



Tutorial review

Microstructure characterization via stereological relations – A shortcut for beginners



Willi Pabst*, Eva Gregorová, Tereza Uhlířová

Department of Glass and Ceramics, University of Chemistry and Technology, Prague (UCT Prague), Technická 5, 166 28 Prague, Czech Republic

ARTICLE INFO

Article history:

Received 2 February 2015

Received in revised form 7 April 2015

Accepted 8 April 2015

Available online 14 April 2015

Keywords:

Stereology

Mean chord length (mean intercept length)

Jeffries grain size

Grain size numbers (linear planar)

Delesse–Rosinlaw law

Saltykov relation

ABSTRACT

Stereological relations that can be routinely applied for the quantitative characterization of microstructures of heterogeneous single- and two-phase materials via global microstructural descriptors are reviewed. It is shown that in the case of dense, single-phase polycrystalline materials (e.g., transparent yttrium aluminum garnet ceramics) two quantities have to be determined, the interface density (or, equivalently, the mean chord length of the grains) and the mean curvature integral density (or, equivalently, the Jeffries grain size), while for two-phase materials (e.g., highly porous, cellular alumina ceramics), one additional quantity, the volume fraction (porosity), is required. The Delesse–Rosinlaw law is recalled and size measures are discussed. It is shown that the Jeffries grain size is based on the triple junction line length density, while the mean chord length of grains is based on the interface density (grain boundary area density). In contrast to widespread belief, however, these two size measures are not alternative, but independent (and thus complementary), measures of grain size. Concomitant with this fact, a clear distinction between linear and planar grain size numbers is proposed. Finally, based on our concept of phase-specific quantities, it is shown that under certain conditions it is possible to define a Jeffries size also for two-phase materials and that the ratio of the mean chord length and the Jeffries size has to be considered as an invariant number for a certain type of microstructure, i.e., a characteristic value that is independent of the absolute size of the microstructural features (e.g., grains, inclusions or pores).

© 2015 Elsevier Inc. All rights reserved.

1. Introduction

Almost all solid materials around us, whether natural or man-made, are heterogeneous in the sense that they contain – apart from the external surface of material bodies – internal interfaces. Among the few exceptions, i.e., examples of materials that are not necessarily heterogeneous, are monocrystals and some amorphous materials (e.g., glass). The internal interfaces can be just grain boundaries between crystallites of different orientations, as in the case of dense, single-phase polycrystalline materials, or phase boundaries, i.e., interfaces between different phases (in multiphase materials, i.e., composites), including solid–fluid interfaces and free surfaces, as in the case of porous media.

The macroscopic overall properties of heterogeneous materials are called effective properties [1]. They include mechanical properties such as elastic constants, thermal properties (e.g., thermal conductivity), thermomechanical properties (e.g., thermal expansion coefficients) and many others, including electrical, magnetic and optical properties and combinations of the latter three with mechanical or thermal properties. Most of these properties are microstructure-sensitive [2], i.e., they depend not only on the chemical composition, but also on the phase composition and all microstructural details.

It is clear that modern materials science and engineering, in contrast e.g., to some more traditional fields like geology or sedimentology, requires not only a qualitative description, but also a quantitative characterization of microstructural features. Only on the basis of quantitative microstructural information it is possible to provide quantitative estimates of the effective properties in the form of bounds or model predictions [1–3]. Such quantitative microstructural information can principally be obtained using probes of different dimensionalities. The most sophisticated tool available for probing is of course tomography, a 3D probe technique [4] with a strong theoretical foundation [5], or its real time variant that may even record the temporal evolution of microstructures and thus would correspond to a “4D probe” [4]. Currently, X-ray computed tomography (X-ray CT) equipment with micron- and submicron-resolution is available [4]. Thus, in principle, the voxel information contained in tomographic images can be directly used today as a data input into software for the numerical calculation of effective properties [6,7]. Nevertheless, although the spatial resolution of X-ray CT is attacking the few-hundred-nm range, there are intrinsic resolution limits to this method that make the use of electron microscopic techniques based on planar sections indispensable in many cases.

Moreover, it has always been a principal aim of engineering science to reduce the amount of information available and to extract certain key parameters which are of major importance for the microstructure and the values of which may lend themselves to analytical calculations of

* Corresponding author.

E-mail address: pabstw@vscht.cz (W. Pabst).

effective properties, because this is a precondition of a physical understanding of microstructure–property relations. However, this is not easily done with the enormous amount of data obtained from tomography. It has to be emphasized that for materials science – in contrast e.g., to medicine – it is not the primary aim of tomography to obtain nice 3D pictures of the internal microstructure, but to obtain global microstructural parameters (descriptors) that are not accessible by lower-dimensional probes, i.e., topological parameters (e.g., the number of objects per unit volume N_V or the 3D Euler characteristic that connects this parameter with the connectivity or genus per unit volume C_V), and correct size and/or shape distributions of 3D objects. In any case a meaningful reduction of information of 3D voxel images, i.e., the extraction of global microstructural descriptors and their correct interpretation requires a thorough understanding of the underlying procedures used with lower-dimensional probes. And last but not least, a more subtle – but not less important – point that will become clear below concerns the errors of measurement. In fact, it is not easy to provide realistic estimates of expected errors for measuring techniques, especially in the case of 3D probes, while for counting techniques, especially in the case of 0D probes (point probes), but also for 1D probes (line probes) and 2D probes (planar sections), this is much easier.

