INTRODUCTION

Optical imaging for visualization of strain distribution in two and three dimensions using digital image correlation (DIC) techniques has been widely used to investigate various material systems.\(^1\),\(^2\) This is noncontact procedure, which does not require complicated facilities, and the sample preparation process is easy compared with conventional procedures. DIC is widely used for understanding cumulative failure of composite materials.\(^3\)–\(^6\) However, its application is limited at room temperature. Quantification of strain (stress) distribution in local areas at high temperature is difficult, and direct observation in such conditions is also limited due to the thermal radiation from the specimen surface at temperatures above \(1000^\circ\text{C}\). Various systems using blue LED light sources and acoustic waves were reported as reliable measurement systems at high temperatures above \(1000^\circ\text{C}\).\(^7\)–\(^9\)

Recently, the group of Kakisawa proposed a new optical system using an ultraviolet light (UV) source for surface observation at high temperature.\(^10\)–\(^12\) They demonstrated that this optical system is suitable for the measurement of surface strain distribution at high temperature in combination with DIC because this system enables to maintain a grayscale distribution of the captured images at elevated temperature up to \(1200^\circ\text{C}\).\(^10\)–\(^12\) In the field of ceramic matrix composites (CMC), visualization of deformation mapping is rather important to understand the mechanical behavior of structural materials in severe working environments (e.g., in gas turbine engines, nuclear reactors, and re-entry vehicles).

Recent studies also showed that DIC is an effective tool to understand heterogeneous deformation of fiber-reinforced CMCs and ceramic coatings at high temperature in various scales \(^9\); however, application of DIC at high temperature is limited by heat resistance of the speckle pattern. Various

Optical imaging of surface strain distribution up to 1500°C: Development of micro-speckle pattern

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Abstract
Micro-speckle patterns were fabricated using \(\alpha\)-\(\text{Al}_2\text{O}_3\) and SiC micro-particles for in-plane strain distribution measurement at temperatures up to 1500°C using an ultraviolet high-temperature observation system (UV-HTOS) combined with a digital image correlation (DIC) technique. The experimental results showed that the measured mean surface strains of \(\alpha\)-\(\text{Al}_2\text{O}_3\) were in good agreement with the calculated values obtained from thermal expansion coefficient and temperature difference. The proposed speckle pattern could be used for strain distribution measurement without degradation even after heating up to 1500°C. The results also demonstrated that the pattern fabricated in this study is effective to understand the heterogeneous deformation mechanism in composite materials applied in high-temperature structural components.

KEYWORDS
digital image correlation, in situ observation, speckle pattern, strain distribution, ultraviolet
materials were investigated as spraying particles, for example, Ti (tested at 650–800°C),\textsuperscript{13} Au (tested at ~700°C),\textsuperscript{14} amorphous silica and TiO\textsubscript{2},\textsuperscript{8} and CoO (tested at 1100°C)\textsuperscript{15,16}; however, usability of these materials is limited below 1200°C.\textsuperscript{17} Tungsten coatings deposited by atmospheric plasma spraying (APS) were also used to improve the thermal stability of speckle patterns.\textsuperscript{18,19} This material is easy to decompose in contact with air. In addition, speckle patterns were also made using abrasion\textsuperscript{9,10} and sandblasting\textsuperscript{16}; however, these methods are not suitable for brittle materials because the defects created act as stress concentration sources under loading. Among the speckle patterns developed in the literature, commercially available Al\textsubscript{2}O\textsubscript{3} and ZrO\textsubscript{2} paints are good examples due to their thermal stability above 1200°C.\textsuperscript{9}

In Ref. [10], the observation and strain measurement at 1400°C are conducted by a pattern formed by scratching surface (denoted as scratch pattern). Although scratch pattern is useful for high-temperature strain measurement because patterns are stable, the damages on the surface cause stress concentration.

The objective of this study is to develop speckle patterns for strain distribution measurement with optical imaging using UV light and CCD. The optimum ceramic particles for the speckle patterns were determined based on the interaction between quality of photograph and materials. Then, thermal stability of the developed pattern at high temperature was also evaluated. Strain measurement was done using a calibration material (polycrystalline Al\textsubscript{2}O\textsubscript{3}) to understand of the effect of speckle patterns on the accuracy of strain measurement. The effectiveness of the developed pattern was also discussed.

