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Crystal Nucleation Control Using Microscopic Online Imaging

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Agenda

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- History of Imaging Crystallization Processes
- Pros & Cons of Different Technologies
- New Technology
  - Equipment
  - On-Line Real Time Measurement
  - Output Data/Process Control
  - Visual Verification
- Successful Applications
- Summary/Conclusion
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Introduction

An innovative approach of inline microscopic imaging has been developed to allow crystal viewing and measurement of size, shape, color, and concentration in a variety of settings (batch or continuous lab reactors, pilot plant crystallizers, and full production vessels).

Monitor from nucleation to full growth in real time, high resolution viewing at a pressure of up to 10,000 psi and temperature range of -450° to 800° F with the ability to see from .7 micron with no upper limit. This is accomplished using rugged fused glass and high output fiber optic lighting (cold light).

Advanced software captures and analyzes particles and compiles the data to determine distribution by length, width or volume along with the PPM concentration. Data can be outputted in a variety of formats and can be presented in tabular and graphical formats.

Monitoring the crystal process through this vision based system allows for the precise control of the nucleation process, which improves quality, yield and subsequent profitability.

History of Crystallization Imaging

To date imaging crystallization processes has been limited to post-process analysis. Particle size distribution can be constructed after a process has ran; if it is a bad product, this has wasted time and resources. In-process sampling typically disrupts production conditions; for example changing temperature and pressure can lead to agglomeration of crystals.

Methods of analysis in the past include:

- Turbidity
- Inverted microscopes
- PVM (particle vision measurement)
- FBRM (focused beam reflectance measurement)

Turbidity

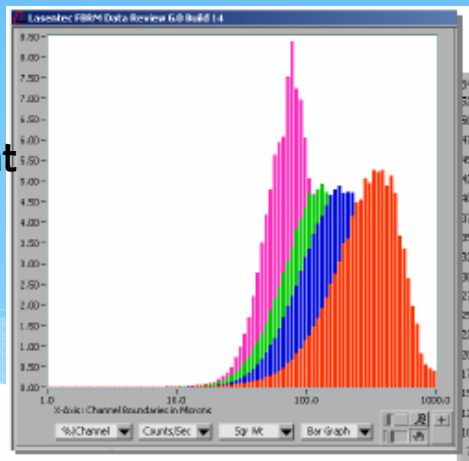
- A basic measurement of abundance or degree of crystallization present.
- Does not provide any indication as to the size or shape of crystals.
- Lends no information to the nucleation process– information for seed is unknown.

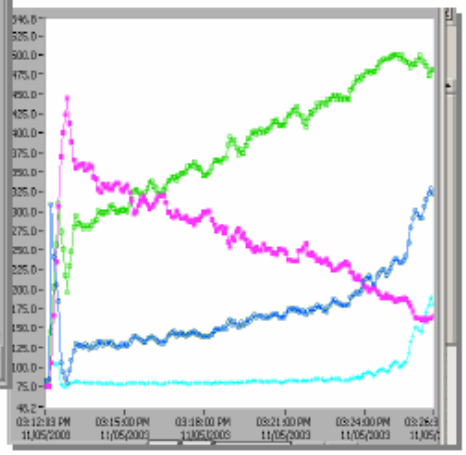
Inverted Microscopes

- Offer better imaging, ability to size down to the several micron scale.
- Do not utilize the most efficient lighting.
- Big, bulky, & costly.

PVM and FBRM

- Particle Vision Measurement provides real time, in process analysis by using a probe emitting six lasers in a vessel to create the live image.
- Focused Beam Reflectance Measurement involves a probe that emits a laser that measures a series of chord lengths and compiles into a distribution.
- Only see to 2 microns due to insufficient front-lighting methods, limited temperature range of operability.





