



Standard Practice for Evaluation of Metals Exposed to Carburization Environments¹

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^{ε1} NOTE—Editorial changes were made throughout in October 1996.

1. Scope

1.1 This practice covers procedures for the identification and measurement of the extent of carburization in a metal sample and for the interpretation and evaluation of the effects of carburization. It applies mainly to iron- and nickel-based alloys for high temperature applications. Four methods are described.

<i>Method A</i>	Total Mass Gain
<i>Method B</i>	Metallographic Evaluation
<i>Method C</i>	Carbon Diffusion Profile
<i>Method D</i>	Change in Mechanical Properties

1.2 These methods are intended, within the interferences as noted for each, to evaluate either laboratory specimens or commercial product samples that have been exposed in either laboratory or commercially produced environments.

1.3 No attempt is made to recommend particular test exposure conditions, procedures, or specimen design as these may vary with the test objectives.

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 3 Methods of Preparation of Metallographic Specimens²
- E 8 Test Methods for Tension Testing of Metallic Materials²
- E 10 Test Method for Brinell Hardness of Metallic Materials²
- E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials²
- E 23 Test Methods for Notched Bar Impact Testing of Metallic Materials²
- E 139 Practice for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials²
- E 290 Test Method for Semi-Guided Bend Test for Ductility

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 *Annual Book of ASTM Standards*, Vol 03.01.

of Metallic Materials²

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens³

3. Terminology

3.1 Definitions:

3.1.1 *carbon potential*—the amount of carbon available for reaction in an environment. This amount depends upon the chemical balance of the carburizing and decarburizing agents in the system such as carbon monoxide, hydrogen, carbon dioxide, water vapor, methane, and nitrogen.

3.1.2 *carburization*—the absorption of carbon atoms into a metal surface at high temperatures. The carbon may remain dissolved or form metal carbides. This may or may not be desirable.

METHOD A—TOTAL MASS GAIN

4. Summary of Method

4.1 This method provides a relatively fast, simple, and inexpensive technique for comparing material or environmental variables. The total mass gain of the sample during exposure is considered as a first approximation of total carbon pickup.

5. Significance and Use

5.1 This method has an advantage over the other three, which are destructive single-determination techniques, in that successive measurements at selected time intervals can be made without destroying the sample. If unwanted reactions (such as sulfidation and oxidation, which are usually minor under intentionally carburizing conditions) are not important, a mass gain plot versus time can provide some additional insight about carburizing rate or intermittent variables, or both.

6. Interferences

6.1 The mass change of a sample may not be entirely the result of carbon pickup. The environment may contain some other corroding species, such as oxygen, that may react with the metal surface to form corrosion products which also affect mass change. This type of data also gives no indication of carbon distribution within the material which may be of more

³ *Annual Book of ASTM Standards*, Vol 03.02.

importance than total pickup. Considering its limitations, this method is best used in combination with at least one of the other methods described in this practice or when considerable knowledge and understanding exist as to how materials usually perform in the particular conditions of the exposure environment, or both.

7. Procedure

7.1 This method assumes the use of a sample that can be readily measured to obtain exposed surface area and weighed both before and after exposure to obtain mass gain per unit surface area, that is, grams per square metre. See Practice G 1.

8. Discussion of Results

8.1 The successful application of this technique depends primarily upon the ability to measure small mass changes. All weighing should be done to the nearest 0.1 mg. Section thickness is also important in order to approximate an “infinite” solid thus allowing carbon diffusion from one surface to be unaffected by diffusion from any other surface. A minimum section thickness of at least 12 mm is necessary, particularly with cylindrical samples, for short time exposure in most carburizing environments. When calculating carburization rate, it must be assumed that carburization as measured by mass gain is not linear with time.

METHOD B—METALLOGRAPHIC EVALUATION

9. Summary of Method

9.1 The sample is cut, polished, and etched to accentuate the carbide structure. The extent of carbon penetration sufficient to form insoluble carbides is then measured directly on a magnified area.

10. Significance and Use

10.1 The carbon penetration number refers to the point at which insoluble carbides are first formed. It does not indicate the total depth of carbon penetration. Metallographic measurement of carbon penetration can be used by itself for evaluation of materials. It can be particularly useful when combined with total mass gain data to give some indication of the solubility and mobility of carbon in the exposed material as suggested by the following:

Mass Gain	+	Penetration	=	Solubility	and	Mobility
low		low		low		low
low		high		low		high
high		low		high		low
high		high		high		high

11. Interferences

11.1 The major limitation of this method lies in the fact that it is sometimes very difficult to differentiate visually between carbides that have formed from carbon diffused into the metal from the exposure environment and those that formed from carbon inherent in the composition of the alloy. An example of this situation is illustrated by comparing the relatively distinct carburized layer boundary in Fig. 1 with the more diffuse area in Fig. 2. This is particularly true of nominally high carbon-

