SQUEEZE FLOW RHEOLOGY IN LARGE TOOLS

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A 310t hydraulic press has been modified for squeeze flow rheology studies so that large tools and high closing velocities can be achieved. Isothermal squeeze flow experiments of GMT have been performed for closing velocities up to 30 mm/s and maximum closing forces of 1000 kN. Successful power law fits of closing velocity versus closing force data have been performed for the velocity interval 3-30 mm/s.

INTRODUCTION

The need for accurate simulations of the manufacturing processes and mechanical properties determined by the process is becoming increasingly interesting nowadays when the development time has to be shortened. Increased use of compression moulded materials in, for instance, the automotive industry demands higher capability for and accuracy in simulations of the compression moulding process. One cornerstone in mould filling simulation of compression moulding materials, is knowledge of the rheological properties of the material. Rheological constitutive equations provide a coupling between strain rate (velocity gradients) and stress. The complexity of these equations range in general from linear, in the case of Newtonian fluids, to more advanced models including for instance a non-linear behaviour or memory effects of the fluid.

Besides strain rate, other state variables also affect rheological properties, for instance degree of cure or temperature, both of common interest when modelling composite materials. Commonly, rheological models are determined by curve fitting within, for the method, relevant strain rate intervals. Since the models, in many cases, only describes subsets of the full material characteristics extrapolations outside the tested interval may be hazardous.

Numerous methods, for instance different kinds of rheometers, exist for determination of the rheological constitutive equations, and the parameters used within. For compression moulding materials, where the material consists of a mix of fibres and matrix, squeeze flow rheometers have become widely used. A squeeze flow rheometer consists of two parallel plates moving towards each other. The material to be tested is placed between the plates and squeezed either out of (constant area tests) or kept inside the tool perimeters (constant volume). The reason for the popularity of the method, besides the close resemblance of the manufacturing method, is that the large sample size makes it possible to treat a sample consisting of a mix of fibres and matrix as a homogenous material.

Using the continuity equation together with the equation of motion, analytical expressions for the radial pressure profile (and hence the closing force) in the material during squeeze flow have been derived for Newtonian and power-law materials [1]. Using either a slip or a no-slip boundary condition at the mould surface, expressions for the two possible through thickness velocity profiles (plug flow/bi-axial extension or near parabolic/shear) is calculated. Extensive studies on the material using constant area tests in a circular tool have been performed by Kotsikos [2] and co-authors. Successful fits to power-law constitutive equation were performed based upon the analytic expression for closing force and assumption of power-law material.

$$F_{ext} = \pi R^2 A_e(\varepsilon)\left(\frac{u}{h}\right)^n \tag{1a}$$

$$F_{shear} = \left(\frac{2m+1}{m}\right)^m \left(\frac{2\pi A_s R^{m+3}}{m+3}\right)\left(\frac{u^m}{h^{2m+1}}\right) \tag{1b}$$

With F_{ext} or F_{shear} determined by the boundary conditions (lubricated, non-lubricated), R tool radius, u closing velocity, h mould displacement A, m and n are material constants. Flow of bi-axial extension kind was found to be suitable when modelling isothermal squeezing of GMT with the molten polymer forming a lubricating zone close to the boundaries. The core material is deforming bi-axially as shown for SMC by Barone and Caulk [3]. A shear dominated velocity profile is however to be expected if the tool displacement is small [4] with the flow characteristic changing from plug flow to shear dominated [5].

Based upon the governing equations, numerical studies of the closing force both for lubricated and no-slip boundary conditions have been studied by Lee and co-authors [6]. They showed that the radial pressure profile predicted by the analytical solution does not accurately describe the actual pressure profile especially if small tools are used. The cutting of fibres at the boundaries and by that, the reduction of the actual fibre length was a problem pointed out by Kotsikos [4]. The reason for errors in the pressure profile and the non-locality of material was also addressed by Toll and Gibson [7].

The in-homogeneity of the material together with the desire to keep the ratio between the peripheral boundary and the area of the material as small as possible suggest the use of a tool as large as possible. However, since a large tool requires presses capable of producing high loads, lack of experimental data for high velocities and large tool areas exists.

In this paper, the possibility to use a 310t hydraulic press with closing velocities between 3 and 30 mm/s has been investigated. The large compression force and the wide range in closing speeds makes it possible to use large tools for squeeze flow rheology studies under conditions resembling a real processing situation. Isothermal squeeze flow rheology studies at high closing velocities is compared to previously published results in order to extend the interval of knowledge to higher closing velocities.

