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# Fabrication of a Complex-Shaped Silicon Nitride Part with Aligned Whisker Seeds Using LOM Technique

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**ABSTRACT**

A complex-shaped part was successfully fabricated by Laminate Object Manufacturing (LOM) technique using silicon nitride tape with aligned silicon nitride whisker seeds. The ceramic tape was cut using a commercial cutting plotter according to the cross section drafts generated by slicing a 3-D model, and then the tapes were stacked sequentially. In order to improve adhesion between the tapes, stacking was performed under vacuum. After binder burnout, the part was encapsulated using latex emulsion and was cold isostaically pressed under 250 MPa. It was sintered to 98.5% TD at 2148 K for 4 h under 2 MPa nitrogen pressure.

*Key words* : ***Laminate object manufacturing, Tape, Computer aided cutting, Automatic stacking, Latex coating, Cold isostatic pressing, Sintering***

## 1. Introduction

Silicon nitride has been considered as one of the most promising ceramic materials for the structural applications.[1] Although it exhibited good mechanical properties, there was a trade-off between some of the properties due to the fact that the properties are closely related to the microstructure. For example, while silicon nitride with fine microstructure exhibited high strength, its fracture toughness was low. Or silicon nitride with coarse microstructure exhibited high fracture toughness and low strength.[2] One of the ways to achieving both high strength and high fracture toughness is to align the whisker-like seed particles and to allow them to grow.[3,4] Since the seed particles can be aligned by tape casting and some ceramic parts have complicated shapes, it is important to find a method of making parts with complicated shapes using the ceramic tape.

Laminate Object Manufacturing (LOM) has been developed as a Rapid Prototyping (RP) technique that materializes a virtual 3 dimensional model by cutting the corresponding cross-sections and sequentially laminating sheet materials.[5] The technique has been applied to various building materials from the adhesive coated paper to ceramic tapes.[6,7] As far as making ceramic parts is concerned, it is similar to Computer-Aided Manufacturing of Laminate Engineering Materials (CAM-LEM).[8] Since LOM can employ ceramic tapes, it seems a proper method for making complex parts using silicon nitride tape with the aligned whisker seeds. In this study, a ceramic part with complicated shape was fabricated using LOM technique.

## 2. Experimental Procedure

Silicon nitride tape with the aligned whisker seeds was prepared in the same way as described in the previous paper.[9] The powder composition was 89 wt% $\alpha$-$Si_3N_4$ (SN E10, Ube Industries Ltd., Yamaguchi, Japan), 6 wt% $Y_2O_3$ (grade C, H. C. Starck Co., Berlin, Germany), 2 wt% $Al_2O_3$ (Sumitomo Chemical Co., Osaka, Japan) and 3 wt% $\beta$-$Si_3N_4$ whisker (SN WB, Ube Industries Ltd.). The thickness of the tape after drying was about 0.1 mm. Commercial 3 dimensional modeling software (SolidEdge 3.0, Intergraph, Corp., Huntsville, AL, USA) was customized using another commercial software (Visual Basic 6.0, Professional Ed., Microsoft Corp.) for slicing the 3-D model and generating the cross sections thereof. The 3-D model was 12 mm thick. Since the linear sintering shrinkage in the thickness direction was larger than the other directions, thickness of the slice was adjusted considering the shrinkage anisotropy. A commercial cutting plotter (SignPro D610, Summagraphics Europe N. V., Zaventem, Belgium) was employed for cutting the ceramic tape according to the cross sections. A 0.1 mm thick vinyl with adhesive back that was popular in the sign industry was also cut and used as the supports of the ceramic tape. Size and shape of the vinyl cut for the supports were adjusted for easy handling. Another 0.08 mm thick vinyl was cut according to the cross sections and then the cuts were removed. The remaining was negatives of the

