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THE RHEOLOGY OF THORIUM
OXIDE SLURRIES

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OAK RIDGE NATIONAL LABORATORY

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THE RHEOLOGY OF THORIUM OXIDE SLURRIES

IV. THE DESIGN AND CONSTRUCTION OF A CAPILLARY TUBE VISCOMETER FOR MEASUREMENT OF VISCOSITY OF AQUEOUS SLURRIES AT TEMPERATURES UP TO 300°C

In studies of the viscosity of thorium oxide slurries it has been shown⁽¹⁾ that at room temperature and constant rates of shearing strain the apparent viscosity* varies by a factor of $\sim 10^3 - 10^4$ depending on the electrolyte atmosphere in the slurry. It has also been shown⁽²⁾ that at room temperature and with a constant electrolyte atmosphere, the apparent viscosity varies by a factor of $\sim 10^4$ as the rate of shearing strain is varied. It appeared that a measurement of viscosity of thorium oxide slurries would be useful in the characterization of various preparations of thorium oxide. Since no information was available on the viscosities of thorium oxide slurries at elevated temperatures, it also appeared desirable to be able to make measurements over a range of temperatures from ambient to 300°C.

A capillary viscometer, being used by F. Kertesz⁽³⁾ of the Ceramics Division to measure the viscosity of molten salts, seemed to possess many desirable features. These included:

1. Operation required small quantities of material,
2. with simple modifications the system could be operated at 2000 psi and 300°C,
3. geometry of the system permitted rigorous analysis of the data.

Further study uncovered no obvious difficulties; therefore, it was decided to proceed with a detailed design. This report describes the design and construction of the capillary viscometer.

*A viscosity measured with Brookfield viscometer, see CF 55-4-182, April 20, 1955 for a discussion of the difference between the "apparent" viscosity and the viscosity as defined by the Poisseuille equation.

Viscometer Design Principles

Measurement of the viscosity of a fluid with a capillary viscometer utilizes the fact that, for laminar flow, Poiseuille's equation (Equation 1) gives the relation between the capillary dimensions, the volumetric rate of flow, the pressure drop, and the fluid viscosity.

$$\mu = \pi g_c D^4 \Delta p / 128 qL \quad (1)$$

Thus the fundamental requirement that must be met in the design and operation of a capillary viscometer is that flow always be in the laminar regime so that Poiseuille's equation will be applicable. For Newtonian fluids this requirement is fulfilled if the Reynolds number

$$N_{Re} = DV\rho/\mu \quad (2)$$

is less than 2100. Equations 1 and 2 define the limits on the capillary length and diameter. In addition the ratio of capillary length to diameter should be greater than fifty in order to minimize kinetic and end effects.

Analysis of the mechanical design problem showed that the principal uncertainty in the measurement of the viscosity at elevated temperatures was the determination of the volumetric rate of flow. The scheme used in the molten salt viscometer involved two electrical probes, mounted vertically, one being longer than the other. Molten salt contacting the probes completes an electrical circuit, thus giving an absolute measurement of the liquid level.

It seemed desirable to avoid the use of the electrical probe idea for at least two reasons:

1. The difficulty of making a pressure seal for the leads out of the bomb at 2000 psi and in the presence of steam.
2. The requirement of being able to measure the viscosity of pure thorium oxide slurries, i.e., no electrolyte addition being allowed thus giving a fluid of very low conductivity making the operation of the probes questionable.

However, the change of pressure drop across the capillary as the last of the fluid passed through could be used as the signal indicating the completion of a cycle. Preliminary tests (see Appendix A) in a glass mock-up showed that at the end of a cycle the pressure drop across the capillary was halved in about 5 seconds. Although this rate of pressure change is far from instantaneous, nevertheless with sufficiently long cycle times, the pressure drop across the capillary could be maintained essentially constant for 95-98% of the total cycle.

The operation of a viscometer using these principles can be seen by referring to the schematic diagram shown in Figure 1. The slurry is made to flow either from the inner reservoir to the outer reservoir or vice versa under the action of a differential pressure. The differential pressure is measured by a DP cell and the equalization of pressure between the two reservoirs indicate the end of a cycle.

