Designation: A 604 – 93 (Reapproved 2003)

Standard Test Method for Macroetch Testing of Consumable Electrode Remelted Steel Bars and Billets[1]

This standard is issued under the fixed designation A 604; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method[2] covers testing and inspection and is applicable to bars, billets, and blooms of carbon, alloy, and stainless steel which have been consumable electrode remelted.

1.2 For the purpose of this test method, the consumable electrode remelting process is defined as a steel refining method wherein single or multiple electrodes are remelted into a crucible producing an ingot which is superior to the original electrode by virtue of improved cleanliness or lower gas content or reduced chemical or nonmetallic segregation. See Appendix X1 and Appendix X2 for descriptions of applicable remelting processes.

1.3 This test method and the accompanying comparison macrographs[3] are generally applicable to steel bar and billet sizes up to 225 in.[2] in transverse cross section.

1.4 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 381 Method of Macroetch Testing Steel Bars, Billets, Blooms, and Forgings[4]

2.2 *ASTM Adjuncts:*

ADJA0604

Adjunct to A604 Test Method for Macroetch Testing of Consumable Electrode Remelted Steel Bars and Billet[3]

3. Description of Macroetch Testing

3.1 Macroetch testing, as described herein, is a method for examining and rating transverse sections of bars and billets to describe certain conditions of macro segregation which are often characteristic of consumable electrode remelted materials. This test method is not intended to define major defects such as those described by Method E 381.

3.2 This test method employs the action of an acid or other corrosive agent to develop the characteristics of a suitably prepared specimen. After etching, the sections are compared visually, or at a very low magnification, if necessary for clarification of conditions, to standard plates describing the various conditions which may be found. Materials react differently to etching reagents because of variations in chemical composition, method of manufacture, heat treatment, and many other variables.

4. Application

4.1 When material is furnished subject to macroetch testing and inspection under this test method, the manufacturer and purchaser should be in agreement concerning the following:

4.1.1 The stage of manufacture at which the test shall be conducted,

4.1.2 The number and location of the sections to be tested,

4.1.3 The condition and preparation of the surface to be macroetched,

4.1.4 The etching reagent, temperature and time of etching, or degree of etching including any special techniques which must be used, and

4.1.5 The type and degree of conditions or combinations thereof that shall be considered acceptable or subject to metallurgical review.

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[1] This test method is under the jurisdiction of ASTM Committee A01 on Steel, Stainless Steel, and Related Alloys and is the direct responsibility of Subcommittee A01.06 on Steel Forgings and Billets.

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[2] ASTM Committee A01 gratefully acknowledges the help of the AISI Committee on General Metallurgy in preparing the appendix, assembling the macroetch photographs, and assisting with the text of this test method.

[3] A complete set of the 20 macrographs on glossy paper is available at nominal cost from ASTM Headquarters, 100 Barr Harbor Drive, W. Conshohocken, PA 19428. Request Adjunct ADJA0604.

[4] *Annual Book of ASTM Standards,* Vol 03.01.

5. Sample Preparation

5.1 Unless otherwise specified, the test shall be performed on specimens, usually ¼ to 1 in. thick, cut to reveal a transverse surface.

5.2 Disks for macroetch inspection may be removed from billets by a variety of methods including torch cutting, sawing, machining, or high-speed abrasive wheels. Adequate preparation of the surface for macroetching must completely remove the effects of torch cutting or high-speed abrasive wheels.

5.3 Due to the nature of the conditions to be detected, further surface preparation is usually required.

5.4 When such further preparation is performed, grinding, machining, or sanding should be carried out in such a manner as not to mask the structure.

5.5 The surface of the disk to be etched must be free of dirt, grease, or other foreign material which might impair the result of the test.

6. Etching Reagents

6.1 The etching response and appearance is dependent upon the type and temperature of the etching reagent and the time of immersion. These details must be established by agreement between manufacturer and purchaser.

6.2 For illustrative purposes some of the commonly used etching reagents are as follows:

6.2.1 *Hydrochloric Acid*—A solution of 1 part commercial concentrated hydrochloric acid (HCl, sp gr 1.19) and 1 part water is more generally used than any other macroetching reagent. This solution may be heated without significant change in concentration, and may be reused if it has not become excessively contaminated or weakened. Etching is generally done with the solution at a temperature of approximately 160°F.

6.2.2 *Hydrochloric Acid-Sulfuric Acid Mixture*—A mixture containing 50 % water, 38 % commercial concentrated HCl, and 12 % commercial concentrated sulfuric acid (H_2SO_4, sp gr 1.84) is sometimes used in place of the previously mentioned 50 % HCl solution. The statements in the previous paragraph regarding reuse and temperature of etchant are applicable to this reagent.

6.2.3 *Aqua Regia*—A solution consisting of 1 part concentrated nitric acid (HNO_3, sp gr 1.42) and 2 parts concentrated HCl is used on corrosion and heat-resistant materials of the 18 % chromium, 8 % nickel type and higher alloy types. This reagent is used at room temperature.

NOTE 1—The reagents in 6.2.1, 6.2.2, and 6.2.3 should be used under ventilating hoods or with some provision to remove the corrosive fumes.

6.2.4 *Nitric Acid*—This etchant consists of 5 % HNO_3 solution in alcohol or water, and is generally used at room temperature. When this reagent is used, the etch disk must have a smooth surface.

