

The surface energy concept is also useful for understanding the behavior of so-called surface active agents or surfactants. The condition of thermodynamic equilibrium in a liquid mixture means that one component is generally preferentially adsorbed at the surface if the adsorption causes a decrease in the surface energy. The term surfactant is reserved for molecular species which strongly adsorb at the surface even when it is at very low concentration in the bulk liquid. Surfactants are almost always present in natural waters and are very important in many biological and industrial processes.

The interface between a solid and a gas or between a solid and a liquid also has a surface free energy associated with it. Figure 1 a shows a liquid droplet at rest on a solid surface surrounded by air. This system contains three different types of interfaces each with a characteristic surface free energy per unit area. The state of minimum free energy for the system then involves trade-offs in surface area for the various interfaces. The region of contact between the gas, liquid, and solid is termed the contact line. The liquid-gas surface meets the solid surface with an angle θ measured through the liquid which is known as the contact angle. The contact angle attains a value that minimizes the free energy of the system and is thus a characteristic of a particular solid-liquid-gas system. The system shown in Fig. 1 a has a smaller contact angle than that shown in Fig. 1 b. The smaller the contact angle, the better the liquid is said to wet the solid surface.

Measurement of surface tension is important in many fields of science and engineering, as well as in medicine. A number of standard methods exist for its measurement. In many systems of interest the surface tension changes with time, perhaps for example because of adsorption of surfactants. Several of the standard methods can be used to measure dynamic surface tension if it changes slowly with time. Special techniques have been developed to measure dynamic surface tensions for systems that evolve very rapidly.

2. Mechanics of Fluid Surfaces

Sonic methods of measuring surface tension depend on the mechanics at the line of contact between a solid, liquid, and gas. When the system is in static mechanical equilibrium the contact line is motionless, meaning that the net force on the line is zero. Tangential forces acting on the contact line arise from the surface tensions of the converging solid-gas, solid-liquid and liquid-gas interfaces, denoted by γ_{SG} , γ_{SL} , and γ_{LG} respectively (see Fig. 2). The condition of zero net force along the direction tangent to the solid surface gives the following relationship between the surface tensions and contact angle θ ,

$$\gamma_{SG} = \gamma_{SL} + \gamma_{LG} \cos \theta. \quad (1)$$

This is known as Young's equation. The contact angle is thus seen to be dependent on the surface tensions between the various phases present in the system and is therefore an intrinsic property of the system.

As discussed in the introduction, the surface tension of a droplet causes an increase in pressure in the droplet. This can be understood by considering the forces acting on a curved section of surface as illustrated in Fig. 3a. Because of the curvature, the surface tension forces pull (the surface toward the concave side of the surface. For mechanical equilibrium, the pressure must then be greater on the concave side of the surface. Figure 3b shows a saddle-shaped section of surface in which surface tension forces oppose each other, thus reducing the required pressure difference across the surface. The mean curvature of a two dimensional surface is specified in terms of two principal radii of curvature, R_1 and R_2 . A detailed mechanical analysis of curved tensile surfaces shows that the pressure change across the surface is directly proportional to the surface tension and to the mean curvature of the surface,

$$P_A - P_B = \gamma \left(\frac{1}{R_1} + \frac{1}{R_2} \right), \quad (2)$$

where γ is the surface tension and the quantity in brackets is twice the mean curvature. The sign of the radius of curvature is positive if its center of curvature lies in phase **A** and negative if it lies in phase **B**. Eq. (2) is known as the Young-Laplace equation and the pressure change across the interface is termed the Laplace pressure. Measurement of the Laplace pressure for a surface of known curvature then allows a determination of the surface tension.

