Optimal heating trajectories of controlling green tapes of YSZ electrolyte deformation using TMA during thermal processing

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Abstract

The optimal heating trajectories to minimize the time required for the organic additives removal in yttria-stabilized zirconia (YSZ) green tapes were determined using a dynamic optimization method. The removal process model was described by the mass transport of the volatile gas evolved from the thermal decomposition of the organic additives inside the tapes and the kinetics of the decomposition. The pressure buildup of the sample tapes formed by the volatile gas can be estimated by a numerical simulation method; meanwhile, the deformation (strain) of the tape caused by the pressure buildup was measured by a thermal mechanical analyzer (TMA) during the thermal processing. Results show that the formation of the maximum pressure buildup at the center of the cubic tape is influenced by the sample size and heating conditions. In addition, the dynamic strain at the center of the sample measured by TMA agrees with the formation of the pressure buildup estimated by the numerical calculation. Moreover, the optimal heating trajectories determined by the dynamic optimization scheme with the constraint of the formation of the maximum pressure buildup were verified from the tape deformation analysis by the TMA tests.
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Keywords: Dynamic optimization; YSZ green tape; Organic additives burnout; Numerical simulation; Thermal mechanical analysis

1. Introduction

Electrolytes are one of the essential components to transport oxide ions for solid oxide fuel cells (SOFCs). The quality and efficiency of SOFCs relies on the performances of the electrolytes when operated between 600 and 1000 °C. The electrolytes have to be fabricated to achieve some requirements, such as high ionic conductivity, leaking prevention, and thermal-shock resistance. The manufacturing of the electrolyte of solid oxide fuel cells has been developed utilizing a ceramic processing method. The beginning of the process is to prepare slurry by mixing YSZ powders with solvents, powder dispersants, plasticizers and polymer binders. The prepared slurry is coated on substrates to form green tapes of the ceramic components with desired thicknesses or devices using a shape forming technique, i.e. tape-casting. The produced green tapes

are then to be thermally treated by a firing cycle, including binder (organic additives) burnout, ceramic sintering, and cooling steps [1].

Thermal removal of the organic components in green tapes of the organic/YSZ composite is one of the critical heating processes to fabricate high quality of the electrolytes for solid oxide fuel cells. During the removal process, the organic additives are decomposed into volatile gas which evolves through the ceramic component when the operation is above the decomposition temperatures. If a large amount of the evolving gas accumulates inside the ceramic, a large pressure buildup is generated to deform the ceramic body as depicted in Fig. 1. The pressure buildup to generate the defects was proposed due to the formation of the evolving gas and the gas transport out of the ceramic body. Defects, such as cracks, can be formed inside the tapes due to the large buildup pressure formation [2]. Recent studies were focused on controlling the formation rate of the evolved gas (kinetics) with respect to the adopted heating strategy to avoid the forming of the pressure buildup inside ceramic components [3,4]. Therefore, the phenomena of the organic additives

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Nomenclature

E_a	activation energy ($J\,mol^{-1}$)
g	process model
h	constraint
k_0	rate constant (s^{-1})
k_i	Kozeny–Carman parameter
K_i	permeability (m^2)
L_x	length (m)
L_y	width (m)
L_z	height (m)
M	molecular weight ($Kg\,mol^{-1}$)
n	reaction order
P	pressure (atm)
P_0	initial pressure (atm)
r	reaction rate ($Kg\,m^3\,s$)
R	gas constant ($J\,mol\,K$)
S	surface area per unit volume (m^{-1})
t	time (s)
T	temperature (K)
T_0	initial temperature (K)
u	flow rate ($m\,s^{-1}$)
v_b	volumetric ratio of organic additives
v_{b0}	initial volumetric ratio of binder
v_g	volumetric ratio of gas
v_{g0}	initial volumetric ratio of gas
v_s	volumetric ratio of ceramic

Greek letters

α	conversion
β	heating rate ($K\,min^{-1}$)
χ	state variable
μ	viscosity (Pa s)
ρ	gas molar density ($mol\,m^{-3}$)
$\bar{\rho}$	dimensionless gas density
ρ_b	organic additives density ($kg\,m^{-3}$)

removal play an important role on the defect formations in the ceramic manufacturing.

