Instrument for Measuring Thermal and Elastic Behavior of Hide and Modified Hide Materials

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ABSTRACT

An instrument is described for measuring elongation and contraction of skin and leathers, shrinkage temperatures, stress-strain relationships, and torsional deformation under load. Data obtained on a variety of materials are presented.

INTRODUCTION

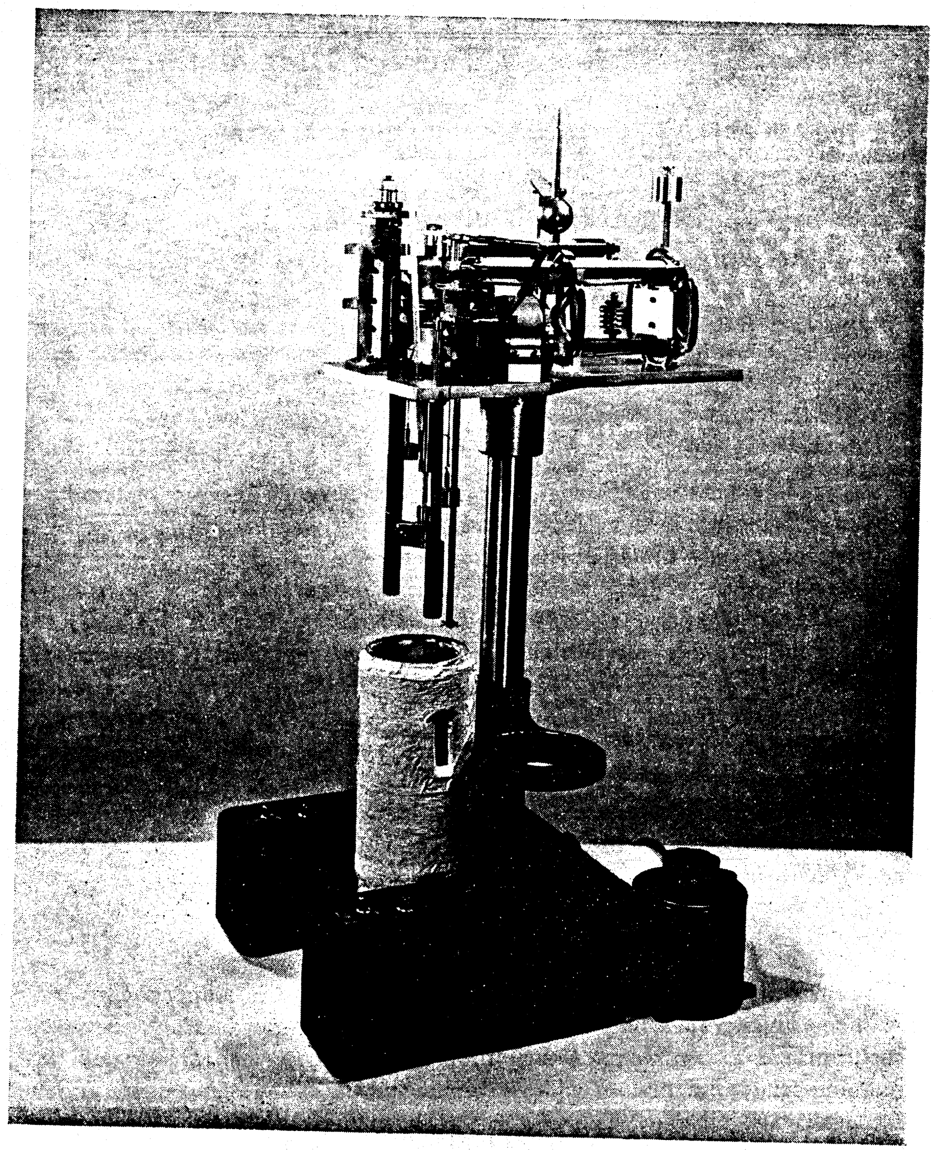
This paper describes an instrument that was designed and constructed to measure quantitatively a variety of the physical constants characteristic of the behavior of hide and chemically modified hide materials. By tests on a single small sample, the instrument permits investigations of: (*a*) elongation and contractile phenomena, (*b*) stress-strain behavior, and (*c*) flexibility in torsion. These studies can be made under various conditions of temperature and in liquid as well as in air. The utility of the instrument in making these investigations is illustrated with observations on cowhide dehydrated with acetone, cowhide tanned with chrome and epoxy resin, and calfskin tanned with canaigre.

