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THERMAL NONDESTRUCTIVE CHARACTERIZATION OF THE INTEGRITY OF PROTECTIVE COATINGS

Protective coatings, such as the thermal barrier coatings developed for use on turbine blades in jet aircraft engines, are often considered "prime reliant" because, in harsh operating environments, failure of the coating could result in the loss of the component it protects. The nondestructive thermal inspection technique of time-resolved infrared radiometry, developed to detect flaws in protective coatings that could lead to coating failure, is an extension of earlier thermal wave imaging techniques developed to examine material microstructure and defects. The technique is completely noncontacting and allows both the coating thickness and the integrity of the bond between the coating and the substrate to be determined simultaneously.

INTRODUCTION

Protective coatings form an integral part of many engineering structures. They play an important role in maintaining the operability of many systems; applications range from providing thermal barrier and oxidation protection in the aerospace industry to corrosion protection in marine applications. Many coating systems are used in all aspects of civil and defense structures, but the common role of all coatings is to protect the substrate from degradation caused by harsh operating environments. In fact, many new materials such as carbon–carbon composites depend on coatings to permit operation in the high-temperature environments for which they have been designed. The effects of a failure of a protective coating can range from cosmetic inconvenience (a poor paint job on an automobile) to loss of a valuable vehicle and personnel (failure of the tiles on the space shuttle).

The availability of accurate, noncontacting, and cost-effective characterization techniques for the nondestructive analysis of protective coatings has important ramifications throughout the life of a coating system. Nondestructive evaluation techniques are needed to monitor the integrity of a coating throughout its operational lifetime and to detect incipient failure before irreparable damage is done to the substrate. The early development stages of a coating system require characterization techniques to determine the properties of various candidate systems and to monitor their behavior during exposure tests. Further, the reliability of a coating system in service depends in a large measure on the quality control that was exercised during its application. On-line characterization techniques can ensure that the application process itself is adequate.

The development of thermal characterization techniques for coating systems—specifically, the method of time-resolved infrared radiometry (TRIR)—has been one extension of our previous work in thermal wave imaging of microstructure and defects in metals, semiconductors, and ceramics, reported in 1986.[1] Some of our early results on coating characterization were reported in 1988.[2] Here, we provide an update of our most recent results.

The TRIR technique is potentially valuable to map areas of disbonded coating as well as to determine physical parameters of coatings, such as thickness and thermal diffusivity. An important aspect of this work has been the integration of an experimental program in technique development and specimen characterization with an extensive modeling effort to provide a quantitative basis for the technique. Much of the thermal technique development has focused on the characterization of thermal barrier coatings used in aircraft turbine engines. Those coatings provide both oxidation protection and thermal insulation for metallic engine components and significantly improve engine efficiency and performance.[3,4] The thermal techniques we have developed for coatings will also have direct applications to other layered and structured systems, such as composite materials and thin-welded structures, for which information on disbonding between layers and details of layer properties is required.

THE TRIR TECHNIQUE

With the thermally based nondestructive TRIR technique, the surface temperature of a specimen is monitored as a function of time during and after the application of a heating pulse. Variations in the surface temperature depend on the details of the heat flow through the specimen and are sensitive to variations in coating thickness and coating disbonds. We study the development with time of the surface temperature for both heating and cooling, during and after the application of a step heating pulse of a specified duration. The technique, implemented with an infrared scanner, allows full-field images to be obtained much more rapidly than is possible with continuous-wave-modulated thermal wave techniques. Since all times are monitored, the thickness of the coating can be measured directly from the experimentally detected thermal transit time of the coating. Therefore, both coating thickness and the integrity of the coating–substrate bond can be determined simultaneously. This differs from the continuous-wave technique, where it is necessary to determine the thermal transit time from the frequency dependence of the surface temperature.

Specific advantages of the TRIR technique include

1. The ability to determine either coating thickness or coating thermal diffusivity.
2. Sensitivity to disbonds between coating and substrate, including those that indicate incipient spallation of the coating.
3. The noncontacting nature of the technique—neither specimen heating nor temperature detection requires sample contact.
4. The parallel nature of the detection process, which allows entire components to be studied in a single measurement.
5. The applicability of image processing techniques to enhance contrast indicating coating defects.
6. The implementation of a practical, field-operable measurement system.

