Estimation of stress generated during co-sintering of porous and dense alumina layers

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Co-sintering was carried out in air at 1350°C for 1 hr to make two types of symmetrical 3-layered laminates. One is PDP (Porous alumina/Dense alumina/Porous alumina) laminate and another is DPD laminate. For the porous alumina layer, the porosity was set to be 20%; however, pore size was changed by adding three sizes of spherical mono-dispersed polymethyl methacrylate particles (5, 15, 30 μm) in raw alumina powder. Young's modulus measurements revealed that cracking occurred in both PDP and DPD laminates. The stress in the layer was estimated from an elastic calculation by using the mechanical and thermal expansion properties of each monolayer. In consideration of sintering shrinkage and thermal expansion of the layer, it was suggested that cracking occurred in the dense alumina layer during heating from 1000 to 1200°C, and in porous alumina layer during heating from 1200 to 1350°C. The cracking was mainly attributed to the mismatch of sintering shrinkage between the layers. From the discussion, the elastic model gave an explanation to crack formation during co-sintering.

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1. Introduction

For functional materials applications, ceramic thin films are sometimes made by powder processing. In this process, thin powder layer is cohesively bonded with a substrate at first; however, in-plane shrinkage is gradually arrested during sintering. To predict its sintering behavior, continuum mechanics model has been used in view of sintering stress and sintering viscosity.^{1)–8)} An isotropic model gave an explanation of densification of film-substrate systems.^{3),4)} And an isotropic finite element method also simulated the constrained sintering of the films.^{5)–8)} Recently, an anisotropic model was proposed, applying to a more complex case in constrained sintering.⁹⁾ In these researches, however, cracking was not explicitly considered because mechanical and thermal expansion properties of sintered materials (viz. the materials in the intermediate stage of sintering) were not fully discussed there.

Alumina has been used as a standard material for substrate applications, and investigated to attain its higher fracture strength. Sintering of symmetrical multilayers has also been studied with experiments on ceramic/metal/ceramic and glass/metal/glass layers.^{10),11)} Various damages were reported in pressureless-sintered $\text{Al}_2\text{O}_3/\text{ZrO}_2$ symmetric and asymmetric laminates made by tape casting and lamination.¹²⁾ However, there were a few reports on such the multilayers composed of different ceramics.

In film-substrate systems and ceramic multi-layers, cracking sometimes occurred during processing.^{13)–19)} One of the reasons for the cracking is the mismatch in thermal expansion between the layers.^{17)–19)} Another reason is the mismatch in sintering shrinkage during co-sintering.^{13)–16)} However, there is not so enough understanding based on both mismatches at present.

In this paper, porous and dense alumina layers were co-sintered to obtain symmetrical 3-layered laminates, and their cracking

behavior was observed. And the mechanical and thermal expansion properties were measured for sintered monolayers (dense alumina and three types of porous alumina with different pore sizes). A one-dimensional elastic model is proposed here based on both mismatches in thermal expansion and sintering shrinkage. By substituting data of monolayers in the model, the elastic stress is estimated in each layer as a function of sintering temperature, and compared with its strength. Finally, validity of the model was discussed together with the cracking behavior of 3-layered laminates.

2. Experimental procedure

2.1 Preparation and the mechanical properties of 3-layered laminates

Alumina powder (TAIMICRON TM–D, Taimei Chemicals co., LTD, Japan) was used as a raw material in this study. Its particle size was 0.19 μm and purity was 99.99%. The crystalline phase was corundum ($\alpha\text{-Al}_2\text{O}_3$). Three types of spherical mono-sized polymethyl methacrylate (PMMA) particles (Techpolymer, Sekisui Plastics co., LTD, Japan) were also used to make uniform-sized pores in the porous alumina layers. The particle sizes were 5 μm (MBX-5), 15 μm (MBX-15), and 30 μm (MBX-30), respectively.

For making porous alumina layer, the alumina powder was mixed with PMMA particles by ball milling for 24 hr in ethanol. The volume fraction of PMMA particles was determined to make the porosity of porous alumina layers be 20%. After eliminating ethanol by rotary evaporator, the mixed powder was dried at 105°C for 24 hr, and sieved with 500 μm opening.