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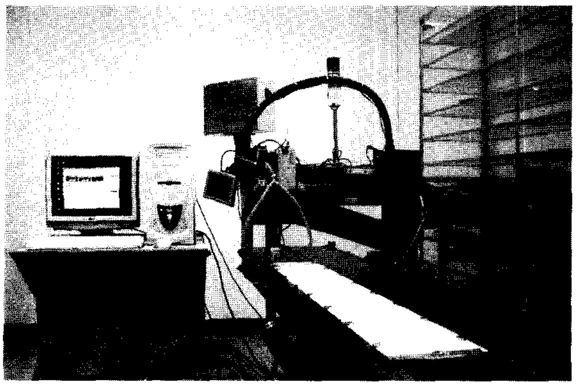
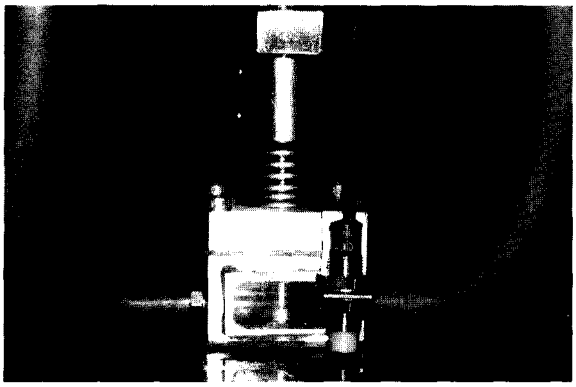


(a)



(b)

**Fig. 1.** In-house built automatic stacking equipment (a) and the vacuum stacking head (b).

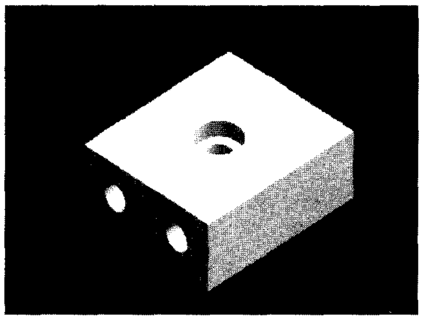
ceramic tape cuts and was used as the frame for both the ceramic tape and the vinyl supports.

After positioning the ceramic cuts and the vinyl support cuts in the vinyl frame, stacking was carried out in sequence at room temperature in an in-house built equipment shown in Fig. 1(a). It is basically an X-Y table with a specially designed vacuum stacking head driven by an air cylinder. The vacuum stacking head consisted of the chamber and punch as shown in Fig. 1(b). Effect of stacking in vacuum on improving the inter-layer adhesion was described in the previous literature.[10] The table is driven by two servomotors. A CCD camera with LCD display for reading the coordinates of the stacking head. The stacking was performed according to the following sequence. First, the coordinates of the cross sections were read in sequence and stored in the computer. Second, the vacuum stacking head was automatically positioned according to the stored coordinates. Third, the vacuum stacking head was down until the chamber fully touched the vinyl frame. Fourth, the vacuum stacking head stopped moving and the chamber was evacuated. Fifth, the punch was down inside the chamber under vacuum and stacking the layer was performed. Sixth, the punch was up and the air flowed into the chamber. Seventh, the vacuum stacking head was up and moved to the next coordinates. In the present study, stacking was performed to make cross-ply microstructure, i.e. orientations of the aligned whisker seeds in the neighboring layers were 90° off.

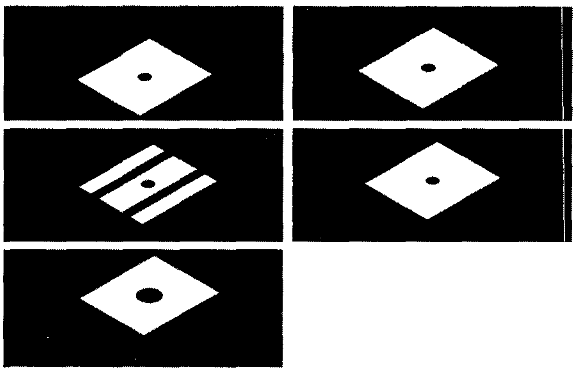
After stacking, the vinyl supports were removed from the stacked body, and the stacked body was cold isostatically pressed under 250 MPa for improving adhesion among the ceramic tapes. Then, the body was packed with the silicon nitride powder and binder burnout was performed. Details of binder burnout procedure were provided in the previous paper.[10] After binder burnout, the green body was coated with a BN spray and was dip-coated with latex emulsion. The coated green body was cold isostatically pressed under 250 MPa again, and sintering was carried out at 2148 K for 4 h under 2 MPa nitrogen pressure. Sintered density was measured using the water immersion method. For comparison, the same body was prepared without cold isostatic pressing. After cutting and polishing, the sintered body was plasma etched and the microstructure was observed using SEM. Also, Vickers hardness and the fracture toughness by indentation crack length method using Evans and Charles equation[11] were obtained under 196 N load. The indentation was performed on the surface parallel to the stacking direction to generate the cracks parallel and perpendicular to the direction. For comparison, silicon nitride without the whisker seeds was prepared by pressing the powder mixture and sintering under the same condition as the above sample.

