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Christopher U. Brown
Alkan Donmez

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# Microstructure Analysis for Additive Manufacturing: A Review of Existing Standards

Christopher U. Brown
*Systems Integration Division*
*Engineering Laboratory*

Alkan Donmez
*Intelligent Systems Division*
*Engineering Laboratory*

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## Introduction

Metal-based additive manufacturing (AM) offers unique possibilities to fabricate metal parts with complex structures. Due to the very rapid melting and solidifying of thin layers of metal at a time, the nature of the material resulting from the AM process has unique characteristics. The National Institutes of Standards and Technology (NIST) project *Characterization of Additive Manufacturing Materials* aims to develop new methods to characterize additively manufactured materials or to expand existing methods applicable for other materials.

Understanding the microstructure of AM materials, i.e. parts made by AM processes, is important for industry to have confidence in AM parts. The focus of this report is to assess the existing microstructure standards for metals to determine if the existing standards are sufficient for characterizing AM metals.

## Scope

Microstructures of all materials, including additively manufactured materials, are significantly affected by various process parameters, manufacturing strategies, post process heat treatments, and environmental conditions, resulting in varying mechanical and material properties. Traditional metallography techniques are used to quantify these microstructures to better understand the effects of multiple factors. The purposes of this investigation are to review existing standards describing the methodologies for microstructure characterization of metallic materials, consider their applicability to additively manufactured metal alloys, and identify, if any, new standards addressing unique characteristics of such AM materials. This report provides a summary of this review and provides recommendations for applying the existing and potential future standards to additively made materials.

In order to perform an assessment of the existing standards related to metal microstructure and determine their applicability to AM metals, a number of standards were reviewed. ASTM and ISO standards were primarily surveyed. Since there are multiple features describing the metal microstructure, a summary is provided of the standards related to each feature with increasing level of microstructure, organized from a micro-level to a more macro-level. Most techniques to analyze metal microstructure require some type of imaging of the specially prepared sample surface. The imaging techniques found in the reviewed standards are optical microscopy, Scanning Electron Microscopy (SEM), x-ray diffraction (XRD), and electron backscatter diffraction (EBSD). There are also non-destructive evaluation techniques found in the standards for characterizing microstructure, which are also summarized in this report.

Microstructures of metallic materials include the following features:

1. Crystal structure - lattice parameters
2. Grain size and grain boundaries
3. Orientation effects - texture
4. Material phases - volume fraction of phase constituents
5. Material phases – precipitates and inclusions
6. Voids, microcracks and delaminations

Microstructure characterization methods require sampling of parts or specific test specimens using special preparation methods. Such preparation methods may include cross-sectioning, grinding, polishing, and etching. There are specific standards describing these methods. These standards are summarized in Table 1.

**Table 1. Summary of standards describing methods to characterize material microstructural features**

| Microstructural features | Relevant standards |
|---|---|
| Crystal structure - lattice parameters | No standards found |
| Grain size and grain boundaries | ASTM E112, ASTM E930, ASTM E1181, ASTM E1382, ASTM E2627, ISO 643, ISO 2624, ISO 4499-2, ISO 14250 |
| Orientation effects - texture | ASTM E82 |
| Material phases - volume fraction of phase constituents | ASTM E562, ASTM E975, ASTM E1245, ASTM E1268, ISO 9042 |
| Material phases – precipitates and inclusions | ASTM E45, ASTM E1245, ASTM E2142, ASTM E2283, ISO 4967 |
| Voids, microcracks and delaminations | ASTM E3, ASTM E165, ASTM E543, ASTM E1208, ASTM E1209, ASTM E 1210, ASTM E1219, ASTM E1220, ASTM E1418, ASTM E2297, ISO 4505, ISO 4494-4, |

In general, all of the following standards appear to be capable of addressing AM manufactured materials. Therefore, we will not address the applicability how each individual standard applies to AM materials. Rather, we will discuss in the conclusion a few specific concerns that the AM community should consider when applying existing microstructure standards to AM metals.

