DEVELOPMENT OF THE IISc SILICON PROJECT

HISTORY

AND

PRESENT STATUS

PRESENTED BY

Dr. H.S.GOPALA KRISHNA MURTHY
PhD - IPC Department
1972 to 1976

PERSONAL INFORMATION

- COMPLETED MSc INOGRANIC CHEMISTRY FROM MYSORE UNIVERSITY IN 1972
- JOINED IPC DEPT. IN Prof. A R Vasudeva Murthy's group in August 1972.
- Guide Dr. M.Subba Rao (and Dr. T.R.N.Kutty)
- Subject: Study of titanyl oxalates of alkaline earth metals-precursors to titanates
- Submitted the thesis in September 1975
- Received Doctorate in October 1976

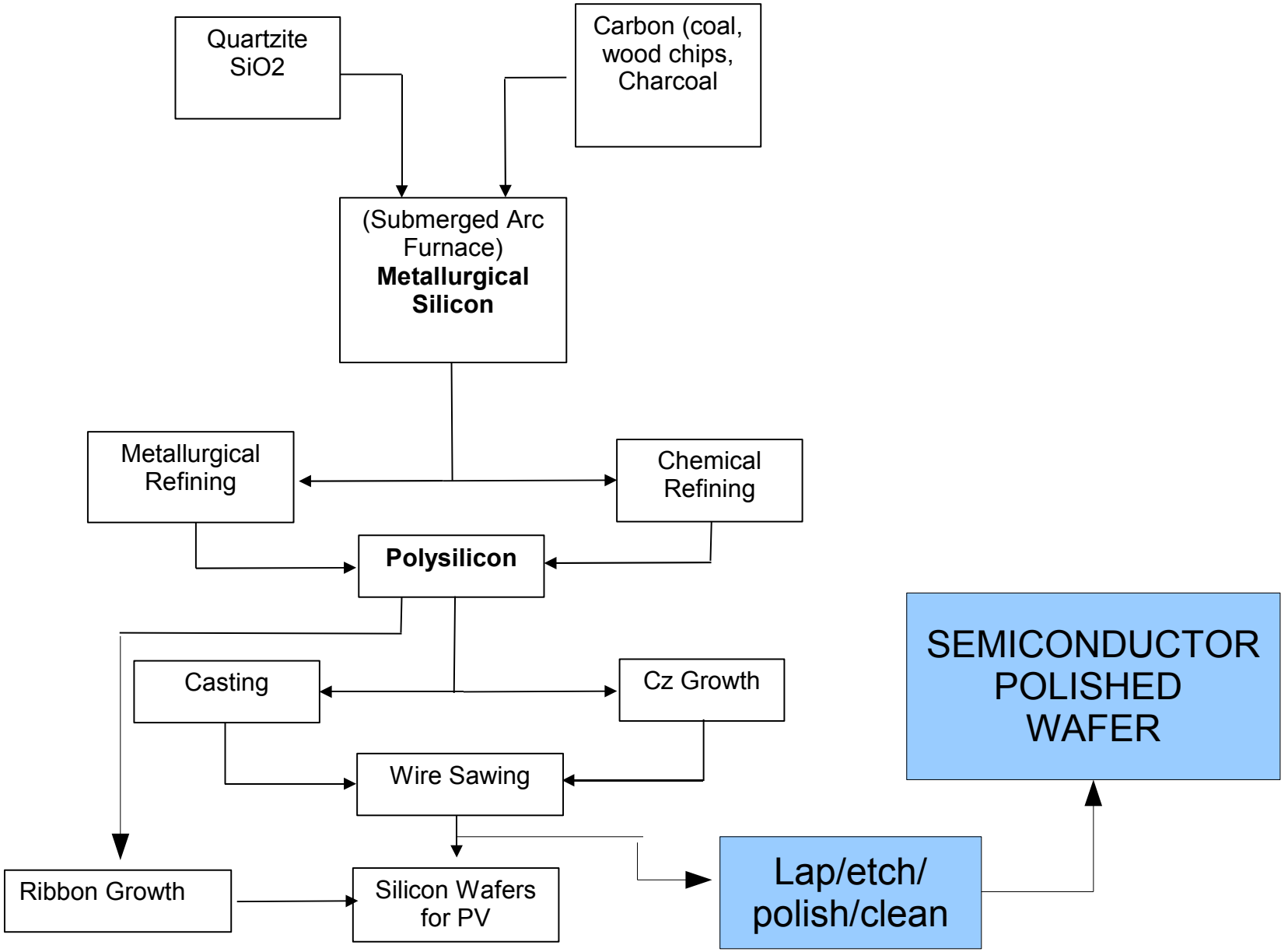
PERSONAL INFORMATION (Contd.)

- Did Post-Doctoral work with CSIR Fellowship from 1976 to 1977.
- Joined as a Project Assistant at IPC/Physics in the Industrial Consultancy Project on Silicon Based Materials sponsored by MCIC in 1977.
- Joined MCIC in December 1978 as Senior Scientific Officer heading their R & D group and worked on silicon based materials for about 27 years
- Left Chemplast in 2004 and running own Consultancy Company ShanGo Technologies

SILICON IS A STRATEGIC MATERIAL

- MICROELECTRONICS IS 95 % SILICON BASED
- PHOTOVOLTAICS USES 87 % SILICON
- ELECTRICAL POWER TRANSMISSION AND CONTROL
- DEFENCE USE IN DETECTORS
- NUCLEAR TECHNOLOGY
- SPACE TECHNOLOGY.

Schematic Silicon Wafer Production Stages



A YOUNG MAN'S DREAM

- Prof ARV as a Second Year Honours student at Central College visited a Ferro- silicon plant at Mysore in 1943.
- He was fascinated with the shining gray material
- He thought it could be a useful material and he should make use of it.
- After completing his MSc at Central College in 1946 he joined IISc as an Analytical Research Assistant when Prof Sanjeeva Rao was HOD

GERMINATION OF SILICON CHEMISTRY

- Prof Sanjeeva Rao's group was involved in chlorination of materials like chromite, ilmenite, and rubber .
- ARV completed his D Sc at IPC in 1953 and was promoted as a Lecturer.
- Prof. K.R.Krishnaswamy was the HOD. He encouraged ARV to study silicon chemistry.
- ARV bought some ferro silicon from Bhadravati where now the ferro silicon factory was located. Chlorine was obtained from MCIC, Mettur Dam.

GERMINATION OF SILICON CHEMISTRY (contd..)

- He made a quartz reactor, added some broken lumps of ferro silicon and after heating with a burner, fed some chlorine from a cylinder.
- The metal started glowing and he could collect more than a liter of silicon tetrachloride.
- He reacted it with ethyl alcohol and made ethyl silicate which he gave to Prof. M.R.Sheshadri of the Foundry Section of the Mechanical Engineering Department for making investment casting. The material worked!.

TENDING SILICON SAPLING

- During 1958-60 IISc was asked by the Government of India to work on silicon
- Prof. Bhagavantam, the Director asked ARV to take up this work. He gave him full freedom to set goals and work.
- Prof ARV teamed up with Prof G Suryan of the Physics Department for this purpose.
- They conducted lab-scale trials on depositing silicon in a small reactor.

GROWTH OF THE SAPLING.

- Both ARV and GS were thrilled with this work.
- Silicon was a rage at that time with considerable work being done all over the world.
- Silicon transistors and the IC chips made using the recently developed planar technology were coming to the market.
- Hence, both decided to continue work on silicon.

INDUSTRIAL CONTACT

- For further work, they needed large quantity of chlorine and hydrogen gas.
- Hence, they decided to approach an industry which was making them in large quantities
- The nearest such industry was MCIC in Mettur Dam, Salem District, Tamilnadu.
- MCIC was pioneer being the first in South India and only the second in the country to produce caustic soda, chlorine and hydrogen.
- Prof ARV wrote to MCIC officials and got an appointment to meet them.

INDUSTRIAL CONTACT

- IISc Director released Rs 60 from a fund he had earmarked for Industrial Development for this visit!
- They went by a bus from Bangalore and reached Mettur Dam.
- They met the General Superintendent of MCIC and started explaining the purpose of the visit.
- As luck would have it, the MD of MCIC, Mr. R.V.Ramani who was a Physicist by training but an Industrialist by Profession, walked in to the room.

INDUSTRIAL TIE-UP

- After the initial introductions, Prof ARV explained the purpose of their visit.
- RVR who had been himself contemplating some diversification on silicon materials was more than glad to hear the two Professors talk on silicon materials!
- Soon an understanding between MCIC and IISc was arrived at for collaboration on silicon based materials.
- MCIC gave the Institute a Project for this work in 1971.