Stereology can generally be defined as the science or art of quantitatively characterizing 3D microstructures on the basis of lower-dimensional probes. Of course, not all microstructural parameters (descriptors) can be obtained in this way. Those that can, are called “metric descriptors”, in contrast to “topological descriptors”, which are inherently 3D parameters and cannot be determined from lower-dimensional probes (e.g., planar sections). If the values of these descriptors refer to the whole material body they are called “global”, otherwise “local”. Only in the case of uniform bodies (i.e., materials without gradients) the values of global and local descriptors are statistically equivalent. Examples of (global) metric descriptors are the phase volume fractions of multiphase materials and certain measures of average grain size (in the case of polycrystalline materials) or (in the case of multiphase materials) the average size of phase regions (e.g., inclusions or pores in the case of composites or porous media, respectively), i.e., in general a characteristic length of the microstructural features (length scale of microstructural heterogeneity).

The historical roots of stereology can be traced back at least to the 17th and 18th century (Cavalieri’s principle of 1635 [8,9], “Buffon’s needle problem” of 1777 [10]), and the development of this discipline is closely related to geometry and statistics, but its first practical application to materials (natural rocks) is by Delesse [11,12] and Rosiwal [13] in the 19th century (1847 and 1898, respectively), who used area fractions of planar sections (i.e., 2D probes) and line fractions on these (polished) sections (i.e., 1D probes), respectively, for determining the volume fraction of mineral phases in rocks. However, it took more than three decades from Rosiwal’s proposal to use line fractions instead of area fractions, until it has been finally recognized by Thompson in 1930 [14] and Glagolev in 1932 [15] that also point fractions (i.e., 0D probes) can be used for this purpose [16]. The latter discovery was the key to further developments, because it enabled for the first time a rigorous statistical treatment of the problem and thus the exact prediction of expected errors before the measurement [17,18]. The historical development of the whole discipline of stereology is very interesting [19–22], but a detailed account would exceed the scope of this paper.

Despite the great practical significance of stereology both for the correct evaluation of 2D planar sections and the correct interpretation of 3D tomographic results, the knowledge of this discipline among materials scientists and engineers is often not adequate. This may be in part due to the widespread – but unjustified – belief that today’s tomography technologies make stereology obsolete, and in part because some of the best works on stereology are not easily accessible to the practicing engineer [23–26]. On the other hand, the early classics in the field of applied stereology [19,27,28] would urgently require updating in order to take into account more recent developments in the field. Actually many of

the currently available less rigorous treatments contain traditional errors and misconceptions, while the more rigorous treatments evoke the impression, that without abstract set theory, Boolean algebra, differential geometry, graph theory and stochastics it makes no sense to indulge into this field at all. As a result, although stereology seems not to lose popularity and continues to be used by many authors, and actually a few nice papers can be found in the modern literature [29–38], most current papers in the field of materials science and engineering, which claim to apply stereological relations as a routine tool, do not take full advantage of the possibilities of this method and do not extract the maximum information out of microscopic studies. This state of affairs is actually regrettable, because as soon as one is willing to accept the highly non-trivial mathematical theorems behind stereology as matters of fact, it is not too difficult and is much more satisfying to apply them correctly and fully instead of incorrectly and only partially, as is common practice.

This paper attempts to convince the reader – with two extreme examples (a dense material and a highly porous material) – that stereology is neither obsolete nor should it be applied in a too thoughtless way, by e.g., just naively using the recipes given in standard norms [39]. In particular, it should be emphasized that up to now there has been no serious attempt to update the international standards with respect to current knowledge in this field. This will become especially clear below when treating the direct measures of grain size (mean chord length, Jeffries grain size [40]), their phase-specific equivalents for multiphase materials, and indirect grain size measures (so-called grain size numbers).

The present paper is organized as follows: Section 2 recalls the Delesse–Rosiwal law for the determination of phase volume fractions, including the conditions under which it can be applied and the calculation of expected and observed errors in the special case of two-phase materials. Section 3 explains the interface density, the grain size measure related to it (mean chord length), the corresponding phase-specific size measure and the mean distance. In an analogous way, Section 4 explains the mean curvature integral density, the related grain size measure (Jeffries grain size) and the generalization of the latter to two-phase materials (Jeffries size). Finally, Section 5 deals with the correlation of the two grain size measures and the corresponding grain size numbers, where it is shown that a clear distinction is needed between the two types of grain size numbers. The standard error concept using the standard deviation in combination with the normalized deviation based on Student’s *t*-distribution is explained and examples of its application are shown throughout the text.

2. Volume fraction determination via the Delesse–Rosiwal law

The finding that (under certain conditions) the volume fraction of a selected phase, e.g., porosity, in a material can be determined from the area fraction of that phase in a planar section, is one of the most important milestones in the history of materials science. The history of this classic theorem goes back to Cavalieri’s principle of 1635 [20] which justified – for the first time – the use of area sections for the approximate calculation (or measurement) of 3D body volumes [5]. Although its practical application in mineralogy (petrography) has been proposed in the mid 19th century [11,12], the problem at that time was the non-availability of practically useful planimeters (the first practically useful “metallographic planimeter” was constructed as late as 1924 [19]). Therefore the idea, also going back to Cavalieri, of replacing the area measurements by line segment measurements and (later) point counting [13–15], were major steps enabling the routine application of this principle for volume fraction determination. However, it was only in the second half of the 20th century that a rigorous proof could be given that the area, line and point fractions are really unbiased expected values of the volume fraction, that the lines can also be curves, that the points need not necessarily lie on a lattice (but can also be randomly “thrown” onto the image) and that the point fraction method (which is based on simple counting) is in fact the most efficient

Download English Version:

<https://daneshyari.com/en/article/1570847>

Download Persian Version:

<https://daneshyari.com/article/1570847>

[Daneshyari.com](https://daneshyari.com)