## 1. Crystal structure - lattice parameters

A crystal structure is the three dimensional ordered arrangement of atoms, ions, or molecules in a solid material. The crystal lattice is the array of sites where atoms sit, repeating in all directions that represents the crystal structure. The unit cell is the smallest unit of volume that contains all the structural and symmetry information to build the solid structure. Lattice parameters include the length along the edge of a unit cell and the angles of the sides of the unit cell. The crystal structure can be described by its unit cell. A unit cell may contain one or more lattice points, and each lattice point may contain one or more atoms. Three common cubic crystals include the simple cubic, body-centered cubic (BCC), and face-centered cubic (FCC) structures.

There does not appear to be microstructural metal standards that address how to characterize structural features at this level. Per the ASM Handbook (Metals Handbook 9$^{th}$ Edition, ASM), a common instrument that is often used is the transmission electron microscope (TEM) to image lattice defects and precipitates. The TEM technique uses a beam of electrons passing through a thin specimen. The electron beam interacts with the specimen to form an image or pattern which is captured on a screen or detector (Ruhle, 1985).

## 2. Grain size and grain boundaries

Grain boundaries are defects in the crystal structure and the interfaces where crystals of different orientations meet. Grain boundaries can disrupt dislocations through a material so that reducing the grain size can improve strength. Crystals, or grains, form during the cooling of materials. Their degree of orientation is referred to as texture. A grain boundary is the interface at which two grains meet. A grain boundary is a single phase interface, where the crystals on either side are identical except with respect to orientation. Grain boundaries may contain atoms moved from their original lattice site, dislocations, and impurities that have migrated to a grain boundary to lower its energy. The higher energy associated with an interfacial excess energy of grain boundaries makes them a potential site for the nucleation of new phases from the solid.

There are several standards that are related to techniques and instruments for grain size measurements. Several ASTM and ISO standards discuss methods to determine various measurements of grain size, including average grain size (ASTM E112, ISO 643, ISO 4499-2, ISO 2624, ASTM E1382, and ASTM E2627), largest grain size (ASTM E930), and characterizing duplex grain sizes (ASTM E1181, ISO 14250). “Duplex grain sizes” are defined as a distribution of grain sizes that are not a single log normal distribution after applying the measurement methods defined in ASTM E112.

ASTM E112 and ISO 643 focus on characterizing grain size using a reflected light microscope using three different procedures: comparison to standard charts to measure grain size, determining the number of grains per unit area, and intercept of grains or grain boundaries with a straight line of known length. These procedures apply to the single phase grain structures but can also be applied to determine the average size of a particular grain structure in a multiphase or multi-constituent specimen.

ASTM E1382 describes a test method for determining average grain size using a reflected light microscope and semiautomatic and automatic image analysis. Grain size measurements are determined from the intercept of a straight line of known length and individual grains and grain boundaries, measurements of grain boundary lengths and grain areas. The selection of specimen orientation and location is important and discussed in detail in ASTM E3.

ASTM E2627 describes how to determine average grain size using electron backscatter diffraction (EBSD). EBSD involves placing a crystalline specimen in a scanning electron microscope (SEM). Electrons are diffracted back out of the specimen and strike a phosphor screen producing a pattern of intersecting bands. From this diffraction pattern, crystal structure and orientation can be determined as a function of position on the samples surface.

A standard test method for estimating the largest grain observed in a metallographic section using a microscope is described in ASTM E930. However this procedure is intended for microstructures containing outlier coarse grains, where their population is too sparse for grain size determination by ASTM E112.

ASTM E1181 is a test method for the specific case of determining if a duplex grain size exists. ISO 14250 also addresses characterizing duplex grain sizes. The use of the term duplex does not imply that only two grain size distributions exist. The test methods separate duplex grain sizes into two classes (randomly varying, and topologically varying), and define specific types of duplex grain sizes within these classes. Examples of randomly intermingled grain sizes include: isolated coarse grains in a matrix of much finer grains, extremely wide distributions of grain sizes, and bimodal distribution of grain size. Examples of topological duplex grain sizes include: systematic variation of grain size across the section of a product, necklace structures, banded structures, and grain growth in selected areas of critical strain.

