

Stereo fabric modeling technology in ceramics manufacture

Hideki Kita*, Hideki Hyuga, Naoki Kondo

National Institute of Advanced Industrial Science and Technology (AIST), 2266-98 Shimo-Shidami, Moriyama-ku, Nagoya 463-8560, Japan

Available online 7 November 2007

Abstract

Authors have been developing a new modeling technology for fabricating three-dimensional (3D) components of various shapes and sizes and for integrating units having precise and complex structures (stereo fabric modeling technology). It is anticipated that this technique can achieve a high size range ratio and excellent enlargement. As a model, regularly arranged protrusions were formed on a substrate surface by reaction with sintered silicon nitride. The contact area between the substrate and molten copper was reduced by these protrusions. It could be further reduced by making the tips of the protrusions flat.

© 2007 Elsevier Ltd. All rights reserved.

Keywords: Modeling; Silicon nitride; Ceramics; Molten metal; Design

1. Introduction

In ceramics modeling, the techniques of extrusion molding, injection molding and press molding are employed depending on the shape, size and required precision of the intended components. It is, however, difficult to fabricate a large, complex-shaped component with a very high precision; this restricts possible designs for production.¹ To solve this problem, we have been developing a new modeling technology for fabricating three-dimensional (3D) components of various shapes and sizes and for integrating units having precise and complex structures. This method is referred to as “stereo fabric modeling” because an entire component is effectively formed by 3D integration. This method increases the degrees of freedom for the structural design of ceramics, particularly for large ceramic components. This paper introduces our research in these areas, in which we developed on a transfer board model that is light weight and has a high rigidity and a low adhesion for molten metal.

2. Design of units

It is possible to produce a wide variety of large components by integrating units having complex shapes. Several such units have so far been designed. In this study, model units having pre-

cisely ordered protrusions on their surfaces and a truss structure in their interiors are described. Such model units are expected to be used as a transfer board for molten or semi-solid metal. The protrusions formed on the surface have the potential to reduce the contact area between the surface and molten metals and thus control the adhesion between them. In addition, transfer boards need to be light weight and have a high rigidity. By optimizing their design we aim to minimize the weight of the boards, while simultaneously ensuring they have sufficient rigidity and strength.

Fig. 1 shows the design of the unit, while Fig. 2 shows a 3D image of an assembly of units. Each unit is hollow and has a truss structure to reduce its weight and to ensure rigidity. To reduce the contact area with the work piece, namely the molten or semi-solid metal that is to be placed on the board, protrusions having a diameter of 0.5 mm and a height of 0.68 mm are formed on the surface.

Interlock structures were formed on adjacent faces during the green stage and units were assembled using these interlock structures. They were fixed together in the green stage, and then dewaxed and sintered in nitrogen gas.

3. Experimental procedures

3.1. Forming

ZrO₂ (TZ-0 grade, monoclinic phase, Toso. Co. Ltd., Tokyo, Japan) and spinel (Showa Denko) were added to silicon powder (Kojundo Kagaku Kenkyuujo, Tokyo, Japan; average particle

* Corresponding author. Tel.: +81 52 736 7122; fax: +81 52 736 7405.
E-mail address: hideki-kita@aist.go.jp (H. Kita).

