Design and Process control of Siemens Poly-silicon CVD Reactor

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Abstract— **The novelty in this paper is to develop a process control for the poly-silicon CVD reactor to achieve optimum productivity of Poly-silicon seed by controlling the process parameters. The production of ingot is done through Siemens process of decomposing Trichlorosilane by Chemical Vapor Deposition on slim tungsten rods. The hardware architecture proposed monitors and controls the systematic sequential stages furnishing dynamics of the plant at a high temperature around 1050°C-1100°C. The HMI communicates through NI's LabVIEW 8.6 package, alarming the user with Process mimic, Report generation, Data and Security management. The plant simulation is realized and verified with LabVIEW 8.6 Version and MATLab 7.5 software tools to obtain the effectiveness of proposed control technique. This GUI based SCADA handles likelihood of fault tolerance, ensuring risk controlled process with optimum productivity of poly-silicon by making system compliant to Industrial standards.**

Keywords—Chemical Vapor Deposition(CVD), Human Machine Interface(HMI), Siemens process, Solar-grade silicon.

I. INTRODUCTION

Awareness of the need to reduce CO_2 emission has only added to the mandate for renewable energy. This public awareness for higher energy prices and global warming problem has opened up the market for solar cell, demanding for energy supply [1]. So have the efforts to make renewable sources as fundamental part of sustained development strategy reinforcing the concern for tight raw material supply of poly-silicon, for two reasons: (1) the scarcity of silicon ingot in the world and (2) India importing SOG-Si (Solar-grade silicon). This brought the surge to conserve energy with a tight raw material supply of poly-silicon ingot, giving an impetus to rapid development of solar cell manufacturing industries to expand large-scale production of Poly-silicon vigorously involving new processing techniques [2-4]. This motivated us to focus towards the development of a process control for Poly-silicon CVD reactor, in this paper, which is mainly aimed to expand silicon reproducible plants in India and to achieve optimized productivity of poly-silicon seed through Siemens process utilizing optimum resources with a vital look on reliable test systems designed for the control of risk process parameters.

The production routes of polycrystalline or solar-grade silicon are of two categories: 1) Chemical route and 2) Metallurgical route. Chemical route is preferred for this proposed siemens model to obtain the purity level extended upto 10N i.e., 99.99999999998%. Siemens process consists of decomposing Tricholorosilane(TCS) by Chemical Vapor Deposition (CVD) on inverse U-shaped hair pin like hot tungsten filaments in a large water-cooled steel bell jar reactor [5],[6]. Architecture of CVD Reactor is illustrated with process stages in Section II. Section III descries the challenges faced during the development phase of poly-silicon crystal. Section IV illustrates PHA studies to overcome challenges posed by CVD Reactor and also describes capability to handle emergency conditions by making the system compliant with required industrial standards. Section V gives the hardware simulation and its results and section VI presents the conclusion and future work.

II. ARCHITECTURE OF POLY-SILICON CVD REACTOR

The proposed hardware architecture for monitoring and control of the sequential processes of poly-silicon CVD reactor is depicted in the Fig. 1.

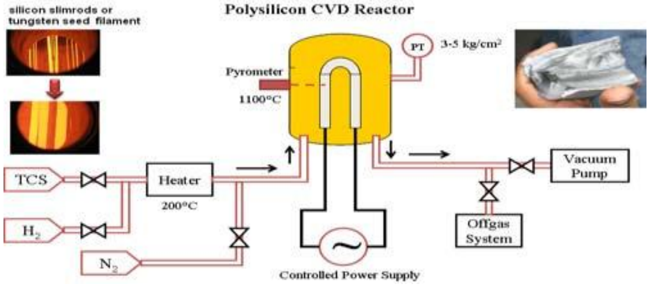


Fig. 1. Block diagram of poly -silicon CVD reactor [7], [8]

In the reactor, a constant rate of hydrogen gas is fed with maintained flow rate of TCS gas from lower to upper portions of the tube giving rise to the following chemical reaction.

