



Tecnológico
de Monterrey

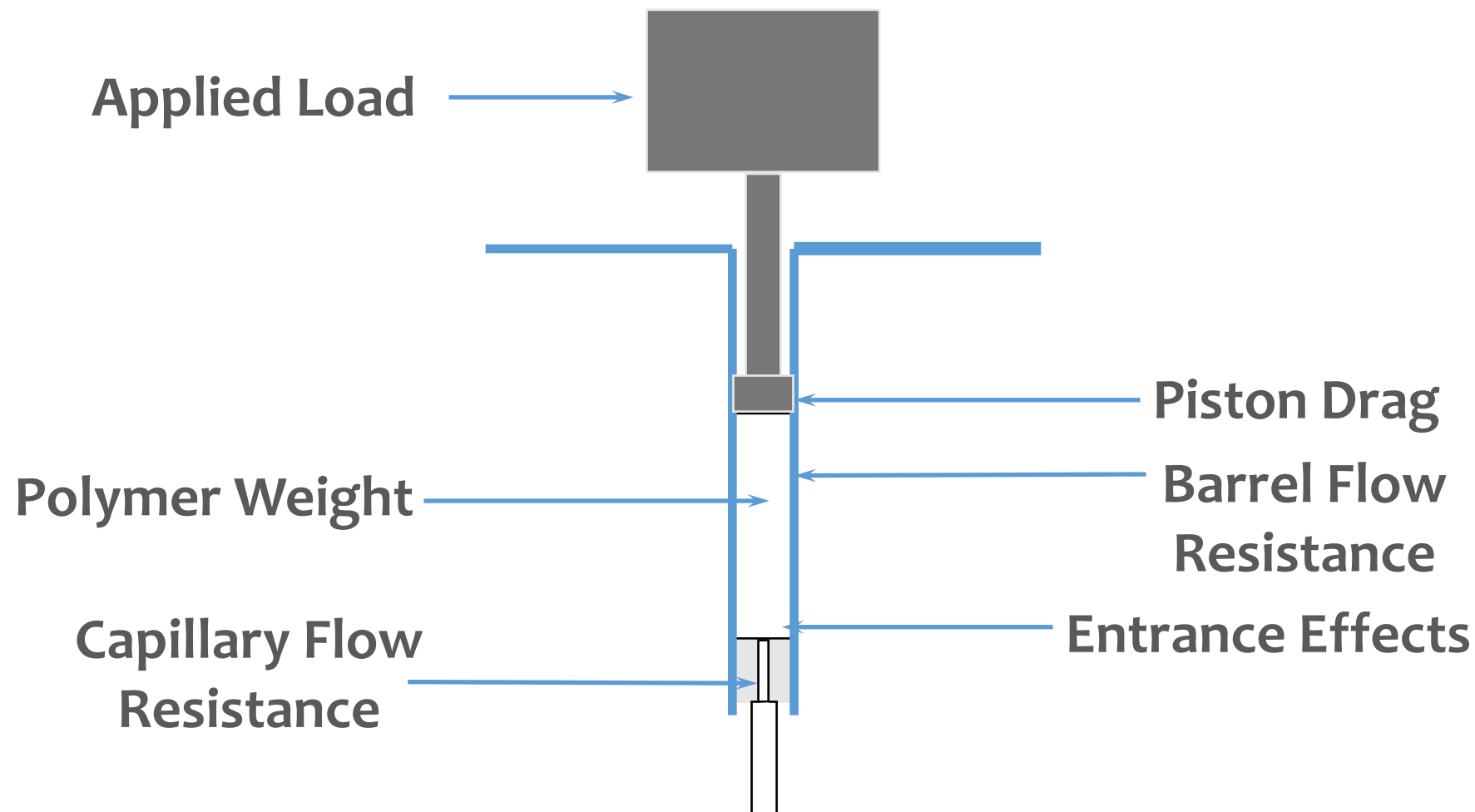


Capillary rheometry

September 23, 2019



Instron Force Balance

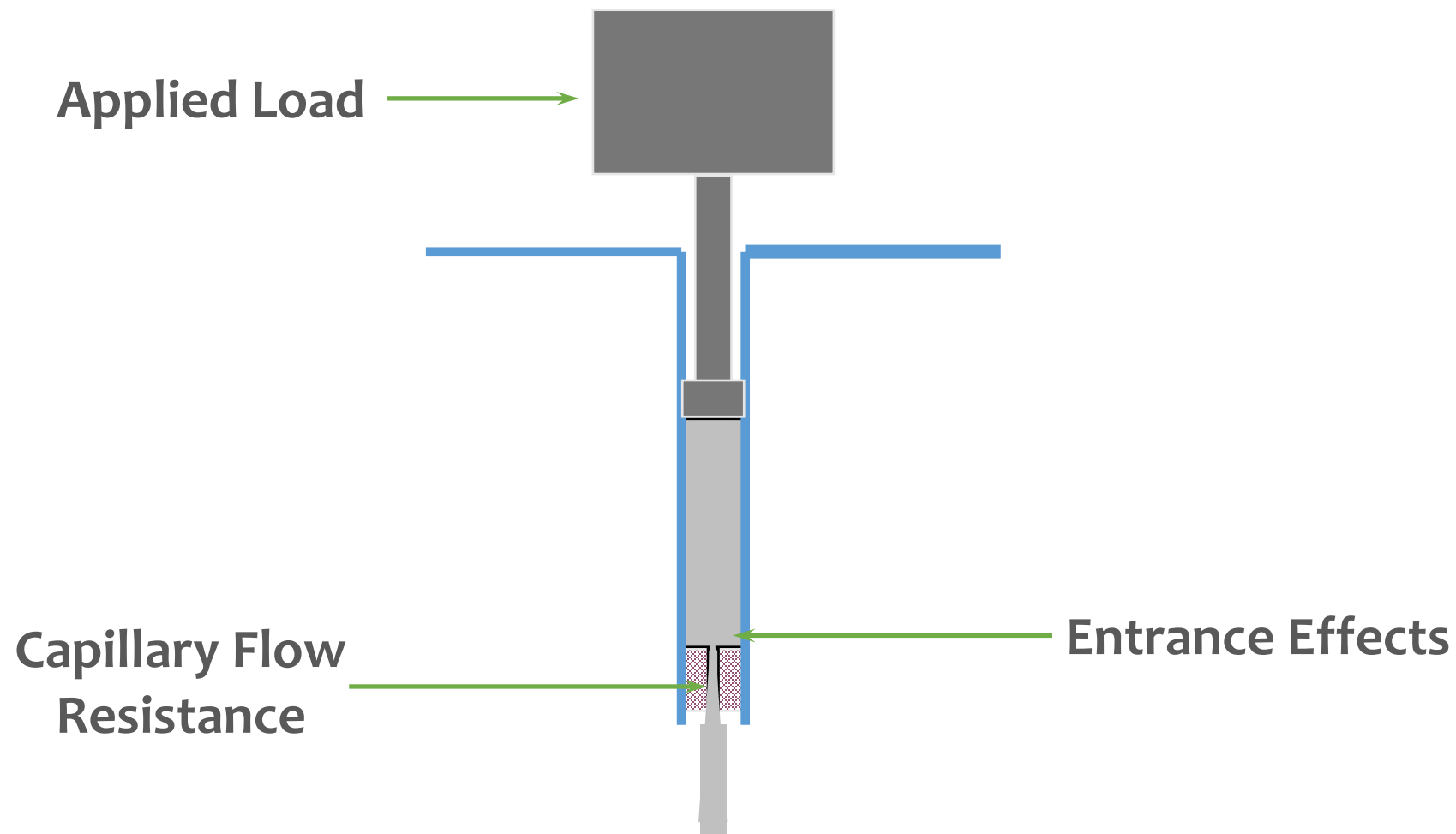


$$F_{\text{applied}} = \frac{0}{F_{\text{drag}}} + \frac{0}{F_{\text{barrel}}} - \frac{0}{F_{\text{resin}}} + F_{\text{entrance}} + F_{\text{capillary}}$$

after dividing by the appropriate areas:

$$\Delta P_{\text{total}} = \frac{0}{\Delta P_{\text{drag}}} - \frac{0}{\Delta P_{\text{resin}}} + \frac{0}{\Delta P_{\text{barrel}}} + \Delta P_{\text{entrance}} + \Delta P_{\text{capillary}}$$

Force Balance



Note: This type of equipment typically handles the information as if the liquid were newtonian and then make corrections to make the data useful for the non-newtonian nature of the polymers.

The Capillary rheometer is a velocity controlled instrument and the things you get from such type of equipment are:

**piston velocity (in/min) (P_v),
load (lbf).**

The volumetric flow (Q) is obtained as follows:

$$Q = P_v \text{ (in/min)} * \text{Piston Cross Section Area (in}^2\text{)} = \text{in}^3/\text{sec}$$

Then, the computer program uses the DIE data (geometry) to calculate the viscosity of the samples and the procedure is based on a force balance on the DIE.

The load (used for the force balance) is measured either at the top of the plunger or just above the die (all depends on the type of rheometer).

Note:

Such load is converted by the computer program to a pressure (F/A).