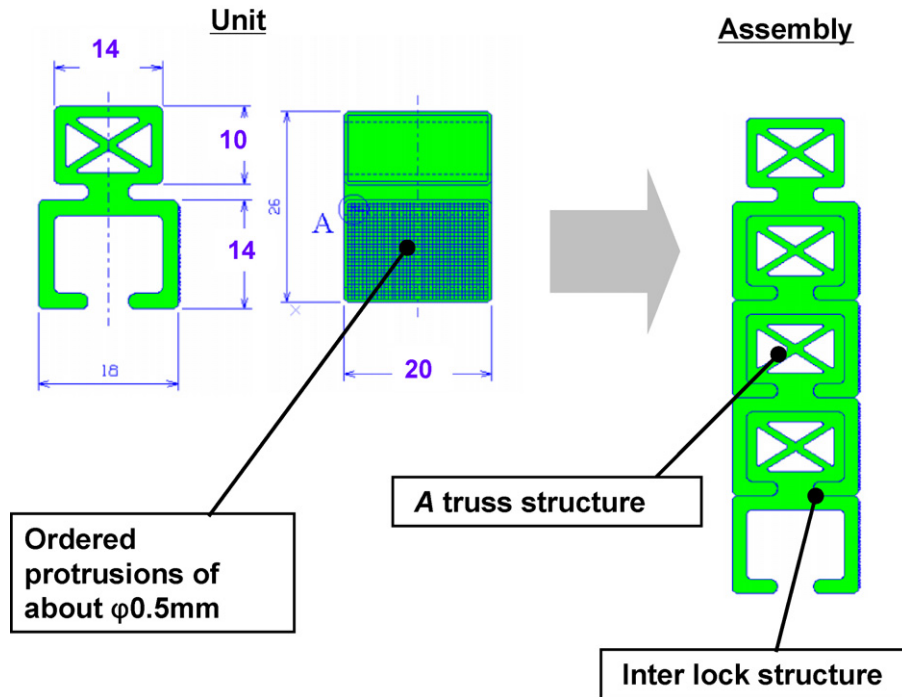


Fig. 1. The design of the unit.

size: $1\ \mu\text{m}$). The mass ratio of ZrO_2 , spinel and silicon was 10:10:80, respectively. The powders were homogeneously mixed by ball milling for 1 h in ethanol using silicon nitride and they were then spray-dried. The mixed powders thus obtained were kneaded with plastic resin and wax, cooled, palletized and then formed by injection molding to produce the units. These units are assembled together by inserting the upper part of one unit into the lower portion of another unit, as shown in Fig. 1.

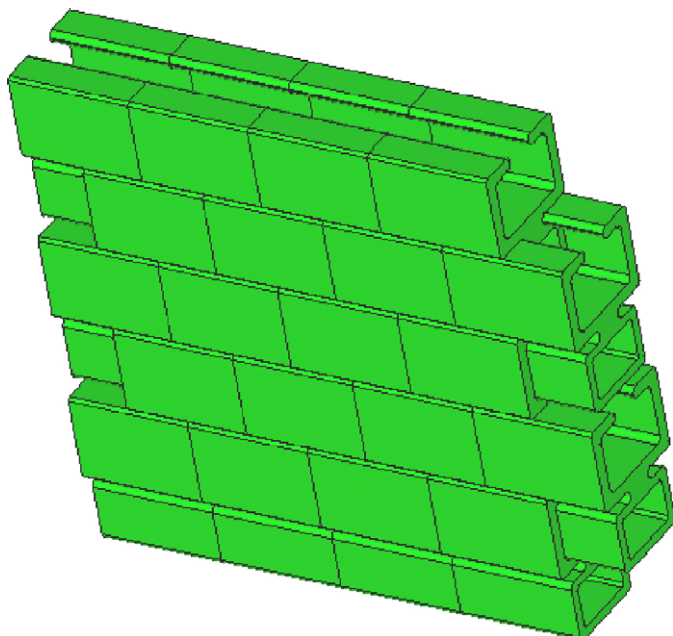


Fig. 2. A 3D image of an assembly of units.

3.2. Sintering

The assembly of green compacts was dewaxed in an air furnace at $600\ ^\circ\text{C}$, and then placed on a carbon plate, which was coated with BN spray and surrounded by an outer graphite crucible. The nitridation of silicon compacts was conducted in a carbon heater furnace (Hi-multi 10000, Fuji Denpa Co. Ltd., Tokyo, Japan) at $1450\ ^\circ\text{C}$ for 3 h with a 2 L/min high-purity nitrogen flow. Thus, obtained sintered body contained about 15 vol.% residual pores.

3.3. Adhesion behavior of molten copper

Adhesion tests were conducted using the sessile drop method. A barrel-shaped copper sample was placed near the center of a ceramic substrate having protrusions, and the sample-substrate system was inserted in the apparatus. The temperature was gradually increased. The copper sample melted at $1100\ ^\circ\text{C}$, became spherical, and then stabilized. The sample was maintained at this temperature for approximately 10 min before the temperature was increased to the maximum temperature of $1185\ ^\circ\text{C}$. During this final stage, images of the sample were acquired using a CCD camera.