Understanding the thermal removal phenomena of the organic additives is a key issue to avoid defects and obtain better quality and higher yield of the products. Kinetics of thermal degradation of binder (i.e. PVB) with inorganic materials has been investigated using analytical approaches. The kinetic
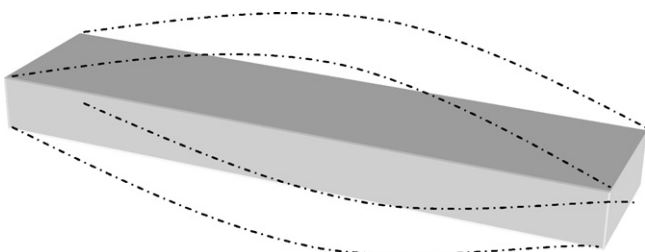

Fig. 1. Deformation effect of a green tape during thermal processing of organic additives removal.

effects of YSZ on the thermal decomposition of organic additives were studied in our previous work [5]. The catalytic effects of the ceramics on the binder decomposition were shown to accelerate the decomposition rate in lower temperatures. These analytical data can be further applied to interpret and demonstrate the formation of the pressure buildup.

The mechanistic description of the volatile gas diffusing from the void space inside the ceramic to the body surface was reported [2]. The fluid flow of the volatile gas in the ceramics was taken as a critical condition of the generation of the internal pressure buildup and the stresses on the ceramic body. These forces have great influences on the formation of the defects and failures on the body [6]. The fluid flow model of the gaseous products inside a porous media had been mathematically developed in three-dimensional space and assumed uniform temperature distribution for a cubic body [7]. The distribution of the internal pressure and stresses within the body were numerically solved using the mass transport model and the kinetics of the binder burnout [8]. The effects of product yield of different shapes of ceramic bodies on the heating process were investigated after carrying out different heating conditions [9]. However, little research was conducted on verifying the assurance of the proposed process model by practical measurements of the sample deformation. If the reliability of the process model is confirmed, the numerical simulation applying the process model can be practically applied on the engineering design and operation for the binder removal process.

In this study, the optimal heating strategies under the constraint of maximum internal pressure formation inside the cubic tape were determined to remove the organic additives by the numerical simulation method. The effects of sample geometries and heating conditions on the thermal treatment of the tapes were discussed. The buildup pressure and the deformation at the center of the cubic tape were evaluated by the numerical calculation and the TMA experimental measurement, respectively. The minimum time required for the organic additives removal by controlling the internal pressure formation was carried out by the optimal heating strategy to avoid large stress formation. In addition, the determined heating strategy was applied on the TMA tests to dynamically correlate the pressure buildup with the tape deformation.

2. Process modeling and optimization strategy

The process model can be described by the physical and chemical theories and phenomena of the thermal removal of the organic additives. The mass transport of the volatile gas generated from the organic decomposition describes the organic removal phenomena during the thermal processing; while kinetics of organic thermal decomposition is an approach to evaluate the generation rate of the volatile gas corresponding to the operating temperatures.

The temperature inside the organic/ceramic composite distributes non-isothermally from a heat transport perspective. However, if the temperature difference inside the small sample size (i.e. $1\,cm \times 1\,cm \times 1\,cm$) is not significant due to the slow temperature ramping during the burnout period, then the
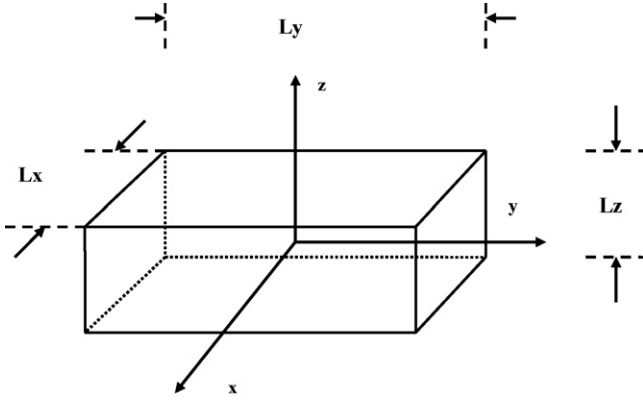
Fig. 2. Geometry of the ceramic cubic and the coordinate system.

temperature distribution can be assumed uniformly inside the body. The process model can be developed as the following.

2.1. Equation of continuity

The flow system in 3D directions for the evolved gas in a rectangular ceramic tape as depicted in Fig. 2 is described as

$$\frac{\partial(\rho v_g)}{\partial t} = -\left[\frac{\partial}{\partial x}(\rho u_x) + \frac{\partial}{\partial y}(\rho u_y) + \frac{\partial}{\partial z}(\rho u_z)\right] + r \quad (1)$$

where ρ is the molar density, v_g is the volumetric ratio of gas, u_i is the flow rate in i-direction, and r is the decomposition rate. The flow rate in porous media can be expressed using Darcy's Law

$$u_i = -\frac{\kappa_i}{\mu}\frac{\partial P}{\partial i} \quad (2)$$

where μ is the fluid viscosity, P is the pressure, and k_i is the diffusion coefficient described using Kozeny–Carman equation as

$$\kappa_i = \frac{v_g{}^3}{k_i(1 - v_g)^2 S^2} \quad (3)$$

where k_i is a constant related to the shape and tortuosity of the pores, and S is the surface area per unit volume of the ceramic body. It is assumed that the S and k_i (typically between 3 and 7) values remain constant due to the small pore size and high ceramic composition of the sample to simplify the parameters determination. From Darcy's law, the fluid flow can be assumed as a laminar and slip flow if the ratio of the mean free path to the porosity radius is less than 1. In addition, if the ideal gas law is assumed, the pressure associated with the molar density can be expressed as

$$P = \rho RT \quad (4)$$

The detailed formulation of a cubic case was derived and found in our previous study [5].

