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Magneto-Mechanically Reconfigurable Metasurface

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14. ABSTRACT
Highly senstive polarizer has been fabricated using the thermal nanoimprinting lithography. Elemental sulfur generated as a by-product of petroleum-refining processes exhibits high intrinsic transmittance at the IR region as well as cost-effectiveness. Using polymeric sulfur synthesized through inverse vulcanization of elemental sulfur, the Au layer was deposited using an E-beam evaporation system under vacuum onto the poly 1D nano-gratings. The poly micropillar arrays are advantageous for actuation due to their low modulus resulting from a low crosslinking density as well as high intrinsic transmittance at the IR region with high sulfur contents. This programmability of magnetic self-assembly will provide on-demand contactless reconfiguration of surface morphology for various systems. The project has been going well and PO expects that further research should be carried out towards the next fiscal year.

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Technical Report

Accomplishments

Research Objectives: Please list the main research objectives of this project

High-speed aerodynamic phenomena have not been fully understood due to the limitation in high-fidelity experimental data that leads to numerical and analytical modeling. Pressure- and temperature-sensitive paints (P/TSPs) are proposed to acquire experimental data in spatiotemporal manner. The objective of the development of P/TSPs is to exceed the temperature to a "high" temperature around 1500 K. If successful, high-fidelity spatiotemporal measurement in pressure and temperature can be achieved that will be used to understand high-speed aerodynamics in high-temperature environment. P/TSP developments in static conditions are focused on in the first year.

Accomplishments

1) major activities
The present project is a collaboration between the University of Notre Dame (UND) and Aichi Institute of Technology (AIT), Japan. The major activities in this project term are to narrow down the candidates of the binding components of the P/TSP.
2) specific objectives
To narrow down the binding materials, temperature calibration with various binding materials was performed.
3) significant results or key outcomes, including major findings, developments, or conclusions (both positive and negative)
Silica-based white paint (Ceracoat 22, Audec Co. Ltd) could give a high temperature tolerance. It can be sprayed on a model surface. With this binding material, nine different phosphors as a temperature-sensitive probe, were used for temperature calibration. It was found that, depending on the phosphors used, the intensity output from the developed TSP varied and six of them could show a response to temperature up to 800 °C (1073 K). See "**Technical Updates**" for details.
4) other achievements. Include a discussion of stated goals not met.
One of the TSPs was applied to the hypersonic wind tunnel at Air Force Research Laboratory, Dayton OH. This test was to understand the current status of the improvements of the P/TSP. Even though it could show spatiotemporal temperature over a wind-tunnel model, the durability was not satisfactory enough. See "**Technical Updates**" for details.

Dissemination

The current outcome of the sensor development was presented at the 2023 AIAA SciTech:

Egami, Y., Matsumura, Y., Shibata, A., Sakaue, H., "Investigation of Thermographic Phosphors Properties for High-Speed Aerodynamics," the 2023 AIAA SciTech Forum, AIAA Science and Technology Forum and Exposition, AIAA paper 2258, https://doi.org/10.2514/6.2023-2258, 2023.

One AIAA paper was accepted at the 2024 AIAA SciTech Forum and one invited presentation will be given at this forum.

Plan Moving Forward

The PI and Co-PI could extend the temperature tolerance up to 800 °C (1073 K). It was found that the radiative heat from the sensor surface will overlap with the sensor signal. To deal with the radiative heat, two-color sensor will be studied to cancel out the radiative heat. To demonstrate the improvements of our project, the second AFRL test will be conducted in the next project term.

Impacts

Other disciplines:
The PI could make a connection to the hypersonic-research professionals at the Air Force Research Laboratory (AFRL Dayton OH). This created a new connection to use the AFRL hypersonic facility for confirmation and demonstration of the developed sensors. In this project year, confirmation tests were performed to identify technical challenges that is documented in the "**Technical Updates**."

