Selective Laser Thermoregulation System for Accelerated Degradation Test of SiC/SiC CMCs

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SiC/SiC ceramic matrix composites (CMCs) are expected to improve the efficiency of aircraft engines. To confirm the reliability of SiC/SiC CMCs, it is necessary to clarify their physical deterioration in a high-temperature and high-humidity environment that simulates an aircraft engine. For the accelerated degradation test of SiC/SiC CMCs, we developed a heating test system, called the Selective Laser Thermoregulation (SLT) system, that uses a fiber laser and a galvano mirror system. This system can adjust the heat input under a steam environment and when the cooling state changes dynamically. We first performed a numerical simulation to design the SLT system and numerically confirmed that it can heat a SiC ceramic sample to over 1400 °C. We then fabricated the proposed system and confirmed that it can heat a SiC ceramic sample to over 1400 °C and safely maintain this temperature.

Keywords: ceramic matrix composites, thermal-mechanical testing, finite difference analysis, galvano scanner, fiber laser

1. Introduction

Air travel is indispensable for tourism and trade, and thus demand for aircraft is expected to increase. SiC/SiC ceramic matrix composites (CMCs), a new material, are expected to replace Ni-based alloys in aircraft engines \[1\] to improve efficiency.

SiC/SiC CMCs are a high-strength fiber-reinforced material that consists of SiC fiber bundles embedded in a SiC ceramic matrix. They have a lower density and a higher heat resistance than those of Ni-based alloys.

The mechanical properties of these CMCs in a high-temperature environment are not fully known. To confirm the reliability of SiC/SiC CMCs, it is necessary to clarify their physical deterioration in a high-temperature and high-humidity environment that simulates an aircraft engine. Many studies have measured the physical properties of SiC/SiC CMCs in a high-temperature environment \[3\]-\[6\]. In an actual aircraft engine, SiC/SiC CMCs will be heated and cooled repeatedly for a long period of time. Therefore, an accelerated heating test system that simulates repeated heating and cooling is required.

In the traditional heating test, it takes a long time to heat and cool the entire volume of the heating furnace. Therefore, it is difficult to quickly perform an accelerated heating test that repeats rapid heating and cooling to simulate repeated heating and cooling cycles. To overcome this problem, an accelerated heating test that uses a CO\textsubscript{2} laser has been developed \[7\]. Using a laser as the heat source makes it possible to realize rapid heating and cooling by controlling the power of laser irradiation because the laser can heat a sample locally (versus entire volume for a heating furnace). However, with this system, it is difficult to heat a sample inside a high-humidity environment because the CO\textsubscript{2} laser has a wavelength of 10.6 µm, which is strongly absorbed by water vapor.

A heating test system for SiC/SiC CMCs that uses a fiber laser with a wavelength of 1.07 µm has recently been reported by Whitlow et al. \[8\]. In their system, the laser beam is shaped into a rectangle (i.e., the shape of the target heating region). The final shaping optics for the laser is a cylindrical lens. However, with this method, the outer edge of the irradiated surface tends to be cooled and thus the irradiated surface cannot be heated uniformly. In addition, if the cooling conditions change due to changes in the atmospheric gas or air flow around the sample, it is difficult to dynamically adjust the input power distribution.

Here, we propose a heating test system, called the Selective Laser Thermoregulation (SLT) system, that combines a fiber laser with a wavelength of 1.07 µm and a galvano mirror system. This system is expected to be able to heat a sample inside a high-humidity environment because water vapor is almost transparent to a 1.07-µm-wavelength laser. We can control the temperature distribution of the target dynamically using the galvano mirror system, which controls the position of the laser spot rapidly and dynamically. Therefore, even if the cooling condition is changed, the system can adjust the input power distribution and con-
control the temperature distribution of the sample. We introduced the concept design in a previous study [9].