2.2. Kinetic equation of binder decomposition

The decomposition rate can be expressed as

$$r = \frac{d\alpha}{dt} = k_0 \exp(-\frac{E_a}{RT})(1 - \alpha)^n \quad (5)$$

where α is the organic species conversion. If let $\beta = dT/dt$, then Eq. (5) becomes

$$r = \frac{d\alpha}{dT} = k_0 \exp\left(-\frac{E_a}{RT}\right)\left(\frac{(1 - \alpha)^n}{\beta}\right) \quad (6)$$

We can also replace α using ρ_b and the volumetric ratio of the organic additives (v_b), then Eq. (6) can be expressed as

$$r = -\frac{d(\rho_b v_b)}{dT} = k_0 \exp\left(-\frac{E_a}{RT}\right)\left(\frac{v_b}{v_{b0}}\right)^n \frac{\rho_b v_{b0}}{\beta} \quad (7)$$

and

$$v_b = v_{b0} - \int_0^t \frac{r}{\rho_b}dt \quad (8)$$

where v_{b0} is the initial volumetric ratio of the organic additives. The additives density, ρ_b, is taken as a constant in this work. The volumetric relationship among the volatile gas, the ceramic, and the additives is

$$v_g = 1 - v_s - v_b \quad (9)$$

where v_s is the volumetric ratio of the ceramic.

The process model can be expressed by the use of continuity equation and the kinetic equation of the polymer decomposition as described above. This problem can be numerically solved using finite-difference method with Crank–Nicholson algorithm to simulate the pressure distribution inside a 3D ceramic body.

2.3. Process optimization

The optimal control problem in this work is described as

$$\begin{aligned} &\underset{\beta}{\text{Min}} \quad t_f \\ \text{such that} \quad &\beta = \frac{dT}{dt} \\ &g(\beta, x) = 0 \\ &h(\beta, x) \leq 0 \end{aligned} \quad (10)$$

where t_f is the total time required to remove the polymer binder, β is the heating rate, x is the state variable, g is the process model, h is the inequality constraint. The process model can be expressed by the use of continuity equation and the kinetic equation of the polymer decomposition as described above. This problem can be numerically solved using finite-difference method with Crank–Nicholson algorithm to simulate the pressure distribution inside a ceramic body. Both cylindrical and cubic samples were selected for the simulation study corresponding to 10-grid points for each dimension in the finite-difference calculation. The optimization approach was utilized the successive quadratic programming (SQP) method which programming was provided by MATLAB Corp. The procedure of solving the dynamic optimization problem can be found in our previous work [5].

3. Experimental

The slurry of the YSZ green tapes was prepared using a commercial yttria-doped zirconia powder (HWA-ZY8, Hanwha, Australia) added into a methyl ethyl ketone (MEK)/isopropanol (ISP) (60/40 vol%) and dispersants mixture. Three dispersants, polyethyleneimine (PEI, Aldrich), fish Oil (sigma), and Terpineol (Fluka) were adopted. After polyvinyl butyral (PVB) the polymer binder was added into the slurry solution and mixed for 1 h, the plasticizer, a mixture of polyethylene glyco1(PEG) and dibutyl phthalate (DBP, Fluka) was added into the solution stirred for 24 h.

The green tapes were produced using the prepared slurry coated on glass substrates by a doctor-blade (Elcometer 3580) method [10]. Then, several green tapes with a thickness about 100 μm were laminated to make it as a required sample size. Thermo-Mechanical Analyzer (TMA) model 7e provided by Perkim-Elmer Corp. was used for the deformation measurement under different heating conditions. A sample size with 0.5 cm × 0.5 cm × 0.5 cm was used for the sample deformation test. A probe sensor was contact on top of the prepared sample to measure the position change during the thermal processing. The TMA can be programmable to be set up as a heating function and control to the required heating rates and recorded on a continuous basis. Thermogravimetric analyzer (TGA) model Q-50 provided by TA INSTRUMENT was used for the TG data collections and measurements. About 10 mg of the sample weight was used in each experiment and the flow rate of the carrier gas was kept at 50 cm^3 min^{-1}. The TG can be programmable to be set up as a heating function and control the heating rates and recorded on a continuous basis.