### 2 | EXPERIMENTAL PROCEDURE

#### 2.1 | Fabrication of micro-speckle patterns

As raw materials, α-Al\textsubscript{2}O\textsubscript{3} (particle diameter: from 40 to 100 nm; Nanostructured & Amorphous Materials Inc., USA) and SiC (particle diameter: ~600 nm; Showa denko, Japan) were prepared. These particles were dispersed into ethanol. The composition ratios of the solutions are summarized in Table 1. The ratio of α-Al\textsubscript{2}O\textsubscript{3} was set to 2 wt%, and that of SiC was varied from 0.25, 0.5, and 1.0 wt% (denoted as pattern A, B, and C), respectively. The solutions were mixed using a planetary centrifugal mixer (AR-100, THINKY Corp., Tokyo, Japan) for 5 minutes.

For strain measurement at high temperature, a polycrystalline α-Al\textsubscript{2}O\textsubscript{3} plate (30 × 30 × 0.5 mm) with average grain size of ~1.0 μm was used because its mechanical and thermal properties at room and high temperatures were known. Specimens (~2.5 × 2.5 × 0.5 mm) were produced from the Al\textsubscript{2}O\textsubscript{3} plate using a diamond impregnated blade. After ultrasound cleaning of the specimens, the mixtures were deposited onto the specimen using an airbrush method reported in the literature.\textsuperscript{10,11} This procedure was selected because this is easy to use and can fabricate speckles on complex-shaped samples. In this study, the distance from the specimen surface and the tip of nozzle (diameter of 0.18 mm) was affixed at 200 mm.

In the present study, we selected a criterion for assessment of fabricated speckle pattern: Mean intensity gradient (hereafter denoted as MIG). MIG ($\delta_j$) is applied for evaluation of the area of interest (AOI) and is given by,

$$\delta_j = \frac{\sum_{i=1}^{W} \sum_{j=1}^{H} |\nabla f(x_{ij})|}{(W \times H)}$$

where W and H are the image width and the image height, respectively. \(\nabla f(x_{ij})\) is the modulus of local intensity gradient vector with $f_x(x_{ij})$ and $f_y(x_{ij})$ are the x- and y-directional intensity derivatives at pixel $(x_{ij})$.

### 2.2 | Evaluation of the performance of speckle patterns

Evaluation of the performance of the speckle pattern was conducted using UV-HTOS. The experimental set up has been already reported in the literature.\textsuperscript{10–12} The authors reported effectiveness of UV-HTOS for in situ observation of degradation of polycrystalline ceramics, composites, and thick ceramic coatings.\textsuperscript{12,20} The system was composed of a UV mercury lamp (USH-500SC2, Ushio Inc) with a pressure range of 0.5-5 MPa and a UV charge-coupled device (CCD) camera. The UV light (wavelength of 365 nm), generated by a mercury lamp, was introduced into the objective lens (NZ-1000, Keyence) through an optical fiber (see also Figure 1). This objective lens was chosen because it has long working distance (~50 mm) and can prevent heating of lens during the test. A light-pass filter (U340-25, Hoya Candeo Optronics Corp) was inserted between the water-cooled CCD camera and objective lens to cut off the light generated by thermal radiation from the specimen surface up to temperatures of 1000°C. Previous studies have shown that the effect of radiation on the image acquired during heating was successfully minimized up to 1400°C with the inserted optical filter.\textsuperscript{10–12,20} The range of field of view (FOV) for the HTOS is approximately 3.7 × 3.7 mm-390 × 390 μm,
which is corresponded to 100x field of view and 1000x field of view, respectively.

The specimen was put into a Pt-holder (~5 mm in diameter and ~2 mm in depth) and heated up to 1500°C and kept at this temperature for 5 minutes using an electric furnace (HT-1500, LINKAM). The rate of temperature increase and decrease was set to be 100°C/min. Surface images of the specimen were captured at intervals of 1s during the heating and cooling stages. The captured images have a resolution of 1004 × 1002 pixel.

