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THERMAL ANALYSIS  
OF  
POLYMER COMPOSITES

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## A. STATEMENT OF THE PROBLEM

### A.1 Introduction

Polymer composites are currently used in a wide variety of structural and thermal protection applications. Although the structural applications of polymer composites have resulted in the bulk of research activities being concentrated on mechanics and mechanical properties at room temperature, there has over the past decade been an increasing interest in the high-temperature, thermal protection application of these materials. In the area of glass-filled polymer composites, the impetus behind this recent interest has been their use in such high-temperature, thermal protection systems as spacecraft heat shields for reentry, missile magazines, rocket motor nozzle liners and blast deflectors. In these applications, the materials are exposed to temperatures high enough to cause decomposition of the solid material. In fact, overwhelming acceptance of this material stems from a high char yield resulting from pyrolysis reactions, coupled with favorable thermal and transport properties of the product char. As a result of the decomposition process, the overall thermally-induced response of the material is quite complex and varies with material composition and/or processing. To design reliable and efficient thermal protection systems, it is necessary to predict the thermally-induced response of these materials a priori. Accurate prediction of this thermal behavior requires the solution of a complex numerical model which includes all of the physical processes that occur in the material over the temperature range of interest. This response is governed, in part, by the material properties which must be accurately determined and modeled so as to be used in the overall numerical scheme.

This research focuses on two widely used glass-filled polymer composites designated as H41N and MXBE-350.

Thermochemical expansion is a property which strongly affects the performance of decomposing, glass-filled polymer composites, and must be accurately determined for input into the overall numerical modeling scheme. Although a voluminous amount of literature exists on the thermophysical properties of these types of materials, there is a dearth of published

thermochemical expansion data over the entire temperature range of decomposition. To study the expansion characteristics of these materials over the entire temperature range of the carbon-silica reactions, it is necessary to carry out experiments to temperatures as high as 2000°C. Therefore, the first objective of this study was the measurement and empirical modeling of the expansion characteristics of the two materials from 20 to 2000°C, at heating rates of 5, 10, 20 and 50°C/min. To the best of the author's knowledge no experimental expansion measurements on these types of materials have been performed in excess of 1500°C.

The existence of local-thermal equilibrium between the solid material and decomposition gas within the tortuous pore network of the material has been an inherent assumption in previous mathematical models dealing with the prediction of the thermal response of these and other similar materials. However, since the thermal response of these types of materials is highly transient, large deviations from local-thermal equilibrium may indeed exist. This suggests that the solid material retains a much larger amount of energy than predicted by current numerical modeling schemes. Clearly, the thermal decomposition of a material is enhanced with increased departure from thermal equilibrium. Consequently, accurate modeling of the thermal behavior of these types of materials should not include the idealized assumption of local-thermal equilibrium. Modeling in the absence of local-thermal equilibrium requires equations for energy conservation in both the solid and gas phase, coupled by what is termed the volumetric heat transfer coefficient,  $h_v$ .  $h_v$  characterizes the rate of energy transfer between the two phases within the tortuous pore network of the material.

The second objective of this study was the design and construction of an experimental apparatus to measure the volumetric heat transfer coefficient. For both materials, volumetric heat transfer coefficients were measured for decomposition temperatures out to 800°C, at a variety of gas flow rates. These data were modeled using a modified correlation in the form of a Nusselt number as a function of Reynolds number. To the best of the author's knowledge no such heat transfer measurements have ever been conducted in the pore network of these types of materials.

Finally, the main objective of this investigation was the development of a one-dimensional, transient numerical model to predict the overall thermally-induced response of decomposing, glass-filled polymer composites. Unlike previous models, the present model does not include the idealized assumption of local-thermal equilibrium existing between the two phases. Using the empirical correlations developed for the expansion characteristics and volumetric heat transfer coefficients, the model was exercised to quantify the overall thermally-induced response of the two materials for a given set of boundary conditions. The model was then exercised with a constant volumetric heat transfer coefficient throughout the domain, large enough to induce approximate local-thermal equilibrium for both materials. From these results, the effects of the assumption of local-thermal equilibrium on the overall thermally-induced response of both materials was determined. Lastly, the model was exercised to quantify the thermal response of H41N and MXBE-350 with a surface heat flux sufficient enough to initiate the carbon-silica reactions.