EQUIPMENT

The Fjellman hydraulic press used in the experiments is a hydraulic laboratory press and has a maximum clamping force of 310 tons. The maximum closing velocity is above 30 mm/s but for the experiments 30 mm/s have been the maximum used. Parallelism and closing velocity is controlled by bi-directional hydraulic cylinders located at the four corners of the lower pressing table. During closing, the corner cylinders move upwards and meet the upper pressing table to control the closure. In this phase (from a mould displacement of approximately 50 mm) the forces acting on the material are the force by the main hydraulic cylinder (diameter 450 mm) and contradirectional forces from the four corner cylinders. In order to calculate the closing force accurately, the forces from the corner cylinders have to be subtracted from force applied by the main cylinder. Applied forces by the cylinders are calculated using measured hydraulic pressure and piston area. Or, expressed as an equation

$$F_{material} = P_{main}A_{main} - \sum_{i=1}^{4} \max\left(0, P_{i,upward}A_{upward} - P_{i,downward}A_{downward}\right) \quad (2)$$

With $F_{material}$ as the force acting on the material, P_{main} the hydraulic pressure in the main cylinder, P_i the pressure in corner cylinder 1-4, A_{main} and A_i represents piston area for the main and the corner cylinders respectively. If the downward force from one corner cylinder is larger than the corresponding upward force, the cylinder will not be in contact with the upper table and no pulling force can be extracted. Totally nine pressure transducers (Dynisco IDA334-3.5C 0-350 bar) are used to monitor the pressure in the hydraulic system. All hydraulic pressures are measured at designated points close to the hydraulic cylinders. Static conditions are assumed so acceleration of the upper pressing table is neglected. The experiments have proven this assumption reasonable, even though some deceleration occur during the final phase of the mould closure. When the mould hits the material, decelerations of 200 mm/s^2 have been recorded corresponding to dynamic forces less than 0.5 kN for the ~ 2000 kg pressing table. Since the closing force during this phase of mould closure is of the magnitude of more than 50 kN, the error introduced by assuming static conditions is less than 1% for the worst case.

The squeeze flow rheology study was performed as constant area tests, i.e. tests with an open mould filled with material at isothermal temperature. During squeezing the material flows outside the tool so that the dimensions of the sample are known throughout the experiment. A circular tool with a diameter of 300 mm equipped with three flush mounted Kistler 6153C piezo electric pressure transducers placed at radius 0 mm, 75 mm and 140 mm, Figure 1 was used. The piezo electric pressure transducers were connected to a Kistler 5037 charge amplifier and further to a data acquisition system. The data acquisition system, (consisting of an A/D converter IOtech 8 MB, 16 bits, 16 channels and an Apple Macintosh 7100/66 AV computer running Labview 3.1.1) was used to monitor the nine hydraulic pressure transducers, the three in-mould flush mounted pressure transducers and a resistive displacement transducer. The sampling frequency used in the experiment was between 100 Hz (low closing velocities) and 500 Hz (high closing velocities), resulting in a few hundred data points per sensor for each experiment.

In order to calculate velocity, the displacement curves are differentiated numerically with respect to time. The high sampling frequency in combination with some background noise in the displacement curve, Figure 2 (although twisted cables were used to increase the signal to noise ratio), resulted in a more noisy velocity curve after differentiation.

Closer study of the signal in Figure 3 indicates that the signal is a combination of an almost linear motion and a superimposed oscillation with a frequency of about 20-30 Hz. The most likely reason for the superimposed oscillations is low frequency vibrations in the equipment. Velocity changes of the upper table of the press caused by changes in the hydraulic pressure seem unlikely as an explanation since the resulting changes in applied force would have been visible in the force curve. The vibrations together with the high sampling frequency will create high apparent velocity changes if velocity is calculated using adjacent terms in time and displacement. In order to exclude the noise, the velocity was calculated using the slope of a fitted line, ranging 25 points (50 ms for the fast and 250 ms for the slow closing velocities) backward and forward in time.

VALIDITY OF USING A 310t PRESS FOR SQUEEZE FLOW RHEOLOGY EXPERIMENTS

Prior to the experiments, tests to investigate the possibility of using a large scale hydraulic press as a scientific instrument were performed. Mould closing velocities and closing forces are programmed in the press' PLC but velocity, displacement and force readings used in the analysis are measured by the data acquisition system and not the values set in the PLC.

In order to investigate the accuracy in the closing force, a circular plate tool was used. The male part described in Figure 1 is also used in the squeeze flow rheometer. The female part was equipped with a pneumatic centre ejector with a ½" air channel running inside the tool from the centre, to the air inlet at the edge of the tool. Filling the channel with oil and mounting a Dynisco IDA334-3.5C (0-350 bar) pressure transducer at the edge provided a fluid filled connection from the centre of the tool to the pressure transducer.

