection either was a gas, which evaporated through the hole, or was later etched out with acids or other nasty solvents. The diameter of the

hole was small enough so the hole could not be seen with a standard jewelers loop. Many of these systems were delivered to the Jewish jewelers in New York City. The removal of the dark spots vastly increased the clarity and corresponding value of many diamond gems.

Some of the gems that were presented to Holobeam for modification not only had imperfections, but also could not be cleaved due to internal strains. We instituted a series of tests to determine if these gems could be sawed using the laser. This could indeed be done, but there were some problems. Removing the strain often caused the diamond to explode, and the cutting laser beam made carbon dioxide out of the carbon in the diamond and the weight of the finished diamonds was less than the starting weight. Weight loss is a very, very, bad concept in the diamond world. We did, however, sell several diamond saws using lasers. Which cut large diamond “roughs” into gem-style shapes.

This application was successful, but most rough diamonds are not large, but are small tetrahedrons. These “roughs” are then sliced into two pyramids in shops in India where young boys monitor several diamond impregnated string saws. We were approached by a diamond company called “Diamond Distributors Incorporated” (DDI) to develop and evaluate a laser based tool to replace the string saws. The laser machine worked very well, there was minimal material loss and high throughput. Working jointly with DDI, we determined that the gems could be cut for half the cost of the Indian

cutters that are using string saws. We assumed that we would sell several systems, but the law of unintended consequences came into effect and the Indians just dropped their price to undercut us. All our technology accomplished was to lower the standard of living of the Indian gem-cutters.

As a side benefit, I was shown into the DDI diamond sorting room in Manhattan and saw the large tables with piles of stones of various clarity being sorted. I was also allowed to see and hold the 25 carat gemstone that Richard Burton was having set for Liz Taylor. (I have never had so many eyes on me as at one time).

Holobeam not only made diamond-drilling systems, but also made, under government contract, one of the highest power portable laser systems, a 1000-watt unit using multiple pump heads with Nd:YAG crystals. It could ignite a plywood panel over 1000 yards away. Shortly after the Israel Olympic team assassination, I was invited to visit one of our laser diamond driller customers for what I assumed would be another order. However, I was ushered through the office and into a back room where we met with Israeli military officials. They wanted to know how fast we could deliver a truck mounted kilowatt laser system. They made it clear that they intended to use it against terrorists. Unfortunately, they wanted a far faster delivery than we could possibly accomplish. This was another peripheral interaction with diamonds, or at least through diamond dealers.

But back to diamonds. After I left Allied and moved to California, I continued working with lasers, first with GTE Sylvania, a military contracts company and then at ILC Technology, a flash-lamp company that I had worked with while at Holobeam. Dr. Paul Lovoi was the director of R&D at ILC and also interested in lasers. ILC decided to focus on flash lamps rather than lasers. I left ILC and started a company to make excimer lasers. I cleverly called it XMR. The first contract we got was from the same Jack Gilman. He was now a vice president of research at Amoco. It was not an excimer laser, but it was a first product. XMR located in a small facility in an industrial park in Santa Clara. The building was far too big for us, but I shared space with George Shukov, who was making computerized machine tools. In the early days, we had little to show, but George owned three antique cars, two Ferraris and a Riley. George assembled precision motion technology systems and we jointly bid on and won several jobs. If we had nothing more to tell the customers, we showed them the cars. George later combined excimer laser technology with his motion technology and developed, patented, and manufactured stainless steel stents for angioplasty procedures. He later sold the patents and became really wealthy, retired and bought more antique cars.

These excimer lasers emit light in the ultraviolet region and had their greatest success in surface processing of materials due to the extremely strong absorption of the ultraviolet. One

of the materials of most interest was, and is, silicon. XMR did a series of studies of melting the surface of crystalline silicon and evaluating the silicon re-growth. The melted layer of silicon, containing whatever other atoms are adsorbed on the melted surface re-crystallizes at the amazing rate of some half-meter per second. This led to the capability of growing very shallow doped layers of silicon on the silicon substrate. By varying the intensity and duration of the laser beam, we could go from cleaning the silicon surface to melting the silicon surface with the rapid re-growth, to totally ablating the surface layer. We did this in air and various gases and also under a vacuum. This resultant silicon surface was very planar, as the surface tension of the molten layer made it smooth before it could cool and re-grow. We called this "laser planarization." The excimer laser did not have enough energy in a single pulse to planarize a large portion of a silicon wafer, but we demonstrated that almost any wavelength laser would work with the proper energy and duration of the pulse.