SILICON BASED MATERIALS

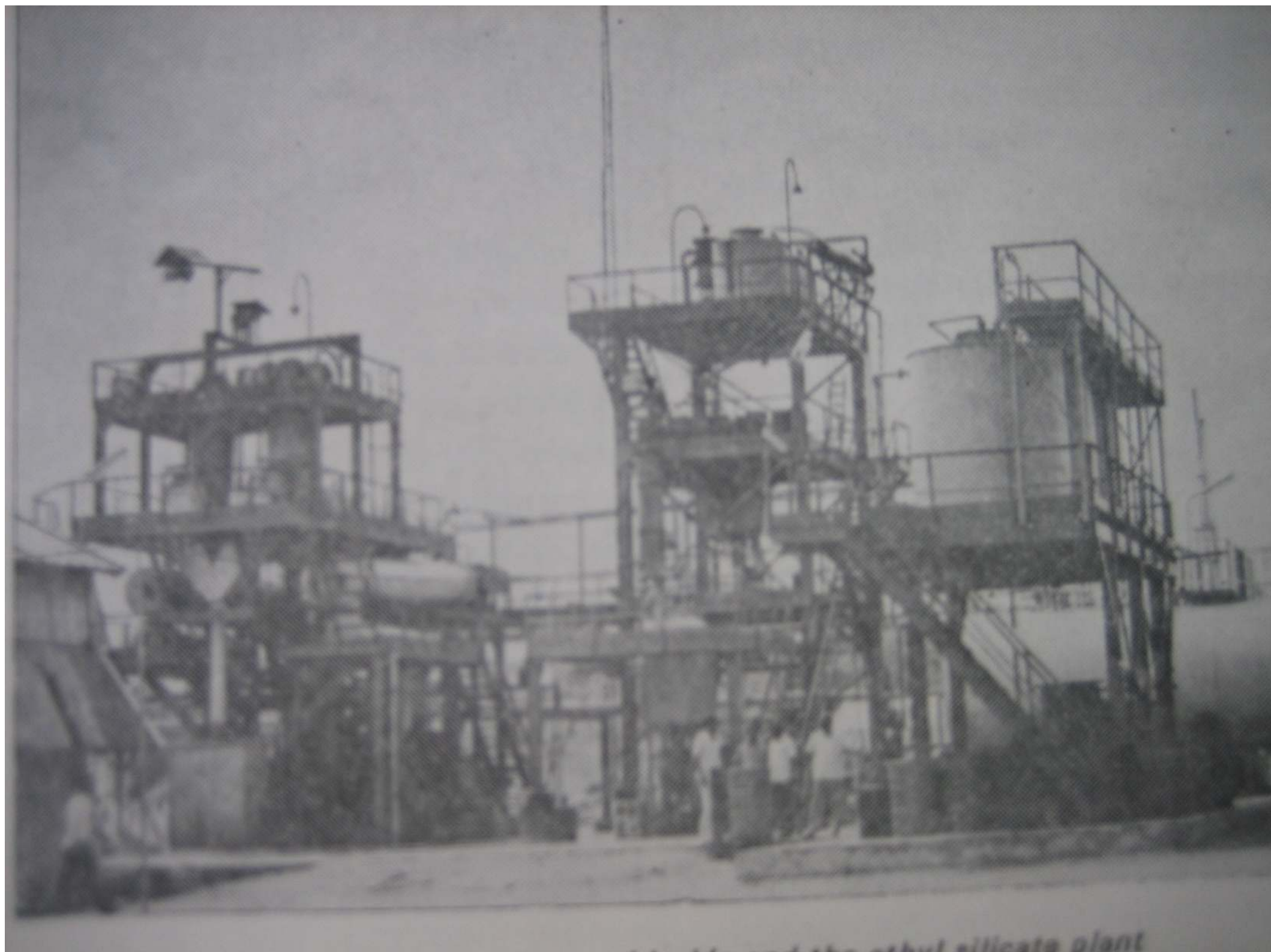
- Sodium silicate
- Fused silica and fused quartz
- Quartz crystals
- Metallurgical silicon and ferro silicon
- Silane and chlorosilanes
- Ethyl Silicate and other silicates
- Organo-chlorosilanes and silicones
- Fumed Silica
- Semiconductor silicon

INDUSTRY ABSORBS SILICON MATERIAL TECHNOLOGY

- RVR at the helm of MCIC was crucial for the strong tie-up between the Institute and MCIC.
- During the course of 1971 to 76, the technology for making silicon tetrachloride and ethyl silicate was implemented in increased scales at Mettur.
- MCIC started supplying Ethyl silicate to local investment casting foundries who were glad to procure it from a local source than undergo the haggles of imports.

EXPORTS NURTURE THE TECHNOLOGY

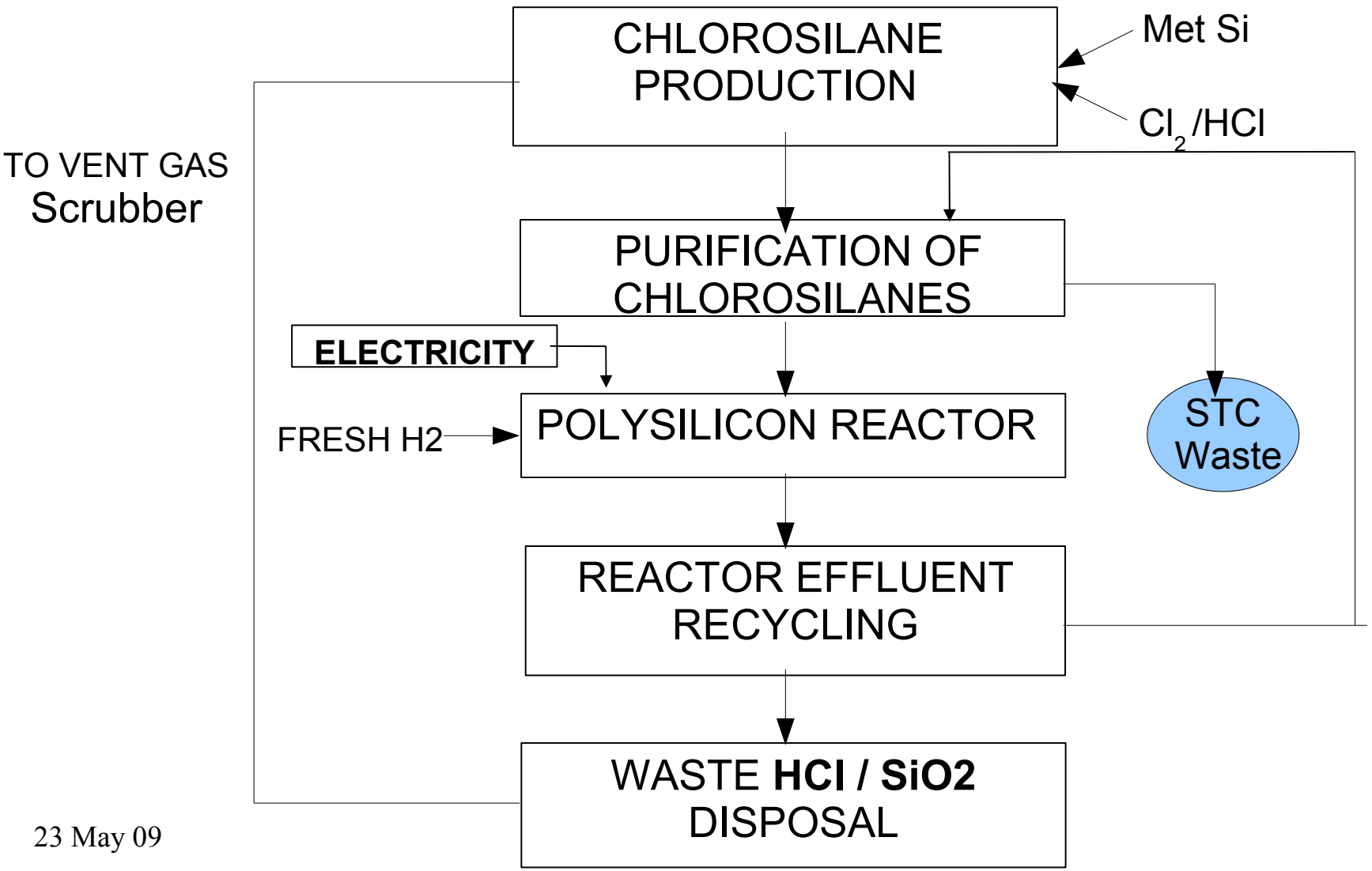
- During his visit to Berbeck College, England in 1977, ARV was approached by a leading user and distributor of ethyl silicate for the material.
- ARV suggested to this distributor to procure the material from Mettur.
- A trial order was placed, material supplied and was accepted.
- A huge order followed immediately.
- By 1977 end a big plant for producing silicon tetrachloride and 1000 tpa of ethyl silicate was started.



