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FULLY ADAPTABLE INTERFACIAL SENSORS AND RECONSTRUCTION MODELING FOR IN SITU HEAT TRANSFER ANALYSIS OF ENERGY-SAVING MATERIALS

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ABSTRACT

Adaptable interfacial sensor technologies are essential to the realization of optimized energy-saving designs through in situ monitoring the material's performance of heat transfer. Previously reported other non-invasive thermosensors can either only monitor part samplings off site or lack signal processing circuitry and sensor calibration mechanisms for accurate analysis of the thermophysical performance. Given the complexity of cutting and sampling, on-the-spot measurement and real-time reconstruction modeling of target materials are critical and requires full adaptability to ensure the accuracy of heat transfer analysis. Here we present a fully adaptable interfacial (that is, no cutting sampling is needed) sensor for in situ heat transfer analysis, which selectively and accurately measures the key parameter reflecting the heat transfer performance, i.e., thermal conductivity, as well as reconstruction modeling based on the thermal conductivity data. Our work bridges the technological gap between signal transduction, amplification and filtering, processing in interfacial thermosensors by merging inorganic/organic-based sensors that interface with the on-the-sport material with integrated circuits consolidated on a printed circuit board for complex signal processing. This adaptably movable system is used to measure the detailed porosity-dependent thermal conductivity profile of materials engaged in energy-related applications, and to make a real-time reconstruction of heat transfer process of the on-the-spot materials. This platform enables a wide range of thermophysical monitoring and reconstruction modeling applications.

KEY WORDS: Nano/Micro scale measurement and simulation, Porous media, Energy-saving materials; Adaptable interfacial sensor; Heat transfer; Reconstruction modeling; 3ω technique

1. INTRODUCTION

Adaptable interfacial thermosensors are devices that can be mated with to closely monitor a material's thermophysical performance, without destroying or limiting the material's integrity [1-2]. Thus interfacial thermosensors could enable in situ analyzing heat transfer process [3-5]. At present, commercially available interfacial sensors are only capable of tracking a material's obvious heat transfer performance, typical indicating via thermal conductivity $k > 0.1$ W/m K, and fails to accurately indicate those k values for energy-saving materials, i.e. thermal insulations, let alone provide insight into the heat transfer process at real-time level. Accurate measurement of thermal insulations could enable such insight, because it could retrieve microscopic structure reconstruction [6-7]. Heat transfer analysis is currently used for applications such as

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structure diagnosis, defect detection, and energy-saving performance optimization [8-14]. For these applications, the sample collection and analysis are operated separately, failing to provide a real-time profile of heat transfer process on the spot, while requiring extensive follow-up analysis. Recently, adaptable interfacial sensors have been developed, with which a variety of thermosensors have been used to measure physical and chemical parameters of interest [1-4].

Given the multivariate modeling that are involved in material heat transfer, an attractive strategy would be to devise a fully adaptable interfacial sensing system to extract the thermal conductivity evolution trend and then implement reconstruction modeling. Here we present an adaptable interfacial thermosensor (AITS) for selectively and accurately measurement of thermal conductivity (Fig. 1a). Our solutions bridges the existing technological gap between signal transduction (electrical signal generation by sensors), conditioning (amplification and filtering) and processing (derivation of target parameters) in interfacial thermosensors by merging commercially available integrated-circuit technologies, consolidated on a printed circuit board (PCB), with interfacial and conforming sensors technologies fabricated on inorganic/organic substrates. This approach decouples the stringent practical requirements at the sensor level and electrical accuracy requirements at the signal conditioning and processing levels, and at the same time exploits the strengths of the underlying thermophysical parameter extraction technology, i.e. 3ω technique [15]. This platform is a powerful tool with which to advance on-the-spot and real-time thermophysical studies by facilitating the measurement of thermal conductivity in energy-saving materials.

2. EXPERIMENTAL METHODS

As illustrated in Fig. 1a, the AITS allows selective and accurate thermophysical measurement of energy-related materials in practical situation as well as heat transfer process rendering based on the obtained thermal conductivity evolution profile. By fabricating the sensors on a thermally insulated substrate while thin silicon nitrides (Si_3N_4) as an interfacial layer, a stable sensor-material contact is formed, while the PCB technology is exploited to incorporate the critical signal conditioning and processing functionalities using readily available integrated-circuit components (Fig. 1b). Additionally, high-precision electrical source, amplifier and electric capacitors are mounted to reduce the fluctuation in the readings of the harmonic voltage signals (see supplementary Information for selection of these components).