Although many instruments for making measurements of this type have been described (1), none of them incorporates all the desired features mentioned above.

APPARATUS AND METHOD

This instrument is purely mechanical in nature and is comprised of two interlinked units which may be operated separately or coordinately. Simply, it incorporates a component for producing and measuring torsion and an adjustable scale and torsion balance for measuring changes in length under constant or variable load. Referring to the photograph and simplified diagram (Figures 1 and 2), one unit consists of a torsion balance, *A*, with a load capacity of 2 kilograms, and a sensitivity of 0.3 gram, in which a spindle, *B*, has been attached to the modified end of the balance beam, *C*. The spindle consists of a cylindrical rod ½ inch in diameter, one end of which has been transformed into a jaw, *D*, for gripping the upper end of the specimen, *Y*. The lower jaw, *D'* is attached to a screw-operated sliding bar, *E*, ½ inch

*One of the laboratories of the Eastern Utilization Research Branch, Agricultural Research Service, U. S. Department of Agriculture.



Courtesy of U.S.D.A. Photo by M. C. Audsley.

FIGURE 1.—Instrument for measuring thermal and elastic properties of hide and leather.

square, by means of an adjustable jacket clamp, *F*. Vertical displacement of the sliding bar is produced by turning a screw by means of a knurled knob, *H*, at its end. The amount of this displacement may be read to 0.001 inch from a scale, *I*, equipped with a vernier. Position of the lower jaw, *D′*, may be changed independent of the scale by loosening the jacket clamp, *F*, and

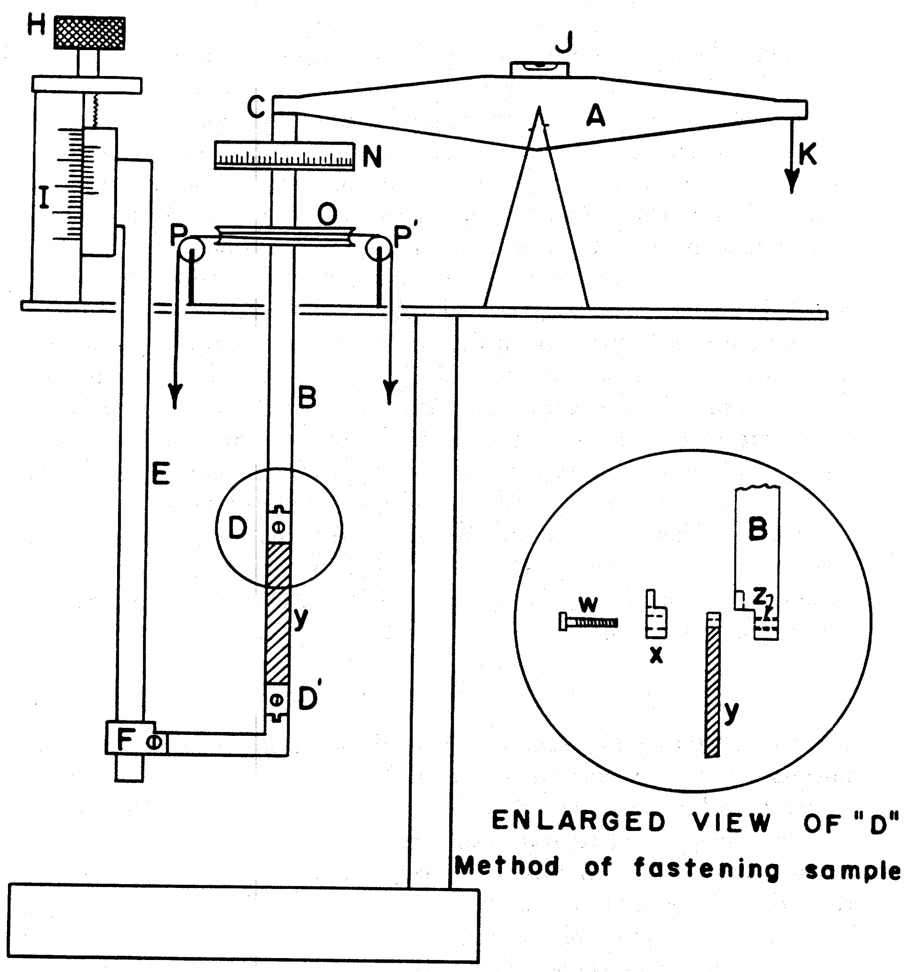


FIGURE 2.—Simplified diagram of instrument showing essential parts.

