Design of a Shear Rheometer to Determine the Viscoelastic Properties of Natural Polymers

Casey Directo, Jerry Le; Mechanical Engineering
Kellogg Honors College Convocation 2013
Mentor: Dr. Vilupanur Ravi, Professor and Chair, Department of Chemical and Materials Engineering

Problem Statement

To determine the ideal composition of *yop* used in the Chumash Indians' *tomols* by investigating the influence of composition on the viscosity of melted *yop,* dynamic mechanical analysis must be employed. Using dynamic measurement methods to calculate rheological properties such as the storage and loss moduli G' and G'' as well as the loss tangent tanδ, the viscoelastic behavior of *yop* can be better quantified compared to non-dynamic methods. However there are several obstacles to performing any type of analysis on *yop*.

Laboratory experience has revealed two properties of *yop*: first that *yop* is a very strong adhesive, and second that melted *yop* will quickly solidify at room temperature. Thus to investigate the rheological properties of *yop* requires a rheometer capable of operating at elevated temperatures and offers a means to handle solidified *yop*.

Background

About the Chumash and *Yop*

The Chumash Indians originally were a collection of 150 independent Native American communities spread across the south central coastal region of California who spoke different but related languages and shared a common culture. One of the hallmarks of Chumash culture was extensive trade between inland and coastal settlements that utilized shell-beads as currency[1]. In order to trade between island and mainland communities the Chumash had to build canoes strong enough to traverse the waters of the Santa Barbara Channel. The most prestigious and prized canoe was the *tomol;* a canoe constructed from planks carved from driftwood and bound and sealed using natural fibers and an adhesive called *yop.*

Yop is a natural polymer made from a mixture of hard asphaltum and pine pitch used by the Chumash as an adhesive and caulking agent in their *tomols.* The Chumash would create *yop* by first breaking large asphaltum rocks into smaller pieces, which were then placed in a soapstone crucible called an *olla* and heated. After the asphaltum melted, ground pine pitch would be stirred into the mixture. If the *yop* was too runny, more asphaltum would be added; too thick and more pine pitch was added. Once the *yop* achieved the correct thickness brushes would be used to apply it between the planks of the *tomol*.

Sadly, with the Spanish missions much of the Chumash culture and knowledge was lost. Most of what is known today about creating *tomols* is thanks to the work of John P. Harrington, who recorded the knowledge of the last Chumash Indian who knew how to make a *tomol*[2]. However very little is known about the exact composition of *yop* used in *tomols*, as Harrington's informant did not know what the exact thickness of *yop* should be. Anthropologists would like to know the composition of *yop* that was ideal for boat construction, and if this composition was chosen for ease of use (e.g., optimum viscosity) or for strength.

Theory

One consequence of viscoelasticity is the high mechanical damping characteristics in polymers. These damping characteristics are known to be dependent on the frequency of forced oscillations or vibrations in a polymer sample. Thus the response of a polymer to a forced oscillation at a specific frequency can be used to characterize the dynamic viscoelastic properties of a polymer.

Suppose a polymer specimen is subjected to an oscillating shear strain γ at an angular frequency ω,

$$\gamma(t) = \gamma_0 \sin \omega t \, , \tag{1}$$

where γ_0 is the amplitude of the shear strain and t is time. The resulting stress response σ for a linear viscoelastic polymer (i.e. at relatively low frequencies) will also be sinusoidal but out of phase with the strain

$$\sigma(t) = \sigma_0 \sin(\omega t + \delta) \, , \tag{2}$$

where σ_0 is the amplitude of the stress and δ is phase angle. Due to the damping properties the strain will lag behind the stress by the phase angle. If equation 2 is expanded to

$$\sigma(t) = \sigma_0(\sin(\omega t)\cos\delta + \cos(\omega t)\sin\delta), \tag{3}$$

it is clear that there are two components in the stress; one in phase with the strain (cosδ) and the other 90° out of phase (sinδ). The stress can then be related to the strain using the definition of the shear modulus G,

$$\sigma(t) = \gamma_0[G'\sin(\omega t) + G''\cos(\omega t)], \tag{4}$$

or

$$\sigma(t) = G'\gamma + \frac{G''}{\omega}\frac{d\gamma}{dt}, \tag{5}$$

where G' and G'' are the in-phase and out-of-phase components of the complex modulus G^*, respectively. G' and G'' are defined as