Afterwards, the rheometer software might give you the following information

Apparent shear rate,

Apparent shear viscosity,

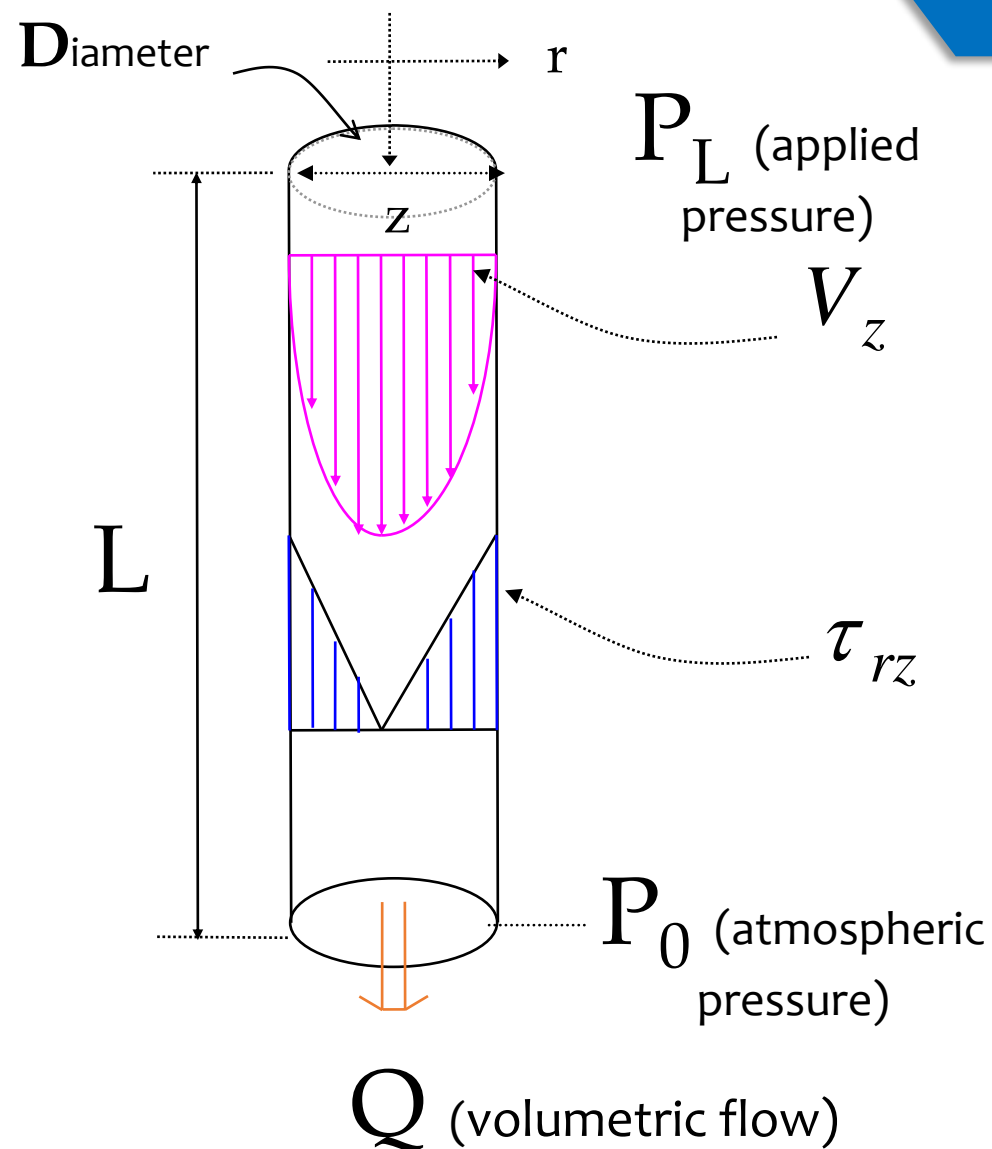
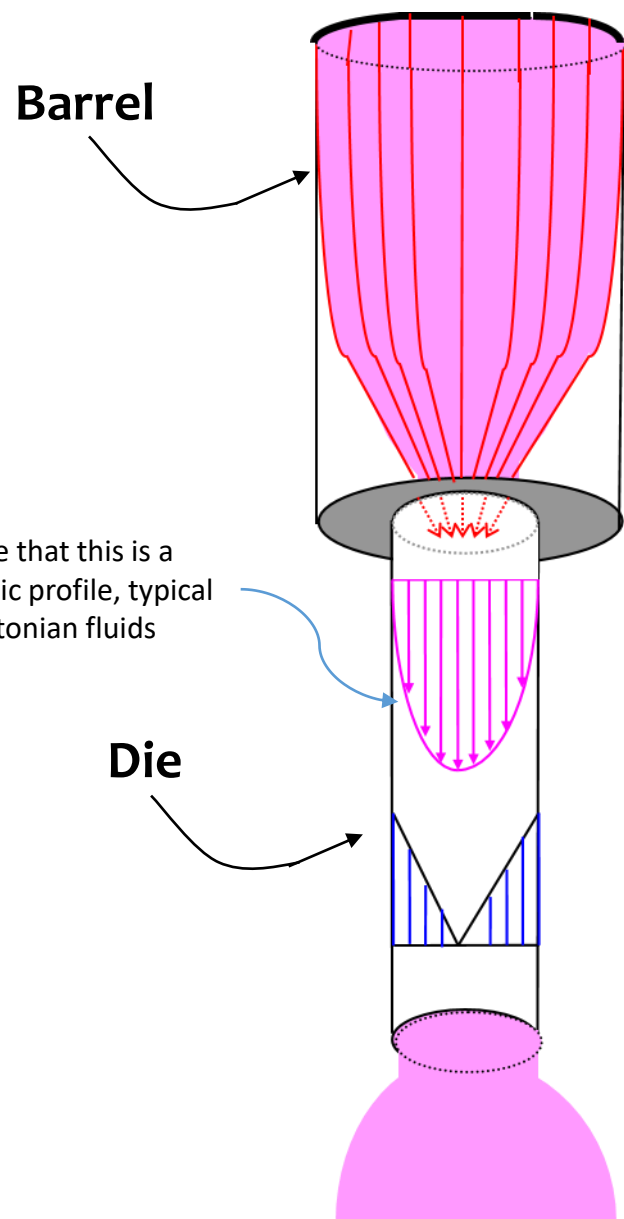
Corrected shear rate,

Corrected viscosity.

(?????)

To better understand this let's take a closer look of polymer flow in the barrel and in the die...

Observe that this is a
parabolic profile, typical
of Newtonian fluids



Remember the meaning of shear stress?

