New characterization method for macrostructures of ceramics hybrid material

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Abstracts

This paper presents new powerful characterization tools for ceramic and hybrid materials based on optical microscopy. Specimens are made transparent by thinning or the immersion liquid and the structure is observed in transmission mode. The tools can reveal detrimental defects, which are characteristically very few in number but large in size. Confocal laser scanning fluorescent microscopy and infrared microscopy are also applied to extend the potential of the tool.

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1. Introduction

Characterization of structure is the starting point in the fundamental study of ceramic and its hybrid materials. Ceramics contains many defects in its structure and one of them behaves as a fracture origin, governing the strength [1]. Full characterization of large processing defects is the key for understanding the strength and its variation in ceramics. Unfortunately, however, current characterization techniques are not well suited for examining these large defects. They are small in number, and cannot be found by SEM and TEM in many cases. Without knowing the characteristics of defects, it is impossible to eliminate them through scientific research. Characterization tool for large defects is essential in the progress of ceramics and their hybrid material.

Smart application of optical microscopy has been reported for characterizing large defects [2]. Ceramics can be examined by the transmission mode of optical microscope, since most of them are made of transparent material. This mode of observation is indispensable to identify defects of extremely low concentration. Many difficult problems have been solved with this microscopy in ceramics. Even the strength of ceramic can be predicted with the information of defects obtained with this method.

This paper shows the new characterization techniques as well as examples of macro- and micro-structures obtained with them. It also reviews the new optical microscopy for the examination of macro-structure of green compacts before firing [3]. Full understanding of green compact is crucial in ceramics, since it governs the macro- and micro-structure and thus the properties of ceramics.

2. Characterization tools

2.1. A tool for examination of ceramic macrostructure

The specimen for examination is a thin section of ceramics. Various methods are available for the preparation. Typically, a thin piece (1 mm) is cut from a bulk ceramics with a diamond tool. The piece is thinned further by grinding. Finally, both faces of the thin section are polished with diamond powder (1 μm). The specimen is placed on a sample stage of an optical microscope and the internal structure is observed with the transmission mode. Typical thickness of the specimen should be about 0.1 mm and depends on the translucency of the ceramics.
2.2. Tools for examination of ceramic green compact

In the examination of green compact, a thin specimen is also used. It can be prepared by grinding a small piece of green body on a sand paper [4]. Then the specimen is made transparent with an adequate immersion liquid. The internal structure is observed with various techniques of optical microscopy.

The selection of liquid is the key for the successful observation, since it governs the transparency of the specimen. In general, the ratio of refractive indices (RI) of relevant phases controls the reflection of light at the interface. No reflection of light happen when the refractive indices matches for the solid and the liquid. Liquids of various refractive indices are available. Liquids with the RI under 1.79 contain safe chemicals only. Those with high RI may be highly toxic. The highest RI attainable is around 2.05 in the liquid at the room temperature. In the visible light range, the refractive index of solid should be under this value. In the infrared region, the transparency is greatly improved.

A regular optical microscope [3,4], an infrared microscope [5] and confocal laser scanning fluorescent microscope (CLSFM) [6] are used for observation. The infrared microscopy extends the range of materials to be examined significantly. With this tool, there is no practical limitation in the examination for systems made of fine powders. A slight disadvantage of IR microscopy is a slightly reduced resolution of optical image. Nevertheless, IR microscopy has very high potential since the size of important defects is very large. In CLSFM, a fluorescent dye is dissolved in the immersion liquid, and its distribution is visualized. The image represents negative image of particles in the green compact at very high resolution; i.e. dark image corresponds to regions of high packing density of powder particle, and bright image the region of low density.

The requirement on immersion liquid varies with the observation method, i.e. the mechanism of image generation. With the polarized light microscopy, the best matching is desirable for refractive indices of the liquid and the solid. The quality of optical image increases with increasing transparency, since the optical contrast is developed by the anisotropy in optical property of solid. Liquid having slight mismatching of refractive index is needed to observe pores and cracks with the normal transmission mode. The optical contrast is generated by the residual scattering of light at the interface of liquid and solids. No structure can be seen when the matching of refractive index is complete. The requirement of refractive index matching is much reduced in IR microscope. The light with long wavelength reduces the reflection of light at the interface. The best matching is needed in the examination with CLSFM.

3. Macrostructure of green compacts and ceramics

Fig. 1 shows the macrostructure examined with the liquid immersion technique for the alumina granules, their compact and the sintered ceramics [7]. The granules contain dimples. These granules are typically formed through spray drying of well dispersed slurry. The flocculated slurry tends to produce
the solid granules. It is of interest to note, that the shapes of these granules are almost identical. Even the small granules contain dimples. This result must be very important to discuss the formation mechanism of these dimples in a future study. The traces of dimple are left in the green compact, which was formed even with uniaxial pressing at low pressure and subsequently CIPed at high pressure. Cracks are noted in some of the pressed granules in the compact. Many darks spots are noted in the sintered ceramics. Clearly, cracks located at the center of deformed granules grow in the densification period and develop defects in sintered bodies [2].

Fig. 2 shows the infrared micrograph of silicon nitride compact made by uniaxial pressing [5]. This micrograph is much clearer than the corresponding micrograph taken with the normal optical microscope (not shown here). The transmission of infrared light is much higher than the visible light for the present system, where the refractive index is high and the particle size is comparable to the wavelength of visible light. The structure observed in this micrograph is very similar to that of alumina. Clearly, the structure of compact made by pressing method is very similar regardless of the difference in material. The dark image surrounding each granule is binder. The binder has a refractive index different from that of the powder. This result suggests that the regular and infrared microscopy can be a powerful tool to examine the distribution of various materials in ceramic hybrid material. Another merit of IR microscope is that thick specimens can be examined [8]. This may open a new way for non-destructive evaluation of green compact.

Fig. 3 shows the CLSFM micrograph of alumina compact. The specimen is similar to that shown in Fig. 1(b). The structure is noted much more clearly and detailed. The cracks at the center of compacted granules are visible. The difference of brightness at various regions in the microstructure shows the difference in the packing density of powder particles. In this microscopy, the brightness decreases as the packing density of powder particles increases. Clearly, the packing density is non-uniform in this green compact.

4. Summary

Various kinds of microscopy were developed to examine the structure of ceramic hybrid materials and their green compacts. They have very high potential for detailed analysis of structure and are capable of revealing various kinds of structures in compacts for the first time. The detailed structure analysis directly shows that there are much to be controlled in the current processing of ceramics. Better control of processing can lead to production of better ceramics.

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