APPLICATION NOTE

Testing the flow behavior of ceramic injection molding compounds

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Introduction

Powder injection molding (PIM), comprising metal injection molding (MIM) and ceramic injection molding (CIM), is an advanced manufacturing technology for complex, high-volume precision components. An example is the automobile industry's current evaluation of ceramic turbine blades with respect to engine heat for the development of more fuel-efficient automobiles.

CIM feedstocks are compounds of ceramic powder and a polymeric binder. In addition to the polymeric filler, molecular weight and the molecular weight distribution also influence the flow characteristics of the final feedstock. To achieve high-precision end products, manufacturers using PIM must determine the material characteristics of CIM feedstocks prior to processing.

Problem statement

A melt flow index (MFI) tester and a mixer sensor were used to characterize two different CIM feedstocks. Although this characterization showed no differences between the two batches, final parts made from these two feedstocks showed significant differences in mechanical properties. Obviously, there were differences between the two feedstocks.

The goal of this subsequent test was to differentiate between the two CIM compound samples using a torque rheometer with appropriate hardware and software accessories. The extruder capillary rheometer tests provide flow curves which can be examined to see what is different between the two samples.

Test equipment

Thermo Scientific™ HAAKE™ PolyLab™ OS Torque Rheometer system including:
- Thermo Scientific™ RheoDrive™ Software with Thermo Scientific™ Rheomex Single Screw Extruder 19/25
- Extruder screw with a compression ratio of 2:1
- Rod capillary die with a diameter of d=1.50 mm and length of l=30 mm
- Thermo Scientific™ PolySoft Capillary Rheology Software
- Circulator SC150-A10 to control temperature in feed zone of the extruder

The capillary rheometer is uniquely suited for the measurement of shear viscosity at process-relevant shear rates. The HAAKE PolyLab OS System can measure rheological characteristics under actual process conditions when used in conjunction with an extruder sensor and appropriate rheological capillary die. In addition, the PolySoft OS Capillary Rheometry software enables the process by helping users to pre-define test procedures, running measurements automatically, performing necessary corrections and allowing regression analysis for modeling of flow channels and molds.

Test conditions

Extruder temperatures:
· Feed zone: 20 °C
· 1st zone: 95 °C
· 2nd zone: 110 °C
· 3rd zone: 135 °C
Die temperature: 135 °C

Test method

To characterize the flow behavior of each CIM feedstock, a sample from each batch was plasticized in the extruder. Then it was transported to the die and pressed through the capillary. Different shear-rates were obtained by setting different extruder screw speeds.

The melt pressure in front of the capillary was measured using the pressure transducer. The connected balance automatically measured the corresponding melt output. After reaching constant pressure and melt throughput values, the data was recorded for calculation of rheological values. The software automatically triggered the subsequent drive speed that was set. Shear rate and viscosity were calculated from the measured data and displayed in a viscosity curve. The software automatically applied the necessary rheological corrections according to Bagley and Weissenberg/Rabinowitsch.