Describe the impact in this reporting period on the development of human resources
This project is an international collaboration between the University of Notre Dame (UND) and Aichi Institute of Technology (AIT), Japan. Graduate students in each institution are the main drivers to perform the present project. To accelerate the performance of the project, the FY23 AFOSR International Supplemental Student Exchange Program was awarded. Through this supplemental program, a US student at UND stayed at the AIT laboratory for one week. This student could also visit two other research laboratories in Japan to see and discuss related research with professionals at these institutions. A Japanese student at AIT stayed for one week at UND and also additional one week at AFRL facility to join the wind tunnel test described in 3) significant results. Through this program, graduate students in each institution become familiar with the fabrication and characterization systems at each institution. Even though students originally had a language barrier, they could improve their communication skills by meeting in person and spending time together.

Changes

The Arc Heated Tunnel at UND is under re-construction of the arc-heater. Instead, the PI could build a research connection with AFRL personnel to apply the developed sensors to the AFRL hypersonic tunnel for the confirmation demonstration. The PI and Co-PI have applied one of their developed sensors for confirmation test this year. By the end of the project year, the final product will be applied to the AFRL tunnel for demonstration. This will provide a better impact on the progress of the project to Air Force facilities.

Technical Updates

To extend the capability of luminescent sensing technology in extreme flow conditions, an investigation of thermographic phosphors applicable from 100 °C (373 K) to 800 °C (1073 K) was carried out. Under the same binding material of silica-based white paint (Ceracoat 22), nine different thermographic phosphors were investigated as a high-temperature sensor (Table 1). Each phosphor was mixed with 40 wt% of the binder paint. Distilled water was also added to the paint at 10 wt% to achieve a viscosity suitable for spraying. The prepared paint solutions were stirred well with a magnetic stirrer for one overnight. Then, the paint solutions were sprayed onto an aluminum oxide ceramic plate using a spray gun (Minijet 3000 B HVLP, SATA GmbH). The prepared thermographic phosphor samples were cured in a vacuum chamber for one overnight. The thickness of the applied thermographic phosphor paint was about 30 μm.

Table 1 Thermographic phosphors used in this study

	Phosphor	Excitation [nm]	Emission [nm]	Vendor
(a)	$(SrCa)_5(PO_4)_3Cl:Eu$	280	446	Nemoto
(b)	$BaMgAl_{10}O_{17}:Eu$ (BAM)	330	460	Nemoto
(c)	$BaMg_2Al_{16}O_{27}:Eu,Mn$ (BAM-G)	330	505	Nemoto
(d)	$CaSc_2O_4:Ce$	430	516	Mitsubishi Chemical
(e)	SiAlON:Eu	400	544	Mitsubishi Chemical
(f)	$Lu_3Al_5O_{12}:Ce$	350, 455	515	Nemoto
(g)	$YVO_4:Dy$	325	480, 575	Phosphor Technology
(h)	$Y_2O2_S:Eu$	345	626	Phosphor Technology
(i)	$Mg_{3.5}FGeO_5:Mn$ (MFG)	410	660	Nemoto

Figure 1 shows the photographs of prepared MFG- and BAM-G-based samples. Two photographs on the left show samples under white light, and the two on the right show emission images of samples excited by a 365-nm UV light. Figure 2 shows prepared samples excited by 365-nm UV light. The emission images of the nine phosphors used in this study are shown in Fig. 2. The excitation and emission wavelengths of each phosphor are shown in Table 1.

