THERMAL PRESSURE CONTROL FOR MANAGING RESIN VOLATILITY DURING RTM

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Introduction

We consider resin transfer molding (RTM) using a benzoxazine/epoxy blend, an experimental resin that requires a two-stage cure cycle to prevent the formation of volatile-induced surface porosity. The resin contains residual solvent [1], which cannot be fully removed by vacuum-degassing, but can be held in solution with ≥200 kPa of hydrostatic pressure during cure. If only a single-stage cure cycle is used, in which the temperature is ramped directly to the final cure temperature (185°C), cure shrinkage causes a loss of cavity pressure, and colder regions in the mold cavity (where the cure lags, even by a small amount) become susceptible to volatile-induced void growth [2]. The pressure drop cannot be prevented by external hydrostatic resin pressure through the inlet line, once the resin in that location gels. Conversely, if the resin within the mold is gelled at a lower temperature, the cavity pressure becomes dependent on resin volumetric changes, and a temperature ramp can be used to maintain cavity pressure by exploiting the effect of thermal expansion to temporarily counteract cure shrinkage.

In this study, we measure resin cure shrinkage and thermal expansion using an in-house built piston-type dilatometer, and develop a model for mold cavity pressure based on thermal and chemical density changes. The model is used to predict cure cycles that delay the (inevitable) pressure drop until the resin has reached a higher degree of cure, where volatile release is no longer possible. To validate the utility and predictive capabilities of the model, the timing and extent of porosity formation are compared, for the baseline and improved cure cycles, through manufacturing trials with a lab-scale RTM that contains a glass tool plate to enable *in situ* observations of porosity formation.

Hybrid RTM/Dilatometer

We describe the design of a new experimental tool that mimics the RTM process, with the added capability of varying the cavity thickness to maintain constant pressure in the presence of resin chemical and thermal strains. This piston/cylinder-type hybrid RTM/dilatometer is mounted in a mechanical test frame (Instron 8858, in load-control mode), features temperature and pressure sensors, and uses an extremely precise non-contact displacement sensor to monitor the mold cavity thickness. Through measurement of variations in part thickness, the resin volumetric strains are quantified, which in turn enables prediction of mold cavity pressure for the traditional (constant-volume) RTM process. The concept was largely inspired by a similar device described by Boyard et al. [3], although ours contains modifications to more closely resemble RTM (e.g. ports for resin injection). Furthermore, by modulating the pressure imposed by the load frame, the relationship between resin volumetric strains and mold cavity pressure is quantified.

Process Modeling and Optimization

Using a combination of traditional thermochemical characterization techniques (calorimetry, rheology, etc.) and the hybrid RTM/dilatometer, we are developing a model for the RTM process that predicts the occurrence of volatile-induced porosity formation. The model includes a one-dimensional temperature and degree of cure simulation (to capture the effects of a through-thickness temperature gradient), and computes the evolution of the part thickness due to chemical and thermal strains (i.e. the "constant-pressure sample thickness"). By comparison of the difference between the constant-pressure sample thickness and the thickness of mold cavity (in a constant-volume RTM tool), the variations in pressure are predicted. Finally, the model predicts the timing, location, and severity of volatile-induced porosity formation by applying previously-determined volatile release criteria [2]. If the pressure falls below the "critical pressure" of 200 kPa, before the resin has reached the "critical degree of cure" that

prevents volatile release (near vitrification), porosity formation can be expected to occur at that time and location.

The process model is used to develop optimized cure cycles that minimize both porosity formation and total cycle time. The model acts as an objective function, whose inputs are the parameters defining a two-stage cure cycle (time and temperature of the intermediate dwell and ramp rate to the final dwell), and whose outputs are the total cycle time and the predicted extent of porosity formation. An optimization procedure is used to find cure temperature cycles that minimize these outputs, and a "Pareto front" is generated to show the trade-offs between these (conflicting) objectives based on varying relative weight factors. Finally, the utility of this approach is demonstrated by fabricating RTM parts in a constant-volume molding tool using the baseline and optimized cure cycles, and comparing the quality of molded samples.

