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Quantification and Uncertainty in Particle Morphological Analysis

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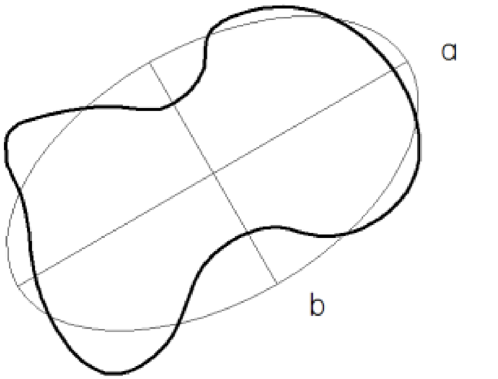
Quantification of morphology is complex

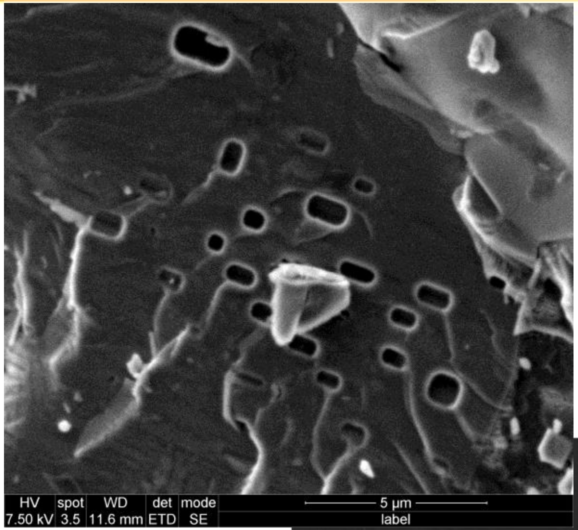
- Fundamentals: 3D objects imaged in 2D
- Many morphological parameters exist
  - For example, MAMA software has 15, IP+ has more than 50

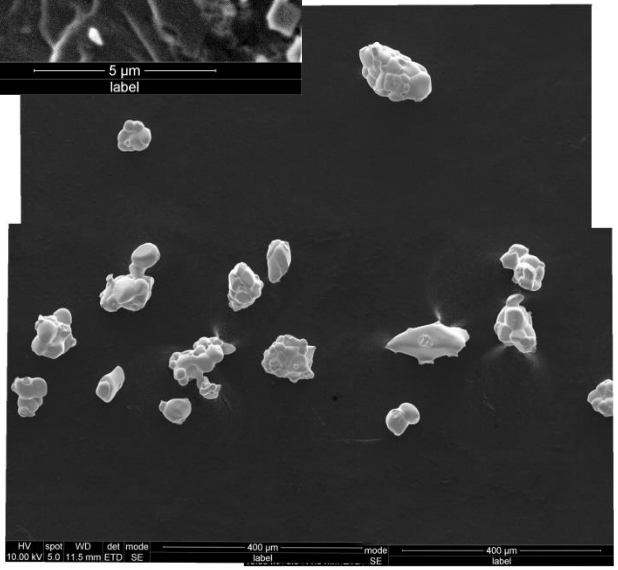


- Definition of objects
  - Where are the edges of the particles?
  - Intimately tied to imaging conditions and particle morphology

Quantification of morphology is complex

- Group vs. Individual
  - Statistics vs. description
- Relative importance depends on application
  - Attribution
  - Quality control
- Sample size
  - $^{1}/_{\sqrt{n}}$ rule for normal distributions: 1000 particles are needed for ~3% precision of mean





Error and uncertainty in particle analysis

Three fundamental sources of uncertainty

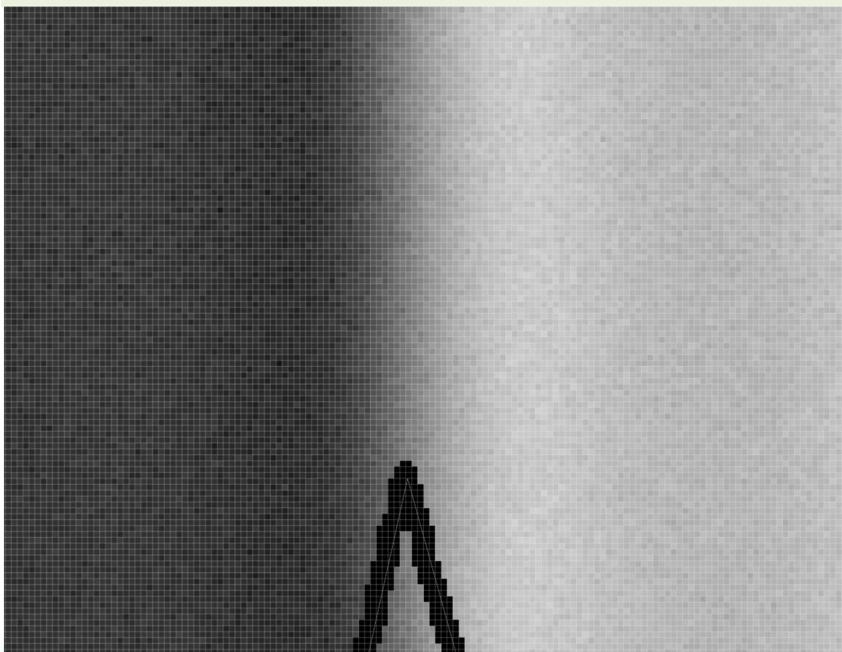
1. Sampling of particles: how do we know the subset of particles we analyze represent the parent?
2. Imaging: the process of creating a 2D digital image from the particles introduces uncertainty
3. Image processing: quantitation of morphological parameters introduces error and uncertainty