2.3 Strain distribution measurement of the standard specimen

Surface strain distribution was measured using a commercially available DIC software (VIC-2D, Digital Solution Inc., USA). AOI was set to 800 × 800 μm, which corresponds to 600 × 600 pixel. The resultant resolution was ~1.3 μm/pixel. The size of the subset and analytical step was set as 75 × 75 pixel and 2 steps, respectively. The strain distributions along the x- and y-directions were analyzed. The strain measured using DIC was compared to that calculated for polycrystalline α-Al2O3 (see also section 3.2).

3 RESULTS AND DISCUSSION

3.1 Evaluation of as-fabricated micro-speckle patterns

Figure 2 shows typical optical micrographs of as-fabricated speckle patterns using a mixture of α-Al2O3 and SiC particles. The images show good contrast, which originates from the difference of reflectance between particles against UV light. For UV light with wavelength of 365 nm at a temperature of 25°C, the reflectance of α-Al2O3 and SiC is ~8% and ~20%, respectively. 21-23 In addition, the absorption coefficients of α-Al2O3 and SiC are ~10−2 cm−1 and ~500 cm−1, respectively. 21-24 Therefore, SiC particles and Al2O3 appear
black and white in the UV images, respectively, because the difference in absorption is much higher than that in reflectance.

The grayscale histogram of each pattern in AOI is also shown in Figure 3. Most intensities (>99%) within AOI are distributed in a similar range (from 25 to 236). In pattern A, two peaks around 100 and 200 (hereafter denoted as P1 and P2) are clearly identified. For all patterns, the intensity of P1 is higher than that of P2. As expected, the intensity of P1 increases and that of P2 decreases with the increase of SiC content. It seems that each speckle has isolated each other in patterns A and B (0.25 and 0.5 wt% SiC); however, the aggregation leads to connection of black speckles for pattern C (1 wt% SiC). Especially, P2 in pattern C has almost disappeared, which implies that the contrast obtained in pattern C is much lower than that for pattern A and B. Table 2 shows the calculated value of MIG of each image at room temperature (25°C). For reliable measurements, high MIG is preferred because mean bias error becomes small. At room temperature, the value of MIG is 20-30 and it is sufficient to act as patterns for DIC because displacement errors for some patterns with different MIG have been evaluated by Pan et al., and displacement errors for MIG 20-35 are ~0.01 pixel.19

3.2 Measurement of strain distribution up to 1500°C

In the present study, we define horizontal and vertical axis as x- and y-axis, respectively. Counter maps of strain distribution along x- and y-direction during heating and cooling are shown in Figures 4 and 5, respectively. Nonuniform strain distribution is observed in both x- and y-directions. These results are natural because thermomechanical properties of α-Al₂O₃ depend on grain orientation and temperature.

The coefficient of thermal expansion (CTE) for a-axis and c-axis of α-Al₂O₃ at room temperature (25°C) is 7.6 × 10⁻⁶ K⁻¹ and 8.3 × 10⁻⁶ K⁻¹, respectively.25 In addition, the CTE for a-axis and c-axis at 1,500°C is 8.6 × 10⁻⁶ K⁻¹ and 9.5 × 10⁻⁶ K⁻¹, respectively. Here, thermal strain of the specimen, εₜ, is defined as.

$$\varepsilon_\tau = \alpha(T)\Delta T$$  (2)

| TABLE 2 | MIG of as-fabricated speckle patterns |
|---------|--------------------------------------|
|         | Pattern A | Pattern B | Pattern C |
| MIG     | 24.25     | 29.88     | 21.54     |

FIGURE 3 Grayscale distribution of as-fabricated patterns: A, 0.25wt% SiC particle B, 0.5wt% SiC particle, and C, 1.0wt% SiC particle

where ΔT and α(T) represent temperature difference before and after heat exposure and CTE depending on temperature, respectively. α(T) of α-Al₂O₃ along a- and c-axis
(defined as $\alpha_a$ and $\alpha_c$) is also varied at high temperature as follows:

\[
a_a(T) = 7.419 \times 10^{-6} + 6.43 \times 10^{-10} T - 3.211 \times 10^{-6} \exp(-2.59 \times 10^{-3} T)
\]

\[
a_c(T) = 8.026 \times 10^{-6} + 8.17 \times 10^{-10} T - 3.279 \times 10^{-6} \exp(-2.91 \times 10^{-3} T)
\]