## 3. Results and Discussion

Fig. 2(a) and (b) show a 3-D model and its slices, respec-

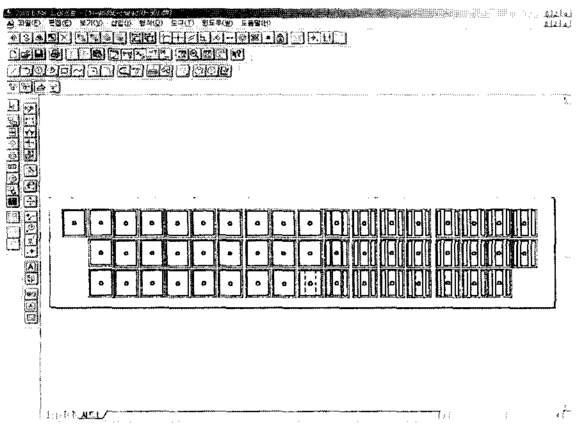


(a)

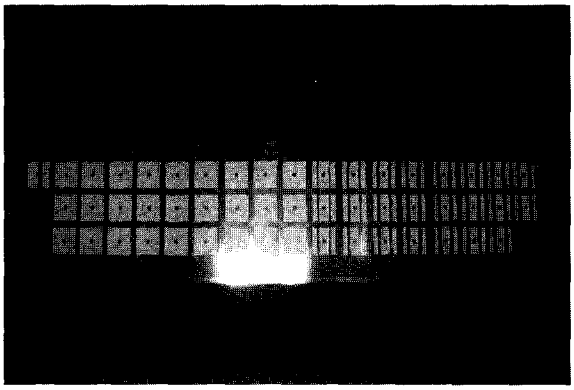


(b)

**Fig. 2.** Virtual 3-D model (a) and its slices (b).



(a)



(b)

**Fig. 3.** Cross sections of the 3-D model (a) and silicon nitride tape after cutting according to the cross sections (b).

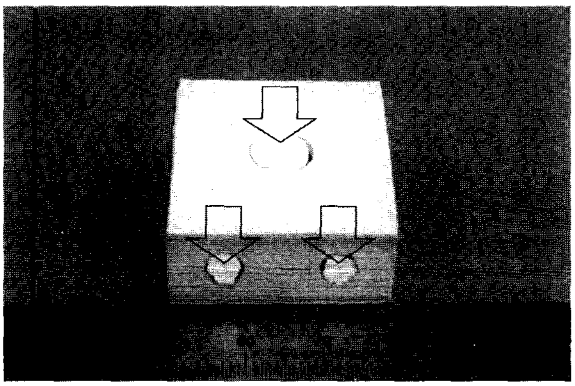
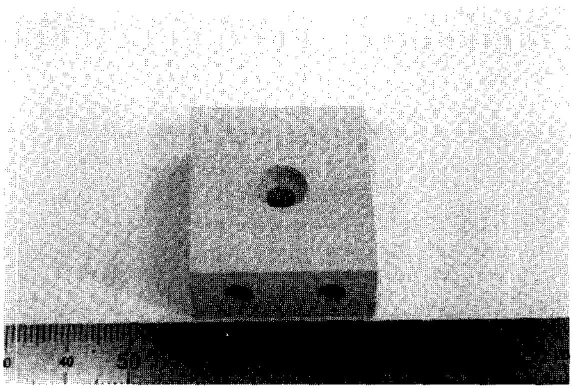
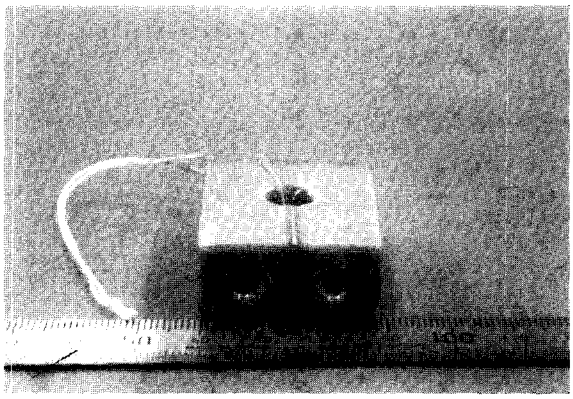


