On-Line Particle Size Analysis by Video Capture

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ABSTRACT

Particle size analysis in fluid bed processes by sieving requires taking samples, slow and labor intensive sieve analysis and conversion of the raw data to meaningful information. For high purity systems with hig value product, the samples are not usable and represent a significant cost. Thus, it is common to sieve a small amount from a relatively large batch primarily to ensure the batch passes specifications which in turn are set as broad as possible to avoid costly rejection.

Because of the increase in computer speed and video capture technology, a relatively new and inexpensive method of sampling for particle size has been examined. This method of on-line particle size analysis by video capture and computer image analysis alleviates the tedious process of sieving. It also supersedes bench testin methods of particle size analysis and provides for continuous monitoring without costly samples. The overall optimization of the system will be discussed with an emphasis on flexibility and the recognition of the rapid pace of advance in CPU speed and video camera technology. If the long term operational experience supports initia testing, it is expected that this technique will become routine for most fluid bed reactors because of the benefits of knowing the particle size distribution, average particle size and sphericity on a continuous basis in control of a fluid bed reactor.

INTRODUCTION

In the granular polysilicon market, the product is inherently of high value ($50/kg) and the customers value a narrow size distribution and consistent average particle size. The small beads are more contaminated and irregularly shaped. The large beads, while more spherical, plug the feeding equipment. It is easier and cheaper t design downstream systems for a standardized product. Unfortunately, production of granular material in fluid beds produce a wide particle distribution as fluid beds are traditionally well mixed. It is possible to introduce various segregation techniques to reduce the particle size distribution, but these introduce some instability to the reactor thus making rapid measurement and control of particle size essential.

ASiMI and SML Associates recognized this problem early in the development of ASiMI's new fluid bed process for granular polycrystalline silicon. Being in the semiconductor business, they recognized that this problem could possibly be solved by use of machine vision systems similar to those used for defect detection and automatic product grading. This brought the choice of a general purpose machine vision system with customized software versus a dedicated system with predeveloped software. Dedicated systems were preferred although more expensive because of a philosophy to buy rather than develop support technology. Two dedicated systems

were identified and passed initial lab testing by the manufacturer, but only one (Canty Vision) had an establishe record with installed systems.

A system was obtained from Canty Vision for further development and was found to be very dependent on obtaining high contrast at the bead edges (grey scale) in order to accurately select the beads, thus lighting of the window proved crucial. Several locations for the window were considered and two were tested. Key process issues were coating of the window, time for video capture and analysis, CPU speed, bead flow rate, fraction of beads measured, time lag from reactor outlet to measuring location and temperature at location. The key parameter for vision system performance was discovered to be pixels per frame as this led to a tradeoff of pixel per bead versus number of beads per frame. More pixels per bead improved accuracy of bead characterization but meant a smaller sample size which decreased the overall accuracy. Safety issues were important because of the potential presence of flammable gases and the possibility of the light source being an ignition source.

The standard software was modified to incorporate the calculation of sphericity thus enabling the online calculation of the minimum fluidization velocity. Future development may include online calculation of the void fraction. The system went into service in July '97 and comparison of its online performance will be compared t the bench testing and to backup sieve analysis of selected samples is currently being evaluated.

SYSTEM REQUIREMENTS

The equipment requirements for the Canty vision system include a personal computer with at least a 486 CPU, a Canty vision camera, a light source, a light housing for the camera, and a digital readout or display screen, and a computer monitor.
The capacity of the CPU and the camera must correspond in order for the system to function at its best. Two factors contribute to the sampling time: the number of frames needed for a stable particle size average and the processing time of the data. Every process will have a different number of frames to achieve a stable average particle size. This specifically depends on the number of beads within a frame and the time it takes for the syste to grab a frame. The system can be configured to take a frame according to the flow rate of the beads flowing into the sampling area. The CPU will then have to process each frame. If the camera is more advanced and ca capture more beads per frame, the same CPU will take longer to process that frame. As a rule of thumb, the more advanced the camera is, the faster the CPU must be. The following hand calculations of sampling time can make things easier when choosing the speed of the CPU using the camera specifications.

Historical sample size = (# frames needed for stable average) * (beads/frame)
Pixels per bead = (pixels/frame) / (beads/frame)
Total pixels in sample size = (historical sample size) * (Pixels per bead)
CPU processing time = (Pixels in sample) * (Pixels/time capability of CPU)
Camera processing time = (# frames needed for ave.) * (time for one frame)
Total sampling time = (CPU processing time) + (Camera processing time)+lag time

A 486 CPU can roughly process one frame/sec which is 540X480 (260000) pixels/sec. Using the ICOMP index, a P2 CPU will process Z times faster than a 486 CPU. Using a faster CPU will subsequently reduce the total sampling time. Note as the quality of the camera increases, the CPU speed must increase in order to compensate for the time in sampling. Also, using a camera with more beads per frame won't have the video quality of a camera with more pixels per bead. Lag time should not be forgotten as it may take several minutes from release of beads from the reactor to the camera.

