Advanced Powder Technology 25 (2014) 154–162

Contents lists available at ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Invited review paper

Processing defects in ceramic powders and powder compacts

Keizo Uematsu *

Nagaoka University of Technology, 1603-1 Kamitomioka, Nagaoka, Niigata 940-2188, Japan

ARTICLE INFO

Article history:
Received 31 August 2013
Received in revised form 7 January 2014
Accepted 11 January 2014
Available online 28 January 2014

Keywords:
Powder
Structure
Aggregates
Large particles
Anisotropy
Ceramics
Characterization

ABSTRACTS

Structure and defects in powders and powder compacts are examined in detail to determine their formation mechanisms, and their relevance to the production of high quality ceramics. New characterization tools that are indispensable in the characterization of defect structure are described with the aid of schematic illustrations. Defects such as aggregates and large particles are present in all powders examined, even after rigorous grinding. These short-range defects degrade the properties and the quality of a sintered ceramic. Long-range defects due to particle orientation and anisotropic particle packing are also common in powder compacts, and are attributed to a shear stress field and/or a directional stress field during forming. Particle packing anisotropy produces long-range defects that are responsible for anisotropic deformation and cracking during drying and sintering.

Contents

1. Introduction

To produce a ceramic, raw powder is formed into a compact of a desired shape and subsequently heated at a high temperature to form a dense cohesive body. The characteristics of the raw starting powders as well as the processes used to shape the body affect the properties and quality of the resultant ceramic. That is, there is a critical relationship between the properties of a sintered ceramic and the structure of the powders and powder compacts from which the ceramic is produced [1].

Schematic illustrations of possible structures in a powder or powder compacts are summarized in Fig. 1. Powder packing structure affects densification and microstructure development during sintering that directly govern the properties of the ceramic. While microstructure–property relationships are very important in ceramics, our explicit understanding of powder and compact structure is very limited and is often based on empirical data and/or intuition [1]. Consequently, traditional engineering ceramic development and optimization often involves a laborious and time consuming trial-and error approach [2]. Explicit understanding and control on packing structure and critical process–structure–property

* Address: Nagaoka University of Technology, 1603-1 Kamitomioka, Nagaoka, Niigata 940-2188, Japan. Tel.: +81 258350072.
E-mail addresses: uematsu@vos.nagaokaut.ac.jp, keizouematsusan@hotmail.co.jp

http://dx.doi.org/10.1016/j.apt.2014.01.009
0921-8831/

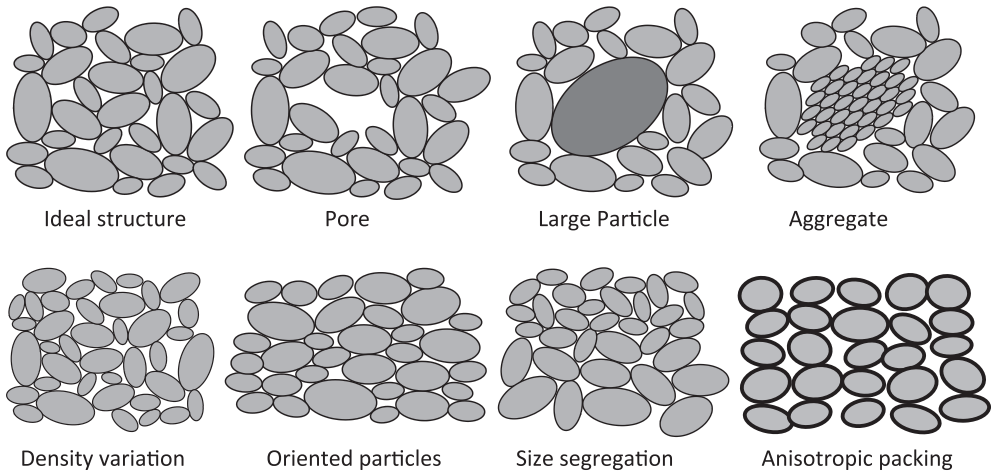

Fig. 1. Representation of packing structures and defects in powder compact.

relationships will significantly improve ceramic quality while decreasing development time.

