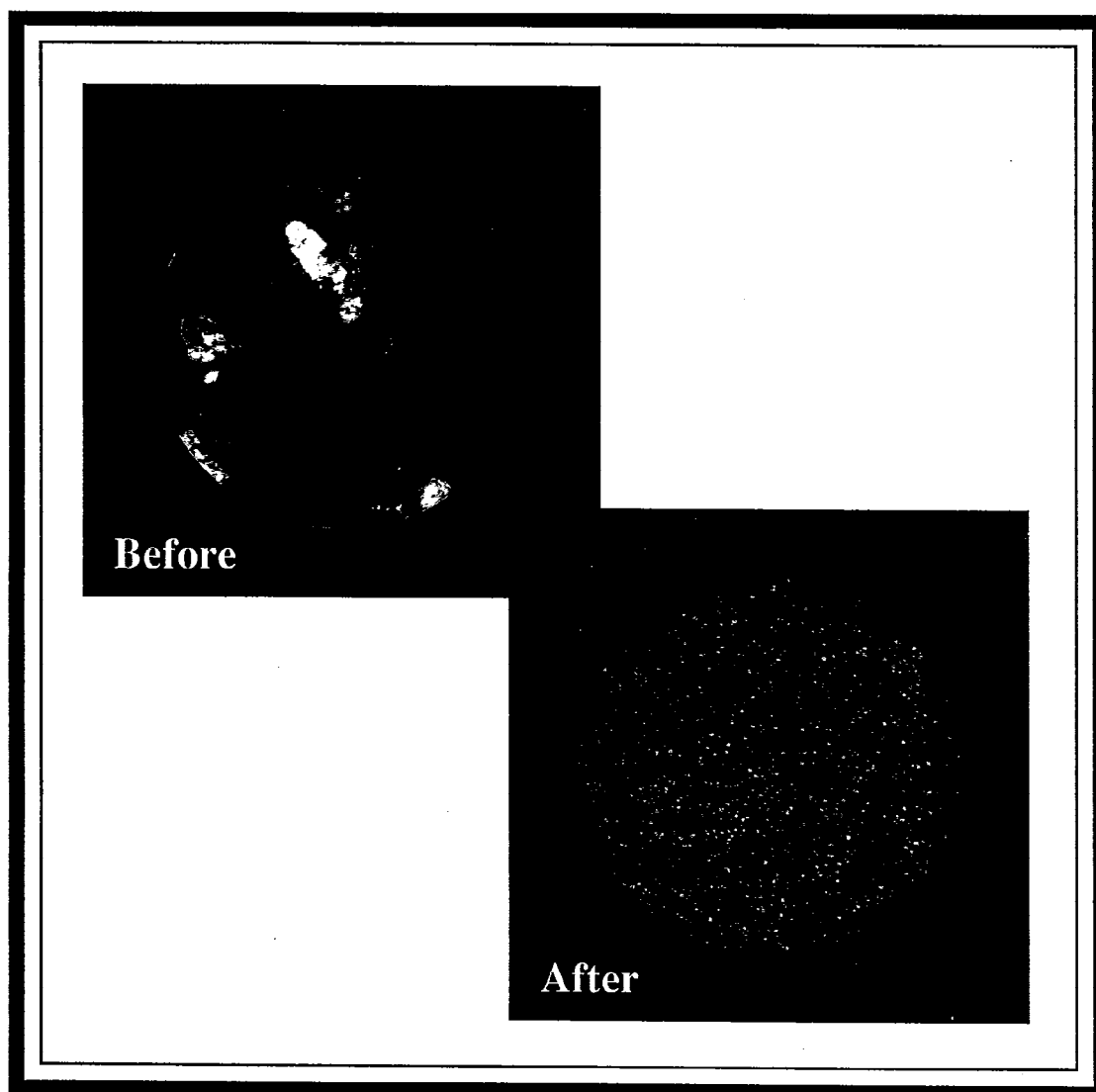


Standard Test Procedure for Aluminum Alloy Grain Refiners 1990

the Aluminum Association
incorporated

**TP-1**

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for Aluminum Alloy Grain Refiners