#### A.2 Material Description

The two composites studied are designated as H41N and MXBE-350, and are fabricated by Ametek, Haveg Division and Fiberite Corporation, respectively. These resins are of the thermosetting variety, meaning they undergo chemical crosslinking during fabrication. Both materials consist of a basic phenol-formaldehyde resin with varying quantities of fillers and reinforcing agents. The fiberglass (reinforcing material) may contain coupling agents and/or binders to insure proper adhesion to the resin network. The fillers improve both the impact strength and flexural modulus of the composite. These materials were chosen for study because they exhibit typical decomposition/length change behavior for glass-filled thermoset composites. Both materials are also of great practical importance since they are used extensively in high-temperature, high-heating rate thermal protection applications.

H41N consists of a phenolic based resin containing short glass fibers and talc. The manufacturing procedure consists of mixing the glass fibers

and talc into the resin, such that the glass fibers are randomly orientated. The material is then placed in a mold, and is either rolled or pressed until its shape conforms to that of the mold. During this procedure the glass fibers become partially orientated in a direction normal to the applied pressure. Afterwards, the material is cured in an autoclave at a specified pressure, under a carefully monitored heating cycle. As a result of this curing process, H41N exhibits transversely isotropic behavior. For example, the material expands significantly in the direction normal to the preferred fiber orientation, while displaying little expansion in other directions.

MXBE-350 consists of acrylonitrile-butadiene (rubber) modified phenolic resin containing fiberglass mat and glass powder. The manufacturing process is initiated by sufficiently wetting the woven glass mat with the resin system. The wetted mat is then partially polymerized in a drying oven (B staged). After B staging, the mats are cut to desired dimensions, stacked several layers deep in a laminating press (to desired thickness), and cured under pressure and controlled heating conditions. It is important to note that the glass fibers in the fiberglass mat, used in MXBE-350, are more randomly orientated than the fibers in standard broadgoods, commercially available for reinforcing laminated composites. As a result, the glass-fibers in MXBE-350 are only partially orientated in the direction normal to the applied pressure. Hence, in some respects these two materials behave similarly. For example, the overall thermochemical expansion behavior of MXBE-350 is similar to that of H41N.

### A.3 Material Behavior

When a polymer composite material is exposed to a surface heat flux, the initial heat transfer is primarily due to transient energy conduction. During this period, the material experiences a small amount of expansion, characterized by the linear coefficient of thermal expansion of the virgin material, i.e.,  $\alpha_v$ . When the material reaches sufficiently high temperatures (200 - 300°C), dependent on material processing, composition and heating rate, chemical reactions begin to occur. These thermally-induced reactions, commonly referred to as pyrolysis reactions, result in the degradation of the

resin component of the composite matrix to residue char and product volatiles. This reaction zone moves from the heated surface through the material. During the initial stages of the pyrolysis reactions, decomposition gases are trapped within the pore network due to low material permeability. This accumulation of gases results in the internal pressurization of the material. This internal pressurization is at least partially responsible for a very rapid and sometimes quite large expansion of the material which occurs at this time. The work associated with expansion is now a means of energy transfer. As a result of the continuing expansion and decomposition the material permeability and porosity begin to increase. This increased permeability, coupled with existing pressure gradients, results in the flow of gas through the pore network. As a result of this flow, large deviations from local thermal equilibrium may exist between the solid material and product gas. The rate of doing work resulting from the flow of gas now becomes an important means of energy transfer.

The gases which flow back through the char structure remove energy by convection with the solid, thus attenuating the conduction of heat to the reaction zone. The gases which flow through the virgin material serve to pre-heat the material. The rate at which energy is transferred between the solid and gas in the pore network is characterized by what is known as the volumetric heat transfer coefficient,  $h_v$ . Our studies indicate that the volumetric heat transfer coefficient is highly dependent on material type, gas flow rate and stage of decomposition.

