



AEROSPACE RECOMMENDED PRACTICE

ARP7302™

Issued

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Sampling, Conformance Strategies, and Recommended Practices for
Closed Loop Processing of Powder for Additive Manufacturing

RATIONALE

New recommended practice where none have previously been released.

1. SCOPE

1.1 Purpose

This SAE Aerospace Recommended Practice (ARP) establishes methods and identifies opportunities to sample used powder feedstock circulating within closed loop equipment of an additive manufacturing (AM) process for the purpose of showing conformance to a powder specification.

Powder within the entirety of closed loop equipment cannot be represented by sampling and testing of discrete, in-process lots. Because powder processing (i.e., reconditioning, conveyance, and storage) is asynchronous with a build cycle, individual samples and their associated tests do not represent the totality of powder committed to a machine. Powder consumed as part of an individual build cycle may only represent a subset of feedstock in circulation within such equipment. Therefore, regular testing to substantiate conformance to a powder specification is required to assert conforming feedstock was consumed during individual build cycles of the AM workflow to fabricate parts or preforms.

Operation of some installations of closed loop equipment may not have sufficient control because they lack identified, monitored, and measured key process variables (KPVs). This recommended practice is intended to support such unmonitored operations where maintenance of system attributes is difficult or impossible. In such circumstances, the substantiation evidence to establish conformance of powder feedstock to its specification is required with every build cycle when producing parts.

Other specifications may establish a framework to identify and set limits on control variables to sustain system attributes required to assert continued conformance of circulating powder to requirements (e.g., controlled atmosphere and its impact on surface reactive elements). However, such a control framework as part of an aerospace process specification is out of the scope for this recommended practice.

This document also establishes terms and definitions that may be referenced in other SAE documents and specifications where closed loop equipment operation is invoked or controlled.

1.2 Application

These recommended practices should be considered when solid-state processing of used, metal powder for reuse in the AM of aerospace parts is performed within the circuit of the closed loop equipment itself. Typical applications for used powder include, but are not limited to, powder bed fusion, directed energy deposition, or binder jetting AM processes. References to laser powder bed fusion and AMS7003 as the applicable process specification are frequently made throughout the body of this document, but the recommendations made are not limited to that one process and may be made for any powder-based AM process.

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This recommended practice is limited to applications where powders to be processed are pre-alloyed or commercially pure, but not powder mixtures of different, nominal compositions (e.g., mixing of powder lots that are each different alloys).

This document applies generally to all closed loop equipment to assess powder delivered and consumed within a single build cycle but without allowances to descope the sampling and testing necessary to represent the states of future build cycles. The repeatability of these systems is not assured if control mechanisms and tracked KPVs are absent throughout the entire equipment circuit.

Sampling opportunities to collect and test powder for conformance to powder specifications are limited in closed loop equipment and may not represent the totality of powder being managed and conveyed. Therefore, this document only applies to sampling made as part of individual build cycles.

This recommended practice has notes/guidance narratives interspersed throughout. These notes/guidance narratives are identified by a [header] and by text in *italics*.

1.3 Safety - Hazardous Materials

While the materials, methods, applications, and processes described or referenced in this specification may involve the use of hazardous materials, this specification does not address the hazards that may be involved in such use. It is the responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

2. REFERENCES

2.1 Applicable Documents

The following publications form a part of this document to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this document and references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

2.1.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or +1 724-776-4970 (outside USA), www.sae.org.

AMS7002	Process Requirements for Production of Metal Powder Feedstock for Use in Additive Manufacturing of Aerospace Parts
AMS7003	Laser Powder Bed Fusion Process
AMS7025	Metal Powder Feedstock Size Classifications
AMS7031	Batch Processing Requirements for the Reuse of Used Powder in Additive Manufacturing of Aerospace Parts
AMS7032	Machine Qualification for Fusion-Based Metal Additive Manufacturing
AIR7359	Additional Guidance for Metal Powder Feedstock for Additive Manufacturing
AS7766	Terms Used in Aerospace Metals Specifications

2.1.2 ASTM Publications

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org.

ASTM B212	Apparent Density of Free-Flowing Metal Powders Using the Hall Flowmeter
ASTM B213	Standard Test Methods for Flow Rate of Metal Powders Using the Hall Flowmeter Funnel
ASTM B214	Standard Test Method for Sieve Analysis of Metal Powders
ASTM B215	Standard Practices for Sampling Metal Powders
ASTM B329	Standard Test Method for Apparent Density of Metal Powders and Compounds Using the Scott Volumeter
ASTM B417	Standard Test Method for Apparent Density of Non-Free-Flowing Metal Powders Using the Carney Funnel
ASTM B527	Standard Test Method for Tap Density of Metal Powders and Compounds
ASTM B703	Standard Test Method for Apparent Density of Metal Powders and Related Compounds Using the Arnold Meter
ASTM B822	Particle Size Determination of Metal Powders by Light Scattering
ASTM B923	Standard Test Method for Metal Powder Skeletal Density by Helium or Nitrogen Pycnometry
ASTM B964	Standard Test Methods for Flow Rate of Metal Powders Using the Carney Funnel
ASTM E203	Standard Test Method for Water Using Volumetric Karl Fischer Titration
ASTM E2281	Standard Practice for Process Capability and Performance Measurement
ASTM E2587	Standard Practice for Use of Control Charts in Statistical Process Control
ASTM F1877	Standard Practice for Characterization of Particles
ASTM F3522	Standard Guide for Additive Manufacturing of Metals - Feedstock Materials - Assessment of Powder Spreadability
ASTM F3592	Standard Guide for Additive Manufacturing of Metals - Powder Bed Fusion - Guidelines for Feedstock Re-use and Sampling Strategies
ASTM D6128	Standard Test Method for Shear Testing of Bulk Solids Using the Jenike Shear Tester
ASTM D6773	Standard Test Method for Bulk Solids Using Schulze Ring Shear Tester
ASTM D7891	Standard Test Method for Shear Testing of Powders Using the Freeman Technology FT4 Powder Rheometer Shear Cell