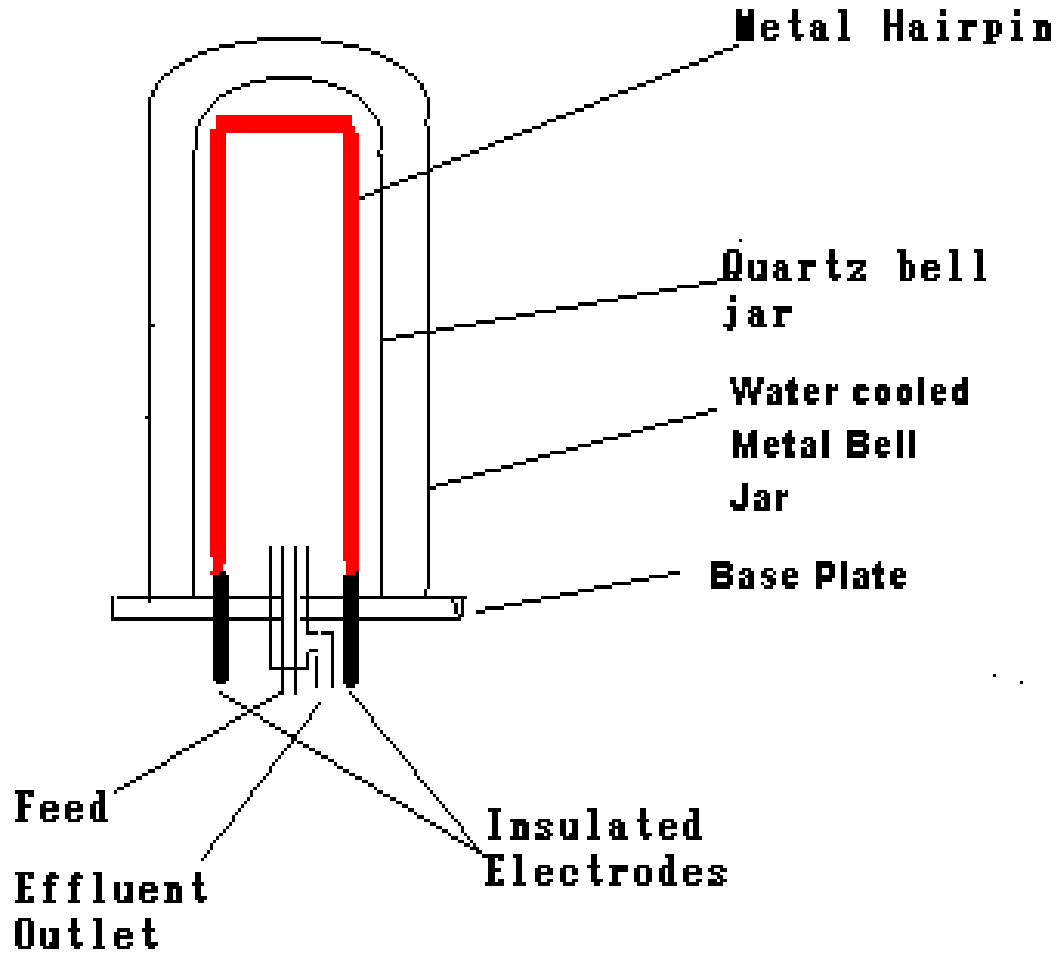
EXPORTS PROMOTE SILICON

- The export business was good. It generated valuable foreign exchange to the Country and good profits to the Company!
- Soon exports to other countries including USA and Australia started.
- MCIC realized the importance of silicon based materials and decided to do further work.
- Since silicon tetrachloride was readily available and hydrogen was available in plenty in the plant, combining the two to produce silicon was the logical next step.

POLYSILICON PROCESS



POLYSILICON REACTOR



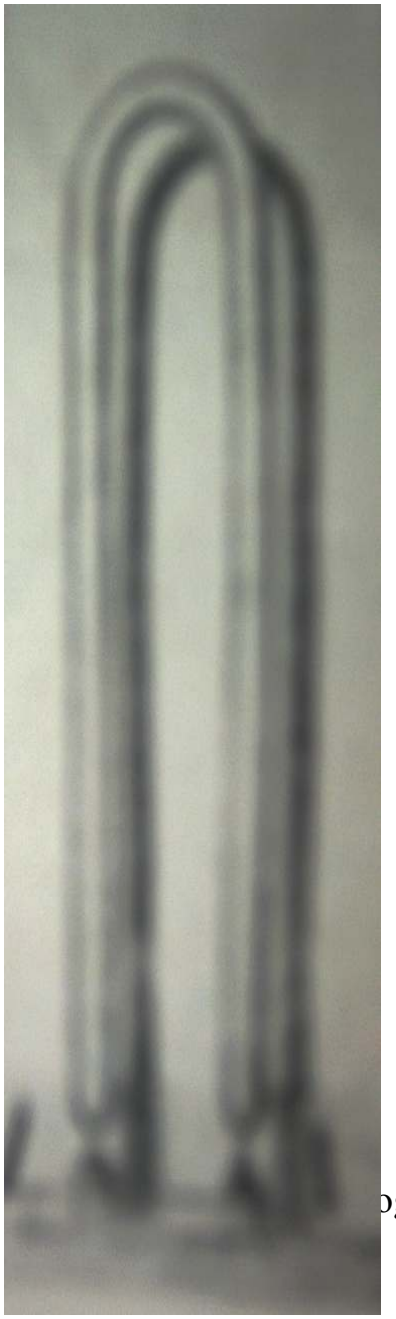
MOVING TO POLYSILICON

- In February 1978, a small set up was made for the purpose. It comprised:
- A purifier for silicon tetrachloride by distillation
- A dryer for hydrogen gas using sulphuric acid
- An Electric Power supply for the poly reactor.
- A small poly reactor, fabricated at IISc with aluminium base plate and quartz bell jar
- Tantalum hair-pins for deposition.
- Optical pyrometer for measuring temperature.

FIRST POLYSILICON PRODUCED

- The entire set up was located in the maintenance shed of the boiler room
- The reactor was run for 24 hours.
- It was stopped because the current reached the maximum of 400 Amps of the power supply.
- The hair-pin was removed and weighed on 19 February 1978.
- 100 gm of Polysilicon had deposited.

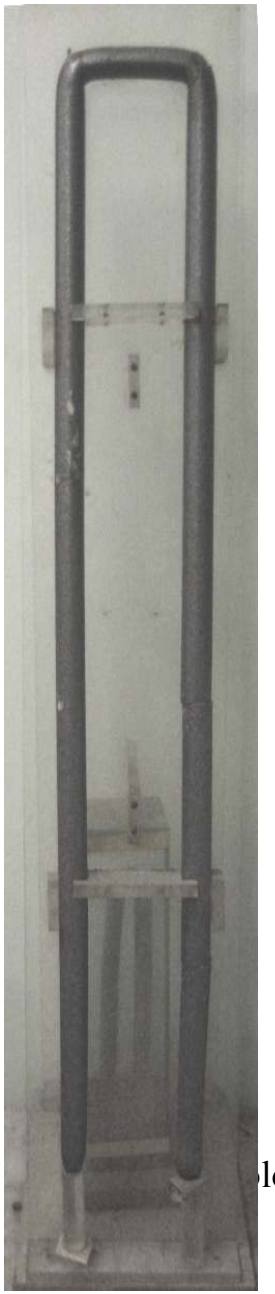
THE FIRST POLY PRODUCED



FURTHER WORK

- Encouraged by the first trials, scaling up of the reactor was taken up
- Next size reactor produced about 1 kg of poly
- The next scale up was for 10 kg
- By 1982 a 100 kg size reactor was being operated.
- Situation was thus ripe for a commercial venture in polysilicon manufacture