XMR was delivering laser hardware to various customers with Amoco being one of the largest, along with IBM. Dr Gilman was interested in high power, efficient, lasers to drive chemical processes that might be of value in Amoco's chemical division. As XMR's business increased, Amoco, through its Venture Arm, invested in, and later, purchased XMR. Amoco, however, was not really interested in surface processing or the semiconductor business and later decided

to divest all of the small high-tech companies it had invested in. It closed the semiconductor processing part of XMR, repurchased all of the semiconductor equipment we had shipped and finally closed XMR. Jack Gilman left Amoco and joined UC Berkley. I left XMR, at Amoco's strong suggestion, and Amoco closed down XMR. My interest in growing diamonds, however, continued.

I had also co-started another laser company, a medical company called "Laserscope" which used green laser light to treat human tissue, from skin resurfacing to prostate tissue ablation and removal. This company was successful and extended my interest in laser interactions with surfaces. I had continued to work with Paul Lovoi and we jointly started another company, SVC, later called "Candescent," to make flat thin television displays incorporating ceramic and lamp technology. We teamed with Hewlett Packard and obtained other venture capital partners and grew this company to where we had a proto-type factory making 13-inch diagonal television displays using field emitter technology and low voltage phosphors, along with many other advances. We investigated using diamond tips on our field emitters. This continued my interest in the deposition of diamond and the subsequent growth of diamond material. We discontinued this work as we discovered that alternate cheaper materials made equally efficient field emitter tips.

These field emitter displays were far more efficient then the existing tube displays The next step would have been a full

production factory, but the price tag for that was about one billion dollars and our timing was bad as the dot-com bust prevented anyone in this country or elsewhere from investing such an amount, even though we had already raised and spent seven hundred million dollars.

After the resultant bankruptcy of Candescent, I was unemployed had time to think, and diamonds returned to the forefront. I had done nothing on diamonds for a few years. I was now working at Light Engineering Inc., a motor manufacturing company I helped start that used Metglass for high efficiency generator and motor systems.

While I was at XMR, we had done work with Arco Solar improving poly-crystalline silicon solar cells by laser irradiating their surface. We obtained good results and patents, but Arco never implemented the process. A few years earlier, I had met Dr Jerry Torrance, a scientist at IBM, who was taking early retirement from IBM research and going into consulting. We worked together on a variety of projects and jointly became interested in Solar Collectors. We started a company called Solar-N to investigate the use of organic dyes and plastics to capture solar energy and to transfer the energy via total internal reflection to the edges of the large panels where the optical energy was some five times concentrated, requiring five times less photovoltaic cells. This process, Luminascent Solar Collector (LSC), looked like it could be less expensive than existing silicon cells. We worked on this process and obtained some good

experimental results. We partnered with Dr. Istvan Gorog, an ex-RCA scientist that I had worked with, but we never were able to find a dye-manufacturing partner to optimize the LSC dye, or a venture investor.

I was now basically retired, but still helping a little on a couple of medical startups, one for sinus surgery and another diagnostic for detecting potential brain aneurisms as well as the amorphous metal based motor company, LE. These consulting jobs decreased with time, the motor company moved everything to China, LSC could not compete with crystalline silicon photo-voltaic cells regarding cost efficiency, both of the medical startups got funding and moved forward.

We had moved into a new house in Cupertino, which sustained considerable damage during the Loma Preita earthquake. I spent a fair amount of time repairing, upgrading, and expanding the house, but the swimming pool and the retaining wall continued to have a tendency to slide down the hill. Eventually we sold the house and moved to San Francisco. This Cupertino house had a large room over the three-car garage, where I had built a fairly large Lionel train layout. This layout went in storage, mainly in my son's attic, with the hope that I would find room at some later date to rebuild it, as there was no room in our San Francisco House.