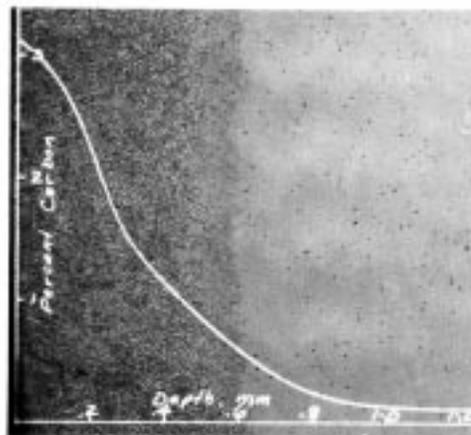


FIG. 1 Microstructure of Carburized Sample with Superimposed Carbon Diffusion Profile (75×)

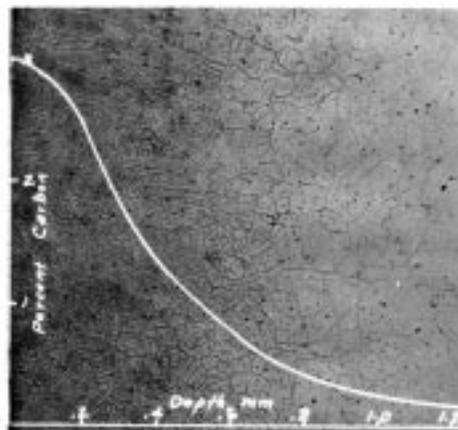


FIG. 2 Microstructure of Carburized Sample with Superimposed Carbon Diffusion Profile (75×)

content alloys. In these cases, the depth of carbon penetration becomes a judgment based on density of the precipitated phase.

12. Procedure

12.1 Success with this method requires that close attention be paid to Methods E 3. The sample is first cut so that the final viewing axis will be perpendicular to the direction of carbon diffusion. After polishing, the specimen is usually etched with a suitable acid mixture to delineate carbides. Some particularly useful etchants are listed in Table 1. The sample is viewed at a magnification of between 50× and 100×. The depth of carbide precipitation is then determined with the microscope’s measuring recticle or other system such as a glass screen and appropriate scale. For example, the sample shown in Fig. 1 appears to have a carbide precipitation depth of about 0.6 mm. Carbon penetration may in some cases be very uneven due to intergranular or other localized acceleration of diffusion. The penetration depth shall thus be taken as at least the average of three measurements each in several areas. Some measure of variability is also necessary such as a standard deviation or other indication. In all cases preview the entire mounted

TABLE 1 Typical Etchants Used to Accentuate Carbide Structures in Iron- and Nickel-Based Alloys

Etchant	Composition ^A	Remarks
Nital	HNO ₃ : 1–5 mL CH ₃ OH or C ₂ H ₅ OH: 100 mL	Use colorless acid and absolute alcohol. Electrolytic microetch 5–10 V, 1–5 s. Make specimen the anode.
Oxalic acid	HOOCOOH: 10 gm H ₂ O: 100 mL	Electrolytic microetch as above.
Glyceresia	HNO ₃ : 10 mL HCl: 20 mL glycerol: 30–40 mL	Microetch. Immerse or swab specimen for 30 s to 5 min with freshly prepared solution.

^A Use concentrated acids.

specimen prior to measurements so that any areas of nonuniformity can be identified. It is helpful to compare photomicrographs of exposed samples with a standard that has received the same temperature and time exposure but without the external carbon potential. Alternatively, if the exposed sample has a large enough cross section, the surface carbide density can be compared with the unaffected core area.

13. Discussion of Results

13.1 Comparisons of carbon solubility and mobility indications are most accurate and meaningful when the boundary between the carburized and uncarburized areas is uniform and well delineated. When this boundary is vague or highly variable, results can be misleading. Statistical analysis cannot necessarily salvage vague measurements. It is best to avoid this technique unless the measurements can be made easily and unequivocally.

METHOD C—CARBON DIFFUSION PROFILE

14. Summary of Method

14.1 In general, this method involves the analysis of consecutive layers of an exposed sample. This can be done by removing and collecting material with a suitable machining technique such as milling or turning. Wavelength dispersive X-ray analysis can also be used in conjunction with the metallographic mount prepared for microexamination in Method B.

15. Significance and Use

15.1 Typical diffusion profiles determined by this method are shown in Figs. 1 and 2. The curves obtained by this method provide a more direct and meaningful measure of carbon solubility and mobility in a metal than can be achieved by a combination of Methods A and B. For instance, the carbon percent versus depth profile may show a difference in alloys due to mobility as compared to strong carbide forming tendency near the surface. It also provides a good quantitative graphic comparison of alloy variables.

16. Interferences

16.1 This method assumes that the excess carbon found at any particular point came from one direction only. This may not be the case, particularly with corners and thin or small

cylindrical shapes. Care should be taken while examining particularly the inner portions of a carbon profile to consider whether at least some of the carbon found might have arrived from other surfaces.