Well, that shear stress at the wall is related to the force exerted by the fluid on the barrel as well as in the die wall:

$$\frac{F_z}{A} = \tau_w \quad (1)$$

The force (F_z) is exerted by the piston on the top area of the molten polymer

$$\text{where } A_{\text{die}} = 2\pi R L \quad (2)$$

$$\text{and } F_z = \Delta P \times A_{\text{perpendicular to } F_z} \quad (3)$$

$$F_z = 2\pi R L(\tau_w) = \pi R^2 \Delta P \quad (4)$$

The stress at the wall is related to the force exerted by the fluid on the barrel's wall . Such force is the same as that exerted by the plunger which is related to the pressure head.

But, how can you relate that to the viscosity of the material and to the volumetric flow ?... Some equations will do it for you...

A momentum balance

gives:

$$\frac{d(r\tau_{rz})}{dr} = \left(\frac{P_o - P_L + \rho g_z}{L} \right) r \quad (5)$$

$$\int d(r\tau_{rz}) = \left(\frac{P_o - P_L + \rho g_z}{L} \right) \int r dr \quad (6)$$

$$\tau_{rz} = \frac{\Delta P_g}{2L} r + \frac{C_1}{r} \quad (7)$$

Since τ_{rz} can not be infinite, the value of C_1 must be zero and then...

$$\tau_{rz} = \frac{\Delta P_g}{2L} r \quad (8)$$

For a Newtonian Fluid

$$\tau_{rz} = -\eta \dot{\gamma} \quad (9)$$

and since:

$$\dot{\gamma} = \frac{dV_z}{dr} \quad (10)$$

and by substitution of equation 10 in 9,

$$\tau_{rz} = -\eta \frac{dV_z}{dr} \quad (11)$$

then the stress at the wall can be calculated using equation 8 at $r = R$

$$\tau_w = \frac{\Delta P_g}{4L} R \quad (12)$$

We can manipulate equation 11 to look for an expression for the flow rate (Q=Average Velocity x Cross sectional area)

$$-\eta \frac{dV_z}{dr} = \frac{\Delta P_g}{2L} r \quad (13)$$

And by integrating we get equation 16:

And the integration constant can be obtained by evaluating the boundary conditions: BC1: $V_z=0$, when $r=R$ BC2: $V_z=\max$, when $r=0$

$$V_z = -\frac{\Delta P_g}{4\eta L} R^2 \left[1 - \left(\frac{r}{R} \right)^2 \right] \quad (17)$$

However, the maximum velocity is a very crude way to calculate the flow rate, since the flow pattern is parabolic for a newtonian fluid, therefore we need to look for an weighted average velocity using the following equation:

$$\langle V_z \rangle = \frac{\int_0^{2\pi} \int_0^R V_z r dr d\theta}{\int_0^{2\pi} \int_0^R r dr d\theta} = -\frac{\Delta P_g}{8\eta L} R^2 \quad (19)$$

The average velocity can be multiplied by the cross section area and

$$Q = \langle V_z \rangle \pi R^2 \quad (20)$$

Substitute equation 19 into equation 20:

By rearranging equation 21,

Observe that the LHS of the equation has units of $1/s$, then the RHS should also have the same units. The RHS, except by eta, is the same as equation 12 and has units of Pa, therefore the units of eta should be equal to ???

therefore:

let's:

then:

Remember that this treatment is for a Newtonian fluid but at the shear rates at which we measured the polymers in the capillary rheometer, the flow is non-Newtonian (as it is in many polymer processing conditions)

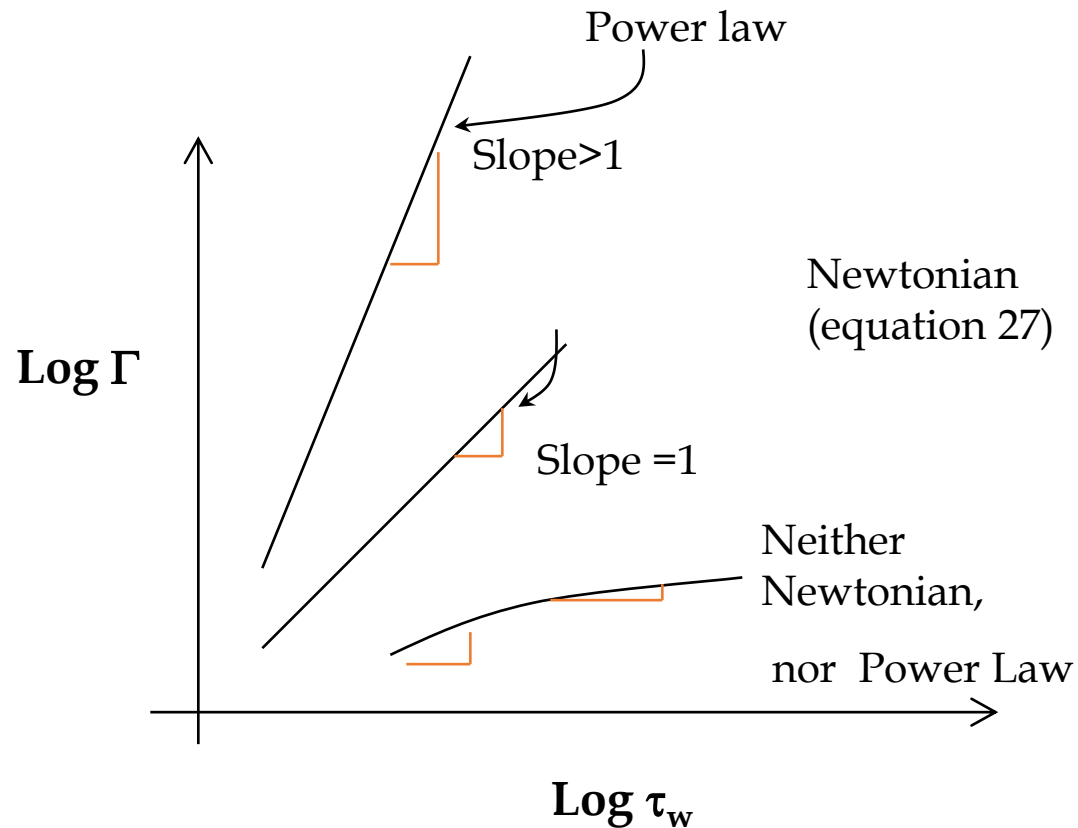
THEN

what can be done to get the data corrected?