In order to make 3-layered laminates, as shown in Fig. 1, pure alumina powder and the mixed powder were alternatively put into a mold (60 mm $\times$ 25 mm interval) and lightly pressed by hand with a die for each layer. Here, by the order of packing, there were two types of laminates such as Porous alumina/Dense alumina/Porous alumina laminate (hereafter PDP laminate) and

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Fig. 1. Stereo-micrographs of 3-layer laminates (Porosity 20% and pore size 30 μm). In PDP laminate, there are two macroscopic cracks in D layer. In DPD laminate, obvious cracking is not seen. A black line in upper D layer in DPD laminate is a marking by pensile (not a crack).

also DPD laminate. And the outer layers were weight 9 g and the center layer was 7 g due to the loss of outer layers by surface grinding. Following that, uniaxial pressing was conducted at 10 MPa for 2 min. The green compact was de-waxed in nitrogen flow at 400°C, and then sintered (viz. co-sintering) in air at 1350°C for 1 hr with the heating and cooling rate of 5°C/min. The co-sintered materials were surface-ground with a diamond wheel (#140) and cut into 5 mm $\times$ 5 mm $\times$ 48 mm in size so as to make the thickness of each layer almost the same. If it is exactly so, the total porosities of PDP and DPD laminates are 13.2 and 6.6% in calculation. The bulk density was measured by the mass and dimensions of the specimen. Cracking in the bending specimen was checked throughout its side surfaces with a stereomicroscope under a magnification of around 10–40. Finally, the specimens were chamfered for bending tests.

3-point bending test was conducted to measure bending strength and Young's modulus of PDP and DPD laminates. The span was 30 mm and crosshead speed was 0.5 mm/min. The calculation was made in reference to JIS1601 and 1602. The number of specimens was around 5–6 for each material.

2.2 Characterization for monolayers

To make monolayers of dense alumina and three types of porous alumina (with different pore sizes), each powder was directly compressed at 10 MPa for 2 min. Only the green compacts of porous alumina monolayers were de-waxed in nitrogen flow at 400°C. All the compacts were sintered in air at 800, 1000, 1200, 1350°C for 5 min, and 1350°C for 1 hr, respectively. The dimensions of the monolayer were measured before and after sintering, and the dimensions at the sintering temperature were estimated in consideration of thermal expansion coefficient of the sintered monolayer. The sintering strain $\varepsilon_i^{\text{SI}}(T)$ at the sintering temperature T , was defined as the logarithmic strain as below,

$$\varepsilon_i^{\text{SI}}(T) = \ln \frac{\ell_i(T)}{\ell_i(T_0)} = \ln \frac{\ell_i^{\text{CA}}(T_0)\{1 + \alpha^{\text{CA}}(T - T_0)\}}{\ell_i(T_0)}, \quad (1)$$

where $\ell_i(T_0)$ is the dimensions before sintering, $\ell_i(T)$ is the dimensions at the sintering temperature, $\ell_i^{\text{CA}}(T_0)$ is the dimensions measured at the room temperature T_0 after sintering, α^{CA} is the average thermal expansion coefficient of the sintered

monolayer, $i = 1$ for dense alumina monolayer and $i = 2$ for porous alumina monolayer.

For all the sintered monolayers (4 types of monolayers $\times$ 5 sintering conditions = 20 testing conditions in total), 3-point bending test was conducted in the same way as 3-layered laminates. The number of specimens was 4 for each condition. Young's modulus and bending strength were determined at room temperature. Although the mechanical properties at the sintering temperatures are actually needed for precise discussion, the data at room temperature is served to substitute for it in this study. If necessary, a factor (e.g. 0.9 for higher sintering temperatures) may be multiplied to the data at room temperature by referring to previous literatures^{20),21)} on mechanical properties of dense alumina at high temperatures. However, this modification does not cause a remarkable change in the discussion here.

To measure the thermal expansion strain $\varepsilon_i^{\text{TH}}(T)$ of each sintered monolayer, thermal dilatometer was used. The specimen was made by cutting a half piece after the bending test into 2.5 mm $\times$ 2.5 mm $\times$ 10 mm in size. The heating rate was 10°C/min, and the maximum temperature was set to be the sintering temperature of each specimen. The thermal expansion strain $\varepsilon_i^{\text{TH}}(T)$ at the sintering temperature T is defined as below,

$$\varepsilon_i^{\text{TH}}(T) = \alpha_i^{\text{CA}}(T - T_0), \quad (2)$$

where α_i^{CA} is the average thermal expansion coefficient of the sintered monolayer, T_0 is the room temperature.