Thus, strain for a-axis ($\varepsilon_a$), c-axis ($\varepsilon_c$), and mean strain ($\varepsilon_m$) for $\alpha$-Al$_2$O$_3$ is calculated by:

\[
\varepsilon_a = \alpha_a \Delta T \quad (5)
\]

\[
\varepsilon_c = \alpha_c \Delta T \quad (6)
\]

\[
\varepsilon_m = \alpha_m \Delta T = \frac{2\alpha_a + \alpha_c}{3} \Delta T \quad (7)
\]

where $\alpha_m$ represents CTE of polycrystalline $\alpha$-Al$_2$O$_3$. Plots of average thermal strains (hereafter denoted as, $\varepsilon_{xx}$, and $\varepsilon_{yy}$, respectively) for pattern A, B, and C with standard deviation measured by DIC during heating and cooling as a function of temperature are shown in Figure 6A-C, respectively. The calculated thermal strains of $\alpha$-Al$_2$O$_3$ for a-axis, c-axis, and mean value based on Equations 3-7 are also exhibited. The results clearly show that the average strain measured using pattern A is in good agreement with the thermal strain of $\alpha$-Al$_2$O$_3$ calculated by Equations 3-7. The result for pattern B is also similar to the thermal strain of $\alpha$-Al$_2$O$_3$, whereas it is a little smaller compared with pattern A in the temperature range of 600–1000°C. The average strain measured using pattern C is also relatively close to the thermal strain of $\alpha$-Al$_2$O$_3$. However, it above 1200°C during cooling is much smaller than that for the thermal strain of $\alpha$-Al$_2$O$_3$. These results clearly indicate that pattern A is the most effective for the measurement of strain up to 1500°C. This is probably attributed to the distribution of grayscale values and the stability of patterns. The relationship between strain measurement and stability of patterns is discussed in the next section.
3.3 | Stability of speckle patterns

Figure 7 shows the captured images during heating up to 1500°C. Using visible light source, the contrast saturates by thermal radiation from the sample surface because the wavelength of emitted light shortens. In the present case, grayscale remains relatively steady even after heating as discussed in previous reports.\textsuperscript{10–12,20} Speckle patterns made of metal particles, and some oxides proposed in the literature, were decomposed or oxidized and the measurable temperature range was limited; however, the speckle patterns proposed in this study do not change even above 1300°C. Although high-intensity pixels increase up to 1400°C, patterns can be recognized even at 1500°C. Figure 8 shows the captured images during cooling from 1500°C to room temperature (~25°C). The appearance of the patterns is not changed during heating. However, it seems that the contrast of the images for each temperature decreases compared with that during heating. The relationship between MIG in AOI and temperature is presented in Figure 9A and B. MIG for each pattern during heating tends to slightly decrease with the increase of temperature. At least, ~60% of MIG measured at room temperature is maintained even at 1500°C. The displacement errors increase in accordance with the decrease of MIG. However, the increase of displacement error caused by the decrease of MIG is probably negligible because it increased by only 0.01 pixel when MIG decreases from 35 to 12.\textsuperscript{19} Focusing on MIG during cooling, it increases with the decrease of temperature. The distribution of MIG for pattern C is larger than that for pattern A and B.

Figure 10A-F show the changes in the grayscale histogram before, during heating to the maximum temperature, and during cooling for pattern A, B, and C.

