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Development of an Ultra High Shear Recording Viscometer

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DEVELOPMENT OF AN ULTRA HIGH SHEAR RECORDING VISCOMETER

by

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Finally gratitude is expressed to the late Dr. R. A. Diehm in whose memory this paper is dedicated.

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CHAPTER I

INTRODUCTION

Objective

A knowledge of the rheological behavior of fluids and suspensions under severe shear conditions is important to the understanding and the success of the coating processes (1). This comes about as a result of the paper industry adopting high-speed coating methods which forced the use of coating colors with higher and higher solids contents (2). It means that the preparation, the application, the penetration, and the adhesion of coating colors are now influenced greatly by their rheology (3). Rheology is responsible for determining the flow characteristics of coatings, determining how and where films split in the application station, and determining the leveling qualities of the coatings on paper. For example, the coating color in actual operation should have a low viscosity to be smoothly and evenly applied, should be quite viscous so as to stay on vertical surfaces, and yet should flow readily under the small stresses of gravity and surface tension to level away blade or roller marks.

An instrument is needed which will determine the viscous behavior of coating colors under conditions like those found in actual mill operations. The two basic requirements for such an instrument are 1) the use of fresh liquid in the shearing zone (i.e., liquid which has not undergone work-thinning by the time its viscous behavior is determined, as found in the air knife coater), and 2) very high rates of shear, in the order of 10^5 sec^{-1} in many cases (4).

It is the objective of this thesis to develop an improved instrument taking into consideration the above two requirements. The machine will demonstrate its use as a tool for evaluating and characterizing paper coatings.

Definition of Viscosity

Rheology is the branch of science which deals with the deformation and flow of materials when subjected to an applied force, or stress. A major part of this field is the study of viscosity - the resistance of a material to flow. A material of high viscosity has a high resistance to flow and appears very "thick" like molasses, while a material of low viscosity has a low resistance to flow and is "thin" like water.

For a theoretical interpretation of the relationship between the applied stress and subsequent flow, consider a fluid confined between two parallel plates, both area A and separated by the thickness x of a material, as shown in the following diagram:

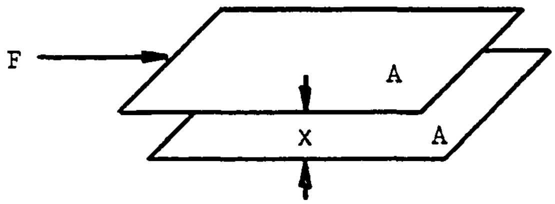

Figure 1. Force on parallel plates.

Imagine the fluid between the plates to be composed of an infinite number of layers, called lamina, parallel to the confining plates of thickness dx and area A.

If a force is applied to the upper plate, the plate accelerates and then assumes a constant velocity. The lamina immediately adjacent to the upper plate move nearly with the same velocity as the plate, but the lamina successively further from the upper plate move with successively lower velocity since the stress is less pronounced at further distances and the resistance to flow is more prevalent. This type of flow in which there is a regular gradation of velocity in passing from one lamina to the next is called laminar flow. The lamina next to the lower plate will tend to pull it along at the same speed unless it has been held stationary, in which case the velocity of the adjacent lamina would be zero.

With this velocity gradient (dv/dx) established between the plates, the confined fluid is subject to shear. The composition of the material being sheared brings viscous effects into the system which exert a drag on the faster moving upper plate. Therefore, the applied shearing stress must be large enough to overcome the viscous drag for flow to result. Thus, the viscous drag is a resistance of the fluid to flow under stress, and viscosity is really a measure of viscous drag. The stress which is necessary to maintain the constant velocity of the upper plate is directly proportional to the velocity gradient between the plates and is, therefore, dependent on the relative rate of motion between the plates and the distance (x) between them. A higher rate of motion between the plates and fluid or a smaller distance between the plates increases the rate of shear. Therefore, using the above considerations, viscosity may be expressed by the following basic equations:

$$F/A = \eta \frac{dv}{dx},$$

where η = proportionality constant, the coefficient of viscosity
F = stress (force) applied to maintain a constant shearing velocity gradient (dynes)
A = area being sheared (cm^2)
$\frac{dv}{dx}$ = rate of shear (velocity gradient)
v = velocity (cm/sec)
x = distance between lamina (cm)

$$V = \frac{\pi (d/2)^2 \; (p_1 - p_2)}{8 \eta l},$$ known as Poiseuille's Law

where V = quantity of flow delivered/second (cm^3/sec)
d = diameter (cm)
η = viscosity in poises (gm./cm·sec)
l = length (cm)
p = pressure (dyne/cm^2 or gm./sec^2·cm)

Basic Terms of Rheology

1. Viscosity ($F/A = \eta \frac{dv}{dx}$) - Viscosity is the property of a material which resists stresses causing flow. Expressed as poises - dyne·sec/cm^2
   - gm./cm·sec

2. Shearing Stress (F/A) - The shearing stress is the force per unit area which is applied to a fluid causing it to flow. It is expressed in dyne/cm^2.

3. Shear Rate (dv/dx) - Often called velocity gradient. The shear rate is the velocity gradient between successive lamina of a material during flow. It is the difference in velocity between adjacent moving lamina of a flowing material.

4. Coefficient of Viscosity (η) - This is a factor of proportionality between shear rates (dv/dx) and shearing stress (F/A). It is usually just termed viscosity. It is proportionality factor (constant) only for Newtonian liquids.

5. Apparent Viscosity - For non-Newtonian fluids the shear rate and shearing stress are not proportional, so the ratio of shear rate to shearing stress is called apparent viscosity. It is intended to indicate that the measured viscosity varies with the shear rate.

6. Yield Value - The yield value is the minimum stress which must be applied to a material to cause flow. Newtonian materials have no yield value and flow at the slightest shear stress.

7. Poise - The poise is the basic unit of expressing the viscosity of a fluid.

   1 poise = 1 dyne·sec/cm^2

   or

   = 1 gm./cm·sec

Basic Literature of Rheology

Newton, in his 17th century writing entitled "Principia" (4), proposed theories which help define one type of fluid. He wrote:

> The resistance arising from want of lubricity in the parts of a fluid is, other things being equal, proportional to the velocity with which the parts are separated from one another.

This means that the shearing stress arising from the viscosity of a fluid is proportional to the rate of shear. This shows that for water, with which Newton worked, the viscosity was just a proportionality constant. This is true though for all Newtonian fluids, upon whose flow properties most instruments are based. Newton made another statement which indicates that work with rotational viscometers in shearing a fluid in the annular gap between two cylinders is analagous to the shear of a material between two parallel plates. The circular motion which is propagated by degree is analagous to the velocity gradient previously discussed. He also brought up the tendency of the impulse of the fluid to carry the outer cylinder which is called its viscous drag. If the outer cylinder were rotated instead of the inner, this viscous motion would then be imparted on the inner cylinder tending to pull it into its motion.

A fluid is said to be Newtonian if in laminar flow η is a constant, independent of the velocity gradient or rate of shear. For this to be true, if shearing stress F/A is plotted against the rate of shear dx/dt, a straight line is developed with slope = η and intercepting at the origin.

In rheological work there are four basic types of flow commonly encountered. These can be clearly represented by a plot of shear rate, dx/dt, versus shearing stress, F/A as seen in the following figure: (5)

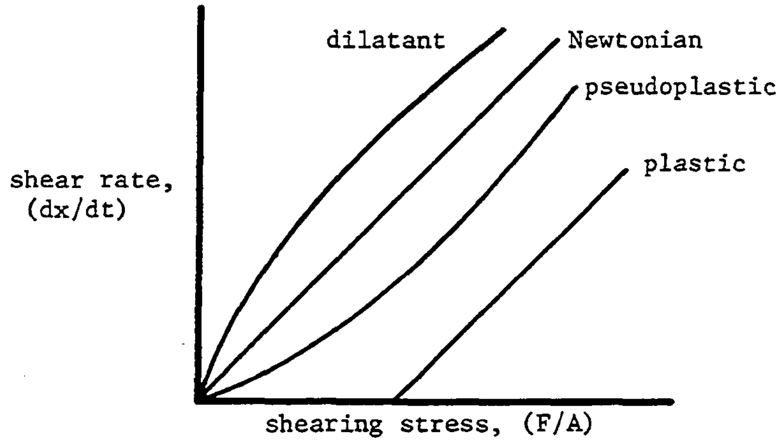

Figure 2. Types of flow.

Newtonian flow shows the flow characteristics of a Newtonian material, which was described previously. That is, for these materials, if the shearing stress is doubled, the shear rate will be doubled. However, in most industrial experiences, excluding work with water and very dilute solutions, most materials do not behave like Newtonian fluids and will exhibit more complicated flow properties.