3. Results and discussion

3.1 Cracking and mechanical properties of PDP and DPD laminates

For PDP laminate, the total porosity was around 18% and for DPD laminate 15%, both of which are larger than the nominal values (13.2 and 6.6% in calculation). As stated in section 3.2, the dense alumina monolayer (sintered at 1350°C for 1 hr) has an experimental porosity of 9.5% (not around 0% in this study) and porous alumina monolayers 18% (almost the same as 20%). By using these values, PDP laminate should have the total porosity of around 15% and DPD laminate 12%. The slight difference (around 3%) in total porosity may be affected by macroscopic cracking itself or densification inhibited by the cracking.

Figure 1 shows the appearance of the side surfaces of 3-layered laminates when the pore size is 30 μm in P layer. In the PDP laminate, some macroscopic cracks can be seen in D layer, whereas we do not see any macroscopic crack in the DPD laminate by this appearance check.

For PDP laminate, the detailed observation gives the information on the average number and length of the cracks, as shown in Fig. 2. The error bar is the standard deviation (If an error bar is not seen, the standard deviation is smaller than the symbol). When the pore size is 5 μm in P layer, there is 1 crack and its length is about 0.1 mm on average. With increasing the pore size, both the number and length of the cracks are increased. This comes from that the strength is lower when the pore size is larger in general.

Figure 3 shows bending strength and Young's modulus of PDP and DPD laminates. The error bar is the standard deviation. In this figure, DPD laminate have almost the same strength values around 250 MPa when the pore size is changed, however, the strength of PDP laminate are strongly dependent on the pore size. Although such the difference seems to correspond to the appearance check in Fig. 1, this must be apparent, because Young's moduli of PDP and DPD laminates have almost the same value around 110 GPa in Fig. 3. It means that DPD laminate was also damaged during sintering. This interpretation is supported by the porosity de-

Fig. 2. Average number and length of cracks in PDP laminate.

Fig. 3. Bending strength and Young's modulus of PDP and DPD laminates.

pendence of Young's modulus of alumina, as shown in Fig. 4, which was obtained by changing porosity of alumina in the same way as described in 2.2. The error bar is the standard deviation. From this figure, Young's modulus of PDP laminate must be around 210 GPa (porosity = 18%), and that of DPD 250 GPa

Fig. 4. Porosity dependency of Young's modulus of alumina.

Fig. 5. Bulk density of dense and three porous monolayers.

(porosity = 15%). However, the experimental values in both laminates are around 110 GPa, which are lower than the values expected in Fig. 4. From the discussion, the DPD laminate must have the same damage as the PDP laminate has, but the cracks were not detected by the appearance check. This is because they were smaller or inside the specimen. Another method for damage inspection will be necessary as a future work.

3.2 Mechanical and thermal expansion properties of the sintered monolayers

Figure 5 shows the change in bulk density of the sintered monolayers. There is not so large difference between three porous alumina monolayers. And the dense alumina monolayer has the same temperature dependency as the porous alumina layers although its value is slightly high. As stated before, the dense alumina monolayer has the porosity of 9.5% and porous alumina monolayers around 18% when they were sintered at 1350°C for 1 hr.

Figure 6 shows Young's modulus of the sintered monolayers. The error bar is the standard deviation. The dense alumina monolayer shows a rapid increase with increasing the sintering temperature, and it becomes 380 GPa when it was sintered at 1350°C for 1 hr. Although three porous alumina monolayers should have almost the same Young's modulus because of equal

Fig. 6. Young's modulus of dense and three porous monolayers.

Fig. 7. Bending strength of dense and three porous monolayers.

porosity (refer to bulk density in Fig. 5), 5 μm porous alumina monolayer shows a higher value than the others. It suggests that pore size also affects Young's modulus in some case, leading to different values at fixed porosity.