Viscometer Design Features

The preliminary tests in a glass mock-up (Appendix A) not only demonstrated the feasibility of the method but also allowed a slurry sample volume to be chosen. Assuming that a 50% reduction in the pressure drop across the capillary would be sufficient to trigger the timer, then a half-cycle time* of 200 seconds is required in order to have a constant pressure drop during 95% of the test time.

Assuming a viscosity of 1 centipoise, a capillary diameter of 13 mils and a density of 2 gram/cu.cm., a sample of less than 30 cu.cm. is required to give a Reynolds number less than 1000.

*Half-cycle time represents the length of time required to force the sample being measured from the inner cylinder to the outer cylinder or vice-versa.

ORNL-LR-DWG 7199A

Figure 1

The dimensions of the inner and outer reservoirs are such that the volumes are essentially the same, so that the rate of change of pressure at the end of each half-cycle should be the same regardless of the direction of flow. The volume of these reservoirs plus associated tubing is less than one percent of the volume of the standard compressed gas cylinder. Gas cylinders can be used as pressurizers and accumulators, and there will be less than a one percent variation of pressure on the system during any half cycle.

The viscometer is heated by inserting it in a furnace made from an 8-inch diameter aluminum casting wrapped on the outside with heater elements and with 3 inches of magnesia insulation.

Provision is made for stirring the inner reservoir with a dash-pot stirrer and the outer reservoir with a rotating magnetic stirrer. If the dash-pot stirrer proves unsatisfactory, a rotating magnetic clutch stirrer has been designed and will be completely interchangeable with the dash-pot stirrer head.

Instrumentation Design Features

Figure 2 is a schematic diagram of the instrumentation required for a complete cycle.* The DP cell determines pressure differentials from 0-200 inches of water at total pressures of 2000 lbs/sq.in., and a 3-15 p.s.i. proportional signal from the DP cell is recorded on a Taylor Miniature recorder whose chart speed is approximately one inch per minute.

Provision is made for recording up to 12 different temperatures on a multipoint Brown recorder. Half-cycle time can be measured to the nearest 1/10 second with a Standard Electric Company timer. Temperature is measured from a

*The philosophy used in the selection of valves and instruments was that the instrumentation must be as simple as possible. As a result, many of the valves are hand operated with the understanding that once the feasibility of operation with slurry is demonstrated, the viscometer operation can be made semi-automatic.

Fig. 2 Schematic Diagram of Capillary Viscometer Instrumentation.

- Manually operated valve
- Solenoid valve
- Rupture disc

thermocouple inserted in the aluminum heater wall and controlled by means of a Simplytrol controller which interrupts power to the heater. Proper sequential operation of the solenoid valves and timer is accomplished through a system of push buttons, selector switches and latching relays.

Viscometer Design and Construction

Having set the general design criteria, a detailed design was made for capillary viscometer to be constructed from Type 347 stainless steel. Allowable stress values used were those recommended for HRP piping⁽⁴⁾. Wall and flange thickness calculations were made using the standard design formulas⁽⁵⁾ (Appendix B). The construction and instrumentation of the viscometer has been completed including a complete dash-pot stirrer. The alternate magnetic-clutch stirrer housing has been constructed with only the bearing assembly remaining to be completed. The viscometer and dash-pot stirrer head have been pressure tested hydraulically to 3800 lb/sq. in.

Figures 3, 4, and 5 show the viscometer disassembled, assembled, and installed in the furnace.

Suggested Operating Procedure

The sequence of valving operations used with instrumentation shown in Figure 2 is as follows:

1. All valves closed. $P_1 = P_2$
2. Open valves 3 and 4; bleed sufficient gas through valve 2 to give the desired pressure drop reading on the DP cell recorder, close valve 2.
3. Open valves 6 and 8, starting first 1/2-cycle.
4. Close valves 6 and 8 at end of first 1/2-cycle.
5. Bleed sufficient gas through valve 2 to give the desired pressure drop reading in the DP cell recorder, close valve 2.
6. Open valves 5 and 7, starting second 1/2-cycle

