

MEASURING GRAIN SIZE

Introduction

The immediate objective of any test or experiment is to obtain reliable, accurate data. Obviously, the quality of this data is directly related to the methods we use to acquire it and these methods involve both good design and proper execution. Fortunately, the design of many tests and experimental methods has already been done and they have already been established as standard testing techniques. There is probably a standard testing technique available for any kind of test you want to do and while each field of study has its own unique set of standardized methods but many are applicable in just about every field. Many of the most widely used standards have been established and are maintained and endorsed by government and industry groups and many are also international in scope.

While we are concerned here primarily with standard testing and measurements, standards in general go well beyond this to include materials and processes, designs and protocols. Yet all will share the following four basic elements:

- C clear statement of the method's intended uses
- C clear definition of all key terms, including the thing being measured
- C method of making the measurements
- C method of reporting the results.

Standards testing methods are essential in science, industry, even everyday life. The most compelling reasons for using standardized testing techniques is that they

- C have been shown to be appropriate for the particular study
- C minimize personal and systematic bias
- C produce reliable, reproducible results when done correctly
- C provide means for evaluating the degree of accuracy.

They also tend to be efficient, as they are often optimized not only for quality results but also for easy of use. But probably most important aspect of standards is that they establish common language and practices that are recognized by peers and critics as the appropriate way to do things.

Whichever field you work in, there will be times when you will be expected to adhere strictly to formal, published standards while at other times you might use a standard method as the basis for a new or novel technique.

This experiment features a standard measurement technique that is widely used in microscopy. It provides a good example of a simple technique that has been optimized for accuracy, efficiency and the elimination of bias. Everyone will use the this technique to measure the size of features on identical images. Will everyone obtain the same results?

Background

Most alloys, ceramics, rocks, etc. are polycrystalline – aggregates of many small, sometimes different types, of crystals. The individual crystals are called grains and their size can influence a number of physical and mechanical properties. Until the field of stereology was established the techniques used to measure the grain size were as different as the people who did the measurements. The results were not very consistent nor were they reproducible. Obviously, there was a need for a reliable, accurate method of making grain size measurements. This led to the establishment of several standard methods. Perhaps the most widely used method is the mean lineal intercept, or Heyn's, technique. The mean lineal intercept length is the average length of a line segment that crosses a sufficiently large number of grains. It is proportional to the equivalent diameter of a spherical grain.

The mean lineal intercept length is determined by laying a number of randomly placed test lines on the image and counting the number of times that grain boundaries are intercepted. Mathematically, it is defined as:

$$\bar{L}_L = \frac{1}{\bar{N}_L} = \frac{L_T}{PM} \quad (1)$$

where N_L is the number of intercepts per total length of the test lines L_T , P is the total number of grain boundary intersections and M is the magnification.

Procedure

Measure grain size in attached figure. Use method described on the next page and the attached form to record your results. Start by noting the magnification of the image and then make at least 5 test measurements.

Results

Calculate the following:

1. Mean lineal intercept length
2. Standard deviation of your measurements
3. Confidence interval at a 95% confidence level

When finished, share your results with the rest of the class.

Analysis

1. How do your results compare to those of the rest of the class?
2. Is having a standardized method sufficient?
3. How would you improve results?

Measurement of Grain Size Using the Mean Lineal Intercept Method

ASTM Standard E 112-88 describes the lineal intercept method in general and the Heyn procedure in particular. The following is an abbreviated version of the Heyn procedure.

1. Magnification, Test Line and Fields

- C Ideally, the length of a single test line and the magnification should be such that 50 intercepts can be counted.
- C The use of multiple fields to obtain 50 intercepts is discouraged, although permitted, due to the bias which will decrease the accuracy as the number of fields increases.

2. Count the intercepts...

- C An intercept is the segment of the test line which overlays one grain.
- C Count 1 for each intercept and $\frac{1}{2}$ for each time an end of the test line falls in a grain.

... or count the intersections

- C An intersection is the point where the test line cuts a grain boundary.
- C Count 1 for each intersection, 1 for each tangential intersection, $\frac{1}{2}$ when an end of the test line ends exactly on a grain boundary and $1\frac{1}{2}$ when the intersection occurs at a triple point.

3. Measurement Strategy

- C Make counts on 3 to 5 blindly selected and widely separate fields to obtain a reasonable average for the specimen. Additional counts may be required to obtain the desired statistics.
- C Use four or more orientations of the test line to eliminate effects due the moderate departures from an equiaxed structure.
- C Use orthogonal sets of parallel test lines if the structure is distinctly non-equiaxed.

4. Tabulate the Results

- C Calculate the average lineal intercept length.
- C Calculate the standard deviation.
- C Calculate the confidence interval for the desired confidence interval.

5. Report the Results

- C When reporting the results include the magnification, length of the test line, total number of intercepts counted along with the mean intercept length and the confidence interval for the stated confidence level.