moving to accommodate samples of different lengths. The design of the jaws is shown in the inset of Figure 2. A ⅛-inch diameter hole is punched in each end of the sample on the center line and ¼ inch in from the end. To clamp the specimen in the jaws a screw, *W*, is passed through the detachable jaw plate, *X*, then through the hole in the sample, *Y*, and finally screwed into the hole, *Z*, provided near the end of the spindle, *B*.

Before the balance system can be operated properly the base on which it is set must be leveled by adjusting the four leveling screws. The null-indicating device of the balance consists of a transit level, *J*, with a sensitivity of 20 seconds. When the sample is placed in position, and all the above adjustments are made, the system operates as follows: A load placed on the free end, *K*, of the balance produces elongation of the sample which in turn causes the balance null-indicator to be off-center. The screw knob is then turned until the level is centered, and the amount of deformation of the sample is read from the scale. When not in use, the balance may be locked in position by means of four grips, whose action is simultaneously controlled by a lever bar,

The torsion unit of the instrument is similar to one described by Clash and Berg (2) and consists of two parallel cylindrical drums, *N* and *O*, one of which is calibrated in degrees and held at zero position by means of a retaining pin (not shown) which fits into a slot on the drum. Attached to the other drum and wound around it both in the same direction are two nylon fishline cords. These cords are passed over two idler pulleys, *P* and *P′*, which are placed at opposite sides of the drum about three inches from it. Equal calibrated weights are then attached to the ends of the cords. The sample to be tested is clamped in the jaws of the instrument as previously described. When the retaining pin is released, the weights fall causing rotation of the drum and spindle which imparts torsional deformation to the sample. The degree of this deformation is measured by direct reading from the calibrated drum, *N*.

OBSERVATIONS

Shrinkage Phenomena.—In investigations of elongation and contractile phenomena, as, for example, in the determination of shrinkage temperature, a sample of hide or leather 2½ inches long and ¼ inch wide is clamped in the instrument and a 10-gram axial load is applied in tension in order to take up any slack in the sample. The length-adjustment screw is turned until the balance-level indicator is centered, then the scale is read. A Dewar flask containing the immersion medium is set in place on a stand so that sample, heater, stirrer, and thermometer are all immersed. When the sample is immersed in the medium, a resetting of the length-adjustment screw is usually required to compensate for the initial swelling. The screw should be repeatedly reset, with a reading after each resetting, until no further changes in the length of the sample take place. Heating and stirring are then initiated. The rate of heating is regulated by means of a variable transformer. The rate of heating chosen for hide materials was approximately 1 °C. per minute for reasons which will be discussed later. The change in length is measured at each desired temperature. The behavior of the sample on cooling may be determined by adding ice or solid carbon dioxide to the liquid medium and following the same procedure as on heating.

Figure 3 shows variation in length with temperature change for *A*, a sample of split cowhide which had been brought to the isoelectric point and then dehydrated with acetone; *B*, a sample of canaigre-tanned calfskin; and *C*, chrome-tanned split cowhide. Upon immersion in tap water, the acetone-

