CAMSIZER®
Dynamic Image Analysis
ISO 13322-2 conform

Measuring the Size and Shape of Frac Sand and other Proppants

Webinar Presentation
Friday, March 9th, 2012

Speaker: Dipl.-Ing. Gert Beckmann
Retsch Technology GmbH

Host: Ian Treviranus
HORIBA Scientific Inc.
Abstract

Measuring the Size and Shape of Frac Sand and other Proppants

The size and shape of frac sands and other proppants plays a critical role in keeping fractures open and at the desired conductivity. Learn how the CAMSIZER has greatly improved the accuracy and speed of proppant analysis. This information will be useful for any petroleum engineer or proppant supplier referencing ISO 13503-2 or API RP 56/58/60 standards.

This Webinar will cover the following topics:

* Faster, more accurate size measurement
* Reporting sphericity and roundness
* Objective results in only 3 minutes
* Example measurement results
Benefits for the Proppant Industry

• Instead of size measurement with sieve shaker and shape analysis by visual inspection you can do a CAMSIZER analysis of both in a much shorter time

• Reproducible results because of high statistics and accurate calibration

• Measuring easily in compliance of API specifications of grain size (90%), oversize and dust (<1%), roundness and sphericity and measure the dust content before and after the crush resistance test

• Measure the increase of layer coating thickness and change of roundness because of the resin coating of sand grains or ceramic proppants

• Also possible: Measuring diameter and length of extrudates
Measuring Principle
CAMSIZER Principle (Two Cameras)

**Advantages**
- Precise full-frame images
- Wide dynamic range: 30µm to 30mm

**Dynamic Factor:**
\[
\frac{x_{\text{max}}}{x_{\text{min}}} = 1000
\]
Two-Camera-System

Measuring Principle

Basic-Camera

Zoom-Camera
Oil and Gas Exploration

Workers at the drill hole of the rig
Oil and Gas Exploration

Principle draft of modern and effective oil and gas exploration
Propped Frac & Acid Frac

open fracture during job (frac width = \( w_f \))

proppant used to prop the frac open

fracture tends to close once the pressure has been released

acid etched frac walls
Different Types of Proppants

The Hierarchy of Conductivity

- High strength: Uniform size and shape, Thermal resistant
  - Tier 1: High Conductivity Ceramic
- Medium strength: Irregular size and shape
  - Tier 2: Medium Conductivity Resin-coated sand
- Low strength: Irregular size and shape
  - Tier 3: Low Conductivity Sand

Image courtesy of CARBO Ceramics
*original located here: http://www.carboceramics.com/hierarchy_of_conductivity/
Different Types of Proppants

Frac Sand (<6,000psi)
- Jordan
- Ottawa
- Brady

Resin-Coated Frac Sand (<8,000psi)
- Cureable
- Precured

Intermediate Strength Ceramics (<10,000psi)

High Strength Ceramics (<15,000psi)
Different Principles to increase the Oil and Gas Conductivity

Image courtesy of Schlumberger
*original located here: http://www.slb.com/
Natural Sand Proppants (Brown and White)
Resin Coated Sand Proppant
Loading Facility of Sand Proppant Trucks
Unloading of a Proppant Railcar
Ceramic Proppants
Ceramic Proppants
Resin Coated Ceramic Proppant
High Strength and High Roundness Resin Coated Proppants

- Ceramic Core
- Growth of Ceramic Proppant during production process
- Resin Coating

Starting Core
Growth of Ceramic Proppant
Resin Coating

Resin Coating
Growth of Ceramic Proppant
Starting Core
CAMSIZER Measurement (Start)

Ceramic Proppant
CAMSIZER Measurement (End)

Ceramic Proppant
Comparison of Methods: Sieving

Advantages
- robust and industrial-suited
- easy handling
- references available from user

Disadvantages
- high amount of time and work
- low resolution, small number of investigatable classes
- limited sample amount
- no shape analysis possible

Worn out sieves
Typical Grain Sizes of Proppants

Figure 1. Some recognised size classes for proppant sands (adapted from Hoagberg & Koerner-Moore)

