Fun Facts about Pitch & the Pitfalls of Ignorance

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ABSTRACT

The various properties of polishing pitches have different advantages and are selected according to the type of work intended. It is important to check pitch properties **before** a pitch lap is poured; to ensure that the final polishing lap properties will be as desired. A simple penetrometer test is utilized as a quality control tool for measuring the hardness (viscosity) of various types of pitch as received from the manufacturer. Only a small sample, 20 grams, is needed for this test. Another simple method for determining pitch quality is the measurement of the softening point. A description of this method and typical results will be described. Lastly, the "tackiness" of pitch and its importance will be discussed.

Lessons learned with pitch preparation, testing, and pouring will complete this broad overview of pitch and some of its properties and applications.

Keywords: pitch, viscosity, softening point, hardness, penetrometer

1. HISTORY AND BASIC FACTS

Polishing pitch has been used successfully by opticians for many years. It can be made synthetically, or can be naturally occurring. The basic types are wood pitch (deciduous and coniferous), rosin based (green and yellow), petroleum based, and asphalt tar pitch (coal based). The characteristics of these different pitches vary depending on the materials they are made from. Some pitches are either formulated from combinations of these and other materials by the manufacturer, or may be blended by the optician to produce desired properties.

1.1 Important Functional Properties

When discussing the properties of pitch, with respect to optical polishing, it is important to distinguish between the properties of the lap versus those of the pitch itself. Two of the most important properties of a pitch polishing lap include its ability to take on a desired form (flat, spherical) and the ability to slightly alter or adjust the form of the lap during polishing. This is referred to as the compliance of the lap. As Norm Brown points out, the ability of a lap to change its surface form is often improperly thought of as merely a function of how hard or soft the pitch is.[1] While the hardness (or, viscosity) of the pitch may strongly influence a lap's compliance, other factors are involved as well, such as the geometry of relief lines/grooves or slurry channels cut into the pitch surface, or the overall thickness of the pitch layer. While the pitch may be very soft or "fluid", if the groove depth is too shallow, the pitch has nowhere to flow and the lap cannot change its form.

Other important properties contribute to the frictional interaction between the lap and the optic. An important property of pitch leads to the lap's ability to hold a "charge", that is, to allow slurry particles to imbed in its surface. In order to maintain a lap, one must regularly recut the relief lines and slurry channels. If the pitch is too "brittle", the surface of the lap will flake, leaving a very rough lap surface, which could lead to poor surface quality in the polished optic. Furthermore, Izumitani describes how the properties of pitch, namely viscosity, affects the removal rate of optics being polished.[2] Clearly,

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the type of pitch used and its properties are crucial to obtaining the best performance from one's lap. Therefore, it is very important to know how to select the proper pitch in order to obtain the desired performance of the polishing lap. Unfortunately, the manufacturers do not publish information on many of these critical properties of pitch, nor are common practices used by those evaluating pitches. The resulting consequence is pitch selection made by trial and error testing by individual opticians and optics shops. Because all the relevant properties are not published, control of variation in properties by the manufacturer is not necessarily controlled to the degree required. Once a particular pitch is chosen for a given polishing process, one must trust that the properties of the pitch lot purchased is relatively the same as the lot purchased a year ago, or ten years ago and that nothing in the processing of the pitch into a lap has significantly altered those properties. As in so many other industries, the supplier relationship can be a key factor in determining which product to purchase. One may be forced to rely on the supplier's reputation.

While we have always had excellent relationships with our pitch suppliers, they may not always be aware of the specific needs of the customer or process. Therefore, reliance on lot consistency and the ability of the pitch to maintain constant properties after pitch melting and lap forming are dangerous assumptions. In our experience, this has lead to one of the most important traps of process control in optics polishing. When it comes to precision optics polishing, Ignorance is not bliss! Know your materials and check them regularly.

What does it mean to know your materials, and how can you know your materials when the pitch constituents are kept as proprietary secrets?