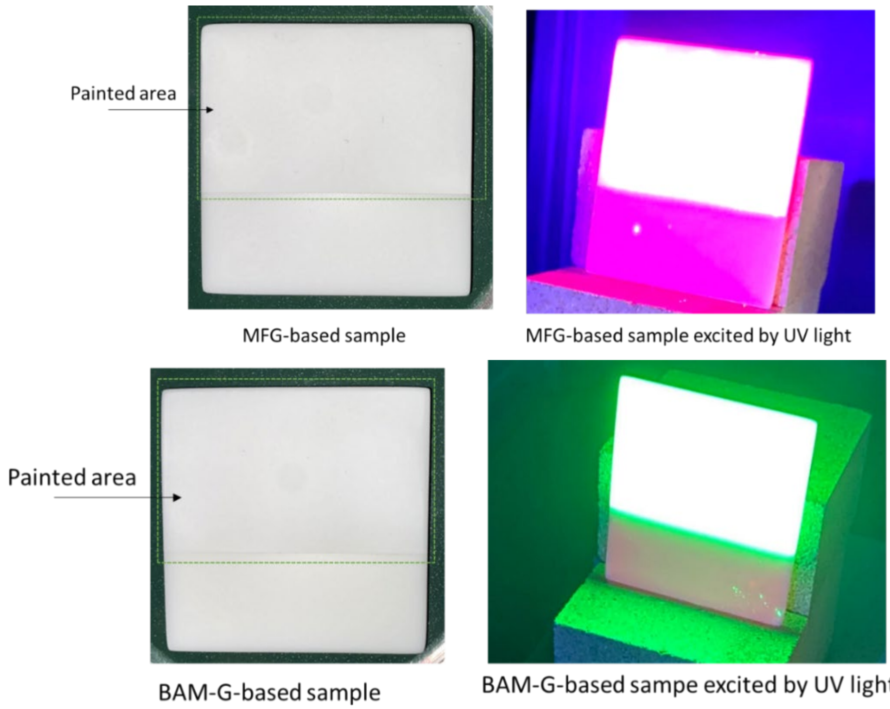


Fig. 1 MFG- and BAM-G-based samples. Right: painted samples, left: emission of samples excited by 365-nm UV light

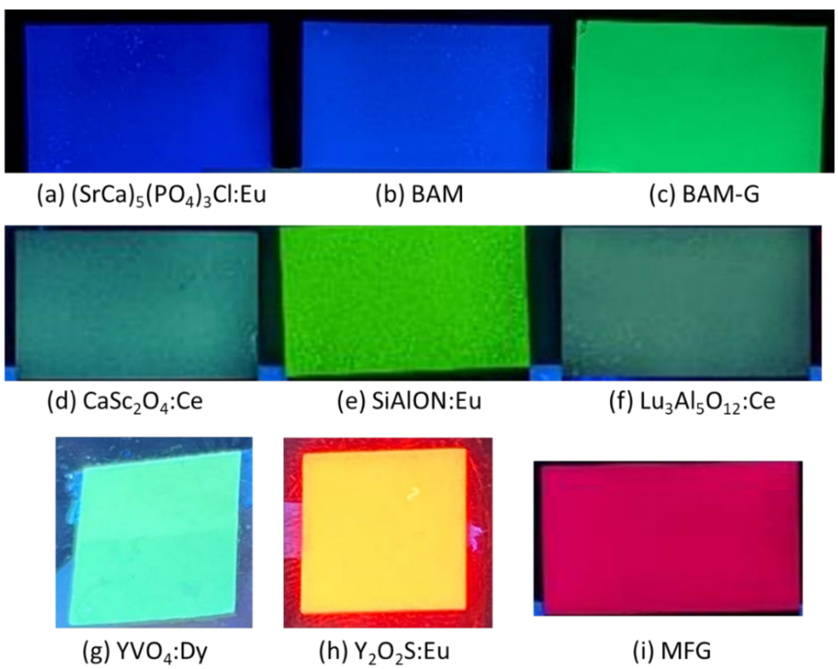


Fig. 2 Prepared thermographic phosphor samples excited by 365-nm UV light.