NEXT SIZE HAIR-PINS

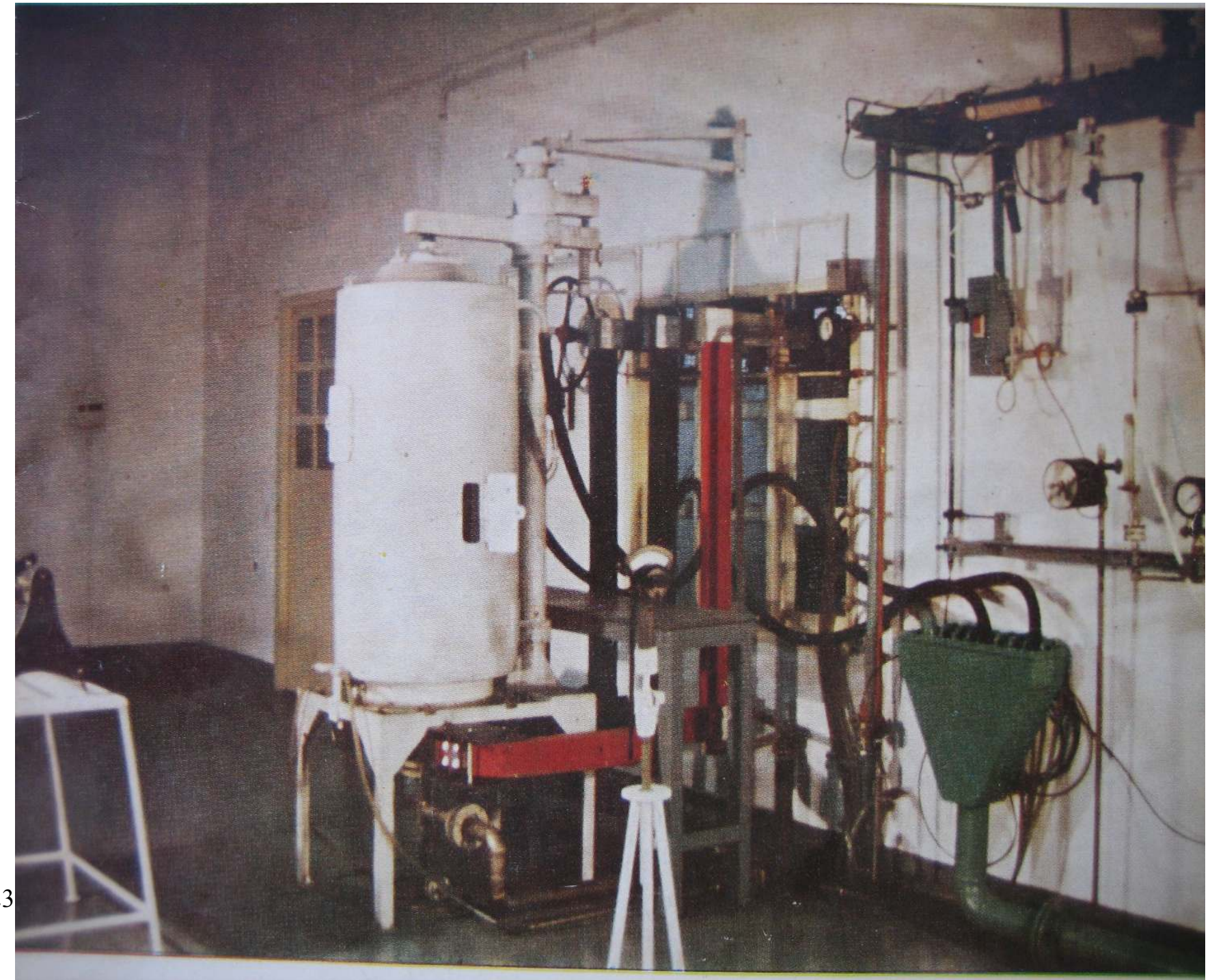


CLOSER VIEW OF NEXT HAIR-PIN

FOUNDATION LAYING

- On 29 October 1982, Mr. Venkataraman, the then defense Minister laid the foundation for the silicon project.
- He visited the R & D set-up and saw the reactor operating and discussed further plans with the Plant Officials.
- He stressed the special significance of silicon to the Defense Department.
- He also stressed the need for Industry and Research Institutions to move closely together.

INAGURATION BY Mr. VENKATARAMAN



CORPORATE CHANGES

- DURING 1983-84 HECTIC CORPORATE ACTIVITIES TOOK PLACE.
- MC&IC WAS TAKEN OVER BY CHEMICALS AND PLASTICS INDIA (CHEMPLAST).
- FINANCES FOR THE PROJECT HAD TO BE NEGOTIATED WITH NEW MANAGEMENT.
- SOME TECHNICAL PROBLEMS IN THE POLY REACTOR SURFACED.
- ALL THESE DELAYED THE IMPLEMENTATION OF THE PROJECT

DARK CLOUDS ON THE HORIZON

- Department of Electronics wanted to establish a National Silicon Facility by importing technology for producing high purity polysilicon.
- A high power delegation went round the world visiting polysilicon manufacturers to explore technological transfer and short-listed a few .
- Another delegation visited the short-listed companies and decided on Hemlock Semiconductors USA.
- Several Indian organizations were working on silicon.

NATIONAL EFFORTS ON SILICON

- NATIONAL CHEMICAL LABORATORY
- NATIONAL PHYSICAL LABORATORY
- BABHA ATOMIC RESEARCH CENTRE,
- SOLID STATE PHYSICS LABORATORY
- NATIONAL METALLURGICAL LAB/IIT Kha
- GRINDWELL NORTON/SILTRONICS
- METKEM SILICON

EVALUATION OF NATIONAL EFFORTS

- A 6 MEMBER NEGOTIATING COMMITTEE CONSTITUTED BY DOE FOR ESTABLISHING NSF VISITED ALL THESE FACILITIES
- THE COMMITTEE REPORTED THAT THE VARIOUS EFFORTS WERE:

RUDIMENTARY;
AT INITIAL STAGES;
NOT SCALABLE;
NOT PROVEN; OR
NOT ECONOMICAL;

THUS NOT WORTH CONSIDERING

IISc-MCIC HOTLY CONTESTED DOE CONCLUSIONS ON SEVERAL GROUNDS

- **Outlay of Rs. 200 crores was too high.**
- **The Capacity of 200 tpa too high for the expected demand.**
- **No down-stream facilities to use poly produced existed.**
- **Cost of production was high**
- **Technology to be provided was incomplete. Technology for production of TCS and disposal of STC produced was not included in the offer.**
- **No effluent handling procedures were available.**
- **Purity of material would be lower as STC used by Metkem gives better purities**

SILICON CONTROVERSY

- Two belligerent groups evolved
- The first group sphere-headed by DOE vehemently supported the import of Technology.
- The second was the newly formed DNES which supported IISc-MCIC technology.
- DNES wanted silicon to be under its jurisdiction in view of huge Si demand for PV.

DOE BULLDOZING

- Undeterred by these, DOE went ahead with its discussions with Hemlock.
- It justified that the outlay of Rs. 200 crores was not for the poly plant alone but for an R & D center also.
- It signed an MOM with Hemlock for the Project by late 1983?
- The Contract stipulated stiff penalties in case DOE rescinds the contract.
- DOE started preliminary work at IPCL Baroda

US GOVERNMENT VOLTE-FACE

- US Government refused permission for transfer of technology in early 1984 since silicon is a material of strategic importance.
- In early 1985 (17 Jan 1985)SILTEC a leading Silicon equipment supplier in California got Metkem's Silicon tested by MIDAC Corporation USA. The report stated that this material was as good as made any where else!
- Coincidentally within a week of this announcement, US Government released silicon from the list of restricted technologies not to be transferred to India!

DOE SIGNS WITH HEMLOCK

- Immediately DOE signed a contract with Hemlock for supply of technology and equipment for a 200 tpa polysilicon plant at a cost of US $ 14.2 M.
- It paid US $ 6.7M for technology.
- The plant was to be established at IPCL Baroda in about 4 years time.
- It is to be noted that Hemlock is a manufacturer of Poly. It buys its reactors and related equipment from other manufacturers.

DEVELOPOMENTS IN OCTOBER-NOVEMBER 1984

- Smt. Indira Gandhi was assassinated in October and Rajiv Gandhi became PM
- He promised to foster indigenous technologies.
- Taking a cue, Prof ARV and GS wrote a letter to Rajiv Gandhi complaining that the indigenous technology for silicon was being killed by DOE.
- Rajiv Gandhi, as a Pilot knew the significance of electronics and silicon.
- He therefore took personal interest in this subject.

ASSESSMENT OF METKEM

- Rajiv Gandhi called the Professors for discussion.
- Professors and RVR explained the issues involved.
- As NSF would take at least 4 years to come up, Professors pleaded that they be given a year to demonstrate the indigenous technology.
- Rajiv Gandhi agreed that this was reasonable.
- He told DOE to hold NSF for a year till Metkem Technology is evaluated. This in spite of Varadarajan Committee Report favouring NSF

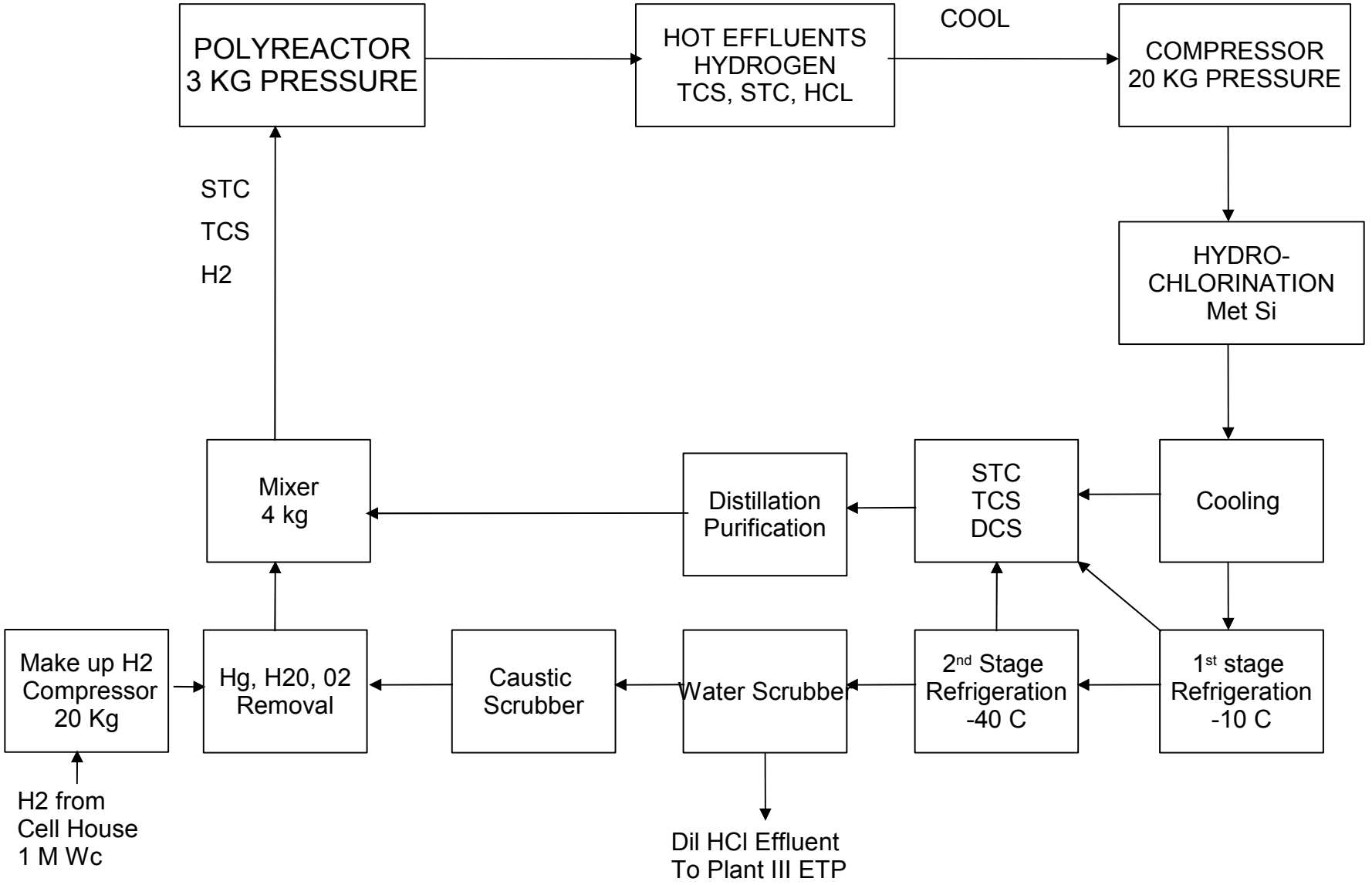
ONE YEAR TO DEMONSTRATE!

- Metkem was given one year to put up a 25 tpa polysilicon plant and produce the material
- Though this time is too short for such a task, (which involved Basic Engineering, Detailed Engineering, Civil work, Equipment ordering/fabrication, Erection, Piping and instrumentation, Utilities, Testing and commissioning) this was taken as a challenge
- Work was started in April 1985.
- Work was completed by February 1986.

TEETHING PROBLEMS

- TNEB refused to supply power as their was power shortage.
- Argon gas required was not available.
- Prime Minister's office intervened to remove these hurdles.
- The plant was successfully commissioned in March 1986,
- First production was reported in early April 1986.

POLYSILICON CVD PROCESS 1986



TASTE OF THE PUDDING

- The polysilicon had to be tested for quality. Metkem had still not installed crystal growth and wafering facilities.
- DNES got a crystal grown in Calcutta with Metkem poly which was wafered at BHEL Bangalore who also made solar cells out of the wafers and the results were satisfactory.
- 30 kg of material was sent to Japan for evaluation by DNES. Excellent results.

SUCCESSFUL DEMONSTRATION

- A rumor was floated that Metkem had not made poly at all but had clandestinely imported some quantity which was given for evaluation.
- Representatives of DOE and DNES visited Metkem, stayed for a few days, observed the operation of the plant and saw the actual production taking place!
- They were fully satisfied with the working of the plant, the material and cost of manufacture

METKEM STANDS TALL

- They reported to the PM's office that the plant at Mettur was running and is successfully producing polysilicon at competitive pricing.
- Prime Minister was convinced that the indigenous technology was good and hence, there was no necessity for import of technology.
- DOE was asked to abrogate the contract with Hemlock.

DOE Plight

- DOE abrogated the contract with Hemlock
- US $ 6.7 M already paid however was lost as a penalty.
- This was severely criticized by CAG in 1997.
- Investigations were made by CBI on this fiasco.
- DOE funded to establish a facility for characterization of local silicon at the Physics Department of IISc.

METKEM DEVELOPMENTS

- GOI decided to encourage Mettur as a center for silicon development.
- Grant-in-aid and loans were provided by the Government to improve the process and reduce cost of production (probably for the first time to a private Company!)
- Two projects were taken and successfully completed between 1997 and 2000.
- Mekem got the admiration of DNES as one of rare examples where Projects were successfully completed and targets fully met.

DNES OFFICIALS AT THE PLANT

PRIME MINISTER VISITS METKEM

- On 20 JUNE 1978, Rajiv Gandhi visited Metkem.
- Though the visit was for 20 minutes, he spent more than100 minutes in the factory.
- He saw the poly reactors, asked very penetrating technical questions.
- He appreciated the plant layout and saw the operations of the crystal growth, wafering and polishing departments also.
- He and Mrs. Sonia planted saplings of “Pride of India” in memory of the visit.

INDEGENOUS TECHNOLOGY

- At a hurriedly called press conference at the plant, Rajiv Gandhi declared “Here is a indigenous technology defeating foreign technology”
- He promised full support to Metkem.
- He had lunch with all the plant personnel and showered praise and encouragement to all of us.

RVR EXPLAINING TO RAJIV GANDHI

Rajiv Gandhi Walking in the Poly Plant Area

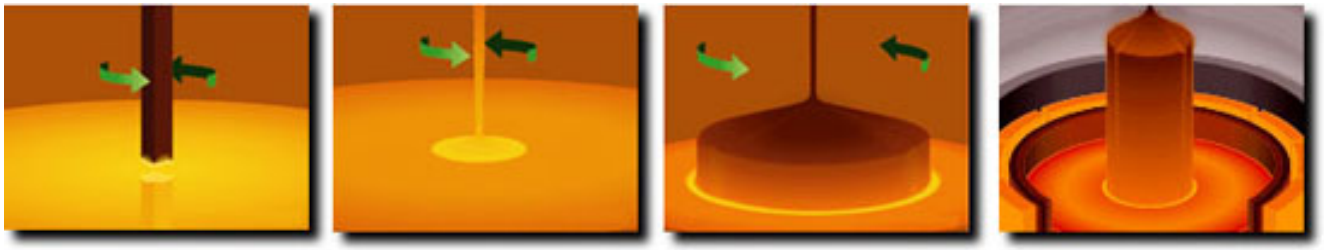
GROUND REALITIES

- Within a few months of starting, the plant had produced more than 10 tons of poly silicon.
- Metkem had just commissioned its crystal growing furnace and wafering machines.
- In-house requirement for one crystal growing furnace was about 5 tons only.
- Inventory of poly build up was because there was no other taker for the poly in the country.

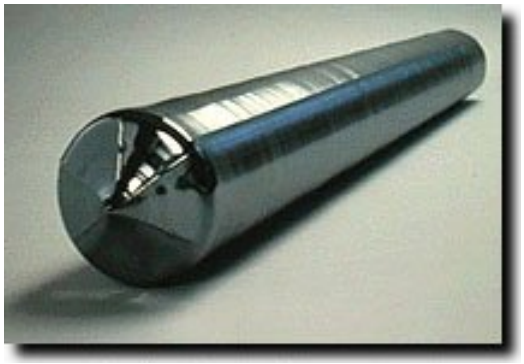
POLY SILICON IS NOT THE END

Polysilicon is itself, not useful.
It has to be converted in to single crystal.
The ingot has to be cut in to wafers.
Wafers are used for making devices and solar cells
Thus making polysilicon is only the starting point.
Down stream facilities of crystal growth and wafering are essential for using polysilicon.

