Measurement of the Pearlite Content in Ductile Iron Microstructures

AFS Ductile Iron Division
Quality Control Committee (12-E)

ABSTRACT
Different methods of making an exact determination of the percentage of pearlite present in a microstructure were compared by members of the AFS Ductile Iron Quality Control Committee. The point count method was found to offer an acceptable level of accuracy while requiring a minimum expenditure in time and equipment. Further work is planned to develop a set of reference photomicrographs as an aid in making rapid visual comparisons.

In many applications some other method of evaluation will better serve the purpose described. This might be a hardness test or some type of service test measuring hardenability, machinability or strength under actual conditions of use and use. Such tests overcome some of the difficulties mentioned above. Also, they can often predict the performance to be expected of a casting in service on a direct basis, rather than inferentially as is the case when examining microstructure.

However, recognizing the limitations listed and adjusting test procedures to minimize them, the examination of microstructure still remains a well-accepted and popular method of determining the properties of a casting or group of castings.

In fact, certain service properties are still best controlled by placing limitations on the percentage of a given constituent in the microstructure. These might include a maximum level on ferrite to ensure good response to surface hardening, a maximum level on pearlite to reduce casting growth at elevated temperatures or a maximum level on pearlite to prevent brittle fracture at reduced temperatures.

Investigation—Recognizing the fact that the number of specifications containing a microstructural specification are increasing, but hopeful that their application could be made less of a problem, the Quality Control Committee set out to evaluate the different methods of measuring, rather than estimating, the percentage of pearlite present in a ductile iron microstructure. The methods considered were:

Weight Method—in which a photomicrograph of the structure is enlarged to a common 8 x 10 in. The different constituent areas are outlined with a pencil or other marker and a small manicure scissors used to cut the different areas out of the photomicrograph. All areas representing each constituent are weighed on a laboratory balance and percentages are then calculated.

Planimeter Method—in which an instrument, commonly used to measure irregularly shaped areas in topography, thermodynamics, and other fields, is used to measure similar areas on an enlarged photomicrograph. Design of this instrument is based on the principle of a time-area by integration of linear dimensions. Different constituent areas in the photomicrograph are outlined and the stylus of the instrument is moved to follow the outline of each area of interest in the constituent. The area is then marked as counted, the instrument reading recorded and the instrument zeroed to begin measuring the next area. After all areas have been measured, instrument readings are totalled and their percentage of the total area calculated.

Point Count Method—in which a photomicrograph of the structure is enlarged to a common 8 x 10 in. The different constituent areas are outlined with a pencil or other marker. A grid of evenly placed lines is placed on the photomicrograph. The number of grid line intersections that fall on the constituent areas of interest are counted. Then their percentage of the total number of points is calculated.

Improved speed and reproducibility can be achieved with this method by construction of a transparent plastic template with the grid of lines drawn on the plastic sheet. The template can then be laid on the photomicrograph and an ink marker used to indicate each intersection counted. In this way intersections will not be missed or counted twice, a count can be interrupted when necessary and there is no need to keep track of the count figure in the observer's head as he proceeds. A small manual counter may be useful for keeping the count figure intact during the counting process. After all the intersections have been marked and counted, a percentage can be calculated and the instrument readings are drawn at 1/4 in. intervals. (See insert.)

Line Intercept Method—in which a number of evenly spaced calibrated lines are superimposed on the microstructure. The number of calibration units that fall on areas of the constituent in question are counted. Then the percentage of the total number of calibration units is calculated.

A count is usually made with the microscope directly on the polished specimen without the production of a photomicrograph. The structure is slowly scanned at a constant rate along one axis, with the observer operating some sort of counting device whenever the scanning point is crossing an area of the constituent in question. Several parallel scans are made at evenly spaced intervals and a percentage of the total travel is calculated.

This method was considered but not included in the comparisons carried out in this investigation. It was recognized that such measurement would be quite time consuming and would require microscopic equipment not available in most laboratories.