<table>
<thead>
<tr>
<th>Screen size (mm)</th>
<th>Mesh #</th>
<th>8/12</th>
<th>10/20</th>
<th>20/40</th>
<th>70/140</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>6</td>
<td>8</td>
<td>16</td>
<td>40</td>
<td></td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>10</td>
<td>20</td>
<td>70</td>
<td></td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>16</td>
<td>30</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>20</td>
<td>40</td>
<td>140</td>
<td></td>
</tr>
<tr>
<td></td>
<td>16</td>
<td>30</td>
<td>50</td>
<td>200</td>
<td></td>
</tr>
<tr>
<td>pan</td>
<td>pan</td>
<td>pan</td>
<td>pan</td>
<td>pan</td>
<td></td>
</tr>
</tbody>
</table>
Sieving problems (here Overloading)

1. Move
2. Sliding friction
3. Static friction

Round particles are captured without release
Sieve Correlation ⇔ CAMSIZER of Natural Brown Sand Sand

Comparison of sieve (black) and CAMSIZER data (red). The agreement is excellent. The CAMSIZER measurement can directly replace the sieve analysis, without changing the product specifications.
CAMSizer Results of Different Natural (Sand) Proppants

Size analysis of 5 different natural sand proppant samples (#12/20, #16/30, #20/40 and #30/50). Each sample was measured twice. The repeatability is excellent. One sample of white sand #40/70 is shown for comparison.
Size analysis of 9 different proppant (sand and ceramics) samples (#20/40). Some have wider, some have more narrow size distributions. One ceramic proppant sample had a bimodal distribution (red “Ceramic-Prop-EP-20-40-Mesh-40”).
Shape Comparison

Shape comparison between natural sand proppants and ceramic proppants. There are two clearly different ranges of Aspect Ratio (Krumbein’s Sphericity). Analysis of other shape parameters are possible as well (Convexity for ceramic bead twins, Symmetry for good and broken ceramic beads, Krumbein’s Roundness etc.)
Grain Size Analysis of Ceramic Proppants
Shape Analysis of Ceramic Proppants

![Graph showing Q3 (%) against Aspect Ratio with different material types and mesh sizes as data points.]

- Ceramic-Proppant -16-30Mesh_xc_min_001.rdf
- Ceramic-Proppant -20-40Mesh_xc_min_001.rdf
- Ceramic-Proppant -20-40Mesh_xc_min_002.rdf
- Ceramic-Proppant -20-40Mesh_xc_min_003.rdf
- Ceramic-Proppant -30-60Mesh_xc_min_003.rdf
- Ceramic-Proppant -30-60Mesh_xc_min_004.rdf
- Blasting-Product-40-120Mesh_xc_min_001.rdf
- Proppant + Interlocking Grains-10-20Mesh_xc_min.rdf

[Graph legend showing different materials and mesh sizes with Q3 (%) on the y-axis and Aspect Ratio on the x-axis.]
Hydraulic Fracturing

Principle draft of modern and effective oil and gas exploration
Proppant Flowback
Ceramic Proppant with Interlocking Particles

Amounts of Proppant Beads and crushed interlocking particles in a mixture

Measurement Results
Measurement Results

Ceramic Proppant with Interlocking Particles

CAMSIZER can find the mixing ratio of Proppant Beads and Angular Interlocking Grains

\[ Q_3 \text{ (round particles)} = 32.8\% \]

\[ 1 - Q_3 \text{ (b/l=0.9)} = 32.8\% \]

\[ Q_3 \text{ (angular particles)} = 67.2\% \]
Fines Migration & Plugging

Ceramic Prop after API single cycle crush test at 6000 psi
CAMSIZER – Advantages
Measuring Broken Beads

© Retsch Technology 2012 - 40

Applications – Ceramic Proppants

Symmetry \sim \min \left( \frac{r_1}{r_2} \right)

20% broken
17.4% broken

21min~

22
21
20
19
18
17
16
15
0.948 0.949 0.950 0.951 0.952 Symm

Q3 [%]

0x0

r

20% broken

good product

broken particle

r1

r2
Particle Shape

- **Breadth-/Length-ratio**

- **Roundness**

- **Symmetry**

- **Convexity**

Measurement Results
Optical Process Control
analysis for size and shape

Measurement Results

x-values in mm
Nr=0, d=2.148,
 xf_min=2.148, xf=2.203, xf_max=2.241,
 xMa_min=2.134, xMa=2.199, xMa_max=2.241,
 xc_min=2.148, xc=2.200, xc_max=2.241,
 Symm=0.9827, b/l=0.9480,
 SPHT=0.9818, Convex=0.9997