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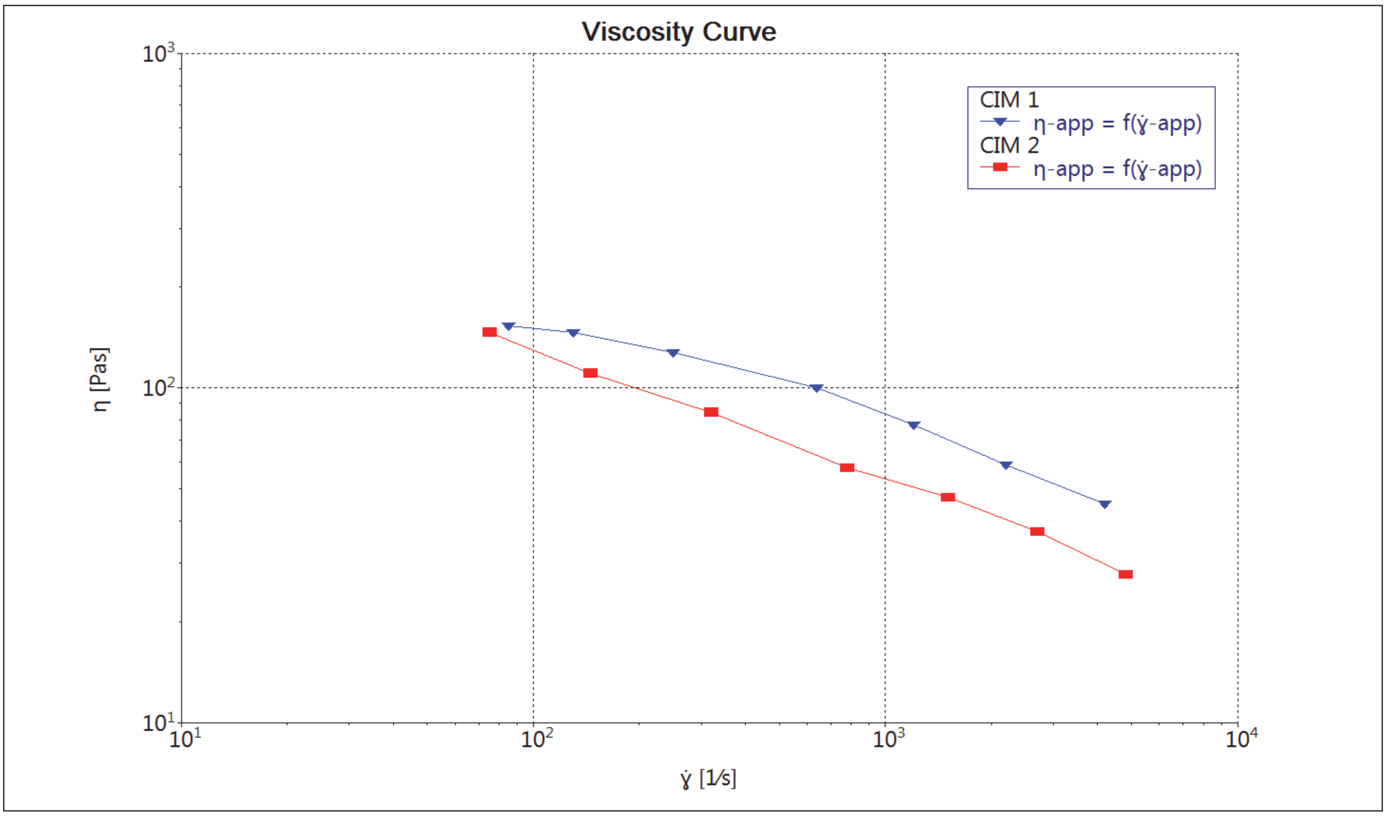

Graph 1: Viscosity curve of two different CIM feedstocks (CIM1 and CIM2).

Test results

The viscosity curve (Graph 1) shows the viscosity measurements of both batches of CIM feedstock. The graph displays the shear stress τ and the viscosity η above the shear rate γ. The binder for the two thermoplastic ceramic feedstocks was based on a polyolefin, so the curves show a shear thinning typical for polymers, i.e., a decreasing of viscosity at an increasing shear rate. Feedstock 2 (CIM2) exhibited more shear thinning than feedstock 1 (CIM1). The measured values are very similar at shear rates < 100 sec^{-1}, but they differ at higher shear rates. The viscosity value at 1000 sec^{-1} for feedstock 1, for example, is 42% higher than for feedstock 2.

Conclusion

The viscosity measurements explain why both feedstocks couldn't be differentiated with an MFI tester and a mixer sensor. Shear rates for that testing setup are normally below 100 sec^{-1} where both feedstocks looked similar. The extruder capillary rheology test allowed measurement at higher shear rates, so it was more comparable to what actually occurred in the extrusion process or during injection molding. The results show that feedstock 1 could exhibit problems and not fill the mold completely, or feedstock 2 (with too low viscosity) may not achieve the necessary form stability.

The extruder capillary rheology system delivered the right data needed to properly assess material properties for manufacturing complex high-volume precision components.

Summary

The HAAKE PolyLab OS system equipped with a capillary rheology measuring set-up provided fast, reliable testing for the flow properties of ceramic feedstock under conditions similar to those in manufacturing. Important material characteristics could be seen, which were not visible with simple MFI testing. That means the HAAKE PolyLab OS system can save time in manufacturing and ensure quality by establishing an automated, standardized test routine. The PolySoft Capillary Rheology Software can be tailored to run a test program suited for many different applications.

Find out more at **thermofisher.com/extruders**

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