INTERNATIONAL

Designation: B935 – 20

Standard Guide for Steam Treatment of Ferrous Powder Metallurgy (PM) Materials[1]

This standard is issued under the fixed designation B935; 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 This guide is intended as an aid in establishing and maintaining a procedure for the steam treatment, also referred to as steam blackening, of sintered ferrous PM materials and the appropriate use and evaluation of these materials. Additional information concerning the effect of this process on ferrous PM material properties is contained in Appendix X1.

1.2 *Units*—With the exception of the values for density and the mass used to determine density, for which the use of the gram per cubic centimetre (g/cm^3) and gram (g) units is the longstanding industry practice, the values in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B243 Terminology of Powder Metallurgy

3. Summary of Guide

3.1 A normal sequence of steps for batch processing is: *(1)* preheat the load; *(2)* introduce a superheated steam-rich atmosphere; *(3)* heat the load to the processing temperature and maintain the temperature for the duration of the processing cycle; and *(4)* cool the load to a temperature suitable for handling. This process will produce a layer of black iron oxide (magnetite) on the surface of the parts and on the surfaces of the interconnected porosity by the reaction.

$$3Fe + 4H_2O\ (gas) \leftrightarrow Fe_3O_4 + 4H_2\ (gas)$$

3.2 For continuous steam treatment, parts are transported through the furnace on a continuous mesh belt, and the thermal profile of the furnace (temperature settings of the various zones and the speed of the mesh belt) is set to ensure that parts are clean and at the appropriate temperature before they are exposed to the superheated steam.

4. Terminology

4.1 Definitions of powder metallurgy (PM) terms can be found in Terminology B243.

5. Significance and Use

5.1 The performance and quality of steam-treated materials depends upon the surface cleanliness of the material prior to steam treatment and the adequacy of the processing. Steam treatment can be used as a decorative coating, producing a blue-gray to a blue-black appearance. It can reduce the susceptibility of ferrous PM materials to further oxidation and corrosion, thus providing better shelf life. More significantly, improvements in apparent hardness, compressive strength, wear characteristics, and some mechanical properties (see Appendix X1) can be observed due to steam treatment. The hardness of magnetite (Fe_3O_4) formed during steam treatment is typically equivalent to 50 HRC, and when present in sintered materials, their wear resistance can be improved significantly. Steam treatment is also used to seal parts or provide a base material for additional coatings. Steam treated ferrous PM materials are used in many industries, including automotive, marine, home appliances, and lawn and garden applications.

6. Apparatus

6.1 The material can be processed in either a batch-type furnace or a continuous belt-type furnace. The furnace must be capable of heating the load, maintaining it at the processing temperature, and maintaining a steam atmosphere free of air

[1] This guide is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.05 on Structural Parts.

Current edition approved Oct. 1, 2020. Published November 2020. Originally approved in 2005. Last previous edition approved in 2016 as B935 – 16. DOI: 10.1520/B0935-20.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard**

leaks. Both batch type and continuous furnaces shall meet the same criteria described in the following procedure section. While comparable performance may be obtained from batch and continuous furnaces, the process conditions used may not be exactly the same.

7. Procedure

7.1 *Batch Processing*

7.1.1 Place the load in the furnace and preheat in air until the temperature of the entire load is above 212 °F (100 °C) but not in excess of 800 °F (430 °C). A temperature of 600 ºF (315 ºC) is typically used.

7.1.2 Once the load is preheated, introduce superheated steam into the furnace until all of the air is purged from the furnace or processing zone. This steam atmosphere shall be maintained until the processing cycle is complete.

NOTE 1—The temperature of the parts shall be in excess of 212 °F (100 °C) before the superheated steam is introduced. If it is not, water will condense on the parts and rust spots will form. The temperature shall not exceed 800 °F (430 °C) before all of the air has been purged from the system by the superheated steam or the parts will be covered with a smooth even coating of rust caused by air in the furnace atmosphere.