OK

x-values in mm
Nr=0, d=1.966,
 xf_min=1.970, xf=2.546, xf_max=3.063,
 xMa_min=1.889, xMa=2.419, xMa_max=3.047,
 xc_min=1.966, xc=2.419, xc_max=3.060,
 Symm=0.9209, b/l=0.6361,
 SPHT=0.9066, Convex=0.9995

OK
Fig. 14. Diagrams illustrating the measurements made for determination of: (A) particle roundness (Wadell, 1932); (B) angularity (Loes, 1964a); (C) circularity (Riley, 1941); and (D) irregularity (this paper). See text for further explanation. The same particle is also illustrated in Fig. 1.
Fig. 2.—Camera lucida drawings, a, of a grain retained on the 0.125-mm. sieve, b, of a grain retained on the 0.061-mm. sieve. (The figures give the dimensions in millimeters.)
Visual Inspection and Comparison with Krumbein’s Chart
Areas of Application

Trend Analysis
Quality Control

>90% within specification => API conform
Maintenance-Free by Venturi-Flushing
12 Years CAMSIZER

~ 600 installed CAMSIZER Instruments worldwide:
Nearly on all continents
For many applications/industries:
API conformity, Brown sand, Ceramic proppants, Extrudates, Grains, Proppants, Resin coated proppants, Sand, White sand ........
Particle Size of Drilling Fluid

### Formula Manipulation (1)
\[ D_{v,0.5} = \frac{9.56806 \mu m}{9.56723 \mu m} \]

### Formula Manipulation (2)
\[ \% < 6 \mu m = 35.292 \%
\% < 6 \mu m = 35.2231 \%
\% < 6 \mu m = 35.3086 \% > 75 \mu m = 1.8668 \%
\% > 75 \mu m = 1.90562 \%
\% > 75 \mu m = 2.36741 \% 

---

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>File Name</th>
<th>( D_{v,0.1} )</th>
<th>( D_{v,0.5} )</th>
<th>( D_{v,0.9} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Drilling Mud Sampling 2</td>
<td>200911301126035.NGB</td>
<td>1.211</td>
<td>9.568</td>
<td>38.289</td>
</tr>
<tr>
<td>Drilling Mud Sampling 2</td>
<td>200911301126036.NGB</td>
<td>1.212</td>
<td>9.602</td>
<td>38.490</td>
</tr>
<tr>
<td>Drilling Mud Sampling 2</td>
<td>200911301127037.NGB</td>
<td>1.211</td>
<td>9.567</td>
<td>39.830</td>
</tr>
</tbody>
</table>

Average

- \( \% < 6 \mu m = 35.292 \% 
- \% > 75 \mu m = 1.8668 \% 

Std Dev.

- \( \% < 6 \mu m = 0.001 \) 
- \% > 75 \mu m = 0.048 

CV (%)

- \( \% < 6 \mu m = 0.020 \) 
- \% > 75 \mu m = 0.206 

ISO 13320-1 (20.0, 15.0, 20.0)

**PASSED** **PASSED** **PASSED**
Surface Area

Size, shape and specific surface area (SSA) affect catalyst performance

Measure SSA using BET: SA-9600

- Flowing gas BET method
- Low price, operating costs, maintenance
- Easy to use, fastest measurement time
- No vacuum system required
- Single or multi-point
- Up to three samples simultaneously
Petroleum Analysis

Many instruments to measure the quality of oils, fuels, etc.
SLFA-20 ASTM D4294 for crude

MESA-6000 ASTM D7220 for low ppm sulfur and chlorine
CAMSIZER
for elongated particles (extrudates)
CAMSIZER® length definitions for elongated particles (extrudates)

\[ X_{Fe\ max} \]

\[ X_{Ma\ min} \]

\[ X_{Fe\ rec} = X_{length} \]

\[ X_{Ma\ min} \]
Length + Diameter of Implants

Length measurement $x_{length} \sim 26$mm
Diameter measurement $\varnothing \sim 0.85$mm

validation and test
1. with plastic and
2. steel cylinders

measurements of produced implants (release time 1 month)
Slides Shown During the Q&A Session
Fitting of CAMSIZER result to Sieving

fitted result

CAMSIZER-measurement $x$ (red) to sieving $*$ (blue)
Digital Image Processing
Measuring of Width ⇔ Sieving

