**CMMT(MN)041**

# Measuring The Extensional Flow Properties Of Polymer Melts Using Stretching Methods

## Summary

In many polymer-forming processes the polymer undergoes significant extensional or stretching flow, for example in blow moulding, vacuum forming and film extrusion. The extensional flow behaviour of materials will therefore have a significant effect on their processability and also on the properties of the moulded product. However, established rheological techniques predominantly characterise the behaviour of polymers in shear and it can prove difficult to relate the behaviour measured in shear with that observed in processing. An ability to characterise their behaviour more appropriately will have benefits in materials development and in process and product design.

The techniques most suitable for characterising the extensional flow behaviour of polymer melts are converging flow methods that use capillary extrusion rheometry data and stretching flow methods. These techniques have been the focus of recent research at NPL. The methods were critically reviewed [1, 2]. These reviews also identify sources of information on testing of a range of materials. Evaluation of the converging flow approach to determine extensional viscosity has been carried out and is reported in detail elsewhere [3].

To address measurement issues concerning stretching flow methods an extensional rheometer was developed at NPL. This note briefly describes that instrument and presents results obtained using it on a range of polyethylenes.

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## The Method

The NPL has developed an instrument for measuring the extensional viscoelasticity of polymer melts, Figure 1. The principle of the instrument is to stretch a molten specimen between a rotating clamp and a fixed clamp. The fixed clamp is used to measure the tensile force developed in the specimen during stretching. It has been used up to 230°C, and has a maximum operating temperature of 250°C, limited by the silicone oil into which the specimen is immersed. The method is described in further detail elsewhere [4].

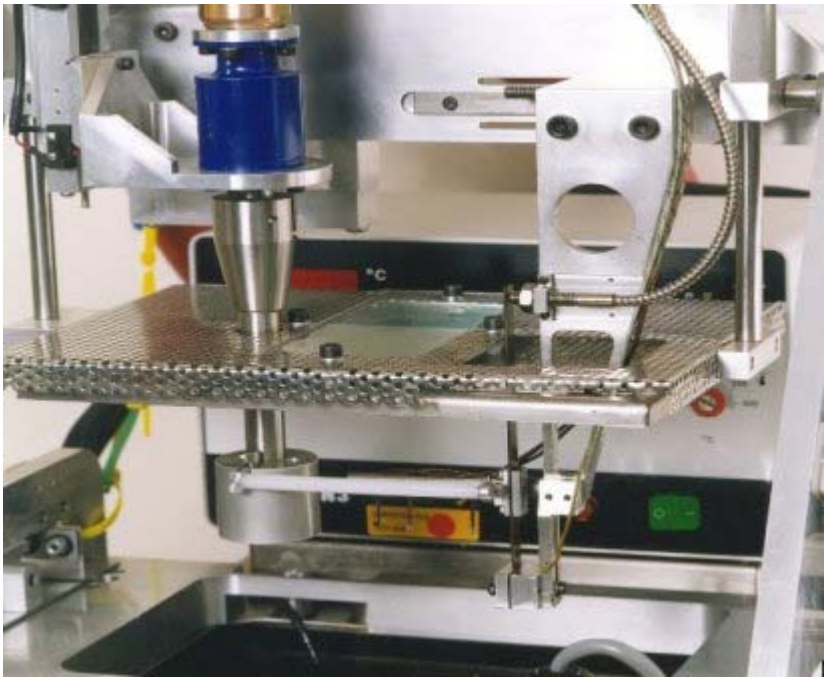

**Figure 1. Extensional rheometer**

# Extensional Flow Characterisation Results

Results are presented in terms of tensile stress growth coefficient data, defined as the ratio of tensile stress to Hencky strain rate. It is thus a transient extensional viscosity.

The use of different specimen length to diameter ratios in the range ≈ 7 - 30 (constant diameter) indicated there was little effect on results at strains above 0.5, Figure 2. However, at low strains of the order of 0.1 there was a possible correlation of aspect ratio with measured values. The use of shorter specimens resulted in higher tensile stress growth coefficient values, although the total variation was within approximately ± 10%. This trend is as expected as the effect of end-errors would increase for shorter specimens.

The repeatability of measurements, as illustrated for example in Figure 2, is considered to be good with variations typically less than ± 10% for strains above 0.1.

