AN EXPERIMENTAL INVESTIGATION OF THE PLASTICATING AND FRICTION BEHAVIOR OF PET CHIPS

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Abstract

An experimental study of the plasticating and frictional behavior of dried and preheated PET chips on a moving, steel surface has been conducted using an experimental apparatus called the Screw Simulator. The Screw Simulator has been specifically designed to reproduce the primary plasticating [melting] and solid conveying mechanisms of single screw extruders. The screw simulator permits the direct observation and measurement of a materials melting and frictional properties. The PET resin plasticating and frictional properties were studied as a function of metal surface temperature, surface velocity, sample width and applied load. In addition the materials bulk density as a function of pressure at several preheating temperatures was measured. These experimental findings have important implications in the design and scale up of single screw designs

Introduction

Melting within an extruder was for many years an unknown mechanism. It was not until the pioneering screw freezing experiments of Bruce Maddock[1] that the standard mechanism of single screw melting was described [Figure 1]. Once the melting mechanism was understood there was an explosion of modeling work to solve the melting problem for use in screw design calculations[2, 3, 4, 5]. The difficulty with the initial melting models was that there was no independent way to check the accuracy of the assumptions and the subsequent melting rate calculations. All of the models were checked by estimating the overall performance of the extruder, which combined the assumptions of an extrusion model [solids conveying, melting and metering model combination] over laid on the assumptions of the melting model. Consequently, the melting model verifications were all indirect verifications leaving the correctness of the melting model assumptions and the accuracy of the calculations in question[6, 7].

This was the situation until the development of the "Screw Simulator" by Chung[8] [Figure 2]. The screw simulator is a device specifically designed to measure the frictional forces[9] and the melting rate of polymers[10] using the solid conveying model of Darnell and Moll[11] and the idealized melting model of single screw extruders [Figure 3]. Prior to the onset of melting the polymer rubs against the barrel surface and controls the solid conveying of the polymer into the screw. In the idealized mechanism, melting occurs only at the interface between the solid plug of polymer and the barrel surface. This is the location where the power of the motor is dissipated by viscous drag to generate the thermal energy used to melt the polymer in a single screw[12]. The solid plug assumption can also be replaced by a compacted bed of pellets[13].

The screw simulator, while similar in concept to the device of Sundstrom and Young[14] allowed direct experimental measurements of friction and melting at conditions typical of operating extruders. Figure 4 shows the melting polymer system as implemented with the screw simulator. The applied load P_0 forces the compacted solid plug of width X_0 against the heated [T_b] moving [V_b] roll surface (the relative velocity of the solid against the metal surface). The solid velocity in the y-direction [V_{sy}] is measured directly by the LVDT and the mass of solid melting per unit area [Ω] is equal to the amount of melt dragged from under the solid plug on the barrel surface. The molten polymer layer is removed by the scraper to mimic the action of the pushing screw flight in the melting mechanism of Figures 1 and 3. The screw simulator for the first time permitted the development and verification of the assumptions and accuracy of the various melting models for plasticating extruders[7].

The friction and melting measurements are important as they are used to determine the performance and design parameters of operating

extruders. The measurements confirmed for the first time that in the limit, single screw extruders are melting limited apparatus[15, 16]. Also, they can be used to estimate the expected output of a single screw extruder based on the screw dimensions, details of the screw design, polymer type and operating conditions. In particular the measurements have been used in a scientific screw design method described by Chung[17, 18]. Melting data for screw design calculations can be obtained either experimentally or estimated using the melting model of Mount, Watson & Chung[8] for fully compacted solid beds or Kuo & Chung[13] for granular solid beds.

Experimental

The Screw simulator was used to measure the bulk density, frictional properties and the melting properties.

The resin sample was a polyethylene terephthalate (PET) chip of 0.60 IV. Prior to testing, the resin sample was dried and crystallized in a vacuum oven heated to 165°C to 177°C [330°F to 350°F].

Bulk density measurements are obtained by introducing a weighed sample to the screw simulator sample cell (2.54 cm by 7.62 cm), the sample plunger is inserted and the initial sample height measured at 6.89×10^{-2}-1.38×10^{-1} MPa [10-20 psi] with the screw simulators LVDT to give the initial sample volume. Pressure is increased in a stepwise fashion and the sample volume calculated as a function of pressure to give the sample bulk density.

