DEPARTMENT OF COMMERCE
BUREAU OF STANDARDS
George K. Burgess, Director

THE TESTING OF RUBBER GOODS

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THE TESTING OF RUBBER GOODS

ABSTRACT

This circular describes the methods used at this bureau in the testing of rubber goods. The physical tests commonly employed are described in detail and the machines used for this purpose, many of which were designed at this bureau, are illustrated and described. Data are given showing the effect of different conditions on the tensile properties of rubber. Special attention is given to the effect of temperature on the physical tests. The circular also contains a brief outline of the methods of collecting crude rubber and the processes used in the manufacture of various rubber articles. The standard procedure of physical tests and chemical analyses may be found in Federal Specifications Board Specification No. 59 or Bureau of Standards Circular No. 232 issued by this bureau.

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I. INTRODUCTION

The testing of rubber goods is receiving increased attention, and the necessity of maintaining standards of quality has resulted in the preparation of master specifications for the purchase of rubber goods by the Government. The purpose of this circular is to describe the methods used for testing rubber at this bureau, and in order that the fundamental principles involved in these tests may be appreciated, reference is made to the sources and preparation of rubber, and a brief description of some of the manufacturing processes is given.

Much remains to be done in the refinement of testing methods now in common use and in the development of new tests, the results of which shall more nearly express the particular qualities desired in rubber. This applies to crude rubber as well as to materials fabricated from rubber. The technical staffs of the rubber industry are working together along this line, and it is hoped that more exact criteria of quality will result.

II. MATERIALS USED IN THE INDUSTRY

1. CRUDE RUBBER—SOURCES, COLLECTION, AND PREPARATION

Crude rubber is obtained by the coagulation of a milky fluid called latex which is contained in a special cell system (laticiferous system) of certain trees, vines, shrubs, and roots, and also from the cellular tissue of certain shrubs and vines. The laticiferous system, which is distinct from the sap-bearing cell system, generally lies between the outer bark and the cambium, the soft, rapidly growing tissue from which new wood and bark are formed; by cutting through the bark the latex is obtained as a white to cream colored, more or less viscous, fluid containing ordinarily from 30 to 35 per cent of solids. This operation is termed "tapping." Rubber exists in the latex as small particles varying in size from 0.5 to 3 microns (0.00002 to 0.00012 inch), which are suspended in a serum. The rubber is separated from the latex by coagulation. This is accomplished on the plantation by acetic acid. The coagulated rubber is separated from the serum, washed thoroughly, and dried.

Rubber is also obtained by spraying the latex into a heated chamber. The latex flows upon an elevated, horizontal, rapidly revolving disk, which hurls it into a current of heated air. The water is thus removed and the rubber descending in fine particles collects on the floor of the chamber in the form of a sponge. The sponge is compressed into blocks for shipment. Latex sprayed rubber contains all the water-soluble material originally present in the latex.

Latex, with a small amount of ammonia added to prevent coagulation, is used to impregnate fibrous materials, such as fabrics and cords for automobile tires. When it is used as a cement the danger resulting from the employment of inflammable organic solvents is avoided.

(a) WILD RUBBER

(1) Sources.—Rubber-bearing species are indigenous to considerable tracts of the tropical and subtropical zones of South and Central America, Asia, Africa, and Australia. The chief botanical orders are the Euphorbiaceæ, Apocynaceæ, Urticaceæ, and Compositæ.

(*a*) *Euphorbiaceæ.*—The most important genus of this order is the "Hevea," and from the species *H. brasiliensis* at least threefourths of the world's output of rubber is obtained. From this we get the famous Pará rubber. There are two main districts in which fine Pará rubber is prepared: (1) The "islands" at the mouth of the Amazon River and (2) the "up river" regions near and above Manáos.

The scraps of rubber adhering to the trees and tapping cups are compressed into irregular masses and sold as "negroheads." Upriver negroheads are generally termed "scrappy." Island negroheads go by the name of "Sernamby." A third variety of negrohead, the Cametá, comes from the district of that name in southwest Pará.