## 3. Orientation effects – texture

Texture is the distribution of the crystallographic orientations of the grains that make up a microstructure. A material with a random distribution in the orientation of its grains is classified as having no texture. A substance with a certain degree of orientation is classified as having weak, moderate or strong texture.

ASTM E82/E82M describes the back-reflection Laue procedure used to determine the orientation of a metal crystal. An incident beam from the x-ray source passes through a pinhole aperture, strikes the specimen crystal structure, and is diffracted back to the detector located between the x-ray source and the specimen. The detector shows a pattern of white spots which are the x-ray beams diffracted by the atomic planes within the specimen crystals. The pattern of white spots and their positions in space are used to compare to standard stereographic projections of crystals to determine the crystal orientation.

## 4. Material phases - volume fraction of phase constituents

Determining a volume fraction of a constituent or phase by systematic manual point count method is described in ASTM E562 and ISO 9042 using a light microscope or SEM with a point grid. Two common grid arrangements, a circular or square array, are typically used. Although this is a manual method, similar characteristics can be determined using the automatic method described in ASTM E1245. A test grid is superimposed over an image produced by a light microscope, SEM, or micrograph. The number of test points is counted that fall within the phase or constituent of interest. Samples selected should be representative of the general microstructure at a specified location within a lot, heat, or part.

Volume percent of retained austenite phase in steel using integrated intensities (area under the peak above background) of X ray diffraction peaks using chromium and molybdenum x ray radiation is described in ASTM E975. The method applies to carbon and alloy steels with near random crystallographic orientations of both ferrite and austenite phases. Retained austenite with a near random crystallographic orientation is found in the microstructure of heat-treated low-alloy, high-strength steels that have medium or higher carbon contents. Although retained austenite may not be evident in the microstructure, its presence can have significant effects on material and mechanical properties, performance during service, wear resistance, and corrosion behavior.

ASTM E1268 describes procedures to qualitatively characterize the appearance of banded structures, procedures for characterizing the extent of banding, and a micro-indentation hardness procedure for determining the difference in hardness between bands in heat treated specimens. Banding is the segregation of elements into distinct layers during solidification. Microstructural banding or orientation can affect the mechanical properties of a material. These methods are described to measure the number of bands per unit length, the inter-band or inter-particle spacing and the degree of anisotropy or orientation. Stereological measurements are made by superimposing a test grid on a projected image from a reflected-light microscope.

## 5. Material phases – precipitates and inclusions

Inclusions are foreign particles that may be contained in the grain boundaries between grains, may be non-metallic particles, and can affect the mechanical properties of a solid. Characterizing the inclusion content in a material is therefore important. There are several standards that describe methods to count different types of inclusions. Precipitates are secondary phases within a microstructure, and are often used as a strengthening mechanism. Typically the precipitates will increase a metals strength or hardness by preventing the movement of dislocations or defects in a crystal's lattice structure.

ASTM E45 describes test methods for determining the nonmetallic inclusion content in wrought steel or other alloys. Methods using a light microscope characterize inclusion size, shape, concentration, and distribution, rather than composition on a polished specimen surface (see ASTM E175 for standard terminology for microscopy). Inclusions are first classified into four types based on their shape (not their chemical composition despite the Type names): Type A – Sulfide Type, Type B – Alumina Type, Type C – Silicate Type, and Type D – Globular Oxide Type. Secondly, the inclusions are categorized as 'thin' or 'heavy' based on thickness or diameter. Thirdly, the inclusions are categorized by severity level which is based on the number or length of the particles present in a 0.50 mm x 0.50 mm field of view. ISO 4967 includes these same four types but also has a fifth group, Group DS – Single globular type, a circular particle with diameter greater than 13 μm.

A disadvantage of this process is that the measurement field size (0.5 mm x 0.5 mm) is small so that many fields may need to be measured to accurately represent the specimen. Only the specimen area examined is represented by the rating since steel often differs in inclusion content not only from heat to heat, but from ingot to ingot in the same heat and in different portions of the same ingot. For wrought steel, the sampling should not be done larger than one heat.