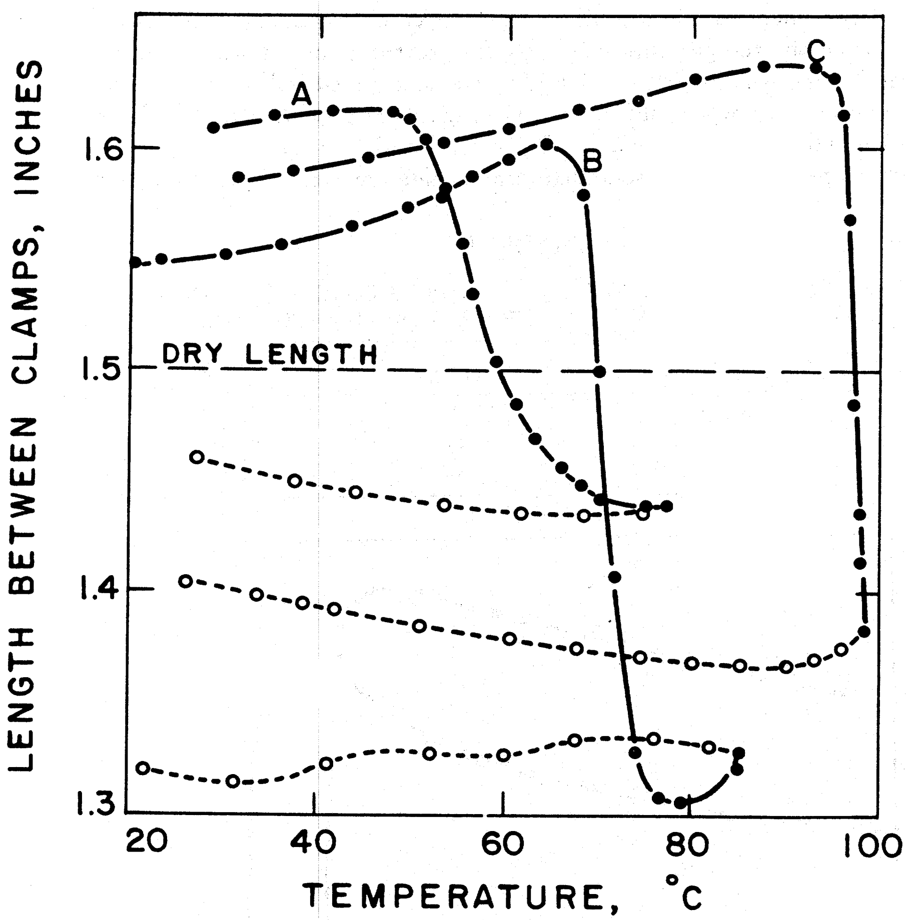


FIGURE 3.—Change in length in water as a function of temperature for: *A*, acetone-dehydrated cowhide; *B*, canaigre-tanned calf; and *C*, chrome-tanned cowhide. Broken line represents cooling curves.

dehydrated hide sample shows the greatest increase in length over dry length, and after 62 minutes in the bath at room temperature no further elongation is noted. The canaigre-tanned calf sample continues to elongate for 36 minutes after immersion and shows the smallest increase in length. The increase in length of the chrome-tanned sample is about midway between that of the acetone-dehydrated hide and canaigre-tanned samples and appears to require the same amount of time to attain constant length as the latter. When no further change in length at room temperature occurs, heating is initiated. All the samples exhibit a further increase in length up to the point where shrinkage occurs, the amount of this increase depending upon the chemical treatment of the sample. The shrinkage temperature, *Ts*, is considered to be the temperature corresponding to the point at which the first decrease in length is observed. The *Ts* values obtained fell in the expected order, that is, chrome-tanned hide, 94°C; canaigre-tanned calf, 65°C; and acetone-dehydrated hide, 49°C. The values obtained for the samples of canaigre-tanned calf and acetone-dehydrated hide are rather low. On cooling

TABLE I

EFFECT OF HEATING RATE ON TEMPERATURE AND RANGE OF SHRINKAGE OF ACETONE-DEHYDRATED COWHIDE

Heating Rate, °C/min	T_s, °C.	Shrinkage Range, °C.
2	55	55–62
1	55	55–60
0.2	54	54–56