4. Results and discussion

The weight loss of the organic additives in the green tape can be analyzed using TG data as shown in Fig. 3. The TG curves show that the significant weight loss of the organic additives distributes in two regions for different heating conditions during the thermal treatment. The first weight loss period begins at 370 K and ends at 450 K due to the thermal decomposition of small molecular species of the additives; the other period is between 470 and 670 K for the thermal decomposition of large molecular species. Kinetic parameters of the thermal decomposition of the organic species can be analyzed using the TG data and an iso-conversion principle. The analytical results of the parameters were determined in our previous study and listed in Table 1 [5]. Besides, the operating parameters of the thermal process are listed in Table 2. These data are further utilized to estimate pressure buildup by the numerical simulation in the following study.
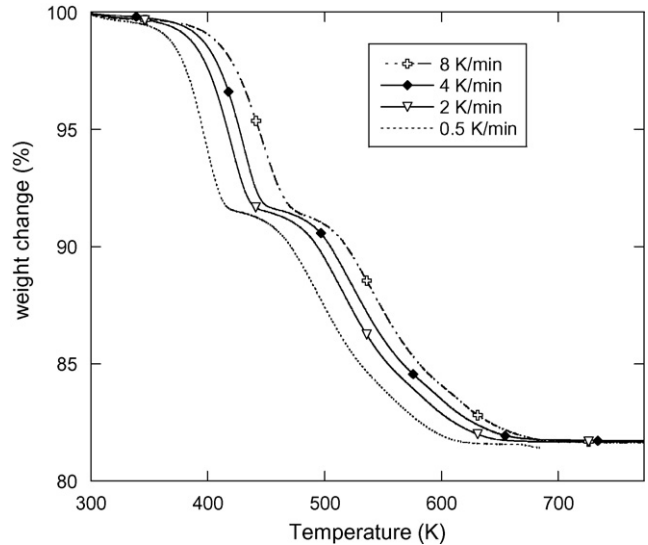

Fig. 3. TG curves of thermal degradation of the green tape samples in different heating conditions.

Table 1
The operating parameters for the process simulation

T_0	300 K
P_0	1 atm
v_s	0.482
v_{b0}	0.375
v_{g0}	0.143
β	0.5, 2,4, 8 K min^{-1}
ρ_b	1100 Kg m^{-3}
μ	0.025 × 10^{-3} Pa s
S	10.8 × 10^6 m^{-1}
k_i	5
L_x, L_Y, L_z	0.5, 0.5, 0.5 cm
M	0.044 kg mol^{-1}

4.1. Pressure buildup and deformation of the green tape

The distribution of the pressure buildup inside the ceramic body was first numerically solved using the 3D process model and the kinetic equation of the thermal decomposition of the organic species. The maximum internal pressure formed inside the ceramic body was observed at the center of the cubic sample during the thermal treatment. Therefore, the buildup pressure distribution at the center was taken as a critical point and used in this work.

Fig. 4 shows the internal pressure distribution at the cubic center for the treated green tape (5 cm × 5 cm × 1 cm), associated with temperature at different heating rates. A large internal pressure was generated for the thermal treatment for operating under a high heating rate condition, i.e. 10 K min^{-1}, inside the

Table 2
Kinetic parameters of the thermal decomposition of PVB/ceramic samples determined using TG data

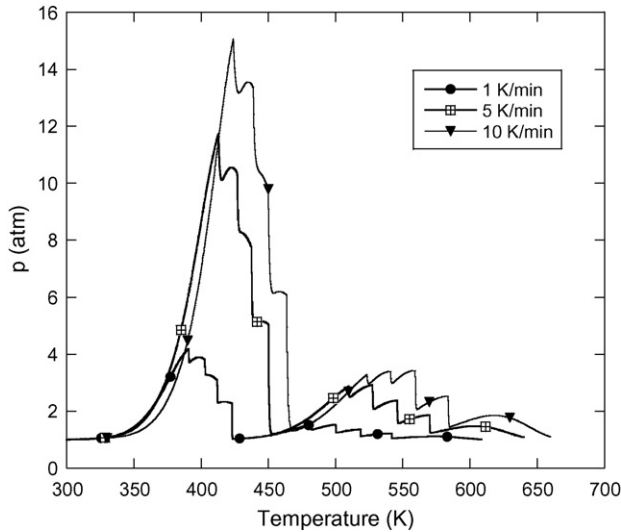| α	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
E_a (KJ mol^{-1})	89	87	85	84	110	124	133	138	142
ln (A) (min^{-1})	22.5	21.7	21	19.9	22.9	25.9	27.1	27.3	26.6

Fig. 4. The formation of the pressure buildup for the tape sample (5 cm × 5 cm × 1 cm) heated at different heating rates.