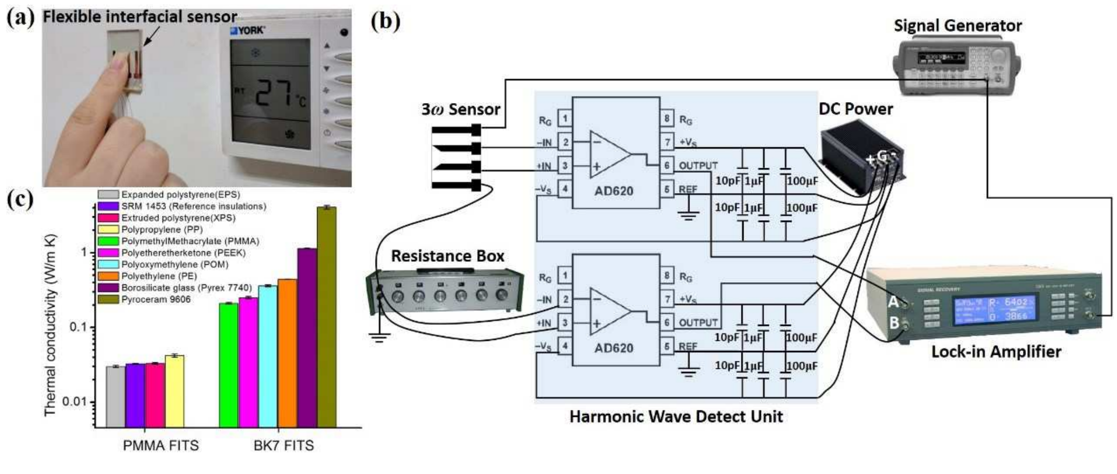

Fig. 1 Images and schematic illustration of the AITS for heat transfer analysis.
a, Photograph of a flexible interfacial AITS on a material's surface. (All photographs in this paper were taken by the authors.) **b**, schematic of the total measurement system for energy-saving materials. The Signal Generator is an Agilent model 33220A, which also provide a reference signal for the commercial Lock-in Amplifier (Ameteck Signal Recovery 7265). High-precision DC Power Source (Aerospace Changfeng Chaoyang Electrical Source Corp. 4NIC-X30), Instrument Amplifier (AD620), Electric Capacity (10 pF, 1 µF and 100 µF)

are mounted in a circuit board. **c**, thermal conductivity measurement results of varied reference specimens for the calibration of two types of AITS fabricated.

Figure 2 illustrates the schematic of the fabrication processes of AITS for heat transfer analysis; fabrication processes are detailed in Methods section. Here, AITS are based on BK7 glass or polymeric methyl methacrylate (PMMA) as backing, whose smallest surface roughness (< 100 nm) and excellent surface finish [16-17] ensure successful generation of continuous hundreds of nanometers thick Au/Cr strip, the core part of the thermosensor. The Joule heat released from the strip after driven by electric current is the probing signal for thermophysical measurement. Four rectangular pads serve as electrodes for connecting wires outside the sensor. The use of good thermal insulators as the substrate, i.e. BK7 glass and PMMA block the majority of the probing heat signal and thus ensure high sensitivity for the energy-saving materials which typically renders low thermal conductivity. Since the temperature rise of the strip only relates to the thermal conductivity and heat capacity of the materials to be measured and the known substrate [17-18], it is convenient to calculate the thermal conductivity of the materials from the readings of the transduced voltage signal of the AITS system.