Among the other varieties of rubber from *H. brasiliensis* may be mentioned:

(*a*) Matto Grosso, fine and entrefine,
(*b*) Matto Grosso, virgin sheets,
(*c*) Matto Grosso, negroheads.
From the Province of Matto Grosso, Brazil.
(*d*) Mollendo (Bolivian Pará).
(*e*) Peruvian (fine, coarse, or scrappy).

Although the rubbers referred to above consist mainly of *H. brasiliensis*, the latices of other species of Hevea, such as *spruceana*, *itaube*, *discolor*, *similis*, and *speciosa*, are also employed to some extent in their preparation.

Another genus of the Euphorbiaceæ is the Manihot. The most important commercial variety is the Manicoba or Ceara, which comes from the Province of Ceara, Brazil.

(*b*) *Apocynaceæ.*—The bulk of native African rubbers belongs to this order, and the main genera are Funtumia, Landolphia, and Clitandra.

The only species of Funtumia which is of commercial importance is *F. elastica.* The natural habitat of the tree is on the Gold and Ivory Coasts, in Uganda, and in other parts of tropical Africa. The rubbers are known as Gold Coast lumps, Ivory Coast lumps, Niger niggers, Benin lump, Congo, and Cameroon.

The species of Landolphia are all creepers or vines, which attain considerable size. They yield red and black Kassai, Upper Congo balls, and Equateur from the Congo region; virgin sheets and pinky from Madagascar; Sierra Leone niggers, twists, and cakes, all from the Sierra Leone and southern rivers; and Konakry, Sudan, and Bassam niggers and twists from French West Africa.

The species of Clitandra are likewise vines and are largely distributed throughout Africa.

In certain Brazilian Provinces the Hancornia, which yields the rubber known commercially as Mangabeira or Matto Grosso sheets, occurs in considerable quantity.

(*c*) *Asclepiadaceæ.*—A rubber of high resin content termed Jelutong is obtained from species of Dyera, the most common being *D. costu-*

lata. Species of Dyera are among the commonest forest trees in Borneo, the Malay Peninsula, and Sumatra. Pontianak is a variety of Jelutong grown in South Borneo.

(*d*) *Urticaceæ.*—The most important species of this order occur in tropical Asia, Mexico, and South and Central America.

Ficus elastica is found mainly in Asia (Burma, Ceylon, Malaya, Java, and India). The principal commercial brands are Assam, Rangoon, Java, and Penang.

The species of *Castilla* (principally *C. elastica* and *C. ulei*) represent the indigenous rubber trees par excellence of Mexico and Central America. We have Peruvian "caucho" rubber from Peru, Negro caucho from Ecuador, and Mexican strips and the different "West Indian" or Centrals from Costa Rica, Nicaragua, Honduras, San Salvador, and Guatemala.

(*e*) *Compositæ.*—The name guayule is derived from the Spanish "hay" and the Indian "hule" or rubber yielder. The rubber-bearing species is *Parthenium argentatum.* It is a gray woody shrub of spreading habit, growing generally between 1 and 3 feet high. It differs from other rubber-bearing plants in that it has no latex but contains the rubber in the cellular tissue of the epidermis and to a small extent in the branches and leaves. The plant, when cut to the ground, will send up new shoots if rain falls within a certain time.

Guayule is now cultivated scientifically on plantations in California and southern Arizona. The quality of the product has been much improved in recent years.

(2) COLLECTION AND COAGULATION.—In the Amazon district the trees are tapped by means of a small iron hatchet having a blade about 1 inch broad. The incisions are generally made in the form of V-cuts or oblique lines. The first tappings are made at a height of from 6 to 7 feet, and subsequent incisions at roughly 1½ to 2 inches below the first one until the base of the tree is reached. About 35 consecutive daily tappings are therefore necessary to complete a tapping line. A fresh tapping line is then commenced at approximately 18 inches from the first one. The latex is collected in small metal or earthenware cups fixed to the tree by means of moist clay. The annual yield of rubber from the average Amazon tree is about 5 pounds. The latex is transferred to pails and is coagulated by a smoking process.