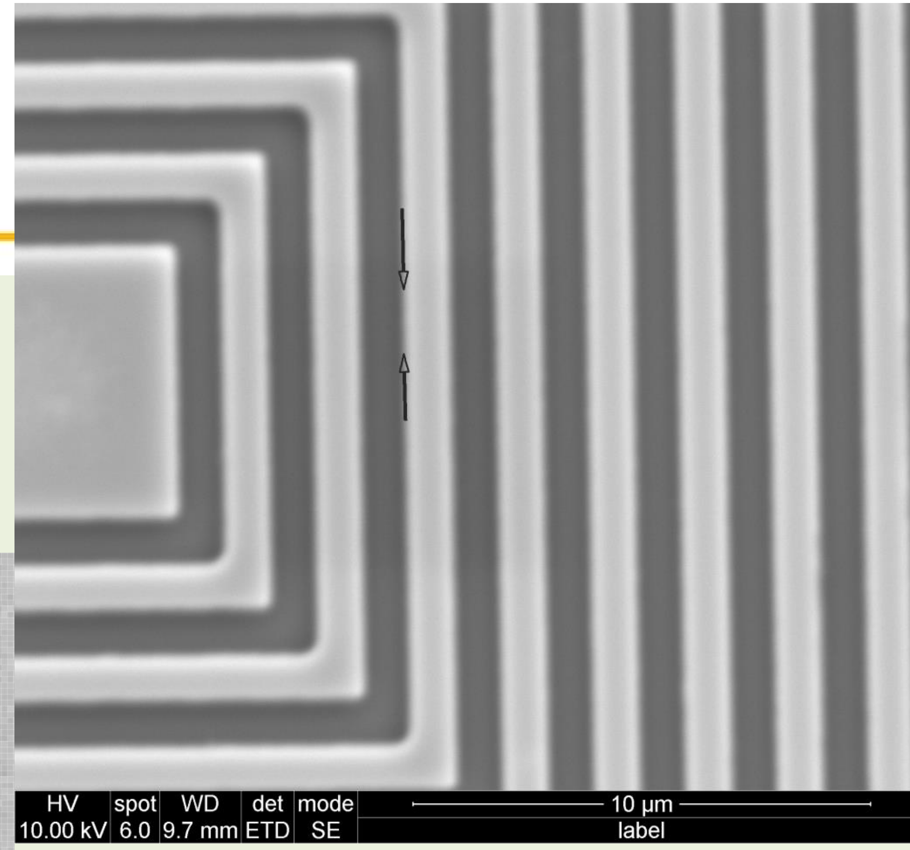
Uncertainty in SEM imaging

Uncertainty source	Effect
Magnification calibration	Accuracy of all measurements
Voltage/spot size	Surface detail and edge sharpness
Apertures and alignment	Resolution and edge sharpness
Focus and stigmation	Resolution and edge sharpness
Contrast/brightness	Edge location
Imaging mode (backscatter detector vs. secondary electron detector)	Edge sharpness
Specimen tilt	Image distortion
Specimen height	Magnification calibration change

SEM magnification

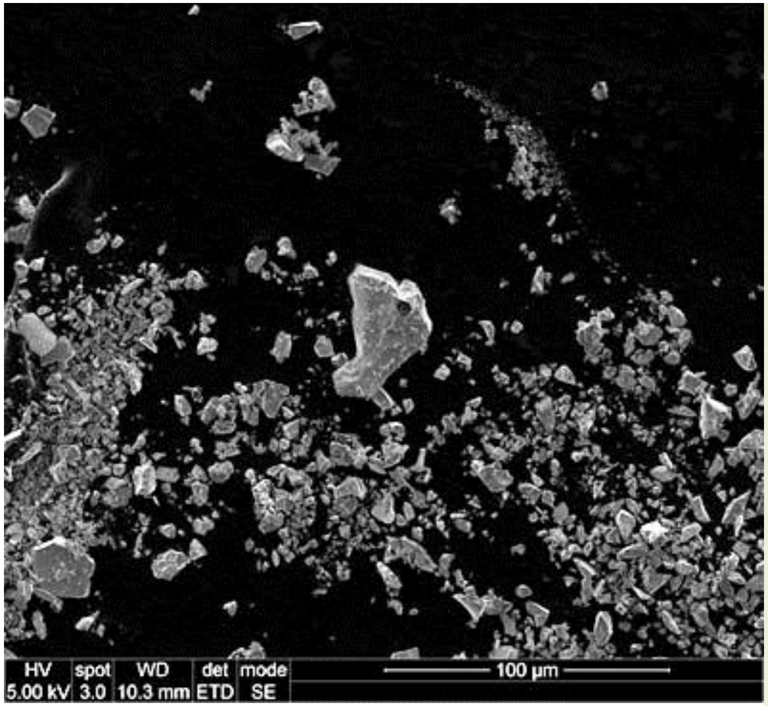
- Defines accuracy of measurements
- ~ 0.5% accuracy