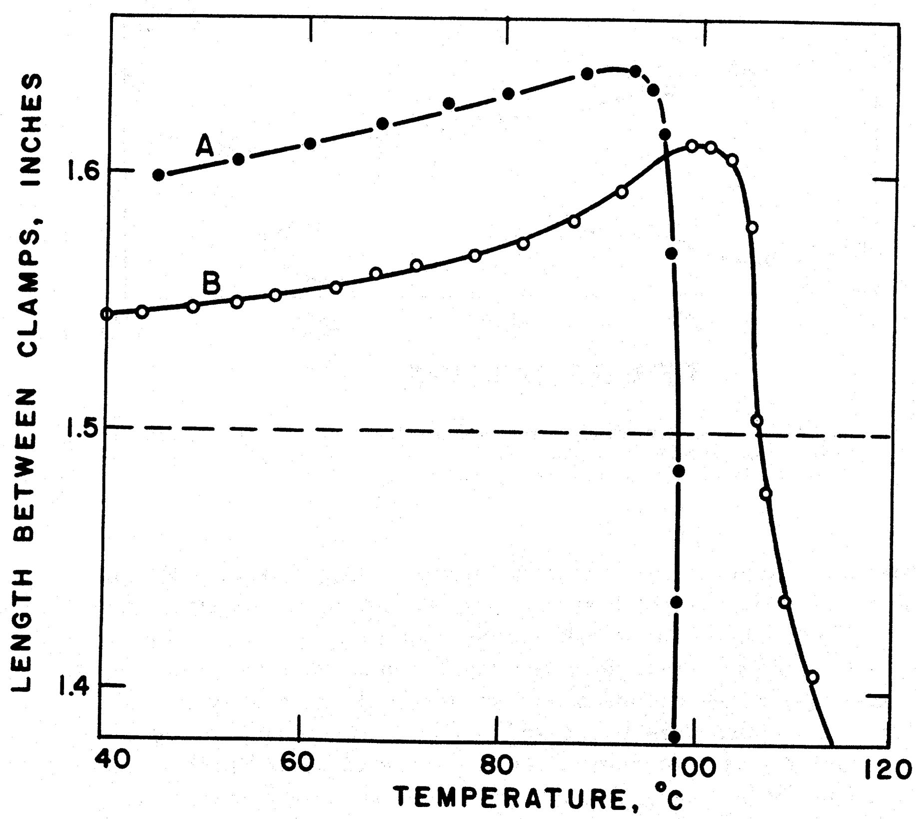


FIGURE 4.—Change in length as a function of temperature for chrome-tanned cowhide; *A*, in tap water pH 7; and *B*, in 25% water 75% glycerol solution.

at a rate of 2°C. per minute, the shrunken samples of acetone-dehydrated hide and chrome-tanned cowhide exhibit a slight increase in length, while the length of the canaigre-tanned sample remains essentially unchanged.

To show the effect of heating rate on the *Ts* value and rate of shrinkage, three test specimens were cut adjacent to each other from another preparation of acetone-dehydrated hide. These specimens were all tested in distilled water by the method just described, except that each was heated at a different rate. The results are shown in Table I. From these results we may conclude that shrinkage temperature is practically independent of heating rate, at least at rates below 2°C. per minute with only the 10-gram load acting on the sample. The shrinkage range, however, varies significantly with heating rate.

In comparison with values obtained by standard methods the values presented in Table I are approximately 5°C. low. This is probably due in part to