**\$10.00**

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**ALUMINUM ASSOCIATION
STANDARD TEST PROCEDURE FOR ALUMINUM ALLOY
GRAIN REFINERS**

1. **Scope:** This standard procedure is applicable for determining the ability of grain refiners to reduce grain size during solidification of aluminum alloys in specified compositions under standardized conditions, and for examining the structure of the grain refiner for uniformity and freedom from defects. This standard is not intended to predict the quantity of grain refiner needed in a specific alloy cast by any casting process or in a specific ingot size.
2. **Applicable Specifications and Registration Records**
 - 2.1 The following specifications or registration records (the issue in effect on date of material purchase) form a part of this standard test procedure:
 - Registration Record of Aluminum Association Designations and Chemical Composition Limits for Aluminum Hardeners
 - OSHA - 1910.1200 Hazard Communication Standard
 - Federal Test Method Standard No. 151 - Metals Methods 111.2 and 112.2
 - ASTM E112 Estimating the Average Grain Size
 - Metals Handbook^R Ninth Edition, Volume 9, Metallography and Microstructures Aluminum Alloys, p. 351
3. **Sampling**
 - 3.1 A sample for chemical analysis shall be taken from a randomly selected waffle ingot or coil of rod for each heat.
 - 3.1.1 For waffle ingot, the sample shall be comprised of drillings from the selected ingot. The drillings shall be obtained from opposite end segments and one centrally located segment and shall include equal weight of drill chips from top, bottom and middle of ingot. The drillings shall be mixed to form the sample.
 - 3.1.2 For rod, the sample shall consist of milled or clipped chips representative of the full cross section from at least two different non-adjacent pieces of rod.
 - 3.2 For grain refining test and metallographic examination, samples shall be selected as follows:

(2)

- 3.2.1 Waffle ingot - one randomly selected waffle ingot from a heat shall represent the heat. The sample for grain size test shall consist of one piece of the proper weight (Section 7.8), including top and bottom surface from the center portion of the ingot (Figure 1).
- 3.2.2 Rod - a length of rod shall be taken from each end of a coil to represent a heat. The sample for grain size test shall be of the required length, of full cross section to provide the weight required (Section 7.8). For metallographic examination, length of rod sufficient for longitudinal and transverse samples shall be provided (Figure 2). For mechanical property evaluation, a 12" (300 mm) length shall be provided.

4. Chemical Composition

- 4.1 The grain refiners shall conform to the chemical composition limits shown in Registration Record of Aluminum Association Designations and Chemical Composition for Aluminum Hardeners or the applicable purchase order.
- 4.2 The samples selected in accordance with section 3.1 shall be analyzed in accordance with Federal Test Method Standard No. 151.

5. Grain Size Test Description - The Aluminum Association grain refining test procedure can be used to evaluate the performance of grain refiners in rod or waffle ingot form. The test was selected to provide a sample piece with a range of cooling rates (Figure 3) that would duplicate the cooling rates found in Direct Chill (DC) cast ingots. The cooling rate at the test sample location selected is similar to the cooling rate expected in the center of a 7 inch (180 mm) diameter ingot.

6. Grain Size Test Material Supplies and Equipment

- 6.1 Supply of 99.7% pure virgin aluminum that has not been subjected to grain refining treatment with the following limits on impurities:

Silicon	.07% max.
Iron	.15-.20%
Iron to silicon	3 to 4.1
Titanium	.005 max.
Boron	.0005 max.
Others each	.03 max.
Total	.10 max.

Other aluminum alloys mutually acceptable may be used.