Compression of a circular lubricated rubber disc with a 2 cm circular hole in the centre creates a hydrostatic pressure inside the tool measured by the edge mounted pressure transducer. The pressure in this reference transducer was then compared with the clamping force divided by tool area, Figure 3. A linear least square curve fit was applied to the force-pressure data resulting in an applied force divided by tool area to measured pressure relation of $y=1.02x+1.59$. The offset of 1.59 bar corresponds to having a weight of the upper press table of 1150 kg instead of the actual weight of, according to the design manual, 2000 kg. The difference between the weight of the pressing table and the measured weight is believed to be caused by the two return cylinders mounted to the upper press table acting upwards. Calibration of the Kistler in-mould pressure transducers was performed in the same way as a validity check of the applied force.

The charge amplifier was automatically short-circuited between each individual experiment to ensure non-drift in the piezo-electric pressure transducers. The Dynisco pressure transducers were calibrated by short circuiting designated positions at the pressure transducer to produce an output signal equal to 80% of the maximum output. The deviation in linearity of the Dynisco pressure transducers are less than 0.1 % and regarded to be unimportant for the squeeze flow rheometer.

EXPERIMENT

The equipment described above was used for isothermal constant area squeeze flow experiments in the 300 mm open circular tool. Closing velocities ranging from 3-30 mm/s were investigated for Symalit GM40PP, GMT material. For all experiments, two sheets of raw material (cut to the size of the mould) with an initial thickness of 3.8 mm (in the unheated state) were placed on top of each other. The angle of the material relative to the main direction of the raw material sheet was shifted 90° between the layers to make the material more isotropic.

Due to the design of the press, special procedures have to be taken during heating of the material. The fact that only closing velocity and clamping force are programmable parameters and not position, steel cylinders had to be placed at the lower press table to keep the upper table at a fixed position during heating. The material was heated by conduction from the lower mould half and initially, convection from the top, the distance between the two mould halves was approximately 17 mm leaving space for the material to loft when the polypropylene was molten. The temperature of the material was measured using Iron-Constantan thermocouples placed at the centre of the material between the two sheets and at the top surface. A heating time of 1h was found sufficient and isothermal conditions were reached after approximately 40 minutes. During heating, insulating material was placed around the mould to speed up the heating time and to provide as constant temperature in the material as possible. After heating, the upper press table had to be moved upwards to remove the steel cylinders and the insulation. After that, as soon as the pressure in the hydraulic system was sufficient for squeezing (<15 s), the squeezing phase started. Automatic trigging of the measurement was done using magnetic switches at a distance approximately 60 mm prior to mould closing.

During closing, the distance between the two mould halves, the closing velocity (calculated from the displacement data) and the closing force were measured as well as the in-mould pressure. Deviations between the set velocity in the PLC and the velocity registered by the data acquisition system existed as well as velocity changes during the closing phase. Deviations between the set and measured velocity is not a problem since all measurements are based upon the actual readings and not by the set values. The non constant closing velocity is probably caused by lag in the hydraulic system. The mould was heated by conduction from electrical heaters in the table of the press so no rapid change in mould temperature was possible, after each experiment excess Polypropylene and fibres were removed from the mould surfaces. Repeated measurements were not conducted after each other to further check for deviations based upon drift or other time-depending occurrences.

RESULTS AND DISCUSSION

Squeeze flow rheology experiments were conducted using Symalit GM40 PP of a temperature of 180°C at four different closing velocities. Three repetitions for each set velocity were used. The closing forces and the actual velocity for the experiments are presented in Figure 4 and Figure 5 respectively. Especially for the higher closing velocities, the velocity is decreasing during the final phase of closure an effect probably related to the response time in the press. However, the repetitiveness in closing force, Figure 4 and closing velocity, Figure 5 within each set velocity is very good especially for the low closing velocities. This repetitiveness together with the foregoing studies of the press force contra the in-mould pressure suggests that the method is robust in terms of the equipment and homogeneity of the material to be tested.

Note the velocity change at 10 mm, in Figure 5, this deceleration occurs when the upper mould half hits the material charge. The time between impact and the time until the set velocity is reached again is approximately 0.15 s for the 30 mm/s case. During this phase, a maximum acceleration/deceleration of 200 mm/s^2 is present but dynamic forces are still small compared to the closing force and are not taken into account. During the rest of the squeezing phase, the velocity is relatively constant for the lowest velocity while a velocity change of about 10-20% occurs for the higher closing velocities.

The original thickness of the two sheets of material that are stacked on each other is 7.6 mm. No major change in force or velocity is visible when the lofted material is fully compressed to original thickness and the actual squeezing is started. Since a constant velocity was impossible over the full velocity range, studies of closing force for constant velocities were impossible to do. Instead, closing force was plotted against closing velocity for different mould displacements during the whole closure phase. Closing force as function of closing velocity for different mould displacement is presented in Figure 6. A fitted power-law equation is included in the plot and the agreement to the least square fit is very good in the first part of the velocity interval. For higher velocities however, the deviation is larger but analysis of residuals in a normal probability plot does not show any major deviations from the normal line. The power-law model has to be considered a successful model for the range of velocities used with the power law exponent continuously decreasing for decreasing mould displacement. The values of the power law index of 0.5 agrees well with experimental data by Kotsikos and co-authors [4] who assumed a power-law material and bi-axial extension flow kind but who used strain rates a decade lower and a smaller mould.