Friction coefficients were obtained by:
1. Presetting the roll speed and roll temperature
2. Preheat the chip to 83.3^{o}C
3. add a known weight of polymer pellets to the sample cell
4. apply the desired load to the sample
5. allow sample/roll interface to equilibrate
6. Activate recording equipment and rotate the roll for several seconds to obtain applied load and frictional force.

The roll is only rotated to give an initial frictional force as extended operation generated significant frictional heating of the interface which affects the measurements.

Melting experiments are performed in the following manner:
1. The roll velocity, temperature and load are preset before the measurement is started
2. The recording equipment is set to measure LVDT position and the sample restraining force [shear stress]
3. Preheat the chip to 83.3^{o}C
4. A pellet sample is introduced to just fill the sample chamber, the plunger inserted and the load applied and allowed to equilibrate for 10-15 seconds to compress the sample
5. The roll rotation is started and the melting rate measured. During the melting measurement, the roll surface is scraped to remove the melt layer from the roll surface
6. The height of the solid sample is recorded with the Screw Simulator LVDT and plotted as a function of time during the experiment to obtain the solid velocity in the direction of the roll surface (Y direction)
7. At the end of the experiment the load is released, the roll stopped and the recording equipment stopped.
8. The melting velocity cm/min is measured as the slope of the LVDT position vs. time on the strip chart recording, the melting shear stress is determined from the measured sample chamber restraining force.

Melting rate, Ω [kg/min/cm^2] is calculated as the product of the solid velocity [cm/min] measured as the slope of the LVDT chart recording and the pellet bulk density (gm/cm^3) at the load applied to the sample.

Melt viscosity was measured as a function of temperature and shear rate using an Instron 3211 capillary rheometer. Temperatures were 260 ^{o}C, 270 ^{o}C, 280 ^{o}C, 290 ^{o}C and 300 ^{o}C. Data was obtained using a 0.1275 cm [0.0502 in] diameter by 7.623 cm [3.0037in] 59 L/D long capillary with a 90 degree entry angle. Prior to the viscosity measurements, the pellet samples were dried for 9 hours in vacuum at 165°C. Because the PET melt viscosity is

Newtonian, the apparent viscosity is presented and the data was not Rabinowitsch corrected.

Experimental conditions of surface temperature, sliding velocity and applied load were varied one variable at a time while maintaining the other two at a constant value. For the friction measurements, the applied load, P_0, was help at 6.89MPa (1000 psi) as representative of most expectations for solid conveying pressures. For the melting experiments an applied load of 2.76MPa (400 psi) was selected as representative of the pressure where the equilibrium melting rate was observed. The sliding velocity of 55.9 cm/sec was chosen as representative of a 114.4 mm diameter screw, rotating at a screw speed of 93.4 rpm. For the friction experiments, a surface temperature of 121°C, as representative of a feed zone temperature, and for the melting experiments, a surface temperature of 276 °C and 293 °C, as representative of melting and metering barrel zone temperatures were used.

Calculations

The viscosity data is plotted in Figure 1 and was curve fit to Equation 1. The regression results are found in Table 1.

$$\ln(\eta_{ap}) = \ln(m_0) + (n-1)\ln(\dot{\gamma}_{ap}) \qquad \text{Eqn. 1}$$

The bulk density was calculated for the compressed solid volume and the initial weight of the sample. The data is plotted in Figure 2 and was curve fit to Equation 2. The regression coefficients are listed in Table 2.

$$\rho_b = A_o + A_1 P_o + A_2 P_o^2 \qquad \text{Eqn. 2}$$

The melting and frictional data were obtained as described and the experimental data used to calculate the frictional coefficient [Equation 3] and the solid melting velocity, V_{sy} in the Y-direction [Equation 4].

