QUANTITATIVE IMAGE ANALYSIS TECHNIQUE FOR DETERMINING LOCAL DENSITY VARIATION

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ABSTRACT

The PMPA Standards Committee is developing a new test method for determining the porosity of powder metallurgy products by image analysis techniques. This technique would be used to evaluate the local density variation in complex P/M parts. An inter-laboratory study was conducted to estimate the uncertainty of this new measurement technique. The results found the accuracy of the test method to be determined by test specimen preparation technique and the image analysis procedure. Such factors as magnification, number of fields examined, field-to-field variability and the detection level setting all had a role in the repeatability and reproducibility of the test method. Results are discussed for both an FC-0208 and SS-316L.

INTRODUCTION

Researchers use microstructure analysis to characterize materials, assist in understanding the cause of failures, determine phase constituents, etc. Quantitative image analysis (QIA) is a technique used to quantify these key microstructure features, e.g., grain size, inclusion or carbide size, porosity size and amount, or the amount of various phases (ref. 1,2). ASTM E 562 describes a standard test method to manually perform these counts. Many a metallographer or graduate student has toiled long hours performing these tedious counts. About 25 years ago, with the advent of the microprocessor and then the PC, the tedium of manual point counts was replaced by the computerized image analysis method. A recent ASTM standard practice, ASTM E 1245 describes a procedure to determine stereological measurements of inclusions or second-phase constituents of metals by automatic image analysis. The results of this analysis are affected by several factors:

1. specimen preparation technique
2. gray-level intensity level used to discriminate features
3. number of fields examined
4. total surface area examined
5. image magnification

In the field of powder metallurgy QIA has been frequently applied to measure porosity (amount, pore size, pore shape and pore size distribution) and microstructure phases (% martensite, bainite, pearlite and ferrite in steel alloys). One of the authors, Tom Murphy, has routinely used QIA in numerous analyses of P/M Steels, having prepared a metallography manual for P/M steels (ref. 3). He has participated in several seminars on the subject (ref. 4) and co-authored numerous technical papers where QIA has played an important role in interpreting the results (ref. 5-11). QIA has been used to better understand high temperature sintering (ref 6, 12), fatigue properties of P/M steels (ref 7-10), and sinter-hardening (ref. 11).

More recently QIA has been identified as the most promising technique to verify the density distribution predictions developed through computer simulation modeling of the compaction process (ref. 13-15). The QIA technique can be used to prepare "density maps", a visual rendition of the density distribution in the part cross section. The question, however, is whether QIA is an effective and accurate tool when used by a variety of researchers. Are the % porosity measurements developed by one research group equal to values determined by another? Without good measurement reproducibility the method will not be useful for verifying the output from the computer model. Therefore, this study was undertaken as a cooperative effort between Concurrent Technologies Corporation and the PMPA Standards Committee to determine the applicability of using QIA for determining the % porosity distribution in P/M compacts. Since process modeling provides more robust tool designs, more uniform mechanical properties and reduced design/development costs, the P/M industry must a have a low cost, accurate means to confirm the output of these computer simulation models.

EXPERIMENTAL PROCEDURE

A group of P/M parts producers, powder manufacturers and interested outside parties volunteered to participate in this study (see Appendix). A statistically meaningful number of companies was necessary in order to yield acceptable results. The low number of participants (only 10) was indicative of the number of companies in the industry with QIA equipment.

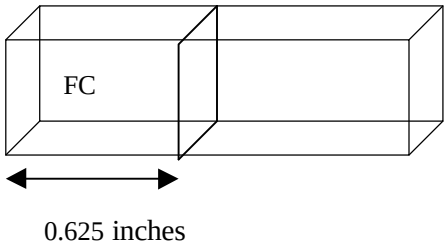
Two sets of standard transverse rupture bar test specimens were prepared by Powder-Tech Associates, one using FC-0208 and one using SS-316L. The stainless alloy represented a softer material, which tend to be more difficult to prepare metallographically and yield an accurate rendition of the open porosity. The FC-0208 was pressed to a nominal 6.7 g/cm^3 density while the stainless steel samples to a 6.4 g/cm^3 density. Sufficient test specimens were prepared so that each participant received one sample from each material. While this approach introduced some variation into the study (since even under the best of laboratory conditions the ten specimens will not be exactly alike) we believed it was most expeditious to send each participant a specimen rather than wait for the participants to transfer a single specimen from lab to lab.