The temperature calibration was performed by varying the temperature from 100 to 800 °C under a constant pressure of 100 kPa. Figure 3 shows the experimental setup for temperature calibration. The prepared samples were placed in an electric furnace (ROP-001W, As One) with an optical window. The samples were excited with a 365-nm UV-LED (HLDL-50UV365-FN, CCS Inc.), and the luminescence from the samples was observed with a 16-bit CMOS camera (C11440-22C, Hamamatsu photonics) with an optical bandpass filter in front of the camera lens. The bandpass filter was chosen to be appropriate for the emission wavelength of each Phosphor. Obtained image data were processed with MATLAB® (MathWorks). To remove the effect of infrared radiation, the luminescent intensity, *I*, was calculated by subtracting the dark image without excitation light from the luminescence image with excitation light at each temperature. The luminescence intensity change by temperature can be described as the polynomial of the second order in Eq.(1):

$$\frac{I}{I_{\mathrm{ref}}} = A_0 + A_1 T + A_2 T^2 \qquad \text{(Eq (1))}$$

where, A_0, A_1, and A_2 are calibration constants. The temperature sensitivity, S_T, is defined as the slope of I/I_{ref} at a reference temperature of $T_{ref} = 100$ °C:

$$S_{\mathrm{T}} = \left.\frac{d(I/I_{\mathrm{ref}})}{dT}\right|_{T=T_{\mathrm{ref}}} = (A_1 + 2A_2 T_{\mathrm{ref}}) \times 100\ [\%/\mathrm{K}] \qquad \text{(Eq (2))}$$

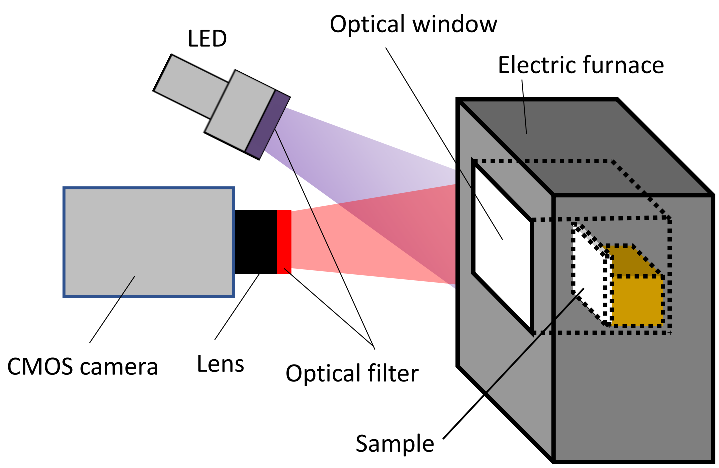


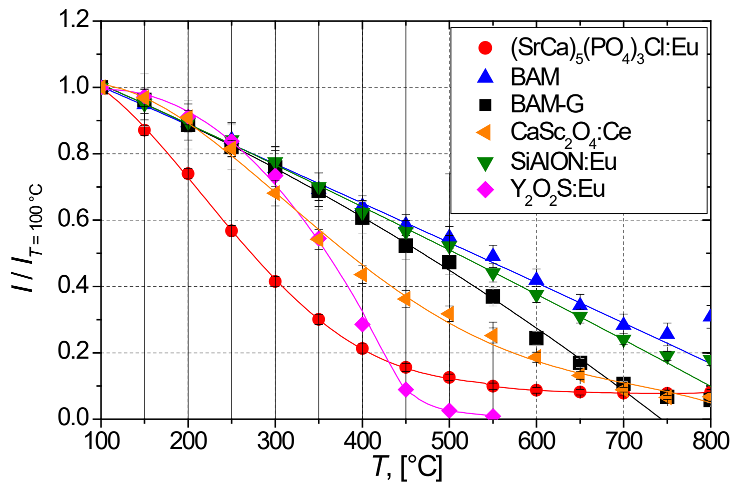
Fig. 3 Schematic view of temperature calibration