It is evident from the plots of the pseudoplastic and dilatant flow in the preceding figure that the slope is not constant and that the viscosity is dependent on the shear rate. It is also apparent that the shear rate and shearing stress are no longer linearly proportional as with the Newtonian flow. For these materials there is no true viscosity as it will achieve different values at each different shear rate. For these materials the term "apparent viscosity" is employed to denote the viscosity value at a given shear rate.

Materials exhibiting plastic flow characteristics, sometimes referred to as Bingham plastic flow, will not flow until the applied shearing stress exceeds a certain minimum value called the "yield value". However, once the yield value has been exceeded, the material acts Newtonian with the rate of shear and the shearing stress directly proportional. This results in a constant viscosity, whose value is determined by the slope of the linear portion of the plastic flow plot in the preceding figure.

Rheopexy and thixotropy are two more ways in which the viscosity of a material is portrayed. These two quantities are time dependent rather than shear dependent which was illustrated in the previous figure. Rheopexy means that the viscosity, η, will rise with time while thixotropy shows a fall in η with time, as shown below.

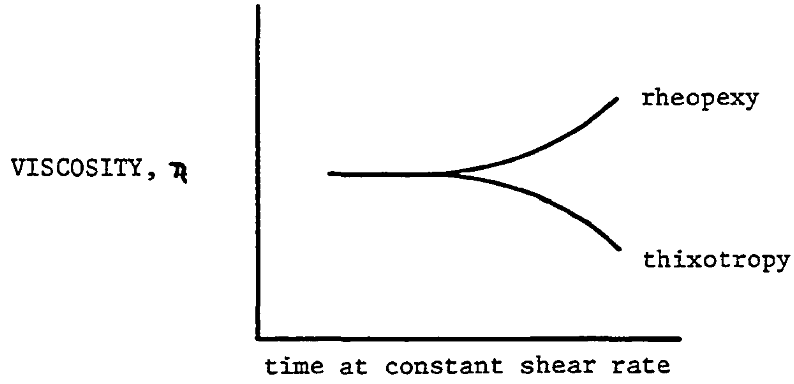

Figure 3. Characteristics of liquids.

Experimentally, it is observed that thixotropic materials become less viscous upon mixing, but then regain their original viscosity on standing. This is often called a "gel-sol-gel" transformation, and the change in flow properties is termed "thixotropic breakdown".

Rheology in Paper Coating

All of the above introduces the reader to rheology. It connects rheology and paper coating, and indicates that study is needed to obtain the proper flow of the paper coating in order to insure uniformly coated paper. Without knowledge and control of flow behavior, coatings will vary considerably, wasting manpower, equipment and raw materials. In addition, the feeling of a job well done will be lacking.

CHAPTER II

LITERATURE REVIEW

Significance of Rheology to Coating

> "We can only guess what men thought and did by the results they obtained throughout the eons of prehistory. For instance, in order to make the many artifacts of pottery which are found today, primitive peoples must have had a knowledge of the working properties of various clays and effects of adding more or less water. True, this knowledge need not have been more than required by present day housewives, but who knows what thoughts unknown rheologists had?" (6)

Indeed, whatever thoughts previous generations had, a foundation was laid which paved the way for more experimentation and the accumulation of knowledge on the subject of rheology as it applies to paper coating. It has been known for a long time that the flow behavior of coatings must be correct in order to insure proper coating of paper commercially (1,7). Smith, Trelfa and Ware (8) were among the first to recognize this. And Green (9) pointed out that many instruments and various methods are available to measure and define flow properties.

Unfortunately, Brookfield viscometer measurements are not indicative of the viscosities encountered in the high shear conditions of a blade coater. Higher shear rate data is necessary in order to regulate the coating conditions for proper operation of a blade coater (10-11). For example, experiments with a laboratory blade machine running at up to 3000 rpm have created rates of shear of 10^4 to 10^6 sec^{-1}. A study by Windle and Beazley has indicated that measurements of color viscosity show that the time factor in the measurement is not as important as the rate of shear at which the measurement is made (12). They have also shown that customary measurement of color viscosity is a reasonable means of controlling hydrodynamic forces when a color passes through a nip of a blade coater (13).

Knowledge such as this can only aid the investigator in finding new and improved ways to coat paper. Thus it can be said that previous literature is a definite boon to the investigating scientist.

Instruments for Rheology

Various instruments have been and are being used to measure rheological properties. Following are some of the more common viscometers and a description of each.

MacMichael

The MacMichael viscometer employs the principle of rotational viscometry as straight forward as possible. The cup rotates (1-50 rpm) and the viscous drag is transmitted to the bob which is suspended by a torque wire. Deflection of the wire, which is measured in degrees MacMichael (290), results in a direct measurement of torque since there is no friction developed. Although this simple, inexpensive instrument permits very precise viscosity measurements, its use as a tool for rheological studies of coating mixtures is limited. Shear rates developed are extremely low and temperature control is difficult.

Hercules

The Hercules viscometer was designed specifically for use with pigment suspensions. Its chief limitation is lack of temperature control. The design is such that the instrument gives a continuous curve of shear stress versus rpm. Power is supplied to a rotating bob immersed in a cup of a given radius through a variable, continuous speed drive. The viscous drag caused by rotation of the bob is sensed by two coiled springs attached to the cup. A pen, which is affixed to a string from the cup, makes a tracing (x-axis) on the recorder which moves in the y direction with rpm. Viscosity is readily calculated from the following equation:

$$n = \frac{9.55TS}{rpm}$$

where T = torque (deflection in cm x spring constant)
9.55 = instrument constant
S = bob constant

Stormer

This is one of the few instruments in which the shear stress rather than the rate of shear is held constant. Stress is applied by attaching weights to a string connected to a drum, which in turn is connected through gears to the rotating bob. The gear ratio is 11:1, thus the torque applied to the rotor is:

$$M = \frac{R_b \times 980}{11} \times W$$

where M = torque in dyne cm
R_b = radius of the bob
W = weight applied in grams

although the torque can be readily obtained, the complex design of the rotors and cups precludes mathematical interpretation of shear rate. Consequently, constants for the calculation of viscosity must be determined experimentally with standard oils for each cup and rotor combination.

Rotovisco

The Rotovisco viscometer is considered by some to be one of the most versatile instruments currently on the market. It not only has a variety of cups and rotors, but it is also equipped with a cone and plate system, and a system to measure visco-elasticity. Shear rates can be varied from 10^{-2} to 10^{4} sec^{-1}.

Although sufficient dimensions are not supplied with the cone and plate attachments, it is presumed that the rate constants are derived from the equation

$$dv/dx = W \text{ ctn } \alpha$$

where W = angular velocity in radians/sec
α = angle formed between the plate and cone in radians.

This provides nearly a true expression for the rate of shear, since this system produces essentially the same constant rate of shear throughout the gap. Consequently, the shear rate is not dependent on the rheological behavior of the material under study as is the case with rotational viscometer cups and bobs. However, care must be exercised in the use of the cone and plate to assure that the sample does not dry out during the measurement. Also, the system is limited to suspensions having particle size below 20 microns in diameter.

Brookfield

The Brookfield viscometer, because of low cost, ease of operation, and portability has become one of the most widely used viscosity instruments. It is a rotational instrument that measures the viscous drag produced on a spindle rotating at a constant speed in the sample being tested. Viscosity is readily obtained by applying a factor to the torque reading obtained directly from the dial. Although it is widely used and is considered a good control instrument, it has a serious limitation. Cup size is not specified, i.e., it is designed to operate in any size container. Consequently, the annular gap, which is vital to accurately calculating shear rate, is not known. Numerous equations have been developed for the "infinite" annular gap of the Brookfield. However, they yield, at best, only an approximation of the actual shear rate.

Ferranti-Shirley

The Ferranti-Shirley viscometer is a cone and plate instrument that is highly desirable for the measurement of the properties of non-Newtonian materials. It is equipped with good temperature control and provides a curve of torque versus rpm from an auxiliary recorder. A major advantage of the Ferranti-Shirley viscometer is that the rate at which shear is applied can be varied rapidly.

Viscosity is easily obtained from the plot of stress versus rate from:

$$\eta = \frac{K \cdot S}{rpm}$$

where K = cone-spring constant
S = stress in dyne-cm.

Rate of shear is determined by the equation:

$$dv/dx = \frac{W}{\alpha}$$

where W = angular velocity in radians/sec
α = angle formed between cone and plate in radians

Band Viscometer

The Band Viscometer, new and fairly recently developed, is capable of generating rates of shear of the order of 300,000 sec^{-1} (4). This instrument also uses fresh liquid at all rates of shear (14). Thus it is more in line with the operation of a paper coating machine (1). It eliminates difficulties such as work thinning encountered with the Brookfield or Hercules type viscometers. The new instrument uses the National Printing Ink Research Institute (N.P.I.R.I.) Band Viscometer blocks (14), and provides a complete rheogram over a wide range of rates of shear in one experimental run.