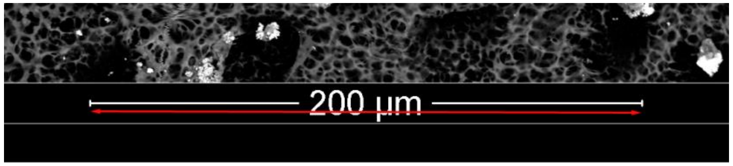
- The edge of the fiducials are not perfectly defined
- Geller magnification standard certified error ~0.8% (better than ~5% error for NIST SRM 8820)

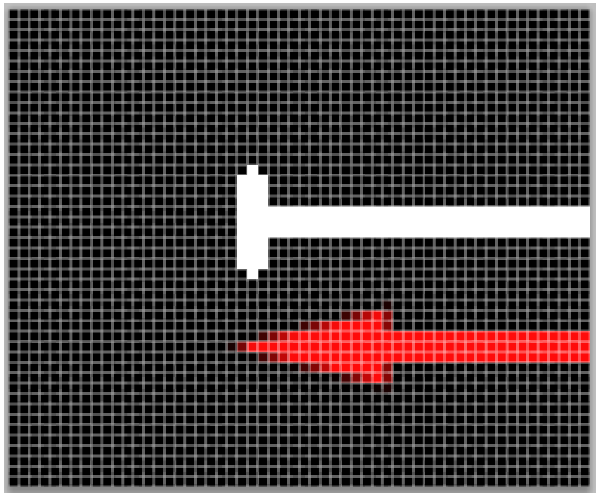
Sampling uncertainty



- Crushed and unsieved particles will have a wide range of sizes
  - Does the distribution of particles deposited on the SEM sample mount reflect the bulk sample?
    - Only extensive mounting using different techniques will answer the question
    - This is the largest potential source of error

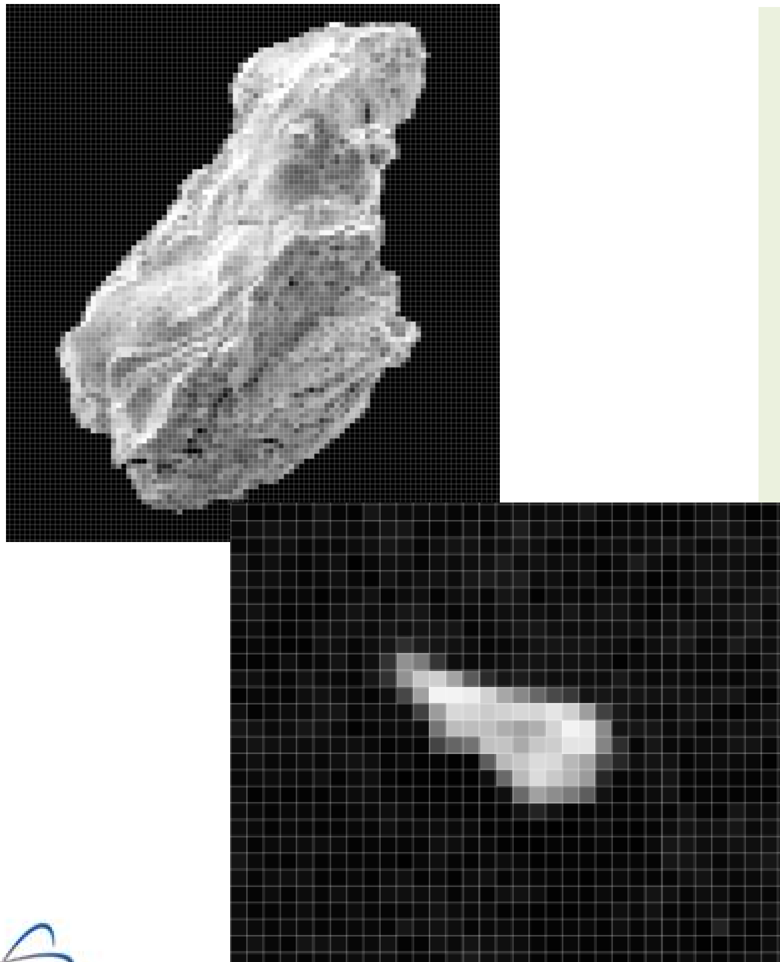
MAMA measurement uncertainty





- Accuracy is based on transfer of magnification from digital image
  - Can be done *exactly* by inputting pixel/µm value from SEM calibration
  - Manual method in MAMA ~ 0.1% error, with practice