$$H_2 + 3HSiCl_3 \xrightarrow{\mathbf{(1050°C\text{-}1100°C)}} 2Si + 5HCl + SiCl_4$$

The hydrogen flow agitates leaving the deposition of poly-silicon around the surface of tungsten increasing the diameter from 3 to 100mm or 150mm. This continuous process of 4 to 5 days is powered with a distribution system, an incomer panel that supplies a 3-phase 11KV mains which is stepped down to voltages of 100-300V at 5000A by an auto-main transformer with On-Load Tap Changer (OLTC). Thyristor controls the load power by varying the firing angle. The optional pyrometers placed around the reactor in three directions sense filament temperatures ranging from 600-1400°C and gives out 4-20mA depending on the amount of glow on the filament. The Cooling water supply to the reactor jacket with flow switches fitted on water manifold reduces temperature of the reactor. Pressure transmitters of -1 to 10 kg/cm^2 are used to sense reactor pressure. Filaments heated upto 1050-1100°c face challenges posed by process parameters like temperature, pressure, vacuum & gas flow rates and electrical parameters like voltage, current, reactive and active powers.

Two silane compounds (TCS & STC) are intermediates in solar-grade silicon production process via chemical route. The Hydrochloric acid formed during the process is sent to a recycling chamber for conversion of TCS gas. Conversion efficiencies and Film morphology depends on operating conditions like source gas mixtures, pressure and reactor temperature. The uniformity in film is built with the increase in pressure with maintained gas flow ratios. Since the homogeneous gas chemical reactions of silane decrease the linearity, the application of feedback control technique is required. After a permissible growth of thickness about 100 or 150mm, the process is completed by slicing down the ingot to Polycrystalline silicon seeds.

III. CHALLENGES OF SEMICONDUCTOR CVD REACTOR

The system is composed by developing the sequential stages of Reactor evacuation and Leak test, TCS and H_2 gas feedrate schedule, Temperature control at around 1050°C-1100°C followed by shutdown process to obtain the sliced poly-silicon ingot. These sequential processes are done to protect against toxic, corrosive, highly volatile gases from escaping out of the reactor which causes irritation of skin and mucous membrane. Reactor Evacuation and Leak test is a start-up test to evacuate air inside the reactor to create a vacuum of 10^{-5} torr, in order to avoid its flammable reaction and also to check reactor's leak free status. Feedrate scheduling is the main process followed by Temperature controlling stage. The thickness of the film deposited depends on the feed ratios of gases to achieve the optimized productivity. As feed rate increases, pressure increases giving rise to uniform distribution of gas. Therefore, gas feedrates are changed at different instants of time according to scheduled ratios to control the reaction rates and also to control the pressure inside the reactor. Thus, this continuous process demands a controlling system for complex thermodynamic reactions to take place at a high temperature with desired productivity. If the temperature decreases below the range, the reaction rate decreases which in turn decreases the rate of deposition of Poly-silicon. If the temperature increases above the range, surface of silicon and tungsten softens losing the stability causing a filament failure. Despite of this, the other challenges faced by the reactor are:

- It is Multi-Input Multi-Output(MIMO) system
- Thermally coupled filaments with radiation heat transfer.
- Continually increased diameter with thermal mass.
- Constantly changed gas mass and their ratios.
- Thermal runaway causes increase in current, increase in temperature & decrease in resistivity.

This developed CVD reactor acquired 10N purity level with a vacuum evacuation reaching 10^{-5}torr followed by supervisory check list ensuring leak free reactor status. A closed loop control system ensured the flow of TCS & H_2 gas ratios as scheduled to achieve the required thermal kinetics at 1050°C-1100°C. A state model is designed to realize the plant dynamics at different temperatures to obtain the transient and frequency response with respect to the plant dimensions. HMI designed handles and alarms the user regarding the likehood of fault tolerance and remedies to give a multi-layer protection to the events of siemens CVD system by making it compliant with required industrial standards. After permissible poly-silicon growth, the process is shut down by reducing the temperature profile with the current profile down to zero with a change in feed rates. Thus, the developed control system ensured handling of emergencies with uniform poly-silicon growth and kinetics by controlling the process parameters.