A standard test method protocol was developed and sent to each participant with the test specimen. The test results are identified exclusively by a unique participant ID number in order to maintain participant confidentiality. The data was analyzed using the ASTM E 691 Procedure for Interlab Study of Test Method Precision.

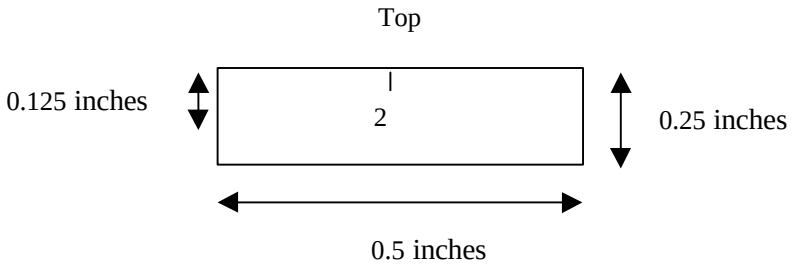
TEST METHOD PROTOCOL

1. Prepare each test sample for mount and polish. If you normally impregnate with resin or epoxy please follow that procedure. It is recommended that the samples be impregnated to help retain pore shape during polishing.

2. Section and mount the test samples so that the "top" is known for orientation. Sectioning should be as close to the midpoint of the 1.25 inches length as possible. Mount the sectioned sample so the freshly cut 0.500 inches x 0.250 inches face is prepared for evaluation—see below:



3. Identify two locations on the 0.500 inches x 0.250 inches face for porosity measurements, as shown below. Point 1 is 0.020 inches down from the "top" surface and 0.250 inches from the left surface. Point 2 is 0.125 inches down from the "top" surface and also 0.250 inches from the left surface.



4. Measure the porosity at Point 1 and Point 2 using a magnification in the 100-200x range. Three different readings must be taken by the same operator. Reposition the stage and re-set the threshold, contrast, magnification, focus, etc. between the three readings.

5. Repeat the three measurements for both test samples. Record your results on the enclosed data form. Retain the two test samples in case we need to re-check any specific lab's measurements.

RESULTS AND DATA ANALYSIS

A summary of test results is listed in Table 1. All data was rounded to one decimal point accuracy from the original results supplied by the laboratories for consistency of data reporting. The data identified as Position #1 represents the results measured at the 0.020 inches location for both the FC-0208 (Sample #1) and the SS-316L (Sample #2). The data listed under the Position #2 designation are the results measured at the mid point (0.125 inches location) for both materials. Statistical analyses were run for both data sets with the results summarized in Table 2.

The statistical analysis finds no significant difference between the results of Position #1 and Position #2. For these two materials, no measurable porosity difference was found from the surface to the interior position.

The data from these ten laboratories falls within a 95% confidence band with only one laboratory (Lab #7) having a marginal outlier for Position #2 data, as illustrated in the two graphs (Figures 1 and 2). Even though there seems to be a large data range among these ten labs, this does not imply an error by any specific lab (either high or low) but rather the inherent variation in this test method. Note that the results for FC-0208 ranged from a low of 10.4 % to a high of 18.0% or a spread of 7.6%. In contrast the SS-

316L sample ranged from 14.1% to 24.0% a spread of 9.9%. As expected the softer material apparently was more difficult to polish consistently and therefore showed a greater variation. The information requested in the original data submission included whether the test specimen was resin impregnated for specimen preparation, whether the lab had an automated stage on their microscope and the magnification used. It was believed that perhaps one or more of these conditions might help explain any unusual results. As noted below (see Table 3) none of these three conditions appears to have a meaningful influence on the results, i.e. one condition always producing a lower porosity value.