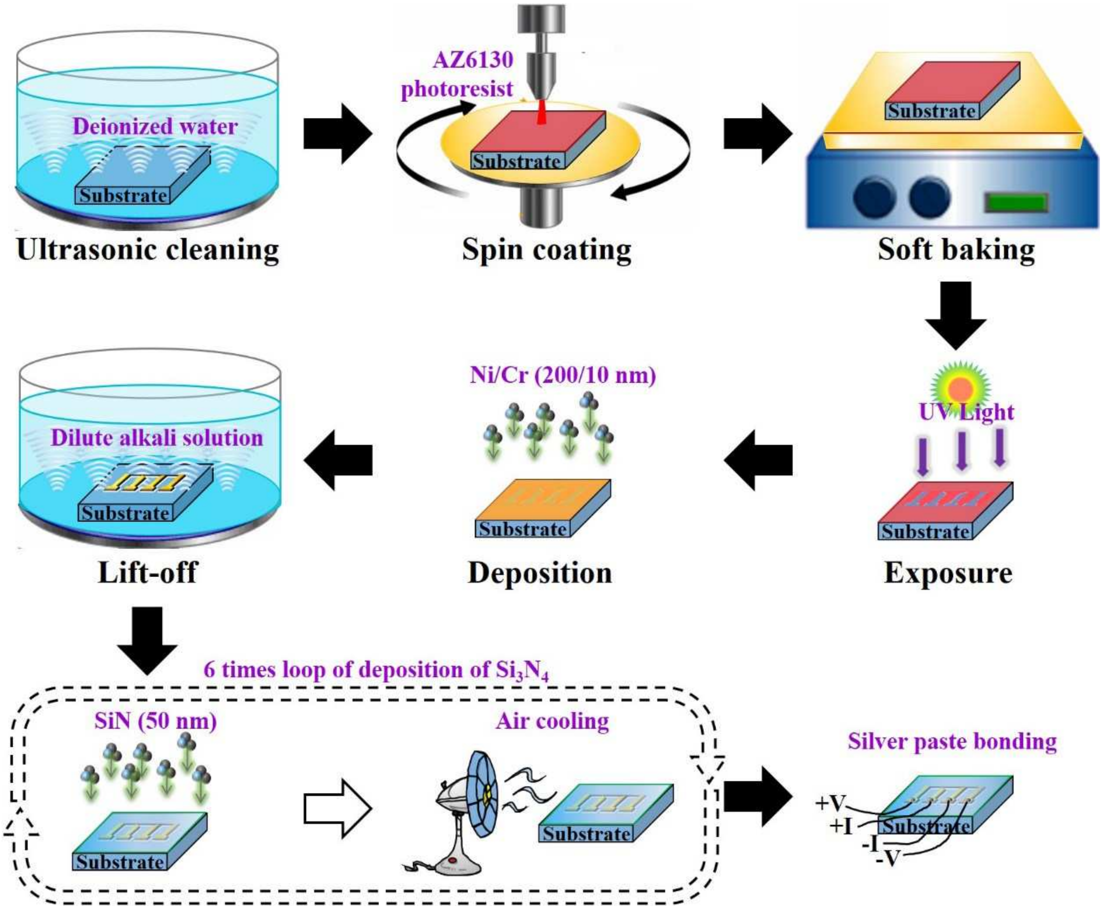

Fig. 2 Process flow for fabrication of AITS for heat transfer characterization.

The performance of each sensor was calibrated separately with different standard specimens with known thermal conductivities. Figure 1c shows the thermal conductivity results for representative reference specimens at room temperature (25 ºC) of BK7 glass and PMMA-based thermosensors. Results of repeatability and long-term stability studies indicate that the measurement error of the thermosensors are within 5.9%. The selectivity of thermosensors is crucial, because various substrate in AITS can influence the accuracy of the sensor. Generally, BK7 glass-based AITS provides good accuracy for measured k range 0.2~10 W/m K, while PMMA-based AITS is applicable to measured k below 0.2 W/m K.

Real-time thermophysical monitoring was performed on a representative energy-saving material, i.e. RS series of polymethacrylimide (PMI) foams, specifically designed to achieve the energy saving in aviation, aerospace, sports equipment for resin injection use and Resin Transfer Molding (RTM) manufacturing parts.

RS foams typically origin from free radical copolymerization of methacrylic acid (MAA) and methacrylonitrile (MAN). Specifically, the copolymerization forms poly (acrylonitrile methyl methacrylate) alternating copolymer (MAA-MAN) sheet, and cyclization and foaming process under heating form the PMI foam. At higher temperature (180~230 ºC), nucleophilic addition reaction occurs between adjacent cyano (CN) and carboxyl (-COOH) in MAA-MAN copolymer, resulting in the formation of the cyclic imide structure with strong polarity and high stiffness, as shown in Fig. 3a. The thermal conductivity of a series of RS foams with various densities, i.e. 30 kg/m3, 45 kg/m3, 100 kg/m3, 120 kg/m3 and 200 kg/m3 (corresponding porosity δ are 97%, 95.5%, 90%, 88% and 80% given the bulk density is 1000 kg/m3) were measured via PMMA-based FITS, and was found to increase the slope of the third voltage vs. frequency with increasing porosity as shown in Fig. 3b. Figure 3c shows the microscopic morphology structure of RS series from scanning electron microscope (SEM), indicating closed rates larger than 99% and the average pore size are statistically around 200 μm.