The basic apparatus consists of two highly polished flat surfaces, parallel to one another, spaced a precise distance apart. When a uniform band passes through the orifice with liquid between it and the sides, the liquid is sheared and a viscous drag is transmitted to the walls of the orifice. In this design the force transmitted to the orifice is measured. With a band velocity of v cm/sec producing a drag of M grams in the liquid, the viscosity, η, is given by the following equation (4):

$$\frac{M \cdot 980}{2 \cdot 1.02 \cdot 1.50 \cdot (2.54)^2} = \eta \cdot \frac{v}{0.001 \cdot 2.54}$$

where the numerator on the left hand side is the total drag force in dynes, the denominator is the total surface area in cm^2, and the denominator on the right hand side is the liquid film thickness in cm.

CHAPTER III

EXPERIMENTAL

Experimental Design

Developing the Band Viscometer and improving Abitibi Paper Company's Band Viscometer consisted of four important stages. These included acquiring the ready made parts of the instrument, forming and constructing a framework to hold the working parts, connecting the various parts so the instrument was operational, and calibrating the instrument.

After that, standard solutions of varying viscosities were used to calibrate and correlate the Band Viscometer, the Hercules viscometer, and the Brookfield viscometer. These standard solutions were Newtonian fluids. Then two coating colors were studied using the Band and Hercules viscometers in order to determine their rheological properties.

Building the Band Viscometer

A schematic drawing of the principal parts of the Band Viscometer is shown in the following figure:

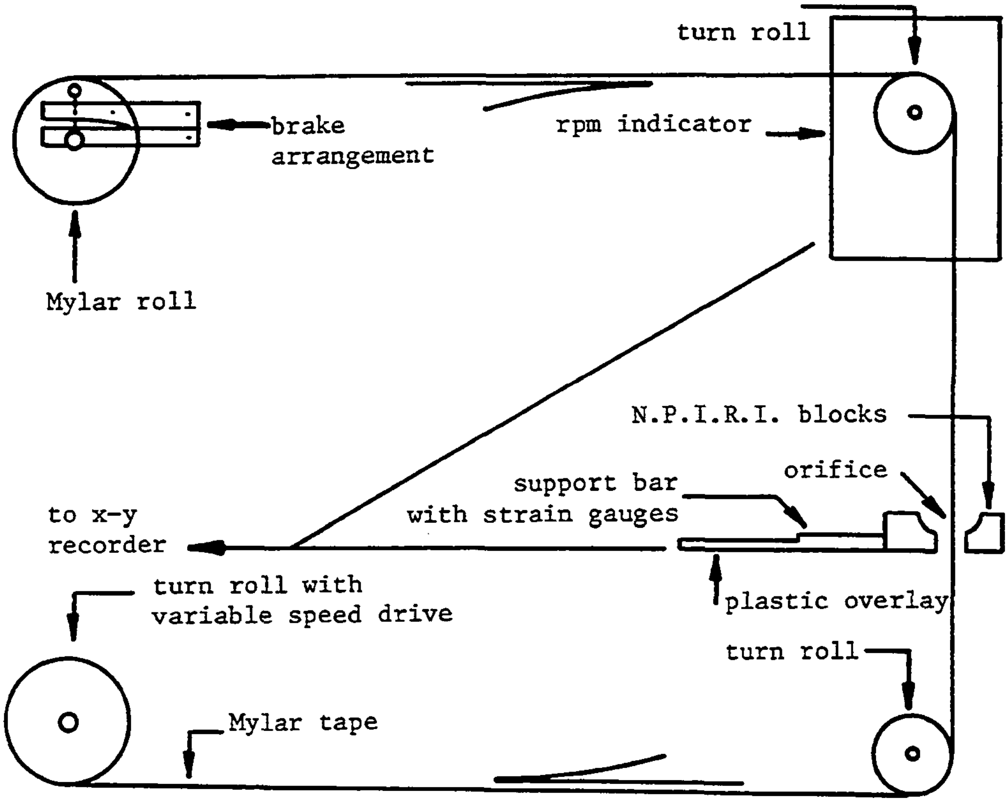

Figure 4. Schematic of Band Viscometer.

The tape moves over the first turn roll, activating the rpm indicator, then moves through the orifice. There any downward movement caused by viscous liquid is sensed by the strain gauges. Signals from the rpm indicator and the strain gauges were connected to the recorder in order to obtain a curve. A variable speed motor draws the tape from the Mylar roll through the orifice to the windup roll.

The primary part of the Band Viscometer is orifice, or nip. It consists of two very highly polished flat metal surfaces, parallel to each other at a specified distance. In the space between the metal blocks, a uniform band and the liquid to be tested pass through, causing a viscous drag which is transmitted through the liquid to the block. A sketch and picture of the nip follow.

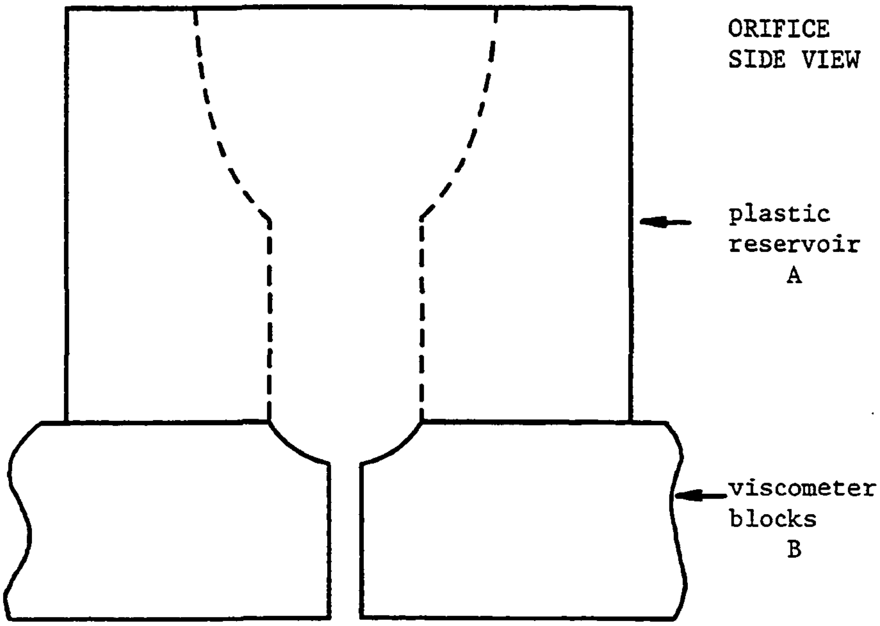

Figure 5. Sketch of viscometer blocks.

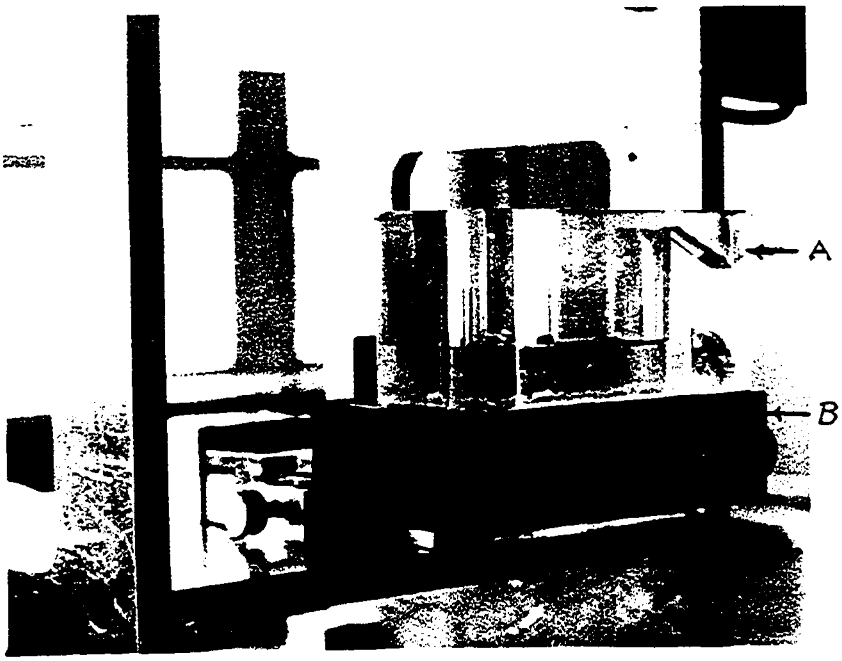

Figure 6. Photograph of viscometer blocks.

Strain gauges were purchased from Micro-Measurements, Inc., Detroit, Michigan. The four gauges were mounted on a steel bar. One end of the bar was bolted to the framework of the instrument and on the other end the orifice and reservoir were bolted. In this manner the strain gauges could sense any downward drag on the bar caused by viscous drag in the nip. The following sketch shows the arrangement of the gauges. Two gauges, one on the top and one on the bottom, are active while the other two dummy gauges complete the Wheatstone Bridge circuit and act to compensate for temperature effects.