The melting rate Ω (kg/sec/cm) and the Melting efficiency Ω/V_b (kg/cm²-cm), are calculated with Equations 4 & 5. The melting efficiency has units of density but are better understood if they are expressed as mass melted per unit area per unit distance slid or gm/cm²-cm.

$$F_c = \text{Frictional force / applied pressure} \qquad \text{Eqn. 3}$$

$$\Omega = V_{sy}*\rho_b \qquad \text{Eqn. 4}$$

$$M_{eff} = \Omega/V_b \qquad \text{Eqn. 5}$$

For calculations based on the Mount et al Model, the dimensionless melting efficiency is described as [Equation 6]

$$M = \Omega/(V_b*\rho_b) = M_{eff}/\rho_b \qquad \text{Eqn. 6}$$

Results and Discussion

Figure 5 presents the bulk density data as a function of pressure and chip preheating temperature. It is clear that the preheat temperature used to dry the chips has an impact on the solids bed compaction.

Preheat temperature will also have an impact on melting rate as it decreases the energy required to raise the solid to the melting point. Consequently, it is important to maintain the chip temperature as high as practical when transporting it from the chip drier to the extruder feed pocket. In the case at hand the chip was expected to drop in temperature from 166°C at the exit of the drier to 83°C at the extruder feed hopper. Consequently, the friction and melting measurements were preformed with a solid temperature of 83°C.

Figures 6, 7 and 8 presents the Solid frictional coefficient (COF) data obtained as a function of metal surface temperature [T_b], Roll surface velocity [V_b] and applied load [P_0].

In Figure 6 the COF is seen to be a weak function of surface temperature for a sliding velocity of 55.9 cm/sec and an applied load of 0.145 Pa. At approximately 175°C the solid is observed to grind [fine, white, powdery pieces of polymer adhered to the roll surface] indicating the beginning of mechanical melting [plastication]. At a surface temperature of 190 °C and 207 °C the sample is observed to be melting (filled circles) with a distinct melt film on the roll surface, indicating an interfacial temperature of greater than the melting point of approximately 260°C.

In Figure 7, the COF remains fairly constant for sliding speeds below 80 cm/sec and then jumps slightly. This is likely due to the increased heat generation at higher sliding speeds and indicates the potential for a change in solids conveying rate at

increased screw speeds. Operating at screw speeds near the 80-85 cm/sec transition could potentially cause surging in systems where the screw speed is controlled by a device such as a melt pump. For constant screw speed operation a jump in feeding would be expected for screw speeds above 85 cm/sec peripheral speeds. This could result in an increase in specific output or surging depending on the screw design and operating conditions. At any rate the observed jump in COF is important to note.

Figure 8 plots the COF as a function of applied load. The COF is seen to remain fairly constant with increasing pressure. At a pressure of 0.06 Pa there is a slight jump in COF. This might be due to packing variations of the pellets but no real explanation of the observation can be given.

In all instances after the frictional measurements, observations of the preheated chip samples at the polymer/metal interface showed a void free interface indicating that the sold bed deformed sufficiently to completely contact the metal surface. At the polymer/metal interface, the solid bed is behaving like a solid plug.

Figures 9, 10 and 11 present the plasticating efficiency as function of metal surface temperature [T_b], Roll surface velocity [V_b] and applied load [P_0] respectively.

Figure 9 Plots the melting efficiency vs. surface temperature [T_b] for a solid temperature of 83.3°C, an applied load of 0.145 Pa (1000 psi) and a surface velocity of 55.9 cm/sec. The plot shows a slight maximum around 275-280°C and would drop sharply for surface temperatures below 250°C to the onset of melting seen in Figure 6 at about 200°C. The melting rate drops above 275-280°C due to the decrease in melt film viscosity and melting stress [Figure 12] and a subsequent drop in viscous dissipation in the melt film. Consequently, there is no advantage to increasing the barrel temperatures much above 270-275°C unless the extruder is torque limited.

Figure 10 plots the melting efficiency vs. surface velocity [V_b] for a solid temperature of 83.3°C, an applied load of 2.76MPa (400 psi) and surface Temperatures of 276°C and 293°C. The plot show a marked decrease in kg/cm²-cm slid and is indicative of a decrease in melting efficiency, not melting rate, with increasing screw speed. What this means is that as the sliding speed is increased, while more polymer is melted per cm², less is melted per cm slid. This is because while more energy might be dissipated in the melt film under the melting solid, less of the energy has time to diffuse to the melting interface and is swept into the accumulating melt pool. This is why melt temperature and torque increases with screw speed.