7.1.3 Raise the temperature of the furnace to the processing temperature of between 800 °F (430 °C) and 1100 °F (590 °C) while maintaining the atmosphere of superheated steam. A typical processing temperature is 1000 °F (540 °C).

NOTE 2—The processing temperature depends on the desired properties. Lower temperatures result in deeper oxide penetration, while higher temperatures produce a thicker surface oxide layer.

NOTE 3—Processes that decrease the amount or the size of the surface-connected porosity such as copper infiltration, grinding, vibratory finishing, sizing, machining, burnishing, shot peening, or polishing will reduce the effectiveness of the steam treatment by limiting the ability of steam to penetrate the part. Increasing the density reduces the pore size, which reduces penetration unless sufficiently large porosity is available to maintain a pathway for the steam. Copper infiltration also coats the surface of iron particles with copper, which prevents steam from reacting with the iron and decreases the effectiveness of steam treatment as a surface treatment.

7.1.4 Hold the load at the processing temperature for 30 to 120 min, depending on the desired properties. Steam treatment builds an oxide layer on pore surfaces and gradually restricts the access of the steam to the interior of the part. Higher temperature causes the metal to be more reactive, producing a thicker surface layer and limiting the penetration of the oxide into the part. Lower temperature produces deeper penetration with a thinner surface layer.

NOTE 4—A discussion of sealing method is recommended for parts requiring additional surface treatments (plating, painting, and so forth).

7.1.5 The oxide produced by steam treatment is slightly porous, which can allow corrosive material to penetrate to non-oxidized iron. While the oxide provides improved shelf and handling life by itself, oil impregnation, an oil dip, or rust preventatives are recommended for applications requiring additional corrosion protection.

7.2 *Continuous Processing*

7.2.1 A pre-heat zone is used to bring the parts up to temperature, while at the same time burning out any residual oil or fluid and cleaning the parts. The pre-heat is typically set to a temperature of 840 to 930 ºF (450 to 500 ºC). Exothermic gas burners are suitable for the pre-heat zone.

7.2.2 The temperature in the steam zone of the furnace is generally set to a temperature of 800 to 1100 ºF (430 to 590 ºC). A typical process temperature is 1000 ºF (540 ºC).

7.2.3 The actual process temperature and the time at temperature depend on the desired properties of the steam-treated parts. Times at temperature in the superheated steam are typically from 45 min to 1½ h.

8. Interferences

8.1 The surface of the material to be steam-treated should be free of contaminants, including red rust, soot, and dirt. Steam treatment cannot reduce existing rust. Soot and dirt attract moisture, which causes rust to form during steam treatment. The soot and dirt will remain as surface blemishes after steam treatment.

8.2 Since steam treatment builds an iron oxide layer on the pore surfaces, it tends to restrict access to the non-oxidized interior iron, causing the reaction to slow until further treatment has little effect on the thickness of the oxide layer.

9. Hazards

9.1 Hydrogen is generated as a by-product of the steam treatment process. This hydrogen must be flushed from the furnace and air ingress must be prevented. Prior to opening a batch-type furnace, the furnace should be flushed with nitrogen or an inert gas unless the parts and furnace are cooled to below the auto ignition point of hydrogen while still in the steam atmosphere.

9.2 **Warning—**Foreign material can interfere with steam treatment and possibly pose a serious health threat. Some chemical compounds, for example, chlorinated hydrocarbons, decompose into products that cause materials to rust during processing. All cutting fluids, lubricants, coolants, and so forth should be removed prior to steam treatment. This may be done during the preheating stage of the steam treatment process. However, the effect of the contaminant and by-products must be considered to ensure they are compatible with the steam treatment process and do not create a hazardous condition.

10. Testing and Evaluation

10.1 Fe_3O_4 can be deposited either heavily on the external part surface or uniformly at the surface and in the porosity. Depending on the objective of the steam treatment, the methods used for evaluation may need to be changed.