(3)

- 6.2 Electric resistance furnace capable of maintaining $1325 \pm 10^{\circ}\text{F}$ ($718 \pm 5^{\circ}\text{C}$)
 - 6.3 Clay-bonded graphite crucible, silicon-carbide crucible or cast-iron crucible, coated with a Dyecoat to avoid iron contamination; 24 lbs. (11 Kg.) capacity
 - 6.4 Calibrated and certified thermocouple and controller indicator capable of measuring temperature up to 1325°F (718°C)
 - 6.5 Two mold/ladles, coated with graphite spray coated only on the outside (Figure 4)
 - 6.6 Oven for warming the mold/ladles to 600°F (316°C)
 - 6.7 Sixty minute timer
 - 6.8 Graphite stirring rod
 - 6.9 Sample tongs
 - 6.10 Quench Tank (Figure 5)
 - 6.11 Protective clothing and face shield
 - 6.12 Notebook
 - 6.13 Water Flowmeter
 - 6.14 Thermometer
7. Procedure for Grain Refiner Test
- 7.1 Use new crucible or clean, used crucible with a wash heat of the aluminum alloy (Section 6.1). Weigh 22 lbs. (10 Kg.) of the aluminum alloy (Section 6.1), place in crucible, heat to $1325 \pm 10^{\circ}\text{F}$ ($718 \pm 5^{\circ}\text{C}$) and stabilize.
 - 7.2 Skim dross from surface with minimum disturbance of the liquid metal.
 - 7.3 Place mold/ladles in oven for preheating to 600°F (316°C).
 - 7.4 Take sample of alloy for confirmation of analysis.
 - 7.5 Turn on water in quench fixture so that water flow rate is one gallon (3.8 liter) per minute. If the water flow is correct, only the bottom one inch (25 mm) of the ladle will be submerged. Record water flow rate and temperature at beginning and end of each test.

(4)

- 7.6 Take sample of the aluminum (Section 6.1) to serve as a control standard for the grain refiner test in accordance with section 7.7.
- 7.7 Record melt temperature. A preheated mold/ladle is lowered beneath the surface of the melt, held for 30 seconds and withdrawn vertically from the melt to ensure that ladle is filled to the bottom of the notch. The filled ladle is lowered vertically into the retaining ring of the quench tank.
- 7.8 Add grain refiner alloy to the melt. The remaining metal weight is determined by adjusting the original weight to account for dross and chemical and control standard samples removed. The weight of grain refiner to be added is determined as follows:

$$\frac{(\% \text{ Ti or B desired}) \times (\text{Weight of Melt})}{\% \text{ Ti or B in Grain Refiner}} = \text{Weight of grain refiner to be added}$$

The grain refiner alloy is added to the melt at $1325 \pm 10^\circ\text{F}$ ($718 \pm 5^\circ\text{C}$) by stirring the alloy into the melt until it has been dissolved (30 seconds).

- 7.9 For ingot grain refiner, the melt is stirred with the graphite rod for 15 seconds only before the first sample is taken. For rod grain refiner, the melt is stirred with the graphite rod for 15 seconds before each sample is taken. The warmed molds/ladles are alternated for each sample taken. Samples are taken as in Section 7.7 above.
- 7.10 For ingot, samples are taken 30, 60 and 120 minutes after grain refiner sample was added. For rod, samples are taken 2, 5 and 10 minutes after grain refiner sample was added. Other times may be used by mutual agreement. The melt temperature is maintained at $1325 \pm 10^\circ\text{F}$ ($718 \pm 5^\circ\text{C}$) throughout the test. Each sample shall be identified and a record kept of residence time and melt temperature.
8. Preparation of Samples for Grain Size Measurement
- 8.1 A transverse section 1.5" (38 mm) from the base (as measured along the sloping side) is the standard plane for examination (Figure 6). To obtain this section the following steps are taken:
- 8.1.1 Measure 1.6" (40 mm) from the base along the side, cut the sample perpendicular to the axis of the casting. Identify this base section on the side with the sample number.