Crystal Growth



Ingot



Further Processing

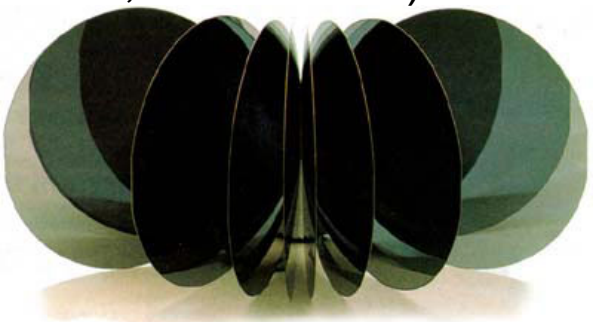
Wire sawing of wafers

Lapping and etching

Thickness sorting & flatness measurement

CMP

Quality inspection (electrical, dimensional)

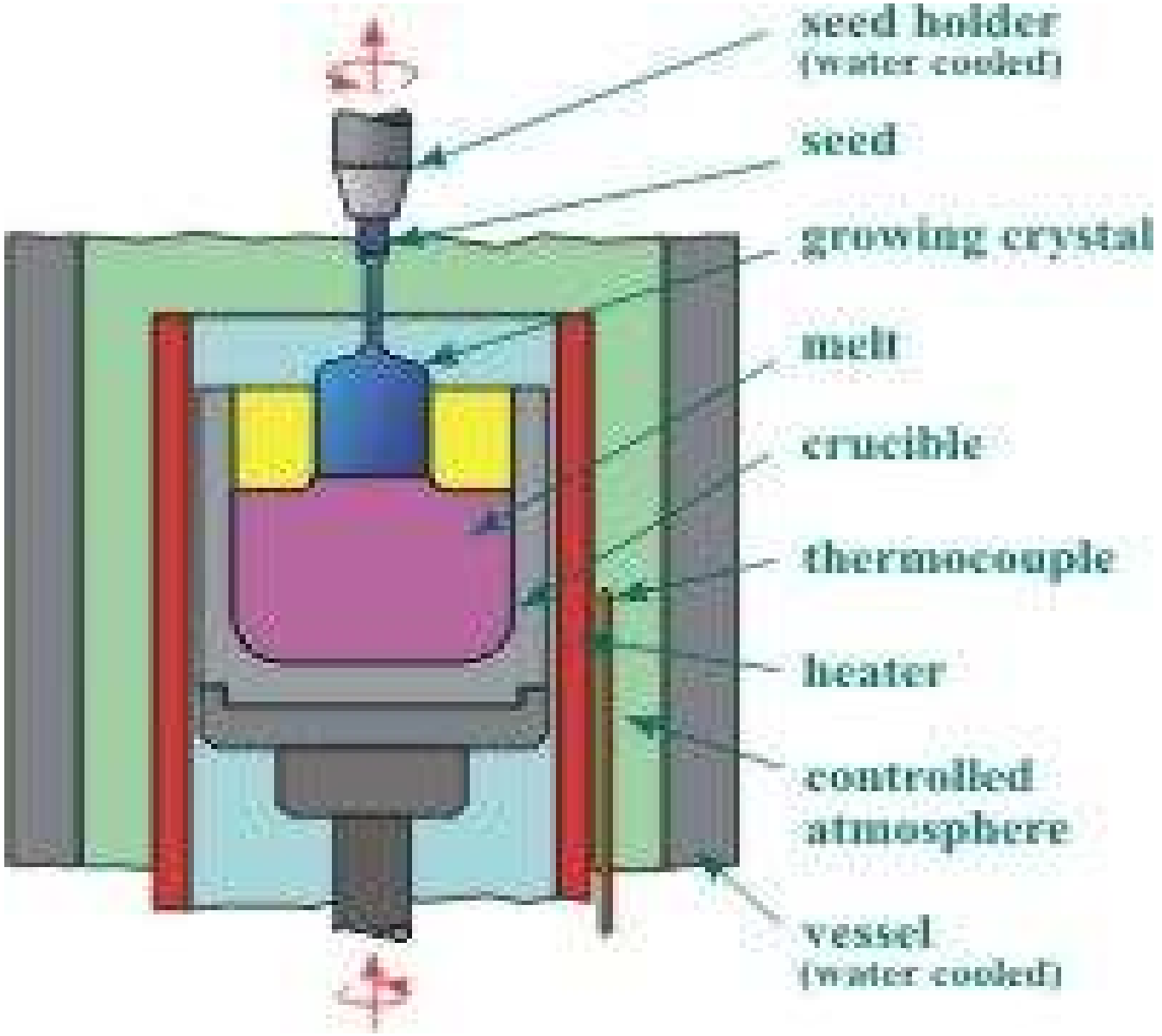


DOWN STREAM OPERATIONS

- While there were considerable imports of wafers by Companies like BHEL, CEL and BEL, for making solar cells,there were no facilities for down-stream operations of crystal growth and wafering.
- Metkem had to invest further on these activities to consume its own poly. Stabilization of all these took time.
- Wafers could be imported freely without any duty. This was to encourage PV growth. Hence, Metkem had to sell wafers at International prices. No Protection!

DIFFICULTIES FACED

- Crystal growth and wafering are highly specialized processes till then not familiar to Indian Industry.
- Metkem with its Chemical back ground had to go through the learning curve of these operations.
- It faced innumerable problems of spares, consumables like silica crucibles, graphite heaters, ID Blades and even supply of pure argon gas which was in short supply.


seed holder
(water cooled)
seed
growing crystal
melt
crucible
thermocouple
heater
controlled
atmosphere
vessel
(water cooled)

OTHER DOWN-STREAM FACILITIES CLOSED

- Siltronics which was established at Hosur during 1980 by Grindwell Norton closed down in 1986 as it could not produce 4” wafers.
- Super Semiconductors Calcutta which had a small facility also closed down an un-viable.
- BEL had long back closed its crystal growing and wafering facilities.
- BHEL which had established crystal growth and wafering facilities also could not operate them.
- Could Metkem survive under these difficulties?

DIFFICULTIES IN SURVIVING

- TNEB which had assured concessional power withdrew this in 1990. The power tariff was heavy and unbearable.
- Metkem could not meet customer requirements in time.
- The only silver lining -- the quality of the wafers was excellent for solar cells.
- CEL gave for successive three years incentives to us for crossing their RFOM compared to Wacker wafers! Then Metkem wafers became their reference!

CEL CMD PLANT VISIT

CEL CMD PLANT VISIT

DGTD AWARD FOR TECHNOLOGY DEVELOPMENT

ELECTRONIC WAFERS ADD TO THE WOES

- Facilities for making electronic wafers was established at a high cost including facilities for lapping, polishing, cleaning and packing of polished wafers.
- Wafers were given to BEL for evaluation
- The quality of the wafers were found to be satisfactory with several batches of wafers.
- However, when it came to buying regular wafers, BEL back-tracked with some lame excuses.
- They accepted to buy only test wafers from us.

UNEXPECTED ASSAULT

- By 1992 the plant was streamlined and could stand on its own legs—at least no operating losses though the accumulated loss was a burden.
- By late 1992 and 1993 Russians started dumping wafers at throw-away prices. All customers started grabbing this material.
- Metkem was forced to match the ridiculously low prices offered by Russians even though the Russian wafers were junk.

UNBEARABLE SITUATION

- Energy consumption in making poly had been reduced considerably and the cost of virgin poly was comparable to world prices even though tariff was high and the plant was small (25 tpa.)
- Still, for making PV wafers sold at low prices the cost of poly was high.
- At this stage, a German manufacturer of poly offered to supply our requirement at very low prices almost at 30% of our cost.
- They offered semiconductor reject poly which however was good enough for solar wafers.

STOPPING OF POLY PLANT

- In 1993, Metkem was the 7th plant in the whole world producing poly.
- The six producers had formed a undeclared trade block like OPEC.
- They never allowed others to come up till 2007
- In the guise of helping Mekem,they wanted to stop Metkem poly operations.
- They succeeded and Metkem Poly plant was stopped by 1993 end. Short term gain overshadowed long term repercussions.

BUSINESS WINS

- By importing poly from Germany the cost of manufacture of wafers was reduced
- In 1994, a new wire saw was procured
- This could produce 30% more wafers per kg of ingot than the ID Saws which were in use.
- Within a year, the wire saw was made to produce 3 times its rated capacity,
- Ingot production did not match the requirements of the wire saw and hence, Ingots were imported from Germany.

COST CUTTING TECHNIQUES

- Argon gas Recovery and reuse
- Local graphite parts manufacture
- Using thinner wires
- Recycling of wire saw abrasive and vehicle
- Recycling of water used for washing
- Improving production and increasing yields to 98 to 99% by educating the workforce

COMMERCIAL SUCCESS

- The cost cutting measures yielded excellent results.
- Though the plant was tiny, still it could produce wafers at low costs. In fact, the cost was one of the lowest in the world
- From 1995, Metkem started making profits
- Additional crystal growing equipment were added in 1995.
- Up to 2002 the unit was making good profits.
- However, Management did not show much interest as its contribution was minuscule