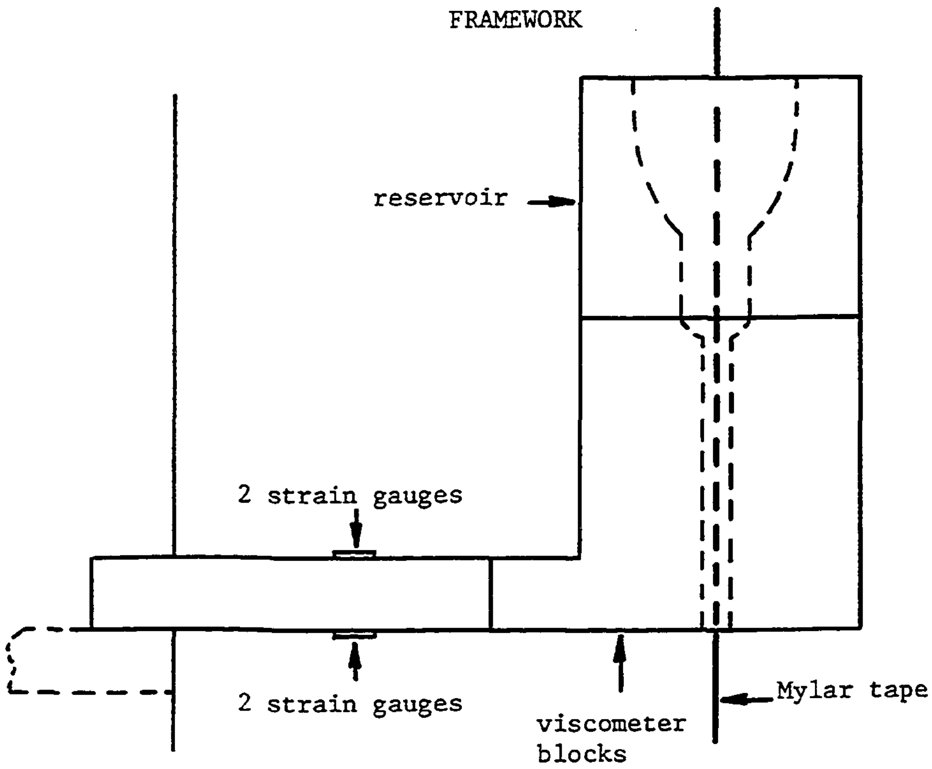

Figure 7. Sketch of strain gauge assembly.

The other variable requiring accurate sensing was the rpm of the DuPont Mylar tape traveling through the nip. This was sensed by a direct drive, Gerbing D.C. tachometer (rpm indicator). It was connected directly to a turn roll over which the Mylar tape passed just before entering the nip. The next photograph shows this.

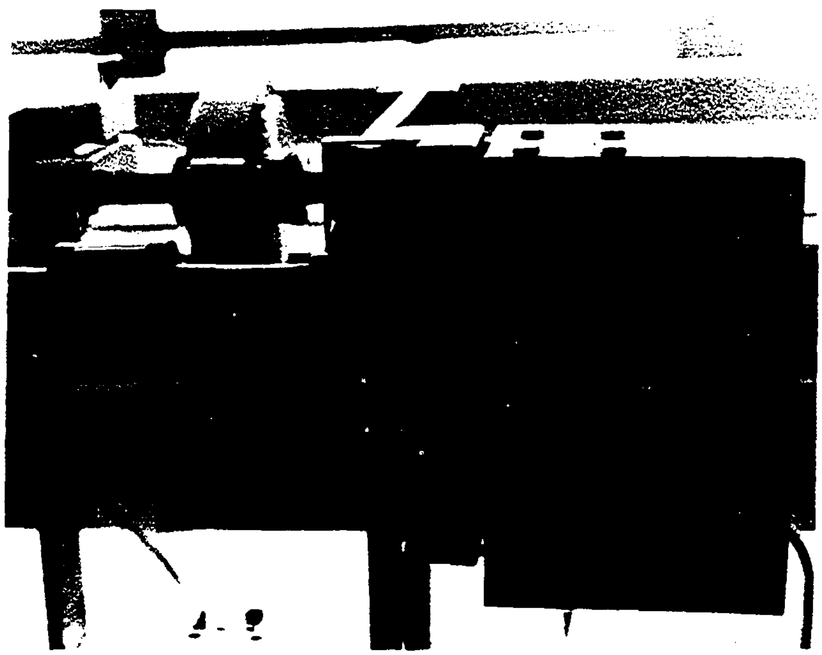

Figure 8. Photograph of rpm indicator.

Signals from the strain gauges and rpm indicator were then routed through a Honeywell Accudata 105 Gage Control Unit to a Honeywell 550 x-y plotter where they were displayed on the two axes. By adjusting the preamplifier and each of the axes of the x-y recorder, a complete rheogram over a range of shear rates was obtainable.

Figure 9 shows the physical setup of the complete Band Viscometer. The Zero Max motor, capable of 4500 rpm is on the left, then the framework with rpm indicator and strain gauges and nip, next the preamplifier, and finally on the right the x-y recorder.

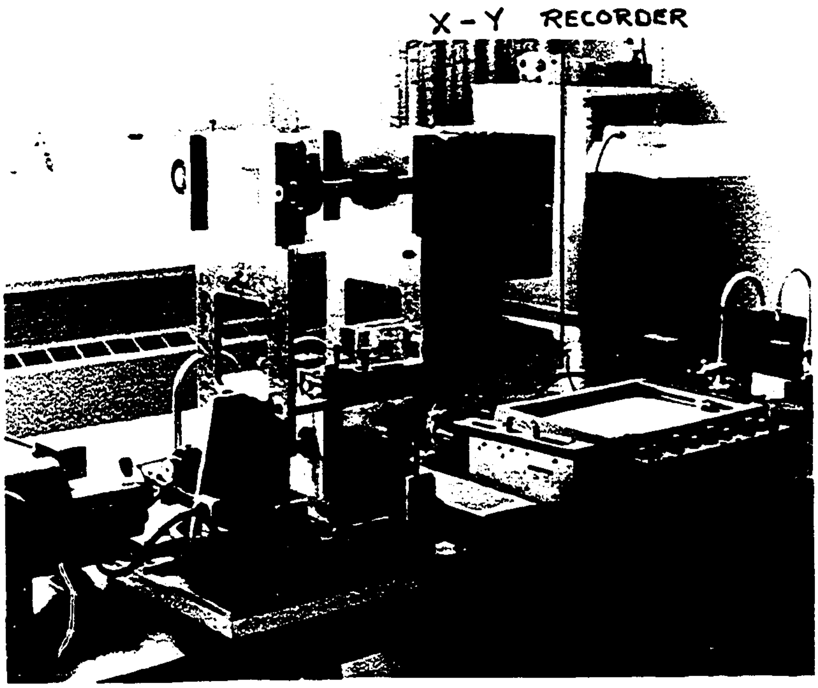

Figure 9. Photograph of Band Viscometer.

The above picture reveals the overall look of the Band Viscometer. The nip assembly was formed with two steel National Printing Ink Research Institute blocks whose sides are perfectly smooth and flat. Metal shims of 0.001 inch thickness were used to bolt the blocks together to yield the required gap. A plastic see-through reservoir was placed on top of the blocks to maintain a supply of the liquid being tested.

In actual experimentation metal shims 0.003 inch thick and DuPont Mylar tape 0.001 inch thick were employed. The space between the blocks and the tape was thus 0.001 inch. Streamline flow was generated with the incorporation of a small radius at the entering edge of the nip. The plastic reservoir also aided in creating streamline flow by helping to center the Mylar tape through the nip. This is accomplished as the shape of the entry radius causes a larger force to be applied at right angles to the direction of the tape travel on the side having the thinner liquid film.

Supporting the Mylar tape and viscometer blocks was a quarter inch thick steel framework of original design. This framework had to be designed so that the 1.50 inch wide Mylar tape and the rpm indicator could move freely. The frame had to be sufficiently massive to minimize vibrations so the strain gauges would not detect them. A number of modifications were found necessary to meet these requirements. Cut-outs made in the upright steel side panels kept the frame from becoming top heavy and allowed easier access to the gauge mounted bar and rear of the viscometer blocks. Triangular shaped steel was placed on each side of the frame to eliminate any swaying movements. These and the vertical sides were bolted to the steel base of the frame to insure strength and unity. Movement was further inhibited with the addition of a 2 inch thick lead base bolted to the bottom steel plate.

After some thought and work, it was decided to mount the motor separately from the basic frame. Connection of the motor to the rest of the unit was accomplished by a direct linkage to the tape windup reel. This decreased noise pickup by the strain gauges immensely.