MAMA measurement uncertainty



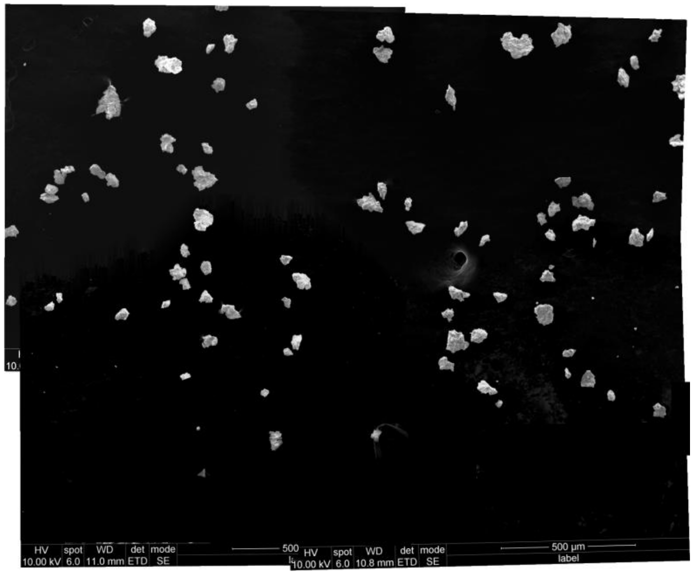
- Errors due to pixelation
  - Above ~1000 pixels typical particles are well represented
  - Morphological parameters lose meaning for small highly pixelated objects
    - "Pixel count" area remains useful
    - Perimeter related measures (circularity, convexity) are particularly bad at low pixel counts

MAMA measurement uncertainty

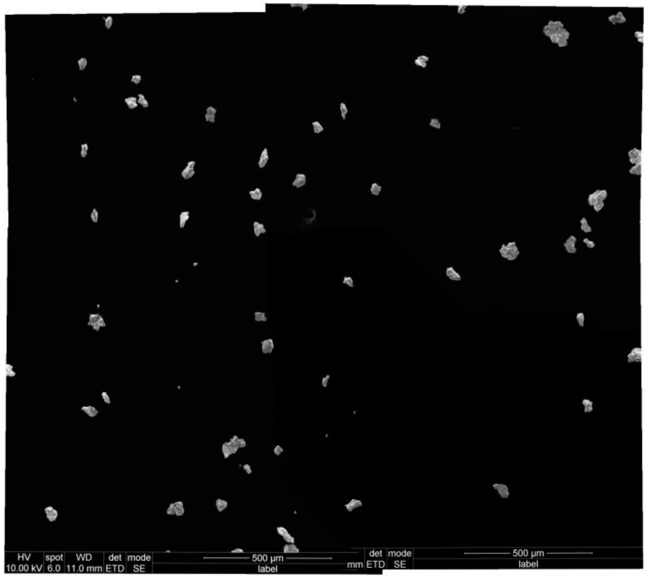
- Key set of morphological parameters for statistical description
  - Pixel count area
    - This is the fundamental measure for 2D images
  - Equivalent Circular Diameter $\sqrt{4A/\pi}$
    - Same information as PCA, reduced to 1D
  - Best-fit ellipse parameters
    - Major and minor axes, aspect ratio (major/minor)
- IAEA document NST018 (National Nuc. Forensics Library)
  - Lattice structure, aspect ratio, porosity, color, particle size and distribution, shape, surface features

Preliminary User Test (Internal)

- 2 clean images were analyzed independently by 4 MAMA users
- Excellent uniformity of results