Figure 11 plots the Melting efficiency vs. applied load for a solid temperature of 83.3°C, a surface velocity of 55.9 cm/sec and a surface Temperatures of 296°C. The melting efficiency is seen to increase up to approximately 2.76MPa (400 psi) and reach a constant value. This is likely due to thinning of the melt film with increasing load to the point where the solid bed is sufficiently compacted to form a "solid" interface at the melting surface, limiting melt penetration into the solid bed. It indicates that for solids conveying pressures above 0.058 Pa the melting process will be in equilibrium with the solids conveying forces. Solids conveying forces higher than 0.058 Pa will not benefit melting and may only result in increased screw wear.

Figure 12 Plots the viscous stress vs. Surface temperature for a solid temperature of 83.3°C, an applied load of 6.89 MPa (1000 psi) and a surface velocity of 55.9 cm/sec. the viscous stress is seen to peak at approximately 275-280°C and then drop at approximately 285°C to a constant value above 290°C. This matches well the observed melting efficiency changes seen in Figure 9 and demonstrates the decrease in viscous dissipation above 285°C leading to the decrease in melting efficiency above 285°C.

Figure 13 plots the viscous stress vs. surface velocity [V_b] for a solid temperature of 83.3°C, an applied load of 2.76MPa (400 psi) and surface Temperatures of 276°C and 293°C. The viscous stress is seen to reach a maximum or slowly increasing stress above approximately 60 cm/sec, perhaps indicating the formation of an equilibrium or limiting melt film thickness. The limiting stress value corresponds to the decrease in melting efficiency observed in Figure 10.

Conclusions:

The experimental observations of the melting performance of 0.6 IV PET pellets highlight the basic frictional and melting behavior of the polymer. The

data can be used to help optimize the performance of PET screw designs for estimating output levels and highlighting limiting behaviors as a function of process settings. For instance, the impact of surface temperature and sliding velocity, on COF and melting efficiency, as well as highlighting transition points such as the sudden increase in COF with temperature, the maximum in melting rate with surface temperature. This information can be used to set optimum barrel temperature settings and screw speeds for solids conveying and melting and avoid settings which might result in extrusion instabilities.

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Tables

Temperature, C	m_0, Poise	n
260	5373	0.9114
270	3975	0.9301
280	2940	0.9448
290	1798	0.9331
300	1402	0.9072

Table 1: Viscosity data regression coefficients for Equation 1 with viscosity in poise. To convert poise to Pa-sec, divide result by 10

Solid temperature, C	$\rho_b = A_o + A_1 P_o + A_2 P_o^2$			
	A0	A1	A2	R^2
23.9	6.91E-01	2.38E-02	-7.63E-04	.997
82.2	7.08E-01	3.39E-02	-1.59E-03	.961
167	6.78E-01	7.38E-02	-2.80E-03	.991

Table 2: Regression coefficients for bulk density [gm/cm^3] as a function of pressure [MPa]
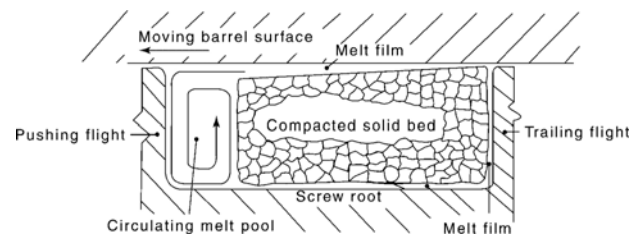
Figures

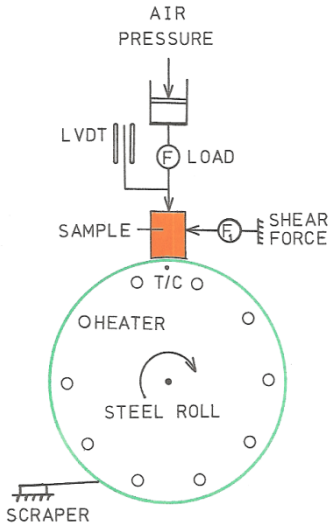Figure 1: Melting mechanism of single screw extruders as determined by Maddock.