2. Configuration of SLT system

A schematic diagram of the SLT system is shown in Fig. 1. A fiber laser oscillator (FEC4000M, Furukawa Electric Co., Ltd.) is fiber-connected to a galvano mirror system (YD-3000M-5.0, Yaskawa Controls Co., Ltd.). Both devices are cooled by a water chiller (RKE5500B-V-2CH-FU, Orion Machinery Co., Ltd.). The wavelength and maximum power of the laser oscillator are 1.07 µm and 4 kW, respectively. The maximum speed of the laser spot controlled by the galvano mirror system is 15 m/s on the target plane. Considering the safety of the experimental environment, the optical axis of the galvano mirror system is inclined 30° with respect to the horizontal. The sample is held by custom-designed clamp units. The distance from the galvano mirror system to the sample is about 1 m. The whole system is controlled by a laptop computer (“Let’s note” CF-SX4, Panasonic Corporation).

The goal of the SLT system is to heat a SiC ceramic sample (30 mm × 30 mm × 2.0 mm (thickness)) to a temperature of between 1400 °C and 1500 °C and maintain that temperature. In this study, we used a SiC ceramic sample instead of a SiC/SiC CMC because their optical and thermal parameters are expected to be similar and the former is easier to obtain.

The target temperature for the system is above 1400 °C and thus we had to design clamp units for the sample. A three-dimensional (3D) model of the clamp units is shown in Fig. 2. The clamp units are hanged on a single rail, allowing for sample size changes. The clamp units hold the SiC ceramic sample (30 mm × 30 mm × 2.0 mm (thickness)) between two blocks made of refractory cement, covering 5 mm of each side of the sample. Refractory cement with a heat resistance temperature of 1300 °C was used for the contact surface with the sample, which is expected to be at high temperature. The refractory cement part is surrounded by a holder made of SUS303, whose heat resistance temperature is 1000 °C. The SUS303 holders are cooled by an attached water pipe. The cooling water is supplied by a second chiller (LTC-1200a, AS ONE Corp.). If the flow rate of this cooling water falls below approximately 90% of the normal level, a custom-made interlock will turn off the laser.

We performed numerical simulations to design the system, including the clamp units, and performed an experiment to confirm the design. The objective of the numerical simulation was to estimate the laser power required to heat a sample to above 1400 °C and to confirm that the clamp units can operate safely, namely that the temperature of all parts is below the respective heat resistance temperature. The objective of the experiment was to confirm that the proposed system can heat the sample to above 1400 °C.

3. Numerical simulation

3.1 Numerical simulation model

To perform the numerical simulation of the SLT system, we simplified the 3D model shown in Fig. 2 to the numerical simulation model shown in Fig. 3. The following conditions were assumed in the numerical simulation.

- The clamp units hold the ends of the SiC ceramic sample (30 mm × 30 mm × 2.0 mm (thickness)) between two blocks made of refractory cement, covering 5 mm of each side of the sample. Refractory cement with a heat resistance temperature of 1300 °C was used for the contact surface with the sample, which is expected to be at high temperature. The refractory cement part is surrounded by a holder made of SUS303, whose heat resistance temperature is 1000 °C. The SUS303 holders are cooled by an attached water pipe. The cooling water is supplied by a second chiller (LTC-1200a, AS ONE Corp.). If the flow rate of this cooling water falls below approximately 90% of the normal level, a custom-made interlock will turn off the laser.

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- The clamp units hold the ends of the SiC ceramic sample (30 mm × 30 mm × 2.0 mm (thickness)), covering 5 mm on each side.
- The sample and clamp units are completely symmetrical. Therefore, only half of the system needs to be calculated.
- The physical property changes in only the x direction are considered. The variations of several parameters in the y and z directions are averaged.
- The power distribution of the laser spot is averaged in a rectangular area of 4 mm × 14 mm. This means that the scanning speed of the galvano mirror system is infinity.
Considering these assumptions, we solved equation (1), which considers the power balance of the target, as the governing equation.

\[ \rho V C \frac{dT}{dt} = L + C(T) + H(T) + R(T) \]  