4. Results and discussion

4.1. Model samples

Figs. 3 and 4 show photographs of the assembly of individual units at the green stage and after sintering, respectively, while Fig. 5 shows the microstructure of the sintered body. This model could be fabricated exactly on the basis of the above-

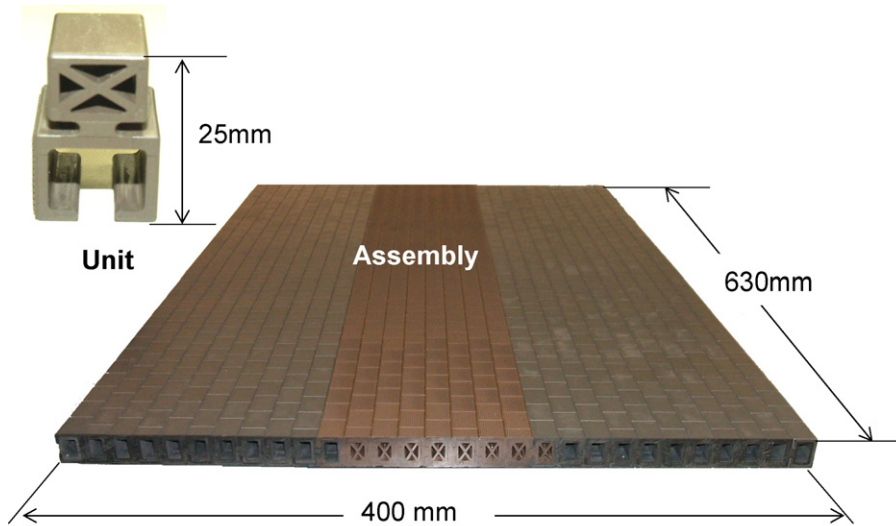


Fig. 3. The assembly of individual units at the green stage.

mentioned design; a precise and complex structure, specifically a hollow structure having a truss and protrusions that were 0.5 mm in diameter and 0.68 mm in height were precisely realized. Individual units were assembled and a large board that was 400 mm × 630 mm was produced. This result implies that the new technology supports large-scale but precise forming that can satisfy a variety of functional requirements for components such as weight saving, high rigidity, heat insulation and less adhesion to molten metal.

4.2. Adhesion behavior of molten copper on the board with protrusions

A copper sample was placed on the substrate and its temperature was increased by heating. Photographs of the profile of the copper sample were taken while it was being heated up

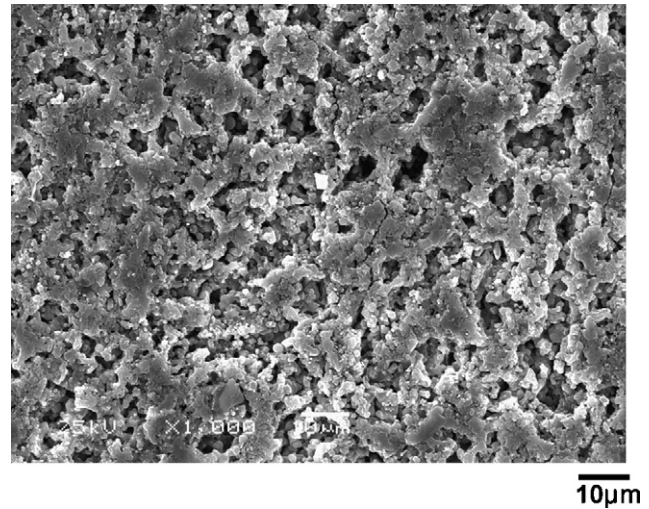


Fig. 5. SEM image of the substrates (after sintering).

Array of protrusions ($\phi 0.5$, 0.68mm in height)

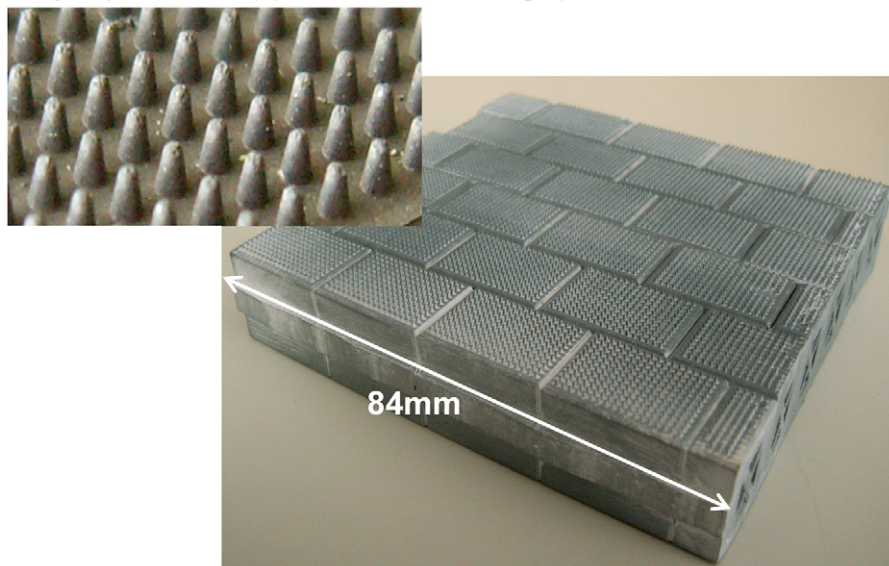


Fig. 4. The assembly of individual units after sintering and close-up of the surface.