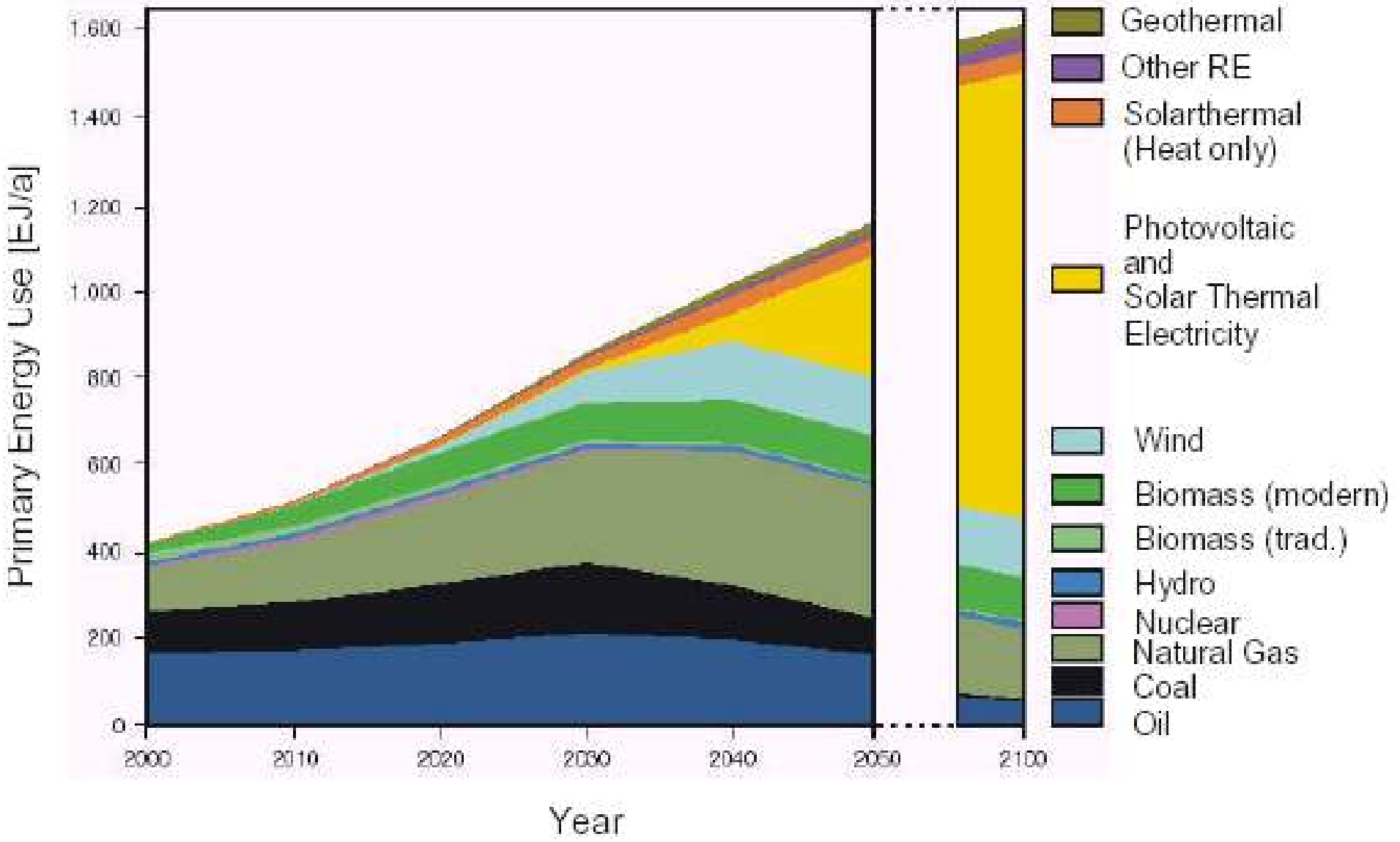
UNIQUE PROCESSES

- Many of the cost cutting processes developed were unique.
- When pressure of reducing costs became important, many others who visited the plant showed interest in such steps
- Chemplast did not show any interest in expansion of the Metkem activities or joining hands with others.
- I left them and started my own Company in 2004 to help clients in implementing silicon wafer projects

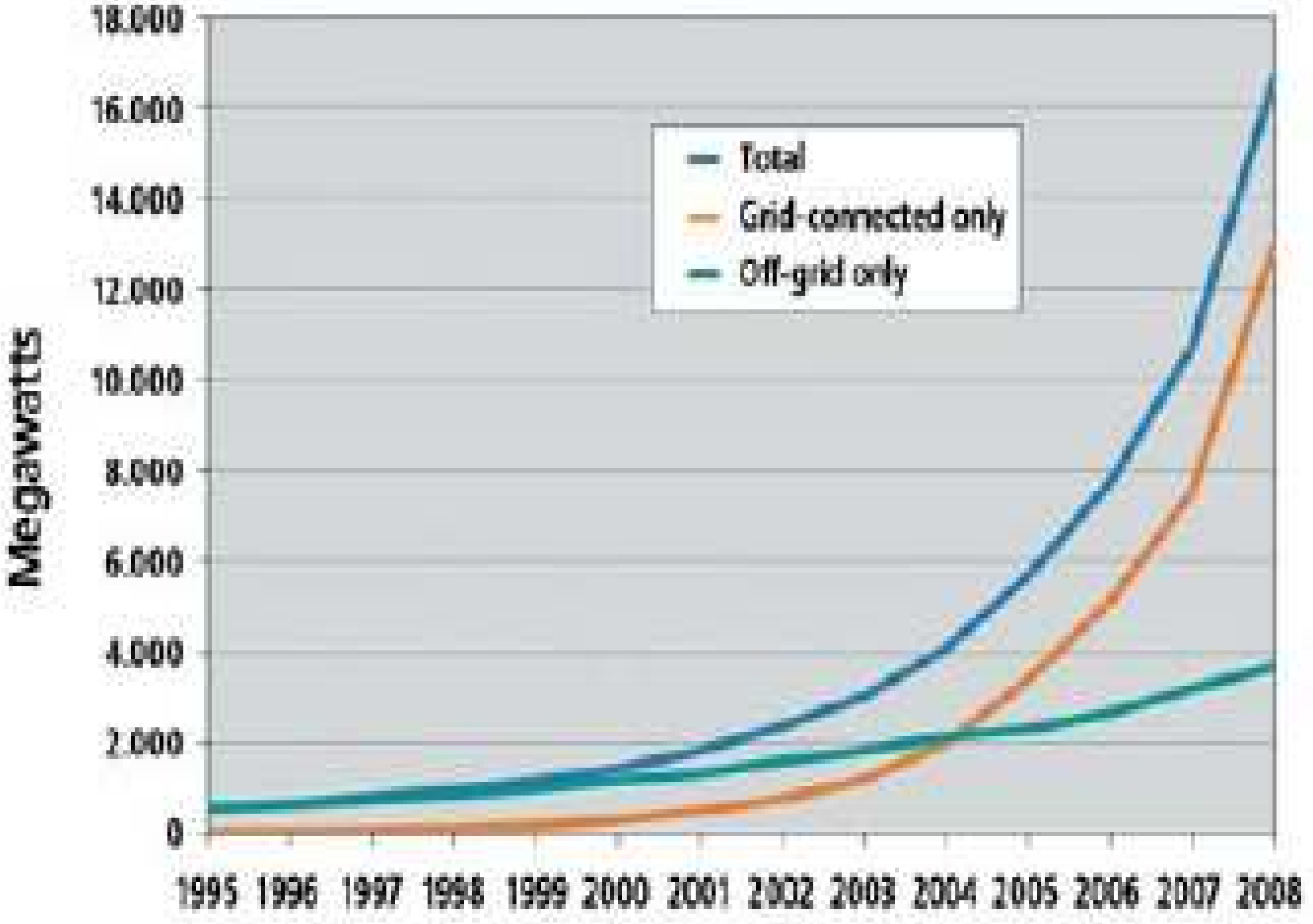
RESTARTING OF POLY PLANT

- From 2003, PV started growing at a rapid rate.
- Demand for poly and wafers increased drastically
- Pricing of poly and wafers galloped.
- Chemplast made good money by selling wafers at high prices.
- However, import of poly became difficult
- In 2005, they wanted to restart the poly plant