2.1.3 ISO Publications

Available from International Organization for Standardization, ISO Central Secretariat, 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, Tel: +41 22 749 01 11, www.iso.org.

ISO 9276 (series) Representation of results of particle size analysis

ISO 13322-1 Particle size analysis - Image analysis methods - Part 1: Static image analysis methods

ISO 13322-2 Particle size analysis - Image analysis methods - Part 2: Static image analysis methods

ISO/ASTM 52900 Additive manufacturing - General principles - Fundamentals and Vocabulary

ISO/ASTM 52907 Additive manufacturing - Feedstock materials - Methods to characterize metallic powders

2.1.4 U.S. Government Publications

Copies of these documents are available online at <https://quicksearch.dla.mil>.

MIL-PRF-23950 Aluminum Powder, Spherical

2.1.5 Other Publications

Jacob, G., Brown, C., and Donmez, A. (2018). *The Influence of Spreading Metal Powders with Different Particle Size Distributions on the Powder Bed Density in Laser-Based Powder Bed Fusion Processes*. NIST Advanced Manufacturing Series 100-17. <https://doi.org/10.6028/NIST.AMS.100-17>.

Saleh, K., Golshan, S., and Zarghami, R. (2018). A review on gravity flow of free-flowing granular solids in silos - Basics and practical aspects. *Chemical Engineering Science*, 192, 1011-1035. <https://doi.org/10.1016/j.ces.2018.08.028>.

2.2 Definitions

The following definitions apply, in addition to those defined in ISO/ASTM 52900, AMS7002, and AS7766:

BUILD CYCLE BATCH: The volume of powder committed to a single build cycle of an AM process. This powder is exposed to the process and partially consumed to produce parts, with the remaining potentially recovered for downstream reconditioning.

CLOSED LOOP EQUIPMENT: The machinery needed to prepare used powder for reuse (refer to AMS7031) while maintaining a closed loop process. Differences between batch and closed loop processes include: the processing equipment is commonly sealed to maintain a controlled environment and one or more insertion/extraction points will be present for inserting new powder or extracting parts/powder.

CLOSED LOOP PROCESS: A powder reconditioning, conveyance, and storage system wherein used powder generated from an additive manufacturing build cycle is processed such that the prepared reuse powder is continuously supplied for the production of parts.

OPEN LOOP PROCESS ("Open Loop"): A powder reconditioning, conveyance, and storage system wherein used powder generated from an additive manufacturing build cycle is processed as a machine batch (refer to AMS7031), such that the prepared reuse powder is supplied for the subsequent production of parts.

POWDER CAPSULE: *In situ* container fabricated concurrently with a build cycle to collect and store powder that is a near representation of the powder consumed during the build cycle used to fabricate it. Alternate language such as "powder coffin," "powder ampoule," "powder containment vessel," "powder crucible," etc., are commonly used in practice.

POWDER LOT: Refer to AMS7002.

POWDER SEGREGATION: The act of separating constituents of powder based on a characteristic. The results of powder segregation may be a non-heterogeneous distribution with a gradient of the characteristic (e.g., mechanical agitation buoying larger particles to the top of a powder column) or intentional removal of a single kind of constituent (e.g., sieving powder to remove oversized particles).

POWDER STRATIFICATION: Distinct layers of powder within a single powder column that are identifiably different by a characteristic (e.g., relative to process exposure reuse powder and virgin powder lots).

PREPARING USED POWDER FOR REUSE: A sequence of activities that involve the reconditioning of recovered powder, the possible blending of this processed material, as needed desorption of accumulated moisture or volatile organics, and inspection for comparison to a material specification before its use in the manufacture of new parts.

RESIDUAL POWDER: Powder that remains along the closed loop equipment circuit despite the machine's proper operation to convey or process such powder into a downstream storage or build cycles.

UNCONTROLLED POWDER: Any lot or batch of powder where there is no traceability back to the original virgin powder lot. This does not necessarily represent powder out of conformance to a powder specification.

USED POWDER: Powder that has been exposed to at least one build cycle and represents a change from the virgin powder state.