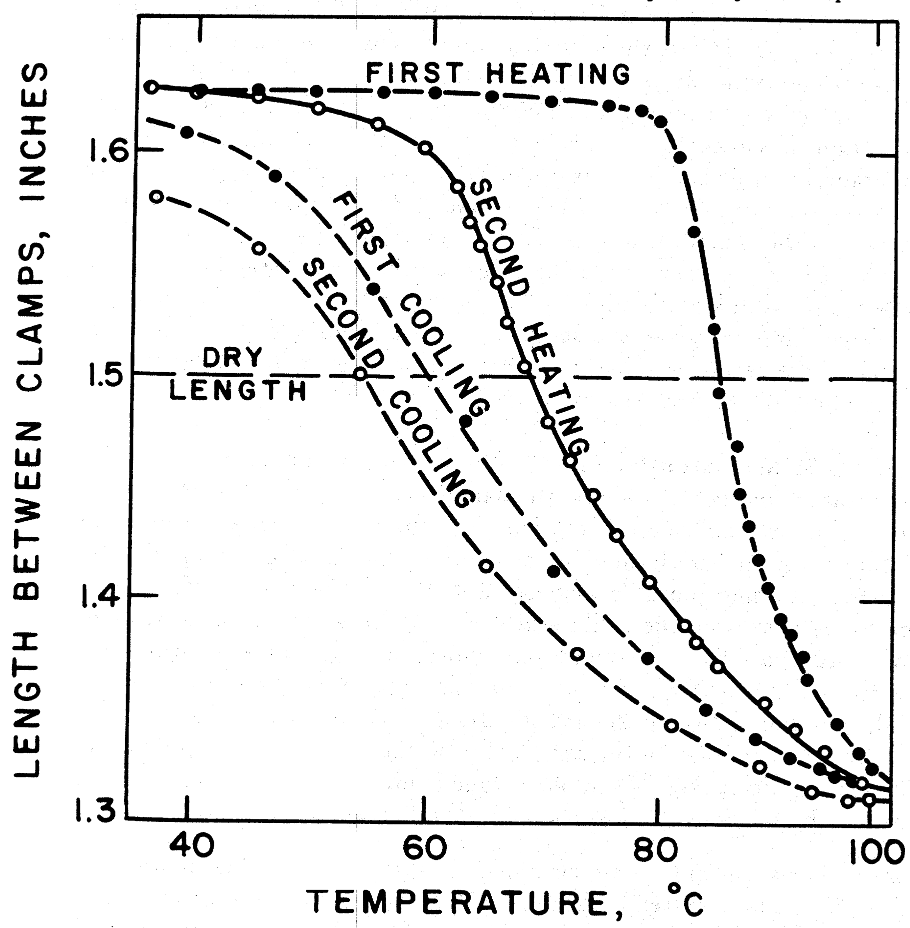


FIGURE 5.—Change in length in water as a function of temperature of cowhide tanned with epoxy resin.

the sensitivity of this instrument which detects changes in length as small as 0.001 inch and also the small load (10 grams) used compared to 100 grams for some of the other methods.

The effect of different liquid media on similar samples of chrome-tanned cowhide may be seen in Figure 4. The values for curve *A* are obtained in tap water while those for curve *B* are obtained using glycerol containing 25% water. The sample tested in tap water shows a much greater increase in length on immersion, and the *Ts* value obtained in the glycerol solution is 8 °C. higher than that obtained in water.

An example of reversible shrinkage behavior is shown in Figure 5, in which length is plotted as a function of temperature for a sample of split cowhide tanned with epoxy resin (3) and tested in tap water. After immersion in water at 28 °C., the sample increases in length for 12 minutes. However, on heating, no essential increase in length occurs; in fact a slight decrease is observed as the shrinkage temperature is approached. At a temperature of 78 °C. a rapid decrease in length begins which continues until the boiling point of the water is attained. On cooling at a rate of 2 °C. per minute, the sample length increases rapidly until the sample is cooled to a temperature of approximately 46 °C.; thereafter a more gradual increase takes place. At the end of the first cooling period, the sample length at 28 °C. (not shown) is the same as the length attained at the start of the heating. On heating a second time, the sample begins to shrink at about 58 °C., approximately 20 °C. below the original shrinkage temperature of the sample. Although not shown in Figure 5, at the end of the second cooling cycle the sample is 0.35 inch shorter than at the beginning of the first heating cycle.

Torsional Measurements.—In determining torsional deformation under load—a measure of the flexibility—the sample is, as before, subjected to a 10-gram "straightening" load in tension. The balance is locked in position to maintain constant length, and the appropriate torsional load is applied by releasing the retaining pin as previously described. The amount of torsional deformation is read from the calibrated drum. In these experiments torque-loads of 0.0125 to 0.03 inch-pound are employed to produce deformations varying from 100° to 500°. Between torsion measurements the balance is unlocked, and the change in length is adjusted as previously described, to maintain constant tension on the sample during the entire test. Applying the formula relating dimensions of sample, torque-load, and degree of deformation given by Clash and Berg (4), a torsional modulus may be calculated.