~ 85 particles



~50 particles

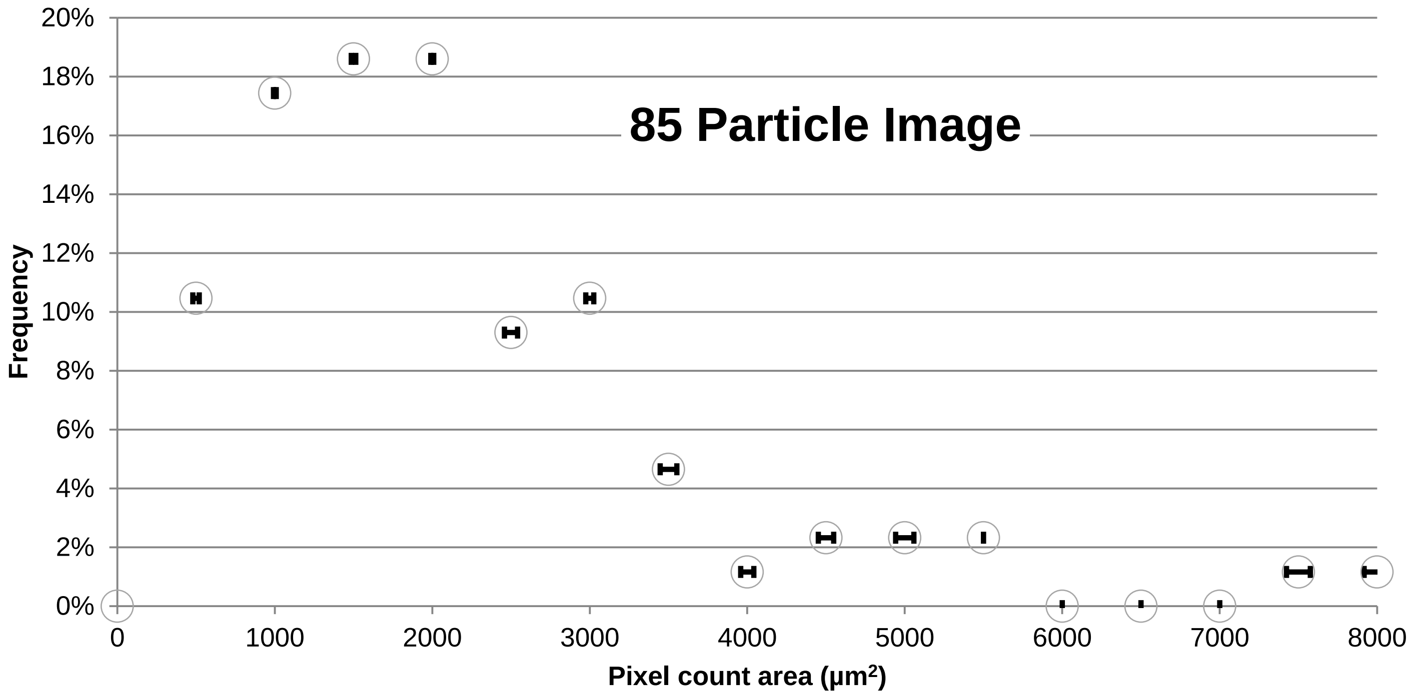
Preliminary User Test (Internal)

Correlation between measured elliptical major axes is excellent

	DSS	ARR	ELT
ARR	0.999899	1	
ELT	0.999789	0.999812	1
KJK	0.999722	0.999651	0.999878

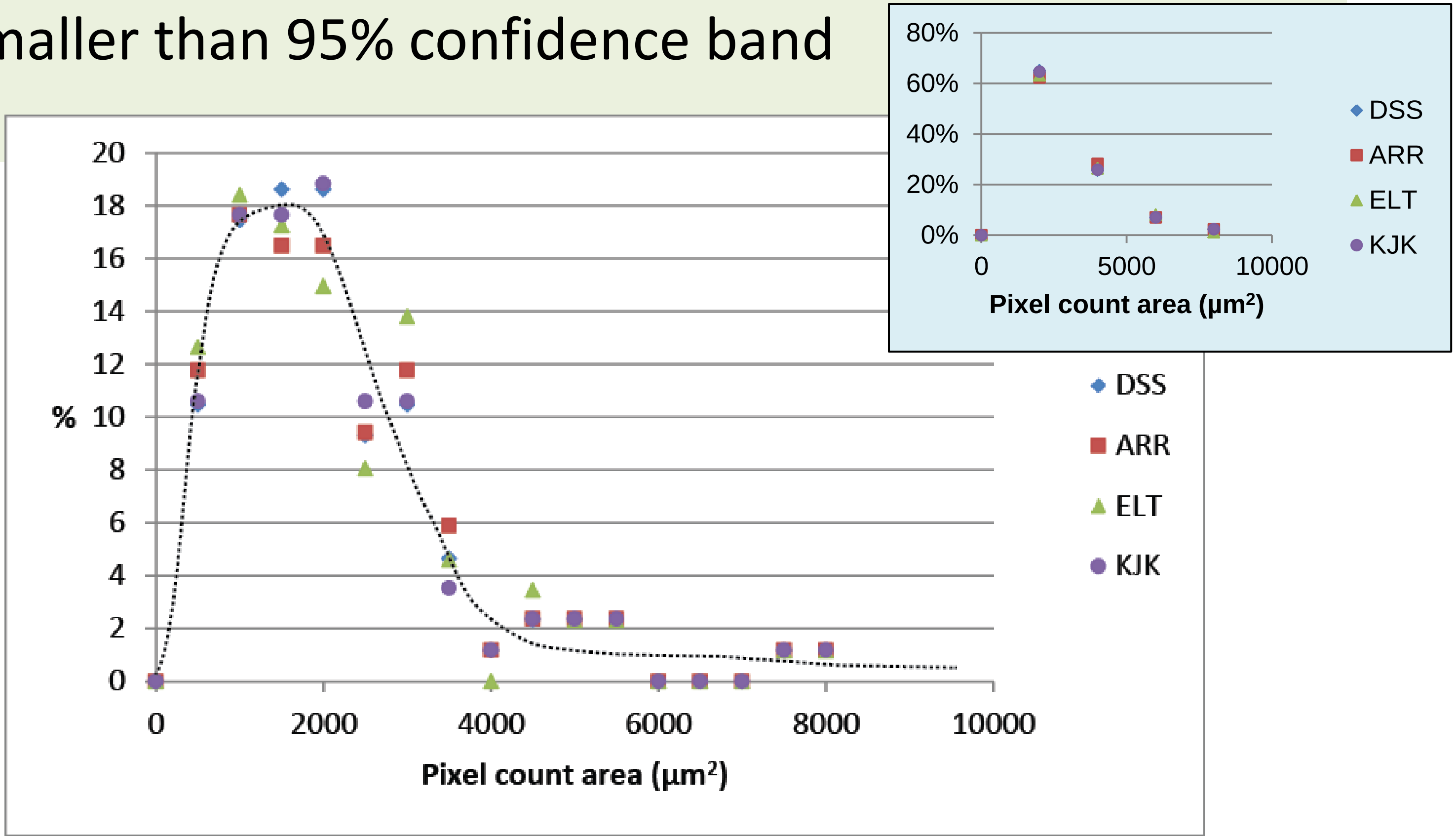
Preliminary User Test (Internal)