Several methods of surface tension measurement are based on measurement of the static shape of an axisymmetric drop or bubble or on the point of mechanical instability of such drops or bubbles. In a gravitational field a drop or bubble which is attached to a solid support assumes a non spherical shape. Figure 4a shows the shape of a hanging droplet, also known as a pendant drop, and Fig. 4b shows a so-called sessile drop. The reason for the deviation of the shape from that of a sphere can be understood from Eq. (2). The hydrostatic pressure changes with depth more rapidly in a liquid than in a gas. The pressure difference across the surface of a pendant drop in air therefore increases from top to bottom requiring an increase in the mean curvature of the surface according to Eq. (2). The drop in Fig. 4a has a neck at the top which means that the two principal radii of curvature have opposite signs and cancel to some extent. At the bottom of the drop the radii of curvature have the same sign thus making the mean curvature larger. The Young-Laplace equation can be written as a differential equation in terms of the interface position for an axisymmetric surface. In a gravitational field as

$$\frac{2}{b} \gamma \Delta \rho g z = \gamma \left[\frac{1}{\phi(1+\phi_z^2)^{3/2}} - \frac{\phi_{zz}}{(1+\phi_z^2)^{5/2}} \right], \quad (3)$$

where ϕ is the distance of the surface from the symmetry axis as a function of the vertical position z , b is the radius of curvature at the apex of the drop or bubble, $\Delta \rho$ is the density difference between the two fluid phases, and g is the acceleration of gravity. Numerical integration of Eq. (3) allows one to compute the shape of an axisymmetric fluid interface. Comparison of computed shapes with experimentally measured shapes of drops or bubbles is a useful method of measuring surface tension. Eq. (3) can be made non dimensional by dividing through by γ/b . The resulting equation contains only one parameter, $\beta = \Delta \rho g b^2 / \gamma$, which is called the Bond number. The shape of an axisymmetric drop, bubble, or meniscus depends only on this one dimensionless parameter. The Bond number can also be written as $\beta = 2 b^2 / a^2$ where $a = \sqrt{2\gamma/\Delta \rho g}$ is known as the capillary constant and has units of length.

Several dynamic methods of measuring surface tension are based on capillary waves. Capillary waves result from oscillations of the liquid surface for which surface tension is the restoring force. The frequency of the surface oscillation is thus dependent on the surface tension and wavelength. Very low amplitude capillary waves with a broad range of frequencies are always present on liquid surfaces owing to thermal fluctuations. Larger amplitude capillary waves can be excited by purposely perturbing the surface.

3. Standard Methods and instrumentation for Surface Tension Measurement

A number of commonly used methods of measuring surface tension exist. The choice, of a method depends on the system to be studied, the degree of accuracy required, and possibly on the ability to automate the measurements. In the discussion that follows, these methods are grouped according to the kind of instruments used in the measurements. Since the information presented

for each method is necessarily brief, readers who are interested in constructing their own apparatus should consult the more detailed treatises in references [1]-[4]. A list of commercially available instruments is given in Table 1 together with vendor names and approximate, 1996 prices.

Capillary rise method

If a thoroughly clean glass capillary tube with a very uniform diameter of less than 1 mm is brought into contact with the surface of a liquid in a container at least 8 cm diameter, and if the liquid wets the tube material with an angle of less than 90°, then the liquid is drawn up into the tube as shown in Fig. 5a. In order to make accurate surface tension measurements by this method the liquid must wet the tube with a contact angle of 0°. The surface tension is directly proportional to the height of rise, h , of the liquid in the tube relative to the flat liquid surface in the larger container. By applying Eq. (2) to the meniscus in the capillary tube, the following relationship is obtained:

$$\Delta\rho gh = \frac{2\gamma}{b}, \quad (4)$$

where, b is the radius of curvature at the center of the meniscus and $\Delta\rho$ is the density difference between liquid and gas. For small capillary tubes, b is well approximated by the radius of the tube itself. For larger tubes or for increased accuracy, the value of b must be corrected for gravitational deformation of the meniscus (p. 12 of [1]). This method is primarily useful for pure liquids and is capable of high accuracy at relatively low cost.