Other ideas incorporated in the development of the instrument included the means for connection of the rpm indicator to the turn roll, design of the braking arrangement, the Mylar tape assembly, and the plastic overlays.

The rpm indicator was connected to the turn roll axle directly with a rubber coupler. The turn roll was located just ahead of the viscometer nip. In this way the speed of the tape as it was about to enter the nip was known.

The braking arrangement consisted of a split piece of plastic in the shape of a "V" resting on top of the axis of the roll to which the tape was connected. A thumbscrew was then placed through one arm of the "V" coming to rest at the other arm which was positioned right over the axle. As the thumbscrew was tightened, more pressure was exerted on the axle, thus keeping the tape roll from unwinding once the motor stopped.

Removing and replacing the tape roll presented another difficulty. Some arrangement was needed so that a new tape roll could be dropped into place as easily as the old roll was removed. As the tape arrived with a cardboard core with a large hole at the middle, it was decided that another core be inserted to bring the core hole down to axle size. A plastic core was made and notched so it could be tightened to the axle. Knowing that it would be difficult to remove the axle from the ball bearings, even though they were of the self-adjusting type, another method was tried and found to work. Two axles were made. One was fitted inside the other. The outer axle was cut in two and fitted with circular aluminum guides the diameter of a new roll of tape. Then this two part axle was placed in the two bearings, one tight and one moveable. The moveable half of the axle slid toward the bearing mounted on the framework, allowing the inner axle with the roll of tape to be inserted. Sliding the axle back into the closed position and tightening it down prevented the tape from moving. Because the other half of the outer axle was stationary, proper feeding of the tape through the instrument was insured.

Finally, pieces of plastic hollowed out from the center to one side were fashioned to cover the wiring needed on the framework. This included the wires to each of the strain gauges and the wires from the rpm indicator. Thus, splashing or spilling that might occur would not disturb the electrical connections. Any improvements upon these basic developments of the Band Viscometer are to be found in a later section of the paper.

Operating the Band Viscometer

The viscometer was operated in the following manner in order to obtain a rheogram:

1. Clean the machined surfaces of the viscometer blocks.
2. Assemble the viscometer with shims of the desired thickness in place. Tighten the viscometer nuts with a torque wrench to 60 in.-lbs.
3. Thread the 1 mil tape through the viscometer orifice and connect it to the motor driven take up roll.
4. Activate the speed sensor, the strain gauges, and the x-y plotter.
5. Fill the supply reservoir with the liquid sample and maintain approximate level throughout the run by manually adding additional liquid.
6. Drive the tape through the orifice at a continuously increasing speed from zero to maximum.
7. The recorder yields a plot of rate versus stress.

CHAPTER IV

PRESENTATION AND DISCUSSION OF RESULTS

Calibration of the Band Viscometer

The curves of viscosity versus rate of shear for the Brookfield Standard Oils and tap water appear graphically in figures 10 through 12. Tables I and II represent the figures numerically.

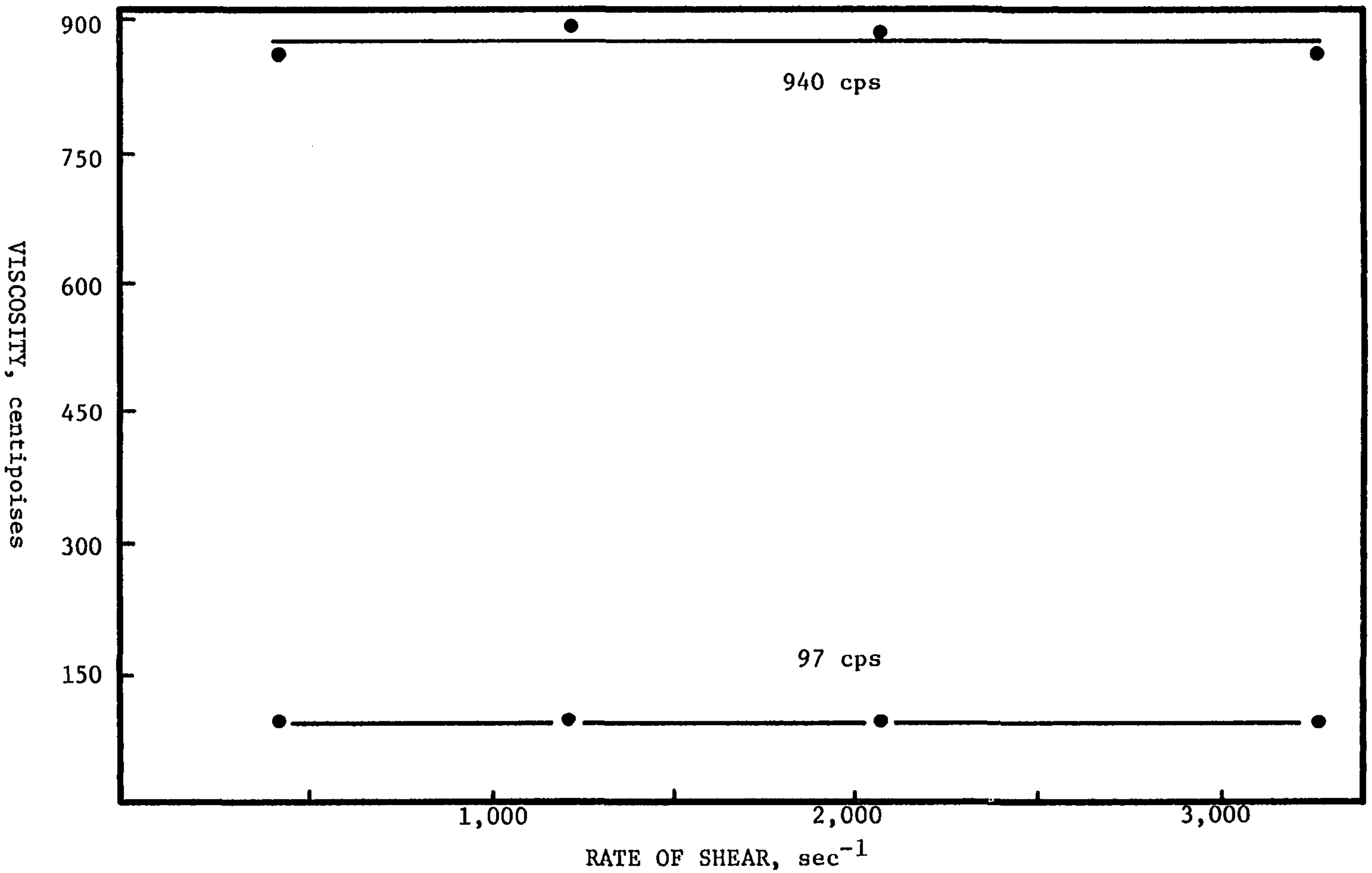

Figure 10. Viscosity versus rate of shear for Brookfield Standard Oil 97 cps and 940 cps on Hercules viscometer.