As the pyrolysis reactions and expansion proceed, the permeability and porosity of the material increase still further. This results in increased gas flow and in a reduction in internal pressure. When this occurs, the material experiences a rapid contraction, primarily due to elastic recovery, followed by a slower contraction which is characterized by the linear coefficient of thermal expansion of the char,  $\alpha_c$ .

At temperatures in excess of 1000°C the carbon based residue remaining from pyrolysis reacts with the silica, present in the glass reinforcement, resulting in considerable additional mass loss. However, no net expansion occurs due to high material permeability and porosity. Deviations from

local-thermal equilibrium may become quite large at this time due to high gas flow rates, limiting the rate of product gas storage.

As with the pyrolysis reactions, the carbon-silica reactions cause significant changes in the thermal, physical and mechanical properties of the material. The carbon-silica reactions have a very adverse effect on the structural integrity of the material as a result of the degradation of the glass reinforcement. In fact, as a result of this degradative process the material exhibits a very severe contraction. Given sufficient incident energy, the active material may entirely be consumed. The final products may include a combination of solid phase silicon carbide and product gases or an entirely gaseous phase.

## B. SUMMARY OF THE MOST IMPORTANT RESULTS

An analytical and experimental investigation has been performed to determine the thermal behavior of two glass-filled, decomposing polymer composites. A one-dimensional, transient numerical model has been developed to predict the overall thermally-induced response of these materials, without including the idealized assumption of local-thermal equilibrium. For both materials, the expansion characteristics and volumetric heat transfer coefficients were experimentally determined for input into the numerical model.

The thermochemical expansion of two similar glass-filled polymer composites has been measured using high-temperature dilatometry. The expansion characteristics of material H41W were measured from 20 to 2000°C. The expansion characteristics of material MXBE-350 were measured to only 1600°C due to its highly reactive nature at high temperatures. For both materials, measurements were conducted at heating rates of 5, 10, 20 and 50°C/min. In both cases, the expansion characteristics have been found to be highly dependent on temperature and heating rate. Both materials exhibit peak expansion during the pyrolysis reactions, while undergoing severe contraction as a result of the carbon-silica reactions. However, the expansion curves for these materials differ both in magnitude and shape. Demonstrated is the fact that even seemingly small differences in materials and their method of preparation have a dramatic effect upon the observed expansion behavior.

The expansion characteristics of the two materials have been successfully modeled using a modified expansion model. This model predicts the expansion behavior quite well, even with the use of average parameters over the heating rates of interest. The use of average parameters greatly simplifies the numerical scheme for the prediction of the overall thermally-induced response of these materials.

An experimental apparatus was designed and constructed to determine the volumetric heat transfer coefficient in these materials. Volumetric heat transfer coefficient data are required for numerical schemes which predict

the thermal behavior of these types of materials in the absence of local-thermal equilibrium. For both materials, the volumetric heat transfer coefficient as a function of stage of decomposition and gas flow rate/pressure drop has been evaluated. For each material,  $h_v$  increases with increasing gas flow rate at each stage of decomposition. For a given pressure drop,  $h_v$  is seen to be greater in H41N than in MXBE-350.

The volumetric heat transfer coefficient data have been correlated in the form of a Nusselt number as a function of the Reynolds number for each material. The characteristic length parameter in both dimensionless groups is the square root of the permeability,  $\sqrt{\gamma}$ . A general correlation is also presented which includes the entire body of volumetric heat transfer coefficient data. It is thought that this general correlation is sufficiently accurate to be used to obtain estimates of expected volumetric heat transfer coefficients in similar composite matrices, especially those with flow fields within the experimental range of Reynolds numbers investigated,  $3.2 \times 10^{-6} \leq Re \sqrt{\gamma} \leq 7.4 \times 10^{-2}$ .

