WHAT METALLURGIST MUST KNOW ABOUT

GRAIN SIZE ASTM E 112

A STEP BY STEP GUIDE for getting fast and accurate results using an Image Analysis Software.

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A Publication of



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INTRODUCTION

What metallurgist must know about Grain Size.

This e-book has been created to provide a practical guide from non-experts to highly experts metallurgist when need to measure grain size according to ASTM standards.

The objective of this guide is to help experienced metallurgists, to be confident using the new digital tools, and for the new generations to have a better understanding of the basics.

Let's say that for experienced metallurgists is the way to be updated and for younger metallurgists is the way to get all the experience behind this measurements.

This is what you get with this guide:

- You will **understand the principles** of measuring grain size.
- You will be able to **identify** a **problem** when measuring.
- You will be able to **measure the grain size manually, semi**automatic and full automatic.
- You will be able to **explain results**.
- You will be able to **ensure the quality** of your measurements.

So let 's start...



CHAPTER ONE

Uunderstanding grain size geometry



Grain size geometry

"Shapes must be space filling." Plateu 1873

What is a grain?

This is the first question that comes to our mind and the answer is quite simple:

"A grain is a particle with a specific geometry"

The ideal grain geometries are:

Tetrakaidekahedron with 14 faces, 24 corners and 36 edges and the *Dodecahedron* is a polyhedron with 12 faces.

The *polyhedrons* are geometric bodies which flat faces enclose a finite volume.



Tetrakaidecahedron: 14 faces



Dodecahedron 12 faces

GRAIN SIZE GEOMETRY

What does it mean ?

It means that a geometry body to be considered as a grain, it must comply with the following requirements:

- Shapes must be space filling.
- Surfaces must exhibit minimum surface area and minimum surface tension. Plateu 1873.

As you can see in figure A), there are a group of individual grains, and because of their geometry, it can be joint to become a bigger particle.

So a metal structure is formed by grains.



Figure A)



Geometry is important because the ASTM E 112 standard provide measuring procedures to determine the average grain size in metallic materials, and the procedures are **geometric based**.

So are independent of the metal or the alloy, because the measurement procedures could be used to the grain size estimation for crystal or cell size for non-metallic materials as well.

This is stated in the introduction of the ASTME112 standard.



CHAPTER TWO

Top 3 Measurement methods



TOP 3

Definition of ASTM GRAIN SIZE

In order to measure the grain size, is necessary to have a scale that represents the grain size.

Imagine giving a result of grain size in microns, or in inches, or millimeters, is kind of complicated don't you think?

ASTM defines grain size as: $n = 2^{G-1}$ n = number of grains / in² at 100X G = ASTM GRAIN SIZE NUMBER

To make it easier, here is a table with a Grain size number and the number of grains / in² at 100X

G	n	G	n
1	1	6	32
2	2	7	64
3	4	8	128
4	8	9	256
5	16	10	512

TOP 3

Definition of ASTM GRAIN SIZE

Now if you are asking, how could I measure it?

Current ASTM E 112 standard describe the use of these 3 measuring methods :

- 1. Comparison charts.
- 2. Jeffries Planimetric Grain Size.
- 3. Heyn / Hilliard / Abrams Intercept method.

Which method should I use?

We will explain in detail how to measure the grain size in the next chapters, but before measuring you have to be sure if the equipment available in the lab can help you or not. Because you need to prepare the sample and then analyze it.

For prepare your sample you will need a cutter, a mounting press, a grinder and polisher and a fume hood for etching the sample.

Keep reading...

TOP 3

Definition of ASTM GRAIN SIZE

To analyze the sample you will need a microscope with reflected light ; it could be Upright or inverted; but make sure the microscope has a 10X objective and 10x eyepieces, to have a 100X magnification.

Also make sure that the microscope can allows you to place a reticle on the eyepieces, or there are some brands and models that you can install a grain size chart between the light path , so you an see the grain size image on the eyepiece and then you can compare it with your sample.

With different type of reticles you can measure manually all the 3 methods.

But nowadays the use of digital cameras for microscopes is more common, so consider to have an Image Analysis System with your microscope.



Upright microscope – Reflected Light





Inverted microscope – Reflected Light

66

Inverted microscope is mostly used on metallography, because the sample is placed down face and that surface is flatter and also because there are no height limit as with the vertical microscope.

Pictures courtesy from Clemex Technologies.

Comparison charts – Planimetric - Interception

Comparison Method

The most simple and easy way to measure the average grain size, is using the comparison charts.

You only have to look on the eyepiece and compare the image you see there with the standard charts, that's it.