7. Etching Containers

7.1 Macroetching must be done in containers that are resistant to attack from the etching reagents. Caution must be exerted to prevent the occurrence of electrolytic couples which can cause uneven attacks and misleading results.

8. Preparation of Etched Surface and Examination

8.1 Upon completion of etching, surfaces of disks should be cleaned by either chemical or mechanical methods that do not affect the macroetch quality. Care should be taken to prevent rusting of the etched surface.

9. Interpretation of Conditions Found by Macroetching

9.1 Four distinct classes of conditions are defined and described under this method:

9.1.1 *Class 1: Freckles*—Circular or near-circular dark etching areas generally enriched with carbides and carbide-forming elements.

9.1.2 *Class 2: White Spots*—Light etching areas, having no definitive configuration or orientation which are generally reduced in carbide or carbide-forming elements.

9.1.3 *Class 3: Radial Segregation*—Radially or spirally oriented dark etching elongated areas occurring most frequently at mid-radius which are generally carbide enriched. This condition may be easily confused with freckles in some materials.

9.1.4 *Class 4: Ring Pattern*—One or more concentric rings evidenced by a differential in etch texture associated with minor composition gradients and ingot solidification.

9.2 Macroetch photographs show examples of each of the conditions revealed by macroetch testing, with five degrees of severity, identified as A, B, C, D, and E for each condition. Degree A exhibits the minimum occurrence of each condition detectable by visual examination of the etched surface, while degrees B, C, D, and E represent increasing severity of occurrence.

9.3 For each condition, or combination of conditions, ratings shall be obtained by comparing each macroetched section with the standard photographs. Bar or billet sections to 225 $in.^2$ cross-sectional area may be rated against these standards. Larger sizes may be rated by agreement between manufacturer and purchaser, but caution must be exercised in interpretation of such results. Figs. 1-20 have been reduced 44 % in area from the standard photographs.

9.4 If the appearance of a given condition does not exactly match one of the five standard photographs, it shall be assigned the rating of the standard that it most nearly matches.

9.5 No standards for acceptance are stated or implied in these illustrations. The extent to which each condition may be permissible varies with the intended application, and such standards should be stated in the applicable product specification, or may be the subject of negotiation between manufacturer and purchaser.