An alternate approach, using a photomicrograph and a visible set of lines is also considered acceptable. Using this approach, speed comparable to the point count method could probably be achieved by construction of a transparent template, similar to that described above. In this case, the template would contain a set of evenly spaced parallel lines and in some manner uniformly calibrated along its length. A count would be made of the number of
calibration units falling on areas of the constituent in question and a percentage calculated.

In this case, accuracy would depend on the proper choice of calibration lengths and line spacings. However, since the mechanics of this method did not seem to offer advantages over the point count method, no attempt was made to evaluate the effect of these two variables.

If a metalograph with the capability of projecting an image of the structure on a ground glass screen is available, both point count and line intercept methods can be carried out by placing the transparent template on this screen. This technique allows the observer to make a count at each of several locations on a specimen. These can then be averaged and greater coverage achieved in less time than would be required for the printing of several photomicrographs. Of course, using this approach a permanent record is not kept of the areas measured.

Automatic Scanning Method—in which the specimen or photomicrograph is systematically scanned to evaluate several hundred thousand individual points. The intensity of light at each point is measured as an indication of whether that point should be identified as one constituent or another. Signals from each point go to a some type of computer where they are tabulated and percentages calculated. Such systems require considerable initial investment but offer great speed and exact reproducibility of results. However, their ability to identify areas as pearlite or ferrite is controlled by the judgment of the operator who determines the intensity levels at which differentiation between the two structures will be made.

A problem arises too in the interpretation of the microstructure of the various cast irons. In many cases, the light intensity levels from pearlite and graphite are so similar that it is difficult to distinguish between them. One technique often used is to scan the unetched specimen first to determine the graphite content, then etch the specimen and scan again. The results of the second scan are subtracted from those of the first to determine the pearlite content in the structure.

When using any of these methods, the practice of outlining all pearlite areas before beginning to measure them seems to be a matter of personal preference. Those who favor the practice claim that it allows the observer to concentrate on the two distinct requirements of identification and measurement separately, rather than simultaneously, resulting in both greater accuracy and greater speed. If the results of a measurement are to be subject to review by a second observer at a later date, outlining the areas measured, for the purpose of reference, would seem to be a wise precaution.

Comparison Of Methods—Six photomicrographs, exhibiting different levels of pearlite content, were chosen for measurement. Each photomicrograph was enlarged to 6 x 10 in. The areas considered by one observer to be pearlite were outlined on each photograph. Then these marked photographs were measured by the weight and point count methods. Two structures were also cross-checked by the planimeter method. The original photomicrographs are shown in Fig 1–6. The marked enlargements of these photomicrographs are shown in Fig 7–12. Values obtained by the three methods of measurement are shown in Table 1. It can be seen that the deviation in percentage reported by the different methods for a given structure was never greater than one and one-quarter percent.

The weight method is considered by some investigators the most accurate of the three methods. However, it is time consuming and tedious—requiring a typical two hours of cutting to measure the pearlite content in a single structure. If one photomicrograph is to be held as a record of the areas considered to be pearlite, two of the same structure are outlined. Finally, this method requires possession of a balance capable of measuring in fractions of a gram, an item not found in every quality control laboratory.
Table 1. Pearlite percentage in structure, measured by different methods on marked photomicrographs.

<table>
<thead>
<tr>
<th>Microstructure</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight Method</td>
<td>15.6</td>
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<td>7.2</td>
<td>18.5</td>
<td>13.4</td>
<td>25.3</td>
</tr>
<tr>
<td>Point Count</td>
<td>15.4</td>
<td>3.9</td>
<td>8.1</td>
<td>18.8</td>
<td>13.5</td>
<td>26.2</td>
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<tr>
<td>Planimeter</td>
<td>16.6</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>25.0</td>
</tr>
</tbody>
</table>

The planimeter method should logically be as accurate as the weight method with time required to make a measurement in the order of 20 to 30 min per structure. Proper technique for operation of the instrument can be easily learned.

The point count method was in good agreement with the weight method in this series of comparisons. A point count can be completed in 5 or 10 min and the only equipment required is the template described above and a felt tip ink marker.