$$G' \equiv \frac{\sigma_0}{\gamma_0}\cos\delta \qquad G'' \equiv \frac{\sigma_0}{\gamma_0}\sin\delta \tag{6, 7}$$

with the complex modulus defined as

$$G^* \equiv G' + iG'' \, , \tag{8}$$

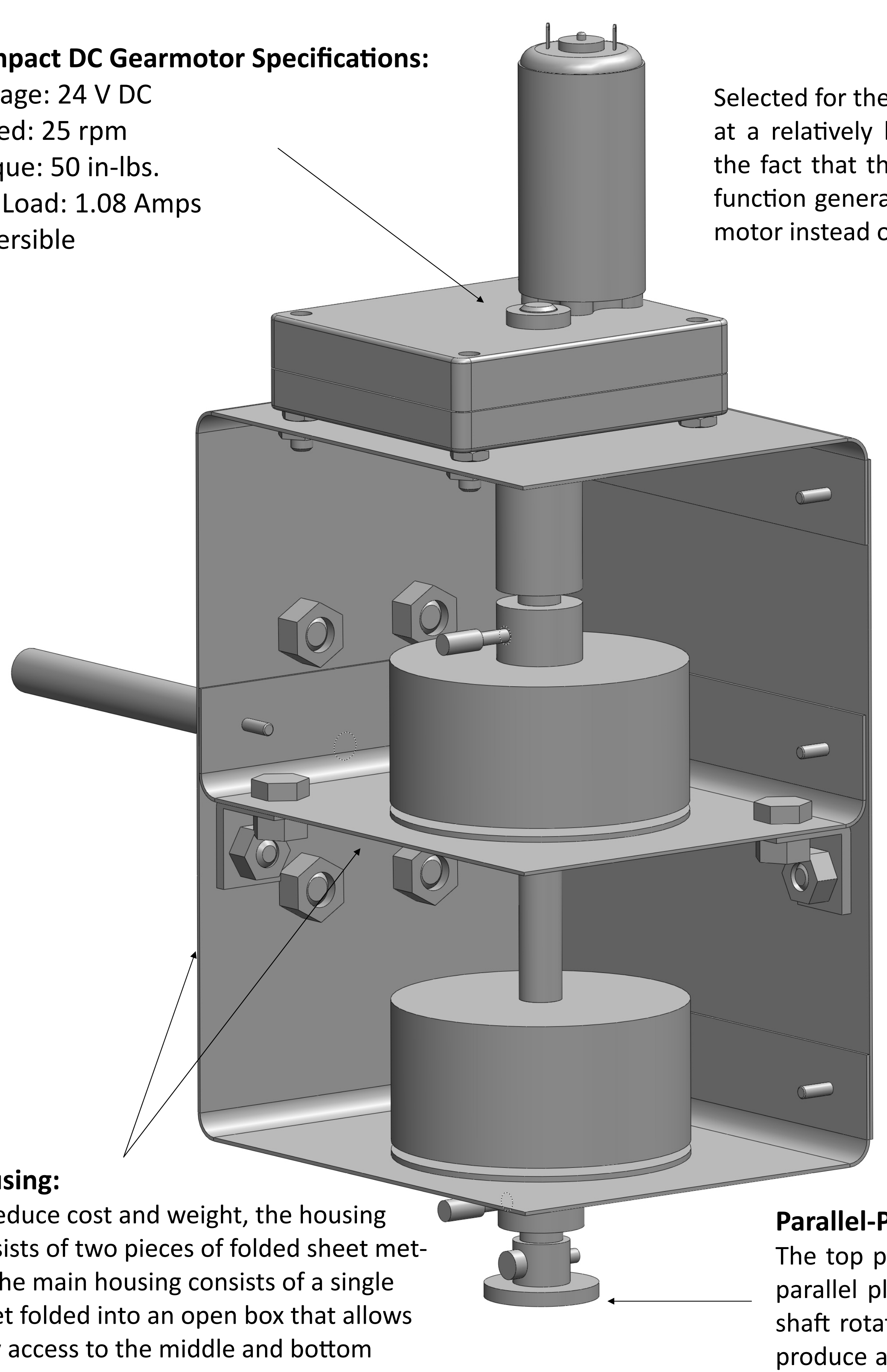
where it is seen that G', the storage modulus, is the real component while the loss modulus G'' is the imaginary component.