Figure 7 shows the bending strength of the sintered monolayers. The error bar is the standard deviation. In the order of dense, 5 μm porous, 15 μm porous, and 30 μm porous alumina monolayers, the bending strength is gradually decreased at each sintering temperature. In section 3.3, these values will be compared to the stress values estimated from a model proposed in this study.

Figure 8 shows the thermal expansion strain of the sintered monolayers. From this figure, we see the thermal expansion strain is in the order of 10^{-2} and there is not so large difference between dense and three porous alumina monolayers.

Figure 9 shows the sintering strain of the sintered monolayers. When we look at this figure carefully, the sintering strain of dense alumina monolayer decreases faster than those of the porous layers from 1000 to 1200°C, however this relation is reversed from 1200 to 1350°C. Compared with the thermal expansion strain, the sintering strain is one order of magnitude larger than it. This means that stress generated during heating may be mainly caused by the difference in sintering strain between dense alumina layer and porous alumina layer. Later (in section 3.3), we

Fig. 8. Thermal expansion strain of dense and three porous monolayers.

Fig. 9. Sintering strain of dense and three porous monolayers.

develop a one-dimensional model to estimate the elastic stress generated during co-sintering.

This sintering strain in Fig. 9 must correspond to the bulk density in Fig. 5. Suppose that a cubic powder compact with mass M_i and length L_{0i} at room temperature T_0 , and this compact is sintered at the temperature T . The bulk density $d_i(T_0)$ before sintering is expressed in the next equation,

$$d_i(T_0) = \frac{M_i}{L_{0i}^3} \quad (3)$$

From the sintering strain in Eq. (1), the dimensions L_i of the compact at the sintering temperature T is given in the following equation,

$$L_i = L_{0i} e^{\epsilon_i^{sl}(T)} \quad (4)$$

By using Eq. (4), we get the bulk density $d_i(T)$ at the sintering temperature T as below,

$$d_i(T) = \frac{M_i}{L_i^3} = \frac{M_i}{L_{0i}^3 e^{3\epsilon_i^{sl}(T)}} = d_i(T_0) e^{-3\epsilon_i^{sl}(T)} \quad (5)$$

The bulk density measurement was done at the room temperature, so that we should consider thermal shrinkage from the sintering temperature T to room temperature T_0 , and then we derive the bulk density $d_i^{CA}(T_0)$ after sintering as below,

$$\begin{aligned}
d_i^{CA}(T_0) &= \frac{M_i}{L_i^3 \{1 + \alpha_i^{CA}(T) \{(T_0 - T)\}\}^3} \\
&= \frac{M_i}{L_{0i}^3 e^{3\varepsilon_i^{SI}(T)} \{1 + \alpha_i^{CA}(T) \{(T_0 - T)\}\}^3} \\
&= d_i(T_0) e^{-3\varepsilon_i^{SI}(T)} \{1 + \alpha_i^{CA}(T) \{(T_0 - T)\}\}^{-3}, \quad (6)
\end{aligned}$$

where $\alpha_i^{CA}(T)$ is thermal expansion coefficient of the sintered monolayer at T . If we estimate the bulk density $d_{\text{DENSE}}^{CA}(20^\circ\text{C})$ of dense alumina monolayer sintered at 1350°C for 5 min, the necessary parameters are the sintering strain $\varepsilon_{\text{DENSE}}^{SI}(1350^\circ\text{C}, 5 \text{ min}) = -2.02 \times 10^{-1}$, thermal expansion coefficient $\alpha_{\text{DENSE}}^{CA}(1350^\circ\text{C}, 5 \text{ min}) = 7.89 \times 10^{-6} \text{ K}^{-1}$, and bulk density before sintering $d_{\text{DENSE}}(20^\circ\text{C}) = 1.84 \text{ g/cm}^3$ (here, the bulk density of dense alumina monolayer before sintering, is approximated by that after sintering at 800°C . When we see Fig. 5, this approximation does not cause any serious problem in this discussion). By substituting these parameters and also $T_0 = 20^\circ\text{C}$, $T = 1350^\circ\text{C}$ into Eq. (6), we get 3.48 g/cm^3 as $\alpha_{\text{DENSE}}^{CA}(20^\circ\text{C})$. This estimate is almost the same as the experimental bulk density (3.52 g/cm^3) after sintering at 1350°C for 5 min. It shows that the bulk density data corresponds well to the sintering strain data.

