Temperature Dependent Flow Properties of Powder Injection Moulding Compounds

Tomas Honek*, Berenika Hausnerova and Petr Saha

[1] Tomas Bata University in Zlin, Faculty of Technology, TGM 275, 762 72 Zlin, Czech Republic

*Email: honek@ft.utb.cz
Fax: x420.67.7541444

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Abstract:
The temperature dependent flow properties of highly filled polymer compounds intended for production of hard-metal parts by powder injection moulding (PIM) technology were studied. The pure binder based on polyethylene, ethylene and butyl acrylate block copolymer and paraffin, and its compounds with hard-metal carbide powder (up to 55 vol. %) were prepared by melt mixing at 180°C. The flow properties were investigated at the temperature range from 140°C to 200°C using capillary rheometer operating flow at a constant piston speed. The measure of temperature sensitivity of PIM compounds, activation energy of shear flow, decreases with powder loading and shear rate. The Arrhenius relation for these materials is only valid in the stable flow region. At the temperatures above 170°C the compounds filled with 45 vol. % carbide powder and higher exhibit an unstable flow of pressure oscillations type at the shear rates above 10^3 s^{-1}. The onset of pressure oscillations is strongly affected by temperature. The relation between critical shear stress for the onset of pressure oscillations and temperature is non-linear.

Zusammenfassung:
Die temperaturabhängigen Fliesseigenschaften von hochgefüllten Polymer compounds, die bei der Produktion von Hartmetallbauteilen mittels der Powder Injection Moulding-Technlogie (PIM) eingesetzt werden, wurden untersucht. Der reine Binder basierend auf Polyethylen, Ethylen und Butylacrylate Blockcopolymeren und Paraffin. Diesem Ausgangsprodukt wurde dann bei 180°C bis zu 55 vol. % HartmetallKarbidpulver beigemischt. Die Fliesseigenschaften wurden im Temperaturbereich von 140°C bis 200°C mittels eines Kapillarrheometers mit konstanter Stempelgeschwindigkeit untersucht. Die Scherströmungsaktivierungsenergie, als Mass für die Temperaturempfindlichkeit der PIM-Verbindungen, erniedrigt sich sowohl mit dem Pulvergehalt und der Scherströmung. Die Arrhenius-Beziehung ist für diese Materialien nur im Bereich stabiler Strömung gültig. Bei Temperaturen oberhalb 170°C zeigen die Verbindungen mit 45 vol. % und mehr Karbidpulver bei Scherraten oberhalb 10^3 s^{-1} eine instabile Strömung in der Form von Druckoszillationen. Das Einsetzen der Druckoszillationen ist stark von der Temperatur abhängig. Der Zusammenhang zwischen der für das Auftreten der Druckoszillationen kritischen Scherspannung und der Temperatur ist nicht-linear.

Résumé:
La dépendance en température des propriétés d'écoulement de polymères hautement chargés, composés entrant en jeu dans la production de pièces métalliques dures par injection-moulage de poudres (PIM), a été étudiée. Le liant pur, basé sur du polyéthylène, du copolymère block éthylène-buthyl acrylate et de la paraffine, et ses mélanges avec de la poudre métallique (jusqu'à 55% vol) ont été préparés en mélangeant à 180 °C. Les propriétés ont été étudiées dans une fourchette de températures allant de 140 °C à 200 °C à l'aide d'un rhéomètre capillaire opérant à vitesse de piston constante. L'énergie d'activation de l'écoulement de cisaillement décroît lorsque la fraction volumique en poudre augmente, et quand la vitesse de cisaillement augmente. La relation d'Arrhenius pour ces matériaux n'est valide que dans la région d'écoulement stable. A des températures supérieures à 170 °C, les composés chargés avec 45 % vol. de poudre et plus, exhibent un écoulement instable à des vitesses de cisaillement supérieures à 10^3 s^{-1}. Cette instabilité est caractérisée par des oscillations dans la pression. L'apparition des oscillations de pression est fortement affectée par la température. La relation entre la contrainte de cisaillement critique correspondant à l'apparition des oscillations de la pression, et la température est non-linéaire.

Key words: PIM technology, highly filled compounds, flow instabilities, temperature effect

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greater in the loaded system, due to the superimposed change in volume fraction of solids associated with the difference in thermal expansion coefficients. Shenoy [22], on the other hand, suggests an opposite dependence of the activation energy on the filler content. His explanation is that the viscosity of filled system is less temperature sensitive compared to the viscosity of pure polymeric binder, which is obviously true because the fillers provide very little free volume change with temperature in relation to the binder. This idea was also confirmed by the work of Liang and Li [23].

The data from our investigations confirm the Shenoy [22] idea that the activation energy of shear flow decreases with increasing filler content against the German's [1] suggestion, which offer the idea of increasing activation energy with rising filler content.

4 CONCLUSION

It has been observed that the flow properties of PIM compounds are strongly temperature dependent - with increasing temperature the shear viscosity decreases. This dependence in less pronounced as volume fraction of solids in the compound increases. The activation energy of shear flow is a decreasing function of both the volume fraction of hard-metal carbide powder and the shear rate. PIM compounds follow the Arrhenius relation in the stable part of their flow curves only.

Temperature and loading were found to be the key factors limiting the onset of the flow instabilities of pressure oscillations type observed for compounds containing 45, 50 and 55 % hard-metal carbide powder. The critical shear stress at the onset of pressure oscillations is shifted toward higher values as the temperature decreases; the dependence is non-linear.

Two different types of temperature dependence of the pressure oscillations amplitude have been found. While for the compounds filled with 50 % carbide powder the amplitude of pressure oscillations increases with rising temperature, the opposite trend can be observed for 55 % compounds.

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