(1)

where \( \rho, V, C, T, \) and \( t \) are the density \([\text{kg/m}^3]\), volume \([\text{m}^3]\), specific heat \([\text{J/(kg K)}]\), temperature \([\text{K}]\), and time \([\text{s}]\) respectively. \( L, \ C(T), \ H(T), \) and \( R(T) \) are functions of the laser input power, thermal conduction, heat transfer to the atmosphere, and radiation, respectively. They all have the unit \([\text{W}]\), which matches the left-hand side of the equation, and are respectively expressed as follows.

\[ L = (1 - R)P \]  

(2)

\[ C(T) = VV(\lambda T) \]  

(3)

\[ H(T) = hA(T - T_0) \]  

(4)

\[ R(T) = \varepsilon\sigma\epsilon\theta^4 - T_0^4 \]  

(5)

where \( R, P, \lambda, h, A, T_0, \varepsilon, \) and \( \sigma \) are the reflectivity of the laser at the target surface, laser power \([\text{W}]\), thermal conductivity \([\text{W/(m K)}]\), heat transfer coefficient, cross-section area \([\text{m}^2]\), background temperature, emissivity, and the Stefan-Boltzmann constant, respectively. The physical parameters of the system components are shown in Table 1.

A symmetrical boundary is applied to the symmetrical axis. The cooling water temperature is fixed at 20 °C. The initial temperature of all parts is 27 °C. The laser power is assumed to be 200 W.

In this paper, other effects such as thermal expansion and chemical reactions are ignored. The size and physical parameters of each material are constant in the simulation.

We developed the solver using Python as the main programming language. The finite difference method of the implicit solver was used to solve the governing equation.

### Table 1 Physical parameters of system components.

|          | SiC  | Refractory cement | SUS 303 |
|----------|------|------------------|---------|
| Thickness [mm] | 2.0  | 17.9             | 17.9    |
| Length [mm]   | 20   | 33               | 10      |
| Width [mm]    | 30   | 39               | 57      |
| Specific heat [J/(kg K)] | 1250 | 1000            | 5900    |
| Thermal conductivity [W/(m K)] | 35   | 1.0             | 16.7    |
| Emission rate | 1.0  | 1.0              | 1.0     |
| Heat transfer coefficient [W/(m² K)] | 8.0  | 8.0             | 8.0     |
| Absorption rate | 0.0  |                  |         |

#### 3.2 Calculation results

The calculation results of the numerical simulation are shown in Fig. 4. The temperature distributions obtained at 10-second intervals are shown. The vertical axis is the temperature and the horizontal axis is the distance from the center of the irradiation surface on the target.

These results show that the system can heat the target to over 1400 °C. The maximum temperature is reached 30 seconds after laser irradiation and maintained, and all parts reach a steady-state temperature within 60 seconds.

### 4. Laser heating experiment using SLT system

#### 4.1 Experimental setup

We fabricated and tested the SLT system, including the clamp units, described in section 2.

The laser power was 400 W. Pulse width modulation with a duty cycle of 0.5 was applied, and thus the average laser power was 200 W. We controlled the focal spot size considering that the optical axis of the galvano mirror system was tilted 30°. The laser spot repeatedly moved up and down at a speed of 20 mm/s over a distance of 10 mm considering several safety factors. The diameter of the laser spot was about 4 mm.

The temperature of the target was measured using a radiation thermometer (PI08M, Optris Infrared Sensing, LLC). The emission rate of the target was assumed to be 1.0. The temperature of the cooling water flowing into the pipe of the clamps was kept at about 20 °C.

#### 4.2 Experimental results

The experimental and calculation results of the change in maximum temperature over time are shown in Fig. 5.

Due to thermometer limitations, there are no experimental results below 575 °C. After 40 seconds of laser irradiation, the SiC ceramic sample was heated to above 1400 °C; this temperature was maintained for tens of seconds.
5. Discussion
The goal of this study, namely to heat a SiC ceramic sample to over 1400 °C, was successfully achieved. The calculation results shown in Fig. 4 indicate that even in the steady state, the temperature of each material of the sample and clamps was below its respective heat resistance temperature. The experimental results confirm that the clamps can be used safely.

