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Microwave Drying of Spontaneous-Coagulation-Cast Wet Alumina Green Body

LIU Wenlong[1,2], ZHAO Jin[1,2], LIU Juan[1,2], MAO Xiaojian[1,2], ZHANG Jian[1,2], WANG Shiwei[1,2]

(1. State Key Laboratory of High Performance Ceramics and Superfine Microstructure, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai 200050, China; 2. Center of Materials Science and Optoelectronics Engineering, University of Chinese Academy of Sciences, Beijing 100049, China)

Abstract: To solve moisture gradients in the conventional drying with controlled temperature and relative humidity, microwave heating was employed to dry wet alumina green bodies shaped by spontaneous coagulation casting. The weight loss, linear shrinkage, surface temperature, and moisture distribution of the green bodies by conventional drying (temperature: 40 ℃; humidity: 60%) and microwave drying were investigated. The time for no further weight loss and shrinkage of the body by microwave drying (power: 250 W) were respectively shortened to 1/6.8 and 1/6 of those by conventional drying. Surface temperature of the green body during the microwave drying increased firstly and then decreased with time, which was strongly correlated with the internal moisture, while the temperature in the conventional drying keeping at 40 ℃. Low-field nuclear-magnetic-resonance (NMR) imaging revealed that the moisture distribution in the green bodies dried by microwave drying was more uniform than that by conventional drying, indicating that drying stress in the former was lower than that in the latter. After sintering at 1550 ℃ for 6 h, alumina ceramics from microwave drying had a higher flexural strength with a smaller deviation than that from conventional drying.

Key words: microwave drying; low-field nuclear-magnetic-resonance imaging; moisture distribution; alumina; coagulation

Spontaneous coagulation casting (SCC) can use a water-soluble copolymer of isobutylene and maleic anhydride (commercial name Isobam) as both dispersant and gelling agent to prepare wet alumina green body at room temperature in the air[1]. This is a new class of near-net-shaped ceramic processing[2]. In the past decade, SCC has received extensive attention in the field of advanced ceramics due to its low addition, non-toxicity, and strong universality. It has been carried out for the shaping of various advanced ceramics, *e.g.*, structural ceramics[3], electronic ceramics[4], porous ceramics[5-6], and transparent ceramics[7-10].

However, drying of wet alumina green bodies by SCC has to face the same difficulties as those produced by other colloidal processes, including gel casting[11] and direct coagulation casting[12-13], because water covers approximately half of the initial volume of the wet green body. In the conventional drying with controlled temperature and relative humidity, a wet green body tends to distort and crack due to inhomogeneous drying[14], especially for the large-sized, complex-shaped samples. This phenomenon hinders the practical application of colloidal processes. In the conventional drying, moisture is transported to the surface of the wet green body by capillary forces. Usually, the internal moisture of the wet green body decreases continuously as the drying processing. When capillary forces cannot transport moisture from the interior to the surface, the drying rate will decrease and the constant rate period (CRP) ends. In the end, a large moisture gradient is generated inside. Therefore, the wet green body is inhomogeneously dried in the subsequent drying process. Large internal stress generated from the inhomogeneous drying would lead to deformation and cracking of the green body[15]. To solve this problem, Yang *et al*.[16] attempted to adjust the network structure of the wet

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Foundation item: National Natural Science Foundation of China (52130207)
Biography: LIU Wenlong (1997–), male, Master candidate. E-mail: liuwenlong20@mails.ucas.ac.cn
刘文龙(1997–), 男, 硕士研究生. E-mail: liuwenlong20@mails.ucas.ac.cn
Corresponding author: ZHAO Jin, lecturer. E-mail: zhaojin@mail.sic.ac.cn; WANG Shiwei, professor. E-mail: swwang51@mail.sic.ac.cn
赵 瑾, 助理研究员. E-mail: zhaojin@mail.sic.ac.cn; 王士维, 研究员. E-mail: swwang51@mail.sic.ac.cn

green body by adding plasticizers, to reduce the internal stress in the drying process. However, the addition of plasticizer may reduce the solids loading in the slurry. Barati *et al*. [17] adopted liquid desiccant to avoid the deformation and cracking of wet body. However, the operation of wet body cleaning and organic solvent recovering are complicated. In addition, it is very important to complete the drying process of the wet body in a short time on consideration of production efficiency.