Fig. 6. The deformation of the samples measured by TMA when heated at different conditions.

cubic sample, where thermal decomposition rate of the volatile gas can be accelerated; whereas, the pressure buildup can be reduced significantly if a lower heating rate, i.e. 1 K min^{-1}, is applied on this case. In addition, two different regions of the pressure buildup distribution were observed as shown in this figure. The apparent pressure buildup was formed for the temperature at 400 K due to the thermal decomposition of the lower molecular organic additives; while the small peak of the pressure were observed in the region around 550 K corresponding to the thermal decomposition of the higher molecular *weight* of organic additives.

Different sample sizes were used to estimate the formation of the pressure buildup at the center of the cubic sample. Fig. 5 shows the pressure buildup distribution for different sample sizes (0.5 cm × 0.5 cm × 0.5 cm). The maximum internal pressure was reduced significantly inside the cubic sample if a

smaller sample size was treated, from the comparison between Figs. 4 and 5. In general, the presence of the maximum pressure formed inside the green tape is one of the critical points for the defect appearance. Therefore, the internal pressure formation has to be seriously considered in order to maintain the perfection of the tape sample during the thermal processing.

The other approach to investigate the effect of the heating conditions on the sample relies on the measurements of the sample deformation, caused by the presence of the volatile gas. The experimental test was utilized a TMA to detect the deformation on the surface of the sample (0.5 cm × 0.5 cm × 0.5 cm). Fig. 6 shows the distribution of the TMA probe position at the cubic center as a function of the heating temperatures for different heating rates. A larger deformation was observed as the heating rate increased. For example, the strain distribution appeared significant for the case of heating rate at 10 K min^{-1} between 400 and 500 K. After 500 K, the position of the probe sensor is dramatically dropped to negative values for all of the cases. This reveals that the ceramic powders surrounding by the organic additives removed thermally were relocated and occupied the available empty space where the additives were removed during the thermal treatment.

The experimental and numerical results of the thermally treated samples were analyzed and compared under different heating conditions. Fig. 7 shows the distribution of both pressure buildup and the sample deformation (strain) for different heating conditions. The sample deformation is related to the pressure buildup caused by the mass transport of the gas evolving from the thermal decomposition of the organic additives. The temperatures of the maximum strain generation appear at 380, 410, and 430 K corresponding to the heating rates at 1, 5, and 10 K min^{-1}. The internal pressure and the strain values became higher when the heating rate increased. Furthermore, the trend of the strain distribution is quite similar to the one of the internal pressure distribution during the thermal treatment except the case of 10 K min^{-1}. The variation of the distribution for the 10 K min^{-1} case is probably due to the instrument limi-
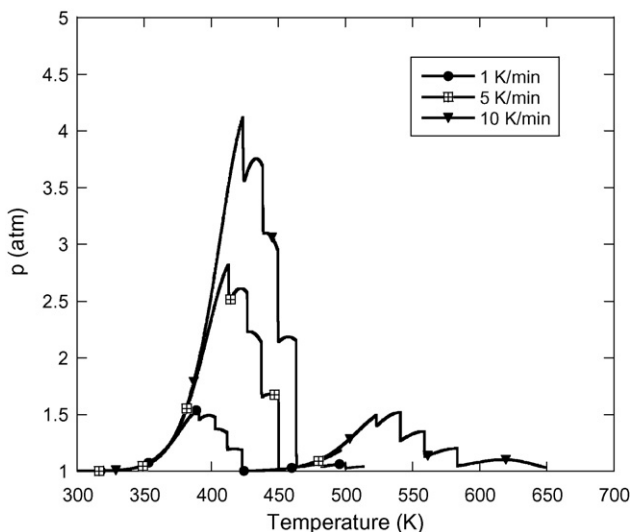

Fig. 5. The formation of the pressure buildup for the tape sample (0.5 cm × 0.5 cm × 0.5 cm) thermally heated at different heating rates.