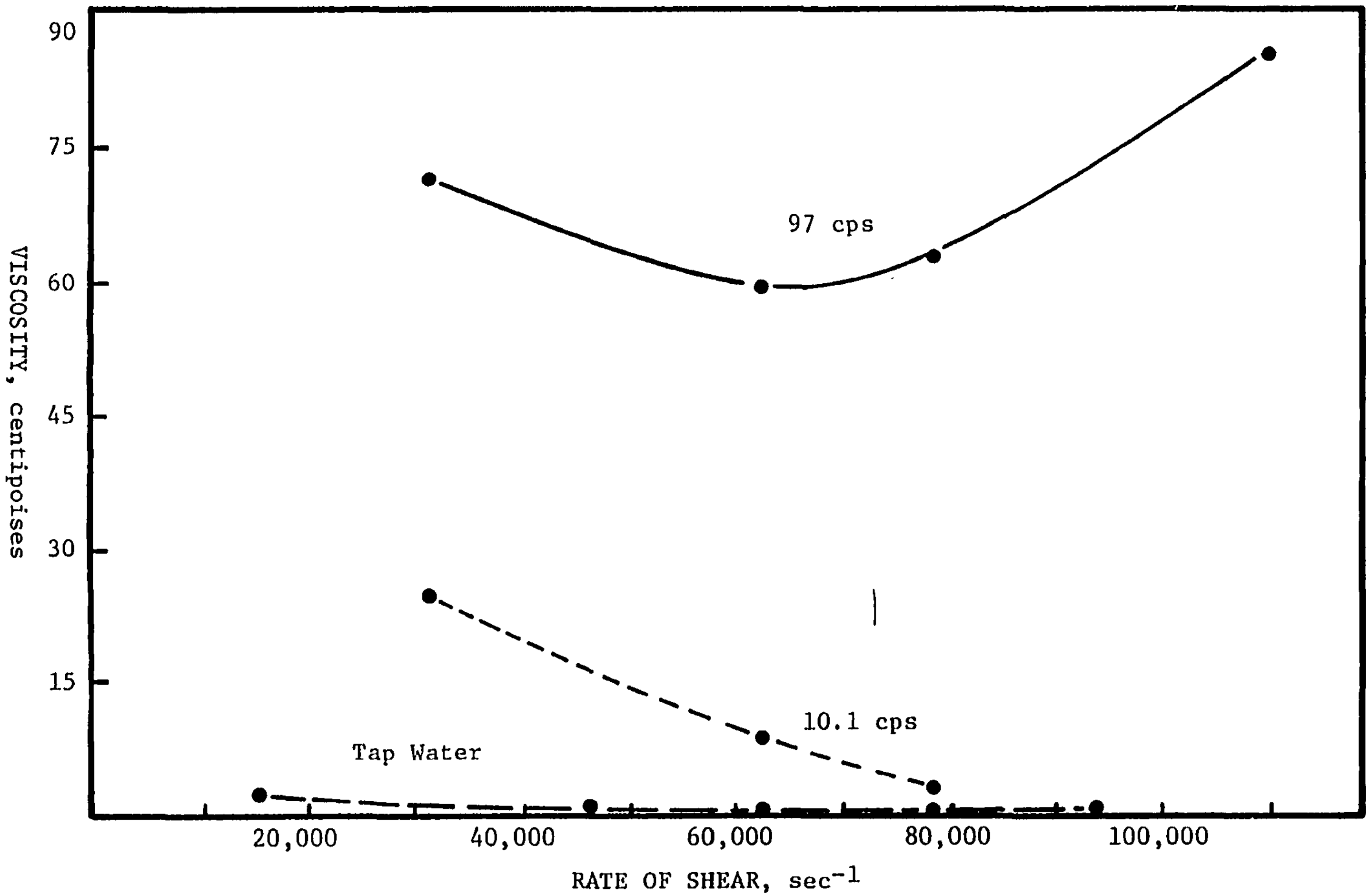

Figure 11. Viscosity versus rate of shear for Brookfield Standard Oil 97 cps, 10.1 cps and tap water on Band Viscometer.

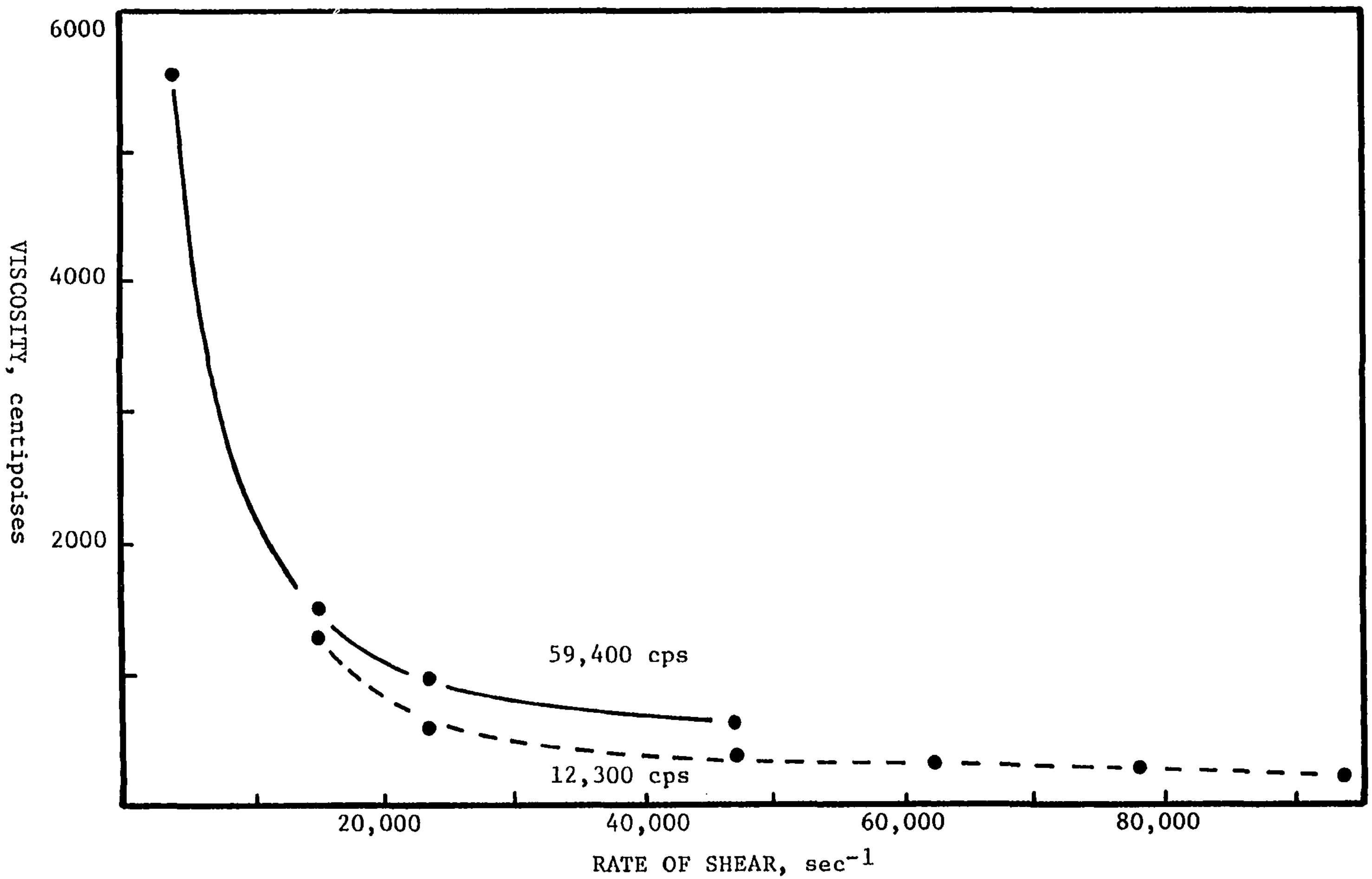

Figure 12. Viscosity versus rate of shear for Brookfield Standard Oil 12,300 cps and 59,400 cps on Band Viscometer.

TABLE I

Hercules Viscometer Results

Material Tested	Rate of Shear (sec^{-1})	Viscosity (cps)
Brookfield Standard	410	92
Oil (97 cps)	1240	92
(see Figure 10)	2070	91
	3310	92
Brookfield Standard	410	860
Oil (940 cps)	1240	890
(see Figure 10)	2070	880
	3310	860

TABLE II

Band Viscometer Results

Material Tested	Rate of Shear (sec^{-1})	Viscosity (cps)
Brookfield Standard	31,300	25
Oil (10.1 cps)	62,500	8
(see Figure 11)	78,200	3
Brookfield Standard	31,300	71
Oil (97 cps)	62,500	59
(see Figure 11)	78,200	63
	109,400	85
Tap Water	15,600	2.4
(see Figure 11)	46,900	1.1
	62,500	.8
	78,200	.6
	93,800	.6
Brookfield Standard	3,900	5630
Oil (12,300 cps)	15,600	1270
(see Figure 12)	23,500	630
	46,900	370
	62,500	320
	78,200	270
	93,800	220
Brookfield Standard	15,600	1375
Oil (59,400 cps)	23,500	970
(see Figure 12)	46,900	620

Figure 10 shows the Brookfield Standard Oil Viscosities when tested on the Hercules viscometer. No appreciable change in viscosity was noted over the shear range studied for the two oils. However, the measured viscosities were slightly lower than standard values of the Brookfield Oils. This could be attributed to a temperature difference.

The Band Viscometer results for the 10.1 centipoise and the 97 centipoise oils differed from those of the consistent Hercules viscometer results. At high rates of shear (31,300 sec^{-1} to 62,500 sec^{-1}) the viscosity decreased steadily, as shown in Figure 11. The 10.1 centipoise oil viscosity continued to decrease, but the 97 centipoise oil viscosity showed an increase. The 97 centipoise oil was the only oil run at the high shear rate of 109,000 sec^{-1}, where the viscosity increase occurred. The coating viscosity results appearing later in this paper also revealed this trend.

Tap water viscosity likewise decreased with the rate of shear. This decrease was probably caused by the increased temperature of the water tested. For as the temperature of water increases, the viscosity drops. At 25°C, the viscosity of water is 0.89 centipoises while at 40°C the viscosity is 0.66 centipoises. The discrepancy between the measured value of 2.4 centipoises and the actual viscosity of water (0.89 centipoises at 25°C) could probably be attributed to the low sensitivity of the Band Viscometer at very low viscosities. The low sensitivity occurred when as much mechanical noise as signal was plotted on the x-y recorder.