Table 1
Position #1 Image Analysis Results

Lab #	Sample #1			Sample #2		
1	11.0	11.0	10.4	15.5	15.6	15.9
2	11.6	14.2	12.8	14.0	14.3	17.6
3	15.8	12.7	13.4	20.1	20.5	20.0
4	16.5	17.0	16.7	21.3	20.8	21.0
5	17.3	17.2	16.2	20.5	20.8	20.5
6	12.3	12.3	12.3	14.2	14.7	14.2
7	16.8	16.9	16.7	15.8	14.8	15.8
8	17.4	18.0	17.2	22.4	21.3	20.4
9	15.5	16.5	15.4	19.2	21.1	17.8
10	14.0	10.3	11.6	16.5	15.6	18.4

Position #2 Image Analysis Results

Lab #	Sample #1			Sample #2		
1	11.8	11.7	11.7	16.2	16.2	16.3
2	11.6	14.3	14.4	13.2	16.9	16.5
3	14.6	15.6	15.9	18.6	20.0	17.6
4	16.6	16.7	16.5	23.9	24.0	24.0
5	13.6	14.7	13.8	18.9	19.0	20.4
6	16.0	16.6	16.7	15.6	15.3	15.8
7	17.8	17.8	17.5	14.1	14.3	14.9
8	18.0	18.1	17.8	20.5	20.5	21.6
9	17.4	18.1	14.8	17.7	17.5	18.0
10	13.2	14.6	12.4	21.4	19.1	19.5

Table 2
Summary of Statistical Results
Position #1

Materials	Average Porosity, %	r, (repeatability), %	R, (reproducibility), %
FC-0208	14.6	2.7	7.2
SS-316L	18.0	2.9	8.1

Position #2

Materials	Average Porosity, %	r, (repeatability), %	R, (reproducibility), %
FC-0208	15.3	2.5	6.2
SS-316L	18.2	2.6	8.4

Notes:
r, (repeatability) is the within laboratory variation allowed for porosity to maintain a 95% confidence in the results.
R, (reproducibility) is the variation among the laboratories allowed for porosity to maintain a 95% confidence in the results.