we need to use

The Rabinowitch correction

Log of Γ is plotted against the log of $\Delta P R / 2L$ then:



The shear rate at the wall is

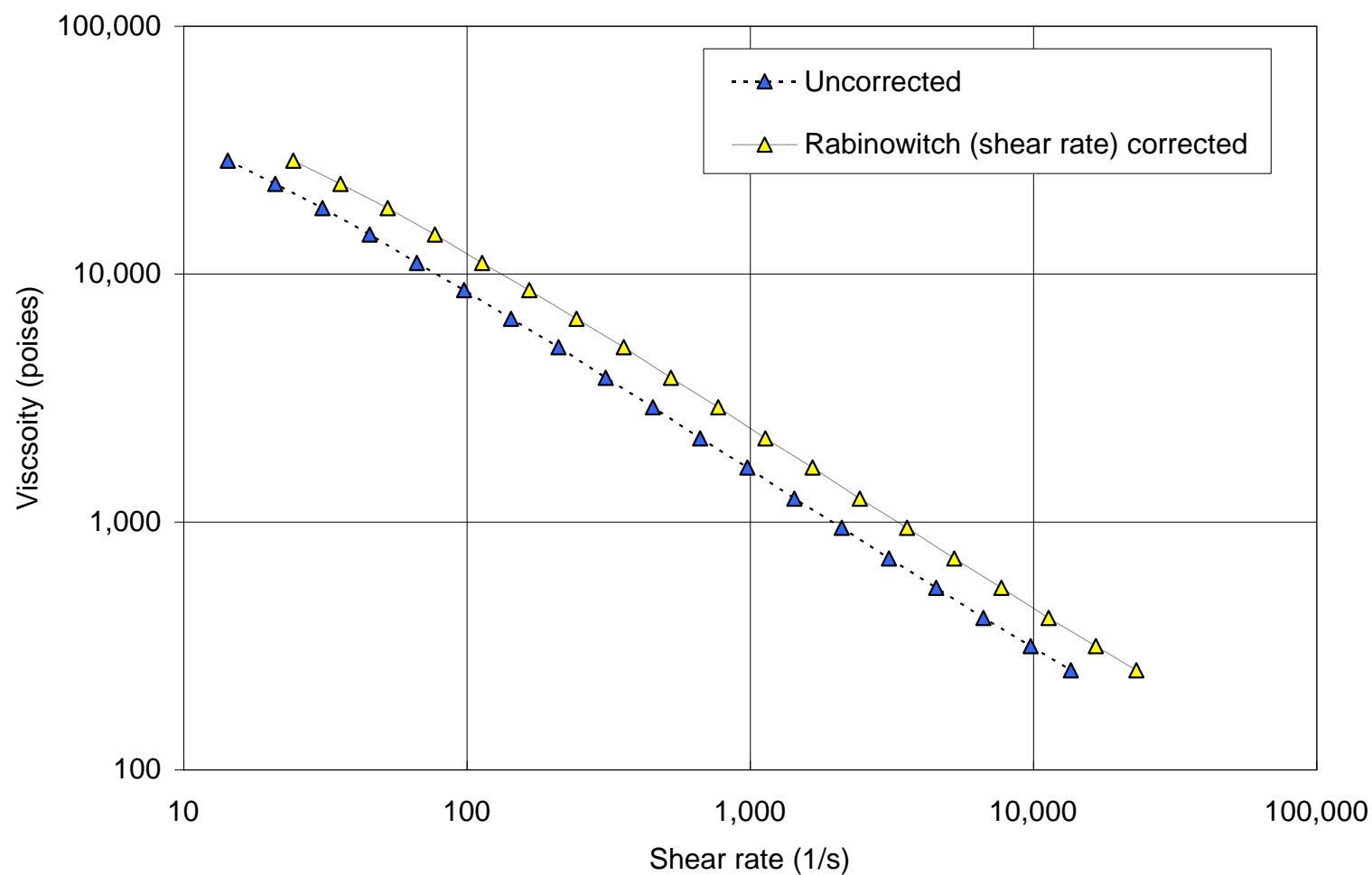
$$\left. \frac{dV_z}{dr} \right|_{r=R} = \dot{\gamma}_w$$

and is related to the apparent shear rate, Γ , by the following equation:

