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The Application of Level and Density Measurement in Polysilicon Manufacturing

Key Words

- Polysilicon
- Photovoltaic (PV)
- Solar energy
- Level
- Density
- Hydrochlorination (HCL) reactor
- Metallurgical silicon
- Silicon tetrachloride (STC) hydrogenation
- Quench column
- Vaporizer column
- Residue stripper column
- Trichlorosilane (TCS)
- Metallurgical silicon (MGS)
- Chemical vapor disposition (CVD)
- Fluid bed reactor (FBR)
- Dichlorosilane (DCS)

Introduction

Solar panels are commonly used around the world to harness the abundant power of sunlight and provide a renewable energy source. A solar panel is comprised of photovoltaic cells which convert the sunlight into electricity. Solar-grade polycrystalline silicon, more commonly known as polysilicon, is a vital raw material used by the photovoltaic (PV) industry to manufacture PV cells and wafers.

Polysilicon production begins by refining quartz or sand into metallurgical grade silicon. Next, the silicon is purified through a series of chemical reactions. To convert silica stone into polysilicon, metallurgical grade silicon (MGS) with a purity of 99% must be manufactured. The MGS is further purified by refining distillation to produce trichlorosilane (TCS). Reduction is performed with hydrogen at temperatures near 1,000°C, resulting in 99.99% pure polysilicon that is deposited in the form of a rod. During the polysilicon production process, measurement of the level and density of the raw materials enables tighter process control and results in less upset conditions, increased production time and better cost control.

Level Application: Hydrochlorination Reactor

The process of refining the MGS takes place in the hydrochlorination reactor where reactions of superheated MGS and hydrogen occur at temperatures in the range of 500°C to 550°C and at pressures in the range of 400 to 500 PSI (i.e., 2800 to 3500 kPa or 28 to 35 bar). The net result of hydrochlorination is a condensed liquid mixture that contains TCS, dichlorosilane (DCS), untreated silicon tetrachloride (STC) and dissolved gases, notably hydrogen gas and hydrogen chloride. Plants monitor the level of the liquid TCS/DCS mixture with the Thermo Scientific LevelPRO gamma level sensor. It is mounted on the external wall of the hydrochlorination reactor to non-intrusively provide precise level control of this mixture, allowing for proper blending of the TCS as it enters the fluidized bed reactor (FBR).

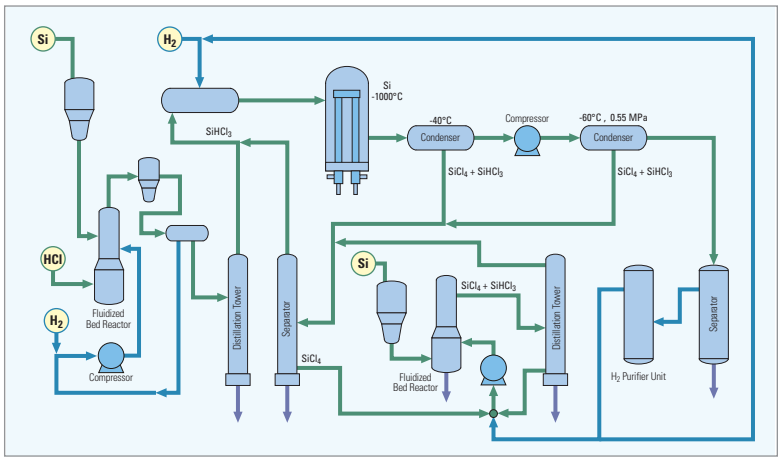

figure 1 – One example of a polysilicon production process.

The liquid TCS is vaporized, mixed with hydrogen gas and reduced in either a chemical vapor deposition (CVD) furnace, resulting in polysilica (PS) rods, or a fluid bed reactor (FBR) that produces PS granules. Either the LevelPRO gamma level sensor or the Thermo Scientific PNF point level gauge is ideal for controlling the level in the FBR. These instruments allow for precise control during manufacturing and blending of the STC and TCS products prior to distillation as well as allow for the control of catalyst fines.