Wilhelmy plate / du Noüy ring methods

Measurement of the pull of a liquid surface directly on a solid object is the basis for two of the standard methods discussed here. In the Wilhelmy plate method the solid object is a flat, thin plate which the test liquid completely wets with a zero contact angle. The plate is suspended vertically from a delicate balance which is zeroed with the plate suspended in air. The test liquid is brought into contact with the bottom of the plate, causing the plate to be pulled down into the liquid by the surface tension force. The force applied to the plate from above is then increased to bring the bottom edge of the plate level with the flat surface of the liquid (Fig. 5b). This avoids the necessity to make buoyancy corrections to the measurement. The surface tension is computed from the force measurement, f , using

$$\gamma = f \cos \theta / [2(l + t)], \quad (5)$$

where l is the length of the plate and t is its thickness. The contact angle θ is often assumed to be zero for common liquids on clean glass or platinum plates but one should be aware of the error caused by non-zero contact angle. No other correction factors are necessary for this method and the fluid density does not need to be known.

The du Noüy ring method is known as a maximum pull method of which there are several variations. The technique is to contact the liquid surface with a ring and then measure the force continuously as the surface is lowered until a maximum force, f_{\max} , is recorded. The maximum force typically occurs just before the ring detaches from the surface. The surface tension is obtained from the formula

$$\gamma = (f_{\max} / 4 \pi R) \cdot F(R^3/V, R/r), \quad (6)$$

where R and r are the radii of the ring and wire respectively as indicated in Fig. 5c, V is the volume of liquid raised by the ring, and F is a correction factor (F is tabulated in Table 5, p. 132 of [4]). The duNoüy ring method requires knowledge of the liquid density, ρ_l , in order to determine V from $V = f_{\max} / \rho_l$. This method requires the liquid to wet the ring with zero contact angle and is not suitable for solutions which attain surface equilibrium slowly.

A single instrument is normally capable of performing either Wilhelmy plate or duNoüy ring measurements. Some commercially available instruments can perform the complete measurement procedure automatically. Computer interfacing with a Wilhelmy plate instrument allows automatic data logging which can be used to follow changes in surface tension with time in surfactant solutions.

Maximum bubble pressure method

The maximum bubble pressure method (MBPM) involves direct measurement of the pressure in a bubble to determine the surface tension. A tube is lowered to a depth t in the test liquid and gas is injected to form a bubble at the tip of the tube as shown in Fig. 5d. The increase in bubble pressure, P_b , over ambient pressure, P_a , arising from the interface is given by the sum of a hydrostatic pressure and Laplace pressure,

$$\delta p = P_b - P_a - \Delta \rho g t = \Delta \rho g Z_b + 2\gamma / b. \quad (7)$$

As a new bubble begins to form Z_b increases while b decreases resulting in an increase in pressure in the bubble. Ultimately b increases as the bubble grows larger thus reducing the pressure. The pressure in the bubble thus reaches a maximum when δp reaches a maximum which in turn can be theoretically related to the surface tension. Equation (7) can be rewritten in nondimensional form as follows:

$$\frac{r}{X} = \frac{r}{b} + \frac{r}{a} \frac{Z_b}{b} \left(\frac{\beta}{2} \right)^{1/2}, \quad (8)$$

where r is the tube radius, X is a length defined as $X = 2\gamma / \delta p_{\max}$, a is the capillary constant, and β the Bond number. The dimensionless quantity r/X depends *only* on r/a , the relationship being determined by numerical solutions of Eq. (3). Tabulations of this relationship together with Eq. (8) are used to calculate the surface tension by an iterative procedure (p. 18 of [1]). The standard MBPM requires a knowledge of the fluid densities, tube radius, and depth of immersion of the tube.

A differential MBPM uses two tubes of different diameters immersed to the same depth. The difference in the maximum bubble pressure for the two tubes, ΔP , is measured which eliminates the need to know the immersion depth and makes the method less sensitive to errors in the knowledge of the liquid density. For the differential MBPM, surface tension is computed from (See [5])

$$\gamma = A \Delta P \left[1 + (0.69 r_2 \rho_l / \Delta P) \right], \quad (9)$$

where r_2 is the radius of the larger tube, ρ_l is the liquid density, and A is an apparatus-dependent constant which is determined by calibration with several standard liquids [6].

Automated MBPM units are commercially available (Table 1) including one which allows for on-line process measurements under conditions of varying temperature and pressure.