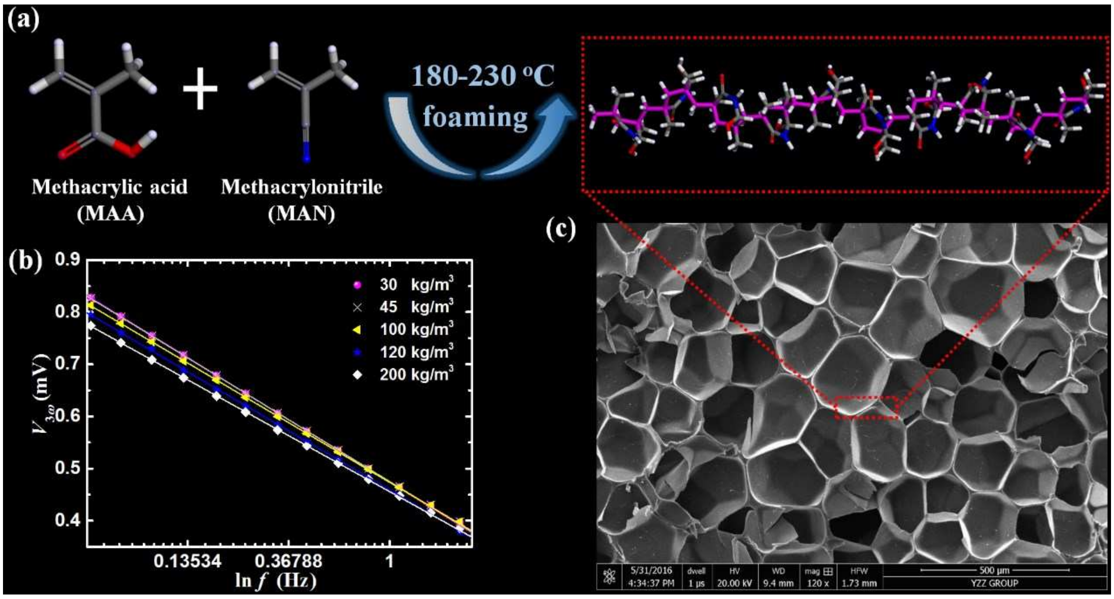

Fig. 3 Synthesis process and experimental data of thermal characterization for representative energy-saving material. **a**, Synthesis process of RS series of PMI foams. **b**, experimental measurement data of RS foams with different densities via the AITS-based 3ω method. **c**, representative SEM image for RS series of PMI foams.

3. REAL-TIME HEAT TRANSFER ANALYSIS AND RENDERING

The second part of AITS technique is real-time rendering of the reconstructed optimum heat transfer model during data processing. Unlike real microscopic morphology structure obtained via SEM technique, the model which could describe the heat transfer process has multiple choice, such as generally used two-dimensional (2D) series model, parallel model, Effective Medium Theoretical (EMT) model, Bruggeman model, and Maxwell-Eucken (ME) model [19-22] (see supplementary Information for detailed introduction of these models). The interactive software companying with AITS embedded the above-mentioned heat transfer models and two three-dimensional (3D) models developed by us. The first 3D model is called hollow cube modeling [23], which was proved a success for accurate heat transfer simulation of aluminum foams [24]. Figure 4a schematically shows the unit of hollow cube model (see Methods for detailed calculation procedures) and Fig. 4b presents calculation results from Fluent® software, i.e. the steady temperature distribution of the model under the temperature difference 10 ºC, which was then used to extract thermal conductivity k for the specimen. Figure 4c shows the k versus porosity trend for RS foams.