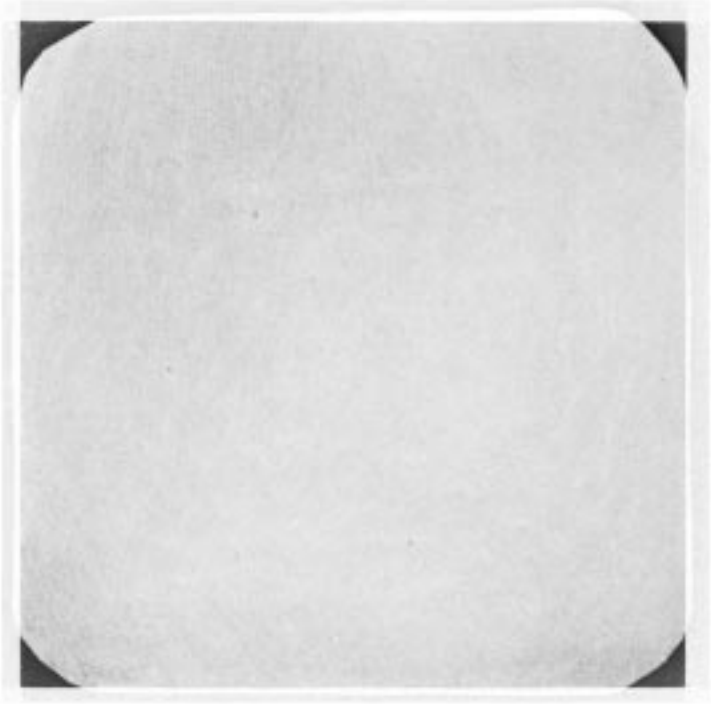

FIG. 1 Class 1—Freckles—Severity A

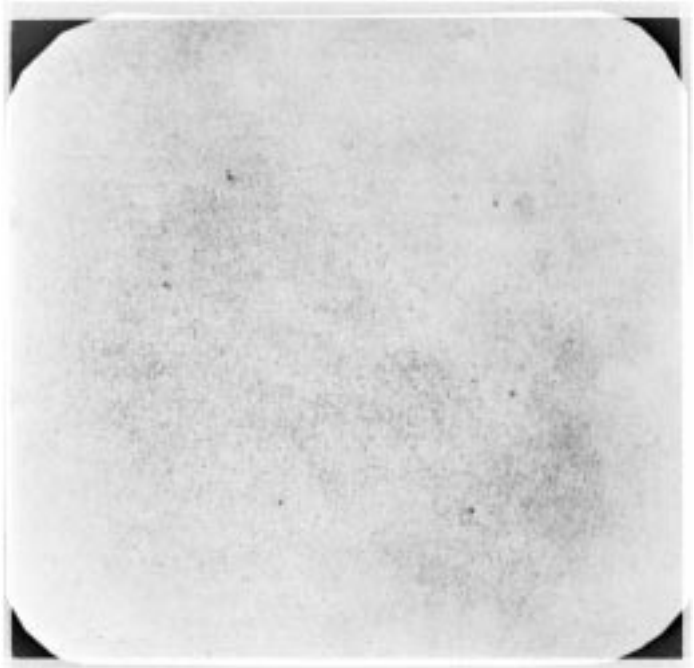

FIG. 2 Class 1—Freckles—Severity B

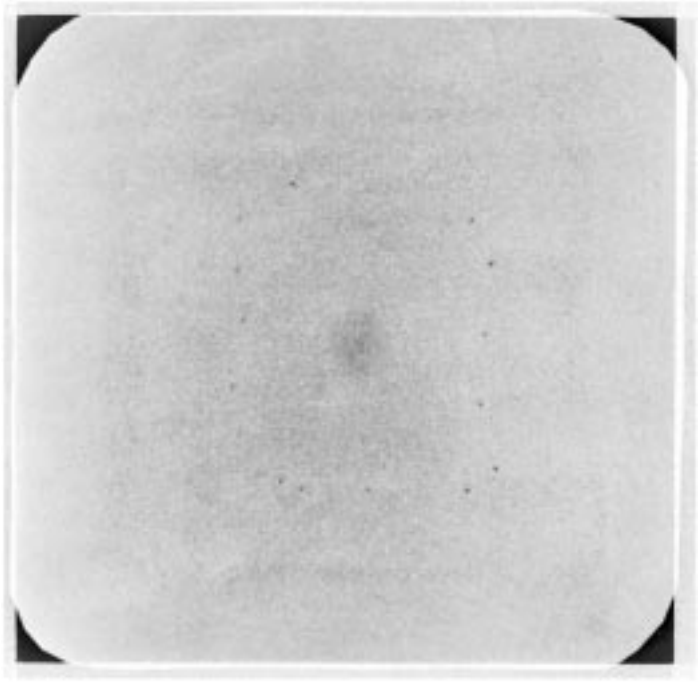

FIG. 3 Class 1—Freckles—Severity C

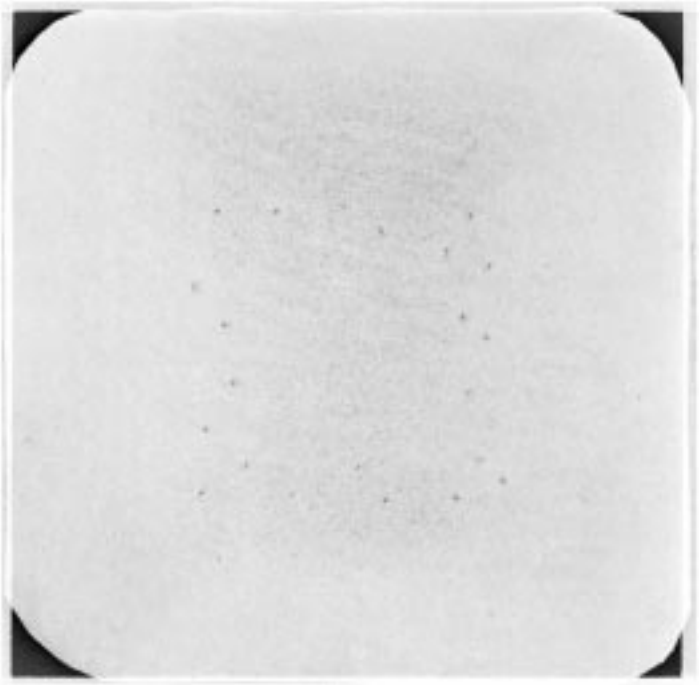

FIG. 4 Class 1—Freckles—Severity D

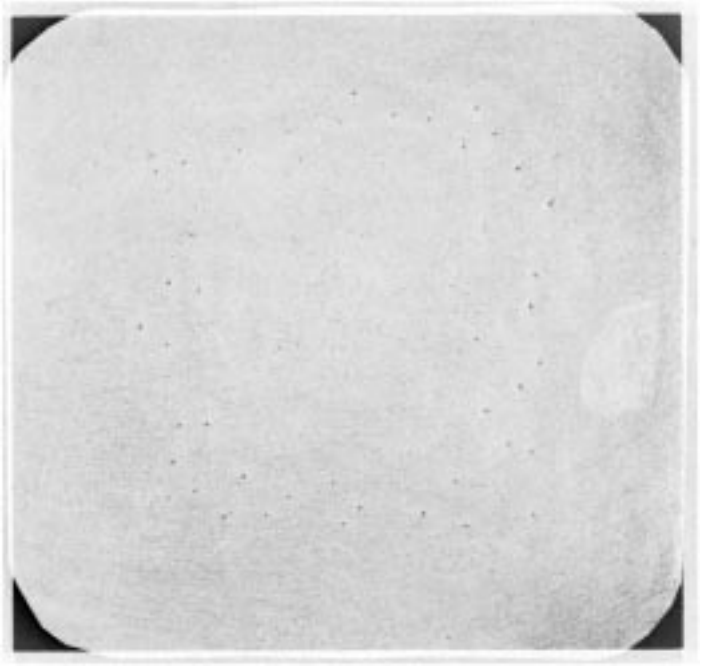

FIG. 5 Class 1—Freckles—Severity E

FIG. 6 Class 2—White Spots—Severity A

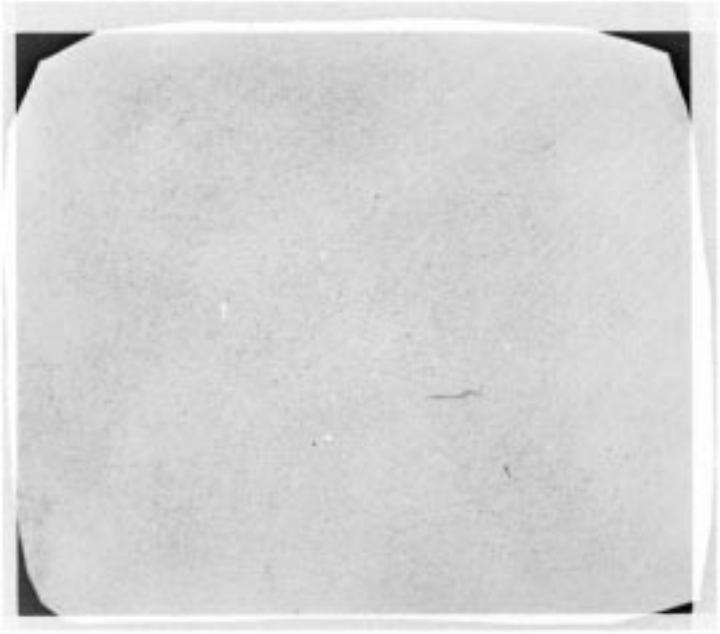

FIG. 7 Class 2—White Spots—Severity B

FIG. 8 Class 2—White Spots—Severity C

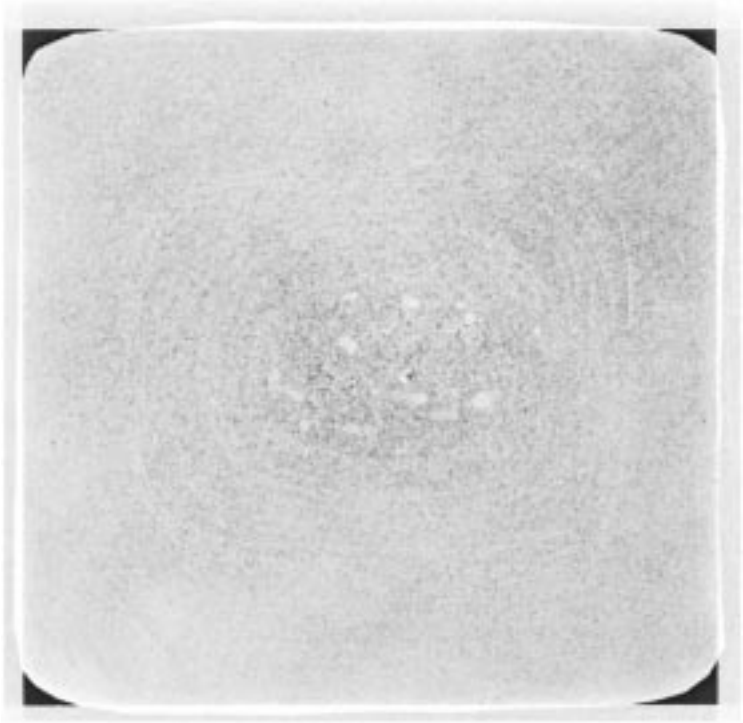

FIG. 9 Class 2—White Spots—Severity D

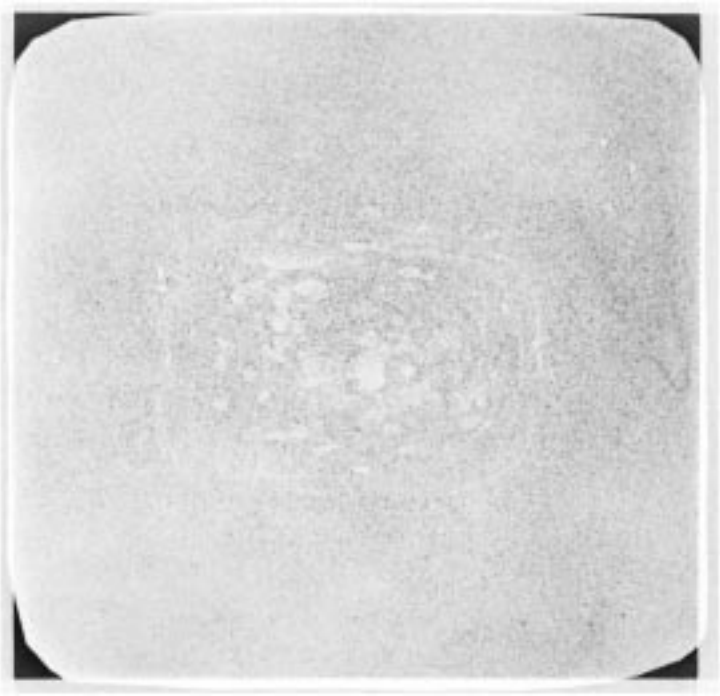

FIG. 10 Class 2—White Spots—Severity E

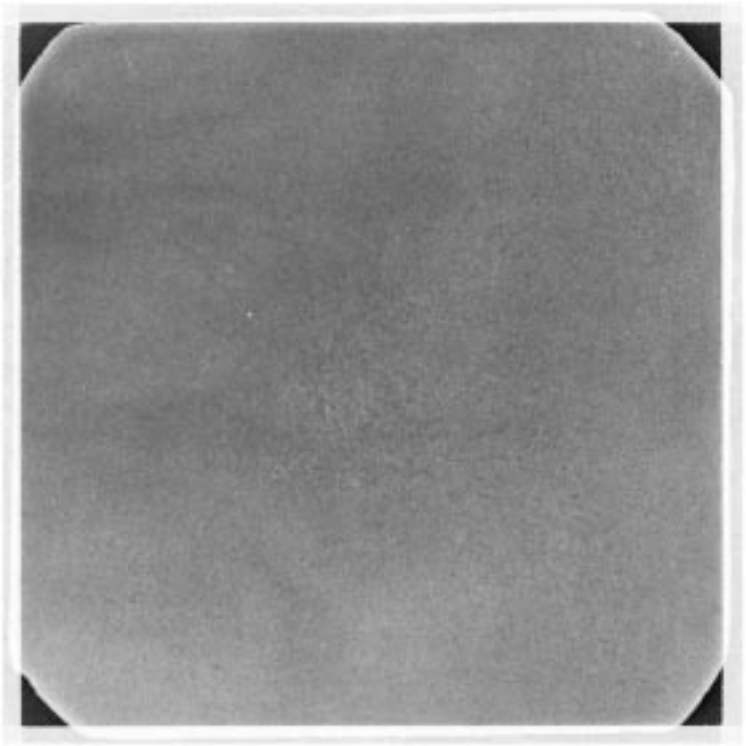