10.2 A simple visual inspection is sufficient for many decorative applications. Density or the change in mass measure the amount of Fe_3O_4 formed. Apparent hardness gives an indication of the surface condition. Metallographic evaluation is recommended to determine the extent of oxide penetration. Microindentation and file hardness testing are not recommended.

10.3 No single test will indicate the effectiveness of steam treatment for all potential applications. It is recommended that the producer and user agree on test procedures to evaluate parts early in the part development process.

10.4 Steam treatment changes the chemical composition and the mechanical properties of the material. Testing for compliance with material specifications should therefore be performed prior to steam treatment, unless it is agreed that the part is to be tested in the steam-treated condition.

11. Keywords

11.1 blackening; black oxide; ferrous PM parts; iron oxide; magnetite; steam

APPENDIX

(Nonmandatory Information)

X1. TYPICAL PROPERTIES OF STEAM-TREATED MATERIALS

X1.1 Table X1.1 and Table X1.2 summarize data for various materials and process cycles. Data are included for material in the as-sintered and for the steam-treated condition (AS = as-sintered; ST = steam-treated). The data should not be taken as a specification or minimum or maximum results that should be expected from steam treatment. Two steam-oxidation processes were used to develop the data. They are identified as Process A and Process B. In Process A, the parts were exposed to superheated steam for 2 h at 1000 ºF (540 ºC), while in Process B the parts were exposed for 4 h at 1000 ºF (540 ºC). The results of this study are based on the average of three (3) test specimens. The data are taken from the following reference: Pease III, L. F., Collette, J. P., and Pease, D. A., "Mechanical Properties of Steam Blackened P/M Materials," *Modern Developments in Powder Metallurgy*, Vol. 21, 1988, p. 275, compiled by P. Ulf Gummeson and Donald A. Gustafson, published by Metal Powder Industries Federation, Princeton, NJ.

TABLE X1.1 Comparison of Properties Before (AS) and After Steam Treatment (ST)

	Process A (inch-pound)											
Material[A]	UTS 10^3 psi		% Elongation In 1 in.		TRS 10^3 psi		Unnotched Charpy Impact ft·lbf		Apparent Hardness HRB		Density g/cm^3	
	AS	ST	AS	ST	AS	ST	AS	ST	AS	ST	AS	ST
F-0000-10	20	22	6	2	48	53	4	2	37	88	6.20	6.52
F-0000-15	19	20	3.5	<1	57	72	4	2	52	90	6.93	7.10
F-0008-25	31	19	1.5	<1	66	63	2.5	1.5	69	108	6.26	6.61
F-0008-30	39	28	1.5	<1	96	95	3	2	87	110	6.78	6.96
FC-0208-40	48	30	1	<1	99	88	3.5	2	87	112	6.27	6.54
FC-0208-50	67	59	1.5	1	138	134	6.5	4	95	111	6.85	7.03
	Process A (SI)											
Material[A]	UTS MPa		% Elongation In 25 mm		TRS MPa		Unnotched Charpy Impact J		Apparent Hardness HRB		Density g/cm^3	
	AS	ST	AS	ST	AS	ST	AS	ST	AS	ST	AS	ST
F-0000-10	140	150	6	2	330	365	5	2.5	37	88	6.20	6.52
F-0000-15	130	140	3.5	<1	395	495	5	2.5	52	90	6.93	7.10
F-0008-25	215	130	1.5	<1	455	435	3	2	69	108	6.26	6.61
F-0008-30	270	195	1.5	<1	660	655	4	2.5	87	110	6.78	6.96
FC-0208-40	330	205	1	<1	680	605	4.5	2.5	87	112	6.78	6.54
FC-0208-50	460	405	1.5	1	950	925	8.5	5	95	111	6.85	7.03

[A] The materials listed are sponge iron powder-based; except for the F-0008-30, a water granulated iron powder-based material, the FC-0208-50 and F-0000-15 are water atomized powder-based materials.