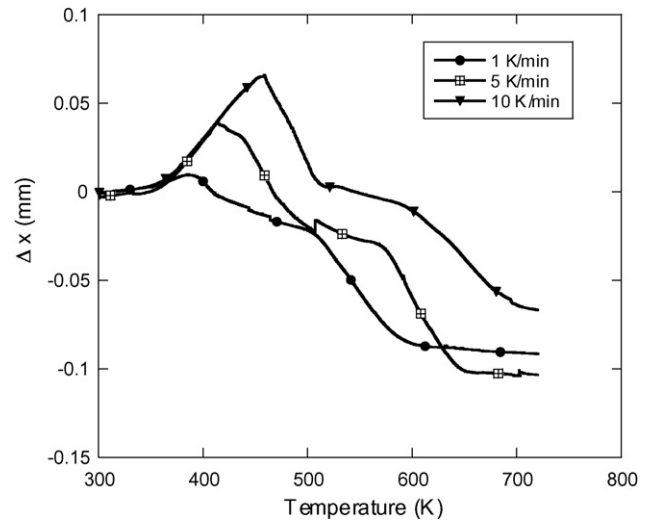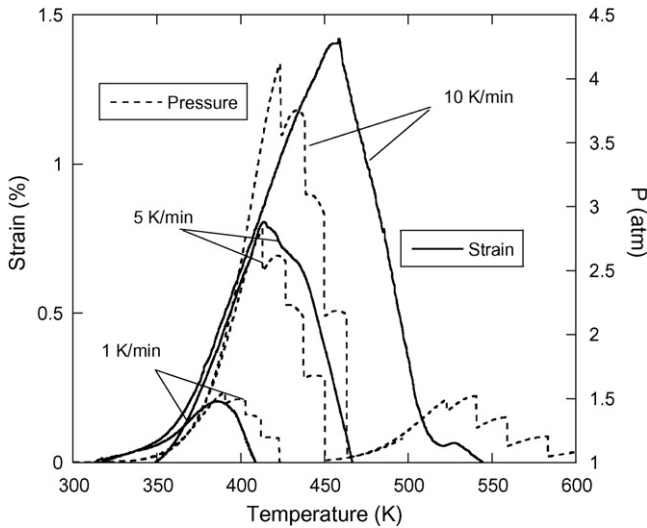Fig. 7. Buildup pressure and sample deformation at the center of the sample (0.5 cm × 0.5 cm × 0.5 cm) estimated by numerical and TMA data, respectively, for different heating conditions.

tation for the fast heating rate condition to maintain the uniform temperature distribution for the sample. This indicates that the pressure buildup estimated by the numerical calculation can be applied to predict the sample deformation measured by the TMA tests. The approach can be further applied on the optimization control of the tape deformation for the thermal processing for the heating rate condition under 10 K min^{-1}.

4.2. Optimal control of the tape deformation

The dynamic optimization problem of the thermal processing is stated as to minimize the required time to remove the organic additives inside the ceramic cubic by adjusting the heating rates to control the maximum buildup pressure formation. The optimization problem can be expressed as

$$\min_{\beta} \quad t_f$$

$$\text{such that} \quad \begin{aligned} \beta &= \frac{\mathrm{d}T}{\mathrm{d}t} \\ g(T, x) &= 0 \\ \frac{P}{P_0} &\leq P_m \end{aligned} \quad (11)$$

where P_m represents the maximum value of the internal pressure constraint.

Fig. 8 shows the optimal trajectories of the heating rates as setting the limitation of the maximum internal pressure (P_m) for the thermal processing. For the case of $P_m = 2.0$, the heating rate starts initially at the maximum value (10 K min^{-1}) until the pressure rises almost to its limitation. The heating rate decreases rapidly from 10 K min^{-1} down to 0 K min^{-1} when the time is at about 9 min to satisfy the pressure constraint as shown in Fig. 8(a). The heating rate rises slowly to keep the pressure under the limitation for the time from 9 to 28 min. In this period, the heating rate was optimally adjusted to satisfy the optimization problem. After the time reaches to 28 min, the heating rate was
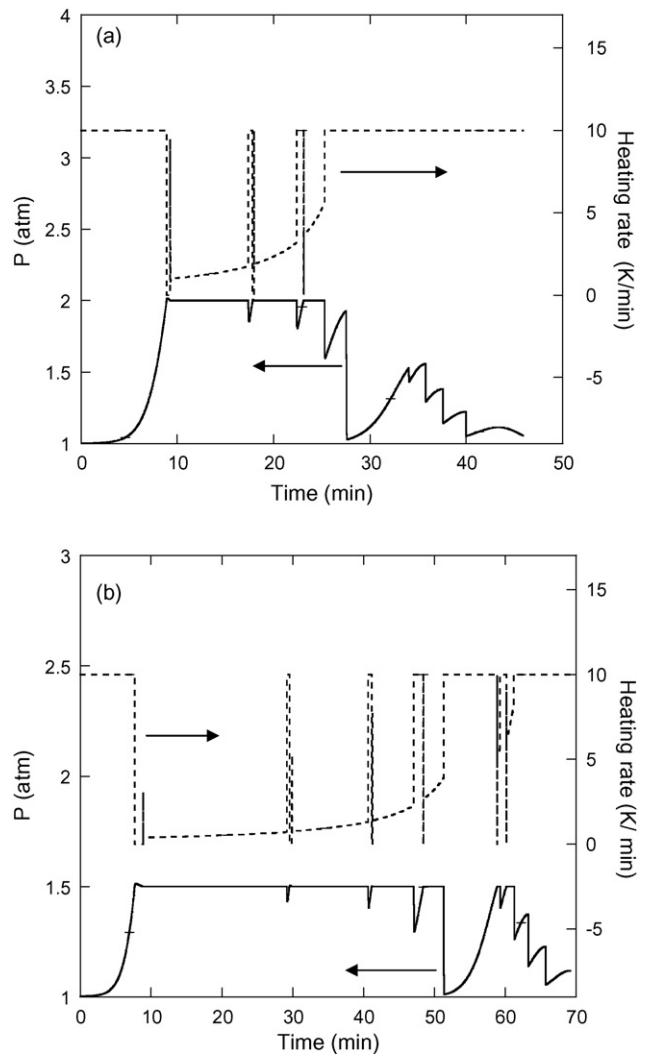