In a small brazier a fire is made from palm nuts. A long wooden rod or paddle is so arranged, one end on a crosspiece, the other on the operator's knees, that it can be rolled either over the top of the chimney and so be exposed to the full volume of smoke, or over the basin containing the latex. The operator pours a small quantity of latex over the wooden paddle and thus forms a thin film of liquid. This is rotated in the smoke until it sets. A fresh quantity of latex is then poured upon the first film, smoke is again applied, and so on until a "biscuit" or ball of rubber of the required size (20 to 100 pounds), consisting of innumerable thin layers tightly adhering to one another, is formed. The ball is then removed from the paddle and is ready for export as the "fine Pará" of commerce.

There is some difficulty in collecting the latex from the Manihot trees, owing to its viscous character and its property of rapidly coagulating. The natives allow the latex to coagulate naturally as it flows down the tree, and the bulk of the rubber is collected in tears and scraps which are stripped off. A certain amount of latex reaches the ground and this is collected on leaves or directly from the soil.

The African rubber trees of the genus Funtumia are tapped in a manner similar to that used on the Hevea trees. The collecting is done entirely by the natives, and relatively little is known of their methods. Funtumia latex coagulates readily on boiling and most native methods are based on this fact.

The methods employed by the natives for coagulating vine latices are of the most diverse kinds. Thus, red Kassai is said to be obtained by smearing the latex on the body and allowing the natural heat to evaporate the water, after which the rubber is stripped off. Black Kassai is obtained by a combined boiling and smoking process. Some of the "ball" rubbers are obtained by applying a coagulant, such as salt, to the cuts made in the vine. The thread rubber thus obtained is wound into a ball of the desired size. More salt is added at intervals so as to maintain constant coagulation. Vines do not readily lend themselves to tapping. They are generally cut down and bled to death.

Jelutong is obtained from a number of species of Dyera. The trees grow to a very large size, those having a diameter of from 4 to 6 feet being quite common. A mature tree will yield about 100 pounds of latex per year with 40 tappings. The latex is rich in solids and will yield about 65 per cent of wet but solid Jelutong. The latex is coagulated by the natives, who use curious mixtures of kerosene, copper sulphate, and alum. The solid matter in Jelutong, however, is quite largely resin and contains only a small percentage of rubber.

The wild guayule shrub is generally collected by pulling up the entire plant, which is baled for shipment to the factory for extraction. Special methods are used to obtain the crude rubber because it is held in the cells of the plant. The dry plant yields about 9 per cent of pure rubber, although the percentage is often greater. When guayule is cultivated the shoots are cut off near the ground without destroying the plant. The yield is from 10 to 20 per cent of the weight of the dry shrub. Three types of processes have been used for the extraction of rubber from the plant: (1) The alkali process, in which the shrub is boiled with a solution of caustic alkali; (2) the solution process, in which the rubber is extracted with carbon bisulphide or some other solvent; and (3) the mechanical process. The great bulk of guayule rubber is obtained by the last process. The shrub is crushed and ground in pebble mills with water. The material is then run into settling tanks where the water-logged woody fiber sinks while the rubber floats and is skimmed off. It is then sheeted and washed on rubber mills and dried. The rubber so obtained is quite dark in color on the surface and contains about 20 per cent of resin, which can be removed. When used with the better grades of rubber, as plantation sheets and crêpes, good results are obtained with modern compounding methods.

(b) PLANTATION RUBBER

(1) Sources.—The plantation rubbers are obtained chiefly from Ceylon, the Federated Malay States, Dutch East Indies, Borneo, and the Pacific islands.

The tree which is now almost exclusively grown on these plantations is the *Hevea brasiliensis.* The Hevea grows in a narrow belt on both sides of the Equator, provided there is plenty of moisture. Before planting, a great amount of work must be done to clear the land, by cutting down the trees and underbrush, which are burned when dry. The deadwood is removed and not allowed to rot upon the ground. From this point on, a vast amount of labor is required to keep out the weeds. The seeds are carefully selected from trees which give the greatest yield of rubber, and extreme care is taken in their propagation. One or two year old plants from the nurseries are set out during the rainy season at from 50 to 200 to the acre. In close planting there may be 400 to 500 trees per acre. The trees increase in height from 6 to 10 feet a year and in girth from 3 to 5 inches. They are not ready for tapping until 5 or 6 years old. The annual yield from such young trees is less than a pound of rubber each, and gradually increases as the trees grow older. The mature trees yield as much as 15 pounds or more of rubber per year.