Figure1

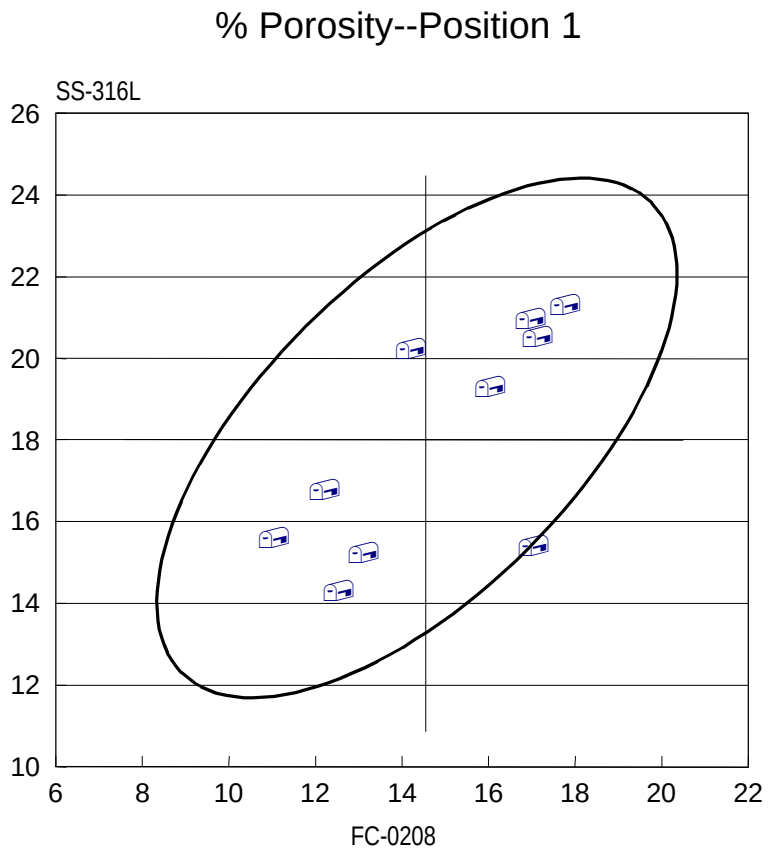


Figure 2

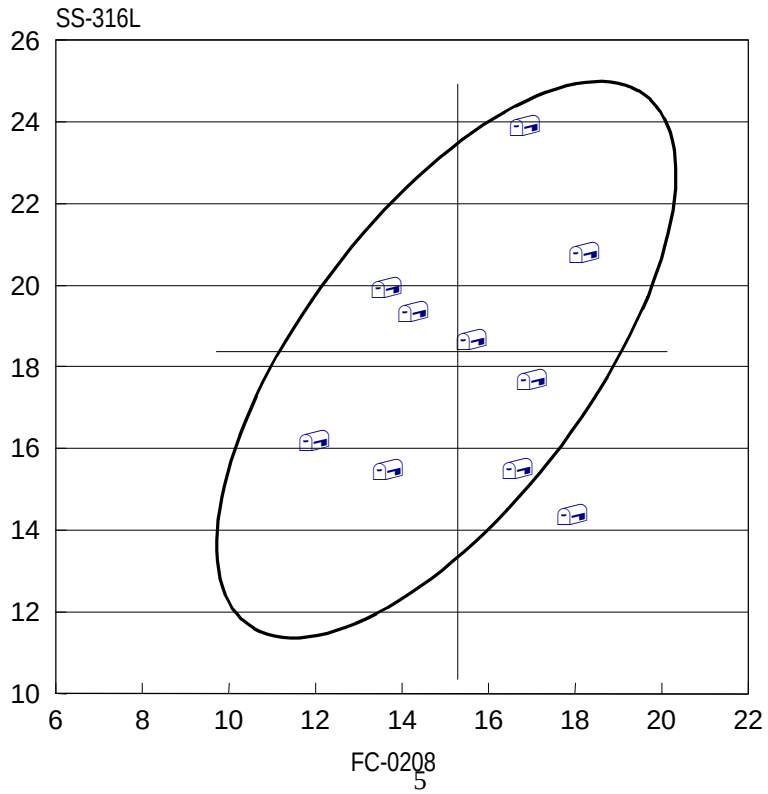


Table 3
Effect of Preparation Parameters

Lab #	Impregnated	Automated Stage	Magnification	Deviation from Mean Value	
				FC-0208	SS-316L
1	Yes	No	100	Neg	Neg
2	No	No	100	Neg	Neg
3	No	No	200	Neg	Pos
4	No	Yes	200	Pos	Pos
5	Yes	Yes	200	Pos	Pos
6	No	Yes	200	Neg	Neg
7	Yes	Yes	100	Pos	Neg
8	No	No	150	Pos	Pos
9	Yes	Yes	200	Pos	Pos
10	Yes	Yes	200	Neg	Pos

DISCUSSION OF TEST RESULTS

The results of this interlab study find the image analysis test method just slightly less reliable than the immersion technique (ASTM B 328) for repeatability (r value of 2% for porosity per ASTM B 328 vs 2.7% average from this study). This means that within lab variation is nearly the same for both test methods. A much greater deviation was found in the reproducibility, i.e., the variation seen between the labs (R value of 4.0% for porosity per ASTM B 328 vs 7.5% average from this study). This can be interpreted to mean that there are meaningful differences in the test methods being used between the ten labs, or, the skills are different, or, the test specimens were different (the local variation in density is much greater than the overall average density variation). Another factor in this larger variation between labs is the lack of a "calibration standard" that everyone can use to ensure their preparation method and instrument are reading correctly, such as the verification test block used with Rockwell hardness testers.

One of the labs with an automated stage also measured the average porosity over the entire specimen. These results found less than 0.5% deviation from the average of their six individual readings for each material. This again confirms that for these two materials and the 0.25 inches thickness of the transverse rupture specimen, the local variation in porosity is quite small. For those laboratories with a continued interest in image analysis techniques the development a transverse rupture size test sample used to calibrate the method and equipment may be a useful tool.

One of the participants commented that sample preparation techniques were the key to consistent results. As noted above, this study included five labs that impregnated their samples and five that did not. Their results did not show impregnation as the key factor in reducing variation: for the FC-0208 samples three impregnated samples had a positive deviation from the mean porosity value and two had a negative deviation: for the SS-316L the results were the same. Therefore it does not appear that impregnation is the answer to consistency in determining porosity by the image analysis method. However, familiarity with the material under investigation may be an important influence for an acceptable preparation technique. Lab #7 with their marginal results was much less familiar with the SS-316L than the FC-0208. Thus, it appears that perhaps localized variation in porosity among ten different samples, familiarity with the material and the lack of a calibration standard may be the real issues in achieving good image analysis results.