An examination of equations 6 and 7 shows that the tangent of the phase angle is related to the storage and loss moduli by

$$\tan\delta = \frac{G''}{G'}, \tag{9}$$

which suggests that materials with greater damping will have larger δ values while materials that are purely elastic will have a δ that is close to 0. Conceptually this makes sense, as the lag between the strain and stress measured by δ is the result of damping, or viscous properties, which is captured by the G'' term.

Rheometer Design



Compact DC Gearmotor Specifications:
Voltage: 24 V DC
Speed: 25 rpm
Torque: 50 in-lbs.
Full Load: 1.08 Amps
Reversible

Selected for the ability to deliver a high torque at a relatively low speed without a gearbox, the fact that the motor is reversible means a function generator can be used to control the motor instead of a costly motor controller.

"HotPot" Rotary Potentiometer Specifications:
Resistance: 100-10k Ω
Resistance Tolerance: ± 20%
Linear Tolerance: ± 3%
Temperature Range: -40°C to +85°C Standard

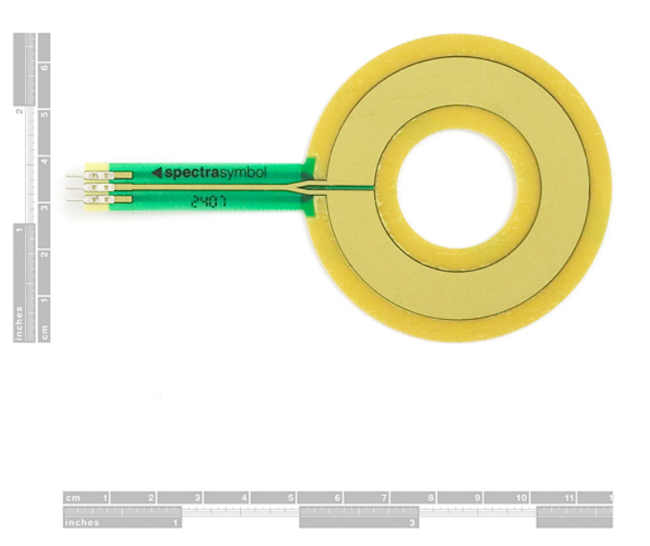


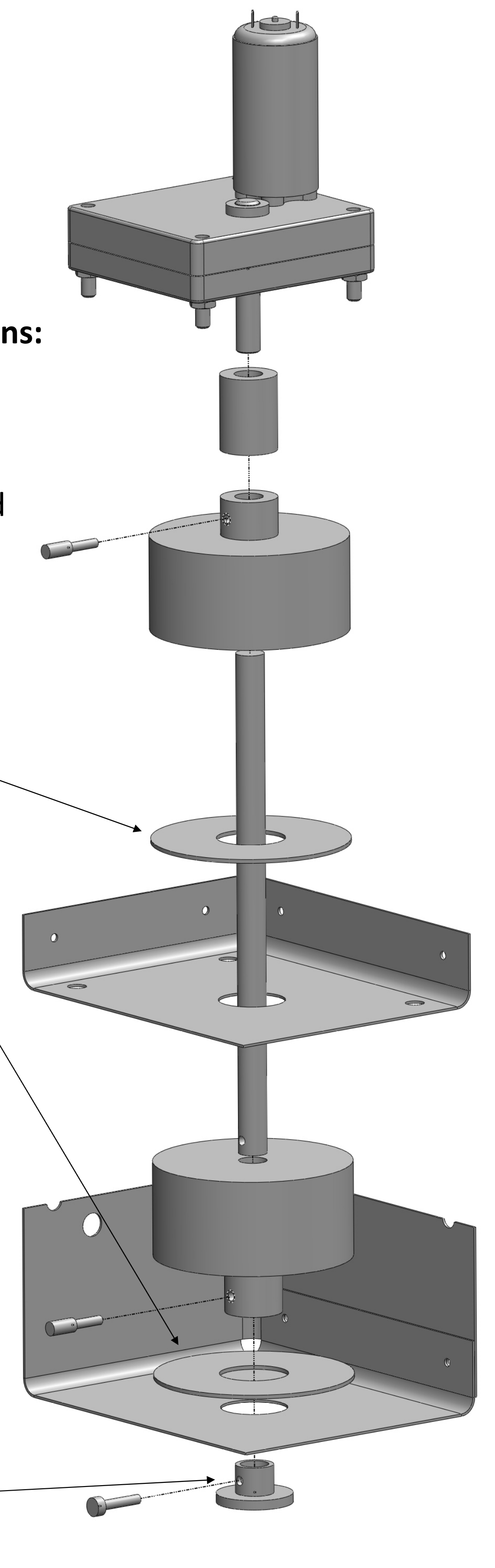
Image taken from Karlssonrobotics.com

The "HotPot" rotary potentiometer were chosen as they could operate at elevated temperatures, offered a theoretically infinite resolution, and had the ability to be placed mid-shaft due to its geometry.

Housing:
To reduce cost and weight, the housing consists of two pieces of folded sheet metal. The main housing consists of a single sheet folded into an open box that allows easy access to the middle and bottom levels for calibration and assembly.

Parallel-Plate Geometry
The top plate at the end of the shaft lies above a fixed parallel plate, sandwiching the sample material. As the shaft rotates, viscous forces that develop in the sample produce a torque on the top plate that is transmitted to the shaft and results in an angular displacement.