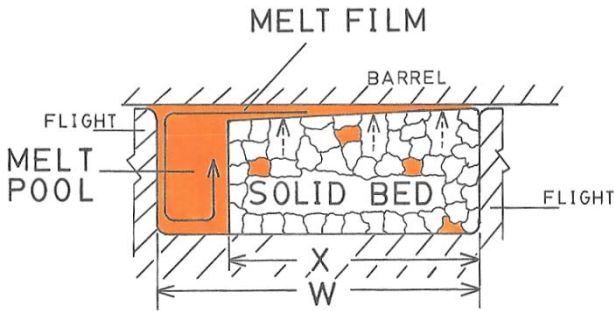Figure 2: Schematic diagram of the screw simulator

Figure 3: Idealized melting mechanism of single screw extruders assuming melting only at the barrel surface.
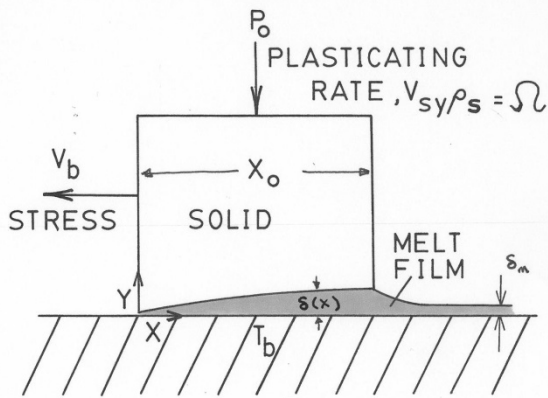

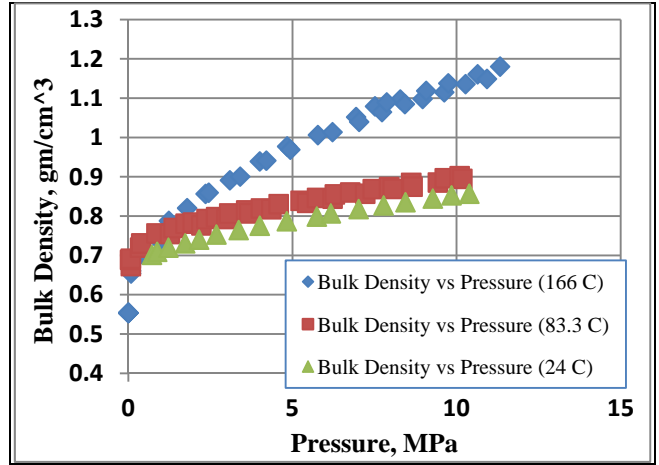Figure 4: Mass balance around melting solid plug at the barrel surface

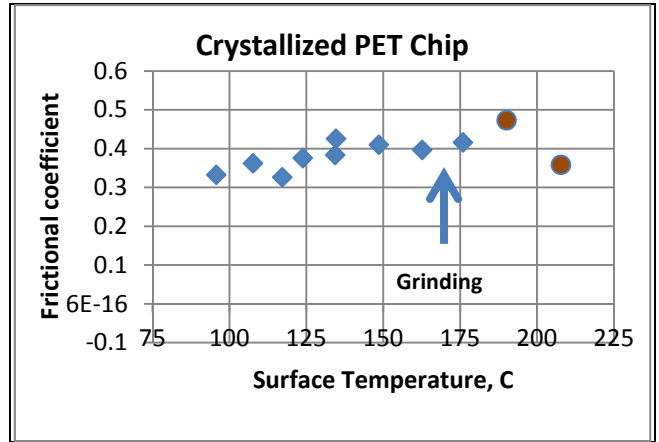Figure 5: Bulk density vs. pressure for PET chip at various solid preheat temperatures