$$\dot{\gamma}_w = \left(\frac{3+b}{4} \right) \Gamma$$

This is the real shear rate at the wall

Rabinowitch for a PS 585 at 204°C

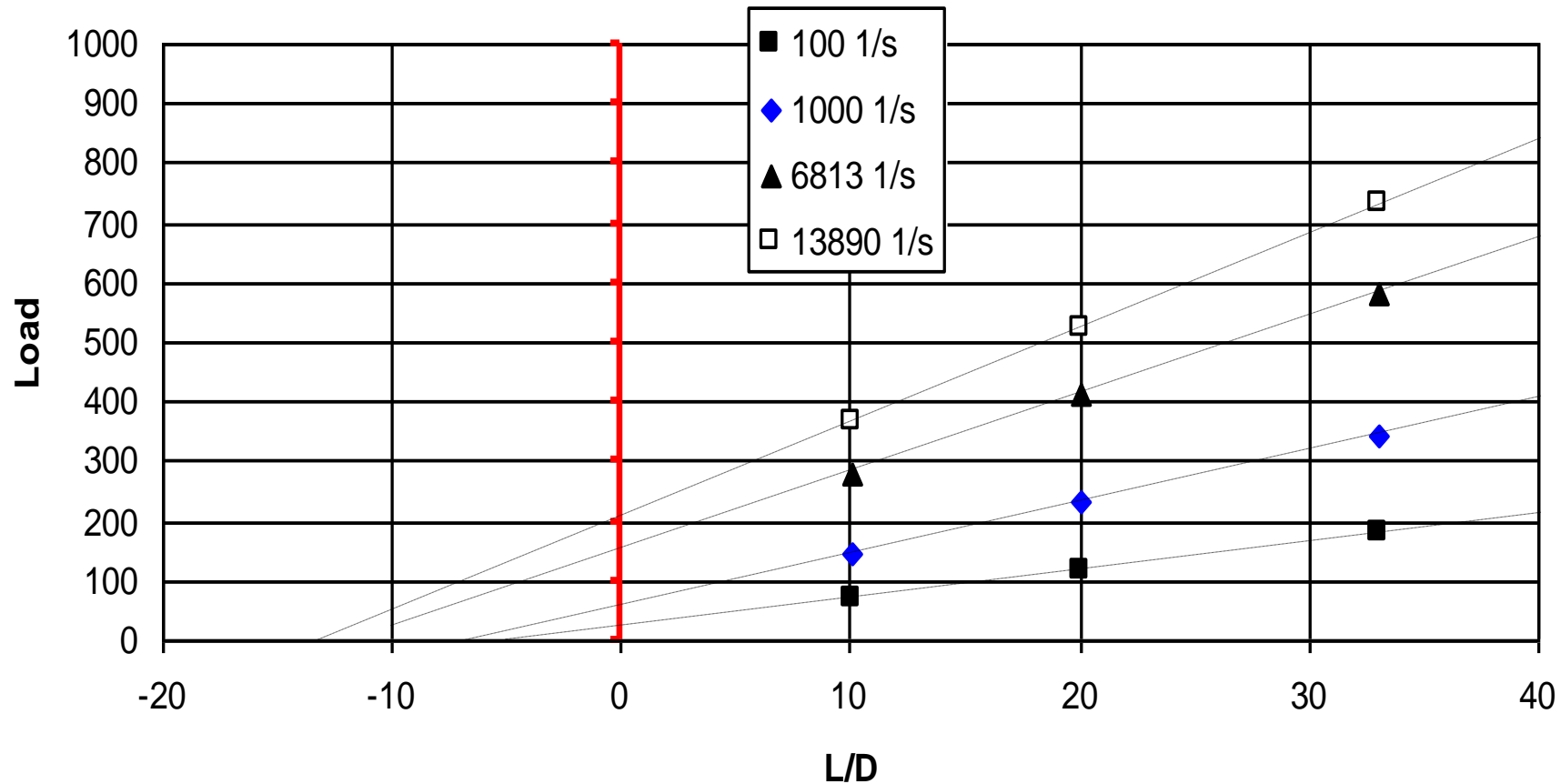


But...

This is just a correction for the shear rate. Another correction is needed because the pressure used in the math presented in the previous pages, assumes that the pressure is that due just to viscous drag while the TOTAL PRESSURE **REGISTERED** BY THE INSTRUMENT IS FOR BOTH the viscous and the _____ of the polymer.