Pendant- / Sessile-drop methods

The shape of an axisymmetric pendant or sessile drop (Fig. 4) depends on only a single parameter, the Bond number, as discussed above. The Bond number is a measure of the relative importance of gravity to surface tension in determining the shape of the drop. For Bond numbers near zero, surface tension dominates and the drop is nearly spherical. For larger Bond numbers the drop becomes significantly deformed by gravity. In principle the method involves obtaining an image of the drop and comparing it to theoretical shapes obtained by integrating, Eq.(3) for various values of β and b . Once β and b have been determined from shape comparison, the surface tension is calculated from:

$$\gamma = \Delta\rho g b^2 / \beta. \quad (9)$$

in practice, the method of shape comparison has traditionally involved the manual measurement of several characteristic dimensions (see Fig.4) of the drop from a photographic print. A ratio formed from these characteristic dimensions is correlated to a shape factor which, together with one of the absolute dimension measurements, is used to calculate the surface tension (p. 27 of [3],[7]). This method is quite accurate but very tedious and time-consuming, in recent years the whole procedure has been automated using digital imaging and computer analysis of these images. Typically several hundred coordinates on the edge of the drop arc located by computer analysis of the digital image and then Eq.(3) is solved numerically for various values of the parameters β and b until a best fit to the experimental edge points is found [8]. A digital image of a pendant drop can be analyzed for surface tension on a desktop computer in one or two seconds [9].

Drop weight / volume methods

A pendant drop will become unstable and detach from its support if it grows too large. The weight of the detached portion is related to the surface tension of the fluid by

$$\gamma = (mg/r) \cdot F(r/V^{1/3}), \quad (10)$$

where mg is the weight of the detached drop, r is the radius of the tip from which the drop hangs and V is the volume of the detached drop. An empirical correction factor, F , is tabulated as a function of $r/V^{1/3}$ (p. 49 of [3]). For Eq. (10) to apply, drops must be formed slowly. Measurements typically involve weighing the accumulated liquid from a large number of drops to determine the average weight per drop. The density of the fluid must be known in order to determine the drop volume and then obtain the factor F . Another method involves measuring the volumetric flow rate of liquid to the tip while counting the drops. The density of the fluid must also be known in order to determine the drop weight. The latter method allows for automation of measurements [10].

Spinning drop method

The spinning drop method is a shape-measurement method similar to the pendant and sessile drop methods. However the deformation of the drop in this case is caused by radial pressure gradients in a rapidly spinning tube. This method is normally used for measuring interfacial tensions between immiscible liquids. A horizontal glass tube with sealed ends is filled with the more dense liquid through a filling port. The tube is then spun about its axis while a drop of the

lower density liquid is injected into the tube. The pressure in the outer liquid increases from the center of the tube toward the walls as a result of the spinning motion. The pressure gradient forces the drop to move to the center of the tube and causes it to elongate, while surface tension opposes elongation. Measurement of the maximum drop diameter, $2r_{\max}$, and length, $2h_{\max}$, together with the angular velocity of rotation, Ω , allows for calculation of the surface tension according to (see [11])

$$\gamma = \frac{(r_{\max})^3}{2r_{\max}^*} \Delta\rho\Omega^2, \quad (11)$$

where r_{\max}^* is correlated to the aspect ratio r_{\max}/h_{\max} . The spinning drop method is particularly suited for measuring ultralow interfacial tensions (10^{-2} to 10^{-4} mN/m).

Specialized methods

Dynamic surface tension

in an aqueous solution of soluble surfactant, the surface tension decreases following creation of new surface area because of adsorption of surfactant molecules. Surfactant adsorption kinetics can be studied by measuring the change in surface tension with time. For a dilute solution, the rate of change of surface tension is often slow enough that automated versions of static methods such as the Wilhelmy plate or pendant drop methods can be used to follow the change in surface tension. In concentrated solutions in which large changes in surface tension can occur within a fraction of a second following surface creation, a dynamic method must be applied. A liquid jet issuing from an elliptical orifice has stationary waves on its surface, the wavelength of which are related to the surface tension. This oscillating jet method has been used to measure surface tension for surface ages as low as **0.6 ms** [12]. A dynamic version of the maximum bubble pressure method has been applied to measure dynamic surface tension at surface ages down to 0.1 ms [13].