The second 3D model newly embedded in our interactive interface is CT scanning reconstruction modeling, based on X-ray tomographic imaging technique, which has been verified powerful for extraction of thermal conductivity of porous materials [25-27]. It is to be noted that this technique is difficult to discern the solid and gas phases for the specimens with extremely small pores, such as RS foams with porosity 97%. But for lower porosity, such as those with porosity lower than 95.5% and generally large pore size, the morphology of porous structures can be excellently reconstructed (Fig. 5a-d). One merit of this technique is the distribution of thermal conductivity along 3D directions could be readily calculated based on finite element method, and the final results is the average value of all directions since the microscopic structure along x, y and z direction are close to each other, as shown in Figure 5e.

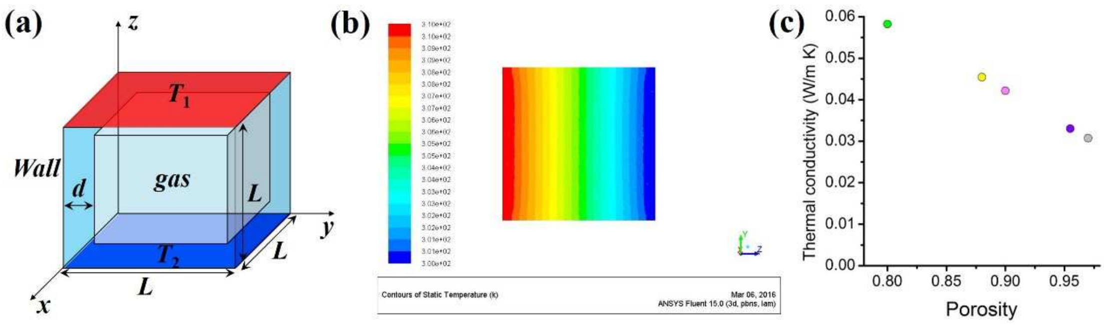

Fig. 4 Hollow cube model for heat transfer analysis. **a**, Schematic for hollow cube model. **b**, the simulated temperature distribution under steady heat conduction. **c**, calculated thermal conductivity versus porosity.

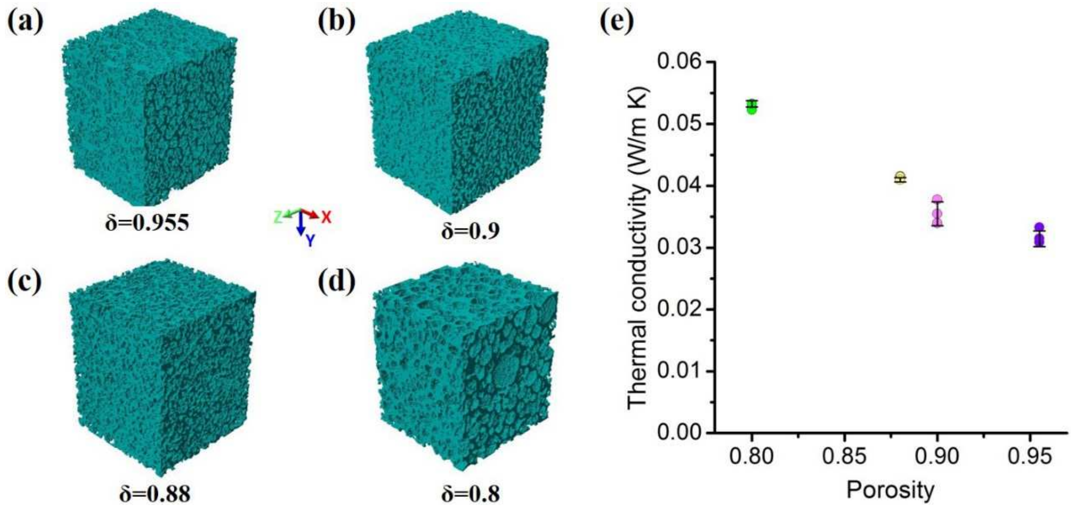

Fig.5 CT scanning reconstruction for heat transfer analysis for RS foams with varied porosities. **a-d**, reconstructed images for further data processing. **e**, the corresponding calculated thermal conductivity.