PREDICTED FUTURE ENERGY SCENARIO

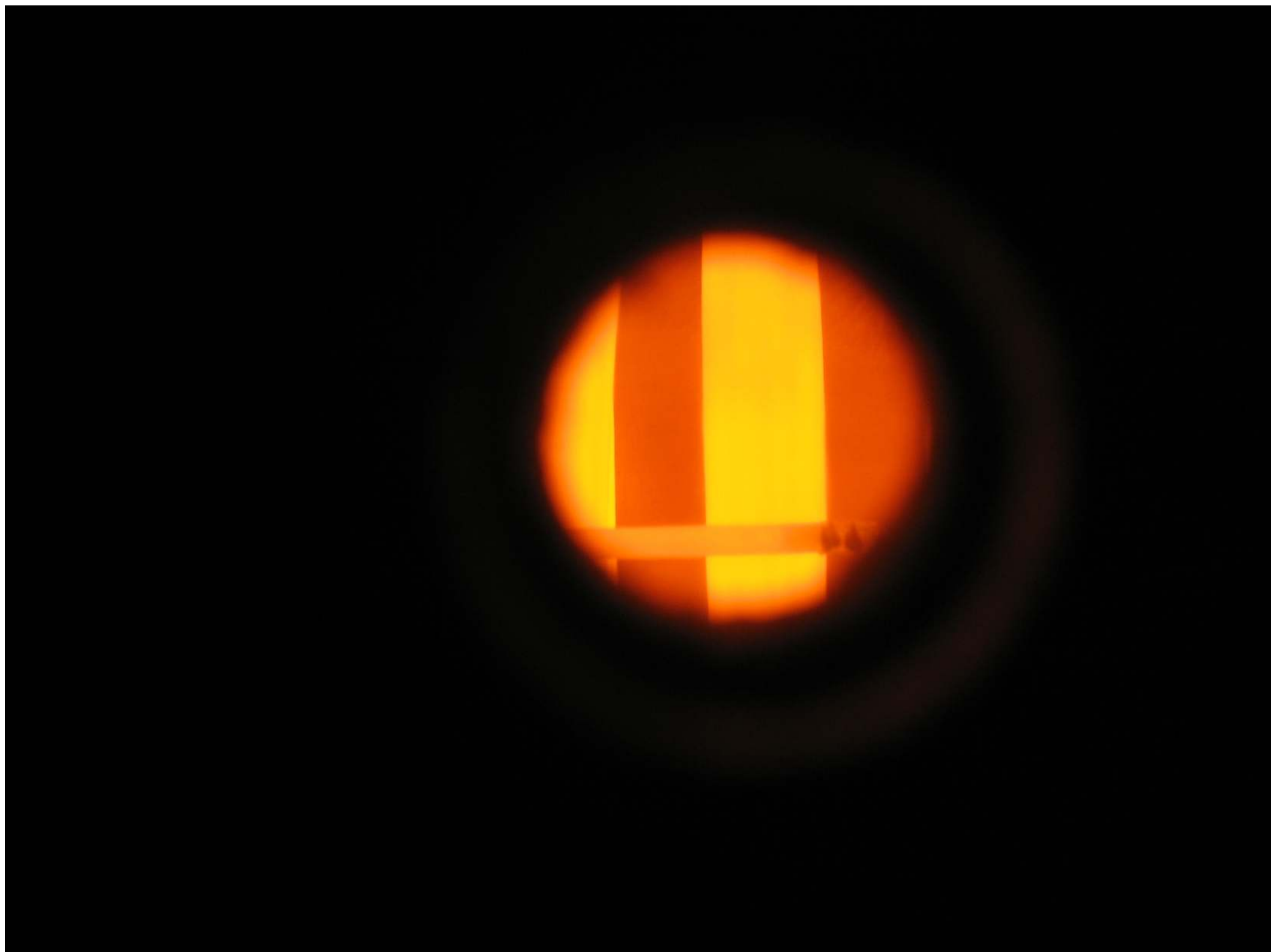


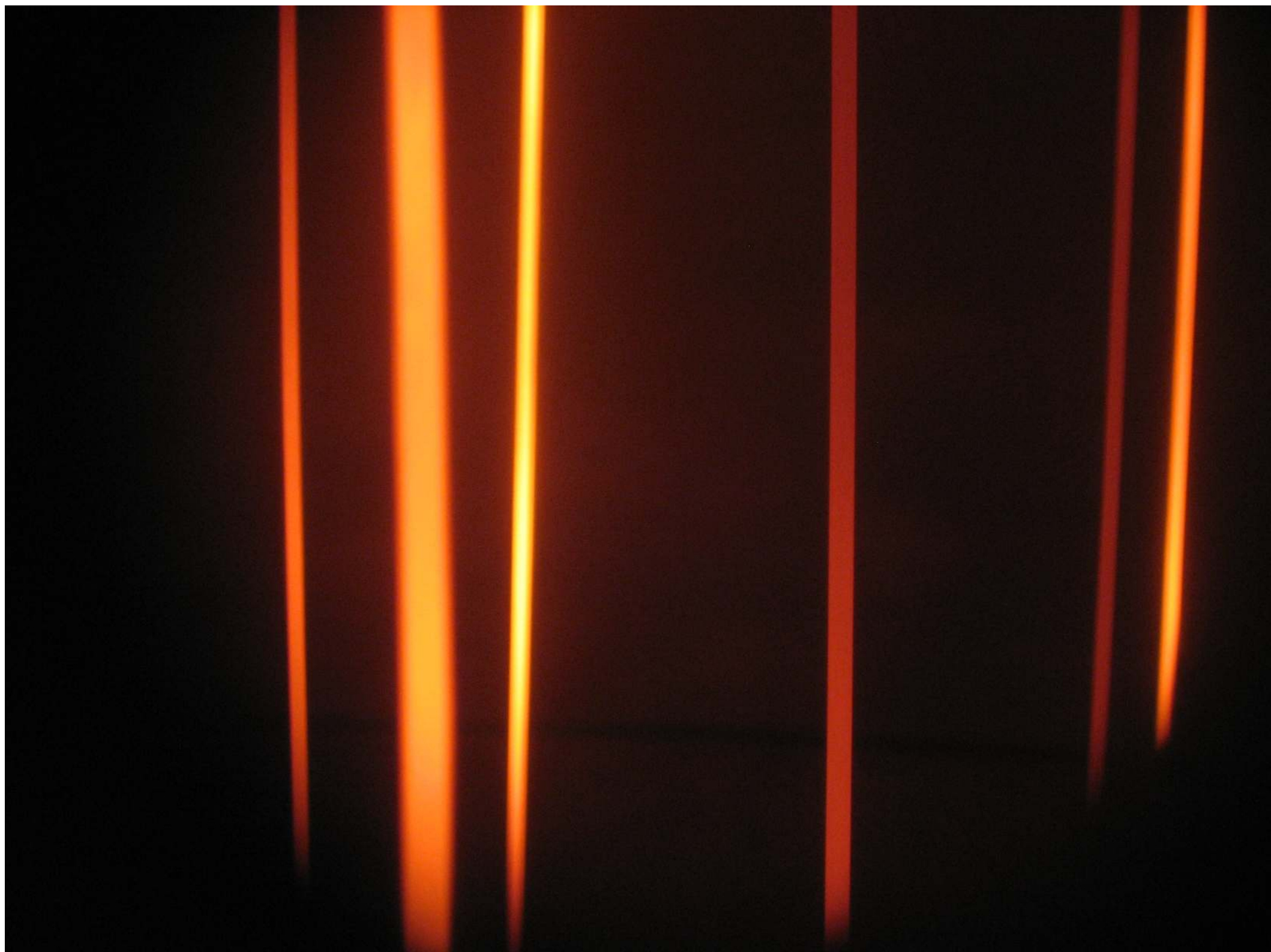
Solar PV, Existing World Capacity, 1995–2008



NEW POLY PLANT WITH NEW CONCEPTS

- From the experiencing of running the Poly plant, I had noted its shortcomings and how they could be overcome.
- I developed better concepts for poly manufacture and gave a new proposal to Chemplast for poly manufacture.
- They constructed a new plant as the the old plant had been sold off as scrap!
- After overcoming teething troubles the plant is in operation for more than a year now.





MAD RUSH FOR POLY

- Till 2006 there were only 6 manufacturers of poly who guarded the technology carefully.
- Because of the tremendous demand for poly, some retired employees or ex-employees of these Companies termed “ROBIN HOODS OF PV” started offering technology for poly silicon manufacture by 2004-5.
- More than 200 new entrants have ventured in to poly manufacturing attracted by the high price.
- The established players have also increased their capacities by getting huge advances from prospective customers.

RECESSION HAS ITS IMPACT

- Because of the present melt-down of economy and deep recession PV is also affected.
- On the one hand availability of poly, wafers and PV modules has eased
- On the other hand, the demand for these has come down.
- This has led to a glut in the market and consequent crash in prices
- Predictions are that only 20% of the new entrants may survive this situation.

SURVIVAL STRATEGY

- Established players have the advantage of mature technology, raw materials sources, customers of long standing and disposal of by-products/wastes
- How a new entrant can compete in the difficult business situation?
- Only when they adopt new and unique technologies with which they can produce the material at a low cost.
- We have a few such unique processes which could help new entrants survive and grow.

LOOKING BACK

- Many institutions started work on silicon in the 70's in the country
- However, only IISc succeeded.

•WHY

- The reason is that at the very early stage, ARV felt that this should be jointly done with an Industry.
- Research Institutions have vast capabilities but inherent limitations.

LOOKING BACK (contd.)

- Industries have their own capabilities, demands, business approaches, financial clouts and reservations on sharing their IPs .
- For commercial success, making a quality product is not enough. It should be at an acceptable cost. Industry knows this and most of the time Research Institutes do not pay attention to this important aspect.
- When an Institute works closely with an Industry, both get attention and therefore the project succeeds.

SUCCEES MANTRA

- Tremendous respect for Intellectual Properties.
- Pressure of business competition making innovations a basic and perpetual necessity.
- This makes Industry work closely with Academics.
- Cross-movement of academics to industry and vice-versa.
- Both know how the other side works and what it can deliver thus developing mutual understanding and respect.

J N TATA'S VISION

- As an industrialist J N Tata knew this very well
- He wanted to create an Institution which would fulfill this need.
- IISc was started with this in mind.
- The success of the Silicon Project is a fitting tribute to the Founder.
- I am proud to have been a part of this.

THANK YOU