Variation Between Observers—In another comparison, the point count method was used by several observers to measure the percent pearlite in each of the structures previously chosen. However, in this case, the identification of pearlite areas was left to the judgment of the individual observers, instead of marked in advance by one party. Values reported by the different observers are shown in Table 2.

The wide variation between certain of these measurements on the same structure points out a problem that will always remain, regardless of the measurement method used. That is the original determination on the part of the observer of what areas he considers to be pearlite. Differences in interpretation between two observers—for example, customer and supplier—must be resolved before agreement can be reached on measured percentages. Outlining the pearlite areas before beginning a point count, then holding the marked photomicrograph for future reference, can help to bring such differences to light.

From data in Table 2, the mean value reported for each structure was calculated. Also, the confidence limits of this mean at the 95% confidence level. This figure suggests where the mean might be expected to fall, if a much larger number of observers were surveyed. These figures are listed in Table 3. The table also contains the standard deviation for each set of data, to indicate the variation of individual observations about each mean. Thus, the mean value reported for microstructure A by a larger number of observers would be expected to fall between 18.5 and 20.3% (19.39 ± 0.95). However, values reported by 95% of the individual observers could only be expected to fall within a wider range of two standard deviations of the mean; that is, between 13.9 and 24.9%.

Table 2. Pearlite percentage in structure measured by different observers on unmarked photomicrographs using point count method.

<table>
<thead>
<tr>
<th>Microstructure</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
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<td>7.0</td>
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<td></td>
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<td>10.3</td>
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<td></td>
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<td>7.9</td>
<td>17.5</td>
<td>11.3</td>
</tr>
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</table>

*observer declined to identify pearlite in this structure because of the poor quality of photomicrograph.
Effect of Metallographic Technique—As individual observers reported the percentages they had determined on the photomicrographs sent to them, it became obvious that the quality of the metallographic and photographic techniques used to produce these photomicrographs could be seriously questioned. Comments from different observers were:

"I found it difficult to achieve satisfactory identification of pearlite areas in structure E. The pearlite in this structure appeared fuzzy, as though excessive nickel or manganese might have resulted in a poor response to anneal. Structure B posed similar problems but was more typical of a commercial ferritic iron."

"Interpretation of structure E difficult, due to poor clarity of photomicrograph."

"Some of the photomicrographs were difficult to interpret. If pearlite is to be determined on an exact basis, I suggest that in the preparation of future specimens the final procedures include at least two light etching and polishing steps before final etch, in order to remove all disturbed metal, which might mask proper identification from a picture."

Table 3. Mean value and standard deviation, pearlite percentages reported by different observers.

<table>
<thead>
<tr>
<th>Microstructure</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
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</thead>
<tbody>
<tr>
<td>mean of reported values</td>
<td>19.39</td>
<td>3.91</td>
<td>7.78</td>
<td>17.06</td>
<td>11.25</td>
<td>25.31</td>
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<tr>
<td>confidence limits</td>
<td>0.95</td>
<td>0.34</td>
<td>0.36</td>
<td>0.83</td>
<td>0.97</td>
<td>0.82</td>
</tr>
<tr>
<td>standard deviation</td>
<td>2.76</td>
<td>0.98</td>
<td>1.05</td>
<td>2.42</td>
<td>2.77</td>
<td>2.38</td>
</tr>
</tbody>
</table>

"The quality of the photomicrographs supplied was not as good as desired for this type of study. I would expect this factor to contribute to the scatter in the results from the individuals participating in the study."

"Photomicrograph E has too much contrast and some of the graphite shapes are degenerate. These factors make it impossible to tell pearlite from graphite, in many areas."

"Samples A and E are rather poor from a photographic viewpoint. I question the use of these samples for anything other than a preliminary report on this procedure."

Rebuttal has been made by some committee members that these photomicrographs are representative of metallographic work produced throughout the industry — whether by customer, supplier or commercial laboratory. However, they agree that photomicrographs of a higher level of quality should be obtained for publication as AFS reference photomicrographs.