Temperature calibration curves of the developed sensors in Table 1 are exhibited in Fig. 4. In addition, the *I* and S_T of each sensor as well as the temperature range of high-temperature sensitivity, are summarized in Table 2. It can be seen that there are two major types of sensors: sensors whose luminescent intensity decreases monotonically with increasing temperature, as shown in Fig. 4 (a), and sensors whose luminescent intensity increases with increasing temperature up to a certain temperature and then begins to decrease, as shown in Figs. 4 (b) and (c). Among the sensors in Fig. 4 (a), where the luminescence intensity decreases monotonically with temperature, BAM, BAM-G, SiAlON:Eu, and Y2O2S:Eu showed high luminescence intensity. BAM, BAM-G and SiAlON:Eu have temperature sensitivity over a wide temperature range of 100 °C to 750 °C, although S_T is not as high as 0.12, 0.15, and 0.13%/°C, respectively. On the other hand, Y2O2S:Eu has a high S_T of 0.43%/°C, but the sensitive temperature range was limited to 200 - 400 °C. Lu3Al5O12:Ce, MFG in Fig. 4 (b) and YVO4:Dy in Fig. 4 (c) showed a maximum intensity at 250, 300, and 300 °C, respectively, and have high S_T of 0.27, 0.66, and 1.67, respectively, even in a limited temperature range. As shown in Fig. 5, YVO4:Dy has several emission peaks.

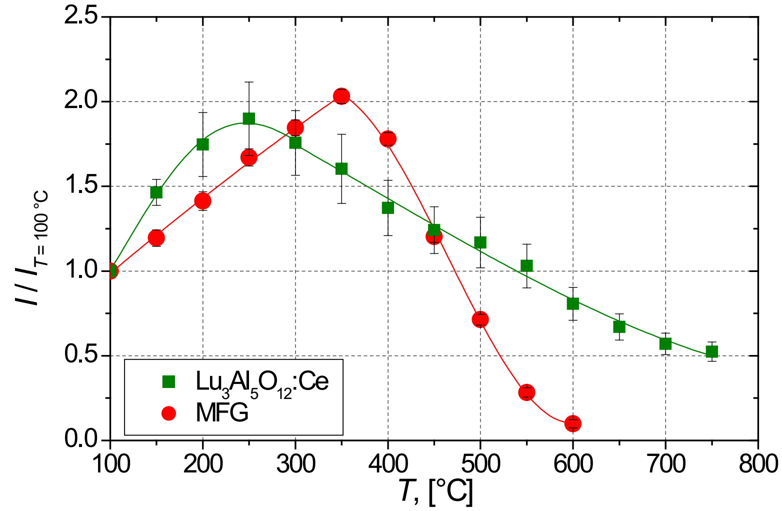
Table 2 Results of temperature calibration of phosphors

	Phosphor	Intensity	S_T [%/°C]	Temperature range with high S_T [°C]
(a)	$(SrCa)_5(PO_4)_3Cl$:Eu	Low	0.27	100 - 400
(b)	BAM	High	0.12	100 - 750
(c)	BAM-G	High	0.15	100 - 750
(d)	$CaSc_2O_4$:Ce	Low	0.17	200 - 700
(e)	SiAlON:Eu	High	0.13	100 - 750
(f)	$Lu_3Al_5O_{12}$:Ce	Low	0.27	250 - 750
(g)	YVO_4:Dy	High	1.67	350 - 450
(h)	Y_2O_2S:Eu	High	0.43	200 – 400
(i)	MFG	High	0.66	400 - 600

(a)



(b)



(c)

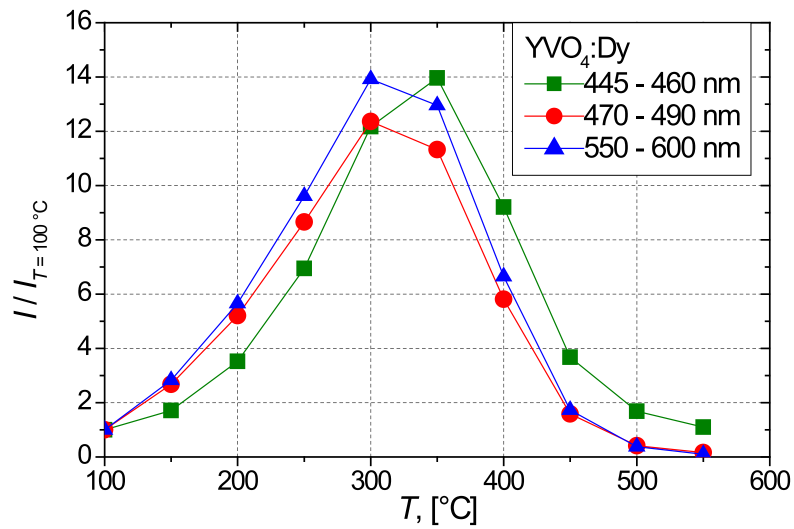


Fig. 4 Temperature calibration of thermographic phosphors.