Exploded Shaft View



Features

Modular Test Fixtures

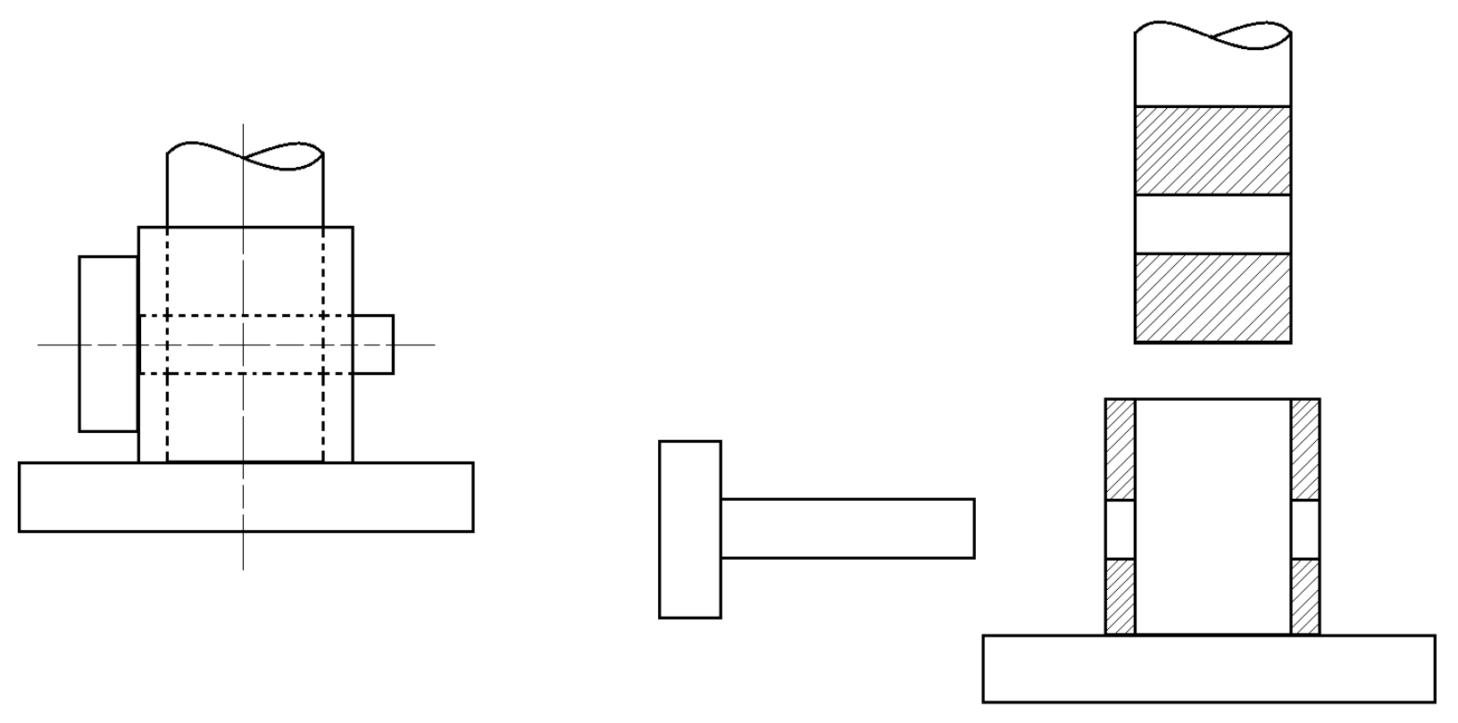


Figure 1: *Shaft end mounting with top plate fixture.*

To handle the adhesive nature of *yop* a modular design was employed for the test geometry. The end of the driving shaft features a hole and pin design to allow the top plate to be quickly removed and replaced. Thus multiple sample compositions can be tested in succession simply by replacing the top plate. Furthermore cleaning or removing hardened *yop* only requires the top plate to be removed, which can be done by removing the pin and raising the rheometer.

In addition to aiding clean up and providing a solution to *yop* hardening between the parallel plates, the modular shaft end allows different sample geometries to be tested. By creating different test fixtures for the shaft, the rheometer can be adapted to run dynamic tests on different materials that require different sample geometries, e.g. solid polymer rods or bars.

