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Standard Guide for Additive Manufacturing — Feedstock Materials — Testing Moisture Content in Powder Feedstock[1]

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1. Scope

1.1 This standard provides guidelines for measuring moisture in powder feedstock used in additive manufacturing (AM). It applies to metallic, ceramic, and polymer AM powder feedstocks.

1.2 This guide provides a description of test methods commonly used to measure moisture and references to their associated standards.

1.3 This guide provides best practice guidance on how to apply the test methods to make them suitable for AM powder characterization.

1.4 This guide is suitable for measuring moisture in AM powder feedstock over the range of 10 µg/g to 10 000 µg/g.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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.*

1.7 *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]

B215 Practices for Sampling Metal Powders

B243 Terminology of Powder Metallurgy

D6869 Test Method for Coulometric and Volumetric Determination of Moisture in Plastics Using the Karl Fischer Reaction (the Reaction of Iodine with Water)

D7191 Test Method for Determination of Moisture in Plastics by Relative Humidity Sensor

E473 Terminology Relating to Thermal Analysis and Rheology

E1131 Test Method for Compositional Analysis by Thermogravimetry

E1142 Terminology Relating to Thermophysical Properties

E1409 Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by Inert Gas Fusion

E1868 Test Methods for Loss-On-Drying by Thermogravimetry

2.2 *ISO/ASTM Standard:*[2]

ISO/ASTM 52900 Additive manufacturing — General principles — Fundamentals and vocabulary

2.3 *ISO Standards:*[3]

ISO 17034 General requirements for the competence of reference material producers

ISO 3954 Powders for Powder Metallurgy Purposes — Sampling

3. Terminology

3.1 *Definitions*—Powder metallurgy terms are defined in Terminology B243. Additive manufacturing (AM) terms can be found in ISO/ASTM 52900. Technical terms in relation with thermogravimetry (TGA) and loss on drying (LOD) are defined in Terminologies E473 and E1142, respectively. Relative humidity sensor terminology is in accordance with Terminology D7191, while terms related to the Karl Fischer method are described in Test Method D6869.

4. Summary of Guide

4.1 Different methods can be used to measure moisture in AM powders. For all methods of interest, the sample of powders are heated in the instrument to vaporize the water. The amount of water is then evaluated using different approaches

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[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.

[3] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

and procedures, depending on the method, as defined in the referenced standards. In coulometric titration methods (for example, Karl Fisher as described in Test Method D6869) and relative humidity sensor methods (as described in Test Method D7191), a dry gas carries the volatiles (evaporated when the samples are heated up) into a titration cell (Karl Fischer) or humidity sensor (relative humidity sensor) to measure the amount of water. For gravimetric or loss-on-drying (LOD) methods (as described in Test Methods D6869, D7191, E1131, and E1868), the weight of the samples is measured during the test and the weight variation is converted into a water content assuming that water is the only compound evaporating from the sample during the test and there is no decomposition or modification of the sample during the test. Presence of other volatiles and the decomposition or reaction of the sample with water or atmosphere may all influence the results.

4.2 In addition to the methodology proposed in standards on coulometric titration, relative humidity sensors and LOD (see Referenced Documents, Section 2), it is recommended to follow the guidelines and recommendations of this guide if measuring moisture in AM powder feedstocks.

5. Significance and Use

5.1 This guide will help manufacturers and users of AM powder feedstocks to identify suitable methods for measuring moisture in the feedstocks.

5.2 This guide will aid control of powder quality and allow powder producers and users of AM machines to assess moisture content of virgin and reused powders.

5.3 This guide is intended to support acceptance and control tests.

5.4 Moisture levels are usually relatively low in metallic powder feedstocks (typically lower than 250 µg/g) but could be significantly more important in polymer and ceramic (typically lower than 10 000 µg/g).

5.5 Moisture may affect powder processability (powder supply and feeding, layer creation) and influence the process and properties of the printed components. As different processes and machines use powders with different characteristics (that is, particle size distribution and shape) and AM machines store and handle powders in different ways, the amount of moisture and its impact may vary significantly depending on the feedstock, process, and machine.