Altogether, this work provides a practical and applied approach to improving RTM manufacturing practices. We aim to bridge the gap between industrial-scale manufacturing and fundamental material characterization by conducting industrial processes at the lab-scale using highly-instrumented custom-designed tools. By these means we gain fundamental understanding of the physical phenomena relevant to volatile-induced defect/void formation, and then leverage that understanding to develop optimized procedures for reliable, robust, and efficient composite fabrication.

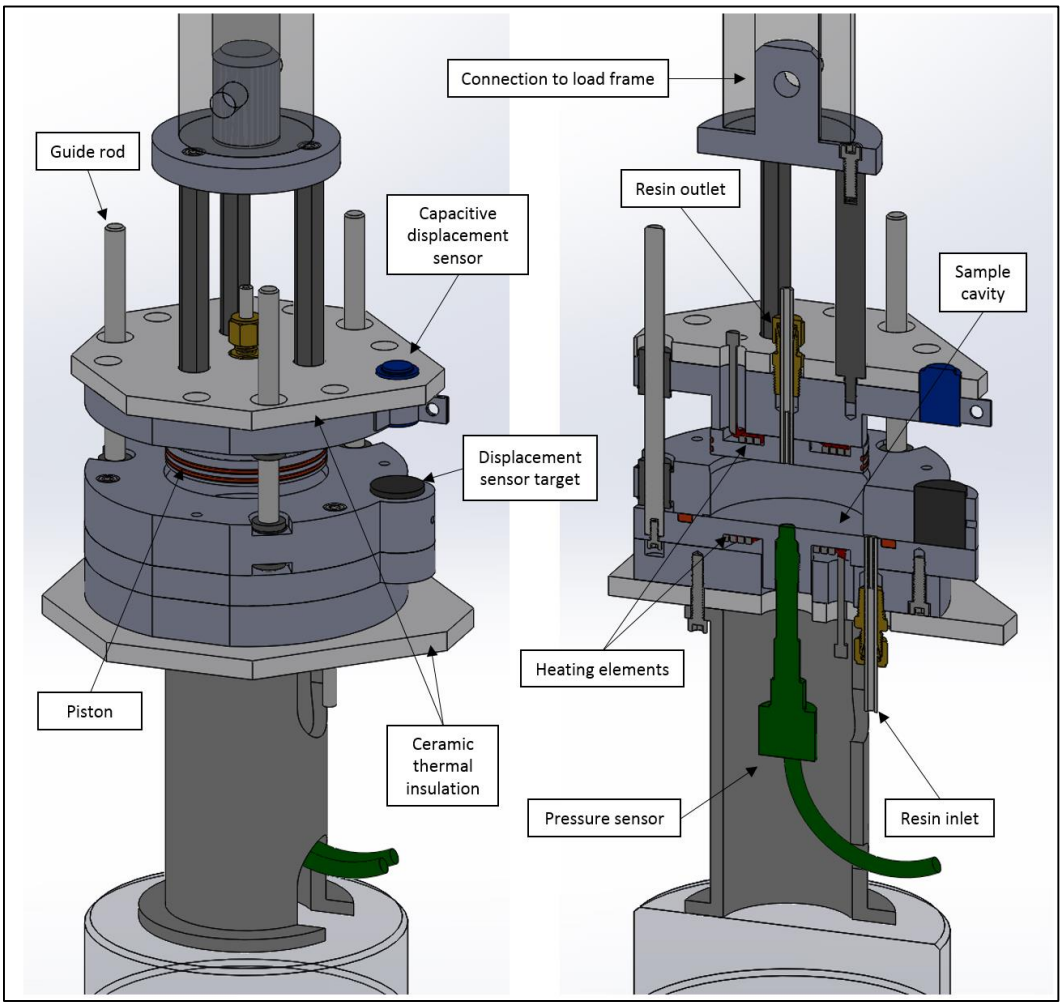

Figure 1: *CAD model of the hybrid RTM/dilatometer tool.*

References

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