Existing terms (refer to AMS7031):

MACHINE BATCH: A subset of a powder that is fully contained and traceable to a single AM machine without in-process additions from the originating source lot of powder, virgin material of a different lot, or blended with a different lot of powder of a different historic process history. This designation is typically used to clarify the subdivision of a single source lot of feedstock amongst many AM machines of the same type.

POWDER BLENDING: The process of combining powder of the same nominal size, chemistry, and physical attributes in such a way that the characteristics of the powder are uniform throughout. The powder to be blended may be from a single machine or multiple machines, originating from a single or set of nominally similar lots of feedstock and with similar process history. Powder blending is limited to powder lots, in-process and/or virgin, where each constituent conforms to the same specification for all compositional and morphological requirements.

POWDER MIXING: The process of combining two or more different batches (or lots) of powder with distinctly different chemical compositions, original particle size ranges, AM process histories, or other physical characteristics in such a way that makes the resultant mixture homogenous and uniform throughout. Powder mixing describes the combining of lots of powder, in-process and/or virgin, where each constituent is described by powder specifications with different (uncommon; not shared/overlapping) compositional and morphological requirements.

NOTE: Powder mixing is not allowed in this specification as inhomogeneities, potentially detrimental to properties, may not be detectable through bulk testing alone. The controlled process history requirements of powder blending are intended to improve consistency.

POWDER RECONDITIONING: The process of altering the characteristics of powder in such a way as to make the powder acceptable for use as feedstock in a powder-based AM process. This typically involves changing physical or chemical attributes of the powder volume on the whole or by removal of a subset of particles (e.g., sieving of oversized particles). Attributes that may be modified include particle size distribution, particle morphology, surface chemistry, and retained moisture. This is not an exhaustive list.

RECOVERY OF USED POWDER: The process of physically removing, collecting, and transporting powder that has been exposed to the build environment of an additive manufacturing machine. The powder is collected from within the build volume, overflow region, or external powder removal equipment. This collected powder is transported to a closed vessel or onto subsequent processing equipment. The means by which the powder is handled may either be using hand tools, pneumatic suction, or by some other mechanized and/or automated method.

RECOVERY OF VIRGIN POWDER: The process of physically removing and transporting virgin powder from within an additive manufacturing machine to a closed vessel or onto subsequent processing equipment. The means by which the powder is handled may either be by hand tools, pneumatic suction, or by some other mechanized and/or automated method.

Unless otherwise defined in the powder handling and storage plan of the additive manufacturing process, virgin powder is synonymous with unused powder and has, at minimum, not been exposed to a build cycle of the additive manufacturing machine. Additional requirements on environmental exposure or control for contamination may be made at the discretion of the CEO in order to maintain the powder status.

NOTE: In some critical applications, powder that has been committed to a machine's hopper or reservoir may no longer be considered virgin powder. Any deviations from the definition of virgin powder as described by ISO/ASTM 52900 must be identified and described in a process control document and agreed upon by the CEO.

REUSE POWDER: Used powder that has seen, at minimum, a single build cycle, recovered from an additive manufacturing process, reconditioned as necessary to modify its physical or chemical attributes, and potentially blended with similar lots of virgin or used powder to be prepared for use as feedstock in the same additive manufacturing process from which it originated.

2.3 Acronyms

AM	additive manufacturing
CEO	cognizant engineering organization
KPV	key process variable
PCD	process control document

3. CLOSED LOOP PROCESSES AND CONFORMANCE TO POWDER SPECIFICATION EXPECTATIONS

A closed loop process introduces additional complexity and uncertainty when managing powder feedstock quality control. The efficiencies of automating actions such as powder recovery, reconditioning, blending, and desorption are a compromise with powder lot pedigree and traceability. These attributes are otherwise maintained in open loop or batch processing methods but are both time- and labor-intensive. In the case of open loop processes, powder is collected and treated volumetrically. Every step is performed for all powder within the operator's custody and therefore maintains a consistent and complete history with clear boundaries. With many closed loop equipment configurations and operational methods, powder intermingling throughout closed loop equipment operation will invariably result in the consumption of powder with various pedigrees and process exposure histories. It is practically impossible with existing machine design to determine volumetric and spatial contributions when multiple lots of powder are committed to a closed loop circuit. There is a strong bias to using a single lot of material when operating closed loop equipment; however, this is all but impractical in long-term operations and/or large format machines. In instances when powder bed processes require conformance to a powder feedstock specification, the clear, evidential basis to substantiate conformance a priori using multiple powder lots is quickly lost. Nevertheless, the opportunity for closed loop processes is that a lack of such evidence does not necessarily equate to a nonconforming condition of powder feedstock or a nonconforming part or preform produced from closed loop AM processes.

For the purpose of this ARP, not all closed loop equipment maintains sufficient sensing capabilities or controllable process variables to verify the repeatable execution of preparing used powder for its reuse. To provide opportunities for these configurations to produce aerospace parts, this document describes methods to sample powder to substantiate conformance to a powder specification but at the frequency of each build cycle that is performed. This scenario for unmonitored equipment typically describes either older or custom machine equipment that still affords efficiencies through automation but requires verification and validation toward powder specification conformance.