At this point, I had time to take another look at diamond growth.

Diamonds are a carbon crystal with tetrahedral symmetry. It is a lattice that likes to add carbon and grow if the conditions are right. Obviously not at all temperatures and pressures, or else diamond gemstones would just grow bigger and bigger. The diamond lattice, at high temperature and pressure, rejects other non-carbon atoms, incorporates carbon atoms from heterogeneous molten surroundings and grows, as in a natural volcanic pipe below the surface. The rate at which the diamond crystal adds carbon probably does not depend critically on temperature, the high temperature in the volcanic pipes probably is necessary to make the liquid magna fluid enough so that occasional carbon atoms become adjacent to the diamond crystal and become part of it.

How long actually does a diamond take to grow in nature? It probably takes a very long time as the seed diamond has to wait for an occasional carbon atom to wander through everything else in the volcanic pipe and finally attach itself to the incipient diamond. I consulted with Jerry Torrance who had worked on surface science at IBM Labs and he conceptually agreed.

But once a carbon atom is adjacent and pressed into the lattice, how long before it becomes an integral part of the tetrahedron? When the conditions are right, crystal growth is very fast. The slow time growth in nature must be due to the slow delivery of carbon atoms to the right place and conditions.

Synthetic diamonds are grown fairly rapidly in hot, high-pressure systems. Diamond filaments also grow very fast in low-pressure chambers. In both cases, the growth rate is probably limited by the problem of maintaining the optimum conditions at the diamond surface as the surface changes. The only atoms present should be carbon, so the graphite must be pure and the surrounding chamber initially evacuated with no gases present

Silicon also grows slowly in the steady state conditions in a furnace, but grows much faster as a thin layer on a silicon lattice surface. Diamonds might well have some of the same characteristics. My new idea was to heat a molten pressurized carbon layer incident on a flat diamond surface and let the molten carbon crystallize and grow the diamond.

This could be done by heating only the carbon layer next to the diamond surface with just the right amount of laser energy. Thirty-nine years ago I suggested at Allied chemical to Jack Gilman that diamonds could be grown using a diamond window on a pressure chamber filled with carbon. The window should grow thicker and thicker with each laser pulse. This was my idea, which was never funded at Allied Chemical Labs.

We know the specific heat of carbon, we can guess the absorption depth of the specific laser wavelength, the thermal expansion of carbon at temperature, and we should be able to model the temperature and pressure wave of the hot carbon liquid onto the diamond window surface.

I decided it was the time to do some experiments.

First I had to locate a facility. I found a vacant corner location in the West Portal neighborhood in an ex-bank building that had been used as a Walgreen drug store. A Walgreen consolidation left it empty. Its location, only one block from our house, made it really attractive to me. It was much larger than I needed, but had a great location. About this time I also realize that if I was successful, I did not want any publicity about the fact. I started thinking about some kind of a cover activity. The Lionel trains came back to mind. I took over the bank building and set up the front of it as a model train display. All of the diamond work was done behind a wall behind the trains.

The trains also gave me something to do while waiting for the experiments to run.

This secrecy may have seemed paranoid, but later proved prudent.

It took about two months to locate and to purchase benches and test equipment and to hire a couple of techs. I located a company to design and fabricate a test chamber and purchased the first diamond window. I then had long discussions with Coherent Inc. a laser company, to determine what would be the right laser. I determined that a diode pumped Neodymium YAG fiber laser would be best. I could operate it either in the Q- switched or normal pulsed mode. This way I could control the thermal pulse into the carbon

atoms immediately adjacent to the diamond both in peak temperature, duration, and shape.

The direct growth of graphite to diamond takes place above 100 K bars and pressure and at some 3000 to 4000° Kelvin. If we can obtain this range in the carbon directly adjacent to the diamond for some measurable time, the diamond should grow. Since diamond also grows at very low pressure in a discharge, I decided to start with the graphite and the diamond in a vacuum chamber.

The lattice constant in diamond is about 4 Å. If each thermal wave generated by a laser pulse grew one atomic diamond layer it would require some four million pulses to grow one-millimeter new diamond thickness. Obviously we hoped for much more growth per pulse.