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PHOTO 14687

Figure 3

PHOTO 14688

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Figure 4

Figure 5

7. Close valves 5 and 7 at end of second $1/2$ -cycle.
8. Repeat step 5.
9. Repeat steps 3 through 7, etc.

NOMENCLATURE

D	capillary diameter	ft.
D_r	root diameter of flange	in.
F	total load taken up by bolts	lb. force
g_c	conversion factor = 32.17	$\text{ft}/(\text{sec})^2(\text{lb. mass})/(\text{lb. force})$
L	capillary length	ft.
N_{Re}	Reynolds number	dimensionless
p	system pressure	lb./sq.in.
Δp	pressure drop	lb./sq.ft.
q	volumetric flow rate	cu. ft./sec.
r	radius	in.
s_b	bending stress	lb./sq.in.
t	thickness	in.
V	fluid velocity	ft./sec.
μ	fluid viscosity	lb./ft.sec.
π	3.1418-----	dimensionless
ρ	fluid density	lb./cu.ft.

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APPENDIX A

Determination of Rate of Pressure Decrease
at End of Viscometer Cycle

A glass mock-up of the viscometer was constructed in order to test the concept of using the rate of pressure decrease to signal the end of a cycle. The viscometer was made from volumetric cylinders, glass tubing, and a glass capillary $3 \frac{1}{32}$ " long and having an average diameter of 466 micron. A mercury manometer was used to measure the pressure drop across the capillary and a water leg was used to regulate the pressure and also to serve as a relief valve for the system. A schematic diagram is shown in Figure 6. The volumetric flow rate was measured at least 5 times during any one up or down test, the average value being used in the calculations.

At the conclusion of a test, the pressure decreased in the desired way. Typical curves of pressure drop against time at the end of a cycle are given in Figure 7. The difference between the up and down curves is due to the difference in volume between the inner and outer reservoir. It can be seen that the pressure drop was halved in about 5 seconds, even with the lowest rate of decrease. This is a sufficiently rapid rate of Δp decrease to be used as a signal for the end of a cycle provided the cycle is long enough so that the pressure drop across the capillary is constant for the greater portion of the time cycle.

As a matter of general interest, readings taken during the steady state portion of the tests are given in Table I.

Fig 6. Schematic Diagram of
Glass Viscometer System.

Fig. 7. Change of Capillary Pressure Drop with Time at End of Cycle.

TABLE I
STEADY STATE VISCOMETER OPERATION WITH A
CAPILLARY PRESSURE DROP OF 135 mm Hg

	Down Cycle			Up Cycle		Down Cycle	
	Cumulative Volume, cc	Time, Seconds	Volumetric Flow rate, cu. cm./sec	Time, Seconds	Volumetric Flow rate cu. cm./sec	Time, Seconds	Volumetric Flow rate cu. cm./sec
1	0	109	0.252	0		90	0.266
2	4.8	90	0.289	20	0.240	72	0.274
3	10.0	72	0.278	39	0.274	53	0.294
4	15.0	54	0.284	58	0.263	36	0.300
5	20.4	35	0.312	80	0.245	18	0.278
6	25.4	19	0.273	101	0.236	0	
7	30.6	0		122	0.248		
Avg.			0.281		0.251		0.283

Using the average flow rate for the up and down cycle (which cancels out the head effects) a capillary diameter of 451 micron was calculated compared with the average diameter, measured with a microscope of 466 micron. (The capillary was slightly elliptical in cross section with a major diameter of 480 microns and a minor diameter of 453 microns.)

APPENDIX B

Capillary Viscometer Design Calculations

The final working drawings are shown in Figures 8-14. Calculations used to determine dimensions are given below, individual components being referred to either by name or by item number keyed to Figure 8.

An allowable working stress of 12,000 psi was used for Type 347 stainless steel operating at temperatures up to $300^{\circ}\text{C}^{(4)}$. Maximum operating pressure was taken as 2000 psi.

I. Capillary Dimensions

Preliminary calculations showed that for $N_{Re} < 1000$, $\Delta p \sim 100$ -in H_2O , $\mu = 1$ cp and $L \sim 2$ -in, a 15 mil diameter capillary would be satisfactory. Commercial capillary tubing was available starting at 6 mils diameter; therefore, an order was placed for 50 ft. lengths of tubing starting at 0.006-in. inside diameter and with diameters increasing by a factor of $10^{0.25}$ up to 0.170-in. inside diameter.

II. Inner and outer reservoir wall thickness (Items 2 and 5)

The inner and outer reservoirs were made from $3/4$ -in. Sch. 80, Type 347 stainless steel seamless pipe and $1\ 1/2$ -in., Sch. 160, Type 347 stainless steel seamless pipe, respectively⁽⁴⁾.