FIG. 11 Class 3—Radial Segregation—Severity A

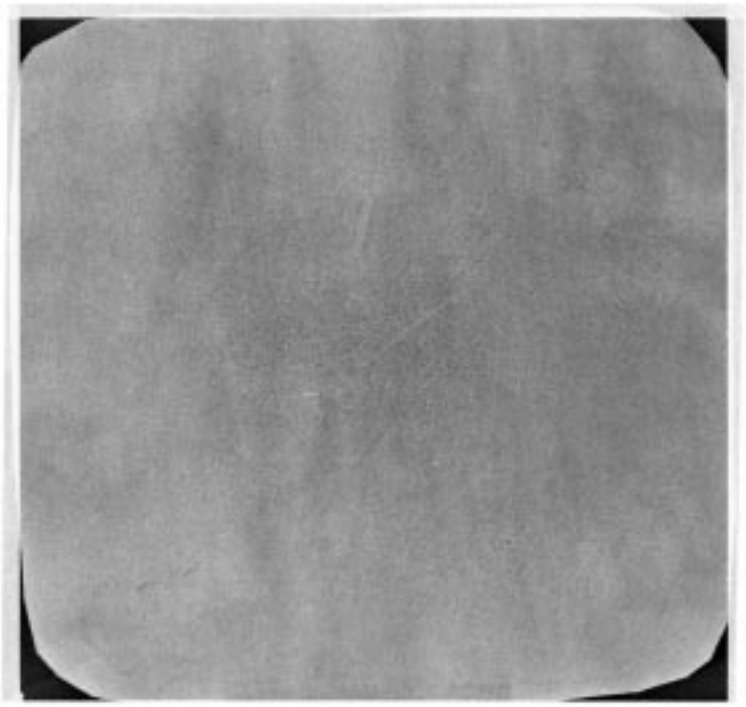

FIG. 12 Class 3—Radial Segregation—Severity B

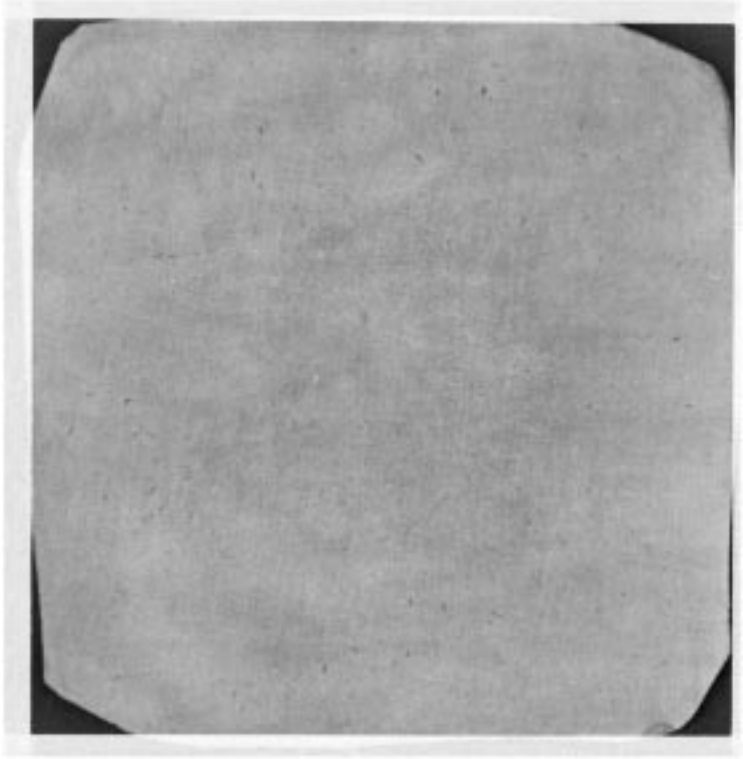

FIG. 13 Class 3—Radial Segregation—Severity C

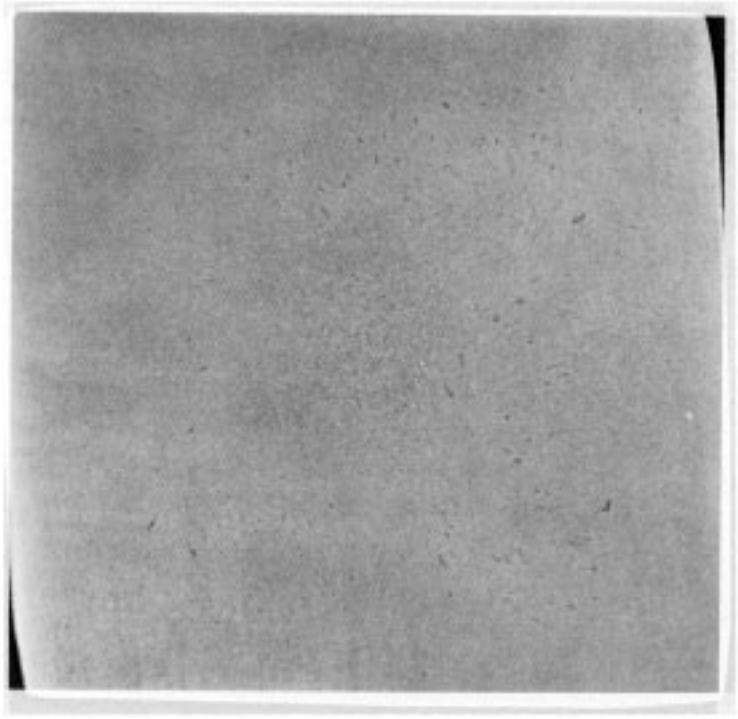

FIG. 14 Class 3—Radial Segregation—Severity D

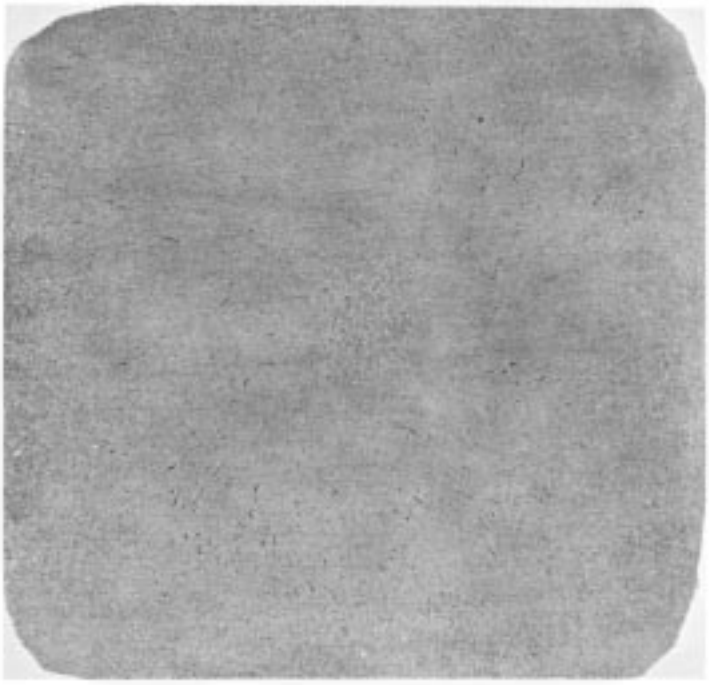

FIG. 15 Class 3—Radial Segregation—Severity E

FIG. 16 Class 4—Ring Pattern—Severity A

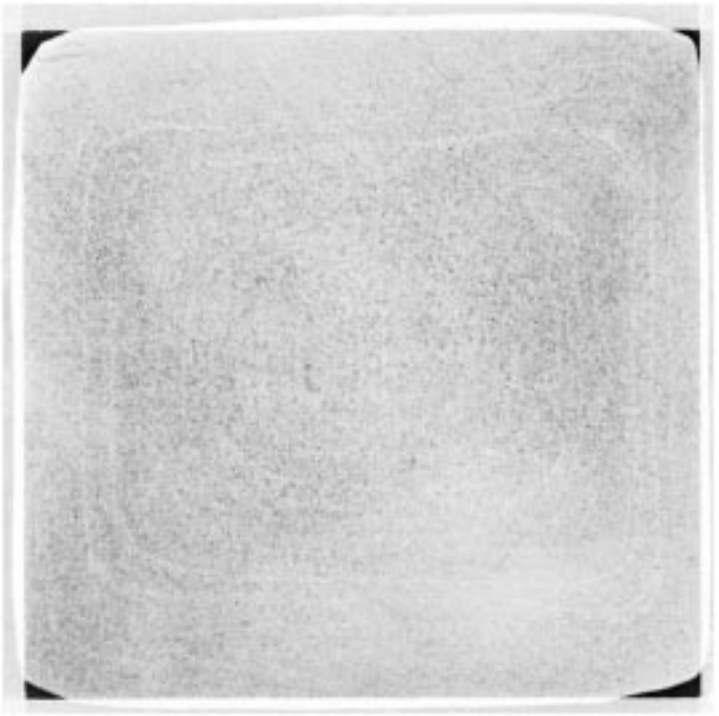

FIG. 17 Class 4—Ring Pattern—Severity B

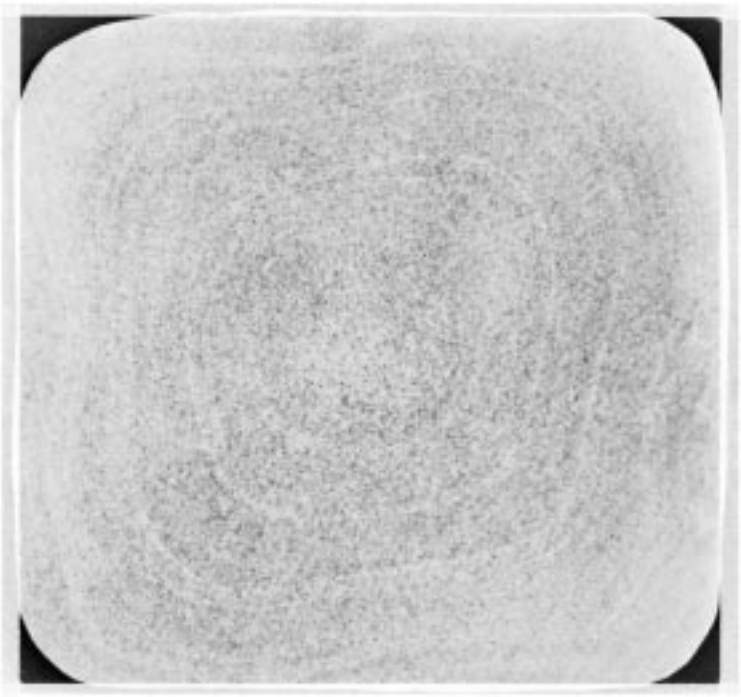

FIG. 18 Class 4—Ring Pattern—Severity C

FIG. 19 Class 4—Ring Pattern—Severity D

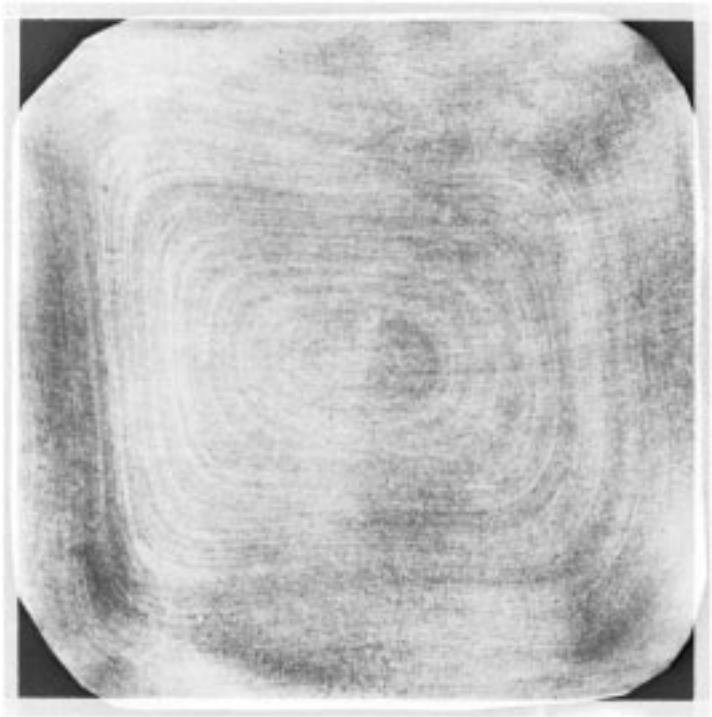

FIG. 20 Class 4—Ring Pattern—Severity E

