On-Line Rheometry for Twin-Screw Extrusion (Along the Extruder) and its Applications

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Abstract:
Due to a number of practical difficulties, both in- and on-line measurements of the rheological properties of complex systems during extrusion are usually performed at the end of the extruder, under very specific experimental conditions. This makes this type of instruments more useful for quality control than for process optimisation, since information about the influence of the geometry and/or processing conditions on the evolution of the material characteristics inside the extruder is not easily gathered. Recently, however, the authors have developed an on-line capillary rheometry system that overcomes most of the existing problems and allows small amounts of sample to be tested in very near real time, along the extruder. The present work aims at illustrating the usefulness of this concept for the study of physical compounding processes and some reactive systems. Two very different systems will be used for that purpose: a reactive extrusion process (the peroxide-induced thermal degradation of polypropylene) and the dispersive mixing involved in the preparation of thermoplastic/carbon fibre composites.

Zusammenfassung:
Wegen einer ganzen Anzahl praktischer Schwierigkeiten werden sowohl in- als auch online Messungen der rheologischen Eigenschaften komplexer Systeme während der Extrusion normalerweise am Ende des Extruders unter sehr spezifischen experimentellen Bedingungen vorgenommen. Dieses Vorgehen macht Instrumente solcher Art mehr für die Qualitätskontrolle als für die Prozessoptimierung nützlich, zumal Information über den Einfluss der Geometrie und/oder Prozessbedingungen auf die Entwicklung der Materialcharakteristika innerhalb des Extruders nicht leicht zu erfassen ist. Kürzlich haben die Autoren ein on-line Kapillarrheometersystem entwickelt, welches die meisten der existierenden Probleme überwindet und es erlaubt, kleine Probenmengen in nahezu Echtzeitbedingungen entlang des Extruders zu testen. Die vorliegende Arbeit zielt darauf ab, die Nützlichkeit dieses Konzeptes für die Untersuchung des physikalischen Verbundprozesses an einigen reagierenden Systemen zu illustrieren. Zwei sehr verschiedene Systeme werden zu diesem Zweck verwendet: ein reaktiver Extrusionsprozess (die Peroxid-induzierte thermische Zersetzung von Polypropylen) und das dispersive Mischen, welches in der Herstellung von Thermoplast-Karbonfasern Kompositen auftritt.

Key Words: on-line rheometry, twin-screw extrusion, reactive processing, termoplastic/carbon-fibre composites

1 INTRODUCTION

Despite its obvious relevance for polymer processing operations, such as the possibility for process monitoring, quality control, process control or process optimisation (see, for example, [1]), in- and on-line measurements of the rheological properties of polymeric systems during processing have been hindered by a number of practical difficulties, specially those associated with accessing the flowing melt in order to make a measurement and with the testing conditions, e.g. required measuring time. In practice, on-line rheological tests are normally performed at the end of the extruder [2-4], after diverting a small amount of melt from the main stream. Most of these devices contain some form of capillary viscometer where the melt shear rate is controlled by means of descending pistons, as in regular off-line laboratory devices, or using gear pumps (see, for instance, Coates [3]).

section already have a *L/D* ratio that is too small for subsequent degradation to have an influence on fibre alignment (and, consequently, the flow properties of the composite). In this case the governing factor in the rheological behaviour is the total fibre area present in the system, which is constant, despite the fibres themselves getting shorter as the melt flows through the section. Alternatively (or, possibly, in addition), the adhesion between the fibres and the matrix may not be high enough to warrant significant differences in the shear behaviour of the composite between the beginning and the end of the mixing section.

Apart from shear flows, it is also relevant to study extension dominated flows, such as the one that occurs in the entry from the reservoir into the capillary, since the stresses involved in these flows are much higher than those involved in shear flows and the results will be probably more sensitive to changes in fibre length. An approximate measure of the extensional viscosity can be obtained from the entry pressure drop that occurs at the contraction (see, for example, the analyses of Cogswell [17] and Binding [18]). This can be done by means of the Bagley correction and the results, expressed as the torque corresponding to entry effects (M_O) versus wall shear rate, are shown in Fig. 8. It is now possible to discriminate the rheological behaviour at locations A and B, i.e. to identify fibre degradation in the composite, since the entry pressure drop for low shear rates (and, hence, lower degrees of alignment with the flow) is much higher at the beginning of the mixing section than at the end. At high shear rates, the fibres are already fully aligned and, thus, again, it is to be expected that fibre length will have little or no influence on the rheology of the composite.