Microwave drying is a method of heating an entire sample simultaneously that has been widely used in the drying of agriculture, food, wood, and sanitary ceramics[18-21]. Chong *et al*.[22] studied the drying of apple slices by microwave and found that the drying rate was greatly improved. Moreover, the difference in moisture content between the external and internal parts of a fruit cube was lower since water diffusion and temperature gradients were consistent. Itaya *et al*. [23] performed simulation by a finite-element method and found that microwave drying had a smaller moisture gradient and lower internal stress than conventional drying. Esin *et al*. [24] used microwave drying to dry clay and confirmed that it greatly improved productivity and reduced the product damage rate. Shirai *et al*.[25] used microwave drying to rapidly dry the wet body prepared by slip casting. Compared with conventional drying, green bodies dried by microwave drying exhibited less deformation and more uniform microstructure. Hemanthakumari *et al*.[26] carried out microwave drying of electronic insulation components by extrusion molding, and the microwave-dried samples exhibited more excellent flexural strength and higher Weibull-modulus values as compared with conventional drying. However, there are less researches on the drying of wet body of advanced ceramics, especially shaped by *in situ* colloidal processes.

Inspired by the above-mentioned studies, we attempted to microwave-dry wet alumina green bodies shaped by SCC and compare them with those dried conventionally. Large-sized samples were expected to be dried by microwaves without deformation and cracking. In this work, 400-mm-long wet alumina green bodies were prepared by SCC. Low-field nuclear-magnetic-resonance (NMR) imaging technology[27-29] was employed to elucidate moisture distribution in the wet alumina green body in the drying process. The effect of microwave power on drying speed, linear shrinkage, surface-temperature changes and moisture distribution of the wet alumina green bodies in the drying process, and mechanical properties of the final ceramics were explored in detail.

1 Experimental

1.1 Materials and sample preparation

Commercial alumina powder (99.8%, AES-11, Sumitomo Chemical Co., Japan) with an average particle size of 0.45 μm was used as the raw material. Isobam 600AF and Isobam 104 (molecular weights 5500–6500 and 55000–65000 respectively, Kuraray, Osaka, Japan) were used as dispersant and gelling agents, respectively. The preparation process of the alumina green body is similar to that previously reported[1]. Deionized water, alumina powder, 0.1% Isobam 104, and 0.2% Isobam 600AF (relative to the weight of alumina powder) were mixed by attrition milling to prepare alumina slurry with 50% solids loading (in volume). The slurry was degassed and poured into molds with dimensions of 400 mm× 50 mm×10 mm and sealed with plastic film to prevent moisture evaporation. After coagulation (time: 12 h; temperature: 40 ℃), the samples were dried in a commercial microwave oven (IS-9DF, INNOV-SOURCE, China) at controlled temperature (40 ℃) and relative humidity (60%). Parallelly, the samples were conventionally dried under temperature of 40 ℃ and relative humidity of 60%. For the measurement of moisture distribution in the drying and mechanical properties after sintering, the alumina slurry was poured into a 40 mm×40 mm×30 mm mold to prepare small samples. After drying, the samples were sintered at 1550 ℃ for 6 h in a muffle furnace.

1.2 Characterization

An electronic balance was used to measure the drying loss (water removal) of the samples. A ruler was fixed to the sample mold to measure the length of the sample. An infrared thermometer was used to measure the temperature at the center of the sample surface. For microwave drying, the weight, length, and surface temperature of the samples (400 mm× 50 mm×10 mm) were recorded at 10 min intervals for the first 3 h and at longer intervals for the following period. For conventional drying (temperature: 40 ℃; relative humidity: 60%), the weight and length of the sample were recorded at 1 h intervals for the first day and at longer intervals for the following period. Low-field NMR imaging (MesoMR23-060H-I, NIUMAG, China) testing was conducted for the small samples (40 mm× 40 mm×30 mm) dried by both methods for different durations. The bulk density of the dried bodies was measured by the Archimedes method. The pore-size distribution of the dried bodies was measured by a mercury porosimeter (Auto Pore IV9500, Micromeritics,

Inc., USA). The fracture surface of the dried bodies was observed using a scanning electron microscope (S-4800, Hitachi, Japan). Flexural strength of the sintered ceramics was measured using three-point bending on bars with dimension of 36 mm×4 mm×3 mm at a crosshead speed of 0.5 mm/min, and 10 specimens were collected to obtain an average value for each sample (INSTRON-1195, Instron Co., USA).