Surface viscoelasticity

A liquid surface covered by a monolayer of surfactant exhibits viscoelastic behavior. In addition to surface tension, the surface rheology is characterized in terms of dilatational and shear elastic moduli as well as dilatation] and shear viscosities. The dilatational properties in particular are important in a variety of situations from foam stability to the functioning of the human lung. Surface dilatation] elasticity is proportional to the change in surface tension for a given change in surface area. This elasticity is seen to depend on the rate of change of surface area for both soluble and insoluble surfactant monolayers which indicates that relaxation processes are active. These relaxation processes give rise to the surface dilatational viscosity. For the case of soluble surfactants, one of the relaxation processes is the adsorption or desorption of molecules at the surface. The equilibrium dilatational elasticity of an insoluble monolayer can be measured by slowly expanding or compressing the monolayer in a Langmuir trough while monitoring the surface tension with a Wilhelmy plate apparatus [14].

Studies of surface rheology at high rates of surface expansion or compression are of interest for both soluble and insoluble surfactants. Methods for studying dilatational viscoelastic properties include transverse and longitudinal capillary wave methods [15- 18], a modified maximum bubble pressure method [19], and the oscillating bubble method [20,21].

Measurements at extremes of temperature and pressure

Several of the standard methods described in this article can be adapted to make surface or interfacial tension measurements at extreme temperatures and/or pressures. The most common methods used to measure the surface tension of high-temperature molten metals and semiconductors are the maximum bubble pressure method [22] and the pendant- and sessile-drop method [23-25]. Measurement of the interfacial tension between oil and a second immiscible phase at high pressure and elevated temperature is of interest for understanding aspects of enhanced oil recovery. The pendant drop method has been applied under pressures of 82 MPa at 449 K [26], while a capillary wave method has been applied at 136 MPa and 473 K [27].

Interfacial tension

Measurement of the interfacial tension between two immiscible liquids can present special difficulties. Measurement by the capillary rise, du Noüy ring, or Wilhelmy plate methods is problematic in that the contact angle is often non zero. The pendant drop [7] and drop weight [28] methods can both be applied provided that the densities of the two liquids are sufficiently different. The pendant drop method in particular is widely used for interfacial tension measurement. Interfacial tension between liquids of equal density can be measured by the maximum bubble pressure method [29] and several other specialized methods [30,31]. Ultralow values of interfacial tension can be measured by the spinning drop [32], pendant drop [33], and capillary wave methods [34].

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Figure Captions

- Figure 1: Illustration of contact angles and wetting.
- Figure 2: Mechanics at the contact line.
- Figure 3: Mechanics of curved surfaces.
- Figure 4: (a) Pendant drop and (b) sessile drop showing the characteristic dimensions used in calculating surface tension from drop shape.
- Figure 5: Geometries for: (a) capillary rise method, (b) Wilhelmy plate method, (c) du Noüy ring method, and (d) maximum bubble pressure method.

Table J: **Commercially available instruments.**

Method	instrument Type	Manufacturer(s)	Approximate Price (Range)
capillary rise	manual operation	Fisher	\$79
Wilhelmy plate / du Noüy ring	manual operation, mechanical balance	CSC, Fisher, Kahl	\$2,000-\$4,000
Wilhelmy plate / du Noüy ring	manual operation, electrobalance	KSV, Lauda, NJMA	\$4,000-\$11,000
Wilhelmy plate / du Noüy ring	automatic operation, electrobalance	Cahn, Krüss, KSV, NIMA	\$9,000-\$24,000
maximum bubble pressure	automatic operation	Kruss, Lauda, Sensadyne	\$5,000-\$23,000
pendant / sessile drop	manual operation	Krüss, Rame-Hart	\$7,000-\$10,000
pendant / sessile drop	automatic operation	AST, FTA, Krüss, Rame-Hart	\$13,000-\$20,000
drop weight / volume	automatic operation	Krüss, Lauda	\$16,000-\$21,000
spinning drop	manual operation	Krüss \$20,100	

Note: Price ranges may reflect differences in degree of automation, the number of accessories included, or variation in price between manufacturers.