In Fig. 8 there is a small but noticeable decrease in M_O between locations A and B of the virgin PP, which is in fact coherent with the slight difference in the shear viscosity at shear rates above 40 s^{-1} that can be perceived in Fig. 7. Although this could be partially due to experimental error, it could also be an indication that some degree of PP degradation is indeed occurring.

4.3 CONCLUSIONS

This section focused on the influence of the compounding conditions on the rheological properties of PP-carbon fibre composites. This was accomplished both via optical microscopy performed on samples collected along the extruder and on-line rheometry. Fibre degradation takes place as the material progresses along the screw and this influences the rheological properties of the composites. It was concluded that fibre degradation occurs predominantly during melting of the thermoplastic matrix, *i.e.*, at the beginning of the mixing section, this effect being so severe that virtually no differences in the rheological behaviour of the composites are observed in shear flow, after that, *i.e.* between the beginning and the end of the mixing section. It is only in extension dominated flows that differences become apparent, since these are more sensitive than shear flows to variations in morphology and, as thus, to fibre length. From the process point-of-view, this means that the processability of these materials will not change significantly, as long as the flow inside the extruder is shear dominated.

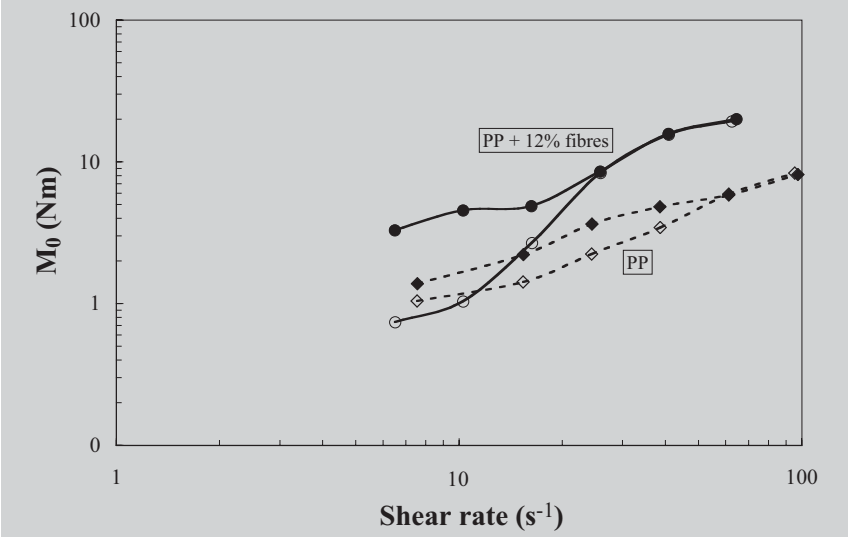

Figure 8: Torque corresponding to entry effects at beginning (full symbols) and end (open symbols) of mixing section for the same study of Figure 6.

5 GENERAL CONCLUSIONS

Since the rheological response of a mobile system can be correlated with its macromolecular structure and morphology, the possibility of carrying out rheological measurements along the barrel of a twin screw extruder provides an important tool for understanding the evolution of the morphology and chemical conversion of specific systems. Process control and optimisation can then be improved, and the experimental validation of modelling efforts carried out. The on-line capillary rheometer used in this work is capable of making quick measurements (in *circa* 15 seconds) of the shear viscosity within a limited shear rate range (typically between 1 and 500 s^{-1} [6]). Therefore, it can be of use to study physical compounding processes and selected reactive systems. Conversely, as with conventional off-line capillary rheometry, the induced

deformations will modify the morphology present in some systems, such as polymer blends, which constitutes an obstacle to the use of the rheometer in the study of this type of materials.

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