APPENDIXES

(Nonmandatory Information)

X1. CONSUMABLE ELECTRODE VACUUM MELTING

X1.1 Process Description

X1.1.1 Consumable electrode vacuum melting (CEVM) of steel has grown from a laboratory process to a major production operation capable of producing ingots in certain grades up to 60 in. in diameter, weighing 50 tons. The available ingot sizes and weights vary from grade to grade, depending upon their complexity and alloy content. Currently, a significant proportion of the ultra-high-strength steels for aircraft and missiles, bearing steels for aircraft engines, and other speciality alloys are being consumable electrode vacuum melted.

X1.1.2 The consumable electrode vacuum melting process is diagramed in Fig. X1.1. To start the melting operation, an electrode produced from conventional air-melted or vacuum-processed steel is suspended in the consumable electrode vacuum melting furnace. The system is evacuated and an arc is struck to a bottom starting pad. Molten metal is transferred across the arc from the electrode to the solidifying ingot contained within the water-cooled copper crucible. As melting proceeds and the ingot solidifies progressively upward, the electrode is fed downward to maintain the proper arc length. As the metal droplets pass through the arc, they are exposed to this vacuum at extremely high arc temperatures, producing extensive degassification, as well as some breakdown and dispersion of inclusions. Due to the rapid cooling provided by the copper crucible, only a portion of the ingot is molten at a time and solidification proceeds in a continuously progressive manner.

X1.2 Product Characteristics

X1.2.1 Essentially, the CEVM operation changes the properties of steel in three ways:

X1.2.1.1 By reducing gas content.

X1.2.1.2 By improving microcleanliness. The nonmetallic inclusion content is rated in a manner similar to that used for air melt except that the level is generally lower and a different chart is used.

X1.2.1.3 By changing the mode of solidification from that of the traditional static-cast ingot to a progressive solidification process, involving high heat input from an arc and rapid heat extraction by the water-cooled copper crucible.