**Fig. 4** Stacked body with the vinyl supports; the arrows indicate the vinyl supports.

tively The cross section draft is simply top view of each slice. Fig. 3(a) and (b) show the cross section drafts stored in a computer and silicon nitride tape after cutting according to the drafts, respectively. Fig. 4 shows the stacked body with the vinyl supports. The vinyl supports reduced the pressure difference resulting from the different cross sectional area during stacking, and thereby decreased a shape change of the body. Fig. 5(a) and (b) show the bodies after binder burnout and after latex coating, respectively. Fig. 6(a) and (b) show the sintered bodies with and without cold



(a)



(b)

**Fig. 5.** Green body after binder burnout (a) and after latex coating (b).

isostatic pressing, respectively. Fig. 6(a) and (b) demonstrate the importance of cold isostatic pressing step prior to sintering. Without CIP'ing, severe delamination occurred. Bender *et al.* also noticed that delamination occurred to the stacked body after binder burnout.[7] They sprayed the organic diluted in the solvent on the ceramic tapes and improved the inter-layer adhesion greatly. However, it was found that spraying the diluted organic uniformly on the ceramic tape was very difficult and the tape was deformed upon contacting with the solvent. Fig. 6(a) shows that the virtual 3-D model of Fig. 2(a) was materialized. The effect of CIPing on the sintered density of the body prepared by the other solid freeform fabrication method, i.e. 3-D printing, was reported by Sun *et al.*[12] Although they also used latex coating for making water proof film on the surface of the body, they used wax filler for the long hollow in the middle of the body. However, since the wax is not as compressible as the green body is, the hollow is suspected to have larger diameter than the original design after CIP'ing. In order to prevent the shape change, it is important to find a way that allows the shrinkage of the hollows as much as the green body during CIP'ing. By preparing and inserting thin latex tubes in the two hollows of the body prior to the coating, water flowed into the hollows and thereby the green body was allowed to shrink under the pressure. In fact, Fig. 7 shows that the linear shrinkage of the diameter of the holes

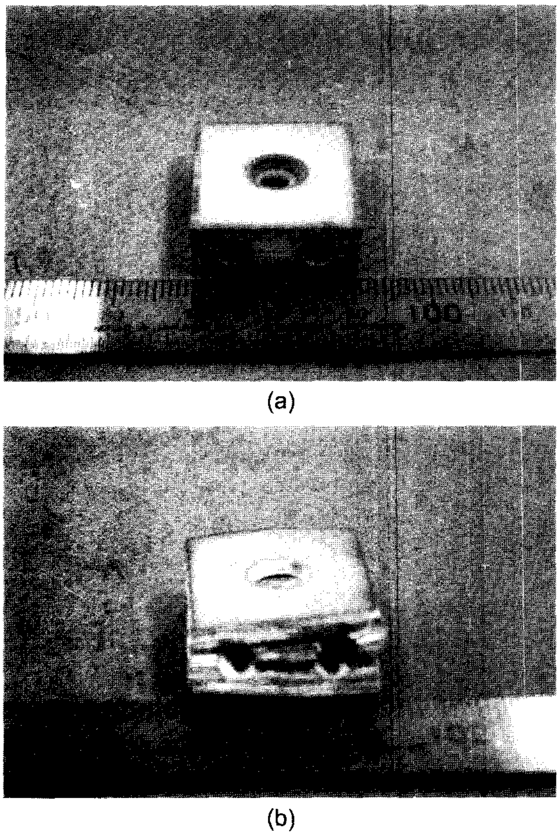
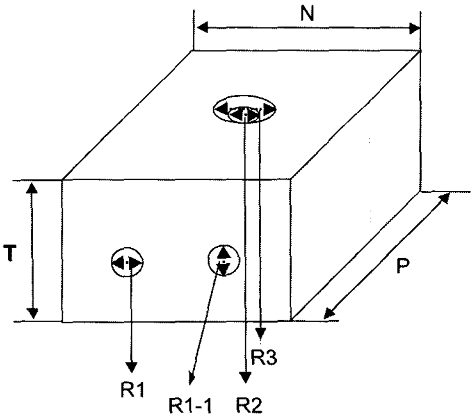