Enlargement Of Photomicrographs—The need for enlargement of standard size photomicrographs to the larger 8 x 10 in. size for these purposes might be questioned by some readers because of the resultant increased cost. However, such enlargement reduces the level of inaccuracy caused by limitations in technique or equipment. For instance, with the weight method, the technologist’s inability to trim pearlite areas with complete accuracy could have a greater effect on the final result, if he were cutting out very small pieces from a small photograph instead of larger pieces from a large photograph. With any method, variations in the width of lines drawn with a felt tip marker on a small photograph might affect the result reported. Outlining areas and counting points, when extremely small areas are involved, can cause problems of mental concentration and eye strain.

The accuracy of the point count method varies with the total number of points used. To have the number of points on a 4 x 5 in. photograph equal the number present on an 8 x 10 photograph, where a ½ in. grid spacing is used, would require a 1/8 in. grid spacing on the smaller photograph. Again eye strain would be a problem.

If the wider spacing was maintained on the smaller photograph, only about one fourth as many points of line intersection would be produced. Such a reduced number of points could also be produced by using a ½ in. grid spacing on an 8 x 10 in. photograph. A comparison between this approach and the original ¼ in. spacing was carried out, producing the results shown in Table 4. Variations as great as 4% pearlite were found when the reduced number of points was used.

Percent of Matrix—The percentages included in this report up to this point have all been the percentage of pearlite contained in the complete structure. Standard practice in more strictly scientific circles is to report the percentage of pearlite present in the matrix alone, excluding the presence of free graphite. When visually estimating percentages such a requirement tends to confuse the problem, but when exact measurement methods are used the amount of additional work involved depends on the method used.

In general, the time required to measure percentage in the matrix will be twice that required to measure percentage in the structure. Since the point count method is the fastest of the three methods under consideration, its convenience will be magnified when measurement of the percent pearlite in the matrix must be made. It is only necessary to make a second count of the points that fall on graphite areas and then subtract these from the total number of points in the structure before dividing to get a percentage figure.

Measurement of percent pearlite in the matrix and percent free graphite were made on the structures originally shown, using both the weight and point count methods. Values obtained are shown in Table 5.
<table>
<thead>
<tr>
<th>Microstructure</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spacing ¾ in.</td>
<td>15.4</td>
<td>3.9</td>
<td>8.1</td>
<td>18.8</td>
<td>13.5</td>
<td>26.2</td>
</tr>
<tr>
<td>Spacing ½ in.</td>
<td>19.6</td>
<td>3.6</td>
<td>7.5</td>
<td>18.9</td>
<td>12.1</td>
<td>26.1</td>
</tr>
</tbody>
</table>

**Conclusions**—Several methods of measuring rather than estimating the pearlite content of a ductile iron microstructure are available. In general, these methods offer comparable accuracy but vary widely in the expenditure of time and equipment. The point count method requires less time and equipment than any of the other methods considered. Therefore, it would seem to offer a practical tool whereby customer and supplier, having agreed on which areas in a given photomicrograph they are going to identify as pearlite, could sit down with two copies of the photograph and each arrive at a nearly identical figure for the pearlite content of the structure.

The members of the Committee feel that reference photomicrographs are necessary for rapid evaluation of structures in production. They look upon the point count method as a practical referee technique when particular questions concerning the accuracy of the reference photo method arise.

**Acknowledgment**—The Committee wishes to express its appreciation to the following organizations that made time, material and facilities available to carry out this investigation: American Cast Iron Pipe Co., John Deere & Co., H. P. Deuscher Co., Ductile Iron Society, Durrkon Co., Inc., Ford Motor Company, Grieve Foundries, Inc., Hamilton Foundry Division and Kuhns Bros. Co. The committee also wishes to gratefully acknowledge the guidance of Paul R. Gouwens, AFS Vice-President – Technology and Ezra L. Kotzin, Manager, Technical Services.