IV. PROCESS SAFETY & EMERGENCY HANDLING

The process and Instrumentation dynamics are key considerations principled with multi-layered protection for system analysis [9],[10]. Each layer of protection consists of grouping an equipment and/or human activity which address an emergency situation to be informed to the community with an engineering approach to control hazard. This decision required for the measurement of Safety Instrumented System (SIS) performance is made acceptable with industrial standards to reach the assignment of target SIL (Safety Integrity Level) with the extension of Process Hazard Analysis (PHA) to mitigate risk associated with each level of CVD Reactor [11]. To establish a safe circuital operation of poly-silicon CVD reactor, ANSI/ISA S5.1-1984(R1992) standard [12],[13] is used for design phase of the thermal plant for a quick and reliable information about process analysis and control of equipment requisites. The electrical design developed follows SEMI S22-0706a standard for safe operation [14]. IEC d61508 is taken as an umbrella standard for Industrial Electrical/Electronic & Programmable Electronic Safety Related System (E/E/PE SRS) [15].

The objective of risk control in terms of likelihood and the severity of hazards were achieved qualitatively and quantitatively with PHA studies with assignment of Target SILs for SIF (Safety Instrumented Functions). Qualitative techniques namely HAZOP (Hazard and Operability) and FMECA (Failure mode, effects and criticality) analysis were adopted to identify failed events and made it capable against the hazardous consequence with remedy to safeguard the system [9]. The checklist verification tells the status of the equipments in order to avoid the hazards before the process starts. Power control & Distribution panel failure (e.g., over current, under current, earth fault, ground fault and voltage fault protections), Air cooling failures, Over pressure and Instrumentation or Equipment failures like Heat exchanger, Valve, Rectifier, Pump failure (e.g., vacuum & water), Auto-focusing failure of Pyrometer and load failures are some failures which occurs posing challenge to the system. Certain factors like Power failure, Power blink, Instrument air/valve, Cooling water & Load failures, Tube ruptures, Manual valve failure, Thermal run -away reactions, Breakage of Quartz window, Malfunction of Vacuum circuit breaker, Reactor leak and Communication failure causes emergency shutdown of the system [16], [17]. Human and In-process thorough monitoring around reactor vicinity reviews and ensures safety against fault occurrence through camera scanning. Quantitative risk analysis using Event tree technique helped in easy identification of hazards and risk associated in the process by suggesting suitable remedies for elimination.

The HMI is designed in NI's LabVIEW 8.6 package [18] for a user-friendly information exchange between man and machine. This HMI alarms for Emergency Off (EMO) operations with a programmed salience guiding the user to take appropriate action is complaint with SEMI E95-1101 Standard.

V. SIMULATION RESULTS OF CVD REACTION

The hardware simulation is developed using National Instrument LabVIEW8.6 Version (Laboratory Virtual Instrumentation Engineering Workbench) for visualizing the sequential plant process. The reactor evacuation and its status were realized with its ideal and practical characteristics as depicted in Fig. 2. Time required for evacuating air inside the reactor from an initial pressure P_i to final pressure P_f is estimated as [19].

$$t = \left(\frac{v}{s}\right)\ln\left(\frac{p_a}{p_f}\right) \tag{1}$$

Where t = Evacuation time [hr], S = Average pump suction capacity [m^3/hr], P_a = Atmospheric air inside the reactor at t_o [torr], P_f = Final pressure at t_l [torr], d = Diameter of the reactor, h = Height, V = System volume [m3]. The values in Table I are practical and real time values obtained during the test. This process is reviewed for 2 or 3 times, till the practical characteristics are identical to ideal characteristics reaching the minor leak check point reading 10^{-5}torr. If iterations prove that reactor is not leak free, jacket is substituted by another steel shield. Evacuation status and its progress with Process mimic, Observation table and Graph is shown in figures (Fig.3, Fig.4 & Fig.5). After start-up stage is verified with leak test which is carried out with Nitrogen and Hydrogen flushing, the process starts with Feedrate scheduling. Both TCS and H_2 gases are controlled by flow transmitters according to their feed schedule to maintain the pressure at 4-5 bars in the reactor. The outlet pressure valve is regulated accordingly to obtain characteristics as shown in figures (Fig.6 & Fig.7).