At this time, I ran into my old laser compatriot, Dr. Jim Murray, who had worked with me at Holobeam and is an expert at laser beam shaping, having worked at Lawrence Livermore for years on their laser system. He confirmed that we could deliver almost any shape intensity and duration laser beam that we might need.

The first diamond window I purchased was 3 mm in diameter and 1 mm thick. The pressure chamber had a 2 mm quartz aperture for the beam to pass through to the diamond window. This chamber could be pressurized to about 1Kbar. We started the actual experiments with the chamber evacuated except for graphite. A mechanical pressure plug

behind the graphite insured the graphite was in contact with the diamond. We used the smallest diameter graphite that we could obtain. We then started a series of experiments increasing the laser power. At lower powers, we saw no effect after some 1000 pulses.

We measured the thickness of the diamond window by reflecting a HeNe laser beam off the window at an angle. There was a reflected spot from the front diamond surface and one from the back surface. The displacement between these two reflected spots was a measure of the diamond thickness and our goal obviously was to see this spacing increase. We continued to increase the laser power and at close to the maximum power, we obtained the first measurable increase in the thickness of the diamond window.

During the next several months we varied the laser pulses in duration and intensity. At the optimum performance, we were able to grow 25 lattice spacing per laser pulse. This equated to 1 mm thickness of new diamond every 100,000 pulses. The laser operated at 1000 pulses per second so we grew one millimeter of diamond every 100 seconds.

7 carats per hour!!!!

A seven-carat very high quality diamond is priced at over $10,000 per carat. This is retail price for the largest and best. CVD grown diamond discs are sold for some $2000 per carat. Our operating cost is some $100 per hour, or $15 per carat.

There is a huge industry related to finding, digging up, and selling diamonds. This laser growth process could totally upset the South African economy, bankrupt DeBeers, and make a large number of people want this not to happen.

I now had to figure out what to do with the success. I decided to keep quiet about these results and determine how far I could take the technique.

There were several interesting technical areas that I wanted to investigate. A first one was finding a way to increase the diameter of the cylinders that I was growing. I rearranged the optics so that I had a divergent beam going through the window and thus managed to grow a diamond crystal that got larger as it got longer.

Once it became large enough, we cut a 1 cm clear aperture diamond window, purchased a larger laser, and developed a machine for growing 1 cm diameter diamond cylinders. The linear growth rate was about the same as the smaller machine. The cost of the diamond was now reduced to around $10 per carat.

Using very pure graphite as a seed material, we grew extremely pure clear diamonds. We then started doping the graphite material with very small amounts of impurities and were able to grow pink, yellow, and blue diamonds. Things just kept getting better and better.

Colored Diamonds

We then demonstrated that we could grow very thin layers of colored diamond by evaporating a thin layer of the desired dopant on the inside surface of the diamond window in contact with the graphite. The first re-grown layer contained the color generating dopant with the subsequent layers again consisting of clear pure diamond.

The next obvious project was to grow stripped diamonds by replacing the pure graphite with doped graphite, growing a millimeter or so, changing the dopant, growing another millimeter, and then finishing off with pure graphite.

Slicing diamonds has always been a problem because the diamond is harder than other materials. It is usually done with the string saw, a string impregnated with diamond dust, or with some form of a laser. Both of these techniques had significant loss and were very time-consuming. Since we could grow a thin layer of colored diamond on the surface of the diamond, we changed the wavelength of the incident laser beam to one that was strongly absorbed by that particular layer. We grew a 1 mm thick layer of pure diamond on top of the colored layer by using the infrared laser. We then reduced the pressure in the chamber, pulsed the strongly absorbing laser, and watched the thin diamond wafer cleave cleanly away from the pure diamond crystal.

All of the above growth work was done with the uniform beam and growing uniform layers. The next demonstration

was to pass the laser beam through a mask and grow a pattern on the backside of the diamond window. By changing the pattern and the dopant between successive shots, we manufactured a junction device on the diamond wafer. This process of laser processing through a re-imaged mask pattern was similar to that we did at XMR for International Business Machines Corporation a few years ago.