3.3 Estimation of stress in each layer based on one-dimensional elastic model

In this section, one-dimensional elastic analysis is used for 3-layered laminates. The volume fractions of two different layers are proportional to the total thickness of two outer layers and that of the center layer. Here, subscripts 1 and 2 denote the dense alumina and porous alumina layers, and 3 kinds of strains are considered; elastic strain ε^{EL} , sintering strain ε^{SI} , and thermal expansion strain ε^{TH} . These strains are expressed by the following equations,

$$\varepsilon_1^{\text{EL}} = \frac{\sigma_1(T)}{E_1(T)} \quad (7)$$

$$\varepsilon_2^{\text{EL}} = \frac{\sigma_2(T)}{E_2(T)} \quad (8)$$

$$\varepsilon_1^{\text{SI}}(T) = \ln \frac{\ell_1(T)}{\ell_1(T_0)} \quad (9)$$

$$\varepsilon_2^{\text{SI}}(T) = \ln \frac{\ell_2(T)}{\ell_2(T_0)} \quad (10)$$

$$\varepsilon_1^{\text{TH}}(T) = \alpha_1^{CA}(T) \{(T - T_0)\} \quad (11)$$

$$\varepsilon_2^{\text{TH}}(T) = \alpha_2^{CA}(T) \{(T - T_0)\}, \quad (12)$$

where T is the sintering temperature, T_0 is the reference temperature (0°C in this chapter), $\sigma_i(T)$ is elastic stress, $E_i(T)$ is Young's modulus, ℓ_i is the dimensions of the sintered monolayer, α_i^{CA} is thermal expansion coefficients of the sintered monolayer.

The balance of force can be given by the following equation,

$$f_1 \sigma_1(T) + (1 - f_1) \sigma_2(T) = 0, \quad (13)$$

where f_i is the volume fraction of the dense alumina layers. And the total strain $\varepsilon_{\text{total}}(T)$ of the laminate is expressed by,

$$\begin{aligned}
\varepsilon_{\text{total}}(T) &= \frac{\sigma_1(T)}{E_1(T)} + \varepsilon_1^{\text{SI}}(T) + \varepsilon_1^{\text{TH}}(T) \\
&= \frac{\sigma_2(T)}{E_2(T)} + \varepsilon_2^{\text{SI}}(T) + \varepsilon_2^{\text{TH}}(T) \quad (14)
\end{aligned}$$

By substituting Eq. (13) into Eq. (14), we obtain the elastic stress in each layer during heating as below,

$$\begin{aligned}
\sigma_1(T) &= \left[\frac{-(1 - f_1) E_1(T) E_2(T)}{f_1 E_1(T) + (1 - f_1) E_2(T)} \right] \\
&\times \{[\varepsilon_1^{\text{SI}}(T) - \varepsilon_2^{\text{SI}}(T)] + [\varepsilon_1^{\text{TH}}(T) - \varepsilon_2^{\text{TH}}(T)]\} \quad (15)
\end{aligned}$$

$$\begin{aligned}
\sigma_2(T) &= \left[\frac{f_1 E_1(T) E_2(T)}{f_1 E_1(T) + (1 - f_1) E_2(T)} \right] \\
&\times \{[\varepsilon_1^{\text{SI}}(T) - \varepsilon_2^{\text{SI}}(T)] + [\varepsilon_1^{\text{TH}}(T) - \varepsilon_2^{\text{TH}}(T)]\} \quad (16)
\end{aligned}$$

During keeping at the sintering temperature T_{max} (1350°C in this study), the elastic stress can be given by the equations as below,

$$\begin{aligned}
\sigma_1(T_{\text{max}}, t) &= \left[\frac{-(1 - f_1) E_1(T_{\text{max}}, t) E_2(T_{\text{max}}, t)}{f_1 E_1(T_{\text{max}}, t) + (1 - f_1) E_2(T_{\text{max}}, t)} \right] \\
&\times \{[\varepsilon_1^{\text{SI}}(T_{\text{max}}, t) - \varepsilon_2^{\text{SI}}(T_{\text{max}}, t)] \\
&+ [\varepsilon_1^{\text{TH}}(T_{\text{max}}) - \varepsilon_2^{\text{TH}}(T_{\text{max}})]\} \quad (17)
\end{aligned}$$