A schematic diagram of the system used for TRIR measurements is shown in Figure 1. An argon ion laser used as the heating source is time-gated by means of an acousto-optic modulator to provide a range of pulse lengths. Either of two heating beam profiles can be selected: a Gaussian profile with a chosen beam diameter, or a line profile obtained by using a cylindrical lens or an acousto-optic deflector. An infrared scanner monitors the surface temperature of the specimen as it changes with time. Synchronizing electronics turns on the laser heating source at a fixed time with respect to the frame rate of the scanner. The data are transferred for analysis to a microcomputer over the IEEE-488 bus.

Three types of TRIR measurements are typically made: temperature–time line scans, X–time temperature images, and X–Y temperature images. The temperature–time line scans monitor the development of the surface temperature at a particular point on the specimen. Analysis of the curves and subsequent comparison with theoretical calculations allow coating thickness to be measured and regions of disbonded coating to be identified. X–time images show the development of the surface temperature with time along a line in one direction (X-direction) on the sample, with time as the vertical axis in the image. Temperature is displayed in pseudo-color, gray-scale, or contour representations. The final data presentation type involves placing the sample on a positioning system stage and collecting a series of X–time images as a function of vertical position on the sample. A particular time slice from each X–time image is used to construct full-field TRIR X–Y images of the sample for any desired time interval.

THEORETICAL AND EXPERIMENTAL RESULTS

Theoretical modeling has been a crucial part of this program and has provided many insights into the varia-

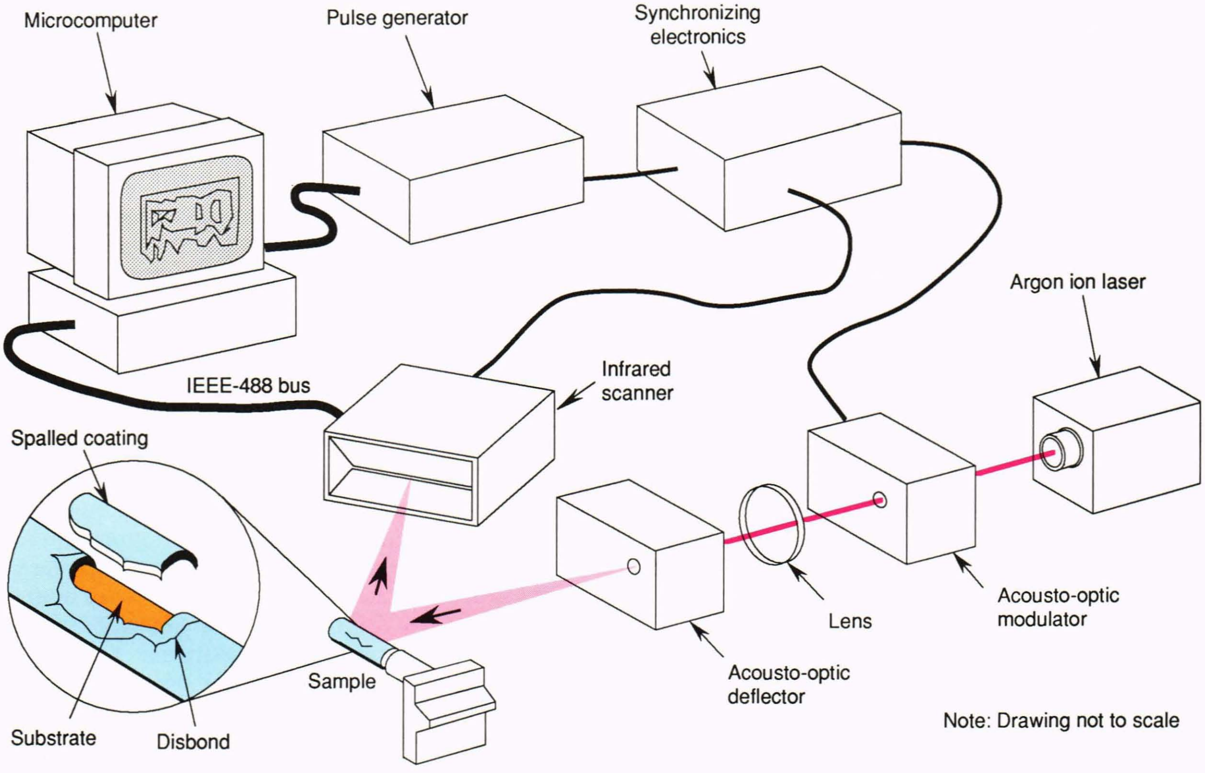

Figure 1. Experimental setup for conducting TRIR measurements.