5.6 A proportion of the water is physisorbed on the surface and can be easily adsorbed and desorbed.

5.7 A fraction of the water can be strongly bonded to the surface of the powder (that is, chemisorbed) and can be difficult to extract even at temperatures significantly higher than 100 °C. Thus, the water may not all be recovered during the moisture analysis and some water may remain in the samples. Consequently, the values obtained during the tests may be underestimated. As water bonds differently to different materials, the evaporation of water as a function of temperature may vary from one material to another.

5.8 Because of the reactive nature of powders, water may react with the surface of the powder and form oxides and hydroxides. Thus, the amount of moisture may change with time even if the powder is stored in a tightly sealed container. Reaction of the powder with water may also happen during the analysis as the powder is heated up. This reaction will reduce the amount of water available at the surface of the powder and may impact the results (that is, underestimate the amount of water measured). If such reactions are expected to occur, their impact on the measurements should be evaluated. This can be done using oxygen analysis to evaluate the amount of oxide formed with time or during a test.

5.9 The amount of water adsorbed on the surface of a powder depends on temperature and relative humidity and is determined by moisture sorption isotherm (water content in equilibrium on a material surface at a given temperature and moisture content). Depending on the temperature and humidity content of the atmosphere, water can adsorb and desorb from the surface of the material to reach an equilibrium with its environment.

5.10 In consideration of 5.6 – 5.9, the amount of moisture in powders may change progressively and be affected by the storage, handling, and conditions of utilization. Thus, moisture content should only be measured at the time of interest (for example, shipping, reception, and usage). If not, evaluating how moisture and oxygen content evolve with time is recommended. This can be done by exposing the powder to humidity and evaluating how the moisture and oxygen content (using inert gas fusion method such as described in Test Method E1409) change with time. The effect of handling can be evaluated by measuring the moisture in test samples before and after a selected operation (for example, sieving, splitting). The stability of the moisture content depends on the nature and specific surface area of the powder and shall be evaluated for every material to be tested.

5.11 The optimum test temperature depends on the material and equipment used. Test conditions should be selected to recover the maximum amount of water while the AM feedstock is not modified or deteriorated. For most equipment and AM powders, the maximum temperature of the equipment is not high enough to recover all the water from the samples and the amount of water is usually underestimated.

5.12 To determine the most suitable test temperature for a specific material, tests can be performed at different temperatures from 50 °C up to the maximum temperature of the equipment. The suitable temperature can be chosen to evaporate the maximum amount of water while avoiding a modification (for example, oxidation or degradation) of the samples during the test. For some materials, it may not be possible to recover all water before the onset of the modification of the material. Consequently, the selection of the test temperature shall be selected case by case.

5.13 Results can only be compared if the tests are conducted under similar conditions (temperature, time, heating rate, gas flow rate, and end criteria) and test methods have been validated and compared with reference materials (see Section 8).

5.14 Depending on the design of the equipment, evaporation conditions (effective temperature seen by the samples, gas flow, and water extraction from the surface of the powder) may differ from one model of equipment to another. Validation of measurements using reference materials should be done before comparing results obtained in different laboratories or with different equipment or procedures to make sure they are comparable.

6. Procedure

6.1 Different methods could be used to measure moisture: Karl Fischer (coulometric titration), relative humidity sensors, and gravimetry (LOD). These methods are covered by different standards (Test Methods E1868, E1131, D7191, and D6869). For these methods, the samples need to be heated to evaporate the water. The equipment shall have a detection limit of 10 µg/g or lower. Procedures should be developed to make sure the equipment and tools are clean and dry before the sampling to prevent contamination. The following subsections describe the different methods.

6.1.1 *Coulometric Titration (i.e., Karl Fisher):*

6.1.1.1 Tests should be conducted following the equipment manufacturer recommendations.

6.1.1.2 Before filling, the empty septum bottles should be dried 1 h at a temperature higher than the test temperature. After drying, the insides of the bottles should be protected from humidity to avoid adsorption of moisture before the measurements.

6.1.1.3 The amount of water coming from the bottles should be measured on three blanks, averaged, and subtracted from the amount of water measured in the powders.