III. Outer Reservoir Bottom Plate (Item 8)

For uniformly loaded circular plates clamped at the edge:

$$s_b = 3p r^2 / 4t^2$$

from which

$$t = \sqrt{3p r^2 / 4s_b}$$

$$t = \left[3 \times 2000 \times (1.338/2)^2 / 4 \times 12,000 \right]^{1/2}$$

$$t = 0.236 \text{ inches}$$

IV. Inner Reservoir Flange (Item 1 and 15)

Flanges are usually computed as cantilevers built in at the root and loaded with a concentrated load at the bolt circle

$$t = \sqrt{6 Fl / \pi D_r s_b}$$

where

$$F = \pi (1.88)^2 \times 2000 / 4 = 2220 \text{ lb.}$$

$$t = \sqrt{6 \times 2220 \times 0.46 / \pi \times 1.05 \times 12,000}$$

$$= 0.394 \text{ in.}$$

Make 7/8" thick to allow for machining after welding.

V. Outer Reservoir Flanges (Item 3, 4)

$$t = \sqrt{6 \times 2000 \times \pi (1.9)^2 \times 0.55 / \pi \times 1.9 \times 4 \times 12,000}$$

$$t = 0.512 \text{ in.}$$

Make 1" thick to allow for machining after welding.

VI. Inner Reservoir Bolts

Calculate as simple load in tension.

$$D = \sqrt{4F / \pi s_t}$$

$$D = \sqrt{4 \times 2220 / \pi \times 12,000}$$

$$= 0.198 \text{ in.}$$

Use 6 - 3/8 - 24NF-2 Bolts

VII. Outer Reservoir Flange Bolts

$$D = \sqrt{4 \times 2000 \times \pi (2.025)^2 / 8 \times \pi \times 4 \times 12,000}$$

$$= 0.272 \text{ in.}$$

Use 8 - 1/2 -20 NF-2 Bolts

VIII. Upper Flange, Viscometer Stirring Head (Item 22)

a) Bolts required (Use 5/8-in. bolts)

Assume 50% of load to make seal with a 2-1/2-in. cavity.

$$1.5 \times 2000 \times \pi \times (1-1/4)^2 = \text{No. of bolts} \times 0.24 \times 12,000$$

No. of Bolts = 5.2, use 6.

b) Bolt circle required

Allow 1 1/2-in. per bolt.

Diameter = 6 x 1-1/2 / $\pi \approx 3$ -in.; use 4-1/8-in. Dia. Bolt Circle

c) Side wall thickness between cavity and bolt hole

HRT Piping 3" nominal pipe = 3.50 OD with 0.406 wall; use 1/2-in. wall.

d) End plate thickness over cavity.

$$t = \sqrt{3 p r^2 / 4 s_b}$$

$$= \sqrt{3 \times 2000 \times (1-1/4)^2 / 4 \times 12,000}$$

$$= 0.255\text{-in.}$$

IX. Lower Flange, Viscometer Stirring Head, (Item 21)

$$t = \sqrt{6 F l / \pi D_r s_b}$$

$$= \sqrt{6 \times 15,000 \times (7/8) / \pi \times 1 \times 12,000}$$

$$= 1.445 \text{ inches.}$$

Fig. 8. Schematic Diagram
of Capillary Viscometer

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Tolerances & Notes

Decimals, ± 5 units in last decimal place shown

Fractions, ± 0.008 "

Material - 347 Stainless Steel

Not to scale

9. 1/4" Dia Type 347 S.S. rod drilled to fit Thermowell Tube

10. 1/4" o.d. Type 304 S.S. tubing - 0.065-in wall; length sufficient to connect to pressure fitting.

Figure 10

1. Insert Armco Iron core in tubing and weld end plates on end of S.S. tubing.
2. Drill hole at center of rod and insert ss capillary tube, welded shut at one end, and weld on outside.

Figure 12

6-5/8-19 NF-2 Case Hardened
Steel Bolts

Weld and Machine Smooth

Weld

15

6- $\frac{11}{16}$ -in Holes
Evenly Spaced

Stirring Head for Viscometer

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Figure 13

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Figure 14

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