ASTM E1245 is similar to ASTM E45 but uses auto image analysis with a reflected light microscope or SEM and an auto image analyzer to measure basic characteristics of the morphology of inclusions in steel or other metals. Measurements include the number of sulfide and oxide inclusions per unit area, area of each inclusion type, and mean distance between inclusions. Inclusions are detected with grey level intensity (see ASTM E986 for beam size characterization).

ASTM E2142 covers procedures to obtain particle size distribution, chemical classification, and ASTM E45 ratings of inclusions in steels using automated SEM with X-ray analysis and automatic image analysis. There are three methods described. Method 1 is equivalent to ASTM E45 but the SEM characterizes morphology and is used to sort particles into classes. Method 2 is based on sorting inclusions by chemistry into traditional classes defined in ASTM E45. Method 3 defines procedures to analyze and report inclusions by arbitrary size distribution and chemical classification.

ASTM E2283 describes a standard practice to estimate the extreme value distribution of inclusions or second phase constituents in steels. Generally the largest oxide inclusions within the specimens are measured but the method can be used to measure other microstructural features. This practice is not suitable for assessing the exogenous inclusions in steels and other metals because of the unpredictable nature of the distribution of exogenous inclusions. This practice is based on using the specimens described in ASTM E45.

## 6. Voids, microcracks and delaminations

The amount of porosity, voids, or pores within a microstructure, cracks or delaminations, possibly between build layers, is important to characterize for AM materials. Some AM materials need to be fully dense for high strength purposes, while other materials require surface porosity. Surface porosity and surface roughness are not covered in this report.

There are at least two general types of methods to evaluate a microstructure for porosity, cracks or delaminations: destructive and non-destructive evaluation (NDE). Destructive evaluation involves cutting a specimen from the main body of the material in order to view a representative sample. ASTM E3 and ISO 4505 describe selection, size, cutting, cleaning, mounting, grinding, and polishing of metallographic specimens for microstructure analysis using an optical microscope or SEM. Many of the standards mentioned already address characterizing features of interest from a cut specimen using either the optical microscope or SEM. ISO 4499-4 and ISO 4505 include reference photomicrographs with which to characterize porosity levels.

NDE involves determining material properties without causing damage to the material. Several factors are important when considering which NDE method to use for detection, for example, the material being examined, or what type of material properties are of interest. ASTM E543 has an appendix listing different NDE methods and the material properties measured. It is a standard useful for NDE testing on any material. It also includes typical flaws detected along with advantages and disadvantages of each technique. ASTM E543 serves as a good reference by including a list of ASTM standards related to each NDE method.

A few of the ‘typical discontinuities detected’ listed in ASTM E543 include voids, the absence of material or a separation not caused by loading or internal stress. Another discontinuity listed is cracks, the separation of the material in any direction usually due to external loading or internal stress. Delaminations, the separation between layers due to external loading or internal stress, are also listed as discontinuities in ASTM E543.

Another type of NDE method involves using fluorescent or visible penetrant and developer materials to visually observe the presence and location of discontinuities open to the surface of metallic materials. ASTM E165 is a good starting point for these methods and lists several ASTM standards describing fluorescent and visible penetrant processes for metals (E1208, E1209, E1210, E1219, E1220, E1418, E2297).

**Sample preparation (cutting, polishing, etching)**

Since the primary objective of examining microstructure metallography is to reveal constituents and structure of metals and their alloys by means of optical microscope or SEM, proper preparation is of major importance. ASTM E3 provides a guide for preparation of metallographic specimens. It recommends a specimen location based on the purpose of the study: for general studies or routine work specimens may be taken to reveal the maximum material variations or from the part ends; for the study of failures, specimens may be taken as close as possible to the fracture or initiation of failure; and for research studies, the nature of the study will dictate the specimen location. Once the specimen location is determined, the specimen orientation(s) is chosen: longitudinal, transverse, and/or parallel to the surface. Three recommendations are given to cut metallographic specimens, sawing, abrasive cut-off blade, or a shear, tools that minimize altering the sample structure. Suggestions for specimen size, cleanliness, mounting the specimens, and suggestions on manual and automated grinding and polishing are also described. ASTM B665 and ISO 4499-1 also describe metallographic preparation for cemented tungsten carbides or other hardmetals.