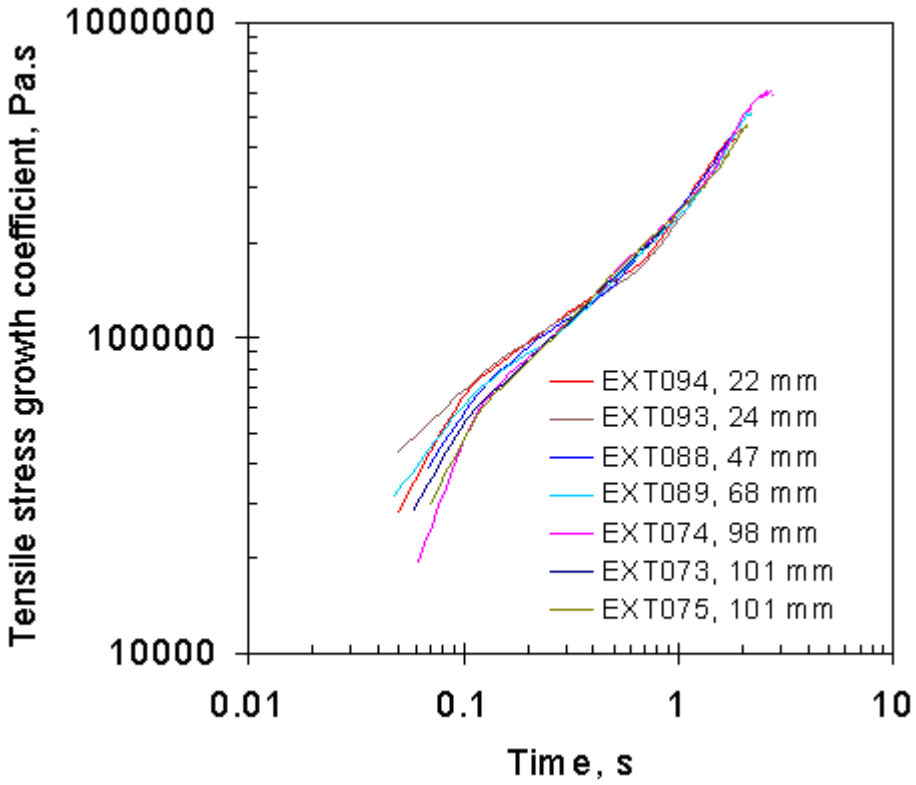


**Figure 2. Effect of specimen length on the tensile stress growth behaviour of a HDPE (HGH) at 150°C and a strain rate of 1 $s^{-1}$. All specimens ≈3.2 mm in diameter.**

Results of extensive testing of a HDPE (HGH[1]) over three decades of strain rate from 0.01 - 10 $s^{-1}$ are presented in Figures 3 and 4. These results clearly show the significant variation in behaviour with strain rate and strain. They also indicate the substantial range of the instrument.

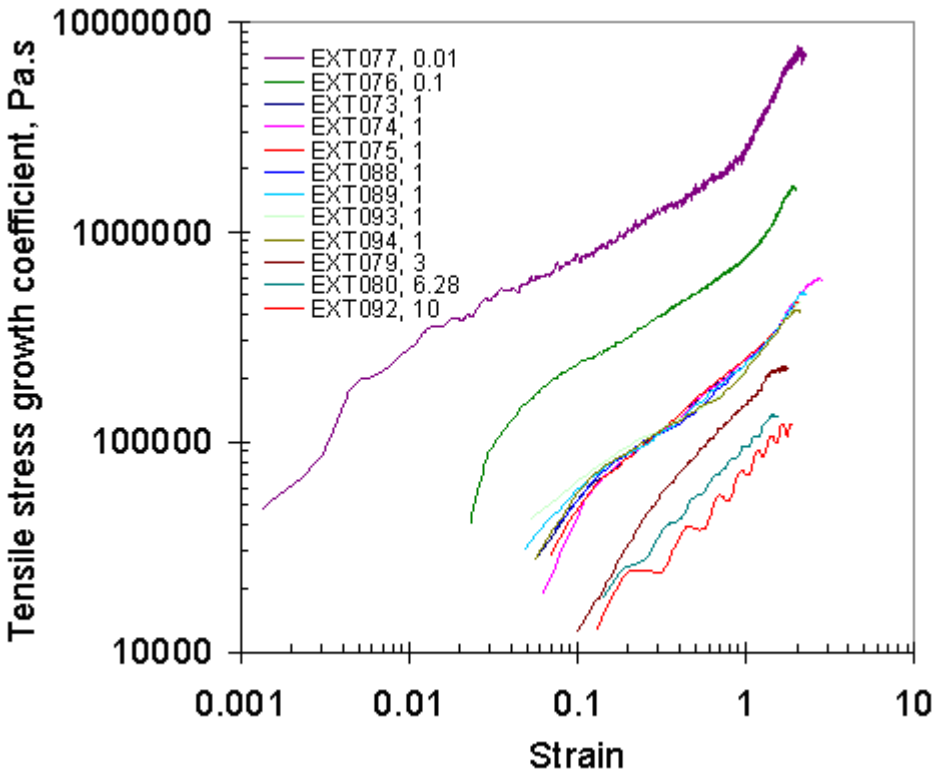

**Figure 3. Effect of strain rate from 0.01 to 10 $s^{-1}$ on tensile stress growth coefficient behaviour of a HDPE (HGH) at 150°C.**

For the high strain rate of 10 $s^{-1}$ it is noted that failure occurred within $\approx$ 0.25 seconds from commencement of the test, indicating the need for high sampling rates to obtain adequate data.

The peak tensile stress growth coefficient values for these four polyethylenes, obtained immediately prior to failure of the specimens, have been plotted as a function of strain rate and clearly indicate the rate dependence of their behaviour, Figure 4. The behaviour of the linear low density polyethylene (LLDPE) is relatively independent of the rate, while the high density polyethylenes (HDPEs) and the low density polyethylene (LDPE) have similar rate dependencies. The tensile stress growth coefficient values varied by over two decades indicating the significant differences in the extensional flow behaviour of the materials.