2 Results and discussions

2.1 Weight loss and shrinkage in the drying

The volume fraction of residual water in the wet alumina green body (400 mm×50 mm×10 mm) with 50% solids loading was plotted as a function of time for conventional drying (temperature: 40 ℃; relative humidity: 60%) and microwave drying (microwave power: 250 W; temperature: 40 ℃; relative humidity: 60%). As shown in Fig. 1(a), by conventional drying, the CRP was approximately 10 h, and the residual water volume fraction in the sample after CRP was 20.4%. The drying was completed in approximately 36 h, and the residual water volume fraction after drying was approximately 0.8%. By microwave drying, the CRP was approximately 1.8 h, and the residual water volume fraction in the sample was reduced to 11.2%. The drying was completed in approximately 5.3 h, and the residual water volume fraction after drying was reduced to 0.6%. Microwave drying accelerated the whole drying process, and the time for no further weight loss of the body by microwave drying was shortened to 1/6.8 of that by conventional drying. Microwave drying was also carried out using microwave powers of 400 and 550 W at temperature of 40 ℃ and relative humidity of 60%. Fig. 1(b) hows that when the microwave power was increased from 250 W to 550 W, the CRP was shortened from 1.8 h to 1.0 h and the residual water volume fraction at the end of CRP was decreased from 11.2% to 7.7%. The time for "complete" drying was decreased from 5.3 h to 1.7 h, and the residual water volume fraction after drying was reduced from 0.6% to 0.4%. The enhanced drying rate was ascribed to the fact that it was easier for the moisture inside the wet green body to transport to the surface and the drying rate was apparently increased with the increase of microwave power. In other words, the increase of microwave power not only decreased the residual water volume fraction after CRP and "complete" drying, but also accelerated the drying rate, as shown in Table 1. On the other hand, as the drying processing, the loss of moisture in the sample caused a decrease in the absorption of microwave power leading to a drying rate decline as seen from Fig. 1(b). These results were in agreement with the study of microwave drying of vegetables[20,30].

Fig. 2(a) shows the linear shrinkage behavior of the wet alumina green bodies. For conventional drying, the shrinkage of the 400-mm-long wet alumina green body stopped at 6.0 h with linear shrinkage of approximately 4.9%. For microwave drying (power: 250 W), the shrinkage of the body stopped at 1 h with linear shrinkage of 5.2%. The time for shrinkage of the body by microwave drying was shortened to 1/6 of those by conventional drying. The shrinkage difference is ascribed to that microwave drying can homogeneously heat the wet green body, making them shrink more uniformly, and resulting in a slightly high shrinkage.

Fig. 2(b) shows the linear shrinkage of the wet alumina green bodies dried by different microwave powers. With the microwave power increasing, the drying shrinkage rate of the bodies increased and the linear shrinkage decreased. For example, when the microwave power increased from 250 W to 550 W, the stop time of drying shrinkage decreased from 1 h to 0.5 h, and the linear shrinkage decreased from approximately 5.2% to 4.8% (Table 2). With the microwave power increasing, water in the wet green body may be vaporized and easily transported to the surface. Capillary force, which is the

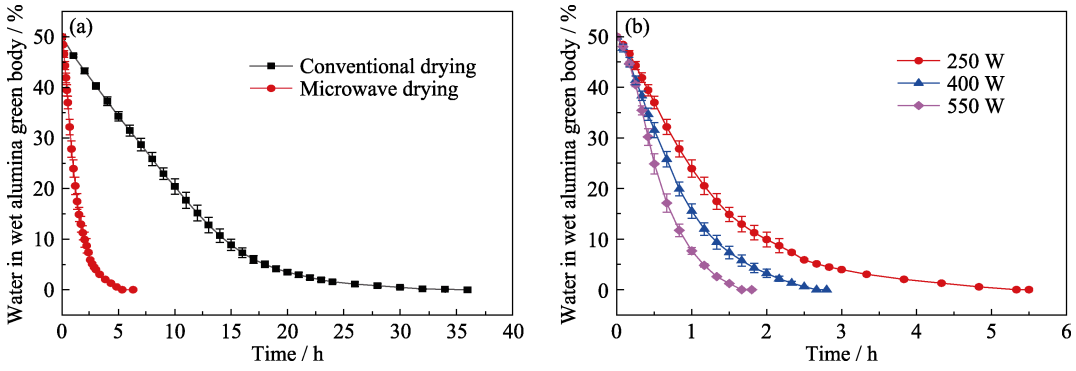

Fig. 1　Effects of (a) drying methods and (b) microwave power on the volume fraction of residual water in wet alumina green bodies

