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INTERNATIONAL
400 Commonwealth Drive, Warrendale, PA 15096-0001

AEROSPACE MATERIAL SPECIFICATION

SAE AMS-2315 REV C

Issued 1963-01-16
Revised 1989-04-01

Superseding AMS-2315B

Submitted for recognition as an American National Standard

DETERMINATION OF DELTA FERRITE CONTENT

THIS REVISION CONTAINS ONLY EDITORIAL CHANGES.

1. SCOPE: This specification covers two methods for determining the percentage of delta ferrite in steels and other iron alloys in which control of delta ferrite is required to ensure that specified mechanical properties after heat treatment will be met.

2. APPLICABLE DOCUMENTS: The following publications form a part of this specification to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other documents shall be as specified in AMS-2350.

2.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096.

2.1.1 Aerospace Material Specifications:

AMS-2350 - Standard and Test Methods

2.2 ASTM Publications: Available from ASTM, 1916 Race Street, Philadelphia, PA 19103.

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.

3.1.3 Machining: Specimens of sufficient size, to provide approximately 0.25 to 0.5 square inch (161 to 323 mm^2) 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 spcimen 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 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. 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, the face of each specimen, perpendicular to the direction of rolling, shall be ground and polished, using standard metallographic polishing techniques in accordance with ASTM E3 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: The percentage of delta ferrite, the value determined as in 3.2.1, shall govern.

3.2.1 Point Count Method: 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. 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 sample surface in a systematic pattern. Measure a sufficient number of fields so that the relative accuracy (See 3.2.1.1.4) is 10% or less.) The relative accuracy of the measurement is influenced by the volume fraction of the ferrite, the magnification, the number of fields measured, and the field-to-field variation of the ferrite phase.

3.2.1.1 Calculation: Calculate the volume fraction of ferrite using the following equation for each field measured:

$$V_{V_{\delta,i}} = \frac{P_{\delta}}{P_t} \times 100$$

where:

$V_{V_{\delta,i}}$ = 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

3.2.1.1.1 Determine the mean value of the volume fraction of ferrite using the following formula:

$$V_{V_{\delta}} = \frac{V_{V_{\delta,i}}}{n}$$

where:

$V_{V_{\delta}}$ = Mean volume fraction of ferrite per specimen

n = Number of fields measured

3.2.1.1.2 Calculate the standard deviation of the field-to-field variation of the volume fraction of ferrite using the following formula:

$$s = \left[\frac{\left(V_{V_\delta , i} - V_{V_\delta} \right)^2}{n-1} \right]^{1/2}$$

where:

s = Standard deviation of the volume fraction measurements

3.2.1.1.3 Calculate the 95% confidence limit (95% CI) of the volume fraction measurements using the following formula:

$$95\% \text{ CI} = \pm 2.0^{*} \frac{S}{(n)1/2}$$

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.

3.2.1.1.4 Calculate the percent relative accuracy (% RA) of the volume fraction measurement using the following formula:

$$\% \text{ RA} = \frac{95\% \text{ CI}}{V_{V_\delta}} \times 100$$

3.2.1.1.4.1 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:

$$n = \left[\frac{(20s)}{V_{V_\delta}} \right]^2$$

3.2.1.1.5 Express the results as the volume fraction, V_{V_δ}, ± the 95% CI and indicate the % RA; e.g.,

V_{V_δ} ± 95% CI (% RA)

3.2.2 Image Analysis Method: Each sample 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. Alternate between the live image and the detected image ("flicker mode") to determine the correct threshold setting. 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. Measure enough fields per specimen so that the relative accuracy is 10% or less.

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 as described in 3.2.1.1. Express the test results in the same manner as described in 3.2.1.1 as content of delta ferrite.

3.2.3 The mean volume percent of delta ferrite shall be used to determine conformance to requirements. A value equal to or less than the specified requirement is deemed acceptable.

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 performing all required tests. Results of such tests shall be reported to the purchaser as required by 4.4. Purchaser reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the product conforms to the requirements of this specification.

4.2 Classification of Tests: Tests in accordance with all applicable requirements of this specification are classified as acceptance tests and shall be performed to represent each lot.

4.2.1 Heat Qualification: Tests to determine 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 in accordance with requirements of this specification 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 the product 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, two of the sample ingots shall be selected from the second usable plate poured. 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 as-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: Not less than three 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 include the percentage of free ferrite for each lot in addition to other information required by the applicable material specification.

4.5 Resampling and Retesting:

4.5.1 Product Other Than Slabs, Plates, Sheet, and Strip: If any specimen used in the above tests fails to meet the specified requirements, disposition of the heat or lot may be based on the results of testing three additional specimens for each original nonconforming specimen; additional specimens shall be as follows: