Effect of Particle Size Distribution on the Flow Behaviour of Powder Injection Moulding Materials

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Abstract: Experimental data relating to oscillatory flow of hard-metal carbide powders compounded with a polymeric binder are presented. Such powder/binder mixtures are used for the production of sintered cemented carbide components via powder injection moulding. The binder composition tailored to a carbide powder consisted of polyethylene, ethylene/acrylic acid copolymer, and paraffin wax. Four kinds of hard-metal carbide powders differing in their particle size distribution were considered to investigate the structural changes during shear deformation. The obtained results reveal that particle size distribution of powder has a considerable influence on viscoelastic properties of highly filled compounds.

Key-Words: cemented carbide powder, particle size distribution, polymeric binder, oscillatory flow

1 Introduction

Currently a wide range of metal and ceramic powders and their alloys are used for sintered parts production via powder injection moulding (PIM). The method represents an effective alternative to machining for the production of small-size complex-shape parts, because of material saving (no scarp technique). PIM parts find their applications in automotive, medical, aerospace, mechanical, IT, and other industrial sectors.

At the start of the process the powder is mixed with a suitable polymer-based binder into a homogeneous compound. Such compound is then formed by injection moulding into so called green part. This stage is followed by debinding, where the binder is extracted and compact is sintered to the final density.

PIM is a multidisciplinary method, where polymer processing techniques are combined with metallurgical approaches. The requirements arising from the particular steps of the process are often contradictory.

This stands in the first place for the characteristic of powder, which is always a compromise among various demands. Spherical or rounded shape brings suitable viscosity during mixing and moulding, but represents difficulties with shape retention of the product during debinding and initial stages of sintering. Particle size of PIM varies between 0.1 and 20 μm [1], which is desirable for rapid sintering, but on the other hand such powders tend to agglomerate, and therefore bring necessity of high shear mixing. Fine powders also improve surface finish as compared to coarser ones.

Ideal powder should combine large and small particles in a tailored particle size distribution, providing high packing, and simultaneously suitable viscosity during moulding. A broad particle size distribution means also less sintering shrinkage, but it causes slower debinding [2].

2 Problem Formulation

PIM compounds represent highly filled materials (close to the maximum packing), and thus their processing is often limited by unacceptable high viscosity. It has been demonstrated, e.g. [3] that high solids loading with a suitable viscosity can be attained using powders having a distribution of particle sizes rather than particles of similar sizes. Obviously, particle size distribution plays a key role among the powder parameters influencing their rheological properties. In our previous papers [4,5] we studied the effect of particle size distribution on a capillary flow (high shear rates) with regard to powder packing limits [4] and temperature and pressure sensitivity [5] of PIM materials. Nevertheless, the processability of PIM compounds is determined not only by the viscous properties, but also by their elasticity. Therefore, the aim of this work is to measure their dynamic viscoelastic properties in order to obtain information on the elastic parameters and distinguish whether the effect of particle size distribution plays a significant role in the nature of the internal structure and its reformation for PIM compounds.

3 Problem Solution

3.1 Experimental

Viscoelastic properties in terms of storage and loss moduli and complex viscosity were measured with plate-plate rotational rheometer (ARES, Rheometrics, USA) equipped with RSI orchestrator software package. Plate diameter was 25 mm, gap between plates was kept 1 mm, angular frequency ranged from 0.1 to 100 rad/s at 140, 150 and 160 °C.

As a model experimental material cemented carbide powders were compounded with a three-component binder in order to prepare 30, 40, 45, 50 and 55 vol. % feedstocks.