You can also use the grain size reticle on the eyepiece to have a more accurate comparison, because the grain size standard charts are printed in the reticle, and you can compare it while observing on the eyepiece.

Is fast and easy, but is only for the average grain size, not for individual grains.





Comparison charts – Planimetric - Interception

Jeffries Planimetric Grain Size

With this method you will know the Grain Size number from calculating the average of the grain area.

The measurement is done in 2 steps.

Step #1 is to calculate the average grain area in mm²
You can do that counting the intercepted grains for the reference circle and counting the entire grains inside the reference circle.
Step #2 Calculate the ASTM Grain Size Number, using the value of the average area. (We will give more details in the next chapters)



Comparison charts – Planimetric - Interception

Heyn / Hilliard / Abrams Intercept method

With these method, you have 2 ways to measure. Using a straight line you can calculate the grain size :

#1.- Counting the grains intercepted by the line.



#2-. Counting the grain boundary intersections.



Comparison charts – Planimetric - Interception

Heyn / Hilliard / Abrams Intercept method

Now, If the structure is no equiaxed , or the shape shows some distortion, you may use 3 concentric circles like these:



But you have to define if you are going to measure the grains intercepted or the intersections of the grain boundaries.



CHAPTER THREE

Comparison method



Comparison method

Method #1

As we say before you only have to look on the eyepiece and compare what you see on the eyepiece VS the standard charts.

How could you be sure is the right number?

That's one of the main complications about this method, because it becomes subjective to the person who is comparing.

And is even more subjective when the grains are smaller and there are slight differences in size .

Imagine a structure where there are 55% of grains are size 8 and 45% are 7, complicated right?

Because as you can only compare the structure as an average, you can not know about the percentage of each size of grain, and you have to made a decision , if you qualify it as 7 or as 8.

Here is what ASTM E112 says about the comparison method.



Figure 8. French NF A04-102 grain size chart for rating McCunid-Ehn carburized samples.

Grain size ratings using the comparison chart method by an individual metallographer will vary within +/- 0.5 G units.

When a number of individuals rate the same specimen, the spread in ratings may be as great as 1.5 to 2.5 G units."

ASTM E 112 - Section 19.8



CHAPTER FOUR

Jeffries Planimetric method



Jeffries Planimetric method

Method #2

Getting the ASTM grain size number by considering the grains quantity within an area.

Now you know some of the limitations of the comparison method, which is easy to apply, but is subjective and affected by the metallurgist criteria.

Here is a more objective method to know the average grain size.

With this method you can get the grain size number in 2 steps.

<u>Step #1:</u> Calculate the average grain area in mm at 100x magnification. (N A = Number of grains per mm^2)

 $NA = f(N_1 + (N_2/2))$

f = Jeffries multiplier ; f = Mag ²/ circle area
N1= Number of grains completely inside the circle.
N2= Number of grains intercepting the circle.

Jeffries Planimetric method

Method #2

Nothing better than a an exercise.

Considering the image below as reference for the exercise:



Where: N1 = 68 and the N2 = 41

The Circle area (A) = 20106.2 mm and Magnification = 100X Replacing the numbers in the equation we can get this: NA = f (N1 + (N2 / 2)) NA = f (68+(41/2)) NA = f (88.5) f = Mag² / A f = (100² / 20106.2)=0.497 NA= 0.497 (88.5) = **44.01 mm²** NA= **44.01 mm²**

Jeffries Planimetric method

Method #2

Now we know the NA (number of grains per square mm) We need to know the grain size number. *NA*= 44.01 mm²

<u>Step # 2</u>

You can know the grain size number in two ways:

- 1. After calculating the NA number, you can go to the table 6 from ASTM E 112 standard and cross the NA number with the Grain size number. For this case the **G is 2.5**.
- 2. Or you can calculate it, using the following formulas:

A= Grain area in mm² = $1/NA = 1/44.01 = 0.0227 \text{ mm}^2$

d = Grain diameter mm= A $^{1/2}$ = 0.0227 $^{1/2}$ = 2.5

G = 2.5

Or you can use this formula:

G= $(-3.322 \text{ Log } \mathbf{A}) - 2.955 = (-3.322 \text{ Log } (\mathbf{0.0227})) - 2.955 = 2.5$



CHAPTER FIVE

Heyn / Hilliard / Abrams Intercept method



Method #3

Here's another objective method to calculate the Grain size number.

With the Heyn / Hilliards / Abrams **Intercept method** you can calculate the grain size number in 2 ways.

#1 Counting entire grains intercepted or,

#2 Counting grain boundaries intersected.