Of course, as optics polishers, we are more concerned with the performance properties and less concerned with the actual ingredients to a given pitch formulation. While one could employ techniques of chemical analysis to determine the constituents, or perhaps to look for variations in proportions of constituents, this can be costly and yield results that are difficult to interpret. However, there are some simple, more direct, means of gathering performance driving information on pitch properties. In order to discuss them we first need to look at a few fundamental material properties. The first is viscosity.

1.2 Viscosity and Shear Modulus

A general definition of viscosity may be described as the resistance of a material to flow. Viscosity is a measure of the internal intermolecular friction in the presence of velocity gradients. Thus, if a shear force were applied to a fluid material, a coefficient of viscosity could be defined as the ratio of applied shear stress to the resulting rate of shear (Equation 1).

(Equation 1: viscosity for a Newtonian fluid)

$$\eta = \frac{\tau}{\dot{\gamma}}$$

$\tau = F/A = \eta V/y = \eta\dot{\gamma}$
τ = shear stress
η = fluid viscosity
F = shear force

The higher the viscosity, the more resistant to flow the pitch will be. As anyone who has ever melted pitch knows, viscosity is greatly dependent on temperature (Equation 2). It is therefore essential to include temperature in any discussion regarding the viscosity of pitch.

$$\eta^* = \eta_0 e^{Q/RT}$$

(Equation 2: exponential relationship of viscosity to temperature)

Q = molar activation energy for viscous flow
R = universal molar gas constant
T = temperature

Another important property of pitch is its elastic behavior. While this is generally a less considered parameter for most pitches, most opticians will complain about pitches that are too brittle, which means a pitch has poor ductility or elasticity.

Elasticity may be expressed by the following equation, which relates applied stress τ, to resulting strain γ, by the Shear modulus, G.

$$\tau = G\gamma \qquad \text{(Equation 3: Shear stress and strain)}$$

For years, opticians have relied on highly qualitative techniques for gauging the "tackiness" or adhesive properties of pitch. This of course, is very important for providing an intuitive sense for the level of friction the pitch will provide in the polishing process. While we do not present any analysis of this property here, we suggest that one could devise a simple test apparatus for measuring the coefficient of friction of the polishing substrate given a set of predetermined parameters.

Good tests exist for characterizing both viscosity and elastic modulus such as the torsional pitch viscosity tester (Brown) or tensile test for elastic modulus. However, for viscosity, opticians have relied upon a penetration hardness test based on an apparatus described by Twyman[3]. A version of this test is routinely conducted at Zygo to verify the condition of our pitch materials. The remainder of this paper focuses on this test and some observations we have made over the past year of testing. The process is described as follows.

2.0 PITCH TESTING METHODS

2.1 Pitch Penetration Test and Results

The pitch sample is crushed and 20g are carefully weighed into a shallow aluminum container approximately 2-inches in diameter. The sample is placed into a preheated oven at 125°C for 30 minutes. The sample is removed and cooled on a flat surface for one hour after which it is moved to the test area and allowed to reach thermal equilibrium for 24 hours before the penetration test is started. The set-up for the test is shown in Figure 1. The 6-mm diameter ball must be well greased to reduce friction. The sample is placed under the indicator and a weight of 500 g is applied for 1 hour. The initial and final indicator readings are subtracted from each other to obtain a penetration depth. Since pitch hardness and viscosity are exponentially dependent on temperature, it is critical that the test is always done in the same environment to obtain reliable and comparable data. At least three measurements should be taken per sample. Table 1 lists penetration depths obtained for a commercially available pitch series. The pitches are listed in order of increasing "hardness". As expected, the "harder" pitch (i.e. Gugolz #91) has a lower penetration depth than the "softer" pitch (Gugolz #55).