TABLE I VALUE OF CHECK POINTS

Control value opened	Ideal Characteristics
Gross leak check point, V_0	335.65mtorr
Gross leak check time, t_0	60sec
Minor leak check point, V_1	0.01mtorr(setpoint)
Leak check start time, t_1	756sec
Leak rate in vacuum, dP/dt (Control valve closed)	0.001mtorr/sec
Minor leak observation time t_2	60sec

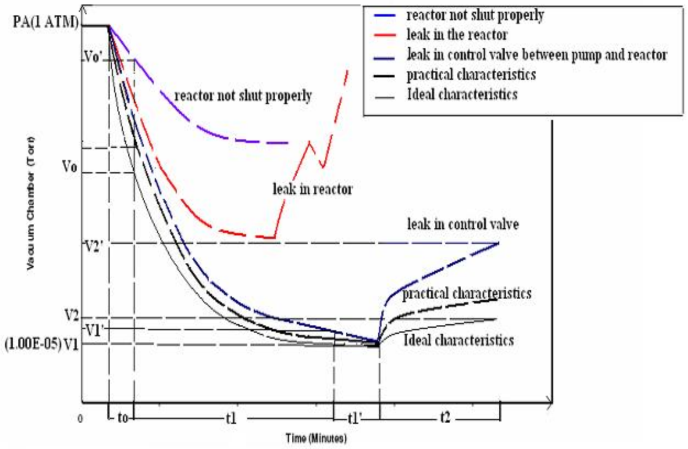


Fig. 2. Illustrates the characteristics of the Evacuation curve under different circumstances.

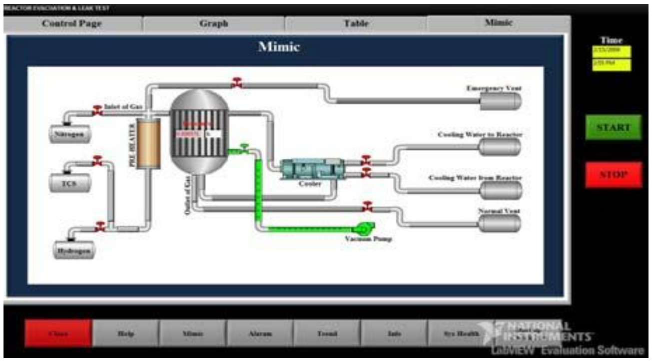


Fig. 3. Mimic page illustrating Evacuation test of reactor

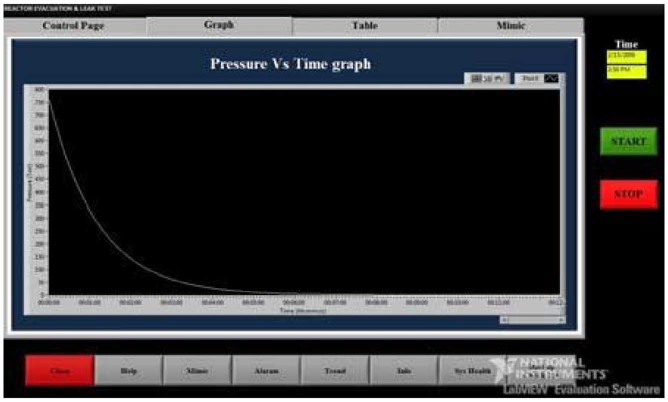
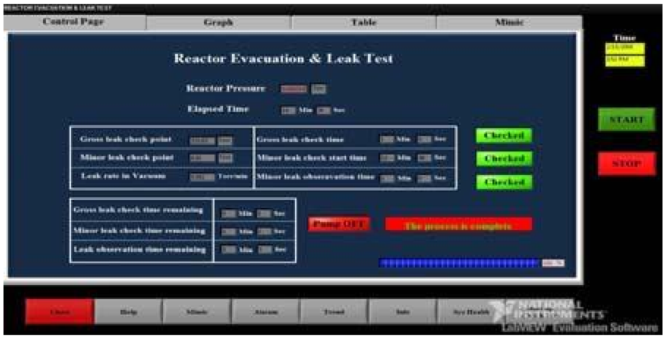