Pros & Cons of Different Technologies

Technology	Pros	Cons
Turbidity	• Assess concentration of crystals present based on reflectivity.	• Size & shape of seed unknown. • Precise start of nucleation unknown.
PVM & FBRM	• In-process, real time analysis.	• Ineffective front-lighting. • Only sees down to 2 microns—critical for nucleation! • Multiple probes used to compile data. • Limited temperature range.
Inverted Microscope	• Ability to size particles down to several microns.	• Ineffective lighting. • Bulky & expensive.
Microscopic Online Imaging	• In-process, real-time analysis. • Ability to size down to .7 micron with no upper limit. • Uses high output, fiber optic illumination & rugged fused glass. • Software allows for customizable output options. • Temperature range of -450°-800° F	

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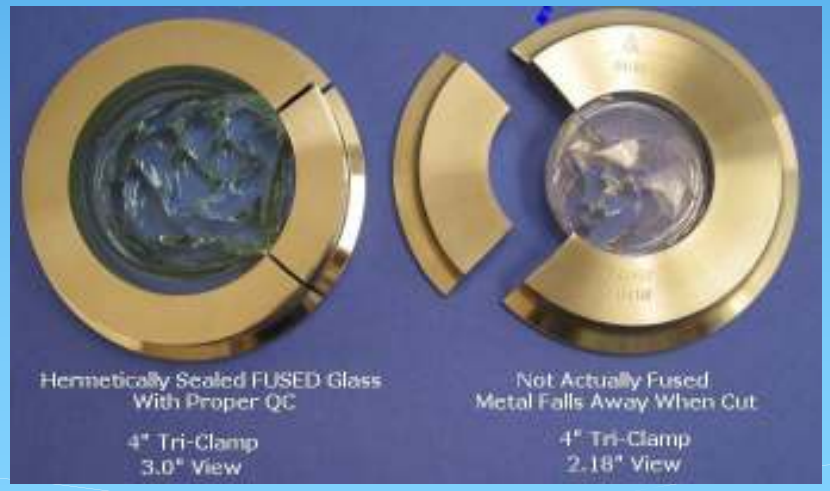
Innovative Microscopic Online Imaging

The way crystallization is observed has been revolutionized with the advent of the following:

- SugarScope
- Glass Reactor Microscope
- InFlow
- CrystalScope



The fundamentals of the system include the fused glass lens technology.

- Creates a rugged, high pressure boundary for the camera and light to see through.
- Glass surface is polished to a mirror finish and because it seals flush with the metal lens tube (or front cap) there are no gasket sites where contamination can build up

The key component to vision based systems is lighting.

- high illumination, "cold light"

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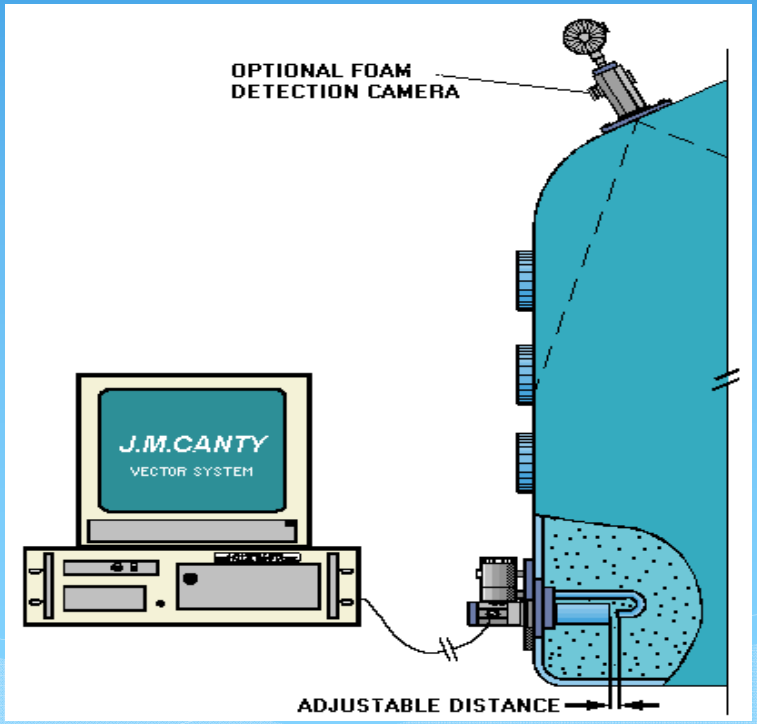
SugarScope