Figure 1 Set-up for the Penetration Test

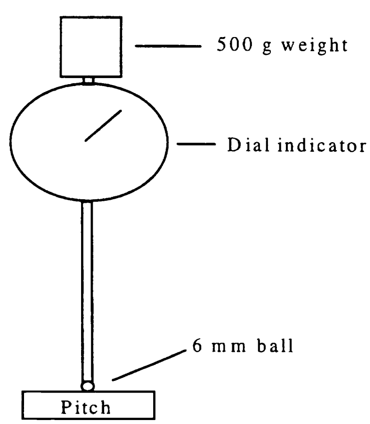


Table 1 Penetration depth of selected commercially available pitch

Pitch	Penetration Depth, Ave. of 3 Measurements @ 22°C
Gugolz #55	0.2624 cm ± 0.003 cm (after 30 minutes)
Gugolz #64	0.1399 cm ± 0.003 cm (after 30 minutes)
Gugolz #73	0.0112 cm ± 0.001 cm
Gugolz #82	0.0064 cm ± 0.001 cm
Gugolz #91	0.0041 cm ± 0.001 cm

The penetration depth can be roughly converted into units of viscosity if so desired, by using the following equation:

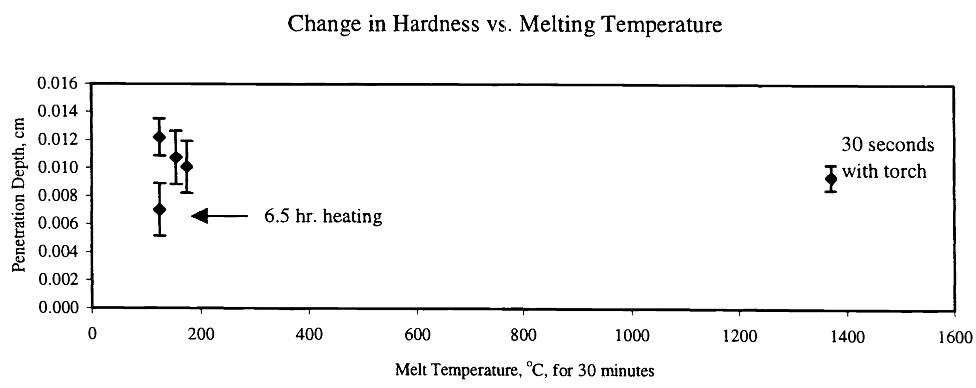
$$\eta = 2.2\times10^{4}\left(\frac{wth^{-1.5}}{\sqrt{D}(1-1.4\frac{h}{D})}\right)$$

Equation 4, viscosity calculation from penetration depth

where h is the penetration depth in cm, t is time in minutes, w is the applied weight in grams, and D is the ball diameter in cm. It is important to note that this equation is valid only for viscosity ranges from 5 x 10^8 to 5 x 10^{10} poise. There should be less than ¼ penetration of the ball diameter, the ball diameter must be 0.64-2.54 cm and the applied loads can vary from a few hundred grams to several kilograms.[1] For the average shop it is much more practical to determine what penetration depths are acceptable at given temperatures for certain pitch types than to use the actual viscosity.

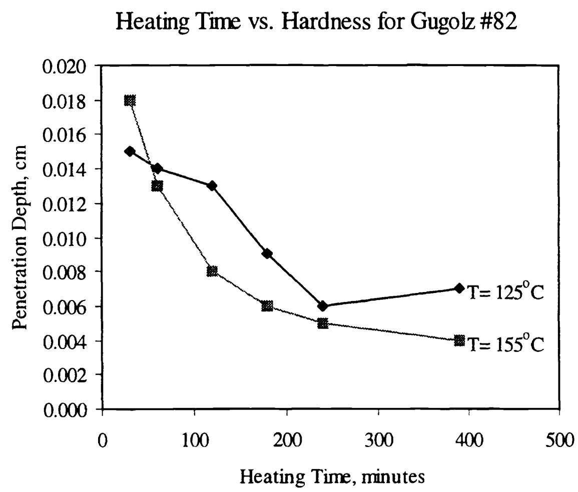
The process used for melting pitch can greatly influence its properties. Using the penetration method, one can determine the effect of both the melt temperature and melt time on hardness. For the pitch tested, we demonstrated a significant change in hardness when the pitch was heated for 30 minutes at 125°C as compared to pitch heated at the same temperature for 6.5 hours (Figure 2). The hardness of the pitch heated longer was two times that of the pitch heated for only 30 minutes. The plot also demonstrates that as the melt temperature is increased towards 200°C, the hardness increases significantly. Also shown on the plot is the hardness of a sample that was measured after being melted using a propane torch for 30 seconds. The sample smoked excessively and even caught fire during preparation. This sample, however, was still softer than the sample melted at the lower temperature for a longer time.