Figure 6: Plot of Frictional coefficient vs. surface temperature [T_b] for a solid temperature of 83.3^{o}C, an applied load of 6.89MPa (1000 psi) and a surface velocity of 55.9 cm/sec. The sample was observed to grind at 175°C and the onset of plastication (circles) was observed above 190°C (melting point is approximately 265°C)
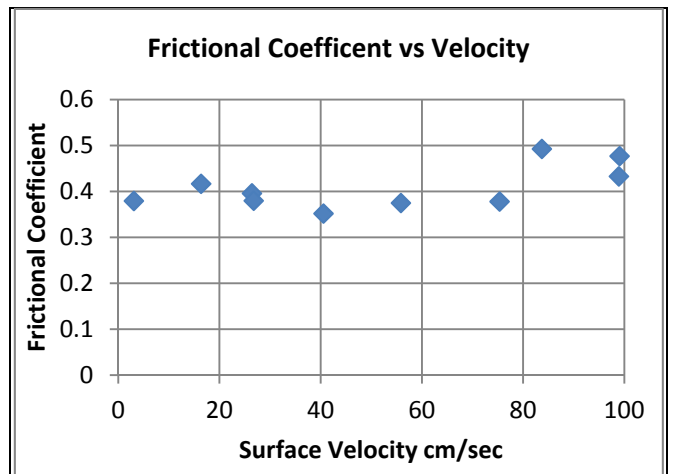
Figure 7: Frictional coefficient vs. surface velocity [V_b] at a preheat temperature of 83.3^{o}C, a surface temperature of 121°C and an applied load of 6.89 MPa (1000 psi).
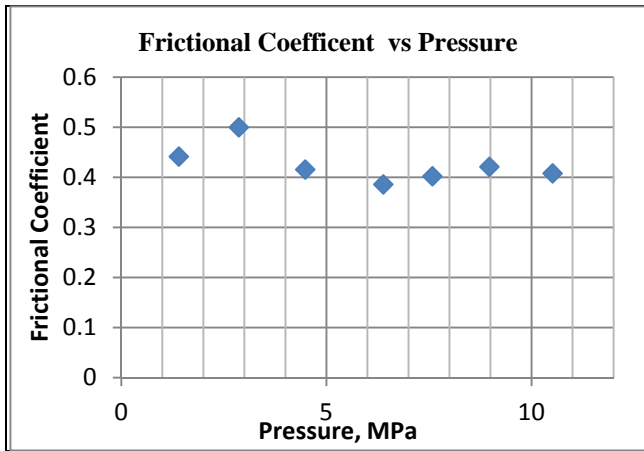
Figure 8: Frictional coefficient vs. applied load [P_0] at a preheat temperature of 83.3°C, a surface temperature of 121°C and an surface velocity of 55.9 cm/sec
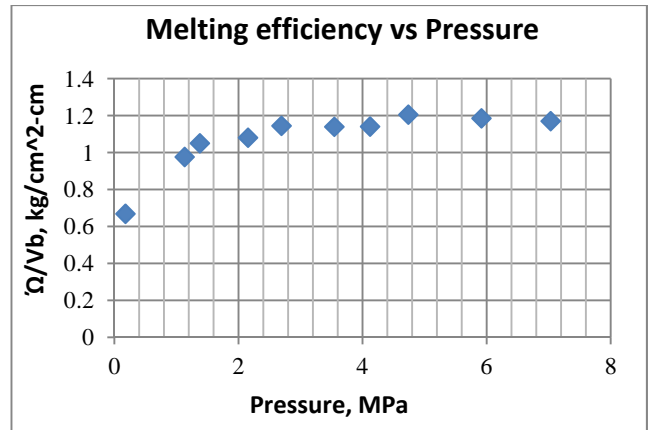

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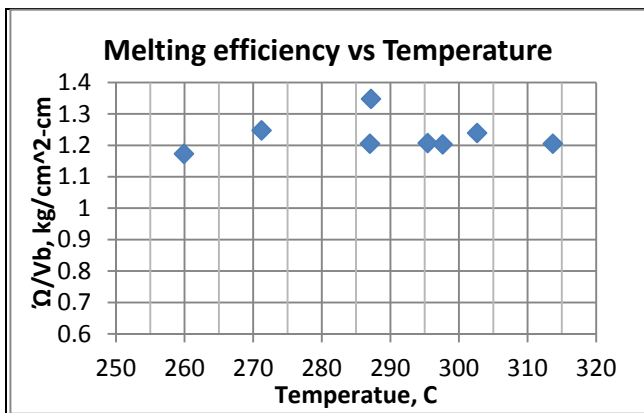

