

Determination of Delta Ferrite Content

1. SCOPE:

This specification covers two methods for determining the percentage of delta ferrite in steels and other iron alloys.

2. APPLICABLE DOCUMENTS:

The issue of the following documents in effect on the date of the purchase order forms a part of this specification to the extent specified herein. The supplier may work to a subsequent revision of a document unless a specific document issue is specified. When the referenced document has been canceled and no superseding document has been specified, the last published issue of that document shall apply.

2.1 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM E 3 Preparation of Metallographic Specimens

3. TECHNICAL REQUIREMENTS:

3.1 Specimen Preparation:

3.1.1 Heat Qualification: Sampling shall be in accordance with 4.3.1. Samples shall be converted into test specimens in accordance with 3.1.3.

3.1.2 Product Qualification: Product from a heat not qualified, based on sampling as in 4.3.1, shall be sampled in accordance with 4.3.2. Samples shall be converted into test specimens in accordance with 3.1.3.

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- 3.1.3 Specimen Selection: Samples of sufficient size to provide specimens having approximately 0.25 to 0.5 square inch (161 to 323 mm²) prepared surface area or of an area sufficient to randomly select the required number of fields to accurately examine the sample, shall be cut from the product as in 3.1.3.1, 3.1.3.2, or 3.1.3.3, as applicable. Each specimen shall be marked in such a manner that its orientation with respect to the direction of rolling, drawing, or extruding is easily identifiable.
- 3.1.3.1 Round, Hexagonal, or Square Product: Specimens shall be selected from an area midway between the edge and the center of the sample.
- 3.1.3.2 Rectangular Product: Specimens shall be selected from an area midway between the longitudinal edge and the center of the sample.
- 3.1.3.3 Tubing and Other Hollow Shapes: Specimens shall be selected from samples which include the full wall thickness of the product.
- 3.1.4 Heat Treatment: Specimens, except as specified in 3.1.4.1, shall be austenitized at the normal recommended temperature for the grade of steel being examined and adequately quenched. The quenched specimens shall be tempered at a sufficiently high temperature to develop good metallographic contrast.
- 3.1.4.1 Solution and precipitation hardenable steels shall be solution and precipitation heat treated to develop good metallographic contrast.
- 3.1.5 Polishing: After heat treatment as in 3.1.4, a face of each specimen, perpendicular to the direction of maximum deformation, shall be ground and polished, using standard metallographic polishing techniques in accordance with ASTM E 3 to produce a surface suitable for microscopic examination.
- 3.1.6 Etching: The polished surface of each specimen shall be suitably etched to reveal delta ferrite.

3.2 Procedure:

Inspect the specimen surface at a magnification appropriate to observe the relative amount and distribution of delta ferrite present. If it is evident that many fields to be measured will have zero value, and no single field will have a value exceeding the material specification maximum, then measure a set number of fields defined in a laboratory procedure sufficient to ensure that the upper control limit of the mean is less than 50% of the material specification maximum at the 95% confidence level as in 3.2.1 or 3.2.2. A 10% relative accuracy level is not required. In case of dispute over the percentage of delta ferrite, the value determined as in 3.2.1 shall govern.

3.2.1 Point Count Method:

3.2.1.1 Field Measurement: Project the image of the microstructure of each specimen onto the ground-glass screen of a reflection-type microscope or metallograph. Place a transparent point-counting grid template either in front of or behind the ground glass. The template should consist of either 100 or 500 systematically spaced grid points, either in the form of fine crosses or a grid lattice. The magnification chosen should be high enough to clearly resolve the delta ferrite grains but not so high that the number of grid points falling in the ferrite grains exceeds one, on the average. For each specimen, count the number of grid points (i.e., the intersection point of the crosses or grid blocks) that fall within the ferrite grains as one and those that fall on the phase boundaries as one-half.

3.2.1.2 Field Selection: Select the fields to be measured blindly, that is, without looking at the image, by moving the stage controls. Do not alter the position so that grid points fall on or miss ferrite grains. Space the fields selected around the specimen surface in a systematic pattern. Measure a sufficient number of fields so that the relative accuracy (See 3.2.1.3.5) is 10% or less. The relative accuracy of the measurement is influenced by the volume fraction of the ferrite, the magnification, the grid point density, the number of fields measured, and the field-to-field variation of the ferrite phase.

3.2.1.3 Calculation:

3.2.1.3.1 Ferrite per Field: Calculate the volume fraction of ferrite using Equation 1 for each field measured:

$$V_{\delta,i} = \frac{P_{\delta,i}}{P_t} \times 100 \quad (\text{Eq. 1})$$

where:

$V_{\delta,i}$ = Volume fraction of ferrite in % of each field

$P_{\delta,i}$ = Number of grid points in the ferrite phase or on the phase boundaries

P_t = Number of grid points

3.2.1.3.2 Ferrite per Specimen: Determine the mean value of the volume fraction of ferrite using Equation 2:

$$\bar{V}_{\delta} = \frac{\sum V_{\delta,i}}{n} \quad (\text{Eq. 2})$$

where:

\bar{V}_{δ} = Mean volume fraction of ferrite per specimen

n = Number of fields measured in the specimen

- 3.2.1.3.3 Calculate the standard deviation of the field-to-field variation of the volume fraction of ferrite using Equation 3:

$$s = \left(\frac{\sum (V_{\delta,i} - \bar{V}_{\delta})^2}{n - 1} \right)^{1/2} \quad (\text{Eq. 3})$$

where:

s = Standard deviation of the volume fraction measurements

- 3.2.1.3.4 Calculate the 95% confidence limit (95% CI) of the volume fraction measurements using Equation 4:

$$\pm 95\% \text{ CI} = \pm 2.0 \frac{s}{(n)^{1/2}} \quad (\text{Eq. 4})$$

where:

2.0 is given as the "t" multiplier for purposes of simplicity. If desired, the exact "t" value for any value can be used instead of 2. Values for t, as a function of n, for a 95% confidence interval are tabulated in most statistical textbooks.

