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The Development of A Novel SR-CT Technique-Originated Equipment for Microwave Sintering

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Abstract

Microwave sintering has been paid a considerable amount of attention by researches as a novel technology for the preparation of dense structural ceramics recently. However, the mature theory has not been established due to the technical difficulties. Synchrotron Radiation X-ray Computed Tomography (SR-CT) technique is a latest non-destructive detection technology. Applying the SR-CT technology on the research of microwave sintering can realize the observation of the evolution of microstructure under microwave and high-temperature field in a non-destructive, 3D and real-time way, provide more accurate experiment data for revealing the kinetics mechanism, and offer direct foundation for establishing the theory of microwave sintering. But due to the high requirements of experiment skills, it is difficult to apply the SR-CT technique into the in-situ observation of the microstructure evolution process during microwave sintering. Especially, duo to the restriction of SR-CT experiment platform, to design a microwave sintering equipment is quite a big problem. In this paper, according to the analysis of the requirements of SR-CT technique and the restriction of SR-CT experiment platform, the corresponding solutions for these difficulties such as separated structure arrangement, two-ply heat insulation and high precise rotation device were put forward, and a microwave sintering furnace exclusively to SR-CT experiment was designed. The testing on the heat insulation structure and the precision calibration of the rotation device was conducted, the results show that the heat insulation effect is good and the rotation precise achieves the required standard. Besides, depending on this equipment, the first observation of the microstructure evolution of SiC during microwave sintering was carried out, and a few sintering phenomena during three sintering stages were clearly observed. The experiment results indicate that the all the specifications of the equipment meet the design requirements.

Keyword: Microwave sintering; synchrotron radiation X-ray computed tomography; microstructure evolution

1 Introduction

Microwave sintering is being developed as a novel technology for the preparation of dense structural ceramics recently [1,2], showing densification processes enhancement, less sintering time and decreasing grains size of

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products compared with conventional sintering [3,4]. However, the mature theory has not been established due to the technical difficulties. To find an appropriate experimental technique to observe the microstructure evolution process during microwave sintering in situ, comprehend the sintering phenomena and reveal the microwave sintering mechanism will provide more accurate experiment data for optimizing the microwave sintering technique and establishing the theory of microwave sintering. There are some optical and electron microscopy techniques [5] generally applied to investigate the microstructure of ceramic materials such as high resolution transmission electron microscope (HR-TEM) and scanning electron microscopy (SEM), which can but acquire high resolution images of the microstructure of the internal faults and supper-thin slices. Not only do these techniques destroy the original microstructure, but fails to observe the characteristics of microstructure evolution. Therefore, it is tremendously helpful to find an appropriate experiment method to powerfully support the mechanism revelation and process optimization of microwave sintering.

The Synchrotron Radiation X-ray Computed Tomography (SR-CT) technique is a latest non- destructive detection technology [6,7,8,9]. Via this technique, in situ observation of microstructure evolution of materials under extreme conditions (e.g., high pressure, high temperature, intense radiation, etc.) becomes possible. The SR-CT technology applied to the research of microwave sintering can realize the observation of the evolution of microstructure under microwave and high-temperature field in a non-destructive, 3D and real-time way, provide more accurate experiment data for revealing the kinetics mechanism, and offer direct foundation for establishing the theory of microwave sintering.

Currently, few scholars have tried to carry out the in-situ investigation of conventional sintering by the SR-CT technique. O. Lame [10], A. Vagnon [11], R.M-Atanasio [12], et al. have adopted this technique to observe the conventional sintering of metallic and ceramic materials. In our research group, we have also carried out the study on the conventional sintering of various ceramic materials [13,14]. Therefore, we are convinced that the SR-CT technique will provide the possibility of in-situ investigation on microwave sintering. However, due to the high requirement of experimental skills, it is difficult to apply the SR-CT technique into the in-situ observation of the microstructure evolution process during microwave sintering. Especially, duo to the restriction of the SR-CT experiment platform, to design a microwave sintering equipment is quite a big problem (such as structure arrangement, the requirement of X-ray passing, high precision rotation device, heat insulation and microwave leak).

In this paper, according to the analysis of the SR-CT technique requirements and the SR-CT experiment platform restriction, the corresponding solutions for these difficulties were put forward, and a microwave sintering furnace exclusively to SR-CT experiment was designed. The testing on the heat insulation device and the precision calibration on the rotation device has been carried out. Besides, the first observation of the microstructure evolution during microwave sintering on SiC was conducted on this equipment. The test and experiment results indicate that all the specifications of the equipment meet the design requirements, and it is feasible to apply the SR-CT technique on the study of microwave sintering.

2 The application exploration of the SR-CT technique on microwave sintering

2.1 The principle of SR-CT technique

The SR-CT technique is a non-destructive testing method by which the specimen passed through by synchrotron radiation X-ray is placed on a rotation device and the projection images of the specimen are received by an X-ray charge-coupled device (CCD). One projection image is collected each time when the specimen turns for an angle. After obtaining a set of projection data, reconstruction algorithm is used to obtain the internal microstructure of the sectional images. The 3-D images of the microstructure can be obtained from a series of sectional images. Reconstruction algorithms applied in the SR-CT technique are mainly filtered by back projection and iterative algorithms. Taking the limited time into account, filtered back projection algorithm is employed in this paper.

2.2 The specifications of the equipment and the technique difficulties

According to the principle of the SR-CT technique, the requirements of high-temperature microwave sintering and the restriction of the SR-CT experiment platform, we believe that the SR-CT microwave sintering furnace should consist of these parties: microwave source, microwave power control systems, microwave cavity, holes for x-ray passing and temperature measurement, heat insulation device, rotation device, etc. The working procedure of SR-CT analysis system that depends on the core equipment of SR-CT microwave sintering furnace is: a).Feed the