You will need to draw a line with a specific length, and you can use the following formula's:

If you want to calculate the grain size **counting the entire grains**.

NL = N / Lt

Or you can use this formula if you want to calculate the grain size **counting boundary intersections**.

PL = P / Lt

Lt= length of the reference line.

Method #3

Way #1 - Counting intercepted grains Here is an example. NL= N / Lt Lt= Line length

This example has 5 Intercepted grains + 2 half at the ends this give us a total of **6 grains**.



Method #3

Way #2 - Counting grain boundary intersections

For this example there are **6 grain boundary intersections**. **#2 Counting grain boundary intersections PL= P / Lt Lt=** Line length



Method #3

So, once we know the N or P value, we need to calculate the NL or PL. Consider the line length about 2 mm.

For NL = N/Lt = 6/2 = 3For PL = P/Lt = 6/2 = 3For this exercise, the NL and PL value is 3.

To know the grain size number we have to calculate using this formula:

G= $[6.643856 \text{ Log}_{10} (\text{ NL})]$ -3.288 <u>Eq.2 Table 6 ASTM E 112</u>

G= [6.643856 Log₁₀ (**PL**)]-3.288 <u>Eq.2 Table 6 ASTM E 112</u>

Or you can calculate it using the Mean Lineal Intercept.

The Mean Lineal Intercept length, which is the average length of a line segment that crosses a sufficient large number of grains, and it can be used to calculate the grain size number with this equation:

G= [-6.643856 Log_{10} (ℓ)]-3.288 Eq.2 Table 6 ASTM E 112

 ℓ = 1/ NL = 1/ PL

Method #3

Now, in case there are not equiaxed grains, or the grain shape present some distortion, you can use more straight lines at different angles.

Or you may use 3 concentric circles as ASTM recommends with a line length of 500 mm.



Method #3

Let's do an exercise with the following data: Lt = 11.4 mm



N= 41 grains outside circle, 25 on the middle cirlce, 20 in the inner circle

Method #3

Applying the formula for NL, this is what we got:

Lt= 11.4 mm N= 41 + 25 + 20 = 86 NL= 86 / 11.4 = 7.54 mm⁻¹ l = 1/7.54 = 0.133 mm G= [6.643856 Log₁₀ (0.133)]-3.288

G= 2.5

We can conclude that the Interception and planimetric methods are reliable and widely used, but present difficulties because is very tedious to get the result and it is more complicated if more area is required.

In order to increase accuracy you need to analyze at least 160 mm² according to ASTM E 112, and this is a difficult task to do it manually.

Keep reading, you are about to discover how to use an Image Analysis System.



CHAPTER SIX

Automatic Measurement using an Image Analyzer



Easier, faster and reliable

How could I measure more fields, faster and with reliable Results?

You got it, using an Image Analysis System.

Below is the list of the **"must know"** principles for any Image Analysis System, please take your time to see the videos, we will use that information for the excercise.

(To see the videos please click on the links)

- 1. <u>Before starting the analysis</u>.
- 2. <u>Setting up the microscope</u>.
- 3. Optimizing Image Quality.
- 4. <u>How the system Works</u>.
- 5. <u>How to calibrate the system</u>.

Easier, faster and reliable

Now you know the principles, you are ready to discover how to measure Grain Size automatically.

Imagine that you have a nice sample and your system is properly setup and calibrated.

You only have to select and detect the grain boundary and allow the system does all the work, this will be done in seconds.

<u>Click here to see it in action.</u>

But, how does the system calculate the Grain Size number?

It uses the Mean Lineal Intercept equation:

G= [-6.643856 Log10 (l)]-3.288 Eq.2 Table 6 ASTM E 112

And the Average grain size per object

G = (3.321928 log10 NA) -2-954 Eq.1 Table 6 ASTM E 112





Easier, faster and reliable

The nex question is, What about the structures like the picture below? Does the system Works the same way?



No, it does not work in the same way, because the binarization is not accurate with the structure above.

For cases like this, you can use the *Intercept method* with the digital tools of the System. <u>Click here to see it in action</u>.



Easier, faster and reliable

Probably you are asking yourself, how do I know that the measurement is correct?

This is a very good question, let me show you something really signifcant from ASTM standard:

ASTM E 112 point 19.8:

"Grain size ratings using the comparison chart method by an individual metallographer will vary within +/- 0.5 G units. When a number of individuals rate the same specimen, the spread in ratings may be as great as 1.5 to 2.5 G units."

ASTM E 112 point 19.14 :

"An individual metallographer can usually repeat planimetric or intercept grain size measurements within +/- 0.1 G units. When a number of metallographers measure the same specimen, the spread of grain sizes is usually well within +/- 0.5 G units."