The Canty system was also connected to a PLC which allows for average particle size to be transmitted to the PLC while historical data is kept within the Canty system. The light source that is used in this system is a fiber optic light pipe. It resembles a small flash light on the end of a fiber optic cord. The light is actually a low voltage bulb incased in a box. An elliptical reflector focuses the light onto the light pipe which is then bounced down the fiber optic cord. This particular type of light source works well in ASiMI's application due to the

safety hazard of a spark source and hydrogen presence. Depending upon the area of classification of the process, a cheaper light source may work just as well.

During setup of the Canty system, it was noticed that in order to get optimal use of the light, a diffusion source was needed to disperse the light evenly around the camera head. Diffused light is much better than direct light to minimize shadows and bright spots in the camera's view. In first tests and setup, thin translucent plastic was conically wrapped around the camera with the light source on the outside of the plastic. This provided even light distribution for the camera. Later during setup of the pilot plant, a design using UHMW, a white translucent plastic, was incorporated allowing the light to disperse through a block of plastic allowing for a soft light hue to penetrate around the camera. This can be seen in the diagram of Figure 1.

SOFTWARE

Software was provided with the Canty system which included a calculation for the average particle size. The particle sphericity and the particle sphericity distribution calculation was of interest and an algorithm for this was provided to Canty from ASiMI which was then programmed into the software. This algorithm was created using measurements taken onsite by the Canty system.

Let $A_{p,m}$ = measured particle area
$D_{p,c}$ = calculated particle diameter from measured particle area
$D_{p,max}$ = maximum measured particle diameter
$D_{p,min}$ = minimum measured particle diameter
And ϕ_p = particle sphericity = measured area/maximum area, upper limit = 1.0

Since $A_{p,m} = (\pi/4)* D_{p,c}^2$,
Then $D_{p,c}^2 = (4/\pi)* A_{p,m}$
And $\phi_p = D_{p,c}^2 / D_{p,max}^2$

$$\phi_p = (4/\pi)* A_{p,m} / D_{p,max}^2$$

CALIBRATION

Calibration is one of the most important steps in setting up an online particle size analyzer. As with any piece of equipment, this step can produce faulty results if not done correctly. There are two types of calibration for online particle size analysis: static and dynamic. Static calibration would include a stationary container in which the bead size was known and the container was mixed before each frame was captured with the camera. Dynamic calibration is fixing the camera in an actual moving process and having the camera capture frames of moving beads. This type of calibration can be done bench top or in situ. Dynamic calibration in inherently better especially if calibrated in situ with the same conditions and equipment as in the actual process.

In ASiMI's case, static calibration was used initially followed by in situ dynamic calibration. This was beneficial because the static calibration served as hands on training and initial trouble shooting tool while the dynamic gave trustworthy calibration results.

The following is a sketch of the Canty Vision Camera Setup for the calibration test.

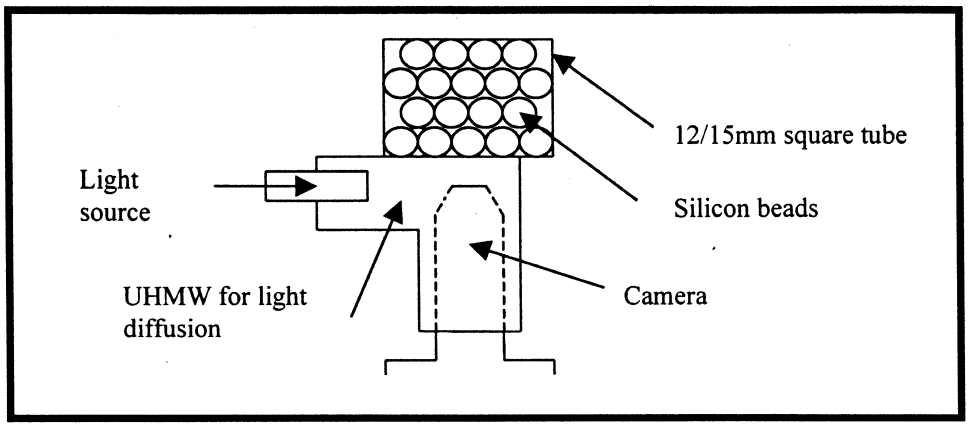

Figure 1. Canty Vision Camera Setup

The static calibration results gave ASiMI enough information to set up the Canty Vision System at the pilot plant location. The calibration was done bench top where the system was completely assembled and dimensions for the location of the camera, light source, reflection piece, and bead sample tube were recorded. Required setpoints and system operating parameters in obtaining good bead images were also recorded. The PLC was tie into the Canty system allowing data for average particle size and sphericity to be collected.