Etching specimens for metallographic examination is important to highlight phases of interest. ASTM E407 contains tables of metals with etchants to highlight specific phases for microscopic evaluation. Also included are the etchant composition and procedure.

Inclusion ratings, done manually using ASTM E45 or automatically using ASTM E1245 are influenced by the quality of the specimen preparation. ASTM E768 provides examples of proven specimen prep methods that retain inclusions in polished steel specimens. It is also a guide to determine if the prepared specimens are of suitable quality for subsequent inclusion ratings. Two preparation methods are discussed for steel metallographic specimens that will be analyzed for nonmetallic inclusions with automatic image analysis (AIA) (Silicon Carbide Procedure and the Rigid Grinding Disk Procedure).

**Conclusion and next steps**

Most of the standards reviewed in this report address characterizing metal microstructural features that are not process specific. These standards therefore appear to be capable of addressing metals made by AM processes. There are some unique aspects of AM materials that may require attention when applying existing microstructural standards. One of these is that the additive manufactured layered build process, such as the layered process used in ‘laser-powder-bed-fusion’ (LPBF), may result in anisotropic material properties. The anisotropic nature will require that manufacturers take additional notes when preparing, sectioning, and during microstructural analysis such that specimen direction is always reported and that microstructure analysis is done in different directions, e.g., parallel and perpendicular to the build direction (ASTM F2971, ASTM F3122, ISO/ASTM 52921).

Another unique aspect of LPBF AM materials is their microstructure based on the AM process of solidifying powder layers to form a part. A result of this layered process can be weaker interfaces between layers leading to delamination. Since delamination is one of the major weaknesses of laminated composites, there are standards that address delamination and crack growth resistance for layered composite materials (D5528, D6115, D6671, E1922, ISO 15024, ISO 15114, ISO/DIS 19927). For metals however, standards exist for crack growth resistance (E399, E1820, E1304, ISO 12135) but not for the unique AM case where the metallic material has been formed in layers.

Microstructure analysis including preparation of metal samples is often a 'trial and error' process. Even minor structural differences can occur among metal samples taken from the same bulk metal section. Existing standards describe general methods or recipes to serve as a starting point to perform microstructure analyses. Metal specimens are prepared mostly based on reference books and past successful personal laboratory practice. The customized nature of this work is well known to metal microstructure experts, but not clear to the AM community trying to understand how to apply traditional techniques to AM metals. A 'best practice' report for using existing standards to characterize the microstructure of AM materials may be the next appropriate step. However, as microstructure analysis continues on AM metals, a need for a specific standard may arise.

As many of the existing standards involve manual or image analysis methods to make measurements, sample selection is important as noted in many standards (E3, E45, E562). A sample should be representative of the general microstructure, or representative of a general location within a lot, heat, or part. Care will be necessary that selected AM samples have a representative microstructure for analysis. Attention to the number of samples, their location, and direction is important to adequately represent a specimen just as it is required for non AM metals analysis. However, unlike traditional metal analysis, AM metal samples will not be categorized by heat or ingot.

The above standards address *how* to assess microstructure or *what* to assess, however, ideas for future AM related standards include *when* or *where* to assess microstructure. A few ideas include:

- A standard recommending *where* along the z axis (height) to analyze, either destructively or non-destructively. For example, at different heights during the AM fabrication process, the microstructure may change depending on the height of the layers. Powder characteristics or laser intensity may also be affected by the height of the build.

- Defining *where* on an AM part to analyze in the x and y axes, either destructively or non-destructively, since powder or laser intensity characteristics may also change depending on these directions within an AM part.

- A standard recommending how frequently microstructure analysis should be assessed. Every AM part could have a complimenting microstructure specimen fabricated next to the part for analysis, or a microstructure specimen could be analyzed after a specific number of AM parts are fabricated.

- A standard recommending a template for the AM part manufacturer/buyer listing possible expectations to be discussed to include how, when, where, and what microstructure assessments will be conducted and by whom.

- A standard recommending how to present microstructure results consistently, and with enough pedigree to provide adequate records of the data.

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