- Comparison of PCA distributions
  - Error bars are standard deviations within the bin



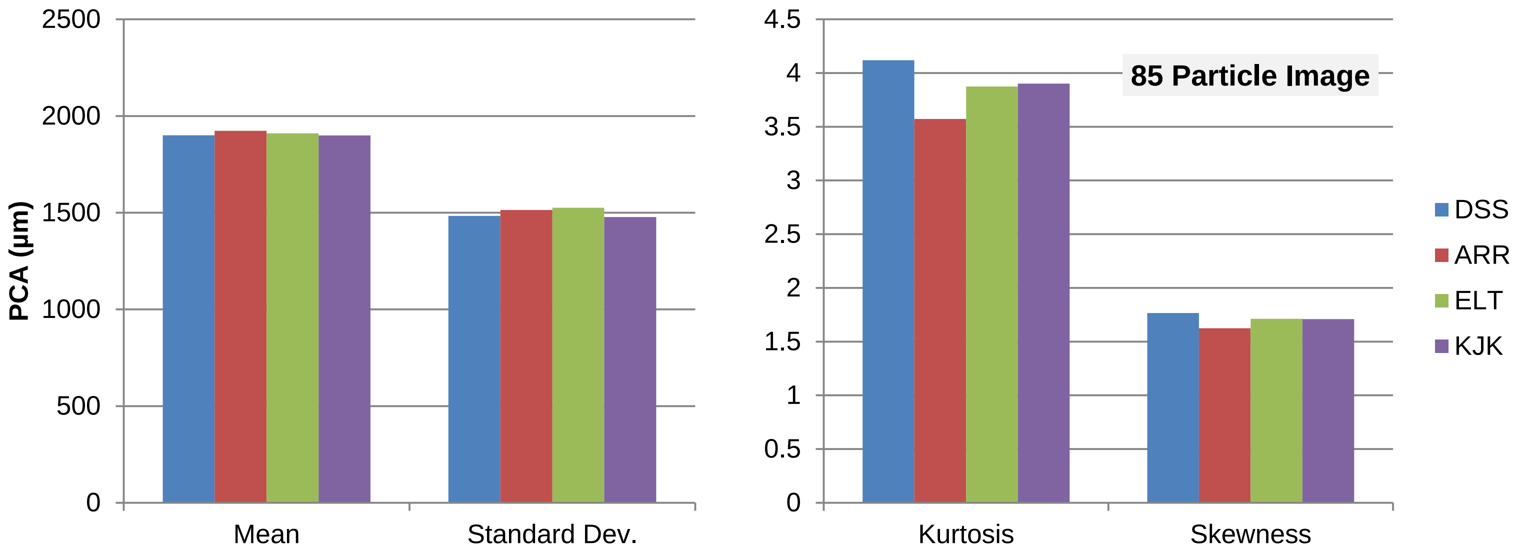
Preliminary User Test (Internal)

- Comparison of PCA distributions
  - Scatter is due to small population (85 particles): bin size is smaller than 95% confidence band



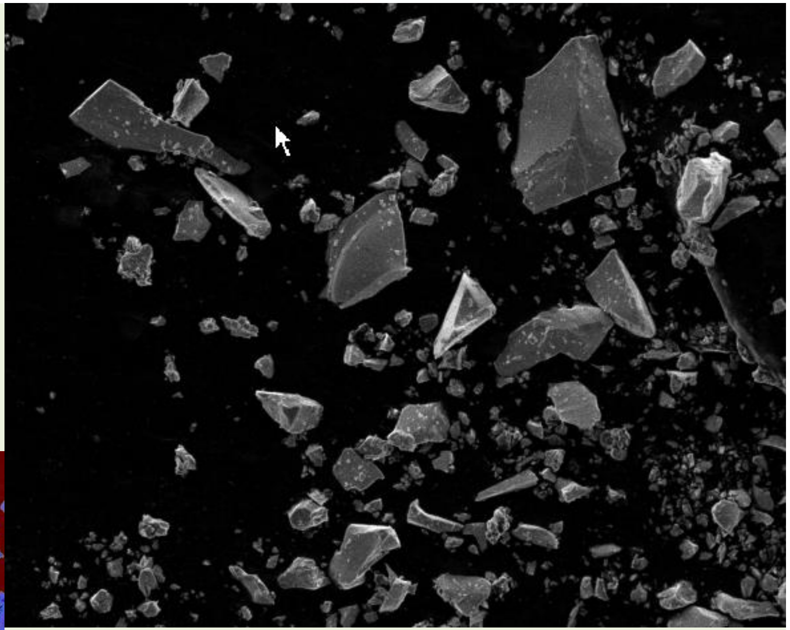
Preliminary User Test (Internal)