The powders employed in the experiments were composites of tungsten carbide and cobalt (cemented carbides) supplied by Sylvania Tungsten, Czech Republic. Four grades were tested - BC10U, BC17S, BC37S, and BC75H - differing in their particle size distributions as follows:

BC10U:	Diameter at 10%:	0.45 μm
	Diameter at 50%:	1.11 μm
	Diameter at 90%:	3.75 μm
	Average diameter:	1.24 μm
BC17S:	Diameter at 10%:	0.43 μm
	Diameter at 50%:	1.36 μm
	Diameter at 90%:	2.71 μm
	Average diameter:	1.24 μm
BC37S:	Diameter at 10%:	1.04 μm
	Diameter at 50%:	3.80 μm
	Diameter at 90%:	7.14 μm
	Average diameter:	3.32 μm
BC75H:	Diameter at 10%:	2.19 μm
	Diameter at 50%:	15.38 μm
	Diameter at 90%:	28.06 μm
	Average diameter:	7.38 μm

3.2 Results & Discussion

In contrast to the behaviour of pure polymers, the rheological properties of filled systems are highly strain dependent even at low strain amplitudes. Strain sweep tests were carried for strains ranging from 0.1 to 100 % at angular frequencies of 0.37, 3.7 and 37 rad/s, and 1 % strain was selected as a representative of linear viscoelastic region.

Viscoelastic properties of compounds represented with complex viscosity reveal that BC10U feedstock (Fig.1) responses to dynamic shear similarly to BC17S for all temperatures measured. These two powders had rather similar particle size distributions – the mean diameter was identical, the portion of small particles very similar, although BC10U had higher portion of larger particles. Further, viscosity of 30 vol. % BC10U feedstock is considerably lower than others, while 50 a 55 % BC10U can be hardly distinguished from each other as well as from the data obtained for BC17S.

The compounds based on BC75H (Fig.2) showed the lowest (about three orders of magnitude) values for 50 a 55 % concentrations among all powders tested regardless of temperature used. Again, it is in accordance with the particle size distribution of this powder represented by large particles, without a portion of small particles (diameter at 10% for BC75H was 2.19 μm, while for BC10U 0.45 μm) prone to agglomerate.

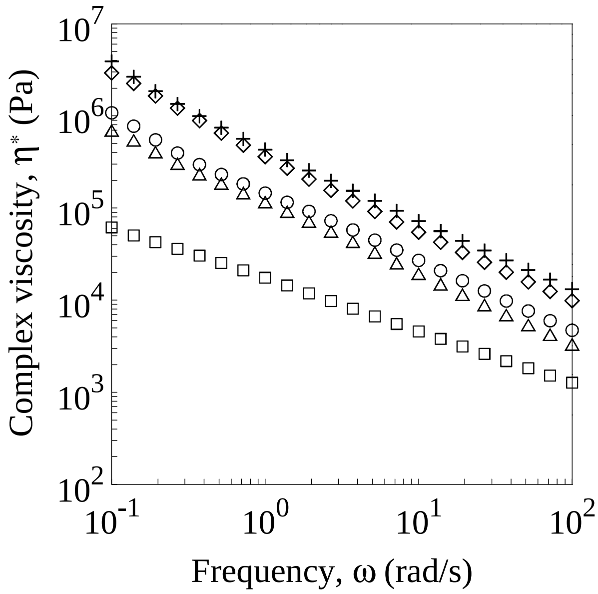

Fig. 1 – Complex viscosity vs. frequency of BC10U compounds containing 30(□), 40(△), 45(○), 50(◇), and 55(+) vol. % of carbide

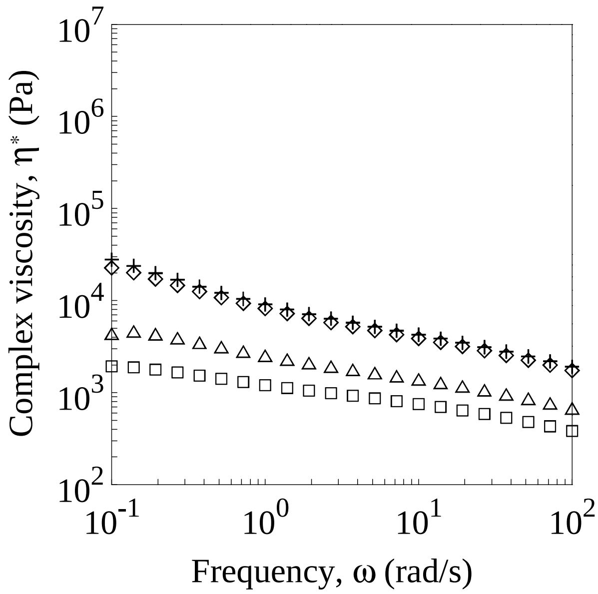

Fig. 2 – Complex viscosity vs. frequency of BC75H compounds containing 30(□), 40(△), 50(◇), and 55(+) vol. % of carbide powder.