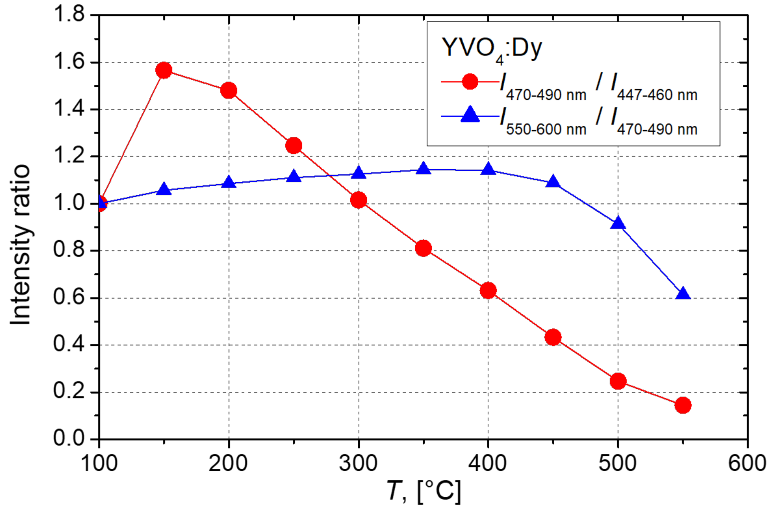
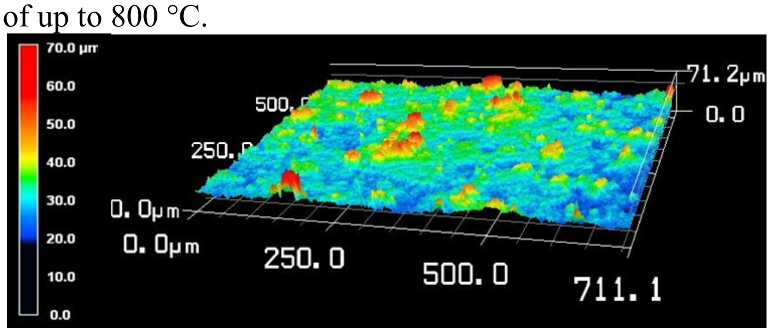
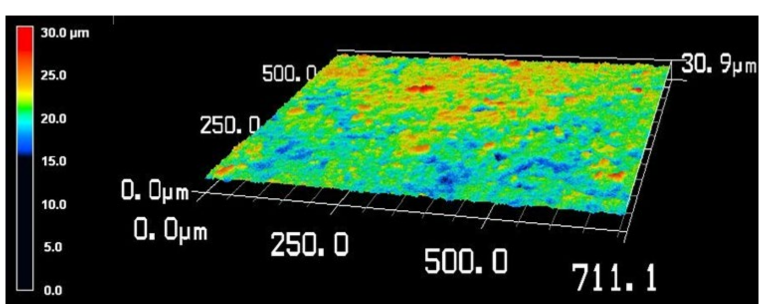


Fig. 5 Temperature calibration curve of YVO4:Dy.

The developed sensors showed sufficient adhesion to the ceramic substrate using silica-based white paint (Ceracoat 22) as a binder. The average roughness of the as-applied sample surface was Ra = 3.92 μm as shown in Fig. 5 (a). The sample surface could be polished with a polishing paper, and the average surface roughness after polishing could be reduced to Ra = 1.44 μm (Fig. 5 (b)). No apparent color change was observed on the surface after heating to 800 °C, nor were any cracks observed. Thus, the Ceracoat 22 used as a binder in this study has sufficient thermal and mechanical durability and good adhesion to the substrate for measurements in the temperature range of up to 800 °C.