Table 2. Manufacturers and suppliers of instruments for surface tension measurement.

AST Products
 9 Linnell Circle
 Billerica, MA 01821-3902
 (508) 663-7652

Brinkmann Instruments, Inc.
 (distributor for Lauda tensiometers)
 One Catiaque Road
 P. O. 130X 1019
 Westbury, NY 11590-0207
 (800) 645-3050

Cahn Instruments
 1001 Fourier rive
 Madison, WI 53717
 (608) 831-5515

CSC Scientific Company, Inc.
 8315 Lee Highway
 Fairfax, VA 22031
 (800) 458-2558

CTC Technologies, inc.
 (distributor for NIMA tensiometers)
 7925-A North Oracle Road, Suite 364
 Tucson, AZ 85704-6356
 (800) 282-8325

First Ten Angstroms (FTA)
465 Dinwiddie Street
Portsmouth, VA 23704
(800) 949-4110

Fisher Scientific
711 Forbes Ave.
Pittsburgh, PA 15219-4785
(800) 766-7000

Kahl Scientific Instrument Corp.
P.O. Box 1166
El Cajon, CA 92022-1166
(619) 444-2158

Krüss USA
9305-B Monroe Road
Charlotte, NC 28270-1488
(704) 847-8933

KSV Instruments USA
P. O. Box 192
Monroe, CT 06468
(800) 280-6216

Rame-Hart Inc
8 Morris Ave.
Mountain Lakes, NJ 07046
(201) 335-0560

Sensa Dyne instrument Div.
Olc-Dyne Research Corp.
P.O. Box 30430
Mesa, AZ 85275-0430
(602) 924-1744