Cavitation at shear rates above 15,600 sec^{-1} in the high viscosity Brookfield Standard Oils (i.e., 12,300 centipoises and 59,400 centipoises) caused lower viscosity readings on the Band Viscometer. Cavitation was visually observed when air was drawn into the plastic reservoir and mixed with the oils before flowing through the orifice. This resulted in non-laminar flow, which is not a true measure of viscosity. Figure 12 portrays these lower viscosity values.

Rheology of Coatings

Both a low and a high solids coating were employed to compare the Hercules and the Band viscometers. Tables III and IV below show the formulation of these coating colors.

TABLE III

Low Solids Coating Color Formulation

Ingredient	Dry Weight, gm. oven-dry
Titanium Dioxide (Rutile)	120
Clay (Huber Hydra-Gloss)	480
Styrene Butadiene Latex (Dow 620)	150
Calgon	0.75 or 1.5
Water	to make 30±1% solids
Defoamer	0.6

TABLE IV

High Solids Coating Color Formulation

Ingredient	Dry Weight, gm. oven-dry
Titanium Dioxide (Rutile)	120
Clay (Huber Hydra-Gloss)	480
Styrene Butadiene Latex (Dow 620)	150
Calgon T	12
Water	to make 57±1% solids
Defoamer	0.6

The results of testing the above coating colors on the Hercules and the Band viscometers are found graphically in Figures 13-15 and are summarized in Tables V and VI.

TABLE V

Hercules Viscometer Results on Coatings

Material Tested	Rate of Shear (sec^{-1})	Viscosity (cps)
Low Solids Coating with 1.5 gm. Calgon (see Figure 13)	400	950
	1200	450
	2100	340
	3300	290
Low Solids Coating with 0.75 gm. Calgon (see Figure 14)	400	1530
	1200	870
	2100	770
	3300	720
High Solids Coating (see Figure 15)	400	6590
	1200	2170
	2100	1300
	3300	860

TABLE VI

Band Viscometer Results on Coatings

Material Tested	Rate of Shear (sec^{-1})	Viscosity (cps)
Low Solids Coating with 1.5 gm. Calgon (see Figure 13)	15,600	150
	31,300	90
	46,900	75
	62,500	65
	78,200	73
	93,800	79
Low Solids Coating with 0.75 gm. Calgon (see Figure 14)	7,800	285
	15,600	190
	31,300	115
High Solids Coating (see Figure 15)	15,600	380
	31,300	330
	46,900	325
	62,500	410

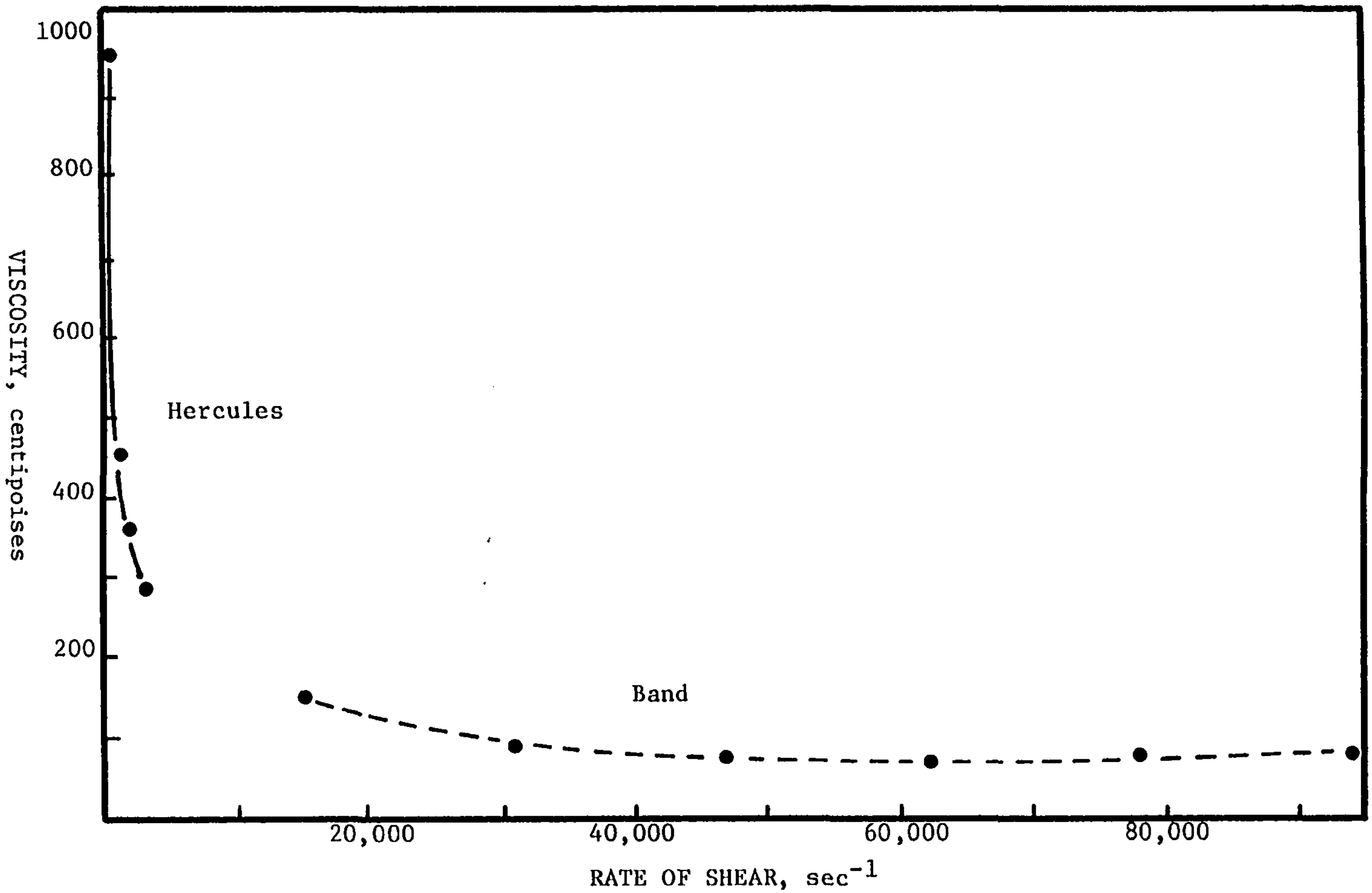

Figure 13. Viscosity versus rate of shear for low solids coating with 1.5 gm. Calgon.