Fig. 8. Optimal trajectories of the heating rate and distribution of the pressure buildup at the cubic center for the maximum pressure constraint set at (a) 2.0 atm; (b) 1.5 atm.

accelerated to the maximum value again and maintained to the end of the process.

If the maximum internal pressure (P_m) was set at 1.5, the optimal trajectories of the heating rates were determined as shown in Fig. 8(b). The heating rate decreased suddenly from 10 to 0 K min^{-1} at 8 min, and maintained at low heating rates for 43 min to satisfy the pressure constraint. After 52 min, the heating rate is controlled at 10 K min^{-1} until the second decomposition period appears at 58 min. The heating rate was reduced again during the short period of time until 62 min. The heating rate rose to 10 K min^{-1} and kept to the end of the organic removal process. It is estimated that the time required to remove the additives is about 20 min longer for the pressure constraint setting at 1.5 than the one at 2.0.

The optimal control scheme of the thermal process as shown in Fig. 8 can be transformed into the temperature distribution for the dynamic optimization problems. Fig. 9 shows the temperature distribution of the optimal heating trajectories for carrying out the TMA experiments. The optimal trajectories of the heat-
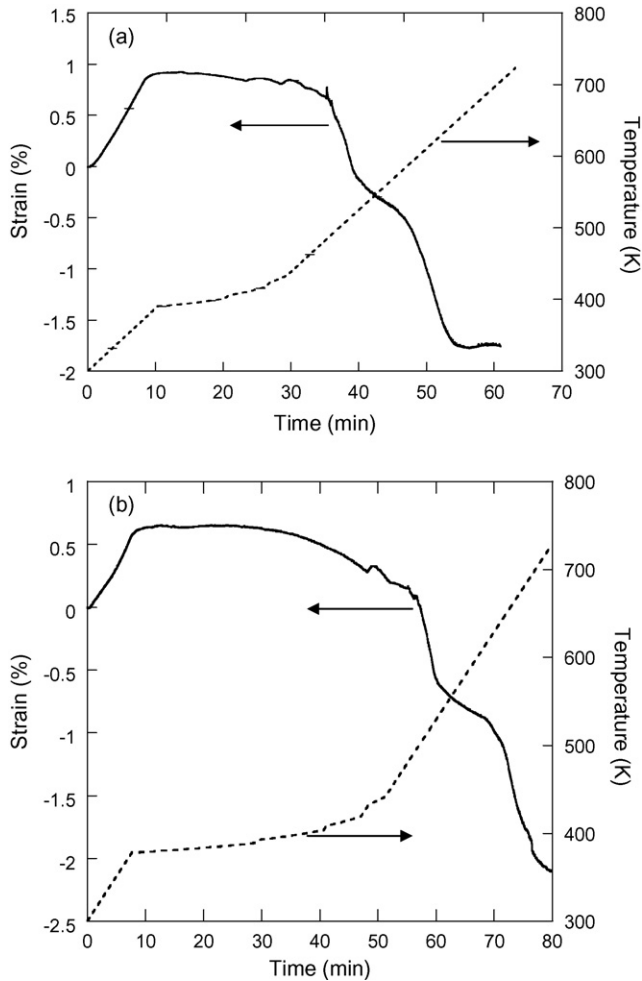
Fig. 9. Optimal trajectories of the temperatures and the tape deformation profiles as functions of time determined under the maximum pressure constraint set at (a) 2.0 atm; (b) 1.5 atm.

ing temperature were influenced by the control of the limitation of the maximum internal pressure generation. For the higher maximum internal pressure setting ($P_m = 2.0$), the strain of the test sample rises to 0.8% and maintain for 25 min based on the optimal heating condition as shown in Fig. 9(a). Fig. 9(b) shows the distribution of the sample strain for the dynamic optimization problem with $P_m = 1.5$. The strain maintain at about 0.5% for a period of time as the optimal temperature was controlled around 400 K for 30 min. This reveals that the deformation of the green tapes can be well-controlled using the off-line optimization control scheme. In addition, longer burnout times are needed for the case with the smaller pressure constraint ($P_m = 1.5$), to satisfy the operating requirement.