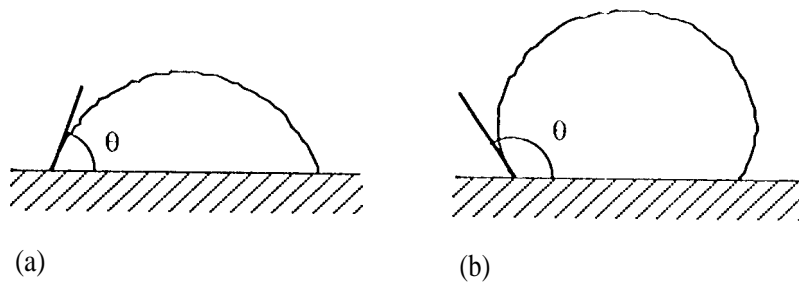


Figure 1

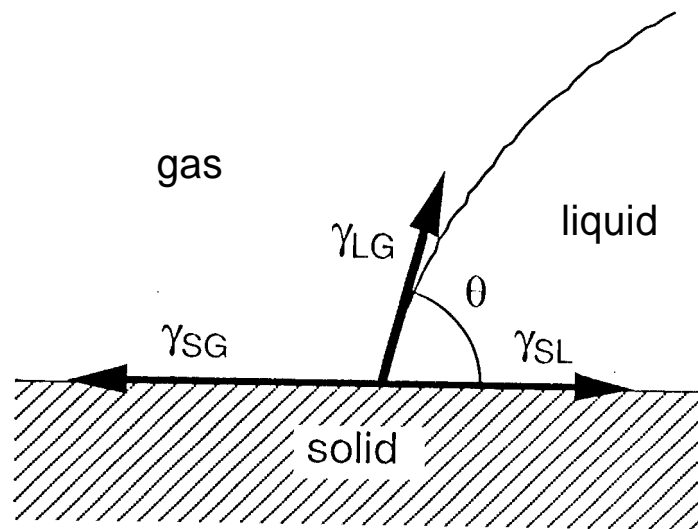


Figure 2

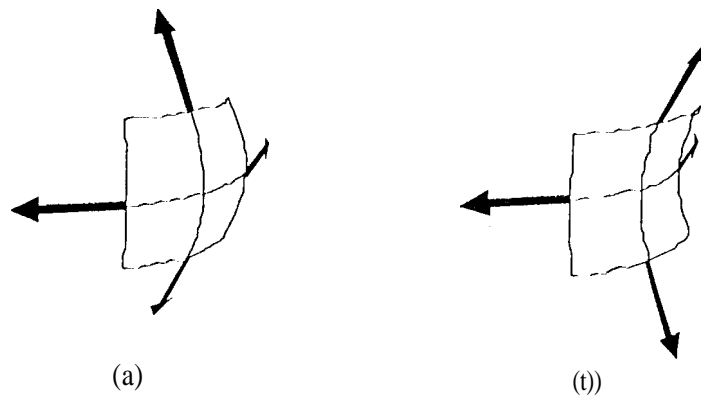


Figure 3

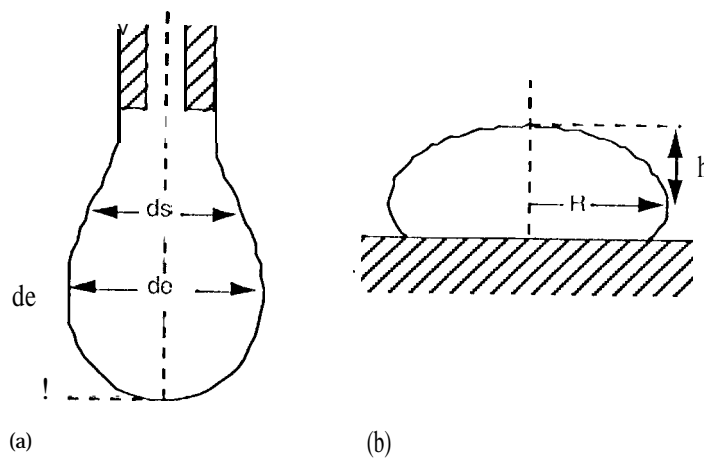


Figure 4

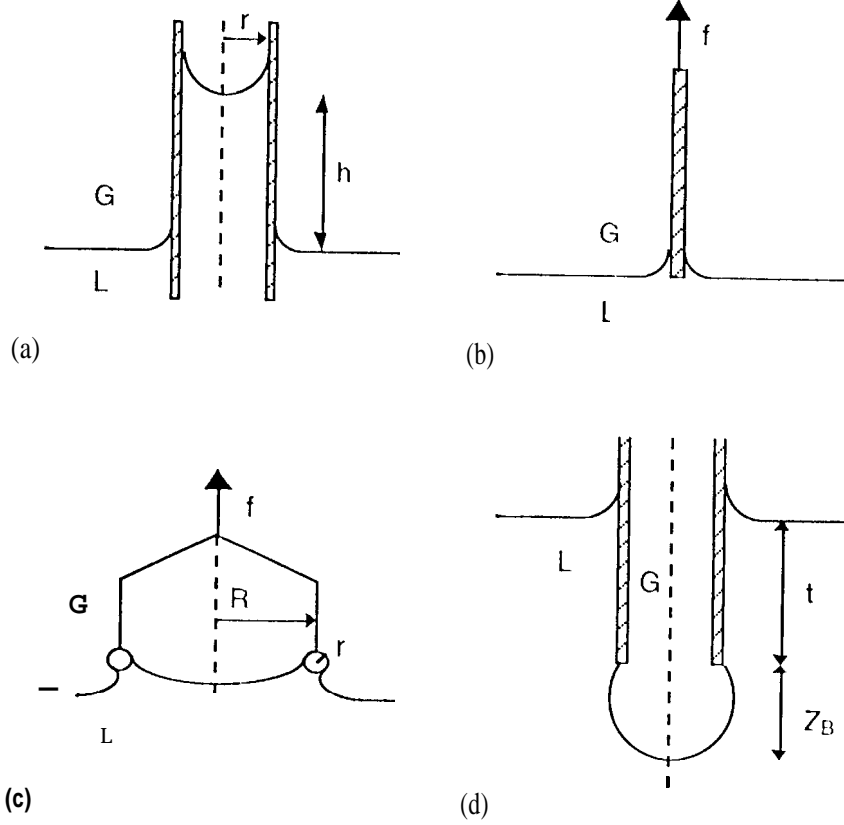


Figure 5