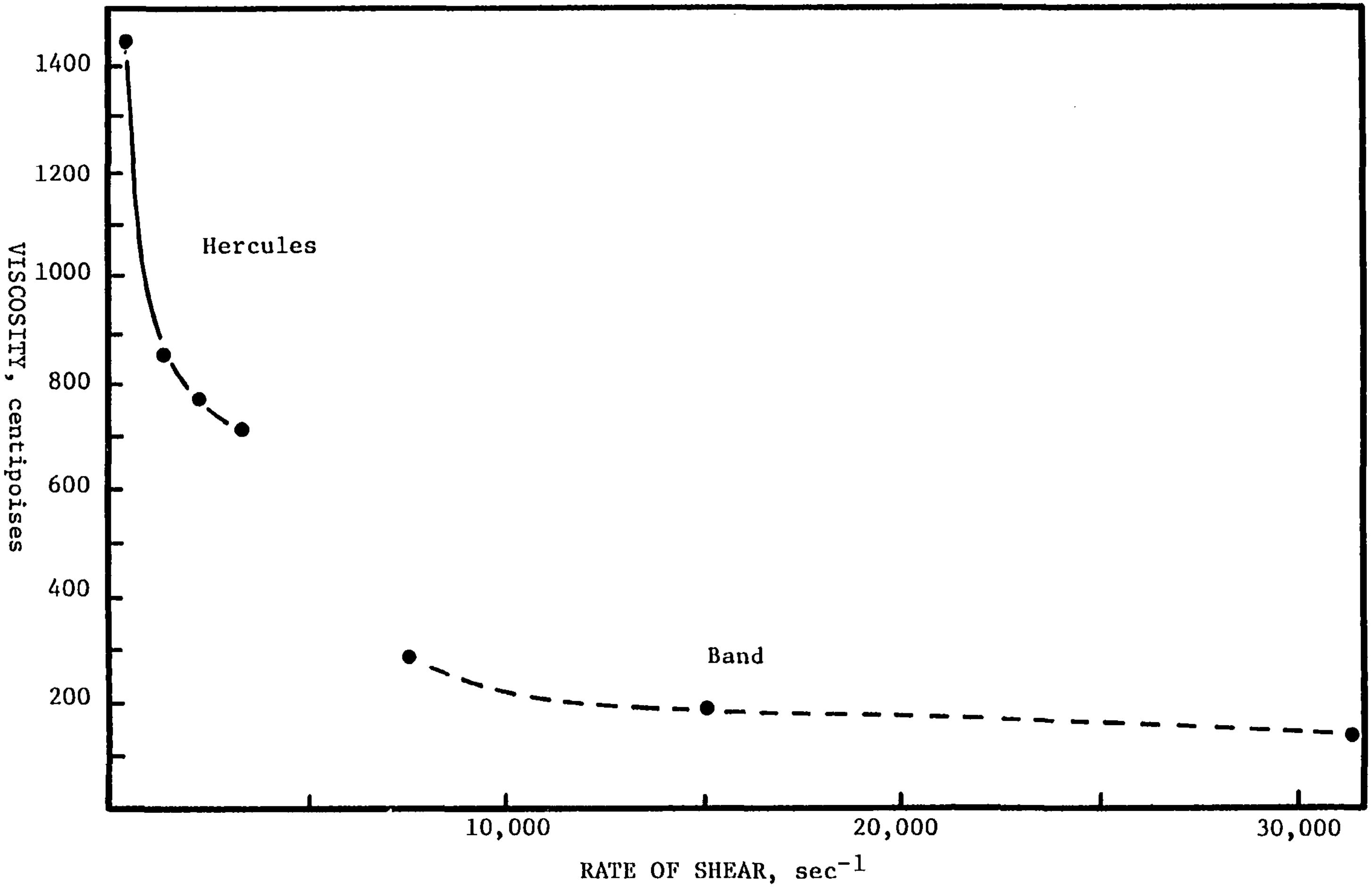

Figure 14. Viscosity versus rate of shear for low solids coating with 0.75 gm. Calgon.

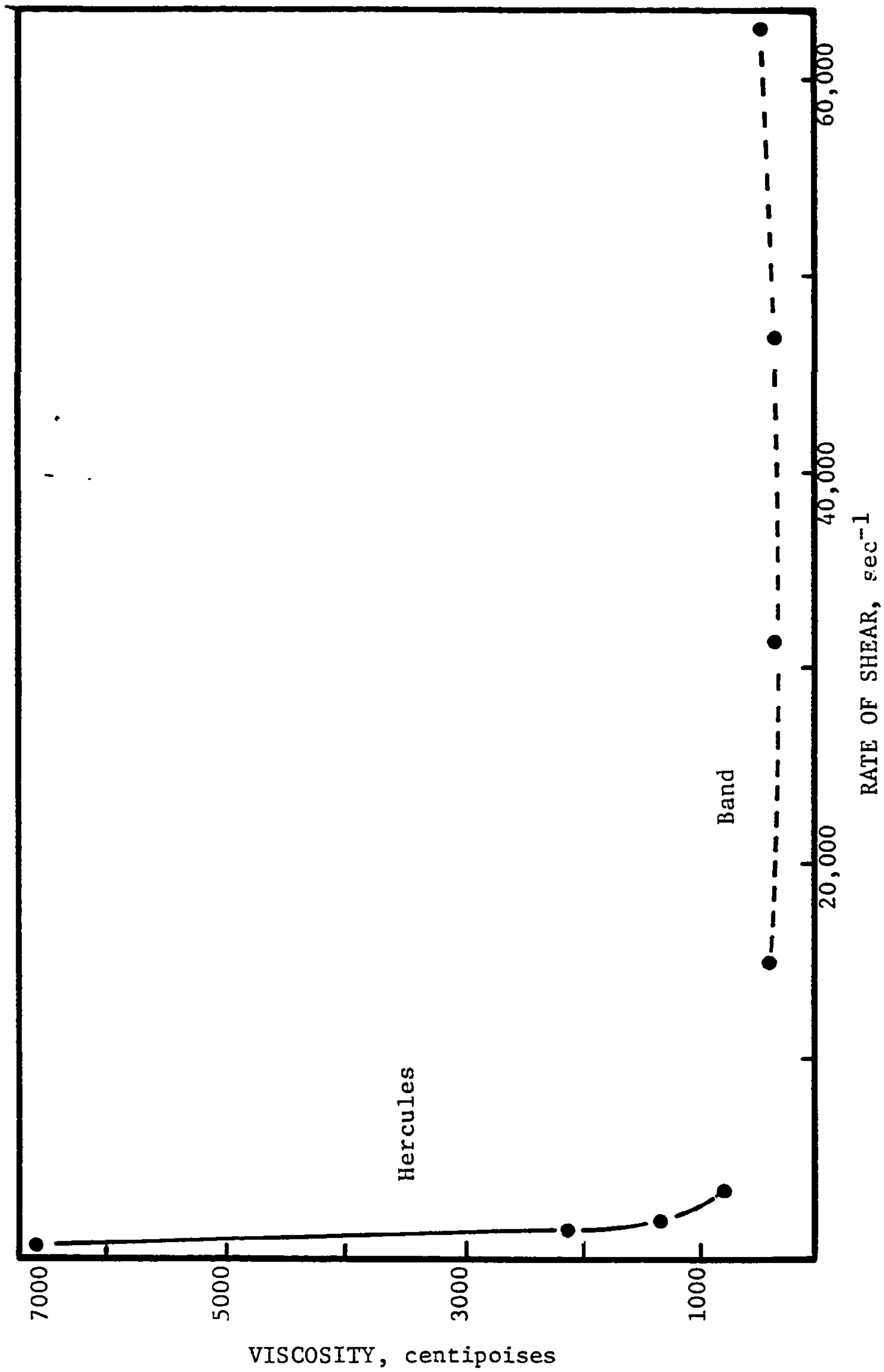

Figure 15. Viscosity versus rate of shear for high solids coating.

These results indicate that the Hercules and Band viscometers are based on sound principles and function well in their own shear ranges (10^3-10^4 sec^{-1} and 10^4-10^6 sec^{-1}, respectively). Both viscometers showed a decrease in viscosity with rate of shear (i.e., pseudoplastic flow). However, the Band Viscometer in its higher range of shear detected an increase in viscosity (i.e., dilatant flow). The Hercules viscometer was unable to detect this increase in viscosity in its shear range. This probably explains why blade coaters have found the Hercules viscometer of limited value in predicting rheological properties.

Specifically, the low solids coating with 0.75 gm. Calgon decreased in viscosity as the rate of shear increased in both the Hercules and the Band viscometer, from 1530 centipoises to 720 centipoises (53%) and from 285 centipoises to 115 centipoises (60%), respectively. Figure 14 shows this graphically.

When 1.5 gm. Calgon was used in the low solids coating color, a definite difference in the Hercules and Band viscometer viscosities appeared. Figure 13 shows the difference graphically. In its shear range, the Hercules viscosity steadily decreased with rate of shear. In the Band Viscometer's higher and larger shear range the viscosity decreased to a minimum, then increased with rate of shear. Where the Hercules viscometer indicated pseudoplastic flow only, the Band Viscometer showed a change from pseudoplastic flow to dilatant flow, which is important in high speed blade coating.

The high solids coating color viscosities paralleled those of the low solids coating color with 1.5 gm. Calgon. The Hercules viscosity decreased with rate of shear, while the Band Viscometer viscosity decreased, and then increased, as shown in Figure 15. Again the Band Viscometer illustrated its value at higher rates of shear than possible with the Hercules viscometer. It revealed the same change from pseudoplastic flow to dilatant flow as shown in the low solids coating.

CHAPTER V

CONCLUSIONS

This work has been successful in the design and construction of an improved Band Viscometer.

The Band Viscometer will be a tool helpful in studying rheological behavior in a research atmosphere and as a guide for the optimization of coatings used in industry. It will be useful expecially at very high rates of shear nearing those rates of shear actually found in normal blade coating operating conditions. This is true for two reasons. One, because high rates of shear are generated in the nip, and two, because the instrument is constantly replacing the liquid being tested with fresh liquid as is the case in commercial blade coaters.

The Band Viscometer displayed the ability to show the change from pseudoplastic flow to dilatant flow of typical coating colors whereas the Hercules viscometer still presents the same coating colors as thixotropic throughout its limited lower shear range.

CHAPTER VI

SUGGESTIONS FOR FUTURE WORK

More practical mill experience will aid in proving the Band Viscometer's usefulness. Also, testing in the laboratory various pigment combinations that are used in industry will add to the knowledge already gained.

Thoughts for possible improvement of the machine itself include finding a way to put one continuous band of tape with a washing, rinsing, and drying system on the machine instead of the disposable tape on a roll now used. Also, a brush to clean the tape of any foreign particles before entering the reservoir is desirable. Placing the rpm indicator on the left side of the instrument (attached to the same turn roll) would allow the operator to see the increase in speed more readily. In addition, the strain gauges could be made more sensitive by mounting all four on one side of the metal bar and activating all four gauges. This would decrease the amount of amplification of the signal that is now needed. More noise would be filtered out, also, rather than recorded. A thinner and/or a narrower metal bar would allow for more strain sensing too. It is a very sound unit, however, these few improvements could refine the machine to a higher degree of operation.

CHAPTER VII

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