- 3.2.1.3.5 Calculate the percent relative accuracy (% RA) of the volume fraction measurement using Equation 5, if required (See 3.2):

$$\% \text{ RA} = \frac{95\% \text{ CI}}{\bar{V}_{\delta}} \times 100 \quad (\text{Eq. 5})$$

- 3.2.1.3.5.1 A 10% relative accuracy is adequate. If the calculated % RA is substantially higher, use the following formula to estimate the number of fields (n) required to obtain a 10% RA:

$$n = \left(\frac{20s}{\bar{V}_{\delta}} \right)^2 \quad (\text{Eq. 6})$$

- 3.2.1.3.6 Express the results as the volume fraction, $\bar{V}_{\delta} \pm$ the 95% CI and indicate the % RA; e.g.,

$$\bar{V}_{\delta} \pm 95\% \text{ CI} (\% \text{ RA})$$

Unless criterion of 3.2 for limited delta ferrite is met, in which case express result as $\bar{V}_{\delta} \pm 95\% \text{ CI}$ without % RA.

3.2.2 Image Analysis Method: Each specimen shall be etched so that only the ferrite phase is darkened preferentially, or everything but the ferrite phase is darkened preferentially. Choose a magnification on the image analysis monitor that permits accurate detection of the ferrite grains. Adjust the threshold setting to detect only the ferrite grains, using an appropriate method such as alternating between the live and the detected images ("flicker mode"). Use automatic stage movement, if available, to select the fields to be measured without bias. If such a system is not available, select the fields by moving the stage controls without viewing the image. Space the fields to be measured systematically around the specimen surface. Measure the ferrite volume fraction by dividing the number of detected picture points per field by the total number of picture points per field and express this value as a percentage. Automatic percentage readout may be used according to the equipment manufacturer's instructions. Measure enough fields per specimen so that the relative accuracy is 10% or less, unless the limited delta ferrite criterion of 3.2 is met.

3.2.2.1 Calculation: Calculate the volume fraction per field, as described in 3.2.2, and the mean volume fraction of ferrite, the standard deviation, the 95% confidence level, and the % relative accuracy, if required, as described in 3.2.1.3. Express the test results in the same manner as described in 3.2.1.3 as content of delta ferrite.

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection:

The vendor of the product shall supply all samples for vendor's tests and shall be responsible for the performance of all required tests. Purchaser reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the product conforms to specified requirements.

4.2 Classification of Tests:

All applicable requirements are acceptance tests and shall be performed to represent each lot.

4.2.1 Heat Qualification: Conformance to "heat qualification" requirements on samples as in 4.3.1, if acceptable, need be conducted only once per heat.

4.2.1.1 Heats which have been qualified as semi-finished product shall be considered qualified for finished product.

4.2.2 Product Qualification: Tests on product not "heat qualified" shall be conducted on product from each lot.

4.3 Sampling:

The sampling procedure described in 4.3.1 shall be performed by the producer for heat qualification. No further sampling by the producer shall be required from a heat which meets the requirements of the applicable material specification. Sampling procedure on product which lacks ingot or strand traceability shall be as described in 4.3.2.

4.3.1 Heat Qualification:

4.3.1.1 Heats of Top-Poured Ingots: Samples shall be taken from semi-finished or finished product representing the top and bottom of the first ingot and last usable ingot from heats having not more than 10 ingots or not over 30 tons (27,216 kg) or from portions of heats within these limits; and from the top and bottom of the first, middle, and last usable ingot of heats having more than 10 ingots or over 30 tons (27,216 kg).

4.3.1.2 Heats of Bottom-Poured Ingots: Samples shall be taken from semi-finished or finished product representing the top and bottom of three ingots. One ingot shall be taken at random from the first usable plate poured, one ingot at random from the usable plate poured nearest to the middle of the heat, and one ingot at random from the last usable plate poured. When a heat is constituted by two usable plates, one ingot shall be taken at random from each plate. When a heat consists of a single usable plate, any three random ingots may be selected.

4.3.1.2.1 If there are less than three ingots in the heat, samples shall be taken representing the top and bottom of all ingots.

4.3.1.3 Strand-Cast Heats: Samples shall be taken from semi-finished or finished product having at least a 3:1 reduction in cross-section from the cast strand (or samples of the cast strand similarly reduced) representing the front, middle, and back of both strands when two strands are cast, or of an inside strand and an outside strand when more than two strands are cast. When a single strand is cast, six samples having at least a 3:1 reduction from the cast strand (or samples of the cast strand similarly reduced) representing both ends of the first, middle, and last usable cuts (blooms) of the strand or product shall be taken.

4.3.2 Product Qualification: Three or more samples shall be taken at random from each lot; a lot shall be all product of the same size and shape from each heat in the shipment.

4.4 Reports:

The vendor of the product shall report the average percentage of delta ferrite (See 8.2) for each lot or heat, as applicable, provided no single specimen value was above the material specification maximum. The data supporting this statement shall be kept on file and available for review for the same period as other data supporting the specification requirements.