$$\begin{aligned}
\sigma_2(T_{\text{max}}, t) &= \left[\frac{f_1 E_1(T_{\text{max}}, t) E_2(T_{\text{max}}, t)}{f_1 E_1(T_{\text{max}}, t) + (1 - f_1) E_2(T_{\text{max}}, t)} \right] \\
&\times \{[\varepsilon_1^{\text{SI}}(T_{\text{max}}, t) - \varepsilon_2^{\text{SI}}(T_{\text{max}}, t)] \\
&+ [\varepsilon_1^{\text{TH}}(T_{\text{max}}) - \varepsilon_2^{\text{TH}}(T_{\text{max}})]\}, \quad (18)
\end{aligned}$$

where t is the keeping time at the sintering temperature.

On the other hand, during cooling, the elastic stress is estimated by the following equations,

$$\begin{aligned}
\sigma_1(T) &= \left[\frac{-(1 - f_1) E_1(T_{\text{max}}, 1 \text{ hr}) E_2(T_{\text{max}}, 1 \text{ hr})}{f_1 E_1(T_{\text{max}}, 1 \text{ hr}) + (1 - f_1) E_2(T_{\text{max}}, 1 \text{ hr})} \right] \\
&\times \{[\varepsilon_1^{\text{SI}}(T_{\text{max}}, 1 \text{ hr}) - \varepsilon_2^{\text{SI}}(T_{\text{max}}, 1 \text{ hr})] \\
&+ [\varepsilon_1^{\text{TH}}(T) - \varepsilon_2^{\text{TH}}(T)]\} \quad (19)
\end{aligned}$$

$$\begin{aligned}
\sigma_2(T) &= \left[\frac{f_1 E_1(T_{\text{max}}, 1 \text{ hr}) E_2(T_{\text{max}}, 1 \text{ hr})}{f_1 E_1(T_{\text{max}}, 1 \text{ hr}) + (1 - f_1) E_2(T_{\text{max}}, 1 \text{ hr})} \right] \\
&\times \{[\varepsilon_1^{\text{SI}}(T_{\text{max}}, 1 \text{ hr}) - \varepsilon_2^{\text{SI}}(T_{\text{max}}, 1 \text{ hr})] \\
&+ [\varepsilon_1^{\text{TH}}(T) - \varepsilon_2^{\text{TH}}(T)]\} \quad (20)
\end{aligned}$$

In these equations, the thermal expansion strain $\varepsilon_i^{\text{TH}}(T)$ is defined as,

$$\varepsilon_1^{\text{TH}}(T) = \alpha_1^{\text{SI}}(T - T_0) \quad (21)$$

$$\varepsilon_2^{\text{TH}}(T) = \alpha_2^{\text{SI}}(T - T_0), \quad (22)$$

where α_i^{SI} is the thermal expansion coefficient of the sintered monolayer (at 1350°C for 1 hr).

Table 1 shows the estimated elastic stress in each layer during heating and also keeping at the sintering temperature, assuming that $f_1 = 1/3$, $f_2 = 2/3$ for PDP laminate, and $f_1 = 2/3$, $f_2 = 1/3$ for DPD laminate. In this table, Roman type face denotes that the estimated stress is lower than the strength of the sintered specimen at the temperature. And Bold Italic type face denotes that the estimated stress is higher than the strength at the temperature, leading to possible cracking. For PDP laminate, at 800°C and 1000°C , the stress is very low in both layers independent of pore size, however, at 1200°C , the stress in the dense alumina layer becomes several hundreds MPa in tension, which is higher than the strength of each sintered specimen. It suggests that the macroscopic cracks in Fig. 1 may be formed during heating from 1000 to 1200°C in PDP laminate. When the temperature reaches 1350°C , high tensile stress is generated in the porous alumina layers and high compression stress in the dense alumina layer, and these stress states are maintained at the end of keeping (at 1350°C for 1 hr) although the stress value is changed. And we see that possible cracking occurs in the porous alumina layers during heating from 1200 to 1350°C . For DPD laminate, the tendency is the almost the same as PDP laminate. This agrees well with the discussion on Young's modulus in Fig. 3.