(a) Before polishing: *R*a = 3.92 μm



(b) After polishing: *R*a = 1.44 μm

Fig. 5 Change in surface roughness *R*a of samples (a) before and (b) after polishing.

As the first hypersonic test, a rhodamine-B based temperature-sensitive paint was used to identify technical challenges. At this test, our most up-to-date temperature sensors were not able to be tested because these were under the development phase. The rhodamine-B based sensor has silica-gel particle and a silicone rubber as binding materials. The paint removal after the first wind-tunnel run was observed. Because of the paint removal, the temperature field under this hypersonic flow was not at the desired high temperature. In the next project year, our developed high-temperature sensor will be applied to this model to demonstrate the durability and the capability of the temperature measurement at a high-temperature region.

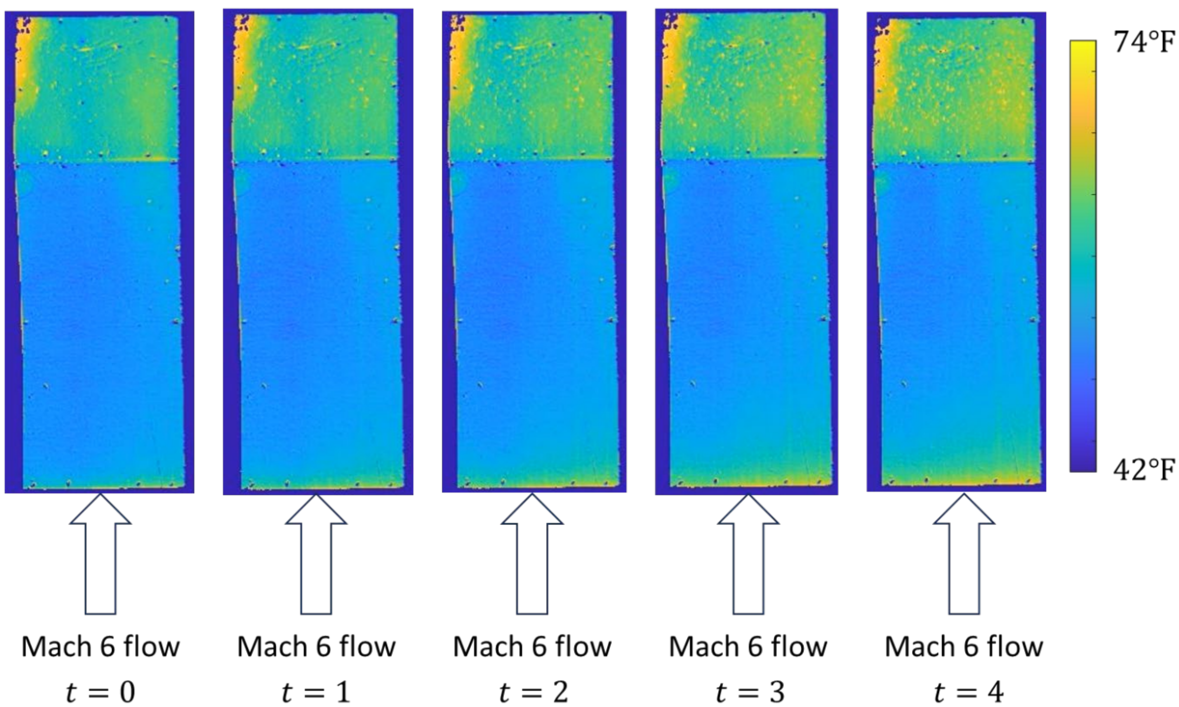


Fig. 6 Spatiotemporal temperature over a wedge model tested at AFRL hypersonic wind tunnel.