After the dissolved gases have been stripped out, the STC is removed from the TCS/DCS mixture by distillation. During the distillation process, several metal chlorides are removed because they are known to plate out on process equipment. The LevelPRO gamma level sensor and the PNF point level switch can be used on the columns to monitor the gravity flow of the distillation process and ascertain a number of key levels. This online monitoring allows for even flow and subsequent process efficiency. The coordinated control of this flow helps to increase production as well as ensures less upset conditions which can increase operational costs.

Level control is ideal in this application because:

- Measurement of fluidized solids content (hydrogen and

figure 2 – Thermo Scientific LevelPRO (left) and DensityPRO (right) sensor systems

chlorosilanes) enables optimal process control
- Process control is also optimized with high/high and low/low alarms which can be achieved via the two SPDT relays on the LevelPRO sensor.

Level Control: Quench Column

Precise level control helps manufacturers optimize the process flow through the quench column. This optimization enables production of a pure product and reduces the amount of recycled additives that are lost. Typically, 18 feet of installed level control on the column enables better monitoring and regulation of the input and outflow.

There are several reasons level control is ideal for the quench column, including:
- It enables control of process inputs and outputs
- It eliminates process upsets
- It ensures purity of the product
- It enables recovery of recycled additives.

Density Application: Quench Column

The quencher sludge is a mixture of unreacted MGS fines, metal chlorides and some STC with minor amounts of TCS. The sludge is richer in phosphorus and arsenic but leaner in boron than the crude TCS. The Thermo Scientific DensityPRO gamma density gauge provides process control of the quencher sludge density, allowing better evaluation of the mixture of chlorides and fines. This blending control via density measurement allows for upstream adjustments to increase production with less downtime.

Density measurement is important in this process because the liquid evaporates and the vapor phase becomes dense. The difference in density between the upper and bottom phase at extreme conditions is much smaller and requires level monitoring in addition to density measurement.

Density Application: Vaporizer Column

Liquid TCS is vaporized and mixed with hydrogen gas before it is sent to the CVD or FBR for production of PS granules. In this manufacturing process, the liquid evaporates and the vapor phase becomes dense in the vaporizer column. The DensityPRO gauge allows for the monitoring of the TCS and hydrogen mixture which is critical for downstream processing to the CVD or FBR. If the mixture is out of balance, these processes will be affected and the entire closed loop system will be out of balance which may cause delays in process production or even process shutdowns.

The key points to consider for density measurement using the DensityPRO gauge in this application are:
- There is a smaller difference in density between the upper and the bottom phase at extreme conditions
- The DensityPRO gauge mounted at vessel high level will provide vapor density-compensated level measurement
- High level is better defined with a lower source size
- Precise density measurement will keep the closed loop system in balance.

Conclusion

Advanced process technologies, including Thermo Scientific level and density gauges, are enabling the polysilicon industry to tightly monitor the production process. Reliable level control of the hydrochlorination reactor ensures the TCS is properly blended before it enters the FBR. Level control is also critical during manufacturing and blending of STC and TCS products prior to distillation and allows for the control of catalyst fines. During the distillation process, level control also helps to ensure the flow remains even to increase production with fewer upset conditions.

In the quench column, close monitoring of the quencher sludge density ensures proper blending control is achieved. The density of the TCS and hydrogen mixture in the vaporizer column must also be monitored to ensure the mixture remains in balance to keep the entire closed loop system in balance. By providing highly reliable measurement, Thermo Scientific level and density gauges are helping polysilicon plants optimize production, lower operating costs and increase plant safety.

In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

China
+86 (10) 8419-3588
+86 (10) 8419-3580 fax

India
+91 (20) 6626 7000
+91 (20) 6626 7001 fax

United Kingdom
+44 (0) 1606 548700
+44 (0) 1606 548711 fax

USA
+1 (713) 272-0404
+1 (713) 272-4573 fax

www.thermoscientific.com