tions in coating–substrate bonding and coating properties that we can expect to detect experimentally. A one-dimensional model has been developed[5] that describes the temperature-versus-time response for step heating of a three-layer system consisting of a substrate, a coating, and the gas layer above the coating. The results for a series of coating–substrate combinations are shown in Figure 2 for (curve a) a semi-infinite specimen of zirconia; zirconia coatings of thickness (curve b) 200 μm, (curve c) 400 μm, and (curve d) 600 μm on a superalloy substrate; and (curve e) a 400-μm layer of zirconia on an air substrate to simulate a disbonded condition. Note that temperature is plotted versus the square root of time from the onset of the heating pulse, because this presentation linearizes the result for the semi-infinite response. The semi-infinite response then becomes a convenient reference curve against which the coating–substrate curves can be compared.

All of the curves in Figure 2 show the same early behavior, but for coatings on the superalloy substrate (b–d), the curves begin to drop below the semi-infinite response at times that are determined by the coating thickness. The characteristic thermal transit time is $0.36\ L^2/\alpha$, where L is the coating thickness and α is the thermal diffusivity. At that time, the presence of the substrate is detected, and because the substrate is more thermally conductive than the coating, the rate of temperature rise decreases. The TRIR technique is also useful when inspecting multilayered coating systems. Theoretical and experimental results[6] have shown that it is possible to detect a thermally insulating layer beneath a thermally conducting substrate layer by means of an increase in slope at a fixed time after the initial slope decrease when the first substrate layer was detected.

Curve e in Figure 2 for a 400-μm layer of zirconia coating on a thermally insulating air substrate shows the same initial behavior as that of the 400-μm coating on the superalloy substrate (curve c). But once the characteristic thermal transit time for this coating thickness is reached, the air substrate data show an increase in slope, in contrast to the decrease in slope for the superalloy substrate data, because of the thermally insulating nature of the zirconia/air boundary. This signal response can be used to monitor coating disbonding in real engineering systems.

Experimental temperature-versus-time line scans are shown in Figure 3 for three locations on a thermal barrier coating test specimen consisting of a 250-μm-thick zirconia coating on a superalloy substrate. The temperature–time curve for a region of coating known to be well bonded shows a response similar to that of curve c in Figure 2, although the thermal transit time is different because of a different coating thickness. Curves a and b in Figure 3 were obtained at two different locations adjacent to a region of coating that had spalled off, revealing bare substrate. These two curves follow the semi-infinite response up to the same thermal transit time as was found for the well-bonded curve; at that point, they show the slope increase predicted for a thermally insulating substrate shown in curve e of Figure 2, indicating that the coating in this region is disbonded.

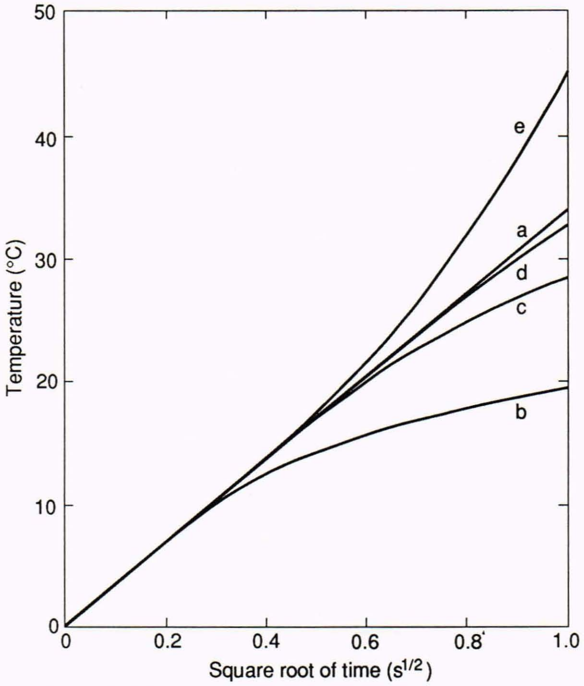

Figure 2. Theoretical curves of temperature versus the square root of time for (curve a) a semi-infinite specimen of zirconia; zirconia coatings of thicknesses (curve b) 200 μm, (curve c) 400 μm, and (curve d) 600 μm on a superalloy substrate; and (curve e) a zirconia coating 400 μm thick on an air substrate to simulate a disbond.