DISCUSSION OF QIA TECHNIQUE

An additional study was performed by one of the co-authors (T.M.) to address some of the questions associated with detection level settings, microscope magnification, and data scatter. In this small study, the testing was separated into two groups. The first dealt with the effects of changing magnification and detection level and how the combination of a change in resolution and the detection of lighter gray levels affect the amount of detection. The second group looked at the field-to-field scatter at two magnifications with a fixed detection level.

With the magnification/detection level tests, the sampled areas came from the center portion of the cross-section. At each magnification, the automated stage pattern was 5 x 5 fields with each pattern starting at the same location. Consequently, areas tested were reduced as the magnification was raised. To compare areas, as the magnification is doubled, the field area is reduced to ¼. Taking this into account, the area measured at 200x was still large enough to average the field-to-field variation and provide an accurate estimate of the percent porosity. The threshold level was increased by a total of 20 points at each magnification. Testing was performed at five point gray scale increments.

With the field variation tests, the sampled areas came from a stripe taken across the center of the bar in the direction of the long dimension (0.5 inches). Starting points of each were the same. The area of the higher magnification traverse was half that of the lower because twice the number of fields were run at double the magnification. Both traverses were run at fixed detection levels. The amount of reflection from the sample surface was kept constant by adjusting the intensity of the lamp as the magnification was changed. The ability of the objective lens to collect light changes with magnification.

Magnification/Detection Level Tests

The amount of scatter around the mean, between the lowest and highest value fields, appears least at the lowest magnification (Figure 3) and becomes greater as the magnification is increased (Figures 4 and 5). This is probably due to the larger field area and smaller amount of field-to-field variation.

As would be expected, the increase in detection level correlates to an increase in the area detected. This can be observed as change in the slope of the individual area % threshold setting lines, of particular interest are the mean lines. The reason for this is that the gray value at the pore edge changes gradually as viewed from the pore interior to the metallic portion of the sample and as the threshold setting is increased (detecting lighter gray values), the amount of detection is also increased. The greatest increase on the mean line is at the lowest magnification (Figure 3) where the as-detected value increases from a low value of 15.59% to a high of 18.65%. This relates to a possible difference in relative density of 0.25 g/cm^3. The slope decreases as the magnification increases indicating less effect of the detection level increase as the resolution of the system is increased. At 200x, the as-detected differences ranges from 14.11% to 15.09%, a difference of <0.1 g/cm^3 in density (see Figure 5).