- High speed video system the captures and displays live video images of sugar crystals in a sugar pan under process conditions
- Enables monitoring without disrupting pressure and temperature for sampling.
- Implements fused glass technology
- Adjustable gap



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Glass Reactor Microscope

- Same measurement capabilities as the SugarScope
- Includes thermal control
- Ability to mix/agitate
- Access for additional instrumentation through the lid
- Real Time Crystal Size Analysis
- Crystal Distribution by Major, Minor Diameter, Area, Perimeter
- Crystal Size & Shape
- Crystal Count
- Density Of Crystals
- Detection Of Seeding Problems
- Automated Temperature & Vacuum Controls During Crystal Growth

InFlow

- Same measurement capabilities as the SugarScope
- Fused glass
- Adjustable flow cell
- Back lighting and camera assembly to create an optimal online particle sizing environment

CrystalScope

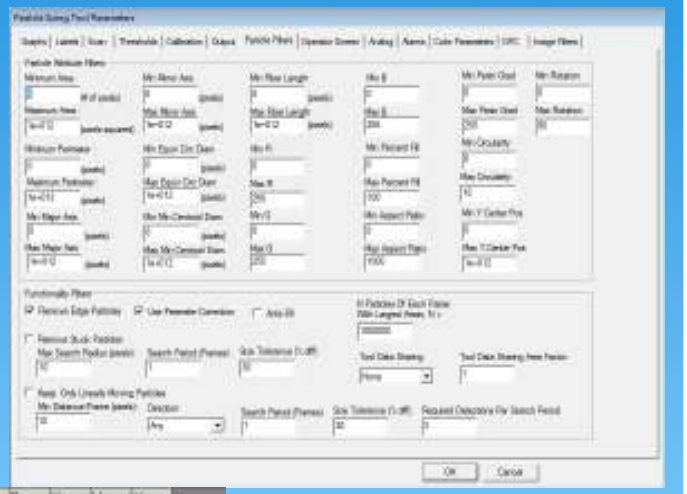
- Same measurement capabilities as the SugarScope
- Real time crystal size analysis
- Uniform backlighting for true shape illumination
- Ethernet controlled magnification for variable lens settings
- Fused glass, high pressure/temperature seal from process
- Adjustable gap