For pattern A (see Figure 10A and B), the grayscale values before heating maintain up to 1000°C. The difference between P1 and P2 decreases with the increase of temperature above 1000°C by increasing of grayscale value of P1 and decreasing that of for P2. The result indicates that the contrast obtained decreases with the increase of temperature. Notably, the intensity of P2 rises above that of P1 for temperatures

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure5}
\caption{Contour maps for stain measurement up to 1,500°C during cooling. 0.25wt% SiC particle, 0.5wt% SiC particle, and 1.0wt% SiC particle are summarized.}
\end{figure}
FIGURE 6 Measured strains by DIC as a function of temperature: A, 0.25wt% SiC particle B, 0.5wt% SiC particle, and C, 1.0wt% SiC particle

FIGURE 7  Captured optical micrographs of surface speckle pattern at room temperature, and during heating to 900-1500°C
The stability of pattern B (see Figure 10C and D) is lower than pattern A because the intensity of P1 increases and decreases repeatedly. Although the grayscale value for P1 and P2 increases with the increase of temperature, the difference between P1 and P2 also decreases with the increase of the temperature. This result suggests that the decrease of contrast occurs during heating. After 5 min at 1500°C, the difference between grayscale value of P1 and P2 considerably decreases, and the difference between P1 and P2 increases with the decrease of temperature during cooling as well as pattern A. Contrarily, the intensity of P1 is higher than that of P2 before and after heating. For pattern C (see Figure 10E and F), the distribution of grayscale value is almost the same up to 1000°C and it narrows at above 1000°C. After heating at 1500°C for 5 minutes and during cooling state, the intensity of P1 decreases drastically, which implies that the contrast during the cooling state also decreases rapidly. In addition, the distribution of grayscale value is quite large compared to other patterns and this phenomenon causes the large distribution of MIG for pattern C.

These results clearly indicate that the contrast of pattern A, B, and C decreases at above 1000°C, which is attributed to the oxidation of SiC particles. A weight gain of 2%-8% is observed for SiC particles with diameter of 0.95-3 μm heated between 950 and 1500°C for 5 minutes owing to the formation of SiO₂ on their surface. Therefore, it is considered that the SiC
particles agglomerate owing to the formation of SiO$_2$, and the aggregation becomes more prominent as the SiC content increases. On the other hand, the degradation of the patterns in terms of optical properties is negligible because the absorption index and transmittance of SiO$_2$ for UV light with the wavelength of 365 nm are ~10$^{-7}$ and over 90%, respectively $^{27,28}$. Thus, strain measurement using UV-HTOS at high temperatures up to 1500°C is realized by high-temperature DIC using patterns composed of sub-micro-SiC and nano-α-Al$_2$O$_3$ particles proposed in the present study. It is desirable that the distribution for grayscale of patterns has two different peaks (black and white) up to 1500°C to obtain the contrast required for the DIC measurement. In other words, the availability of patterns is measurable by evaluation of the distribution of grayscale values. In the present study, pattern A (0.25 wt% SiC) seems an optimal pattern for the measurement. Therefore, using UV-HTOS and the micro-speckle patterns developed in the present study, the application of high-temperature DIC to advanced high-temperature structural materials such as SiC fiber-reinforced SiC matrix composites (SiC/SiC) and ultra-high-temperature ceramic (UHTC) composites, which are used as structural materials at 1200°C or above can be realized.

4 | CONCLUSIONS

In the present study, micro-speckle patterns were fabricated using nano-α-Al$_2$O$_3$ and SiC sub-micro-particles, and the process parameters for fabrication of the patterns at high temperature were optimized. The micro-speckle patterns were evaluated both at room and high temperatures, their performance for DIC and UV-HTOS was assessed, and the following conclusions were obtained.

The ratio of powder mixtures is the most important parameter in the fabrication of speckle patterns for strain distribution measurement by DIC. Especially, the distribution of grayscale values for patterns strongly depends on the SiC content. In the present study, 0.25 wt% SiC-2 wt% α-Al$_2$O$_3$ particles (pattern A) were optimal because peaks for black and white could be isolated during strain measurement up to 1500°C.

A previous study shows (ref. 10) scratch pattern is effective to measure strains up to 1400°C. However, stress concentration is evolved by scratching on the Al$_2$O$_3$ surface. In the present study, strains of an α-Al$_2$O$_3$ polycrystalline plate successfully measured by proposed speckle patterns and UV-HTOS. There was a good agreement with the calculated values using CTE and temperature difference. This clearly indicates that strain distribution of advanced high-temperature structural materials such as SiC/SiC and UHTC composites can be realized up to 1500°C using the pattern developed in the present study.

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