\( x_{c_{\text{min}}} \) “width”

\( x_{c_{\text{min}}} \)

Comparison

CAMSIZER-measurement \( x_{c_{\text{min}}} \) (red) and sieving * (black)
Digitale Imaging ⇔ Sieving
Digital Imaging ↔ Sieving Cubes / Angular Particles

- $x_{\text{Sieve}}$
- $x_{\text{min}}$ Camsizer
- $x_{\text{max}}$ Camsizer

Graph showing %Q3 vs. x [µm] with two curves labeled RT669_3993_Z_LB_05%_xc_min_001.rdf and RT669_RT_3993.ref.

© Retsch Technology 2012 - 66
Digital Imaging ⇔ Sieving

Examples of samples with edgy particles without fitting

CAMSIZER-result $x_{c\min}$ (red)
sieve analysis * (black)
Digital Imaging ↔ Sieving

New elementary fitting with single (narrow) sieve class and entire distribution
Digital Imaging ↔ Sieving

Elementary fitting = Sieve Correlation with single (narrow) sieve class

Samples with similar shape
Digital Imaging ↔ Sieving

Q₃ – Fitting

Elementary - Fitting
Slides Shown During the Q&A Session
Comparison between Static ⇔ Dynamic Image Analysis

ISO 13322-1

ISO 13322-2

© Retsch Technology 2012 - 72

Retsch Technology
Solutions in Particle Sizing

a VERDER company
Slides Shown During the Q&A Session Sampling
Sample Quantity in Mass or Volume in Relation to the Particle Size

ISO 13322-1

Tables 1 to 3 list, as a function of the geometric standard deviation $\sigma_g$ of the test powder, the number of particles to be measured, $N$, the median particle size of a cumulative mass distribution, $x_{50,3}$, the Sauter Diameter, $\bar{x}_{1,2}$, and the average mass diameter, $\bar{x}_{3,0}$, for the precision, respectively. $\delta = 0.05, 0.1$ and $0.2$. Here, the confidence coefficient probability, $P$, is equal to 0.95.

Table 1 - Number of particles to be measured $N$, $\delta = 0.05$, $P = 0.95$

<table>
<thead>
<tr>
<th>$\delta$</th>
<th>$\sigma_g$</th>
<th>$x_{50,3}$</th>
<th>$\bar{x}_{1,2}$</th>
<th>$\bar{x}_{3,0}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>1.10</td>
<td>585</td>
<td>389</td>
<td>131</td>
</tr>
<tr>
<td>0.15</td>
<td>1.20</td>
<td>1480</td>
<td>934</td>
<td>294</td>
</tr>
<tr>
<td>0.20</td>
<td>1.30</td>
<td>2939</td>
<td>1808</td>
<td>528</td>
</tr>
<tr>
<td>0.25</td>
<td>1.40</td>
<td>5223</td>
<td>3103</td>
<td>843</td>
</tr>
<tr>
<td>0.30</td>
<td>1.50</td>
<td>8526</td>
<td>4920</td>
<td>1247</td>
</tr>
<tr>
<td>0.35</td>
<td>1.60</td>
<td>13059</td>
<td>7355</td>
<td>1750</td>
</tr>
<tr>
<td>0.40</td>
<td>1.70</td>
<td>19028</td>
<td>10504</td>
<td>2363</td>
</tr>
<tr>
<td>0.45</td>
<td>1.80</td>
<td>26617</td>
<td>14457</td>
<td>3096</td>
</tr>
<tr>
<td>0.50</td>
<td>1.90</td>
<td>36007</td>
<td>19295</td>
<td>3956</td>
</tr>
<tr>
<td>0.55</td>
<td>2.00</td>
<td>47358</td>
<td>25093</td>
<td>4952</td>
</tr>
<tr>
<td>0.60</td>
<td>2.10</td>
<td>60811</td>
<td>31919</td>
<td>6092</td>
</tr>
</tbody>
</table>