Table 1 CRP and residual water (after CRP and drying) of samples

Drying method	Microwave power/W	CRP/h	Residual water after CRP/% (in vol.)	Residual water after drying/% (in vol.)
Conventional	—	10	(20.4±1.5)	0.8
Microwave	250	1.8	(11.2±1.4)	0.6
	400	1.3	(9.4±1.4)	0.5
	550	1.0	(7.7±0.7)	0.4

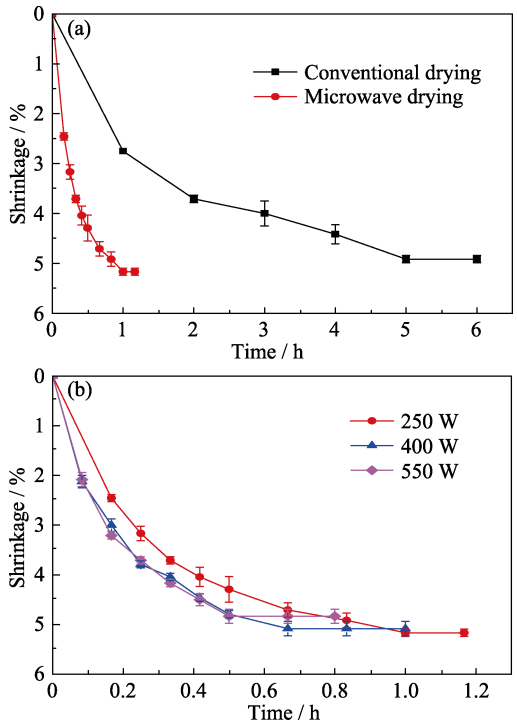

Fig. 2 Effects of (a) drying methods and (b) microwave power on shrinkage behavior of wet alumina green bodies

Table 2 Shrinkage stop time and shrinkage of samples

Drying method	Microwave power/W	Shrinkage stop time/h	Shrinkage/%
Conventional	—	6.0	(4.9±0.1)
Microwave	250	1.0	(5.2±0.1)
	400	0.7	(5.1±0.1)
	550	0.5	(4.8±0.1)

drying force for shrinkage in conventional drying, was decreasingly required to transport water in the high-power microwave drying. Therefore, the linear shrinkage of the wet alumina green bodies caused by capillary force was also reduced (Fig. 2(b)).

Fig. 3 shows the surface temperature of the samples dried by different microwave powers. When the microwave power increased from 250 W to 550 W, the maximum surface temperature of the wet alumina green body increased from 53.1 ℃ to 78.4 ℃, and the time to reach its maximum temperature decreased from 2.3 h to 1.2 h. That is, the higher the microwave power, the higher the maximum temperature of the sample surface and the shorter the time required to reach the maximum temperature. At each microwave power, the surface temperature of the wet alumina green body increased firstly and then decreased. As the drying processing, the loss of moisture caused a decrease in the absorption of microwave power. The generated heat was gradually decreased, and finally lower than that required for the evaporation of moisture and that lost from the surface of the sample. Therefore, the temperature decreased. Pictures of undeformed and intact samples after drying are shown in Fig. 4, demonstrating successful drying of the samples with a dimension of 400 mm×50 mm×10 mm. Fig. 4(a) shows the sample after conventional drying, and Fig. 4(b-d) show the samples dried by microwave drying with powers of 250, 400, and 550 W, respectively. However, 40-mm-thick samples exhibited cracking when dried by microwave powers of 400 W or 550 W, as shown in Fig. 4(g, h). The cracking was ascribed to the quick water-vapor expansion with the increase of temperature, because a higher

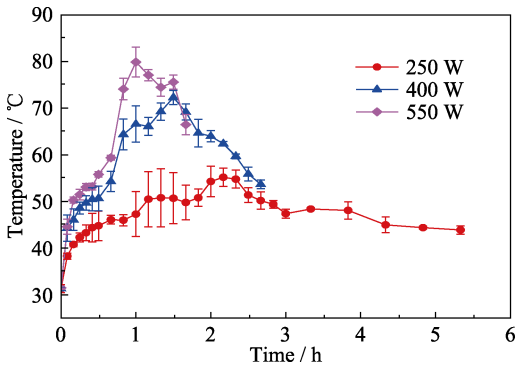

Fig. 3 Effect of microwave power on the surface temperature of wet alumina green bodies