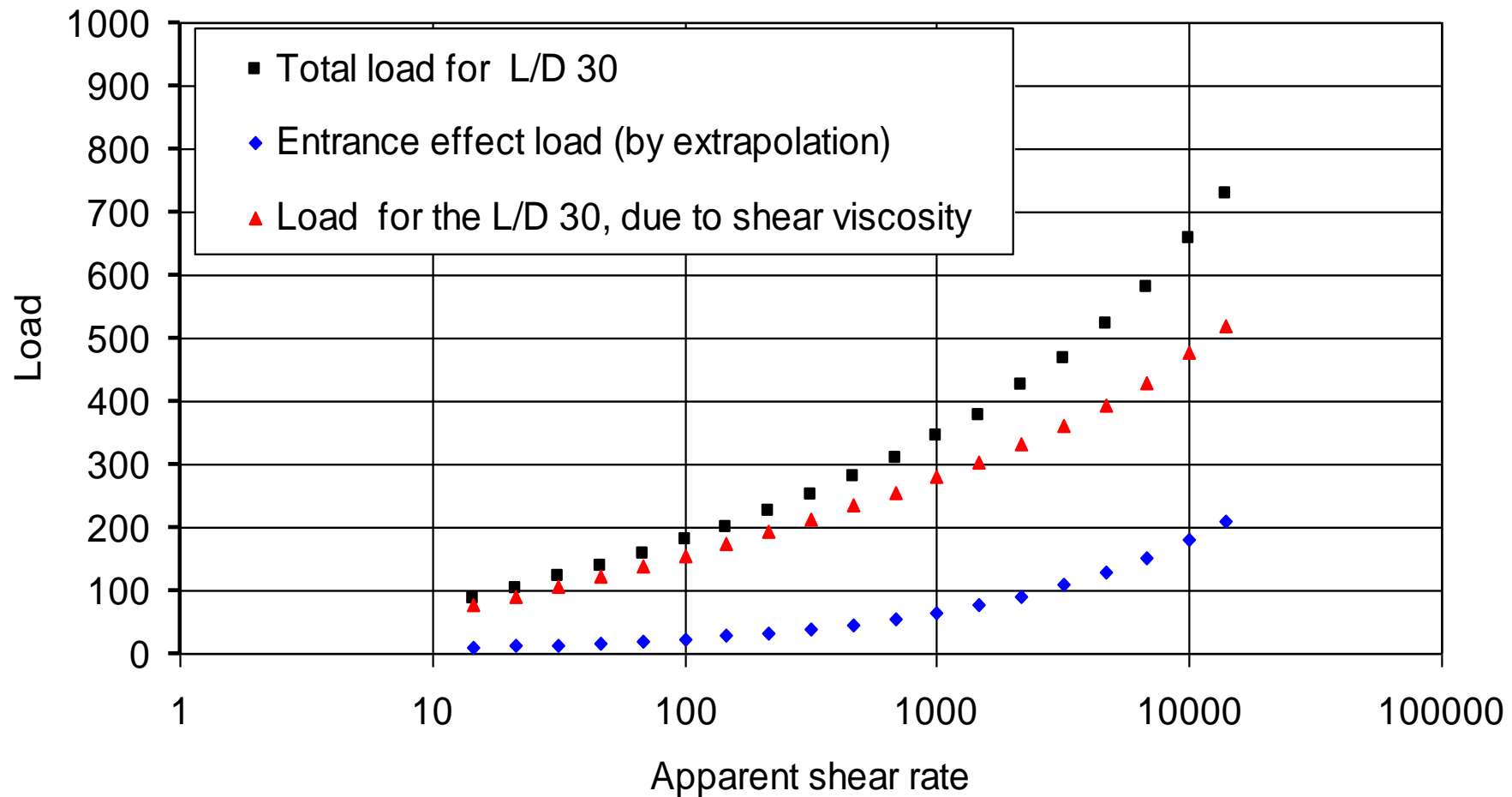
The correction needed is called the...