The first Hevea seeds from which the plantations were started were smuggled out of Brazil and no care was taken to collect them from the best trees. The propagation of the trees by budding is being tried on some new plantations, but it is not yet certain that trees of uniformly high yield can be obtained in this way.

(2) Collection and Preparation for the Market.—Methods of tapping rubber trees undergo frequent change. The herringbone system consists in making a series of oblique cuts which run into a central channel from one side. In the basal V system the cuts meet in the channel from each side. The present practice is one cut per tapping on one-quarter to one-half of the circumference. Cuts

must be made with care. Each cut removes a strip of bark one-thirtieth to one-twentieth of an inch wide. Methods of tapping are still a matter of experiment. Trees are tapped from daylight to about 9 a. m. On some plantations, trees are tapped daily; on others they are tapped on alternate days. Period tapping consists in collecting the latex for a month and allowing the tree to rest for a month. When tapping is resumed, the tree appears to have lost the habit of yielding latex and does not produce its maximum yield for a short time.

Plantation latex is generally coagulated by the addition of a small quantity of acetic acid. For producing pale crêpe advantage is taken of the bleaching action of sulphurous acid by adding sodium bisulphite to the latex. Formic acid has replaced acetic acid as a coagulant on some plantations because only half the quantity is required.

The amount of coagulant largely determines the character of the coagulum, which may be soft like the coagulum of milk obtained with rennet, or may have the consistency of a stiff jelly. The stiffer coagula are rolled and washed with more difficulty than the softer. The treatment to which the rubber coagulum is subjected has an important bearing on the quality and market value of the rubber. One of two courses is usually adopted: (*a*) The rubber is merely sheeted by the action of washing rolls, by which process an appreciable quantity of the nonrubber constituents of the latex is retained. In order to avoid mold or tackiness, the rubber is thoroughly smoked. (*b*) The rubber is converted into thinner sheets called crêpe and thoroughly washed during the process to remove the other constituents of the latex as far as possible. The washing rolls squeeze out the serum, or watery portion from the coagulum and wash out the excess of the other nonrubber constituents of the latex. These rolls are of much the same construction as those used in the factory for washing. (See fig. 1.)

The rubber as it leaves the washing rolls is in sheets about one-eighth inch thick and 10 to 14 inches wide; the length may vary from a few feet up to 30 or 40 feet. These sheets are hung in a room at a temperature not higher than 120° F. until they are dry. Sometimes the room is kept full of smoke during the drying process. Almost any hardwood, coconut husks, or the like may be used to produce smoke. Smoke acts as an antiseptic and preservative of rubber. The sheets of rubber are pressed into blocks for export. Crêpe rubber is air-dried at about 100° F. and dried in vacuo at about 120° F.

It is interesting to note the tremendous development of the production of plantation rubber. According to statistics[1] published

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[1] Rubber Division, United States Department of Commerce.

by the United States Department of Commerce, practically none was produced in 1900; in 1910 about 8,000 tons, and in 1926 more than 580,000 tons were produced. The plantation acreage increased from slightly over 1,000,000 acres in 1910 to about 4,300,000 acres planted area in 1923. The annual production of rubber, mostly wild, from tropical America and Africa in 1910 was about 70,000 tons, and in 1925 was about 40,000 tons. Unless plantation production methods are adopted or unless market prices for the commodity prevail at a high level over long periods this amount will not be increased. The net imports of the United States for each year from 1921 to 1925 have averaged about 73 per cent of the total imports into all countries.

An illustrated description of the production of plantation rubber may be found in a publication of the United States Department of Commerce entitled "The Plantation Rubber Industry in the Middle East."