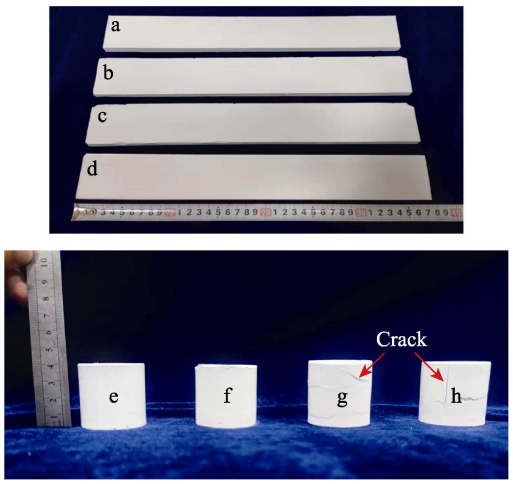

Fig. 4 Photos of alumina green bodies dried by (a, e) conventional drying and microwave drying ((b, f) 250 W, (c, g) 400 W, and (d, h) 550 W)

microwave power was employed in a short time. Therefore, microwave power must be optimized when drying thick samples.

2.2 Microstructure of alumina green bodies

Fig. 5(a) shows the pore-size distribution of the alumina green bodies (400 mm×50 mm×10 mm) dried by different methods. The cumulative pore volume of the alumina green body dried by conventional drying was 0.182 mL/g, which was greatly increased at a pore diameter of approximately 100 nm and then increased significantly at a pore diameter of approximately 8 nm. The cumulative pore volume was reduced to 0.170 mL/g by microwave drying (power: 250 W), which was only increased at a pore diameter of approximately 100 nm, and the slope was larger. This indicates that the pore size distribution among particles is concentrated in the bodies dried by microwave drying, implying more uniform microstructure. In short, the wet green body dried by microwave drying was found to shrink more homogeneously than that dried by conventional drying, resulting in more uniform pore-size distribution and reduction in cumulative pore volume. The bulk densities of alumina green bodies dried by different methods were close to 2.38 g/cm^3 which were determined by the Archimedes method.

With the microwave power increasing from 250 W to 500 W, the cumulative pore volume of the alumina green bodies dried by microwave drying increased from 0.170 mL/g to 0.195 mL/g which was only increased dramatically at a pore diameter of approximately 100 nm, as shown in Fig. 5(b). The pore-volume result was consistent with the bulk density of the alumina green bodies, which decreased from 2.38 g/cm^3 to 2.36 g/cm^3. With the microwave power increasing, the internal water was heated and vaporized, the ability to transport moisture through the vapor-pressure gradient increased and the dependence of moisture transportation on capillary force decreased so that the shrinkage between the ceramic particles decreased. Meanwhile, vapor-volume expansion become stronger with the increase of microwave power, leaving larger cumulative pore volumes in the dried bodies. Eventually, the cumulative pore volume increased, which could explain the decrease in the linear shrinkage (Fig. 2(b)) and the increase in the cumulative pore volume of the wet alumina green bodies after drying, as shown in Fig. 5(b).

Fig. 6 shows the fracture surfaces of alumina green bodies (400 mm×50 mm×10 mm) after drying by different methods. It can be seen that the microstructure of the alumina green body by microwave drying was uniform (Fig. 6(a)), similar to that by conventional drying (Fig. 6(b)). Microwave dried sample showed no microcracks. The pore size distributions of the samples dried by different methods were also similar, which were in agreement with the pore size distribution measured by a mercury porosimeter.