Figure 2 The effect of pitch melt temperature on hardness



To gain more insight into the effect of melting time on pitch hardness, several samples were prepared at 125°C and 155°C and melted between 30 to 400 minutes. The penetration depths of the resulting samples are plotted in Figure 3. For both temperature ranges, the pitch hardness doubles for longer melting times. It also appears that the pitch hardness stabilizes after approximately 225 minutes of melting. This is most likely the time at which all volatile components of the pitch have burned off at the designated temperature.

Figure 3 The effect of heating (melting) time on pitch hardness



Therefore, if during manufacturing or packaging, the pitch is heated for different time periods, or at different temperatures, the properties of the pitch could dramatically change. Furthermore, once in the optics shop, if the melting procedures are not well controlled to maintain a consistent melt time and temperature, one can expect variation in the performance of each new lap poured.

2.2 Softening Point Method (ASTM Softening Point Test)

Another useful test for verifying pitch parameters is the ASTM Softening Point Test.[4] This is perhaps the only standardized test conducted by most manufacturers of pitch.

The American Society for Testing and Materials (ASTM) has a standard procedure that is used to measure the softening point of bitumen, a procedure which is also known as the ring and ball test. The ASTM designation for this procedure is D36-95.[4] This procedure is relatively easy, although time consuming. A small plug of pitch is prepared and placed in a glycerin bath, the temperature is raised at a controlled rate and the temperature at which the pitch flows a known distance is recorded as the softening point. The softening point for most pitches falls around 50°C. Figure 4 illustrates the set-up for this test. Figure 5 shows the softening point of selected commercially available pitch types. Plotting the softening point versus the penetration depth (Figure 6) shows that the pitch with a lower softening point has a higher penetration depth, as expected. In fact, we found a very linear relationship between the two tests. This allows one to check on results published by manufactures, and allows one to make periodic tests of pitches in stock or in use to make sure they still have the flow characteristics of the original specification.

Figure 4 Softening point apparatus

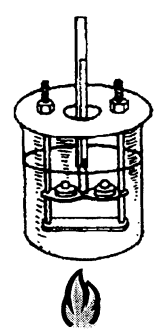


Figure 5 Softening points (using ASTM D36-95) of selected commercially available pitch