Easier, faster and reliable

Measurement errors are always present, a metallographer can make a little mistake while comparing, or counting manually as well as the Image Analysis systems.

But, how could we know the error is aceptable?

The answer, Calculating the **R**elative Accuracy.

According to ASTM E 112 in the point 15.6:

"If the % RA is considered to be too high for the intended application, more fields should be measured and the calculations in 15.1-15.5 should be repeated."

As a general rule, a 10 % RA (or lower) is considered to be acceptable precision for most purposes.

Easier, faster and reliable

You need to process enough data to have a robust analysis, that it means that you need to measure more than 1 field of your sample.

How many fields?

To calculate the Confidence interval, is necessary at least 5 fields.

With 20 fields, you can get a fare enough accuracy.

If you want to reach the highest accuracy, you will need 60 fields. *Not easy to do it manually.*





CHAPTER SEVEN

Relative accuracy and Confidence interval



% R.A. and C.I.

Relative Accuracy and Confidence Interval

To calculate the relative accuracy, first you have to calculate the Confidence Interval.

Here are 2 ways to calculate the CI

It could be based on the fields **(n)** or in the number of grain áreas or intercept lengths. **(N)**

95% $CI=t.s \div \sqrt{n}$

95% $CI=t.s \div \sqrt{N}$

Where:

n= the number of fields measured (for field measurements)

N= the number of grain áreas or intercept lengths for individual measurements

t= Table 4 provide the values for t as function of n or N s= standard deviation from grain measurements.

No. of Fields,	t multiplier	No. of Fields,	t Multiplier
5	2.776 2.571 2.447 2.365	19	2.101 2.093 2.086 2.080 2.074 2.069 2.064
6		20	
7 8		21	
		22	
9	2.306	23	
10	2.262	24	
11	2.228	2.228 25	
12	2.201	26	2.060
13	13 2.179	27	2.056
14	2.160	28	2.052
15	2.145	29	2.048
16	2.131	30	2.045
17	2.120	40	2.020
18	2.110	60	2.000
		00	1.960

DIE 4.05 W Confidence Internal Multipliers & /En 40 and En

A or, number of grains, N.

% R.A. and C.I.

Relative Accuracy and Confidence Interval

After calculating the CI, you can now calculate the % of R.A.

%RA= (95% CI ÷ X) * 100

X= the mean value measured (NA,NL, or PL or I)

Making this manually is not easy, it takes more time to do it if you want to deliver a professional report.

Imagine how useful is that the %RA and CI is being calculated automatically by the software.

Highly convenient, don 't you think? This way you can focus on prepare your report and explain the results.

Results							
<u>File Edit Chart View</u>							
<u>μ</u> x Σ	. 🗂 💑	00	FLDM2 ASTME112-13 - FidN -				
FLDM2 ASTME112-13 - FldNo							
Chart							
7.50 T							
6-							
2 4 50 -							
E 4.50							
W							
15 ³							
1.50 -							
0							
1			2				
FidNo							
FldNo	ASTME112	13					
1	6.94						
Chatiatian							
Minimum	6.94						
Maximum:	6.94						
Rating #:	6.94						
Count:	1						
Under:	0						
Over:	100.00	94					
Field Count:	100.00	76					
Field Area:	794558.80	µm²					
Total Area:	794558.80	µm²					
Relative Accuracy:	0.00	%					
Confidence Interval:	0.00						



CHAPTER EIGHT

CONCLUSIONS



CONCLUSIONS

Relative accuracy and Confidence Interval

After all this chapters, you can now be sure what is necessary to do, to perform a professional measurement for grain size.

The use of the image analysis systems makes your work easier, but is necessary you understand how it Works and how to adjust it to get objective Results.

Also, with this guide you know now , how many fields are required to analyze in order to get a 10% of RA or less.

So regardless the type of material, this rules apply and even in some cases, where the structure is not easy to process digitally; with the use of the image analysis you can get objective information only by measuring more fields.

We expect you have enjoyed this e-book, we try to sintetize a huge ammount of information about this topic in some pages that can be useful and practical.

Disclaimer:

This document can not be used for legal or comercial disputes, and any posible injury, damage to personel or facilities due to the interpretation of this information ,is under user responsability only.

REFERENCES

Grain Size Measurement _ George Vander Voort ASTM E 112-13 Standard ASTM E 1382-13 Standard

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Thank you...

Feedback

Do you like this e-book?

If you found this material, useful and practical, please give us your comments about it.

My comments are...

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