Fig. 4. Graph depicting Reactor Evacuation characteristics



Reactor Evacuation & Leak Test

Reactor Pressure

Elapsed Time Min Sec

Gross leak check point | Gross leak check time Min Sec | Checked

Minor leak check point | Minor leak check start time Min Sec | Checked

Leak rate in Vacuum Torr/min | Minor leak observation time Min Sec | Checked

Gross leak check time remaining Min Sec

Minor leak check time remaining Min Sec

Leak observation time remaining Min Sec

Pump OFF

The process is complete

Fig. 5. Screen shot illustrating completion status of the process

Fig. 6. Screen shot of mimic page for Feedrate scheduling

Fig. 7. Characteristics of TCS and Hydrogen feedrate as scheduled

The reactor vessel with slim tungsten rods are heated up through 230V AC single phase power supply and the current required for heating is controlled with the help of Thyristor control panel. The model of cylinder is shown in Fig. 8. The cylindrical vessel of length (l) and diameter (d) is considered with inner radius of heater r_1, outer radius of cylinder r_2 and with an outer cover of radius r_3. It depends on the chemical composition of the cylinder like Tungsten, Nitrogen gas, Stainless steel and Glass boundaries. Heat flow in a radial direction is realized with its circuit model as shown in Fig.9. The system comprises an electrical heater of heat capacity C_h connected via a thermal resistance R_h to the furnace and to the surroundings with a thermal resistance of R_O and with a heat capacity C_r. The filament kept at temperature T_h transfers heat to surrounding air inside the furnace at temperature T_i, through the thermal resistance R_h. The heat flow in radial direction is exposed to two different temperatures T_i at 900°C and T_o at 1200°C making the model a linear time variant system. The increase in temperature will increase the pressure inside the closed radial reactor. Dynamics of the vessel at that particular time is modeled using state space realization following the conduction & convection properties [23-26] and thermodynamic principles using following equations.

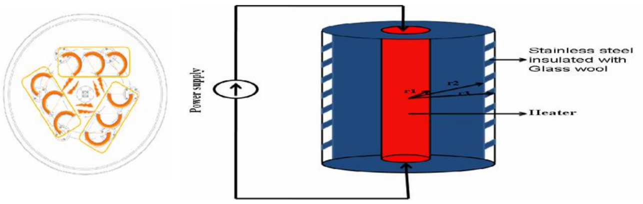


Fig. 8. Structure of the reactor with filaments

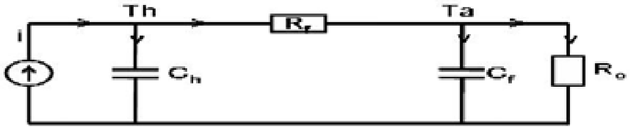


Fig. 9 Thermal model of the proposed reactor

Using Fourier's law [20-21], Heat flux of the radial system is given by $Qr = -kAr\ (dT\ /\ dr)$ (2)

where $A = 2\prod r^2 l$, Area for heat flow in radial system. Heat conduction of the radial system at different temperatures is given by

$$Q = 2\pi kl\left[(T_h - T_a)/\ln(r_1/r_o)\right] \qquad (3)$$

The thermal resistance is

$$R_{th} = \ln\left(\frac{r_1}{r_o}\right)/(2\pi kl) \qquad (4)$$

Heat capacity is given by $(\Delta Q) = m * Cp$ (5)