6.1.1.4 Septum bottles should be filled with the samples before the measurements.

6.1.1.5 The amount of material should be selected to have an amount of water fitting the range of sensitivity of the titration cell (see 7.5). If the order of magnitude of the amount of water is unknown, preliminary tests should be conducted to get an appreciation of the amount of water. If the amount of moisture measured in these preliminary trials is out of the range, the mass of powder should be modified to fit in the range and tests need to be reconducted.

6.1.1.6 As the samples are contained in septum bottles and the titration cell is enclosed, water from the atmosphere should not interfere with the measurements. During the transfer of powder into the bottles, care must be taken to minimize the exposure of the powder with the atmosphere. The transfer must preferably be done under a controlled atmosphere (for example, glove box, laboratory with controlled atmosphere) to avoid adsorption or desorption of water from the atmosphere, unless a demonstration has been made that the handling procedure has minimal impact on the results. This demonstration should be done using the extreme conditions encountered in the laboratory (temperature and humidity) and with each material to be tested (composition and particle size distribution).

6.1.1.7 The samples contained in a septum bottle are heated in a furnace to recover the water.

6.1.1.8 A dry gas (less than 3 µg/g water) carries the water from the septum bottle to the titration cell.

6.1.1.9 Test temperature needs to be as high as possible without deteriorating the sample.

6.1.1.10 As the test temperature has an impact on the amount of water evaporated, results can only be compared together if the tests are conducted at the same temperature.

6.1.1.11 Only the coulometric titration is appropriate to measure the low-moisture content generally present in AM powders. The moisture content is determined in the titration cell using the reaction of water with I_2 and sulfur dioxide leading to the formation of hydrogen iodide and sulfur trioxide. The extent of the $2I- \rightarrow I2 + 2e-$ reaction is evaluated by potentiometry and used to calculate the amount of water in the sample.

6.1.1.12 The volumetric method is not precise enough for AM powders and should not be used.

6.1.2 *Relative Humidity Sensor-Based Methods (see method described in Test Method D7191):*

6.1.2.1 Tests should be conducted following the equipment manufacturer recommendations.

6.1.2.2 Before filling, the empty septum bottles should be dried 1 h at a temperature higher than the test temperature. After drying, the bottles should be protected from humidity to avoid adsorption of water inside the bottle before the measurements.

6.1.2.3 The amount of water coming from the bottles should be measured on three blanks, averaged, and subtracted from the amount of water measured in the powders.

6.1.2.4 Septum bottles should be filled with the samples before the measurements.

6.1.2.5 The amount of material should be selected to have an amount of water fitting the range of sensitivity of the sensor (see 7.5). If the order of magnitude of the amount of water is unknown, preliminary tests should be conducted to get an appreciation of the amount of water. If the amount of moisture measured in the preliminary tests is out of range, the mass of powder should be modified to fit in the range and tests need to be reconducted.

6.1.2.6 During the transfer of powder into the bottles, care must be taken to minimize the exposure of the powder with the atmosphere. The transfer must preferably be done under a controlled atmosphere (for example, glove box, laboratory with controlled atmosphere) to avoid adsorption or desorption of water from the atmosphere, unless a demonstration has been made that the handling procedure has minimal impact on the results. This demonstration should be done using the extreme conditions encountered in the laboratory (temperature and humidity) and with each material to be tested (composition and particle size distribution). Once the sample is in the bottle, the humidity from the atmosphere should not interfere with the measurements.

6.1.2.7 The samples contained in a septum bottle are heated in a furnace to recover the water.

6.1.2.8 A dry gas (less than 3 µg/g water) carries the water from the septum bottle to the titration cell.

6.1.2.9 Test temperature needs to be as high as possible without deteriorating the sample.

6.1.2.10 As the test temperature has an impact on the amount of water evaporated, results can only be compared together if the tests are conducted at the same temperature.

6.1.2.11 The water content is obtained using the integration of the humidity sensor signal over the test duration from the time the specimen is introduced in the instrument and exposed to the heat source until the complete desorption of water at the set temperature (i.e., defined as a threshold desorption rate).

6.1.2.12 The tests should be considered as terminated when a variation rate threshold is obtained.

6.1.3 *Gravimetry or LOD Methods:*

6.1.3.1 Tests should be conducted following the equipment manufacturer recommendations.

6.1.3.2 In most LOD experiments, the samples are not contained in bottles. Thus, the water coming from the sample holder and the surface of the instrument may contribute to the amount of moisture measured and may affect the results.