5. Discussion

The pressure buildup is formed and attributed to the gas evolving from the thermal decomposition of the organic additives inside the tape. The control of the internal pressure formation is one of the critical points to avoid the defect generation during the thermal processing. Therefore, the search for the optimal heating scheme of the thermal processing is to control the pres-

sure formation. The optimal heating trajectories of the burnout process are influenced by several operating conditions, such as the constraint of the maximum internal pressure formation, and properties of the green tapes. The effects of these conditions on the optimal heating scheme were discussed in our previous study [4].

In practical, the deformation of the tape as shown in Fig. 1 can be measured using analytical instruments, such as TMA. The appearance of the tape can demonstrate the deformation (strain) in shape due to the pressure buildup acting on the ceramic (stress). The stress–strain relationship can be described as

$$\tau = Ys \qquad (12)$$

where τ is the stress, s is the strain, and Y is the modulus of the elasticity. The modulus can be determined if the stress–strain relation is known. In this work, the modulus at the center of the tape is to be determined, if the tape deformation and the internal stress normal to the surface can be estimated. However, the normal stresses inside the tape, including the normal stress generated from the gas flow and the buildup pressure, are difficult to be measured. It was reported that the main contribution of the pressure buildup is primary on the formation of the normal stresses of the tape. Therefore, the normal stresses at the center of the ceramic tape can be evaluated by the internal pressure distribution using numerical methods [8]. Therefore, the modified modulus of the tape at the center can be estimated using the internal pressure and the strain data determined in this study instead of the true value of the normal stress.

Fig. 10 shows the internal pressure associated with the strain of the sample transformed from the data in Fig. 7. The distribution of the modified modulus, the slope of the pressure versus the strain, was determined at different heating rates as shown in Fig. 10. The modulus data distribution indicates that the data was influenced by the heating rates and the strain formation at different heating temperatures. The effect of the heating temperature on the modulus is attributed to the organic/ceramic composition
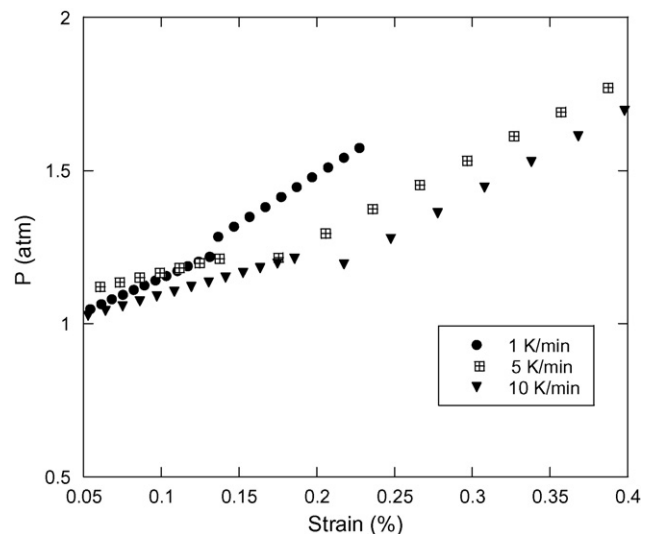

Fig. 10. Relation between the buildup pressure and the strain of the tape samples during the thermal processing.

of the tape at that temperature. The composition of the composite was altered by the heating temperature as shown in Fig. 3, because the organic species can be decomposed and evolved from the sample during the heating process. This implies that the changes of the sample modulus in different stress–strain regions are a dynamic function of the compositions of the green tapes. The strain of the sample is estimated by the value of the modulus which is affected by the tape composition and temperatures and the stress in that operating condition. Alternatively, the optimization case can be changed to control the tape deformation instead of the internal pressure formation. The on-line optimal control approach can be carried out by monitoring the tape deformation to avoid the defect formation during the thermal process for a practical operation.

6. Conclusions

The optimal heating strategies of the organic additives removal with constraints were determined by evaluating the pressure buildup by numerical computation and deformation of the green tapes by TMA measurements. The minimum time required to remove the organic additives was estimated under the constraint of the maximum buildup pressure formation inside the tape during the thermal processing. In addition, the optimal trajectories were influenced by the operating conditions, such as the sample size and the constraint of the maximum pressure

buildup formation. The optimal heating conditions predicted by the numerical simulation were carried out and verified by the tape deformation using TMA experimental tests. The optimization approach can be further applied to the development in the optimization control of the ceramic manufacturing process

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