The results of the numerical study with a prescribed surface heat flux of  $279.7 \text{ kW/m}^2$  indicate that local-thermal equilibrium does not exist in either H41N or MXBE-350. In fact, solid to gas temperature differences of  $500^\circ\text{C}$  exist locally for MXBE-350. While for the tighter, more fibrous H41N differences as high as  $200^\circ\text{C}$  are predicted locally for the times investigated. For H41N, pressures in excess of 10 atmospheres were predicted, and the existence of such high pressures has been verified experimentally.

An investigation into the effects of the idealized assumption of local-thermal equilibrium revealed significant errors associated with the assumption for both materials. Solid temperature differences between the actual and idealized cases were as high as  $100^\circ\text{C}$  and  $180^\circ\text{C}$  locally for H41N and MXBE-350, respectively. As a result, measurable errors are evident in the mass loss and pressure profiles for the idealized case. The errors in the pressure and mass loss profiles are significant because spallation and/or cracking are highly dependent on stress concentration and material strength, which are linked to internal pressure and stage of decomposition,

respectively. From the results of this study, it is evident that accurate numerical modeling can only be achieved when the idealized assumption of local-thermal equilibrium is not incorporated into the modeling scheme.

On exercising the model to investigate the effects of the high-temperature carbon-silica reactions on the thermal behavior of the materials, a surface heat flux of  $2797.0 \text{ kW/m}^2$  was prescribed. Results indicate severe deviations from local-thermal equilibrium for both materials. In fact, for H41N and MXBE-350 solid to gas temperature differences as high as  $800^\circ\text{C}$  and  $1400^\circ\text{C}$ , respectively, are predicted locally. These deviations are about 4 and 2.8 times greater than those predicted for the lower heating rate for H41N and MXBE-350, respectively. From these results it is clear that the errors associated with the idealized case would be far greater for this high surface heat flux. As a result of the carbon-silica reactions both materials exhibit a significant increase in the porosity, a decrease in the solid mass to approximately 50% of the original value and a corresponding severe contraction.

C. LIST OF ALL PUBLICATIONS

"Measurement of the Thermochemical Expansion of Porous Composite Materials," Florio, Jr., J., Henderson, J.B. and F.L. Test, Proceedings of the 11th European Thermophysical Properties Conference, Umea, Sweden, June, 1988.

"Measurement of the Thermochemical Expansion of Porous Composite Materials," Florio, Jr., J., Henderson, J.B. and F.L. Test, High Temperatures - High Pressures, Vol. 21, 1989.

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"Internal Pressure and Temperature Distribution in Decomposing Polymer Composites," Ramamurthy, H., Test, F.L., Florio, JR., J. and J.B. Henderson, accepted for the Proceedings of the Ninth International Heat Transfer Conference, Jerusalem, Israel, August, 1990.

"An Analytical Investigation of the High Temperature Thermal Response of Decomposing, Glass-Filled Polymer Composites," Florio, Jr., J. and J.B. Henderson, Journal of Thermophysics and Heat Transfer (accepted).

"A Study of the Effects of the Assumption of Local-Thermal Equilibrium on the Overall Thermally-Induced Response of a Decomposing, Glass-Filled Polymer Composite," Florio, Jr., J. and J.B. Henderson, submitted to the International Journal of Heat and Mass Transfer.

"Characterization of Forced Convection Heat Transfer in Decomposing, Glass-Filled Polymer Composites," Florio, Jr., J., Henderson, J.B., Test, F.L. and H. Ramamurthy, submitted to the Transactions of ASME Journal of Heat Transfer.

"An Analytical and Experimental Investigation of the Decomposition of Glass-Filled Polymer Composites," Florio, Jr., J., Ph.D. Dissertation, Mechanical Engineering and Applied Mechanics, University of Rhode Island, Kingston, RI, USA, 1989.

**D. LIST OF SCIENTIFIC PERSONNEL**

Name	Degree
John Florio, Jr.	Doctor of Philosophy in Mechanical Engineering and Applied Mechanics

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