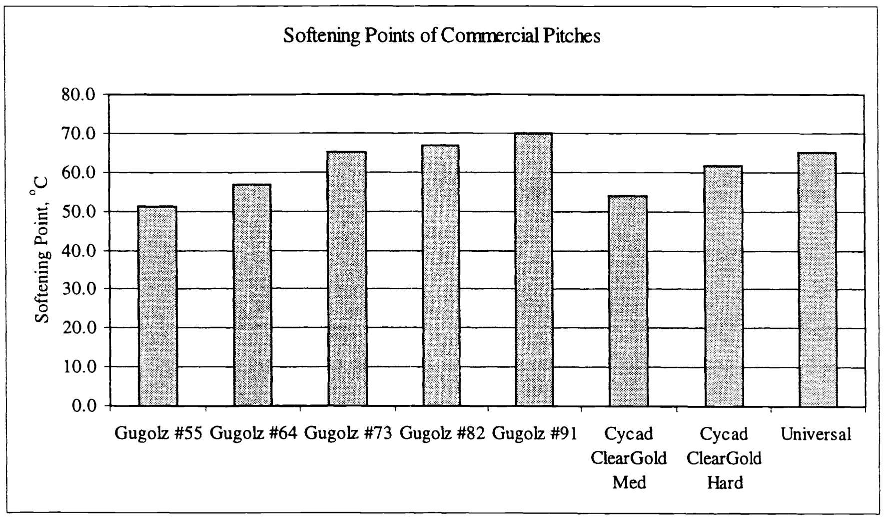
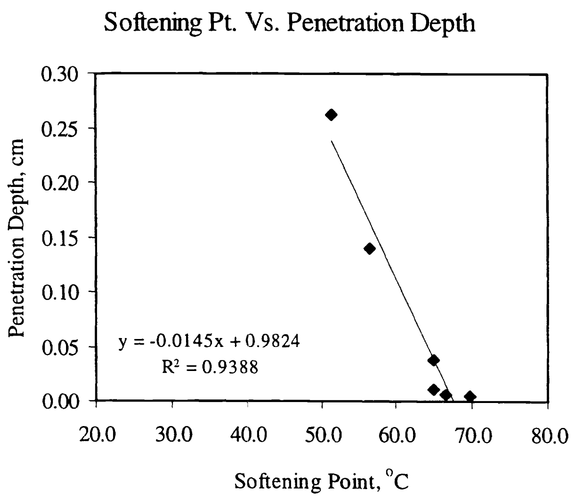


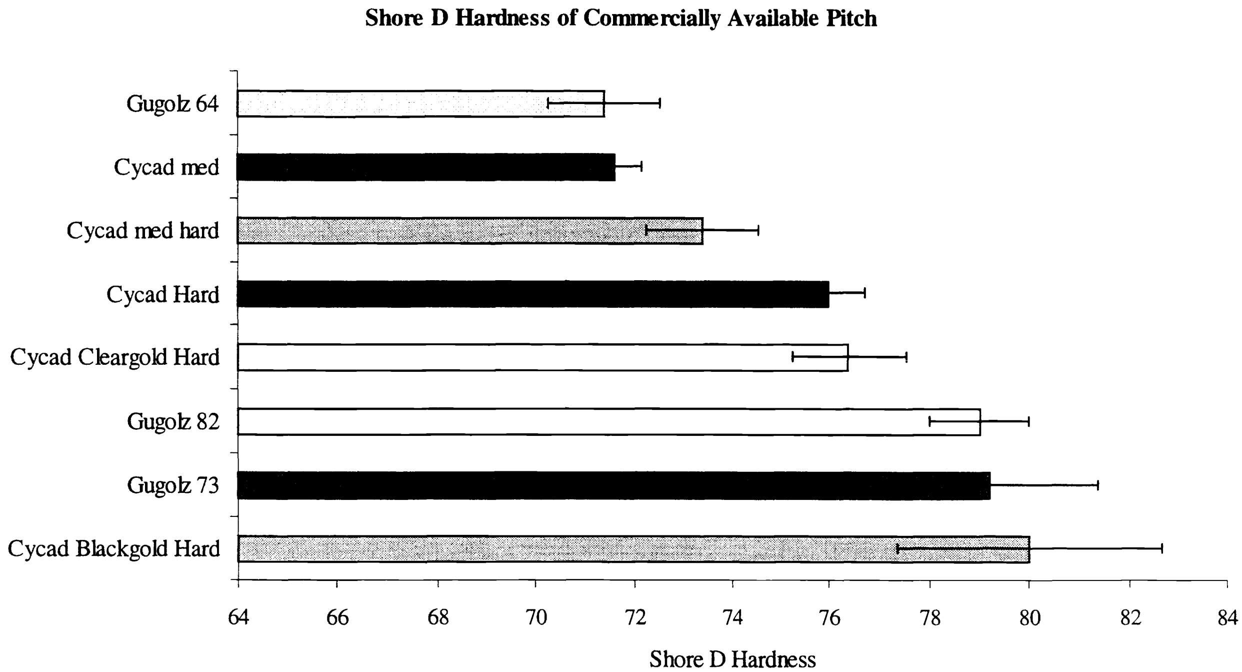
Figure 6 Softening point versus penetration depth



2.3 Shore Hardness

Another method that can be used to verify hardness is a durometer test, specifically, Shore D. This method, however, is not as reliable as the penetration test or the softening point test as evidenced by the high standard deviations shown in Figure 7. It would be very difficult to determine whether a sample was Gugolz #73 or #82 using the Shore D durometer, even though there is a significant difference between the two pitch types.