CONCLUSIONS

Investigations have been performed regarding the possibility to use a large scale hydraulic press for squeeze flow rheology experiments in order to achieve larger closing forces and higher closing velocities than with the commonly used testing machines. The different tests of the equipment shows that the agreement between measured pressure/force in the tool and the applied force is remarkably good. Due to the design of the hydraulic press, including the PLC and the components in the hydraulic system, a lag exists in the force response. This lag leads to a non-constant closing velocity for the highest velocities and incapability to maintain constant closing velocities especially when the closing force is high and increasing. However, using high sampling frequency makes it possible to measure force during closure and to determine a closing force to closing velocity relationship during closure for different mould displacements. This will be sufficient for determination of material parameters for materials that does not exhibit memory effects.

Constant area squeeze flow rheology studies of Symalit GM40 PP GMT material at an isothermal temperature of 180°C has been performed using a large (diameter=300mm) circular open mould at closing velocities ranging from 3-30 mm/s. A power-law velocity to closing force relationship was successfully fitted to the experimental data. The value of the power-law exponent agrees well with previously published data for smaller moulds and lower velocities extending the results.

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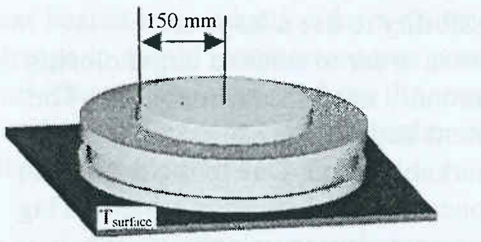

Figure 1, Circular tool used for squeeze flow rheology experiments.

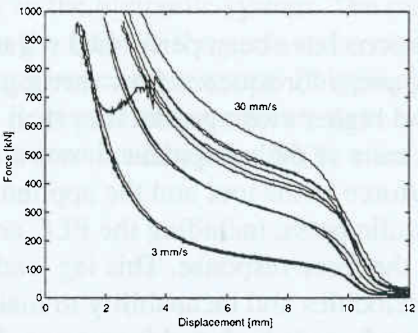

Figure 4, Closing force for different closing velocities for Symalit GM40PP 180°C

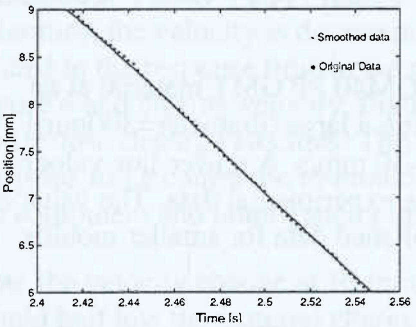

Figure 2, Original and smoothed displacement curves.

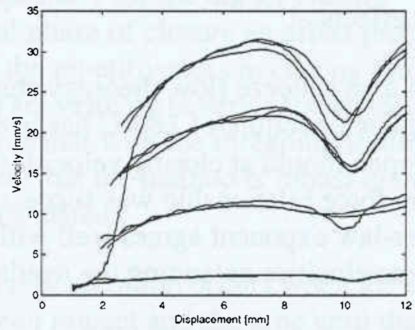

Figure 5, Closing velocities used for the squeeze flow experiment for Symalit GM40PP 180°C

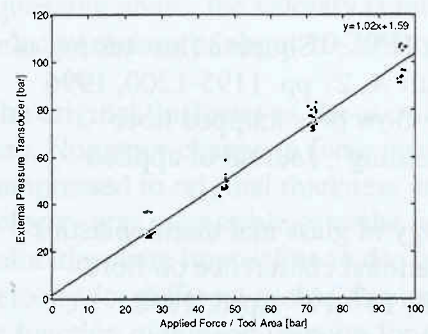

Figure 3, Comparison of the applied force and pressure in the tool.

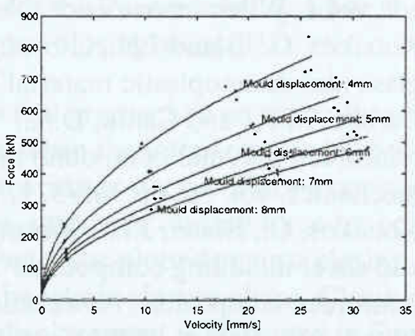

Figure 6, Closing force as function of velocity for Symalit GM40PP 180°C. Dotted lines represents a power-law fit.