Where m is mass and C_p is specific heat capacity at constant pressure. Using state model representation [22], we get

$$C_a\frac{dT_a}{dt} = \frac{1}{R_f}T_h - \left(\frac{1}{R_o} + \frac{1}{R_f}\right)T_a \qquad (6)$$

$$T_a = \frac{1}{C_hR_f}T_h - \frac{1}{C_h}\left(\frac{1}{R_o} + \frac{1}{R_f}\right)T_a + (0)i \qquad (7)$$

Equations (6) & (7) can be represented as

$$\begin{pmatrix} T_h \\ T_a \end{pmatrix} = \begin{pmatrix} \frac{-1}{C_hR_f} & \frac{1}{C_hR_f} \\ \frac{1}{C_fR_f} & -\frac{1}{C_h}\left(\frac{1}{R_o} + \frac{1}{R_f}\right) \end{pmatrix}\begin{pmatrix} T_h \\ T_a \end{pmatrix} + \begin{pmatrix} \frac{1}{C_h} \\ 0 \end{pmatrix}i$$

$$T_a = \begin{pmatrix} 0 & 1 \end{pmatrix}\begin{pmatrix} T_h \\ T_a \end{pmatrix} + (0)i$$

A PID controller in feedback, controls the steady state and transient response as per the requirement. This controller provides the most accurate and stable control, and is best used in systems which react quickly to changes in the energy added to the process. Hence, setting the controller parameters is achieved by tuning it using cohen-coons method. Tuned parameters using the cohen-coons method are $T = 250$, $t_d = 15$sec,K=1. After tuning,

$$K_c = \frac{1}{k}\frac{\tau}{t_d}\left(\frac{4}{3} + \frac{t_d}{4\tau}\right) = 60$$

$$\tau_1 = t_d \frac{32 + 6\frac{t_d}{\tau}}{13 + 8\frac{t_d}{\tau}} = 0.1 Sec$$

$$\tau_D = t_d \frac{4}{11 + 2\frac{t_d}{\tau}} = 1200$$

The process parameters are controlled and plant dynamics with its steady-state and frequency response are as shown in the screen shots of Fig.10, Fig.11 & Fig.12. Practical and real time values obtained during the test is tabulated in Table II.

By adopting the state model, system is made completely observable, controllable and stable. This SCADA system (hardware and software) globally monitors and ensures user friendly process control interface with Real-time data trending along with Storage and easy retrieval of data, Report generation and delivers progress visualization of each level in the process. The diameter measurement is monitored and captured by a camera for a permissible growth of poly-silicon of about 100 or 150mm thickness as illustrated in the Fig. 13.

TABLE II Value of Parameters At 900°C & 1200°C

At 900°C	Tungsten	Glass wool	Hydrogen	Stainless steel
Radius	0.0015m	0.27m	0.15m	0.2m
Density	19300 kg/m3	-	0.06 Kg/m^3	-
Specific heat capacity	150 J/(Kg-K)	-	15150 (J/(Kg-k))	-
Heat capacity	20.453 (J/K)	-	64.2208 (J/K)	-
Thermal conductivity	-	0.04 (W/m*K)	0.18 (w/m-K)	23 ((W/m)*K)
Thermal resistance	-	1.8029*10^{-4} K/w	4.0739 K/W	1.8029*10^{-4} K/W (with glass wool)
At 1200°C	Tungsten	Glass wool	Hydrogen	Stainless steel
Specific heat capacity	156 J/(Kg-K)	-	15770 (J/(Kg-k))	-
Heat capacity	21.2713 (J/K)	-	66.84903 (J/K)	-
Thermal conductivity	-	0.04 ((W/m)*K)	0.18 (w/m-K)	24 ((W-m)*K)
Thermal resistance	-	1.8029*10^{-4} K/W	4.0739 K/W	-