The poor understanding of structure–property relations in ceramics is mostly ascribed to a lack of adequate characterization methods to study particle packing structure. With the exception of more recently developed tomography, scanning electron microscopy (SEM) has historically been the primary tool used to characterize microscopic structure, while porosimetry is used to characterize macroscopic structure [3]. SEM is excellent to examine packing structure in detail in a small region. However, the very small volume subjected to SEM examination severely limitd the detection and characterization of the "unusual" or defect structures responsible for reducing the quality of a ceramic. Likewise, the information acquired from porosimetry is often too general to correlate a specific defect/structure with ceramic performance or quality.

The simple fact is our knowledge of the local structural defects that govern the quality of a ceramic is very limited [1]. The mechanical strength of a ceramic is dictated by the largest defect that behaves as the fracture origin [4]. Consequently, a clear understanding of the source and location of large packing irregularities is key to improving the strength of a ceramic. However, because these defects are extremely rare, their detection and characterization is almost impossible with conventional characterization tools and methods.

Additionally, the overall structures of a powder compact has traditionally been very difficult to characterize including structural heterogeneities such as those due to particle orientation and anisotropic packing that may be responsible for anisotropic deformation and the formation of cracks upon heating [5].

The development of the liquid immersion method (LIM) has created a route to better understanding packing structure at various levels [6]. Using the LIM and the related unique methods developed thereafter, it is now possible to characterize a variety of packing structures explicitly for the first time. This paper discusses powder packing structure at various levels, including how process affects packing structure, and how packing structure influences the mechanical properties of a ceramic and the troubles in production such as cracking and warping.

2. Liquid immersion method and related methods

The LIM was developed over 20 years ago and has been applied in the fundamental research of ceramics ever since. Details of the method have been documented elsewhere [3]. An outline of the method is as follows.

A powder compact is made transparent by immersing it an appropriate index of refraction liquid to observe the internal structure with a proper microscopy. Fig. 2 shows alumina powder compacts immersed in liquids with various refractive indices [8]. The best transparency is noted for the liquid with the same refractive index as alumina. The liquid transforms the alumina/air interface into an alumina/liquid interface to minimize the reflection/refraction.

The thickness of the powder compact affects the quality of the image significantly. Practically, a 0.2 mm thick specimen can be prepared by grinding a small piece of powder compact with a sand paper. The sample can be held with the tip of finger and rubbed on the sand paper. Alternatively, a jig with a successively shallower groove can be used to hold the specimen. Heating at an adequate temperature below the densification temperature gives the compact some strength, which can ease the preparation of a thin specimen.

A variety of microscopes can be used to observe many kinds of structures [6–10]. Table 1 summarizes the structures that can be observed. Characteristics including crystallographic properties can be examined with a polarized light microscope. The high transmittance of infrared light through powder compacts makes IR microscopy the best for the bulk observations in thick specimen as well as in specimens with a high refractive index for which an adequate immersion liquid is not available. Confocal fluorescent scanning laser microscopy (LFSLM) provides high resolution detailed structures. Structures and features that have successfully been identified with these tools include agglomerates, large particles, packing heterogeneities, local particle orientation of particles, and the segregation of additives.

The structure of a ceramic after sintering also can be examined with a variety of transmission microscopy. These methods are

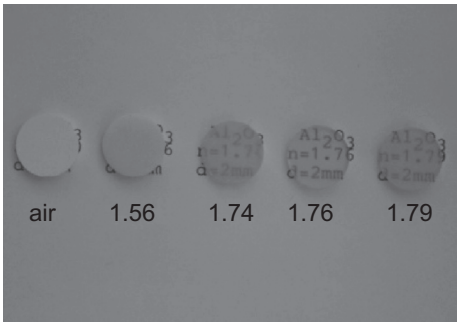

Fig. 2. Images of alumina powder compacts with different refractive index liquids.