The maximum temperatures obtained in the simulation and experiment slightly deviate, as shown in Fig. 5. The average temperature in the last 20 seconds was 1521.0 °C in the simulation and 1479.7 °C in the experiment. This difference in maximum temperature (41.3 °C) can be considered to be reasonable.

However, there was an obvious difference in the rate of temperature increase between the experiment and the simulation. There are two possible reasons for this difference, namely the scanning speed of the laser spot and the temperature dependence of specific heat.

As described in section 3.1, we assumed that the scanning speed of the galvano mirror system is infinity in the numerical simulation model. This simplified modeling might be the reason for the difference between the experiment and numerical simulation.

The temperature dependence of specific heat was not taken into consideration in the numerical simulation. The specific heat of SiC ceramics increases with temperature [10]. Therefore, if the temperature of the target increases, the rate of temperature increase decreases. It is thus necessary to consider the temperature dependency of the specific heat of a SiC ceramic sample to obtain more accurate calculation results.

Furthermore, a small area of oxidization was observed in the experiments. Therefore, the difference between the experimental and calculation results may have resulted from the simulation model not taking into account the effects of oxidation.

In the future, targets of various shapes will be heated because the required laser irradiation conditions, such as scan speed, vary according to target shape.

6. Conclusion
We designed the SLT system and confirmed that it can heat a SiC ceramic sample to above 1400 °C.

A numerical simulation indicated that a SiC ceramic sample (30 mm × 30 mm × 2.0 mm (thickness)) can be heated to above 1400 °C by irradiation from a 200-W laser in a heating area of 14 mm × 4 mm. Furthermore, it was calculated that the temperature at each point of the clamps was below the respective heat resistance temperature when the sample was heated to above 1400 °C and maintained at that temperature.

Experiments confirmed that the SLT system can heat a SiC ceramic sample to over 1400 °C safely when the laser is applied to the center of the SiC ceramic sample. The laser spot repeatedly moves up and down at a speed of 20 mm/s over a distance of 10 mm.

The results indicate that the SLT system can safely heat SiC/SiC CMCs to above 1400 °C.

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References
[1] G.S. Corman and K.L. Luthra: “Handbook of Ceramic Composites” ed. by N. P. Bansal, (Publisher, Boston, 2005) p.99
[2] M. Roode, J. Price, J. Kimmel, N. Miriyala, D. Leroux, A. Fahme and K. Smith: J. Eng. Gas Turbines Power, 129, (2007) 21.
[3] R. J. Young, A. B. L. Broadbridge and C.-L. So: J. Microscopy, 196, (1999) 257.
[4] Y. L. Dong, H. Kakisawa, and Y. Kagawa: Meas. Sci. Technol., 25, (2014) 025002.
[5] A. Haboub, H. A. Baie, J. R. Nasiatka, B. N. Cox, D.B. Marshall, R. O. Ritchie and A. A. MacDowell: Rev. Sci. Instrum., 85, (2014) 083702.
[6] T. Whitlow, E. Jones, C. Przybyla: Composite Structures, 158, (2016) 245.
[7] M.P. Appleby, D. Zhu and G. Morscher: Surf Coat Technol, 284, (2015) 318.
[8] T. Whitlowa, J. Pitzb, J. Piercea, S. Hawkinsc, A. Samuele, K. Kollinsd, G. Jeffersond, E. Jonesd, J. Vernonda, C. Przybylad: Composite Structures, 210, (2019) 179.
[9] H. Koshiji, K. Azato, T. Ohkubo, E. Matsunaga, T. Dobashi, N. Shichijo, M. Sato, Y. Kagawa and K Go-to: IEEJ Technical Meeting on Innovative Industrial System (in Japanese), (2020) IIS-20-007.
[10] M.W. Chase, Jr.: NIST – JANAF Thermochemical Tables, 4th Ed., J. Phys. Chem. Ref. Data, 9, (1998)