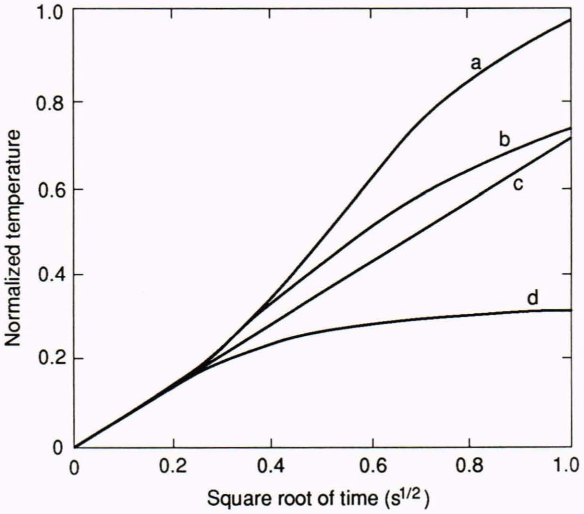

Figure 3. Experimental line scans of temperature versus the square root of time for three locations on a thermal barrier test specimen, showing temperature–time response for a well-bonded coating (curve d) and for two locations in the region of disbonded coating (curves a and b). Curve c shows the semi-infinite response.

The curves in Figure 3 show some features that are not exhibited by the one-dimensional model. By about $0.6\ s^{1/2}$, the two disbonded curves begin to show decreasing slope values, as opposed to the ever-increasing slope found in the one-dimensional theoretical example.

A recent three-dimensional model[7] has shown this to be the result of lateral heat flow in the coating that depresses the peak temperature excursion. As the diameter of the heating beam is decreased, the lateral heat-flow effects become more pronounced. We are investigating a way to measure lateral thermal diffusivity by this effect.

Another feature of interest in Figure 3 is that after the thermal transit time is reached, the two disbond curves do not follow the same dependency. Because of differences in the heat flow from the coating to the substrate at the two locations, resulting from variations in the adhesion of the coating to the substrate, one curve shows a substantially greater temperature excursion than the other. This indicates that the degree of disbonding of the coating between the two locations is different and that the TRIR technique is sensitive to variations in the properties at the coating/substrate interface.[8] Further work is in progress to calibrate the relationship between the TRIR response and the degree of disbonding and, hence, the remaining strength of the bond. This result is potentially very important because, although some ultrasonic techniques can detect coating disbonds, there is no technique that quantifies the strength of a bond.

A specimen was prepared to test the ability of the TRIR technique to detect a simulated coating disbond. A 775-μm-thick layer of a machinable ceramic (Macor) was bonded to an aluminum substrate containing two holes 2 and 5 mm in diameter, as shown in Figure 4A. The theoretical characteristic thermal transit time for this coating is 0.31 s (0.56 $\mathrm{s}^{1/2}$). The TRIR X–Y images produced for the specimen orientation shown in Figure 4A show an intensity variation in the X direction that is due to the spatial nonuniformity of the line heating source. This beam profile effect can be removed and the detectability of the defects improved by dividing an image acquired in the defect region by an image acquired over well-bonded material.[9] The line heating source was successively positioned along the surface of the test specimen with the 5-mm defect at the top and the 2-mm defect at the bottom. The three images shown in Figures 4B, C, and D were produced from time slices of the X–time images at $t = 0.22$ s, which is less than the thermal transit time for the coating (Fig. 4B); at $t = 1.0$ s, which is greater than the thermal transit time (Fig. 4C); and 0.05 s after the end of the heating pulse (Fig. 4D). Three features are observed in images C and D. The middle feature is the 5-mm defect and the bottom one is the 2-mm defect. The top feature is an unintentional disbond between the coating and the substrate caused by poor bonding when the test specimen was made. The features are not found in image B, because the time that image was taken was less than the thermal transit time of the coating.