This demonstration of semiconductor devices on diamond gave me an idea for an exit strategy.

If I could partner with a major semiconductor company, I would potentially have protection from the diamond industry. I proceeded to work with Robert Hill, a local patent attorney and a fellow Michigan State fraternity brother and filed a series of some 20 patents covering all of the above. The patents describe the concepts, but did not reveal that we had actually had any success. I now needed serious help in presenting this idea to a semiconductor company. I contacted Tom Tobiasson, an excellent senior attorney who had worked with me at Laserscope and arranged for him to be our lead legal contact.

He arranged a meeting with Intel, where we displayed a 5 cm long rod, 1 cm in diameter, with colored stripes. We also displayed a 1 cm diameter diamond wafer patterned with a typical semi conductor array.

Our proposal was that they purchase the entire patent package, our technology and all rights for a large initial

payment and a small ongoing fee. I brought John Phillips on board to help negotiate the financial aspect.

We offered Intel the exclusive rights to this process and technology if they made a decision within 10 days. They accepted the negotiating concept and started evaluating.

We calculated the size of the diamond market, the potential profit, and the unknown, but large, potential of diamond-based semiconductors. We used a financial model that I had developed with the expert programming of John Phillips a financial expert retired from NCR. Our initial proposal to Intel was for $1 billion upfront and 5% of the gross sales. We made the argument that they could realize more than that from just the diamond industry and all of the technology applicable to semi conductors would be pure profit.

After signing confidentiality agreements and secrecy agreements, they took the diamond samples for evaluation and testing and set up a meeting for a week later.

At this meeting they presented a proposal involving a much smaller amount of money, which John and I rejected out of hand. They ultimately agreed to our original proposal with the ongoing fee reduced to 2% and with us promising secrecy and agreeing to ongoing consulting with Intel and with Intel owning all future inventions that we might make.

We agreed to five years of secrecy. I am now writing this history under an assumed name from Tahiti. I have no idea where John Phillips is.

If only this story was true! I am still convinced the technical logic is correct, but we never did the serious testing on thick diamond slices.

The Real, Less Exciting Ending

I decided after I had retired from other business activities-that I should try to get a patent on the idea of growing a diamond in this fashion. The basic idea, as I described above, is to bombard a crystalline diamond surface with carbon atoms in a fashion that they will become part of the crystalline diamond structure. The high-intensity short duration laser pulse passes through the diamond and is absorbed by the graphite carbon structure adjacent to the diamond surface. But hot graphite atoms, which are carbon, transfer from the graphite to the diamond.

I worked with a patent attorney friend that named Bob Hill to do the preliminary work on the patent including some searches and drawings. After spending a few thousand dollars, I decided not to attempt to get a patent as it seemed like the idea of the procedure was so obvious that someone must've tried it.

But I was still curious and one day at a laser show, I discovered that a laser company was trying to sell a system with extremely short duration pulses. They were anxious to find some use for the system and offered to loan it to me for a month or so. About the same time I discovered that there was a company actually growing thin diamond discs by

bombarding a diamond surface with hot carbon atoms from a chemical vapor discharge. The diamond evaporation company offered to give me a few 5 mm diameter samples.

My son Peter, had retired, and was racing cars and was looking for something to do and we borrowed an optical table from Dave Horn, a friend from XMR days and set up a test apparatus in Peter's garage. We had no real instrumentation and the laser had fixed energy output pulses we used carbon black as a source of carbon atoms and press the carbon against the diamond surface.

My analysis of our results is that the energy in the laser pulse was large enough that the evaporated carbon atoms generated sufficient heat when they impacted the very thin diamond layer that we used as a substrate, that they locally melted it. We decided that we would need a much thicker diamond substrate and a method to attenuate the laser pulse to be appropriately with the proper energy. Our free loan of the laser was done at this time and the company wanted it back and it seemed like we would need a multi-thousand dollar diamond substrate to continue our experiments.

We mutually decided to abandon any further effort as we both had other interests and the probability of success with a defendable patent seemed slim.

Peter went back to auto racing, I went back to retirement.