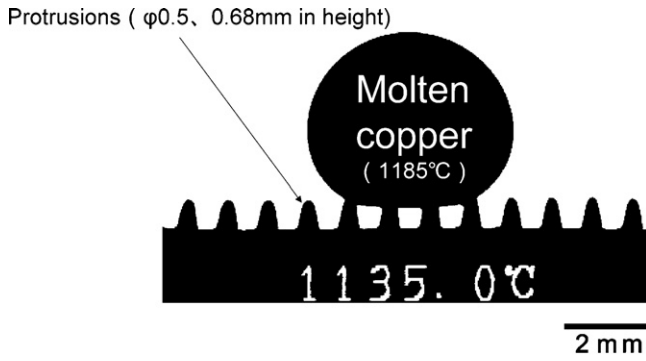


Fig. 6. A molten sample placed on the substrate.

to 1185 °C and they are shown in Fig. 6. The copper sample melted at 1100 °C and became spherical. When a substrate having protrusions was used, air pockets formed between the molten copper and the gaps between the protrusions. This indicates that it is difficult to wet silicon nitride substrates with copper. Consequently, the molten copper is supported by the tips of the protrusions and does not flow into the gaps between the protrusions. The contact area of the copper on the substrate with the protrusions array was 0.38 mm², whereas it was 4.75 mm² for a flat substrate. Thus, the contact area can be reduced by forming protrusions on a substrate that initially has a flat surface.

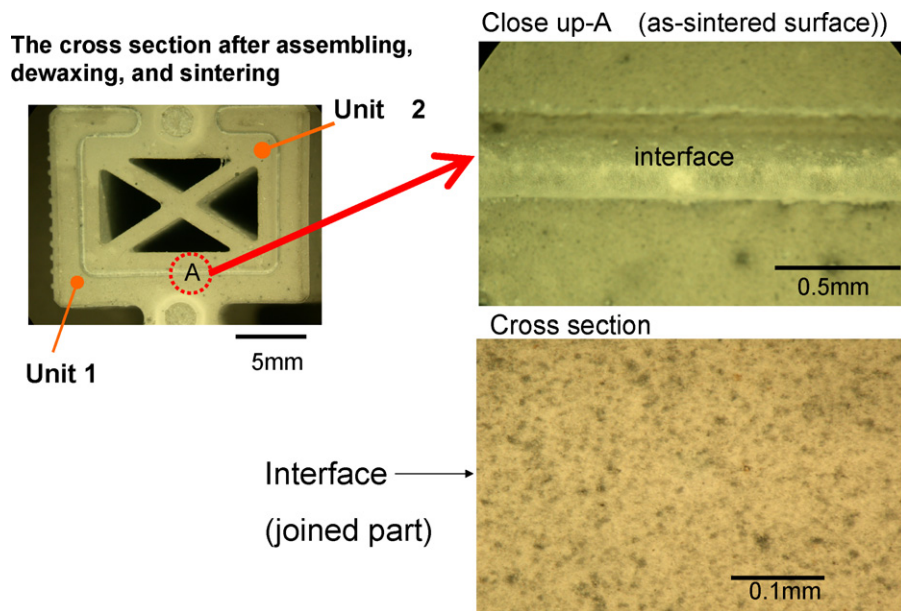


Fig. 7. The appearance and the cross-section of the assembly after sintering.