**Fig. 6.** Body after CIP'ing and sintering (a) and after sintering without CIP'ing (b).




| | T | N | P | R1 | R1-1 | R2 | R3 |
|---|---|---|---|---|---|---|---|
| Before CIP (mm) | 10.34 | 29.3 | 34.51 | 4.7 | 5.4 | 4.78 | 8.26 |
| After CIP (mm) | 12.11 | 27.67 | 32.6 | 4.45 | 5.1 | 4.45 | 7.82 |
| Linear shrinkage (%) | 10.6 | 5.6 | 5.3 | 5.3 | 5.6 | 6.9 | 5.3 |

**Fig. 7.** Linear shrinkages of the sample due to cold isostatic pressing after the latex encapsulation.

by the cold isostatic pressing was greater than 5%.

Density of the sintered body was 98.5% TD. Fig. 8 shows the microstructure of the sintered body. Since the etched surface was parallel to the stacking direction, the elongated grains with hexagonal prism shapes growing from the whisker seeds were aligned parallel and perpendicular to it. Vickers hardness was 13.9±0.3 GPa. The fracture toughness values were 6.1±0.4 $MPam^{1/2}$ and 5.2±0.2 $MPam^{1/2}$ in the directions parallel and perpendicular to the stacking direction, respectively. The comparative sample prepared from the powder compact exhibited Vickers hardness of 14.1 ±0.2 GPa and the fracture toughness of 4.5±0.2 $MPam^{1/2}$.

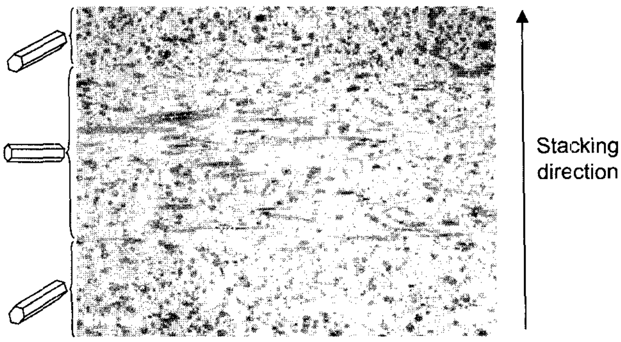



**Fig. 8.** Microstructure of the sintered body; surface parallel to both the casting direction and stacking direction; diagram on the left showing the orientations of the grains growing from the whiskers.

## 4. Conclusions

A virtual 3-D model with a complicated shape was successfully materialized using silicon nitride tape with aligned silicon nitride whisker seeds. In order to reduce the deformation of the ceramic tapes during stacking, the vinyl support was used. Cold isostatic pressing after binder burnout was necessary for making dense and sound body after sintering. The body was sintered to 98.5% TD and Vickers hardness was 13.9±0.3 GPa. The fracture toughness values parallel and perpendicular to the stacking direction were 6.1±0.4 $MPam^{1/2}$ and 5.2±0.2 $MPam^{1/2}$, respectively. The microstructural observation revealed that the layers with the aligned whisker seeds had cross-ply structure.

## Acknowledgement

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