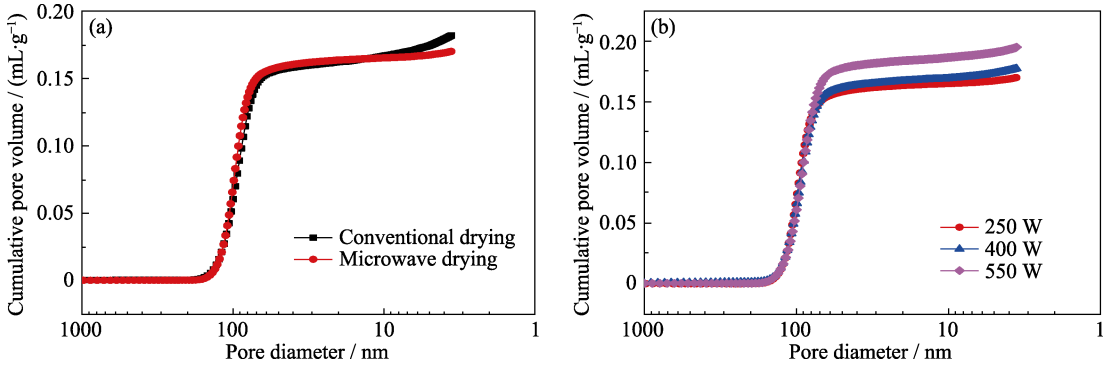

Fig. 5　Effects of (a) drying methods and (b) microwave power on pore-size distribution of alumina green bodies

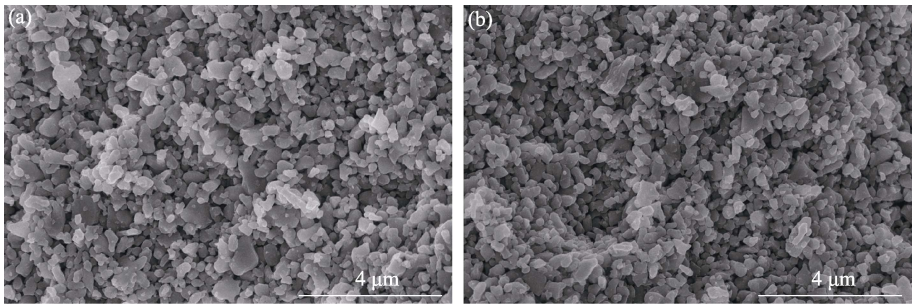

Fig. 6　Fracture surfaces of alumina green bodies
(a) Microwave drying (250 W); (b) Conventional drying

To clarify the mechanism of microstructure uniformity from microwave drying, low-field NMR imaging technology was employed to reveal the moisture distribution of wet alumina green bodies in the drying process. Fig. 7 shows the moisture distribution of the samples with a dimension of 40 mm×40 mm×30 mm dried by the two methods. In conventional drying, the moisture inside the wet green body decreased with time going and moisture gradients existed in the drying process (Fig. 7(a)). However, the moisture inside the wet green bodies dried by microwave drying was more uniform (Fig. 7(b)), similar to the simulation of the microwave drying process of molded ceramics by the finite element method[23]. It is known that microwave drying can homogeneously heat the entire sample, so that the internal moisture is easily transported to the surface with the help of capillary force, and the internal moisture gradient of the wet green body is small. Therefore, microwave drying can dry wet green bodies quickly, uniformly, and safely. Nevertheless, in conventional drying, the capillary force was the main driving force to transport the internal moisture to the surface for evaporation. As the drying processing, the moisture content was decreasing, and the capillary force alone was not enough to transport the internal moisture to the surface for drying. Hence, the moisture gradient inside the sample gradually increased, and the drying and evaporation interface moved inward. It has been reported that the generation of a moisture gradient generates internal stress inside a wet green body[14]. The greater the moisture gradient, the greater the internal stress. The generation of internal stress makes the internal microstructure inhomogeneous, and even cause deformation and cracking.

2.3 Mechanical property

Fig. 8 shows the flexural strength of alumina green bodies and ceramics dried by different methods and then sintered at the same conditions (1550 ℃×6 h). It can be seen that their flexural strength is similar, but the relative standard deviations of the flexural strength of the alumina green bodies and ceramics dried by microwave drying (250 W) are both smaller than those dried by conventional drying. This result illustrates that the microstructures of alumina green bodies and ceramics dried by microwave drying are more uniform with better reliability, similar to that reported previously for the preparation of electronic components[26].