Figure 7 Shore D Hardness of Commercially available pitch [4.65 kg load at 22.6 $\pm$ 0.5°C][5]



3.0 SIGNS OF TROUBLE AND COMMON PROBLEMS

There are many warning signs, readily apparent to most opticians, which can tip one off that there may be a problem with the pitch. We say "may" because the pitch too often tends to be the scapegoat for other problems associated with a given polishing process. However, the pitch utilized can sometimes contribute to problems such as a chronic torroidal/irregular lap shape on a planetary polishing machine, poor control over lap form and thus workpiece form, scratching of the workpiece, and poor removal rate. Table 2 is a trouble shooting guide for common pitch problems.

Table 2 Trouble Shooting Guide

Problem	Symptom	Comments and Recommendations
Pitch too brittle	Fissures in lap	• Pitch overcooked, pour a new lap • Pitch not properly specified, select a different pitch
Poor lap control	Figure not changing	• Poor contact with the part, evaluate "tackiness" and figure • Measure elastic modulus, or friction coefficient • Insufficient lap relief, cut new grooves
	Figure changing uncontrollably	• Pitch is too soft, measure penetration hardness/viscosity • Temperature fluctuations are changing viscosity • Grooves closing too fast, recut more often or specify a new pitch
Poor removal rate	Lap not charging	• Pitch is too hard, measure penetration hardness/viscosity
Scratching of parts	Scratching of parts	• Pitch is too hard, measure penetration hardness/viscosity • Pitch/environment is too cold • Pitch is cut too rough, harbors contaminants
Poor lap figure	Conditioning tool chatter Chronic torroidal lap shape	• Press out lap with tool (increase load, increase slurry temp.) • Poor pitch flow, poor conditioning tool configuration, etc.

4.0 SOLUTIONS AND RECOMMENDATIONS

Once the lap is formed, there is little one can do to fix the basic properties. Knowing and controlling your materials can save a lot of time and money. In order to achieve this, several things may be done. One needs to define what a good pitch "is" for specific applications. Once a good pitch type is established, physical properties should be measured and a quality checksheet listing the parameters for the properties should be produced. This checksheet should be used to verify that each new lot of pitch received is deemed good prior to using it. Manufacturers or distributors may also be able to provide a certificate of conformance. It is important that lap preparation procedures be well defined, then strictly adhered to. Despite this, the first line of defense is still visual observation. However, once the observation is made of a potential difference in pitch/lap performance, one must follow up the observations with tests to confirm or study potential variations in materials to prevent future occurrence.

SUMMARY

The functional properties of polishing pitch were discussed and methods for making penetration hardness tests and the ASTM Softening Point test were presented. While these tests have been used for decades by opticians and pitch manufacturers, this paper is intended to reinforce the necessity for implementing routine measurements of polishing materials to avoid costly mistakes and enhance the level of consistency in any polishing process. Results showed a significant increase in penetration hardness as a function of both melting temperature and duration. Even at low temperatures, extended heating of "melted" pitch can produce changes in hardness of more than a factor of two. This reinforces not only the need to avoid "overcooking" the pitch during melting, but also for standardizing both the temperature and time taken to obtain consistent results. Although not explored in this paper we note that the dependence of hardness on melting time and temperature should also manifest changes in the "tackiness" or frictional properties of the pitch/workpiece interaction. We observed this both in the prevalence of increased cracking in laps made with overcooked pitch and overall "brittleness" of the resulting material.

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