Basic Equations for Sampling Statistics

Arithmetic Mean

$$\mu_x = \frac{1}{n} \sum_{i=1}^n x_i \quad (2)$$

Standard Deviation

$$\sigma_x = \sqrt{x^2 - \mu_x^2} \quad (3)$$

Standard Error of the Mean - the error associated with sampling a population

$$SE = \frac{\sigma_x}{\sqrt{n}} \quad (4)$$

Confidence Interval (95% confidence level)

$$\mu_x \pm 2 SE \quad (5)$$

Data/Calculations

Name _____ Date _____

Sample Number _____

Sample Description _____

Magnification _____ X

Test Number	Intercept Count	L (microns)	L ² (microns ²)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
Sum:			
Mean:			
Standard Deviation:			
Standard Error:			
Confidence Interval:			

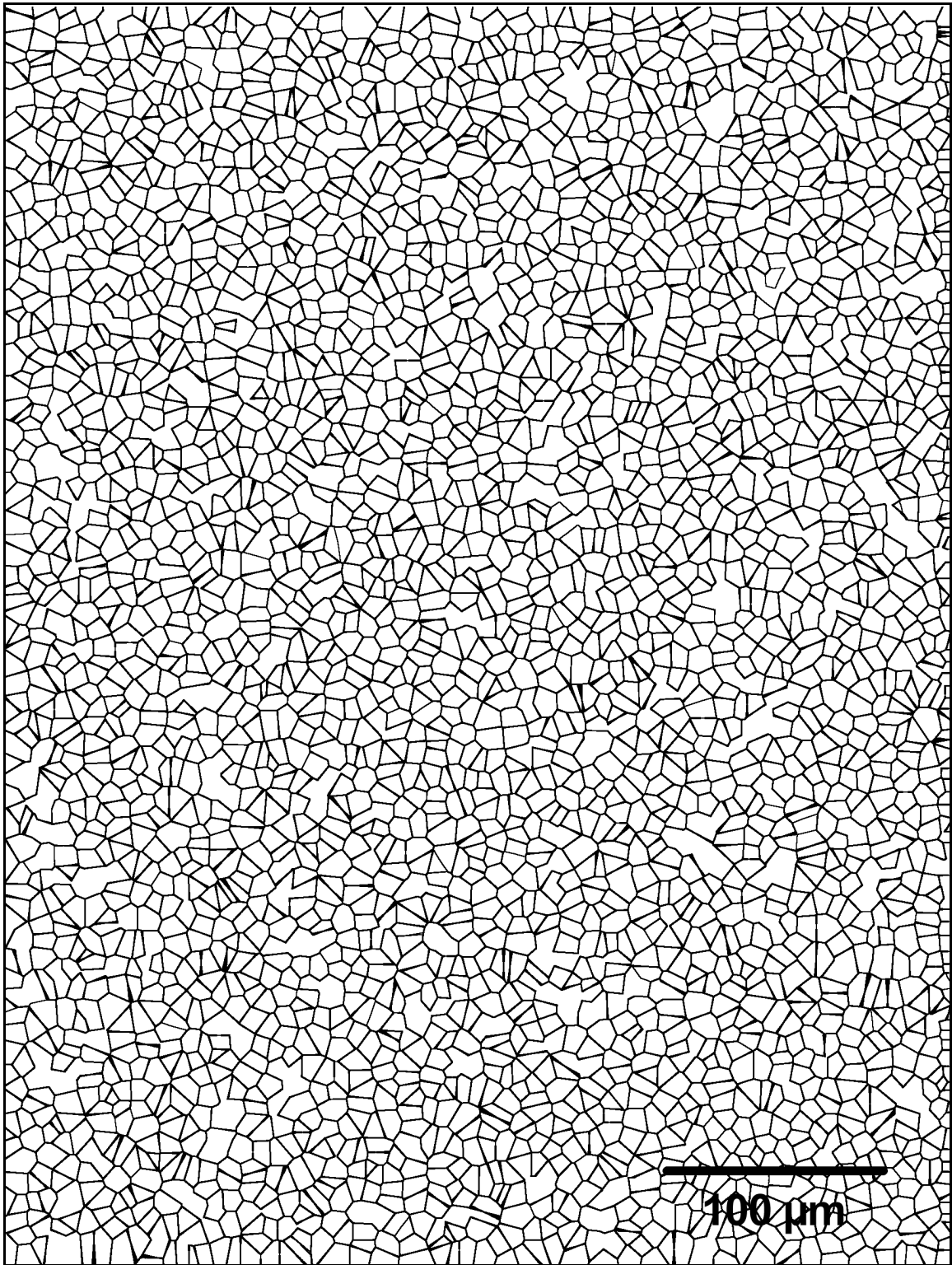


Figure 1 Sample Microstructure #1