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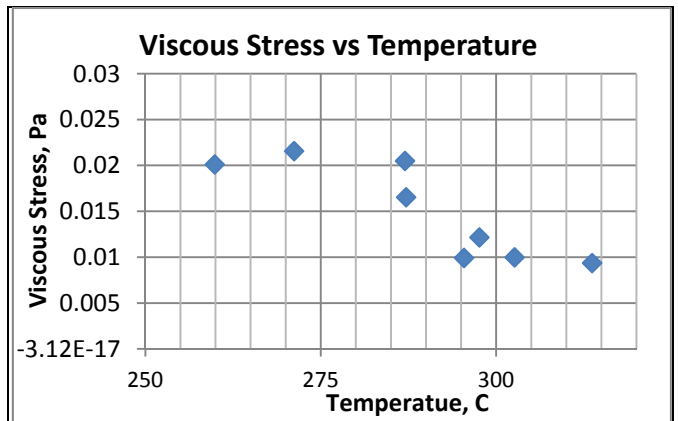

Figure 12: Plot of viscous stress Vs. Surface temperature for a solid temperature of 83.3°C, an applied load of 6.89 MPa (1000 psi) and a surface velocity of 55.9 cm/sec.
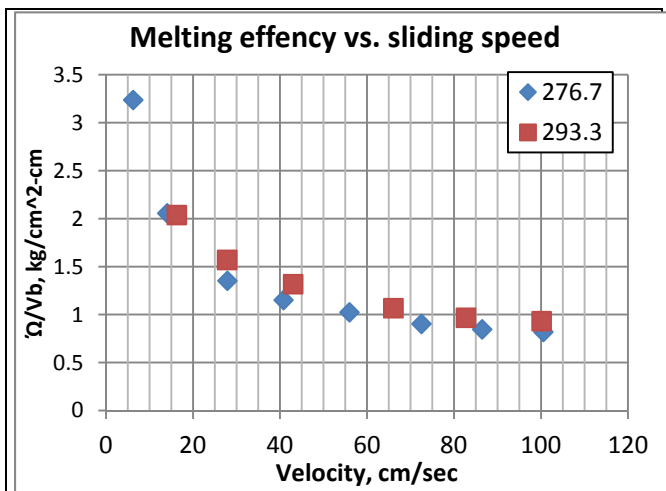

Figure 10: Plot of melting efficiency vs. surface velocity [V_b] for a solid temperature of 83.3°C, an applied load of 2.76MPa (400 psi) and surface Temperatures of 276°C and 293°C.
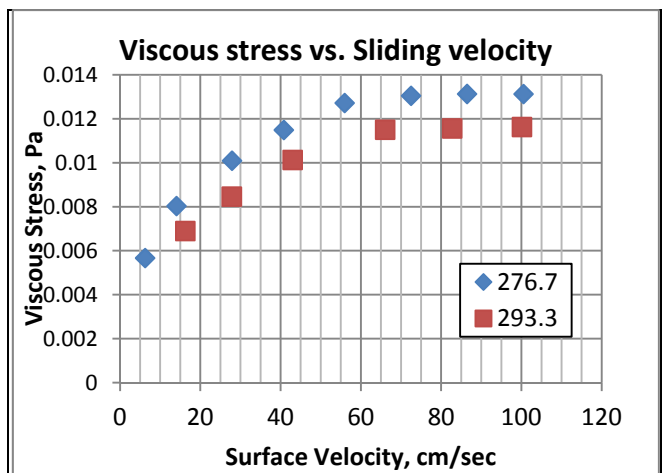

Figure 13: Plot of viscous stress vs. surface velocity [Vb] for a solid temperature of 83.3°C, an applied load of 2.76MPa (400 psi) and surface Temperatures of 276°C and 293°C.