Figure 3

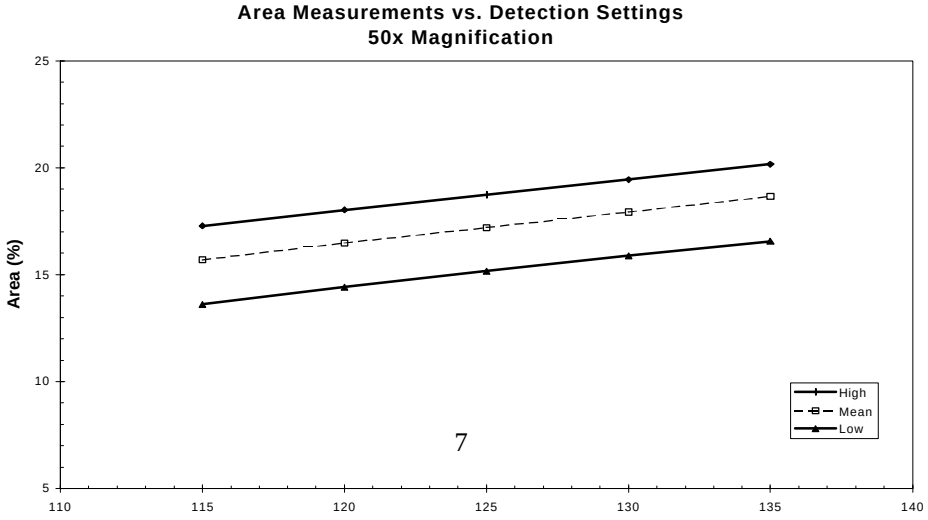


Figure 4