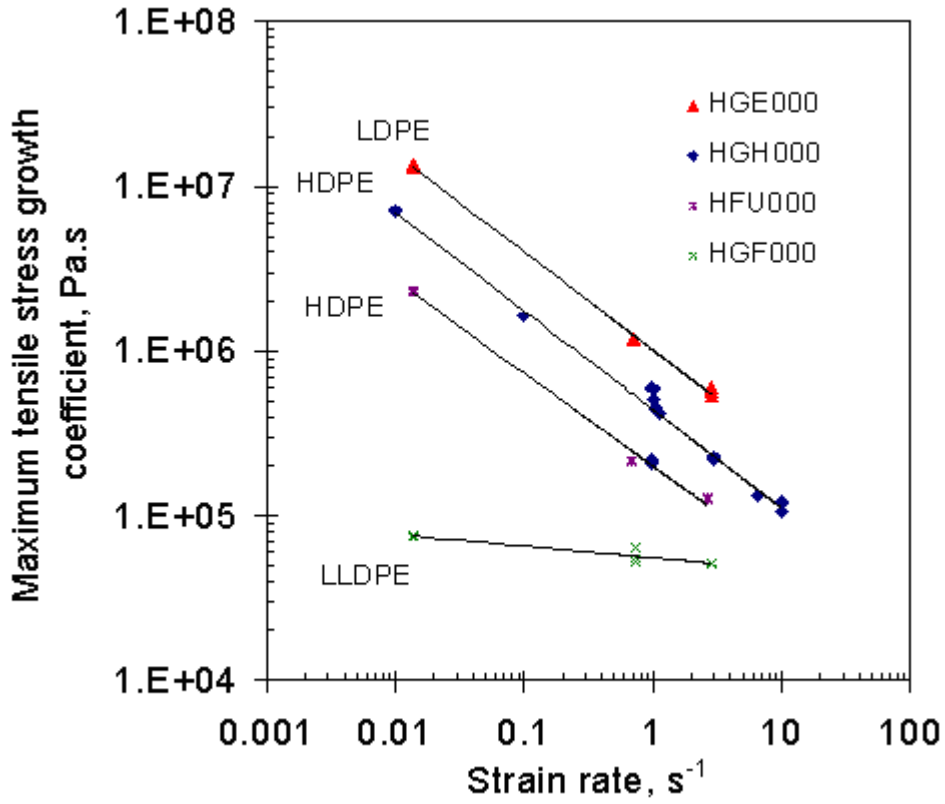


**Figure 4. Comparison of maximum tensile stress growth coefficient values for four polyethylenes at 150°C.**

# Intercomparison Of Stretching Techniques

The results of an intercomparison of stretching techniques indicated that the variation in tensile stress growth coefficient values was estimated to be up to approximately ±60%. The variation in peak tensile stress growth coefficient values was up to approximately ±100%. A high density polyethylene that was stable at 190°C for in excess of two hours was used as the intercomparison material. Measurements were made at 150°C and 190°C [5].

# Analysis Of The Uncertainties In Measurement

Details of the analysis of the uncertainties in extensional measurements are presented elsewhere [4]. The analysis can be used to identify the critical parameters in the measurement method that generate the uncertainties and can thus be used to improve the measurements.

The analysis, using a HDPE (HGH) at 150°C and a strain rate of $\approx$ 1 $s^{-1}$ as an example [4], indicated a significant increase in measurement uncertainties at high strains. This increase was predominantly due to the increase in the uncertainty in the measurement of force resulting from the significant reduction in the cross-sectional area of the

specimen during the test. Uncertainties were estimated to be approximately ± 20% at a strain of 3.5 and ± 50% at a strain of 5.5. It is noted, however, that strains as high a 4, corresponding to a reductions in the cross-sectional area of the specimen by ×55, are rarely encountered in processing.

# Summary

Extensional flow characterisation yields information about the materials not revealed by shear flow measurements. Thus materials characterisation solely on the basis of shear flow measurements is inadequate, particularly when the data are required for predicting the performance of materials in processes that are predominantly extensional flows.

Aspects of the measurement of extensional flow behaviour using stretching methods have been discussed. Results obtained for a range of polyethylenes have been presented to illustrate the measurement of the transient extensional flow behaviour of polymer melts. A Good Practice Measurement Guide on extensional testing has been prepared [4] that:

- discusses various issues related to the need for, and measurement of the extensional flow properties of polymer melts,
- defines approved terminology and presents definitions,
- describes important considerations for stretching flow measurements,
- describes practical considerations for measuring extensional flow behaviour, including the analysis of the experimental data and assessment of the effect of uncertainties on the measured values.

To illustrate the measurement technique, typical data obtained using the NPL extensional rheometer are presented.

# Footnotes

1. NPL references to the materials, e.g. HGH, are used to identify different grades of materials.

# Acknowledgements

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# References

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