Figure 6 shows a graph in which apparent torsional modulus in water is plotted as a function of temperature. These values were obtained simultaneously with those reported in the curves shown in Figures 3 and 5, and therefore they correspond to the same samples. These values are calculated

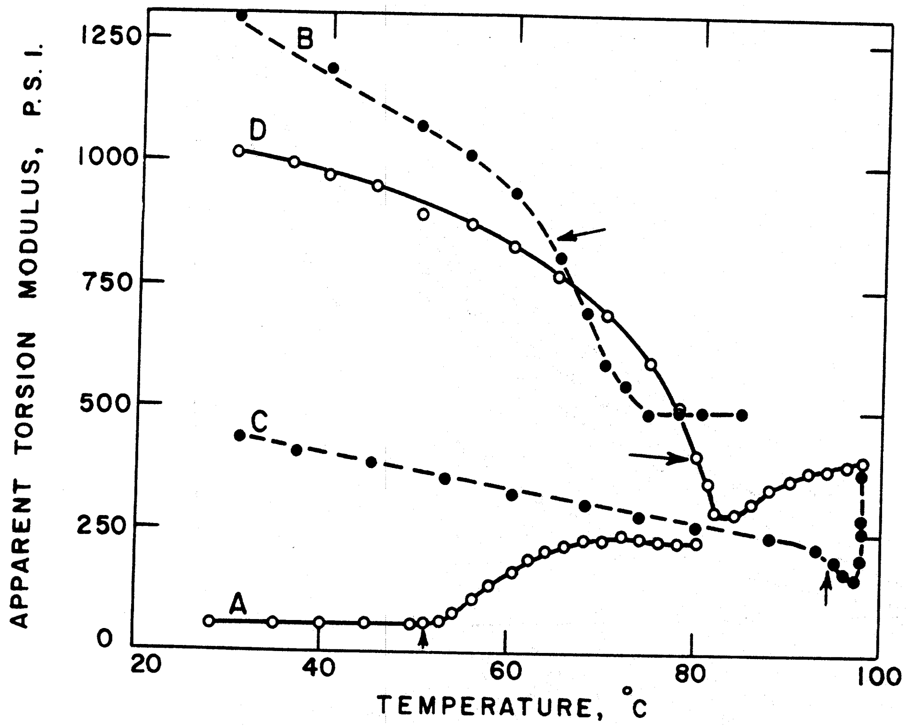


FIGURE 6.—Apparent modulus of elasticity in torsion in water as a function of temperature for: *A*, acetone-dehydrated cowhide; *B*, canaigre-tanned calf; *C*, chrome-tanned cowhide and; *D*, epoxy-tanned cowhide. Arrows indicate shrinkage temperatures.

on the basis of the cross-sectional area of the dry sample and therefore cannot be regarded as absolute, since no correction has been applied to compensate for the effect of the water.

At room temperature the sample with the lowest apparent modulus was the acetone-dehydrated hide (*A*), with the moduli of the samples tanned with chrome (*C*), epoxy resin (*D*), and canaigre (*B*), being approximately 7, 20, and 26 times as great, respectively. These values appear to show that stiffening occurs on tanning and that the stiffness of the wet samples seems to vary with the type of tannage. For the tanned materials the modulus values exhibit a gradual decrease with increasing temperature in the initial portion of the curves, while the modulus of the acetone-dehydrated hide remains constant. When the shrinkage temperature is attained, the tanned samples all show a rapid decrease in apparent modulus. The acetone-dehydrated sample again behaves differently in that shrinkage causes an increase in modulus. The final portions of the curves are all of a different nature: the chrome-