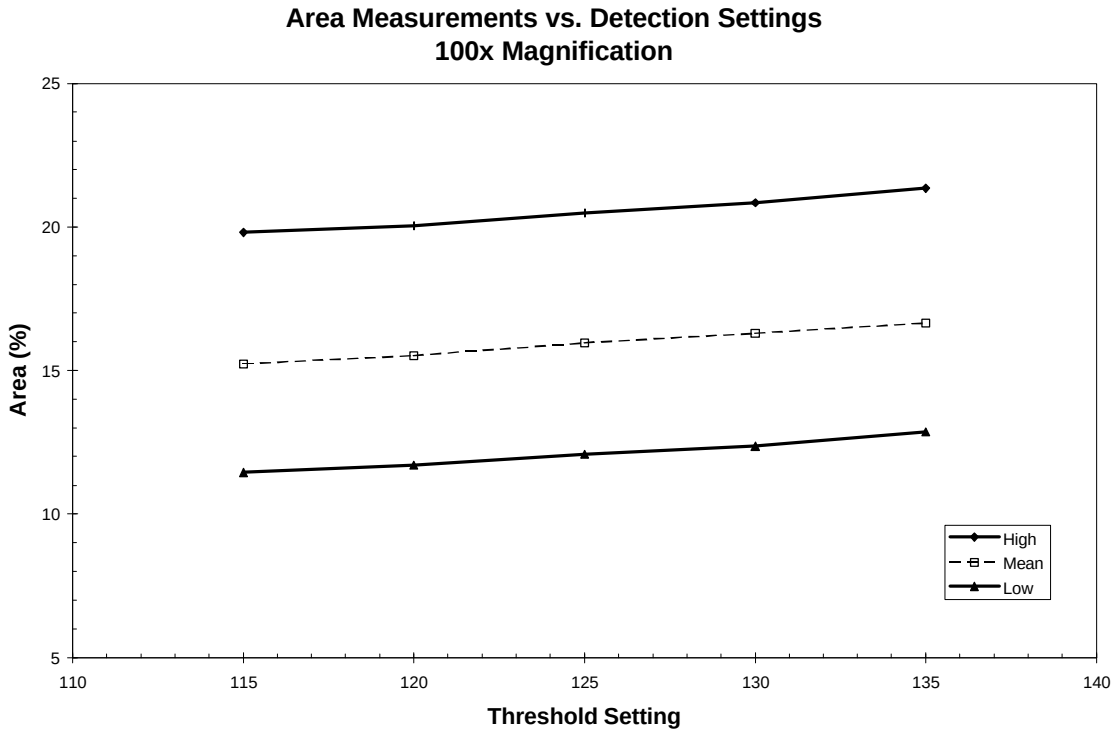
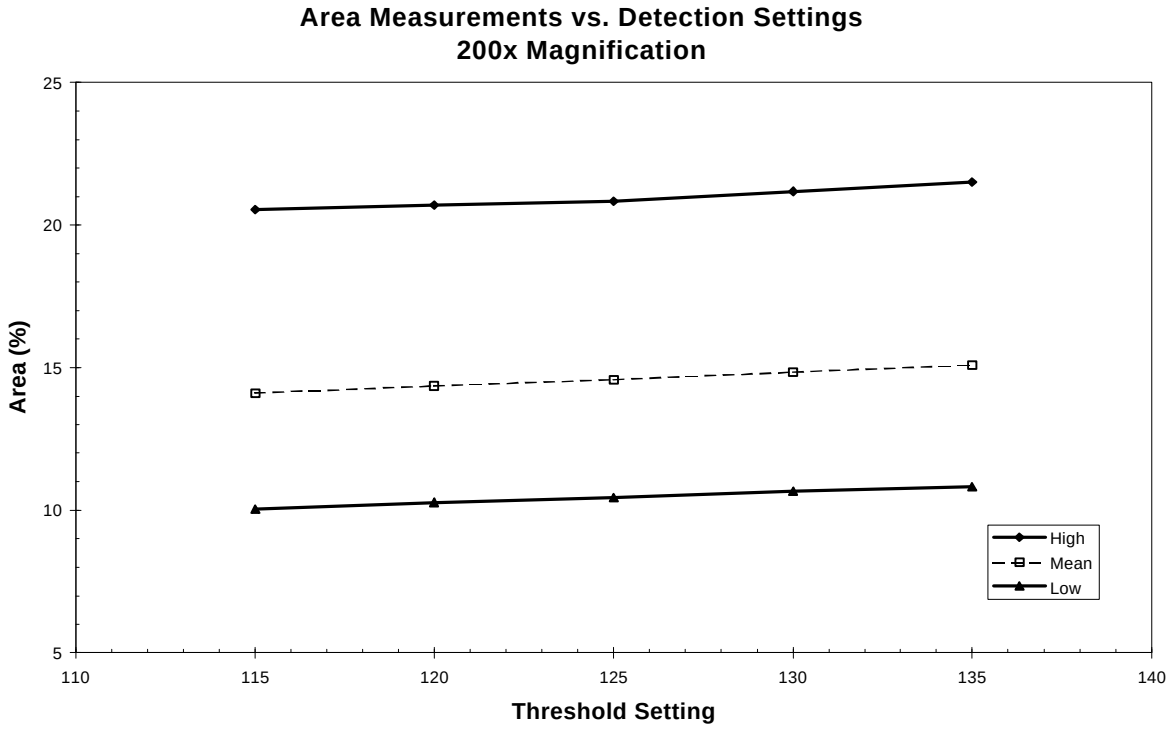