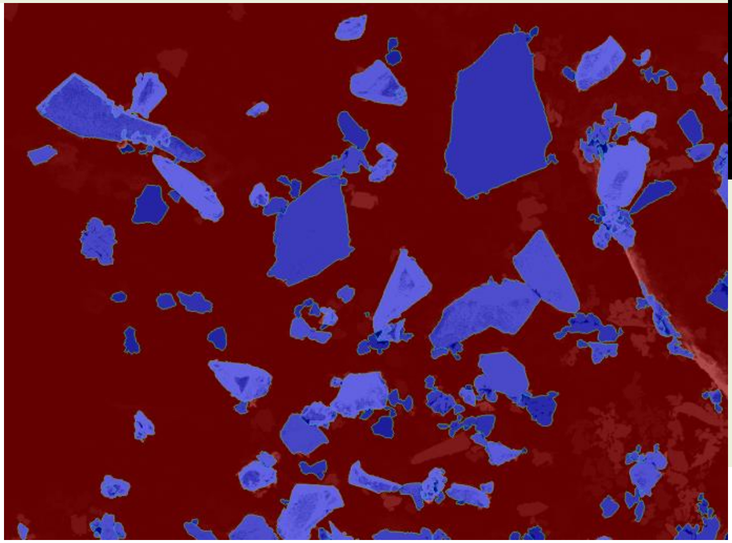
- Comparison of PCA distributions
  - Compare key statistical measures: mean, standard deviation, kurtosis, and skew



Segmentation: Strength of MAMA

- MAMA segmentation is superior
  - Most image processing programs use filters and thresholding: too crude
  - More than 200 particles easily defined in example





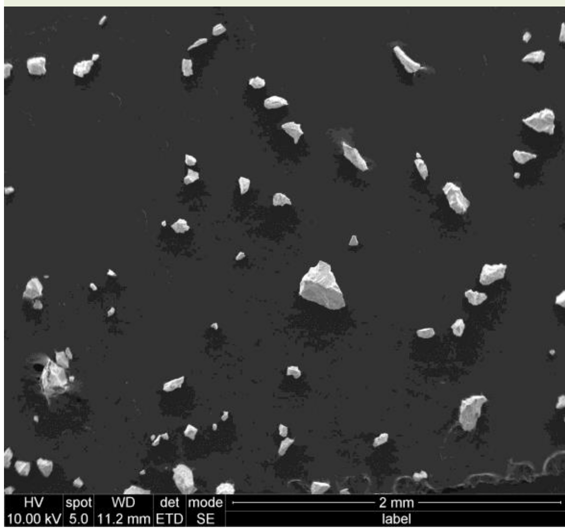
Easy to use and precise segmentation necessary to get high population numbers required for good statistics

Proposed round-robin uncertainty test: 3 levels of rigor

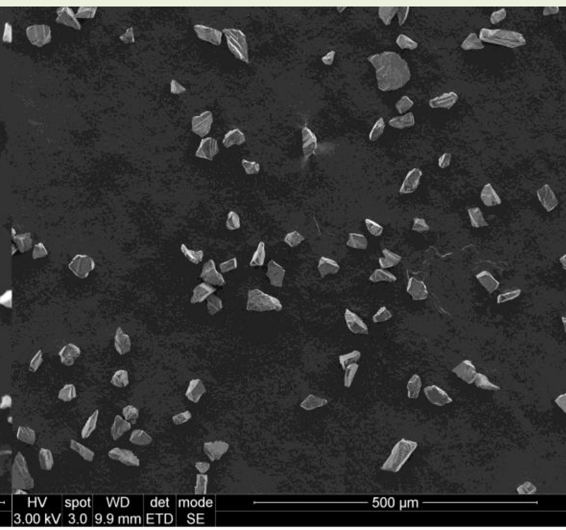
1. Users measure a common set of images made from selected NIST standards using MAMA software. Images will cover a range of difficulty in terms of particle overlap, edge clarity, etc.
   – Tests the user variability associated with using the MAMA software.
2. LANL mounts NIST standard powders on SEM specimen mounts and users create their own images using these, then perform image analysis.
   – Adds the SEM operator variability and SEM calibration variability to uncertainties associated with (1).
3. Users individually mount their own SEM specimen mounts using the NIST standards, create images, then perform image analysis.
   – Adds the variability in subdividing and mounting powders for SEM observation to the uncertainties associated with (1) and (2).