Table 2 shows the estimated elastic stress in each layer during cooling from the sintering temperature. The change in the stress during cooling is within several tens MPa, and the porous

Table 1. Estimated stress in laminates during heating and also keeping at 1350°C. Bold Italic typeface means the estimated stress is higher than its strength, leading to possible cracking

DPD		Estimated stress in DPD/MPa				
Pore size / μm	Temp. Layer	800°C (5 min)	1000°C (5 min)	1200°C (5 min)	1350°C (5 min)	1350°C (1 h)
5	D	-1	-1	98	-460	-240
	P	3	1	-200	930	480
15	D	0	3	210	-260	-930
	P	-1	-7	-430	530	1900
30	D	0	3	90	-33	-790
	P	-1	-5	-180	67	1600

PDP		Estimated stress in PDP/MPa				
Pore size / μm	Temp. Layer	800°C (5 min)	1000°C (5 min)	1200°C (5 min)	1350°C (5 min)	1350°C (1 h)
5	D	-3	-1	230	-1040	-550
	P	2	1	-110	510	270
15	D	1	10	560	-670	-2500
	P	0	-5	-280	330	1300
30	D	1	9	260	-86	-2100
	P	0	-4	-130	42	1000

Table 2. Estimated stress in laminates during cooling from 1350°C. Bold Italic typeface means the estimated stress is higher than its strength, leading to possible cracking

DPD		Estimated stress in DPD/MPa				
Pore size / μm	Temp. Layer	1350°C (1 h)	1200°C	1000°C	800°C	0°C
5	D	-240	-230	-220	-220	-190
	P	480	470	450	440	380
15	D	-930	-930	-920	-910	-870
	P	1900	1900	1900	1800	1800
30	D	-790	-780	-770	-760	-710
	P	1600	1600	1600	1500	1400

PDP		Estimated stress in PDP/MPa				
Pore size / μm	Temp. Layer	1350°C (1 h)	1200°C	1000°C	800°C	0°C
5	D	-550	-540	-520	-500	-440
	P	270	270	260	250	210
15	D	-2500	-2500	-2500	-2500	-2400
	P	1300	1200	1200	1200	1200
30	D	-2100	-2000	-2000	-2000	-1800
	P	1000	1000	980	970	910

alumina layer still has high tensile stress when the temperature reaches 0°C in any case.

By this analysis, we conclude that, in PDP and DPD laminates, cracking may occur in the dense alumina layer during heating from 1000 to 1200°C, and then in the porous alumina layer during heating from 1200 to 1350°C. It leads to almost the same cracking and damage in both laminates. Needless to say, we note that elastic analysis usually gives the overestimate in stress and also that cracking itself affects the stress state in each layer after the cracking. And also we should say that the sintered materials do not always show elastic stress/strain relation but elasto-plastic or visco-elastic one at high temperatures. However, the mismatch term $\{[\varepsilon_1^{\text{SI}}(T) - \varepsilon_2^{\text{SI}}(T)] + [\varepsilon_1^{\text{TH}}(T) - \varepsilon_2^{\text{TH}}(T)]\}$ in Eqs. (15) and (16) determines the sign of stress (viz. tensile or compression),

independent of constitutive equation. The remained term containing Young's modulus and volume fraction is a conversion factor from strain to stress. When we think so, this elastic model captures the essence of stress state in each layer by relatively simple experiments and calculations, and gives the first approximation of stress change in each layer.

4. Conclusion

Porous and dense alumina layers were co-sintered to obtain symmetrical 3-layered laminates, and their cracking behavior was observed. And the mechanical and thermal expansion properties were measured for sintered alumina monolayer (dense alumina and three types of porous alumina with different pore sizes). A one-dimensional elastic model is proposed here based on both mismatches in thermal expansion and sintering shrinkage. By this elastic analysis, it is concluded that cracking may begin in the dense alumina layer during heating from 1000 to 1200°C and in porous alumina layer during heating from 1200 to 1350°C. The cracking was mainly attributed to the mismatch of sintering shrinkage between the layers. PDP and DPD laminates may have the almost the same cracking and damage. From the discussion, the elastic model gave an explanation to crack formation during co-sintering.

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