Figure 5



Field-to-field Variation Tests

The amount of noise, up and down movement in the graph around the mean, is much greater at the higher magnification (compare Figures 6 and 7). The effects of micro-density differences are very apparent, as the field area becomes smaller. At 200x, large differences can be observed in neighboring fields where the largest, between fields 31 and 32, is >8%. In contrast, the greatest neighboring fields difference at 100x is slightly more than 3%.

Figure 6

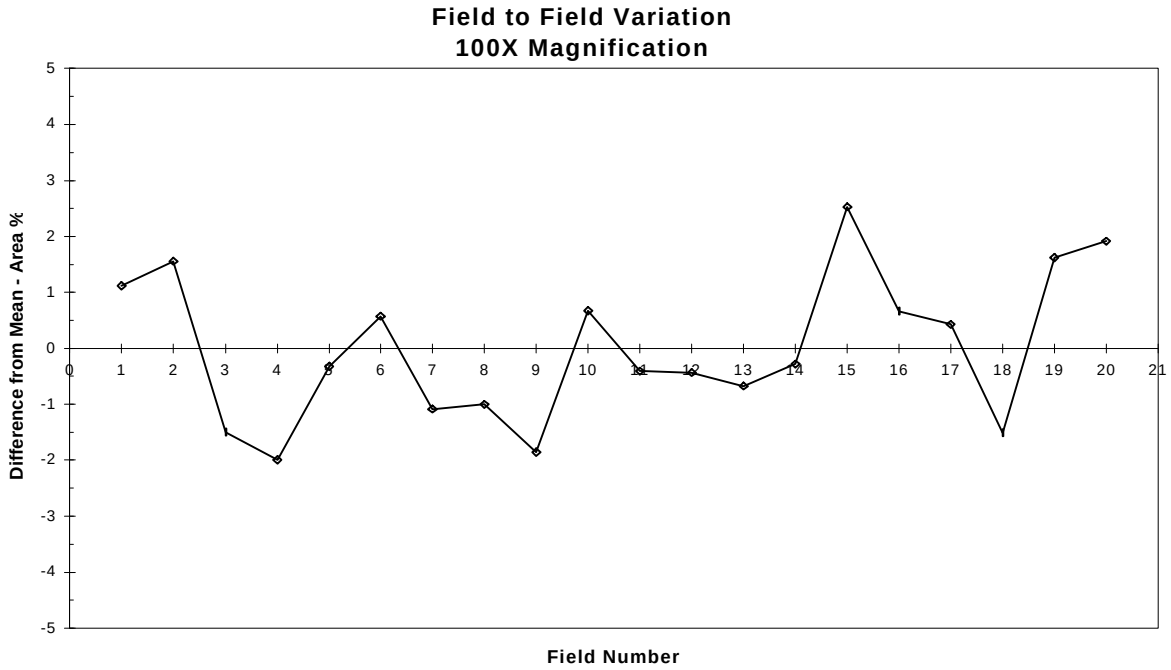
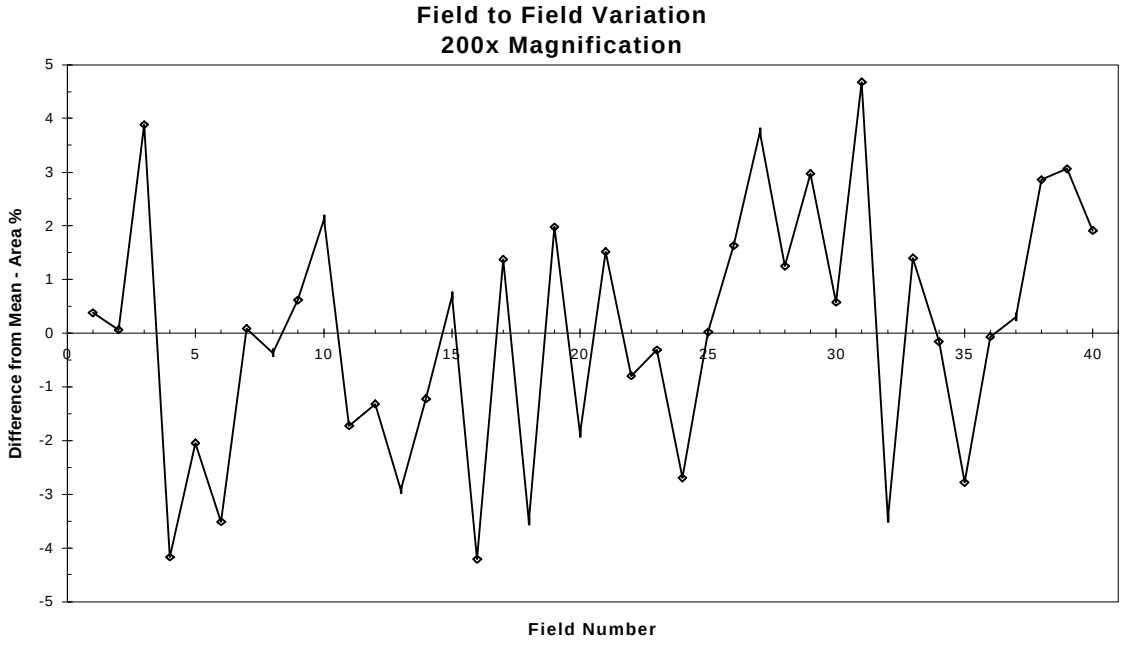


Figure 7



CONCLUSIONS

1. Image analysis methods can be used effectively to determine the local porosity in powder metal materials. The results are more consistent between labs when measuring a harder rather than a softer material.

2. The variation within a laboratory in determining porosity by image analysis is 2.7% (r, repeatability) while the variation between laboratories is 7.5% (R, reproducibility). The r value is comparable to bulk measurements, using ASTM B 328; the R value is nearly double the variation determined by bulk measurements.

3. A more consistent test method may be demonstrated if the same test specimen was circulated among the labs and a calibration sample was used to qualify the preparation technique and equipment.

4. The large variation in results between laboratories was not due to the use of resin impregnation, an automated stage or different magnifications in the 100-200X range.

5. The variables of detection level setting and microscope magnification appear to be the controlling QIA factors in accurate percent porosity measurements when all other factors such as sample preparation and calibration of the image analysis system are correct.

6. The effects of detection level setting are more severe at lower magnifications, but the field-to-field variation is less. Conversely, a higher magnification is more forgiving to minor detection level adjustments, but more fields must be run to compensate for the greater field-to-field variation.

7. When a higher magnification is used, the time to perform an analysis is greater because more fields need to be measured to provide information for the same sample area.

APPENDIX—LIST OF INTERLAB STUDY PARTICIPANTS

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