Ball-Collar Assembly

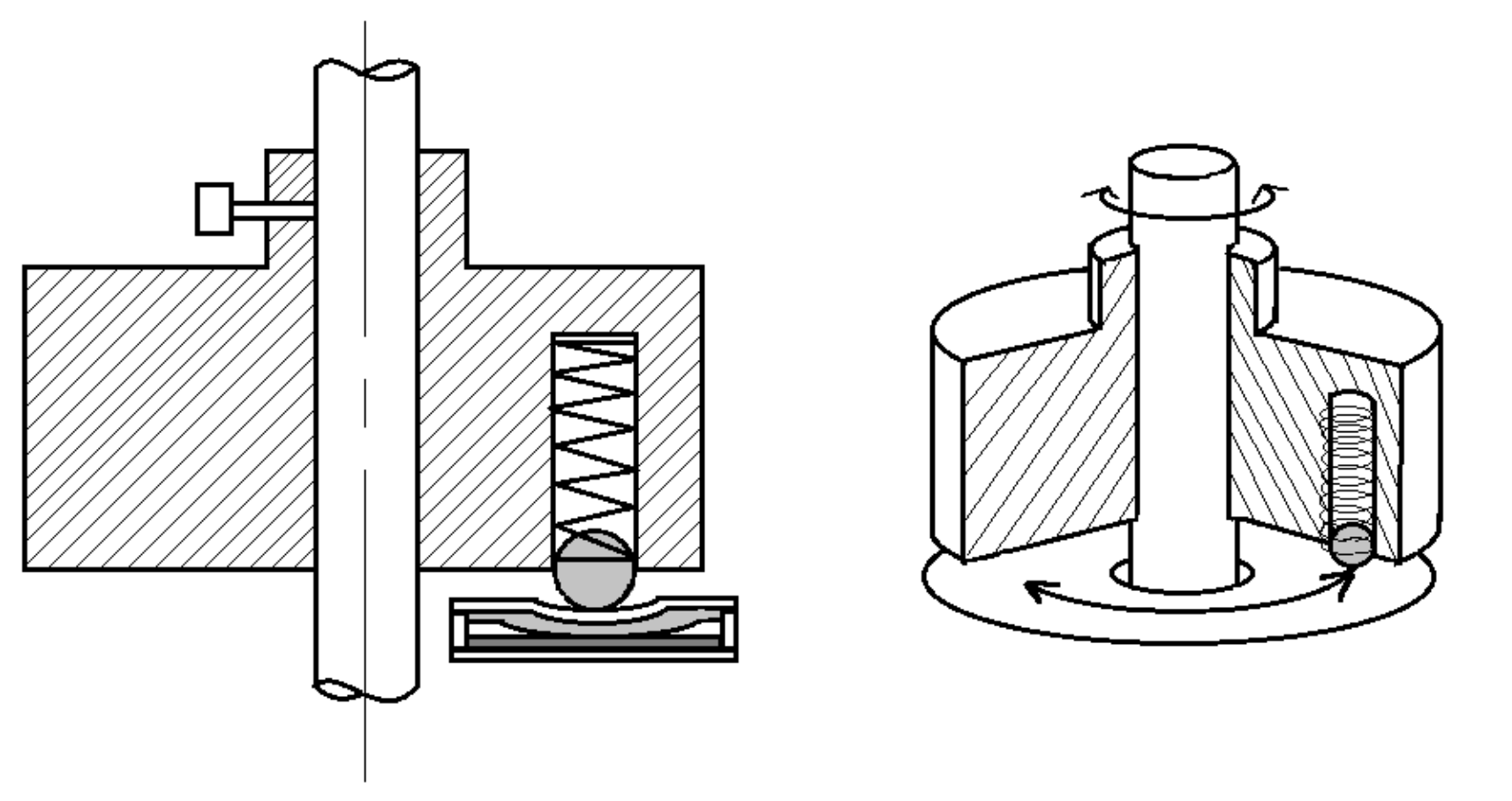


Figure 2: *Ball-collar and HotPot operating concept.*

To measure the angular position of the shaft at different locations, two "HotPot" rotary potentiometers were selected. Due to the HotPot's design, two "ball-collars" were designed to provide the mechanical input to each HotPot.

Each collar is fixed to a point in the shaft by a set screw. A hole cut into the collar fits a spring and 3/8'' chrome ball bearing, which sits on the rotary dial of the HotPot. As the shaft rotates, the collar moves the ball bearing with the spring providing enough force on the ball to close the circuit in the HotPot. Since each collar is fixed to the shaft by a set screw, the angular position measured by each HotPot corresponds to the location of the set screw. This allows each HotPot to be placed away from the ends of the shaft, in positions that are much more accessible or less likely to be damaged by the hot plate.