A Sufficient Sample Quantity is Based on the **Number** of Particles
Additional Slides to better explain Sampling and Sample Splitting
Separation of fine and coarse particles

Separation happens during
- Transport processes (container, train and truck)
- Feeding processes (funnels, vibration feeders, belts)
- and Storage (bulk pile, silo)
Separation of fine and coarse particles

Segregation (separation by size) happens during
- Filling processes (silo)
- Feeding processes (bulk pile)
- Accumulation of fines in the middle of the pile
Different Fertilizer Product Types, Different Production Methods, as well as Different Sizes and Shapes
Sample Splitting
Sample Splitting
Sample Splitting
Sample Splitting
Sample Splitting

1/6 1/8 1/10
Additional Slides to better explain the CAMSIZER Technology
Resolution

Detection of particles

One pixel is element of a projection when **at least half of the pixel** is covered.
Range of use

Shape

Size

30 µm to 30 mm
Advantages

- fast
- reproducible
- precise

measurement principle

maintenance-free & robust
What is the size of this particle?
Particle Size

$X_{c,\text{min}}$ “width”

$X_{\text{area}}$ “diameter over projection surface”

$X_{\text{Fe, max}}$ “length”

CAMSIZER results are compatible with sieve analysis

$A' = A$
Volume Definition

Ellipsoid model leads to better results

\[ V_{\text{ellipsoid}} = \frac{\pi}{6} \cdot x_{Fe \text{ max}} \cdot x_c^2_{\text{min}} \]
Additional Slides to better explain the Dispersion Technology (against static charged particles)
Size, Shape & Density Measurement of Charged Coated Resin Beads using an Electric High Voltage Ionizer
How to separate the dust part from the granules to measure these single dispersed?

Dispersion of coated granules with Ultrasonic or ALU-C Aeroxide
Additional Slides to better explain the Basics of the used Parameters and the Calibration of the Instrument
Particle Distributions
Cumulative distribution $Q_r$

$Q_r$ [%]

Based on
<table>
<thead>
<tr>
<th>Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number</td>
</tr>
<tr>
<td>Length</td>
</tr>
<tr>
<td>Area</td>
</tr>
<tr>
<td>Mass/Volume</td>
</tr>
</tbody>
</table>

Measurement Results

median
Measurement Results

Particle Distributions
Frequency distribution $q_r$

$q_r[\%/\text{mm}]$

\[ p_r = \int_{x_1}^{x_2} q_r(x) \, dx \]

\[ q_r(x) = \frac{dQ_r(x)}{dx} \]

\[ \Delta Q_r(x_1, x_2) = q_r(x_1, x_2) \Delta x \]

Mv(x): mean value of x, determined from the distribution of all particles

$q_r(x)$ is the frequency distribution of particles, and $p_r$ is the cumulative distribution (probability) of particles within a certain size range.
Particle Distributions
Histogram fractions $p_r$

$n$ is the number of the fraction

$Q_r(x = x_n) = \sum_{i=0}^{n} p_{r,i}$
Reproducibility

Calibration with traceable standard

=> Absolute accuracy
Glass Bead-Standard 500µm – 2000µm

1,25mm ± 24µm
95% (* Q₃ 75% )
written in the calibration certificate

CAMSIZER and Sieving (72%)
done with calibrated sieve by hand
Whitehouse Standard XX030

Calibration Results

Certificate of Analysis

IMAGE ANALYSIS STANDARD

Catalogue Number: XX030

1. Graphical Review

2. Tabular Summary

- % Micronage
- Average Micronage (µm)
- Standard Deviation
- Coefficient of Variation
- Skew Factor
- Kurtosis Factor
- Median Diameter
- Upper and Lower Percentiles

3. Additional Information

- Date of Calibration
- Certification Authority

- Dr. G. H. Hine
- Associate Analyst

Although the information contained herein is correct to the best of the company's knowledge, no warranty, express or implied, is made or given by the company with respect to the accuracy or completeness of the information and the company disclaims all liability, whether direct or indirect, which may result from the use of the information contained herein. The information provided is for reference only and is not to be used for sales or marketing purposes without the written permission of the company.

a VERDER company
Thank you for your attention!