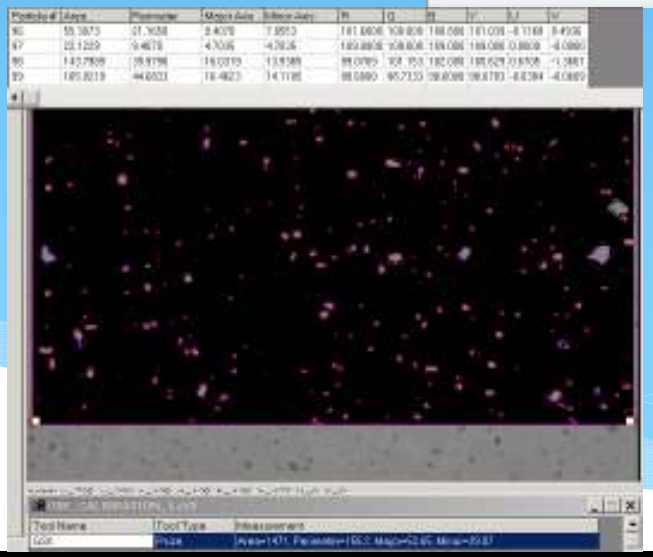
Online, Real Time Measurement

- Non-contact
- Accurate
- Repeatable
- Response Time
- Archived Data

Software Features

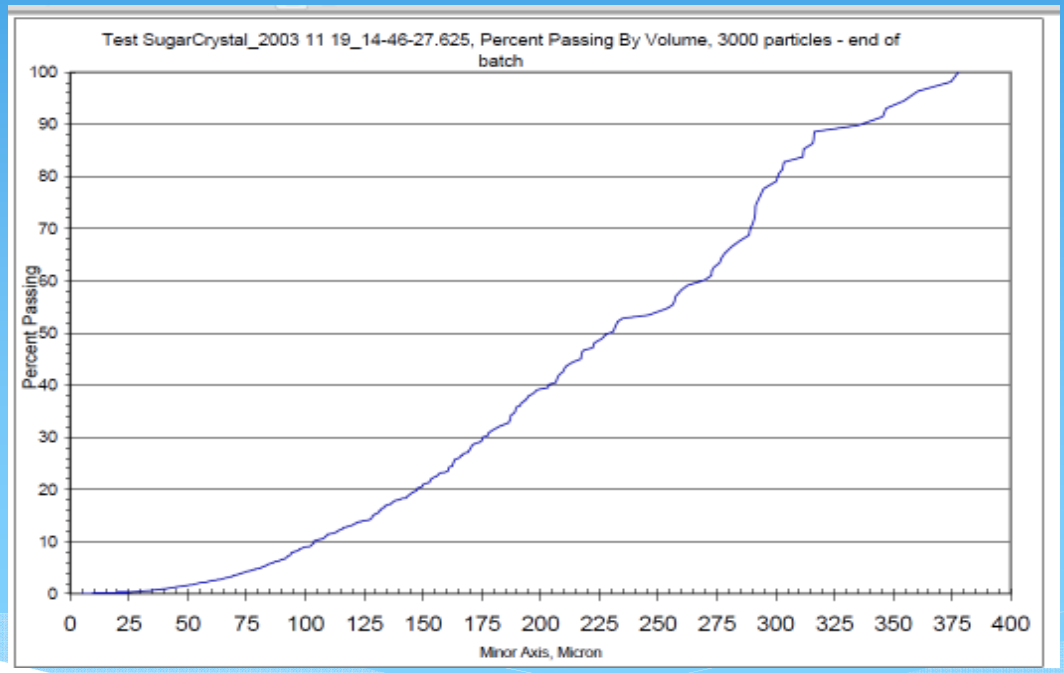
- Major and Minor Axis
- Area and Perimeter
- Max. and Min. equivalent circle diameter
- Centroid diameter
- Fiber length
- Color values, RGB
- Aspect ratio
- Perimeter gradient
- Circularity
- & more
- Allows for customizable distinction between “good” and “bad” product





Output Data/Process Control

- Displays data
  - Graphs (automatically generated)
  - Bin analysis
  - Tables
- Output Signals
  - 4-20 mA
  - Spreadsheet
  - Database
  - OPC interface
  - Modbus
- Process Control

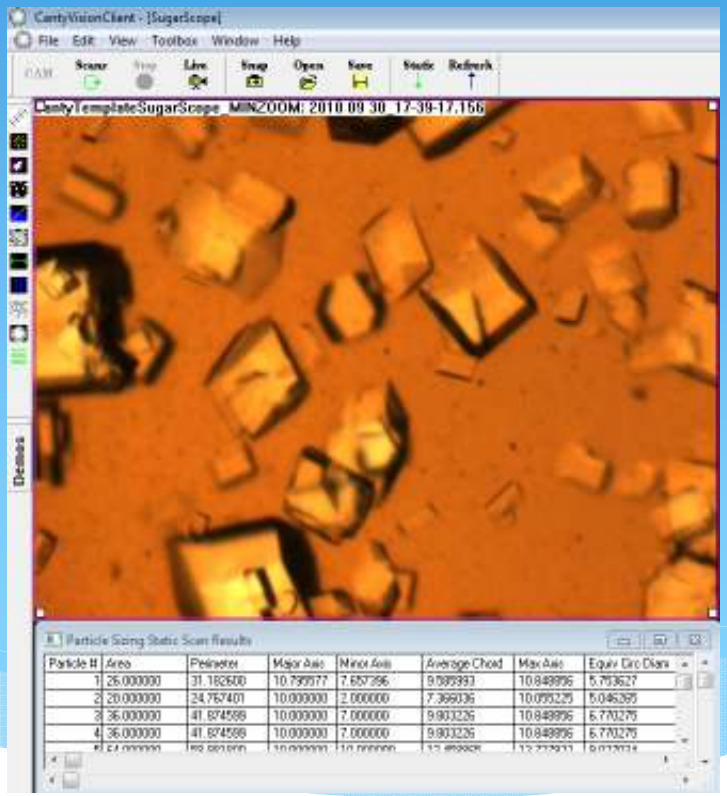


Visual Verification

- Key Advantage
  - Operators view process in real time
  - Cross-hairs/Inverted image
- R&D
  - Recording Files (.AVI and .MPEG4)
  - Images (.BMP)
  - Data (.TXT, .CSV, .XLS)