<table>
<thead>
<tr>
<th>Microstructure</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
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<td>pearlite-weight</td>
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<tr>
<td>pearlite-pt count</td>
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<tr>
<td>graphite-weight</td>
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<tr>
<td>graphite-pt count</td>
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</table>

**Future Plans**—This study should be looked upon as a progress report of the work done by the Committee to date. It is published for the purpose of promoting discussion as well as providing information.

In the near future the Committee will supply sets of unmarked photomicrographs to a greater number of observers for measurement by the point count method. It has been recognized by Committee members that agreement between several observers on a marked photomicrograph should be quite good. The point count technique is simple to understand and apply. Serious disagreement should only arise when different observers exercise their individual judgment, regarding the identity of the different constituents in the structure. By distributing unmarked photomicrographs, it is hoped to determine the magnitude of such disagreement for the structures in question. The final result of this study should be production of a set of reference photomicrographs. Each photomicrograph would be identified as exhibiting a certain most probable percentage of pearlite and the statistical deviation of this percentage stated.

Further, the Committee recently arranged to have the pearlite content of these structures measured by one of the optical scanning devices. Such data should provide another set of guidelines for establishing a most probable percentage figure for the structures under consideration.

**APPENDIX**

**Pearlite in Total Structure**

1. Using a felt tip marker with a fine point, outline all the areas on the photograph that you consider to be pearlite.
2. Center the grid layout of the template on the plate of the photograph. Because of differences in the way individual photomicrographs may be cut, this may not always be the same as lining up the outside edges of the template with the outside edges of the photograph.
3. Use paper clips, another device to keep the template from slipping while you are counting.
4. Count all points where a full intersection of two lines falls on a pearlitic area. Do not include the partial intersections of lines around the edge of the grid.
5. If a point falls on the edge of a pearlite area, count it as pearlite. Such points might only be counted at half value in a more exact method, but since there are a total of more than 1100 intersections on the grid, such a refinement in this case does not result in a significant increase in accuracy.
6. Mark all pearlitic points on the template with a felt tip marker, then go back and count them. In this way a line of points will not be skipped and you will not lose count if interrupted. After the count is complete the points can be removed from the template with a damp cloth.
7. The plates of some photographs may be smaller than the grid layout of the overlay. This will cause the total number of points falling on the plate to vary. The complete grid layout contains 30 intersections along the 8 in. dimension and 38 intersections along the 10 in. dimension for a total of 1140 points. Points not used, because they are off the plate or only on the edge of the plate, should be subtracted from this total.
8. Percent pearlite in the total structure is determined by dividing the number of points falling on pearlite areas by the total number of points, then multiplying by 100. For rapid approximation this can become a simple matter of dividing the number of pearlitic points counted by eleven.

**Note:** To determine percent pearlite in the matrix, the number of points falling on graphic areas must be counted and subtracted from the total number of points. Then the number of pearlitic points can be divided by this reduced total and again multiplied by 100.

**Calculations Involved in Determining Mean Values, Confidence Limits and Standard Deviations:**

<table>
<thead>
<tr>
<th>Term</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$x$</td>
<td>individual values reported</td>
</tr>
<tr>
<td>$Σx$</td>
<td>sum of individual values</td>
</tr>
<tr>
<td>$n$</td>
<td>number of observers</td>
</tr>
<tr>
<td>$x$</td>
<td>mean of values</td>
</tr>
<tr>
<td>$Σ(x-x)^2$</td>
<td>difference between individual values and mean</td>
</tr>
<tr>
<td>$L$</td>
<td>sum of differences squared</td>
</tr>
<tr>
<td>$t$</td>
<td>confidence limit of mean</td>
</tr>
<tr>
<td>$t = 2.634$</td>
<td>A constant value for 95% confidence</td>
</tr>
</tbody>
</table>

**Example (microstructure A)**

Mean $= \bar{x} = \frac{Σx}{n} = 47.6 \times 10^3 = 19.39$

Standard Deviation $= \sqrt{\frac{Σ(x-x)^2}{n-1}} = 2.76$

Confidence Limit $= L = t = (2.634 \times 2.76) = 0.95$