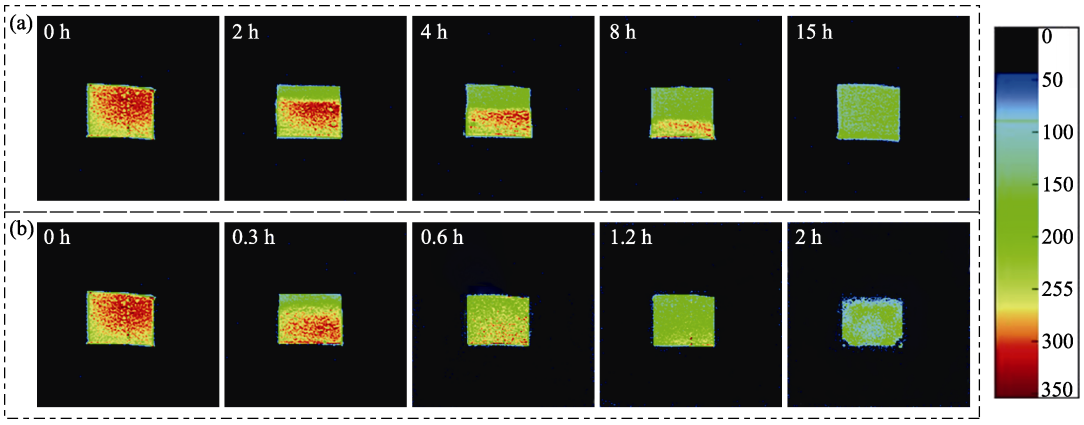

Fig. 7 Moisture distributions of wet alumina green bodies in the drying process
(a) Conventional drying for 0, 2, 4, 8, and 15 h; (b) 250-W microwave drying for 0, 0.3, 0.6, 1.2, and 2 h

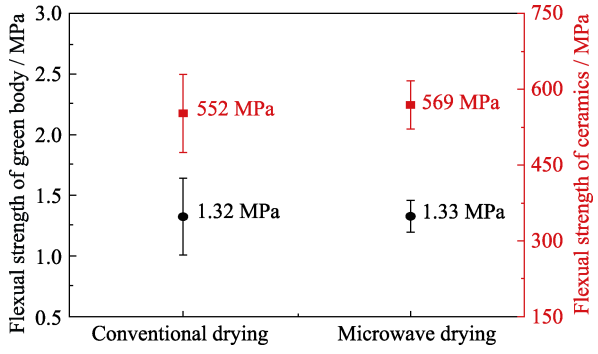

Fig. 8 Effect of drying methods on flexural strength of alumina green bodies and ceramics

3 Conclusions

In this study, microwave drying was adopted to reduce the moisture gradient of wet alumina green bodies prepared from spontaneous coagulation casting to avoid deformation and cracking in the drying. Compared with conventional drying, the residual water volume fraction after CRP was reduced from 20.4% to 7.7%, and the drying rate was increased by 5.8 times by microwave drying. Moreover, using low-field NMR imaging technology, the moisture distribution of wet alumina

green bodies in the drying process was directly observed. A significant moisture gradient appeared in the conventional drying. While, it was more uniform in the microwave drying. Accordingly, this imaging of moisture distribution would be helpful for deeply understanding moisture transportation, drying stress, and microstructure of bodies in the drying. When undergoing microwave drying, the wet green body shrank homogeneously, which greatly reduced the probability of deformation and cracking during drying. Thus, more homogeneous alumina green bodies were obtained, and the flexural strength of the ceramics was improved with a smaller standard deviation.

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微波干燥自发凝固成型氧化铝湿坯

刘文龙[1,2], 赵 瑾[1,2], 刘 娟[1,2], 毛小建[1,2], 章 健[1,2], 王士维[1,2]

(1. 中国科学院 上海硅酸盐研究所, 高性能陶瓷和超微结构国家重点实验室, 上海 200050; 2. 中国科学院大学 材料科学与光电研究中心, 北京 100049)

摘 要: 为了解决常规干燥(控温控湿)过程中坯体出现水分梯度问题, 利用微波干燥法来干燥湿坯。通过对比常规干燥(温度: 40 ℃; 湿度: 60%)和微波干燥的方式, 研究了湿坯的重量损失、线性收缩、表面温度和水分分布。与常规干燥相比, 采用微波干燥(功率 250 W)时, 干燥结束时间和停止收缩时间分别缩短至 1/6.8 和 1/6。在微波干燥过程中, 样品表面温度随时间延长先升高后降低, 与体内水分密切相关。而在常规干燥过程中, 温度保持恒定在 40 ℃。采用低场核磁共振（NMR）成像技术表征湿坯内部的水分分布情况发现: 在微波干燥过程中, 水分分布更均匀, 表明微波干燥时湿坯的干燥应力更低。在 1550 ℃下烧结 6 h 后, 微波干燥制备得到的氧化铝陶瓷具有更高的抗弯强度, 且标准差更小。

关 键 词: 微波干燥; 低场核磁共振成像; 水分分布; 氧化铝; 凝胶

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