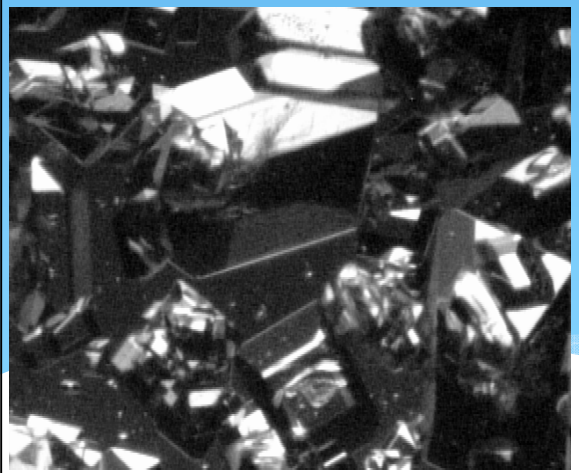
Successful Applications

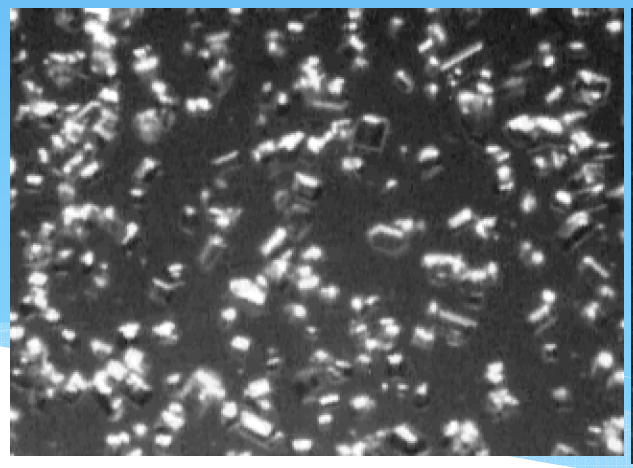
- Batch crystallization
- Lab-based reactor setting
- Wanted to determine the level of seeding required for a range of process parameters
- Simulated process conditions, variable seed rates tested.
- Determined initial point of nucleation
- Determined addition rates to yield an optimum product quality

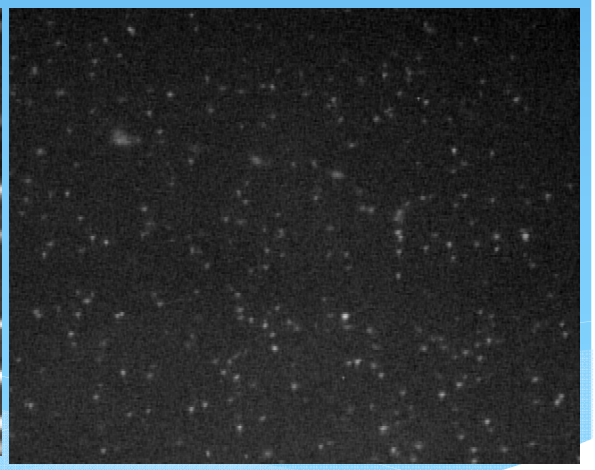


Successful Applications

- In-process setting, continuous
- Ability to track and record dissolution data







Summary

Various crystallization imaging methods of the past have resulted in inefficient and unreliable data collection, leading to less yield and costly decreases in profitability.

Microscope Online Imaging measures in-line, in real time a particle count in each frame to insure crystals are growing uniformly and not agglomerating, generating fines or fragmenting.

Size and polymorph of the crystals is simultaneously measured and broken down into various parameters that can be used for analysis and to control the rate of growth and increase yield.

Conclusion

- Real Time measurement, online
- Repeatable/Accurate
- 2D True size and shape
- Particle count
- Visual Verification
- Output Signals
- Process Control
- Better Quality Product
- Higher Yield