4. RESULTS AND DISCUSSION

Rendering heat transfer model is of the utmost importance to energy-saving materials because this analysis could provide for helpful guidance for optimization of the structure to achieve desirable heat transfer performance. The interactive interface integrated in our measurement system could rapidly indicate the trend of thermal conductivity versus porosity after the in situ measurement of the representative energy-related materials, such as RS foams. Generally, the trend calculated from seven heat transfer models, i.e. series, parallel, EMT, Bruggeman, Maxwell, hollow cube and CT scanning reconstruction models, will emerge timely with the experimentally determined data scattering. Figure 6 shows the on-the-spot measured thermal conductivity versus porosity for RS foams and the prediction from various heat transfer models. The parallel model and series model are still representing the upper limit and lower limit for the thermal conductivity trend as usual. The calculated thermal conductivity results from EMT model agree well with the experimental data for samples with very high porosity but are still remarkably lower for RS foams with relatively lower porosities. The underlying reason is EMT model deploys tiny squares to construct the matrix (see supplementary Information for detailed introduction of this model), which brings about thermal contact resistance between adjacent squares not existing for real structures. Thus, EMT model is close to series model, as shown in Fig. 6.

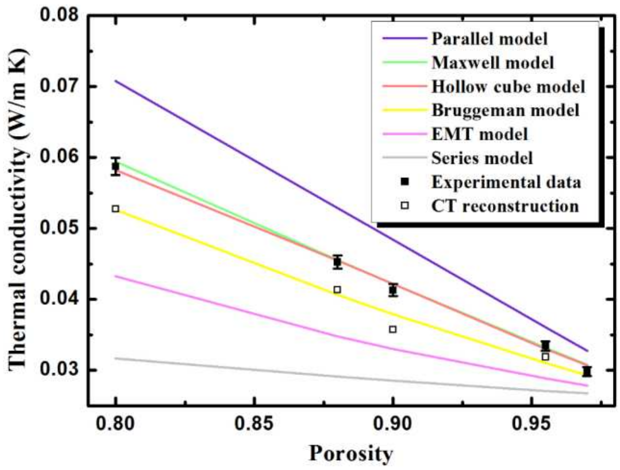

Fig. 6 Comparison of experimental and calculated porosity dependence of thermal conductivity based on various heat transfer models for RS foams.

It is intuitive to draw the conclusion that Maxwell model and hollow cube model give accurate description of the heat transfer process for RS foams investigated from Fig. 6, which indicates they are able to accurately describe the thermal transport of close-cell materials. CT scanning reconstruction method surprisingly deviates remarkably from the real results, indicating the micro-CT scanning reconstruction technique at the present stage fails to discern some tiny connections of solid skeleton and thus increases the solid-gas interface thermal resistance, a main culprit for underestimation of the thermal conductivity. It is to be noted that the result from CT scanning reconstruction agree well with that from the 2D Bruggeman model (see supplementary Information for detailed introduction of these models). The underlying reason is both of these approaches are based on constructing approximately real skeleton model via capturing the gray information of scanned images, which unavoidably losses some tiny solid-solid interfacial connection structures. Maxwell model and hollow cube model, however, are verified the most accurate description for the closed-cell foams investigated. This is because the polyhedral cell consisting of the foam is a rigorously closed structure, similar to Maxwell model and hollow cube model. And although the shapes of polyhedral cell includes quadrilaterals, pentagons, hexagons and heptagons within RS foams, the simplification as circular or cube actually would not induce discernable error for modeling the heat transfer process.

6. CONCLUSIONS

Thus, we have merged fully adaptable interfacial thermosensor (two different sensors) connected with conventional commercially available integrated circuit components and real-time reconstruction modeling for heat transfer analysis at an unprecedented level of integration, not only to accurately and on-the-spot measure the energy-saving materials, but also to render their porosity dependent thermal conductivity relation via reconstruction of the heat transfer process. This application could not have been realized by either of the technologies (adaptable sensors and silicon integrated circuits) alone, owing to their respective inherent limitations. The inorganic/organic-based sensor technologies lack the ability to implement sophisticated electronic functionalities for complex signal conditioning and processing. On the other hand, the silicon integrated-circuit technology does not provide full adaptability to ensure the accuracy required to achieve reasonable heat transfer analysis for thermal insulation materials. Importantly, the entire system is mechanically adaptable, thus delivering a practical interfacial sensor technology that can be used for prolonged on-the-spot test activities. This platform could be exploited to facilitate real-time heat transfer modeling. We envision that the in situ data sets that could be collected through such studies, along with simultaneous heat transfer process reconstruction, would enable in situ thermal conductivity measurement techniques with which to generate predictive model for understanding the heat transfer process and practical needs of energy-related applications.

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