ENHANCED VISCOSITY CHARACTERIZATION FOR FAST CURING EPOXY RESINS AT PROCESS RELEVANT TEMPERATURES

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Introduction

Large batch production of high performance composite parts is still a huge challenge. Resin transfer molding (RTM) or wet compression molding (WCM) have the potential to change this by using fast-curing resins. Due to their very short curing time, fast-curing resins enable rapid processing, but in turn demand a comprehensive process knowledge and well-matched process parameters. In order to find optimal process parameters, numerical simulations are helpful and avoid the need for expensive experimental trials.

RTM and WCM mold filling strongly depend on the viscosity of the resin, which itself is affected by the cross-linking reaction and the process temperature. If a constant mass flow is applied at the inlet, inaccurate viscosity data can lead to significant errors in the prediction of the cavity pressure. This complicates the simulation-driven design of recently developed pressure-controlled RTM process variants [1,2]. For mold filling simulation, it is therefore vital to have accurate viscosity data. However, established measurement and sample preparation techniques are not specifically designed to capture viscosity data during the first seconds of the reaction, which is crucial in case of fast curing resins at process relevant temperatures. The main reason for this shortcoming is the specimen preparation and gap adjustment. In this study, a new automated specimen preparation process is presented, which minimizes this time lag by directly injecting the resin into the measurement system. Subsequently, the results are used for mold filling simulation of a complex composite part.

Automated specimen preparation process

Rheological measurements typically involve a fully manual specimen preparation. Resin and hardener are dosed with the aid of a high-precision scale and subsequently mixed by hand. Afterwards, a small portion of the mixed resin is dosed onto the lower plate of the preheated parallel plate setup using a syringe. From this point on, the resin is in contact with the hot sample holders and consequently curing starts. This instantly affects the viscosity of the resin. Therefore, it is vital to minimize the time between first contact of the specimen with the hot sample holders and the start of the measurement. Reducing this period of time in a fully manual process significantly below 30 seconds is hardly possible, mainly due to the need of time-consuming gap adjustments. However, in about the same period of time, mold filling needs to be completed in a competitive large batch production. Therefore, a faster sample preparation process is needed in order to obtain meaningful viscosity data. In this study, this is achieved by using a lab-scale dosing and mixing unit attached to a rheometer as shown in Figure 1. This setup enables a significant reduction in preparation time to values lower than 10 seconds.

In order to evaluate the specimen quality of the automated process, Differential Scanning Calorimetry (DSC) measurements are analyzed in terms of total heat of reaction and glass transition temperature at the end of the cure cycle. The results show similar values for the manual and automated preparation processes.

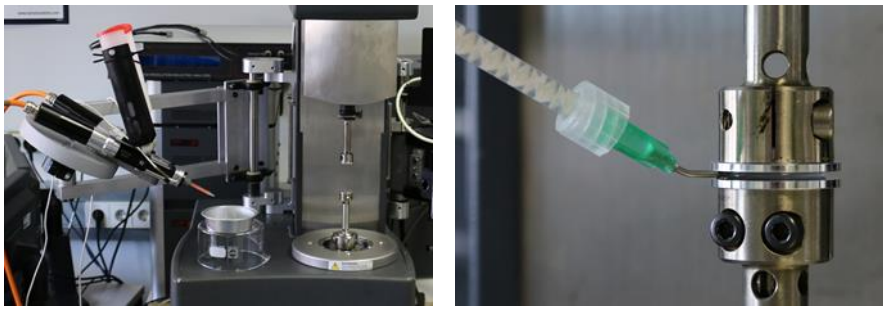

Figure 1: *Automated specimen preparation setup: dosing and mixing unit attached to rheometer (left) and injection of the resin into the gap of the preheated parallel plate setup (right).*

Mold filling and curing simulation of a complex composite part

The influence of the viscosity on the cavity pressure during mold filling is demonstrated by comparing results of two isothermal IRTM simulations. One simulation uses a constant viscosity whereas the other one considers cure and temperature-dependent viscosity (non-constant). In both cases, curing takes place at 110 °C and the resin is injected with a constant mass flow. For the simulation with constant viscosity, the mean value of the viscosity of the non-constant simulation is used. The numerical method is based on a compressible two-phase finite volume solver [3], which is extended in order to take into account the chemo-rheology of the resin. The latter is modeled using the reaction kinetics model of Grindling [4] and the chemo-rheology model of Castro and Macosko [5]. Figure 2 shows the temporal evolution of filling degree, viscosity and pressure at the inlet for both simulations. Although the viscosity of the non-constant simulation exceeds the mean viscosity value of the constant simulation at about 18 s, the pressure remains at a much lower level and does not significantly increase until the end of mold filling. Reasons for this are the complex geometry of the part and the structure of the preform, which consists of several sub-preforms and therefore provokes the formation of local runners. The assumption of a constant viscosity based on the mean value is thus invalid in case of complex composite structures and should be avoided, especially if pressure values are to be used to control the process.

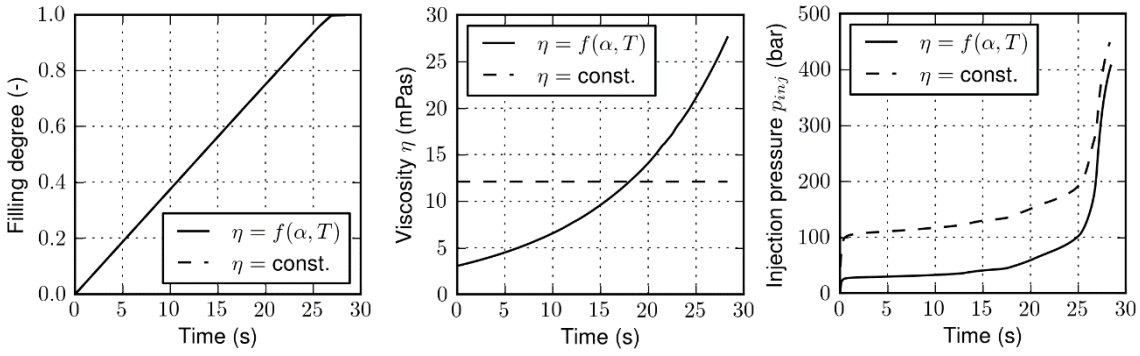

Figure 2: *Temporal evolution of filling degree, viscosity and pressure of mold filling simulations.*

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