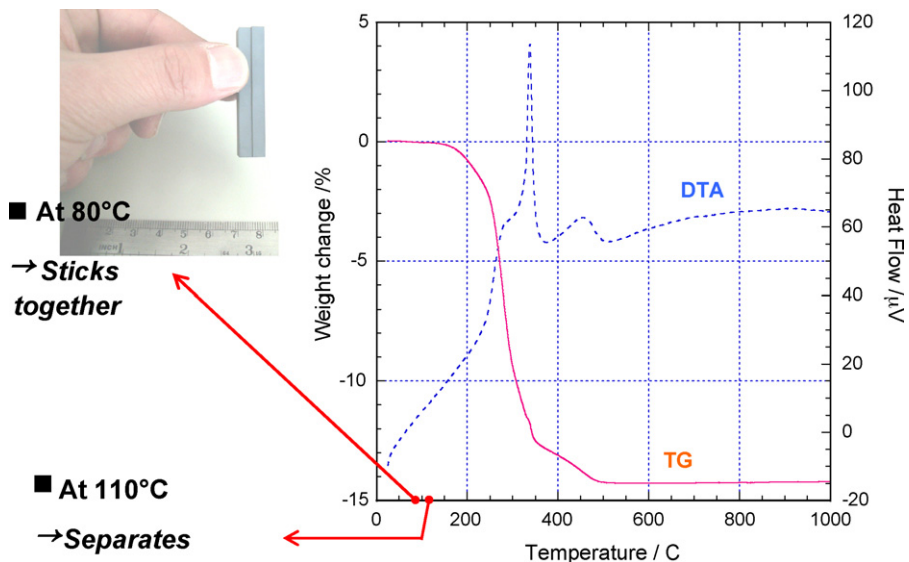


Fig. 8. The TG and DTA results showing the behavior of the binder during dewaxing.

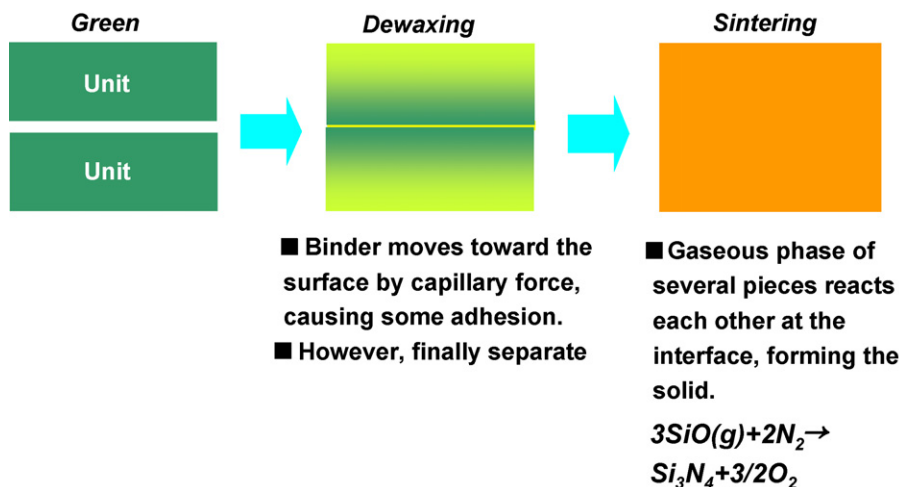
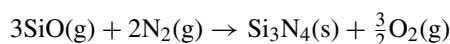


Fig. 9. A schematic diagram of the gap structure during dewaxing and sintering.

4.3. Joining

Fig. 7 shows the appearance and the cross-section of the assembly after sintering. There were gaps between individual units at the green stage; it was, however, confirmed that these gaps became filled with solid during the dewaxing and reaction-sintering stages. Fig. 8 shows the TG and DTA results showing the behavior of the binder during dewaxing. The binder used in this study was removed at 500 °C. Sample bars having the same composition as the model were assembled and placed in a furnace. They were then removed to observe their state of adhesion. At 80 °C the sample bars adhered together, but at 110 °C they became separated. On the basis of these results, the bonding state between units during dewaxing and sintering was hypothesized. Fig. 9 shows a schematic diagram of the gap structure during dewaxing and sintering. During the initial stage of dewaxing, capillary forces are thought to move the binder to the surface, causing some adhesion. However, separation finally occurs in the course of dewaxing. During reaction sintering, the chemical reaction shown below may occur^{2,3}; the gaseous phases of several units would react with each other at the interface, forming a solid that fills the gaps that were present in the green stage.



5. Conclusions

Stereo fabric modeling technology by integrating units having precise and complex structures was introduced. It is anticipated that this technique can achieve a high size range ratio and excellent enlargement. As a model, regularly arranged protrusions were formed on a substrate surface by reaction with sintered silicon nitride. The contact area between the substrate and molten copper was reduced by these protrusions. It could be further reduced by making the tips of the protrusions flat.

References

1. Paulick, C., Steinborn, G. and Waesche, R., In *Joining of Ceramic Components in the Green State, ICCCI 2006*, 2006, p. 135.
2. Riley, F. L., Reaction bonded silicon nitride. *Mater. Sci. Forum*, 1989, **47**, 70–83.
3. Ziegler, G., Heinrich, J. and Wotting, G., Review relationships between processing, microstructure and properties of dense and reaction-bonded silicon nitride. *J. Mater. Sci.*, 1987, **22**, 3041–3086.