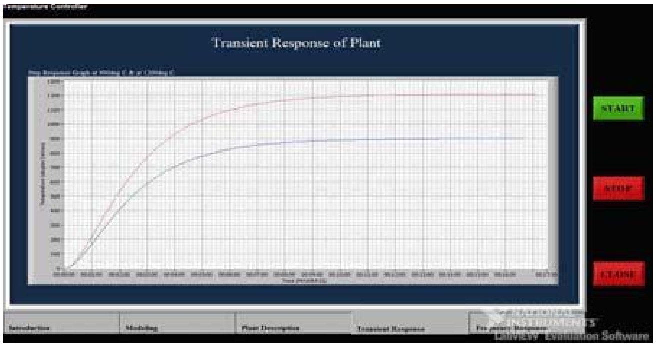


Fig. 10. Screen shot of transient response for temperature model

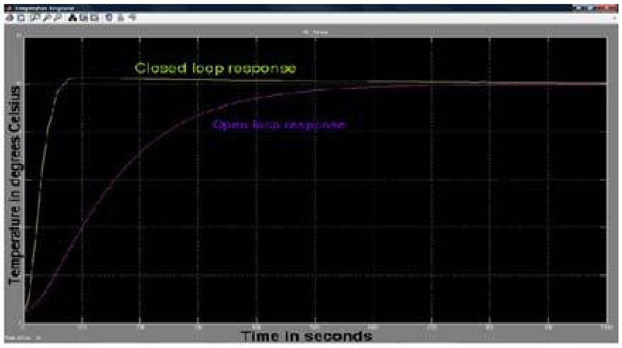


Fig. 11. Simulation of open & closed loop response of plant using MATLAB

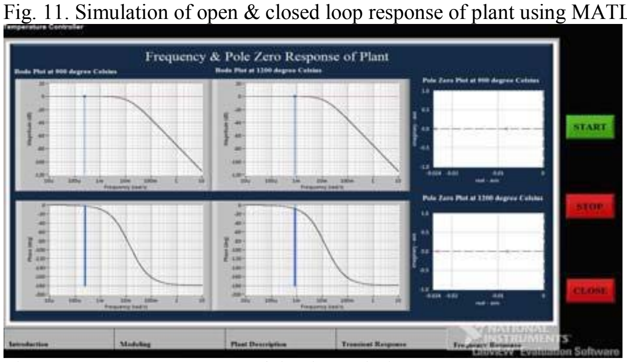


Fig. 12. Frequency response of the thermal plant

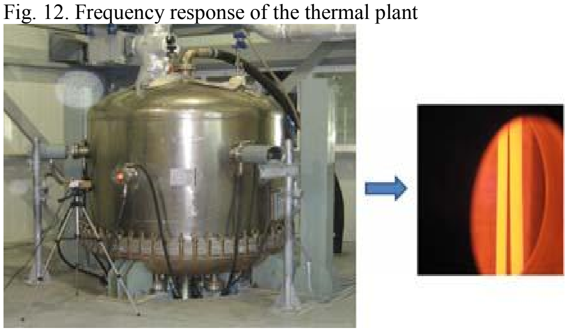


Fig. 13. Vision based Silicon Diameter Measurement

VI. CONCLUSION & FUTURE WORK

The proposed novelty to optimize the control of PVT parameters in a poly-silicon CVD reactor is designed, modeled and developed to achieve productivity of Poly-silicon seed at a high temperature of 1050°C-1100°C**.** with It was observed that the process risk parameters of non-linear time variant CVD system are completely observable & controllable. Also, it si observed that the system is highly stable at a temperature of 900°C & 1200°C which is the worst cases for the seed growth. Specific heat capacity varies slightly from 150-156 J(Kg-K) across 900-1200°C. developing the PVT control model at such a high temperature is very difficult. Using NI LabVIEW, eased the construction of complex thermal structure and also saved time & speed in acquiring response of complex reactor modules immensely. The result is a hazard & risk resistant and reproducible system capable of producing poly-silicon ingot upto 10N purity level via chemical route. The GUI based HMI is reliable to the user with Security & Data management capacity and Emergency handling capability of a process by making the system compliant with the required industrial standards ensuring safety. HMI can also be facilitated with development of wireless remote and touch screen capabilities.

VII. ACKNOWLEDGMENT

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