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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Simple Static Oxidation Testing¹

This standard is issued under the fixed designation G 54; 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.

1. Scope

1.1 *Purpose*—This engineering practice covers determination of preliminary information on the relative growth, scaling, and microstructural characteristics of an oxide on the surface of a pure metal or alloy under isothermal conditions in still air. This test does not necessarily apply to testing of coated specimens.

1.2 *Application*—This procedure may be applied to any pure metal, alloy, or groups thereof that exhibit the formation of a surface oxide structure in still air at the temperature of interest, usually above about 540°C (1004°F). Direct comparison of material at a constant temperature or the effect of temperature on a given material may be investigated. Oxidation is a dynamic time- and temperature-dependent process. The relative resistances of materials to oxidation at constant temperature should, therefore, be determined over at least three time periods.

1.3 Limitations:

1.3.1 Materials usually exhibit one of several basic reactions to a high-temperature oxidizing environment. They may form a protective oxide layer which protects them indefinitely. They may form a protective oxide layer which persists for some finite time after which “scale breakaway” occurs and a scaling rate develops. They may also form a nonprotective oxide which allows rapid oxygen penetration to the metal and subsequent rapid deterioration by internal oxidation, which may render the material brittle and unusable without much observable surface or mass change. Some oxides may be liquid and thereby flux any protective oxides from the surface. Another reaction may be vaporization of the scale or one or more of the reactants. The correct interpretation of this test is thus dependent upon both mass change and microstructural depth of attack data. One should not be used without the other. For materials that form high vapor pressure oxides one must also collect the vaporized oxide by some method if it is required to complete the material balance. Specific methods to do this are beyond the scope of this practice.²

1.3.2 Materials that develop very adherent and protective oxide layers at the test temperature of interest may not be comparable in any practical amount of time because the time to scale breakaway is so long. Some alloys also react very differently to the relatively mild conditions of static oxidation than they do to the cyclic temperatures often found in service where differential thermal expansion may cause accelerated scale breakaway. This general test should thus not be used to predict the quantitative reactions of materials for specific high-temperature applications.

1.4 *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 220 Method for Calibration of Thermocouples by Comparison Techniques³
- E 230 Temperature–Electromotive Force (EMF) Tables for Standardized Thermocouples³
- G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens⁴

3. Significance and Use

3.1 Isothermal Conditions:

3.1.1 It is virtually impossible to maintain a uniform heat zone in a vertical-tube furnace. An open-tube furnace at high temperature might be considered to have still air yet variable convection currents will exist, also a tube furnace with one closed end will have a different flow pattern. Finally, erroneous results are possible when tests are conducted on materials with volatile or liquid oxide species such as WO_3 , M_2O_3 , V_2O_5 , etc. in low air flow conditions.

4. Apparatus

4.1 Furnace and Controls:

4.1.1 *Furnace*, electric-resistance-heated type, capable of heating still air to the temperature of interest. The furnace should be large enough to contain all specimens of a given study without critical regard for location. A small window in the furnace wall or door through which to view the specimens

¹ This practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals, and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

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² For a discussion of oxide vaporization at high temperatures, see Tedman, Jr., C. S., “The Effect of Oxide Volatilization on the Oxidation Kinetics of Cr and Fe-Cr Alloys,” *Journal of the Electrochemical Society*, Vol 113, 1966, pp. 766–768.

³ *Annual Book of ASTM Standards*, Vol 14.03.

⁴ *Annual Book of ASTM Standards*, Vol 03.02.

is helpful. A number of commercial “box” furnaces are available with interior dimensions of about 350 by 200 by 120 mm (14 by 8 by 5 in.) which meet these criteria. The extent and location of the constant-temperature zone within the furnace chamber should be determined for each test temperature. Samples should only be exposed within this zone.

4.1.2 *Thermocouple*—A controlling thermocouple compatible with the temperature being studied appropriately and protected should project into the furnace chamber with its hot junction as close as possible to the specimen location. Consult Method E 220 and Tables E 230 for information on thermocouples.