(c) SYNTHETIC RUBBER

Synthetic rubber has been made by the gradual polymerization of the hydrocarbons butadiene, monomethylbutadiene (isoprene), and dimethylbutadiene (methylisoprene). Each yields a different type of rubber of somewhat different composition and properties. The parent substance of natural rubber is isoprene and many attempts have been made to produce isoprene on a commercial scale. The so-called English synthesis employs higher alcohols and aldehydes which in turn may be obtained from starch. The German synthesis makes use of the coal-tar products cresol and phenol. Acetone is an important raw material which can also be obtained from starch. Several promising syntheses of isoprene based on the use of acetone are possible. The ultimate success of synthetic rubber will not be assured until it is possible to reproduce the condition that is called the degree of polymerization or aggregation of natural rubber. This involves its molecular and physical structure. It is a greater problem than the mere synthesis of a crystalline, organic compound. Synthetic rubber oxidizes readily, requires accelerators for vulcanization, and its physical properties are comparable with those of the poorer grades of natural rubber. Unless synthetic rubbers can be made as good as the natural rubbers and as cheaply, they will not be manufactured commercially.

(d) CHEMICAL AND PHYSICAL PROPERTIES OF RUBBER

Rubber belongs to the class of compounds known to the chemist as hydrocarbons; that is, substances which contain only the elements carbon and hydrogen. Harries, Weber, and other investigators determined that the formula for the rubber molecule is polyprene $(C_5H_8)x$, which is derived from the simpler hydrocarbon isoprene

(C_5H_8). All attempts to make synthetic rubbers are based upon the acceptance of this formula for the rubber molecule.

The specific gravity of clean commercial rubber is about 0.92, but varies with the species, method of coagulation, and purity; the values obtained by various investigators are from 0.91 to 0.97. On warming, unvulcanized rubber becomes soft and sticky and is partly decomposed by the heating. When the rubber is cooled, it does not regain its original properties but remains as a semifluid, sticky mass. At temperatures below the freezing point of water rubber loses its elasticity and becomes rigid, and on immersion in liquid air it becomes brittle. It is insoluble in water, but upon soaking in water for a long time it will absorb considerable quantities. It dissolves in a number of organic solvents, such as benzene, chloroform, carbon bisulphide, naphtha, etc. Rubber is a poor conductor of electricity and heat. It is not affected by alkalies or dilute acids, but concentrated sulphuric acid or nitric acid attacks it. It is gradually oxidized in air, with the formation of resin.

2. GUTTA-PERCHA AND GUTTA-BALATA

These substances are not rubber, but have many properties similar to those of rubber, and are used in the rubber industry.

Gutta-percha is obtained from various trees, belonging to the natural order Sapotaceæ, growing on the Malay Peninsula and the Archipelago. The trees of the species *Palaquium oblongifolium* and *Palaquium obovatum* are the main sources. *Palaquium oblongifolium* is being raised on plantations at Tjipetir, Java. The gutta is obtained from the leaf by a mechanical process. The plantation product has a much higher market value than that of gutta from other sources. The latex from uncultivated areas is collected generally by cutting down the tree and ringing the bark at intervals of 12 to 18 inches along the trunk. The milky fluid fills the grooves, soon coagulates, and is scraped off with a knife. Some latices do not coagulate quickly, and these are collected in vessels. They are gently boiled with or without the addition of water. The raw gutta is cut up, softened in hot water, washed on a washing machine, and then forced through a strainer. After a second washing the inclosed water is forced out by a kneading machine and the mass is sheeted out in 5-foot slabs one-eighth to one-fourth inch in thickness. The method of collecting the latex by cutting down the trees is extremely wasteful.

Commercial gutta-percha is hard and tough, but on warming to 115 to 122° F. it can be pressed into any shape, which it will retain on cooling. Its main use is for the insulation of submarine cables. It is also used for temporary dental fillings, leather cements, and for golf balls.

Gutta-percha consists of 30 to 84 per cent of "gutta," a rubber-like material, and 10 to 60 per cent of resin, which consists of albane, a crystalline resin, and fluavile, a yellow amorphous resin, in the ratio of about 2 to 1.