Bead calibration included small particle size pre-sieved beads 0.5 mm to 2 mm. The calibration results in these small sizes gave reliable data and correlation factors were developed for future test runs. The following table is the calibration results from the static calibration test.

Table 1. Static Calibration Test Results

SCREEN	BEAD SIZE	EXPECTED SIZE	CALIBRATED AVG SIZE	RECOMMENDED SCALE FACTORS
(#)	(mm)	(mm)	(mm)	(mm/pix)
10	2.000	2.180	2.040	0.0131261
12	1.700	1.850	1.790	0.0104780
14	1.400	1.500	1.620	0.0104780
16	1.180	1.290	1.190	0.0106810
18	1.000	1.090	1.070	0.0102010
20	0.850	0.925	0.935	0.0099110
25	0.710	0.780	0.786	0.0101638
30	0.600	0.655	0.629	0.0087480
35	0.500	0.550	0.554	0.0081970

Dynamic calibration results refined the calibration scale factors. It was realized that there is a maximum bead flow rate for this process for the camera to take accurate frames. In this particular process, the maximum bead flow rate is 10 kg beads/hr.

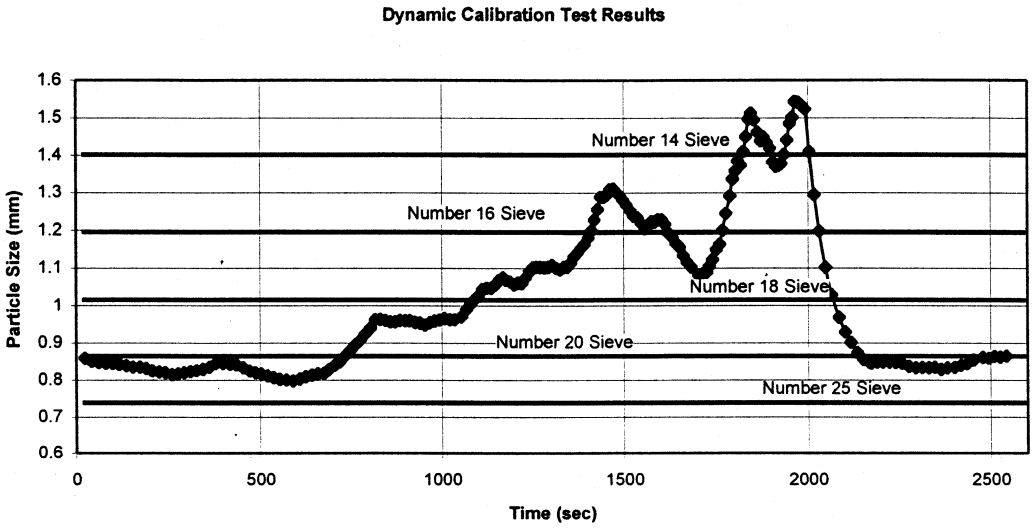

Figure 2. Dynamic Calibration Test Results

The initial dynamic calibration test consisted of adding 5 batches of presieved beads each of 200 gram t the reactor while a small amount of nitrogen was purged in to prevent plugging of the grid. The beads wer added in the order 25 mesh, 20 mesh, 18 mesh, 16 mesh and 14 mesh. Some beads left immediately down th bead removal tube. The mass in the bead removal tube was calculated at 230 grams. It can be seen that the firs 200 grams of 25 mesh showed up clearly with the transition to 20 mesh at about 700 seconds. A further transition occurs at 1000 se onds which probably indicates the finish of the direct fill of the bead withdrawal tube at 230 grams. The graph then shows a series of hills and valleys which probably indicate the variability of mixing in the reactor, while the tail of the graph shows a rapid drop off indicating segregation of small particles on top.

CONCLUSION

In conclusion, on-line particle size analysis is by far the most efficient and inexpensive method of particle size analysis. The tedious alternatives including sieving and bench top analysis do not offer the accuracy of bulk bead measurement within a feasible amount of time. With on-line particle analysis, the fluid bed process can be closely monitored and the system can quickly make adjustments without having to wait for lengthy sieving or bench top analysis.

Optimization of the on-line particle analysis includes choosing the appropriate equipment for the specific process it will be used for. Using a correct camera (beads/frame) companioned with an adequate CPU, minimizes analysis time. These can be chosen with the use of some simple formulas given for Total Sampling Time. Various software can also be chosen for more efficient calculation of needed information. Also, different algorithms can be added to existing software packages. A sphericity calculation was added to this specific vision system and may be important for most granular fluid bed processes. Lighting conditions can be optimized b using a diffusive casing around the light. Calibration was found to be most beneficial when static and dynamic calibration tests are both carried out.

REFERENCES

J.M. Canty web page: www.jmcanty.com, August 1997