4.1.3 *Controls*—Test temperature and the precision of its control are important to the reproducibility and usefulness of the results. An indicating controller calibrated for the control thermocouple and capable of maintaining furnace chamber temperature is adequate. Within the very general scope of this procedure, the recommended control precision should be at least 1 % or $\pm 10^{\circ}\text{C}$ (20°F), whichever is smaller. If furnace design and the thermocouple’s precision allow it, a temperature control precision of $\pm 5^{\circ}\text{C}$ (10°F) is preferred. It may also be necessary to control the input voltage to the furnace heating elements for the most efficient temperature regulation. Over-temperature controls or continuous temperature recording should be used to guard against inadvertent temperature excursions from sticking contacts or thermocouple burnout.

4.2 *Specimen Support:*

4.2.1 Great care must be taken in the selection of this material to avoid contamination of the test specimens. Metal such as platinum, tungsten, or some high-temperature alloys is acceptable if it has been shown from previous work at the test temperature to be nonreactive with the test specimen at points of contact. Ceramic materials such as porcelain, Alundum, or fire clay may also be used if they are inert at the exposure temperature. Some ceramics may cause localized, accelerated corrosion if they contain sulfur, phosphates, or low-melting point metals. Silicon carbide is not suitable as support material due to the formation of a low-melting eutectic with some alloys.

4.2.2 The amount of specimen surface area in contact with the support material should be kept to a minimum. Specimens should also be located on the support so as to allow free circulation of the furnace atmosphere. Depending upon the test objectives, it may be sufficient to locate all the specimens of one test on a single support. Each specimen must be placed in a separate crucible, however, if it is desired to retain any spalled oxide scale.

4.3 *Balance*—An analytical-type balance should be used that meets or exceeds the following requirements:

Sensitivity	0.1 mg
Readability	0.1 mg
Accuracy	± 0.1 mg

4.4 *Metallurgical Microscope*—This instrument should contain a measuring reticle capable of measuring to ± 0.013 mm (± 0.0005 in.) at $100\times$ magnification. A viewing screen appropriately calibrated to ± 0.013 mm (± 0.0005 in.) is an alternative. Light microscopes and other instruments such as microprobes and scanning electron microscopes capable of

much higher magnifications are usually essential to determine the fine structure of oxide scales, but this is beyond the scope of this test.

5. Test Media (Air)

5.1 *Humidity*—This is usually a function of existing laboratory conditions. The oxidation characteristics of some alloy types may be affected by humidity variations. It is thus important to include reference standards in each test series or to expose all materials to be compared at the same time.

5.2 *Heating Rate*—The specimens and their support are charged into the furnace operating at the test temperature and allowed to heat.

5.3 *Cooling Rate*—The specimens and their support are removed from the furnace and allowed to cool in static ambient air.

6. Preparation of Specimens

6.1 *Size*—A specimen having a total surface of at least 400 mm^2 (0.62 in.^2) is recommended insofar as errors in weighing and rounding of corners are a function of specimen size, the largest convenient specimen dimensions are desirable. Sheet or strip specimens are preferred so that edge effects do not mask normal behavior under exposure conditions. Any shape may be used but data obtained from specimens of widely varying shapes or sizes, and thus surface areas, should not be compared directly. Duplicate or triplicate specimens should be exposed when possible.

6.2 *Method of Cutting*—Any practical method of cutting is acceptable but sheared edges should be refinished by machining, filing, or abrading to remove the severely disturbed layer of metal.

6.3 *Preparation of Surfaces*—Surface preparation, or lack of it, may affect the oxide-forming characteristics of some materials, particularly under borderline conditions. Particular research objectives, such as the evaluation of various mill-produced surfaces, may necessitate other surface preparation techniques. Detailed description of the surface preparation should be included when reporting results. For general testing, where surface preparation is not a variable to be investigated, all surfaces of the specimen should be finished with dry No. 120 grit abrasive paper. All traces of oxide scale or burrs must be thoroughly removed and the edges should not be rounded.

6.4 *Identification*—Specimens may be marked by stenciling, engraving, notching, drilling, or any other suitable method as long as the major portion of the surface area is undisturbed. In any case, the mark must be pronounced enough not to be obliterated if heavy scaling takes place. Specimens may also be left unmarked and placed in easily identified positions.

6.5 *Measuring*—The specimens should be measured to ± 0.02 mm (± 0.001 in.) and the total exposed surface area calculated including edges.

6.6 *Degreasing*—The specimens should then be thoroughly degreased in clean acetone followed by 1,1,1-trichloroethane, ether, or other suitable noncorrosive agent, and dried.

NOTE 1—Care must be taken after this step to avoid surface contamination, which may locally accelerate corrosion. Practice G 1 should be consulted.