Gutta-balata is obtained from the latex of the *Mimusops balata* and *Mimusops globosa* trees growing in British, Dutch, and French Guiana. The commercial product comes in slabs about one-fourth inch thick, is horny, and from white to dark cream in color. It is collected by tapping as well as by felling the trees in the same manner as gutta-percha, for which it is the only substitute. It has similar properties, although it is somewhat softer, owing to the fact that it contains more of the softer resin fluavile. It contains the resins in the proportion of approximately 2 parts of albane to 3 parts of fluavile. The main use of gutta-balata is in the manufacture of belting for power transmissions. It is also used in golf balls.

3. RECLAIMED RUBBER

The object in reclaiming rubber is to restore useful properties to the scraps of worn-out articles, so that the material may be used again. The aim is to restore to the vulcanized rubber the properties of crude rubber. This has not been accomplished fully, for no one has yet succeeded in removing the combined sulphur and regenerating the rubber molecule in its original state.

Reclaimed rubber is a plastic product prepared by subjecting vulcanized rubber scrap to processes of grinding, chemical treatment, and refining. Over 140,000 long tons were produced in 1925, and about 175,000 tons in 1926.

The first commercial reclaimed rubber product was known as "rubber shoddy." Old boots and shoes were ground, the loosened fiber blown out, and the residue treated with live steam for 8 or 10 hours. Later, in a modification of the process, the fiber was disintegrated by treatment with sulphuric acid. "Rubber shoddy," imperfect as it was, proved to be of value in rubber footwear, clothing, and chiefly in carriage cloth, in which it gave excellent service. Some rubber manufacturers regrind uncured and even vulcanized factory scrap and use it in new batches of compounds. During 1926 more than 13,000 long tons of such scrap was used in this manner.

Nowadays used rubber is reclaimed in large plants, under close technical supervision. The product obtained is plastic and mixes readily with new rubber, sulphur, and other compounding ingredients. Rubber compounds consisting partly of reclaimed rubber serve a useful purpose in the production of footwear and rubber heels, wire insulation, hose, and tires.

To be successful, the treatment of scrap rubber demands considerable investment in power plant, digesters, rubber mills, and similar necessary equipment. The plants must have ample space for the storage of raw material and products, and must be located close to an adequate and constant source of supply of used rubber. The price of crude rubber to some extent influences the demand for reclaim. However, reclaimed rubber, even during the period of low prices for crude in recent years, has been in constant demand.

The modern process begins with the sorting of the scrap. Although subject to a recognized classification by the dealer, the reclaimer sorts it again before processing. Each assortment is finely ground and the ground waste passed over screens and magnetic separators.

From this point the removal of fiber is accomplished chiefly by the acid process of Mitchell or the alkali process of Marks. By far the largest amount of reclaimed rubber on the market is produced by the alkali process. In the acid process the ground scrap is treated with dilute sulphuric acid in lead-lined vats, at the bottom of which live steam is injected. The fiber is slowly weakened and destroyed. This requires as much as four hours or more. The rubber is washed from the disintegrated fiber, generally passing over rifflers which remove sand and metal. After thorough washing the mass as a rule is mixed with oils, tars, or other softeners and conveyed to large digesters. Live steam is forced into the mixture for 24 to 48 hours at a pressure of 100 to 150 pounds. This treatment plasticizes the rubber and removes part of the free sulphur. The plasticized mixture is then dried.

When the alkali process is used the ground rubber containing fiber is treated in digesters with dilute caustic soda of a strength varying from 2 to 8 per cent. The fiber is disintegrated and dissolved at 150 pounds steam pressure in from 8 to 24 hours. A considerable amount of free sulphur is removed from the rubber. It is washed in tanks until practically free from alkali, sometimes run over rifflers and finally dried. The dry rubber is softened by working on a rubber mill and then forced through a heavy cylindrical strainer attached to a tubing machine. The rubber emerges as strings, free from coarse particles of metal, grit, and similar impurities. The amount of metal removed at this late stage of the process is astonishing. The rubber is then passed between heavy rolls and appears as an extremely thin sheet. These sheets are converted into slabs or cylinders about 4 inches in diameter. The various grades of reclaimed rubber are very well reproduced in spite of variations in the scrap.