X1.2.2 Depending upon the grade of steel and the application under consideration, consumable electrode vacuum melting is reported to significantly improve one or more of the following properties: transverse ductility in aircraft forging billets, fatigue strength or endurance limit, notched tensile strength or fracture toughness, Charpy V-notch impact strength, stress rupture, and creep strength. Furthermore, hot workability and yield of some grades are significantly improved. The CEVM process has also made possible the

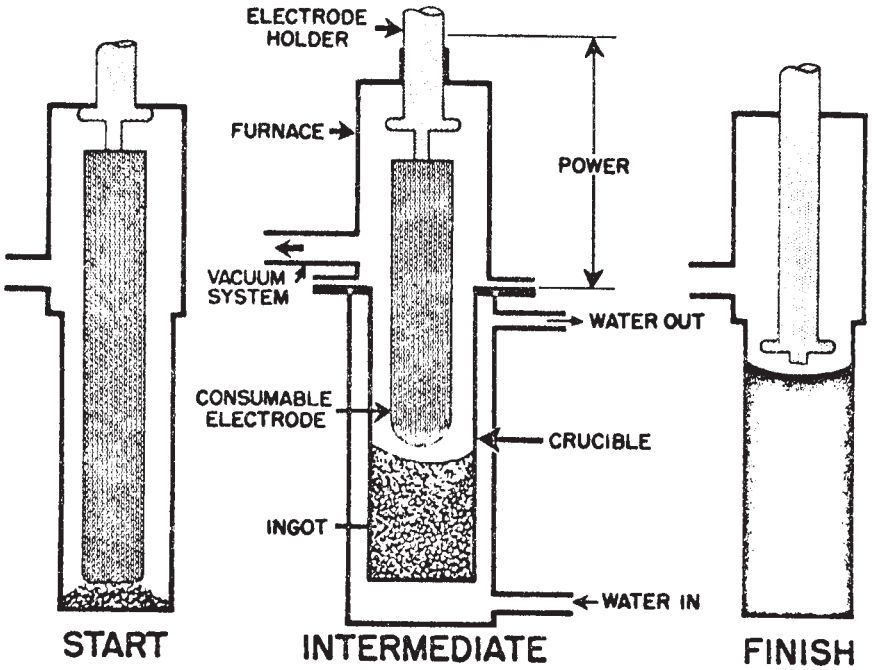

FIG. X1.1 Consumable Electrode Vacuum Melting Furnace

development of new alloys for extremely high-strength or high-temperature applications that did not exhibit satisfactory properties when melted by other methods.

X1.3 Macrotech Characteristics

X1.3.1 Consumable electrode vacuum-melted steels and alloys may contain discontinuities peculiar to this process which are disclosed upon macroetch examination.

X2. ELECTROSLAG REMELTING

X2.1 Process Description

X2.1.1 Electroslag remelting (ESR) was first introduced in an American patent by Hopkins, but most of the published work has been done by Russian engineers. The process has been shown to reduce inclusions, similar to vacuum-arc remelting, with the additional benefit of reducing sulfur content in critical alloys for aerospace and nuclear applications. Many variations of processing parameters, equipment design, ingot sizes and shapes are used.

X2.1.2 The ESR process consists of remelting a consumable electrode through a bath of molten slag using the electrical resistance of the slag to provide the required heat input. The slag composition will vary with the type of alloy and the processor's objectives. Single or three-phase ac or dc current may be applied. Water-cooled ingot molds may be square, round, or designed to produce rough tube rounds. Stationary molds or molds that can be raised as the ingot solidifies are used. Starting the process may be done with cold slag and starter chips or molten slag prepared in a small arc furnace. A diagram of this process is shown in Fig. X2.1.

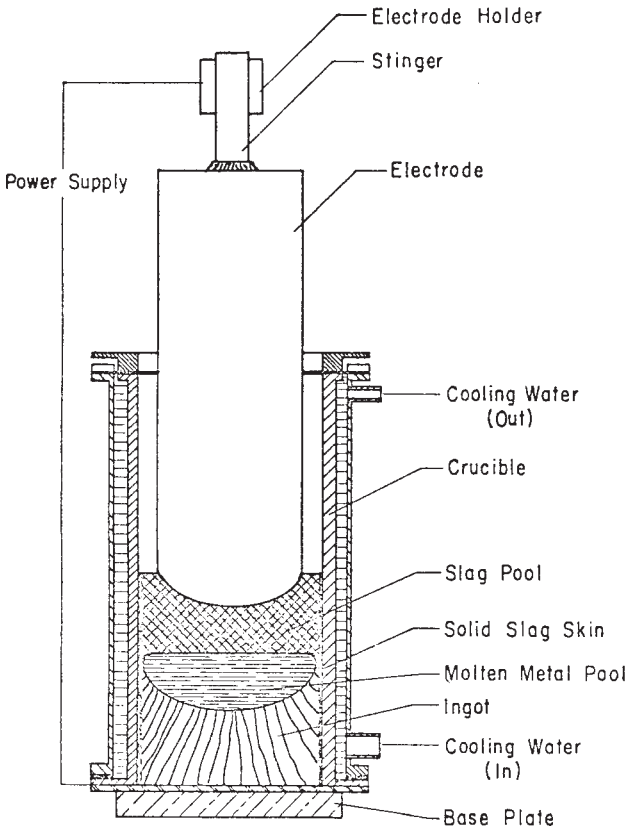

FIG. X2.1 Schematic of ESR Melting Process

X2.2 Product Characteristics

X2.2.1 The ingot surface is protected by a film of slag that solidifies on the ingot as it cools, providing an improved surface.

X2.2.2 Sulfur content may be reduced substantially to improve workability.

X2.2.3 A significant drop in oxide inclusions may be obtained.

X2.2.4 Improved uniformity occurs due to the solidification process.

X2.2.5 Little, if any, loss in alloying elements occurs with appropriate processing parameters.

X2.3 Macrotech Characteristics

X2.3.1 ESR steels and alloys may contain discontinuities peculiar to this process which are disclosed upon macroetch examination