Concerning BC37S samples, their complex viscosity values were determined between those of BC10U (BC17S) and BC75H as its particle size distribution also fits in between.

For storage modulus, similar findings were done. Generally, for unfilled polymer melts storage modulus decreases to zero when the frequency is lowered sufficiently; it becomes independent of the frequency as powder is loaded. The powder enables the compound to store more energy elastically and also to dissipate more energy [6]. Only BC10U and BC17S samples exhibited plateau on their storage modulus vs. angular frequency curves for powder loadings above 30 % and frequency ranging from 0.1 to 1 rad/s, thus remaining the behaviour typical for composites, while the feedstock based on BC75H reveal the modulus vs. angular frequency increasing with angular frequency for all loading levels as it is found for viscoelastic melts.

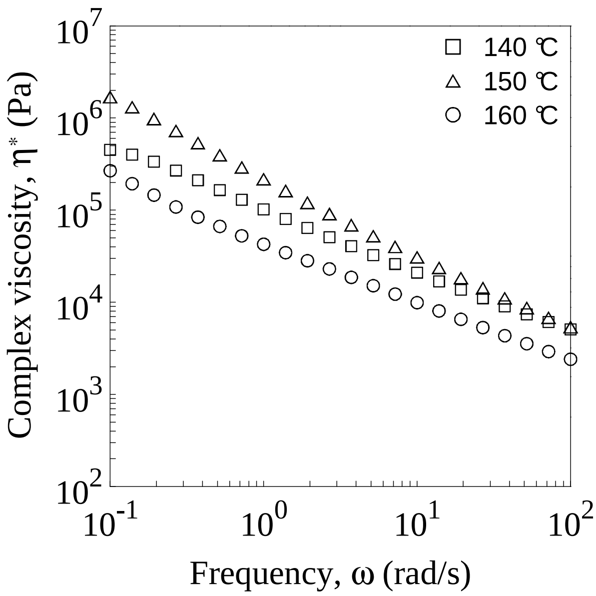

Fig. 3 – Complex viscosity vs. frequency of BC37S compounds containing 50 vol. % of carbide powder as a function of temperature.

Temperature effect in the range from 140 to 160 °C was pronounced only for BC37S compound containing 50 vol. % (Fig.3), for all other compositions it was not distinct (see Fig.4 as an example).

4 Conclusion

Flow properties of powder injection moulding (PIM) compounds are considerably influenced by the size and particle size distribution of a powder used. The compounds based on powders having a high portion of small particles tend to have higher viscoelastic functions, while behaviour of material containing large particles remains the course of a viscoelastic melt.

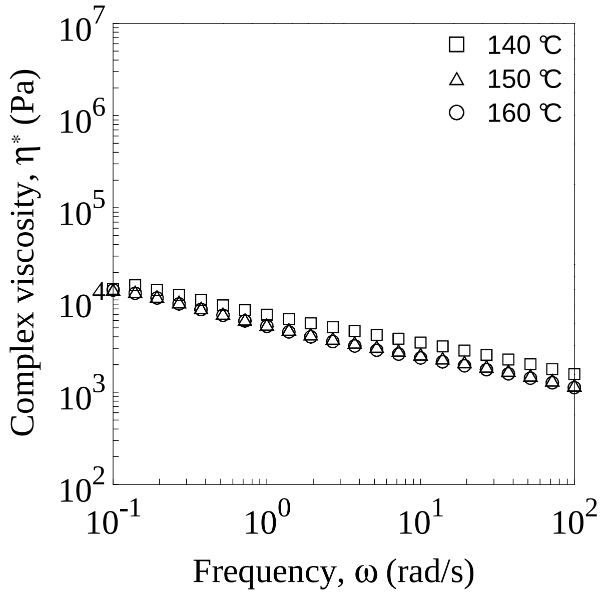

Fig. 4 – Complex viscosity vs. frequency of BC75H compounds containing 50 vol. % of carbide powder as a function of temperature.

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