No reclaimed rubber has yet been produced which is equal to good new rubber. It is more or less inferior in strength and stretch. However, reclaimed rubber is a valuable ingredient in many classes of rubber goods when properly compounded to meet the conditions of service required.

4. RUBBER SUBSTITUTES

No true rubber substitute—that is, no material possessing all the properties of rubber—has yet been produced. The term "rubber substitute" is commonly applied to materials which are produced by vulcanizing certain vegetable oils, such as rapeseed, corn, and cottonseed oils, either by treatment with cold sulphur chloride or by heating with sulphur. The sulphur-chloride process produces "white substitute," while that made with sulphur is known as "brown substitute." These substitutes are used in the manufacture of cheap, soft-rubber articles of low specific gravity. Their use tends to reduce the strength, elasticity, and wearing qualities of the finished product and they are unsuited for making articles which are to be subjected to high temperatures.

The so-called mineral rubbers should not be considered forms or varieties of rubber. They are bituminous materials, either natural products, such as gilsonite or other types of asphalt, or the crude tar residue from the distillation of petroleum.

5. VULCANIZING INGREDIENTS

Charles Goodyear in America and Thomas Hancock in England independently observed the effect of sulphur on rubber at an elevated temperature. Goodyear, to whom priority is given for the discovery, called the process vulcanization. Vulcanization is also called "curing" in rubber factories. Ground roll sulphur is ordinarily used in rubber compounds to effect vulcanization. The vulcanization of rubber by means of sulphur chloride at ordinary temperatures is called cold curing. There are forms of active sulphur which cure rubber at room temperature. The reaction between the gases hydrogen sulphide and sulphur dioxide produces active sulphur. This reaction is used in the Peachey process of vulcanization. Vulcanization has been accomplished by substances other than sulphur, such as selenium, trinitrobenzene, and other organic compounds. Hard rubber requires a larger amount of sulphur and a longer cure in its manufacture than soft rubber.

6. COMPOUNDING INGREDIENTS

The substances that are mixed with rubber in the manufacture of rubber goods comprise a list of materials of widely varying natures. The rubber compounder finds it advantageous to classify these substances into definite groups, although often there is some overlapping between them.

The ingredients may be classified as follows: (*a*) Fillers (1, inorganic; 2, organic); (*b*) accelerators (1, inorganic; 2, organic); (*c*) pigments; (*d*) preservatives.

(a) FILLERS

Certain fillers when mixed with rubber add qualities to the compound which makes their use desirable. A number are added merely as cheapeners and impart no desirable properties. If an excess of fillers is used, their effect on the compound may be detrimental. The cost per unit volume is often the determining factor in the use of one filler in preference to another.

(1) Inorganic Fillers.—Among the inorganic fillers are those which, when added to rubber, impart certain definite properties, such as toughness, increase in tensile strength, hardness, compressive strength, increased insulating properties, and resistance to steam and to abrasion. In general, a filler should be in a state of fine division. The following list contains the most frequently used fillers and some of their general properties:

Carbon black, zinc oxide, clay, lithopone, lampblack, and magnesium carbonate not only slightly increase the rate of vulcanization, but have a decided toughening effect on the rubber. Gas black or carbon black is a valuable pigment in rubber goods. Hancock observed that black stocks resist the action of sunlight, which has a deleterious effect on vulcanized rubber.

A good color may be obtained by mixing 2 per cent of gas black with rubber. The effect of greater quantities depends on the characteristics of the black. Some black produces soft stocks and others have a toughening or stiffening effect on rubber compounds.

Aluminum flake, whiting, barytes, and barium carbonate impart no special properties and are considered as being inert. Some types of clay stiffen or reinforce rubber compounds; others are inert fillers.

Tripoli (infusorial earth), talc, and soapstone tend to make the compound dry and stiff. Talc and soapstone are used mainly for dusting sheets of unvulcanized rubber and molds to prevent sticking during vulcanization. Asbestine is used in heat-resisting compounds; powdered glass and pumice are used in erasers.

Hard-rubber dust is a common filler in hard-rubber goods. It is occasionally used in soft-rubber compounds. Ground fused quartz has been suggested as a filler in certain types of insulation.