CONCLUSIONS

Thermal characterization techniques for protective coating systems play an important role in ensuring the integrity of coating systems and, ultimately, the structures they protect. The TRIR technique provides simultaneous thickness measurement and disbond detection. We are also able to measure multilayer systems and to determine the degree of disbonding. A key requirement in obtaining quantitative information from TRIR measurements is to analyze temperature–time line scans and compare those data with one- and three-dimensional models.

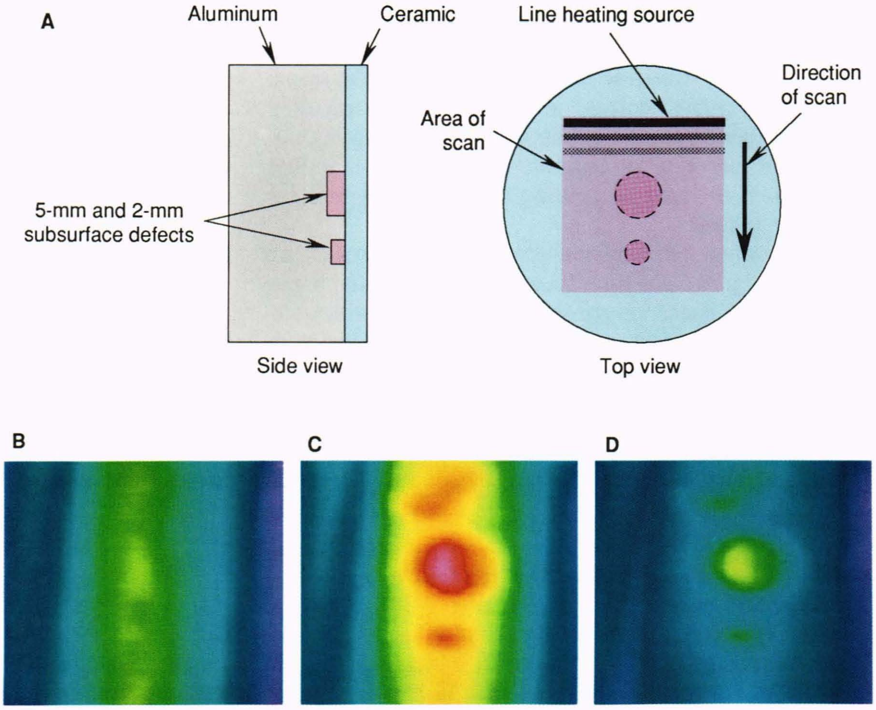

Figure 4. **A.** Scanned region for TRIR X–Y images on the test specimen. **B.** 0.22 s after the start of the heating pulse. **C.** 1.0 s after the start of the heating pulse. **D.** 0.05 s after the end of the heating pulse.

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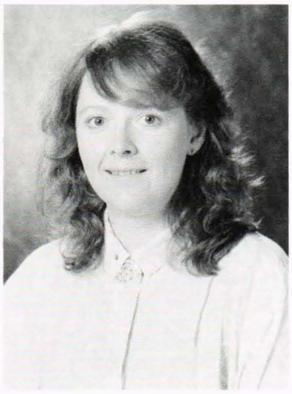

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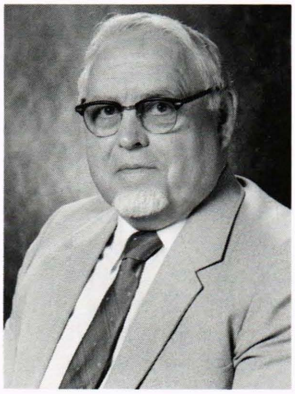

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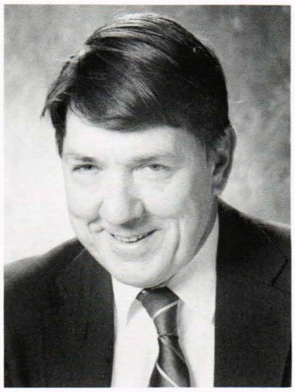

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