6.1.3.3 Adequate drying of the equipment is important. The empty sample holder should be dried 1 h at a temperature higher than the test temperature and protected from humidity to avoid adsorption of water before the measurements.

6.1.3.4 Drying tests (three repeats) should be conducted with reference dry blocks before the analysis to evaporate water from the instrument. The amount of moisture measured during the last drying test should be subtracted from the results.

6.1.3.5 Samples are filled in the specimen holder.

6.1.3.6 The amount of material should be selected to have an amount of water fitting the range of the equipment (see 7.5). If the order of magnitude of the amount of water is unknown, preliminary tests could be conducted to get an appreciation of the amount of moisture. If the amount of moisture measured in the preliminary tests is out of the range, the mass of powder should be modified to fit in the range and tests need to be reconducted.

6.1.3.7 The samples are heated to evaporate the water. The temperature needs to be as high as possible without deteriorating the sample. As the test temperature has an impact on the amount of water evaporated, results can only be compared together if the tests are conducted at the same temperature.

6.1.3.8 The tests should be considered as terminated when a variation rate threshold is obtained.

6.1.3.9 The amount of moisture is evaluated by measuring the weight variations during the test. The weight loss is converted into a water content, assuming water is the only volatile coming out of the samples. Evaporation of other volatiles or weight variations coming from the degradation of the samples may affect the results.

6.2 Coulometric titration (i.e., Karl Fisher) and relative humidity sensor methods can be sensitive enough and suited to measure small amounts of moisture in AM feedstocks. Most gravimetry instruments (LOD) have been developed to monitor moisture in materials having content above 100 µg/g and should be only appropriate for powders with higher moisture contents (preferably ten times or more the detection limit of the instrument).

6.3 As tubing may adsorb moisture, all tubing used in the equipment shall conform to the equipment manufacturer recommendations.

6.4 Results may be affected by differences in the laboratory environment and handling procedures. An evaluation of the effect of laboratory environment shall be done to confirm that the test conditions have minimal impact on the results (that is, reproducibility tests). Tests may consist in exposing the sample to the laboratory environment for different periods of time to see the impact of time of exposure on the results or assessing the moisture adsorbed by a sample after it has been oven dried (before and after cooling and weighing). If comparing results obtained in different laboratories, tests should be conducted with reference materials to confirm that the conditions are comparable. As adsorption and desorption of water may depend on the material, these tests shall be conducted for every type of material to be measured (composition, particle size distribution, shape and production method).

6.5 As the relative standard deviation increases when the moisture content gets close to the detection limit of the equipment, precautions should be taken when comparing results close to the detection limit of the equipment.

7. Sampling, Test Samples, and Test Units

7.1 Samples shall be statistically representative of the feedstock to be tested. Unless otherwise agreed upon by interested parties, the sampling methods and equipment shall follow the practices for powder sampling such as Practices B215 or ISO 3954.

7.2 As water may adsorb and desorb very rapidly from the surface of the powder, the exposure of the samples to the environment shall be minimized before the measurements. Processing the powder with screens, splitters, and funnels may all affect the moisture content in the samples and should be done under controlled conditions (that is, humidity, temperature) to minimize uncontrolled adsorption or desorption of moisture before the measurements. If not done under controlled conditions, recommendations described in 5.10 should be followed.

7.3 Sample containers should be properly sealed after filling with the sample to prevent pickup of moisture before testing.

7.4 A minimum of three repeats should be used but preferably five. The average and standard deviation should be calculated and provided with the results.

7.5 The size of the sample should be adjusted to get an amount of water that fits in the range of the precision of the sensors and instrument. This size depends on the amount of moisture in the samples and the equipment used. The equipment manufacturer manual and procedures should provide tools and procedures to select the optimum size of the samples. If there is any doubt, tests could be conducted to estimate the effect of sample size on the results and select the optimum sample size (within the range where the results are not affected by the mass of the sample).

8. Calibration and Standardization

8.1 *General:*

8.1.1 Suitable instrument calibration methods and standards shall be used as specified by the instrument manufacturer.