Basic Operation

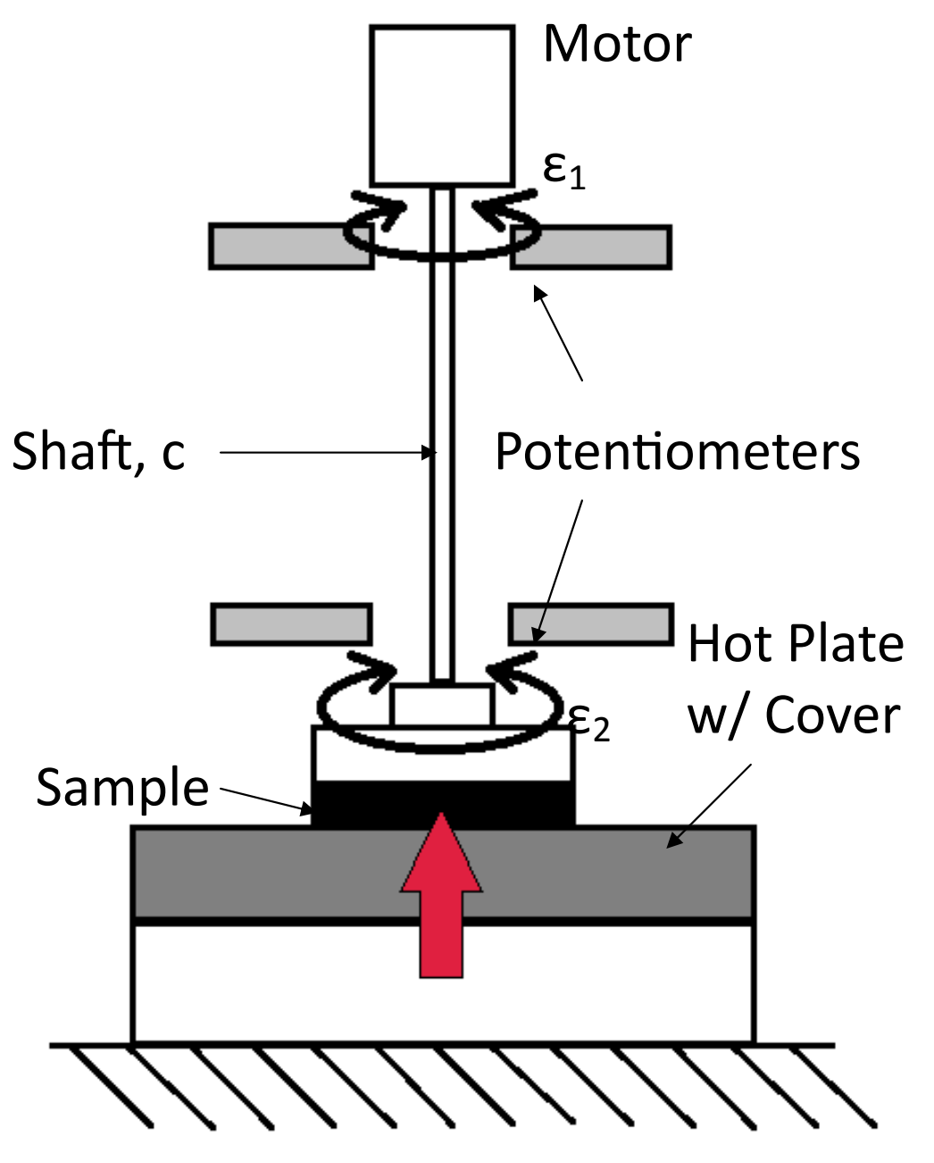


Figure 3: *Simplified rheometer operation.*

The proposed rheometer design is based on the torsion-wire rheometer model utilizing parallel-plate geometry[3]. Figure 3 gives a simplified version of the rheometer operation.

As seen in figure 3, the material to be analyzed is sandwiched between two parallel plates. The bottom plate is fixed to the hot plate while the top plate fixed to the shaft with torsion coefficient *c* is allowed to rotate with the shaft. The top of the shaft is driven by the reversible DC motor running on a sinusoidal voltage to oscillate over an angular range ε_1^0, with the top plate lagging behind and oscillating over angular range ε_2^0. Potentiometers at both ends of the shaft measure the angles ε_1 and ε_2 from which the phase angle ϕ between the two oscillations can be determined. If the mass moment of inertia *I* of the top plate is known, then G' and G'' can be determined using the following equations from Tropea:

$$G' = \frac{cd}{\pi R^4}\left(\frac{\varepsilon_1^0}{\varepsilon_2^0}\cos\varphi - 1\right) + \frac{d(I\omega^2 - c)}{\pi R^4} \tag{10}$$

$$G'' = \frac{cd}{\pi R^4}\frac{\varepsilon_1^0}{\varepsilon_2^0}\sin\varphi \tag{11}$$

where *d* is the distance between the fixed and top plates, *R* is the radius of the top plate, and ω is the driven angular frequency in rad/s. At low frequencies, G' and G'' can be used to calculate the dynamic viscosity η', zero-shear viscosity η_0, and absolute complex viscosity $|\eta^*|$:

$$\eta' = \frac{G''}{\omega} \tag{12}$$

$$\eta_0 = \lim_{\omega\to 0}\frac{G''}{\omega} = \lim_{\omega\to 0}\eta' \tag{13}$$

$$\frac{\left|\sqrt{G'^2 + G''^2}\right|}{\omega} = \frac{|G^*|}{\omega} = |\eta^*| \, . \tag{14}$$

Acknowledgements

I would first like to thank my advisor Dr. Vilupanur Ravi for his support and patience, as well as Dr. Chuan-Chiang "Chris" Chen for his help with the sensors. I would also like to thank the rest of the Chumash Polymer Team Stephen Moser and Greg Barker, as well as Ulus Eckerman and Ted Ibarra for their support. Lastly I'd like to thank everyone from the Kellogg Honors College for the support over the years.

Future Work

- Finish construction and test rheometer
- Design filters for data acquisition
- Establish a test procedure for *yop* testing
- Create different test fixtures for different materials
- Design a mini-furnace with PID temperature controller

References

1) Santa Barbara Museum of Natural History Anthropology Department , 28 Aug. 2009 Home of The Santa Barbara, California Chumash People! <http://www.sbnature.org/research/anthro/chumash/index.htm>
2) Harrington, *et al*. Tomol : Chumash Watercraft as Described in the Ethnographic Notes of John P. Harrington. [Socorro, N.M.]: Ballena Press, 1978.
3) Tropea, *et al.* Springer Handbook of Experimental Fluid Mechanics, Volume 1. Springer,