Bagley Correction: getting pressure entrance effects by extrapolation

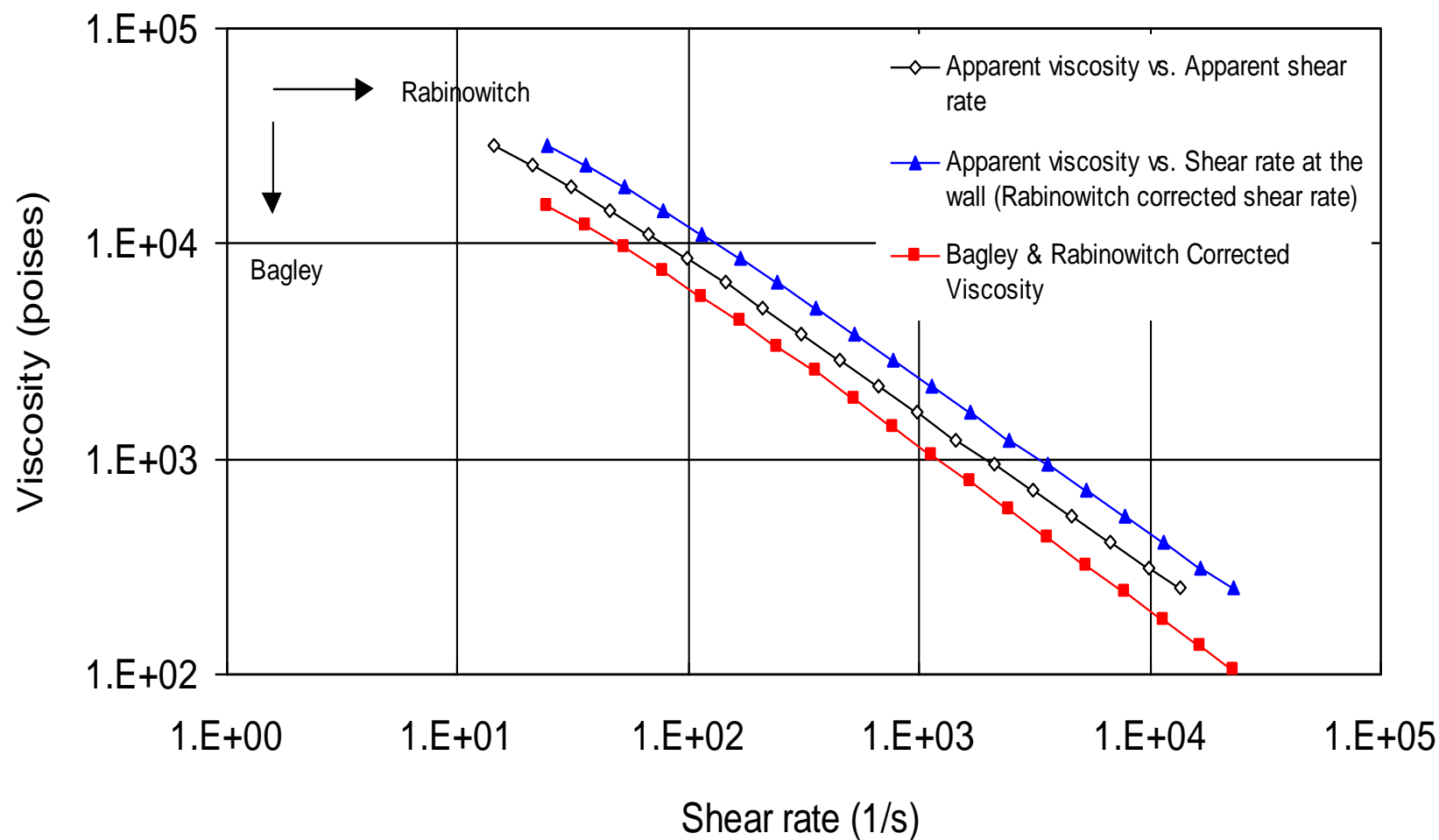


Load versus L/D for various shear rates for the PS 585 resin at 204°C

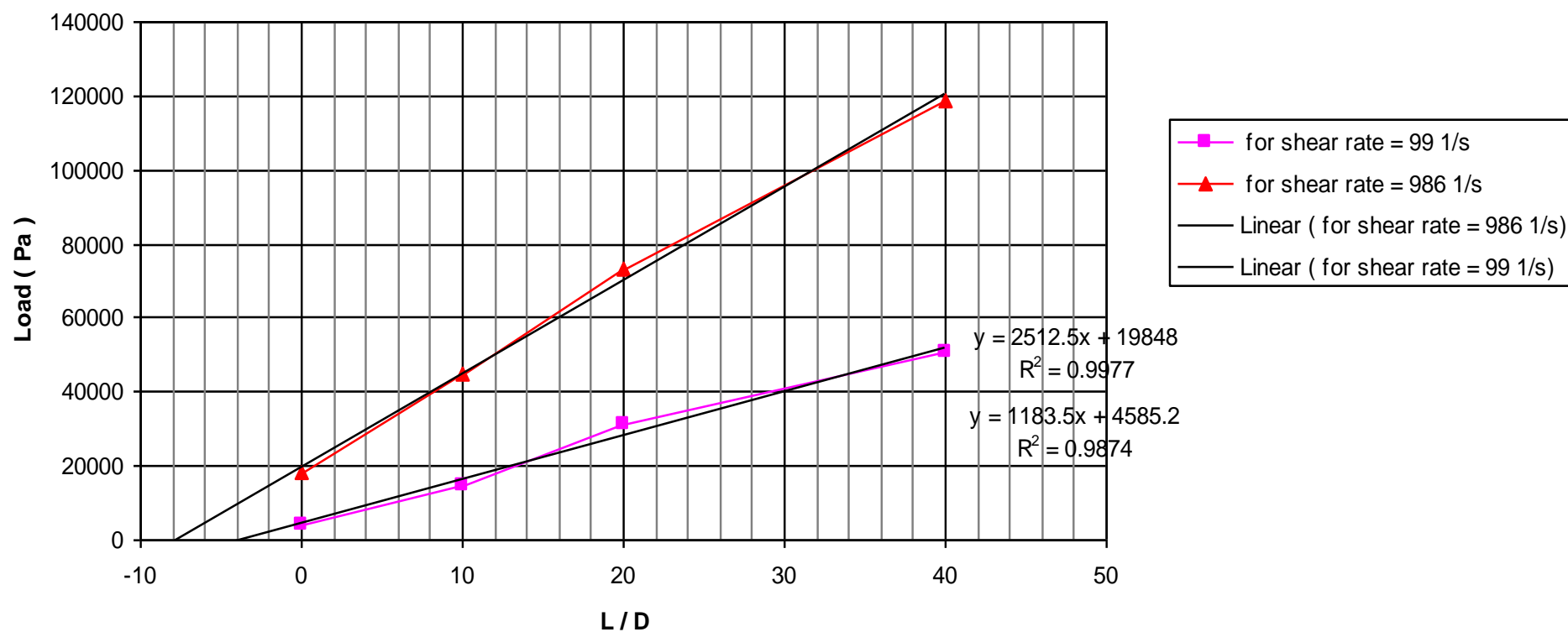
Load (lb_f) versus apparent shear rate ($1/\text{s}$)



Rabinowitch and Bagley corrected viscosity



Bagley correction for a PPRG resin with MFI 4.6.
Shear rates used for the correction: 99 and 986 1/s.
 Rabinowitch correction ignored



Please, answer the following questions:

The apparent viscosity (Γ) is obtained by multiplying the volumetric flow Q by:

Suppose you work all day doing some viscosity tests in the Instron and then you realized that while the instrument is set up for a $L/D = 40$ with a 0.05 in diameter, unadvertedly you use a $L/D = 40$ with a 0.03 in diameter.

How would that affect your results?

What would you do?

Would you throw the results away and start all over again?

What other use you can think of for the entrance pressure data?

Review

Suppose the software in the capillary rheometer computer crashes and the only thing you can read is load and piston speed velocity.

At that moment a customer has a problem and it requires capillary viscosity data. What would you do to report the viscosity with the information you get from the Instrument?

Summarize the methodology

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