Step 1 user test

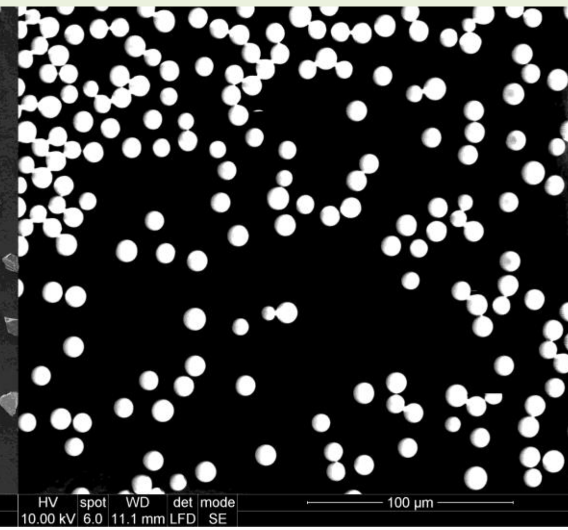
- 3 external users, all from different laboratories
  - Image set includes 1 traceable standard, 1 quasi-standard, and 1 typical specimen



Typical specimen

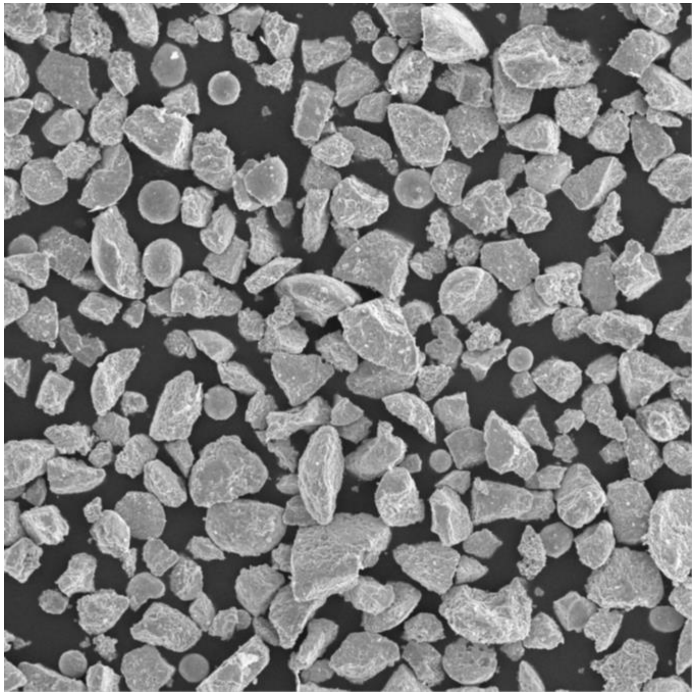


Nominal 325 mesh diamond



10 µm standard poly spheres

Proposed NIST standard powders



- Glass particle standards
  - SRM 1017 – 1021
- W(C,Co) particles
  - SRM 1984, 1985
- ZrO_2 (YSZ)
  - SRM 1978, 1982
- All are very stable and sizes are characterized by NIST

Practical considerations

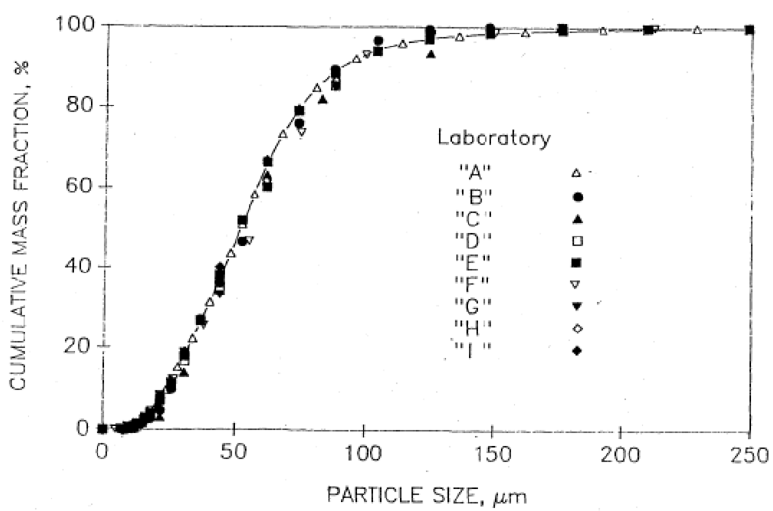


Figure 3. PSD of zirconia by different laboratories using different models of Microtrac R

9 laboratories were involved in standardizing NIST SRM 1982

- Developing a true morphological standard involves a balance of effort vs. payoff
  - How many users?
  - Which morphological parameters?

Future efforts

- Segmentation is the key
  - MAMA already ahead of the pack
    - Ease and precision of segmentation is tied to getting meaningful statistics from powder sets

Abstract

A key part of forensic analysis of interdicted nuclear materials is examining the microstructure of the materials, which are frequently in the form of particulates. We are developing methods for quantifying the morphology of particle sets imaged using scanning electron microscopy. In order to use this data effectively, we need to understand the uncertainties involved in measuring the morphology of particles. This presentation discusses the sources of uncertainty in scanning electron microscopy, and presents the results of an internal user test of the MAMA software package being developed under a DHS Transformational and Applied Research program. The steps required to establish morphological standards similar to NIST standards are discussed.