17. Procedure

17.1 The technique of consecutive layering by machining requires that no lubricant be used. The collected material is degreased if necessary and analyzed for carbon content by a suitable technique such as combustion analysis. The average carbon content of each layer is plotted versus depth of the midpoint of its respective layer. Layering, or other technique of consecutive analysis, is usually continued until the carbon composition approaches that inherent in the unaffected metal.

18. Discussion of Results

18.1 The usefulness of this method is dependent upon both the layering technique and the chemical analysis. The accuracy of the chemical analysis is a function of the interrelationship of the analysis technique, sample size, and carbon content. Surface area and thickness of the layer must be adjusted to minimize thickness while obtaining enough sample material for multiple analyses using the available technique. Generally, the newer instrumental techniques of combustion carbon analysis are more precise and require less time and sample than the primary gravimetric technique. Regardless of the technique used, the layer-cut sample shall be consumed in multiple analyses to provide a good average total carbon content per layer. The values listed in Table 2 were obtained by multiple combustion analyses of a series of consecutive samples, each weighing about 1 g, turned from a cylindrical alloy specimen. As such, they provide some measure of the level of uncertainty of this technique.

TABLE 2 Typical Carbon Determinations Obtained from Duplicate Combustion Analyses of 1 g Samples Consecutively Cut from the Surface of a Wrought Ni-Cr-Fe Alloy Cylindrical Specimen After Exposure in a Carburizing Environment

Layer Number	Layer Depth ^A (mm)	Carbon Determinations, %	
		1	2
1	0.03	3.209	3.184
2	0.13	2.717	2.606
3	0.26	2.368	2.344
4	0.39	2.242	2.247
5	0.53	2.197	2.203
6	0.68	2.168	2.114
7	0.81	2.079	2.059
8	0.93	2.020	2.032
9	1.05	2.003	1.961
10	1.19	1.918	1.912
11	1.33	1.825	1.848
12	1.45	1.729	1.734
13	1.56	1.659	1.623
14	1.70	1.526	1.513
15	1.85	1.333	1.354
16	1.99	1.160	1.175
17	2.12	0.965	0.975
18	2.25	0.747	0.748
19	2.40	0.549	0.527
20	2.56	0.416	0.415
21	2.71	0.367	0.361
22	2.85	0.314	0.318
23	3.02	0.257	0.269
24	3.20	0.219	0.220

^A Distance of layer midpoint from outer surface of specimen.

METHOD D—CHANGE IN MECHANICAL PROPERTIES

19. Summary of Method

19.1 Carburization usually has a great influence on mechanical properties such as strength, ductility, and hardness. Thus hardness, tension, impact, creep, and bend tests of carburized material often yield meaningful results when compared to unaffected material.

20. Significance and Use

20.1 This method provides a straight-forward measure of the effects of carburization on metal properties. When combined with Method B or C and a study of fracture surfaces, it can provide valuable information as to depth of effect. When carburization is only partial, it is difficult to know how the properties of the specimen will relate to those of an engineering structure in the field. The results are, therefore, more or less qualitative and a function of the size and shape of the specimen being tested.

21. Interferences

21.1 The primary limitations of this method are related to obtaining suitable representative specimens of proper orientation and size. It is also important to determine whether bulk properties or surface properties of a partially carburized sample are most important.

22. Procedure

22.1 The mechanical property tests shall be run over a temperature range similar to that of the material in service because properties tend to change drastically with temperature. Use this method in combination with Method B or C to determine the depth of carbide precipitation or increase in carbon content. Always compare the properties of carburized material with those of a reference material representing similar exposure conditions in a nonreactive environment. Use the

appropriate test from the following: for hardness testing, either Test Method E 10 or Test Methods E 18; for tension testing, Test Methods E 8; for impact testing, Test Methods E 23; for creep testing, Practice E 139; and for bend testing, Test Method E 290.

23. Discussion of Results

23.1 Results may vary from specimen to specimen, however, depending on the skill of sample selection and the uniformity of carburization. A statistically significant number of test results should nevertheless be presented as a trend analysis rather than a precise statement of values.

24. Report

24.1 The report shall include detailed descriptions of the specimens and pertinent data on exposure conditions in addition to the data necessary for and obtained from each evaluation method.

24.2 Descriptions of the exposed specimens shall include size dimensions of both the specimen and the product from which it was taken, alloy designation, chemical composition, product form, metallurgical history, surface preparation, color, surface texture, and any post-exposure cleaning methods.

24.3 Descriptions of exposure conditions shall include environment composition and temperature including changes during the test, flow rate of gases, description of apparatus used, duration of exposure, and method of heating and cooling samples.

24.4 If multiple specimens are used, the location of each relative to the others and the gas flow shall be specified. It is also important to differentiate between multiple single specimen exposures and multiple specimens with a single exposure.

25. Keywords

25.1 alloy; carbon; carburization; corrosion; high temperature; iron based alloy; metal; nickel based alloy

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