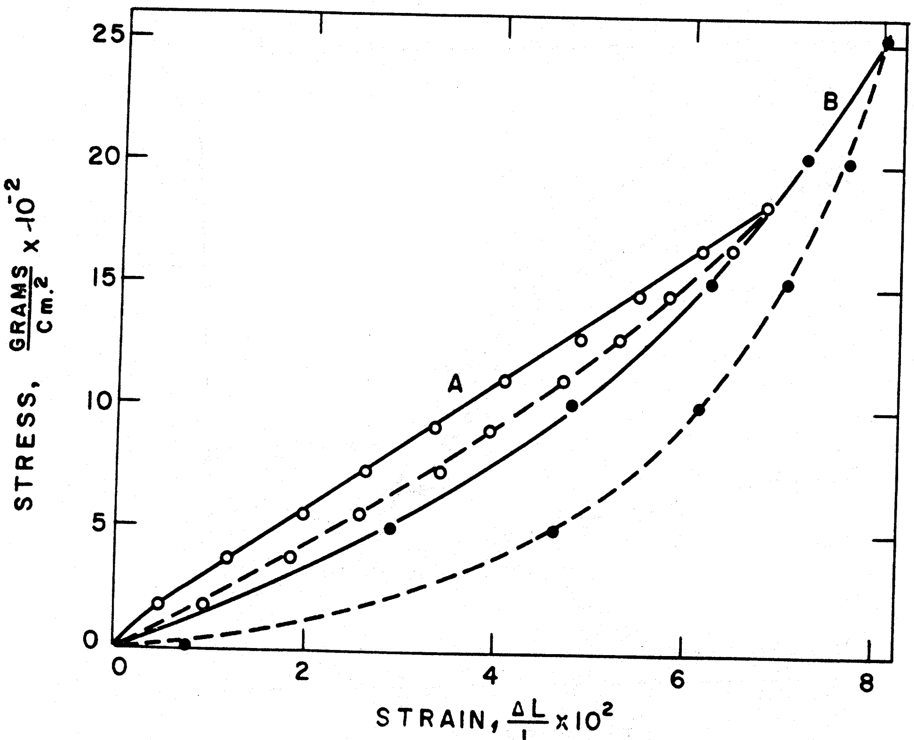


FIGURE 7.—Stress-strain curves for: *A*, cowhide shrunk 3 minutes in boiling water; and *B*, acetone-dehydrated cowhide as received. Determinations were made in distilled water at 25°C.

tanned hide showing a sharp increase and the epoxy-resin-tanned hide, a slight increase, while the canaigre-tanned calf and acetone-dehydrated hide remain constant.

Stress-Strain Measurements.—The instrument may also be employed in measuring the stress-strain behavior of hide and leather samples. This determination is made by mounting the sample in the jaws of the instrument, putting the bath in place, and applying loads to the free end of the torsion balance. The load is increased in equal increments, and the change in length produced by each increment of load is measured by means of the scale and vernier. Figure 7 shows an example of a graph obtained from this type of measurement on two samples of acetone-dehydrated cowhide. Curve *B* shows values for isoelectric acetone-dehydrated hide before shrinking, and curve *A*, values for hide after shrinking in boiling water for three minutes. Both specimens were tested in distilled water at room temperature, and conditioning loads of 1000 and 710 grams respectively were first applied in order to remove most of the nonrecoverable elongation. The cross-sectional areas used in calculating the stress were determined by the method employed by Wiederhorn and Reardon (5).

The shrunken material shows an essentially linear stress-strain relationship on increasing load, and the modulus of elasticity obtained was 425 pounds per square inch. On stepwise removal of the load a hysteresis loop is obtained. On the other hand, the stress-strain curve of the unshrunk sample shows initially a large nonlinear increase in strain per unit stress in the stress range of 0 to 500 grams per square centimeter. At stresses from 500 to 1000 grams per square centimeter the stress-strain behavior parallels that observed for the shrunken sample, and thus the modulus is the same for both in this region. On further increase in stress, above 1500 grams per square centimeter, the modulus of the unshrunk material increases by a factor of two over that of the middle portion of the curve. It may be noted that the modulus values reported here are of the same order of magnitude as those obtained by the torsion method.

It is possible to follow the course of a tanning reaction *in situ* on small specimens by measuring torsional deformation, stress-strain behavior, and changes in length while tanning is taking place. Additional uses for this instrument are found in evaluation of torsional and contractile properties of other polymeric materials such as rubbers and plastics.

SUMMARY

An instrument is described which was designed and constructed in this laboratory for the purpose of investigating hydrothermal and elastic properties of hide and modified hide materials. The apparatus incorporates a torsion-producing and-measuring component and an adjustable scale and torsion balance for measuring changes in length under variable load when necessary. The measurements can be made as a function of temperature in air or liquid media as desired. The apparatus has the following qualities: it is versatile and measures fundamental properties over a useful range of temperature; it is simple in operation, relatively inexpensive in cost, and suitable for routine use in industry; and it requires specimens which are easy to prepare. Methods for measuring shrinkage temperature and amount of shrinkage, effects of tanning on torsional stiffness, and stress-strain behavior before and after shrinkage are discussed, and examples of data obtained are given.

ACKNOWLEDGMENTS

We are grateful to Dr. E. M. Filachione and his colleagues of the Hides, Tanning Materials and Leather Section of this Laboratory for preparation of all samples mentioned herein and to D. R. Killen for making many of the reported measurements.

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