(2) Organic Fillers.—The organic fillers include a large number of oils, vulcanized oils, waxes, paraffins, bitumens, and pitches. They are used mainly to facilitate the mixing of the inorganic fillers with the rubber, to render the compound adhesive, to soften the texture of the vulcanized compound, to soften the compounds which have to go through a tubing machine, etc., to decrease the porosity, increase the resistance to water, gas, acids, and alkalies, and in the manufacture of wire insulation and waterproof material.

Blown asphalts, mineral rubbers, asphalts, pine tar, and coal tar are used in the manufacture of wire insulation and tend to prevent blooming, or the gradual crystallization of free sulphur on the surface. They are also employed, as well as resin and shellac, in compounds that are to be used as frictions. They may be called the adhesive softeners.

Paraffin, ceresin, ozocerite, palm oil, and petrolatum are used to make compounds work more smoothly during mixing.

White and brown substitutes impart to the finished product the soft, velvety feeling of a purer rubber compound.

Ground cotton, leather, cork, and wood pulp are used mainly in the manufacture of articles in which lightness, nonslipping properties, and increased porosity are desirable.

Linseed oil, paraffin oil, and aluminum palmitate are used in the manufacture of waterproofing material.

(b) ACCELERATORS

Accelerators are substances which, when added to a rubber compound, decrease the time required for vulcanization. They also permit the use of a lower temperature. Without their use the output of a factory would be much smaller and the cost of rubber goods proportionately higher. They may be classified in two groups—inorganic and organic.

(1) Inorganic Accelerators.—Litharge, magnesium oxide, and lime are the most frequently used and are especially good for the vulcanization of soft rubbers that are rich in resins and to which have been added oils, waxes, and pitches. White lead (basic lead carbonate) and sublimed white lead (basic sulphate) are used to a less extent.

(2) Organic Accelerators.—Vulcanization in the presence of organic accelerators can be effected in as short a time as five minutes at 212° F. Organic accelerators also have a pronounced effect on the physical properties of the rubber, which may be as important as the increased rate of cure. Increased tensile strength and elongation, and tough, soft, or knotty stocks are obtainable by a proper choice of accelerators, which function as a set of tools in the hands of the rubber compounder. During the last few years the use of organic accelerators has increased enormously. They decrease the time and temperature of vulcanization to a greater extent than the inorganic accelerators. However, because of their activity, care must be taken in their use to avoid overvulcanization.

Accelerators which produce a very rapid cure include piperidine pentamethylenedithiocarbamate, the mono and disulphides of tetramethylthiuram, and xanthogenates. Less rapid accelerators in common use are diphenylguanidine, ethylideneaniline, hexamethylenetetramine, and aldehyde ammonia. For longer cures, much use is made of thiocarbanilide, triphenylguanidine, and formaldehyde aniline. Paranitrosodimethylaniline is frequently used in hard-rubber goods. Aldehyde amine accelerators are produced in the form of resin and function as softeners during compounding. There are hundreds of available organic accelerators, many of which are used for special purposes.

(c) PIGMENTS

These are essentially used for their coloring value, although some, such as zinc oxide, lithopone, lampblack, and certain forms of carbon black, are used for the special properties that they impart to the rubber compound. Antimony pentasulphide is used as a sulphur carrier. The following are used merely as pigments: Red oxide, Indian red, Venetian red, ultramarine blue, cobalt blue, Prussian blue, indigo, chrome green, cadmium sulphide, vermilion, yellow ocher, and chrome yellow.

(d) PRESERVATIVES

A number of so-called antioxidants are said to prolong the active life of rubber. Aminophenols and aminoaldehydes and their condensation products are typical representatives. It is desirable that no accelerating action should result from their presence and that no color changes occur in the rubber. Some preservatives are said to resist the effects of oxygen, and others are said to be more specifically resistant to heat. They may function merely as negative catalysts of oxidation or of the physical disaggregation of rubber. Their action is not fully understood.

III. PROCESSES USED IN THE INDUSTRY

1. WASHING OF CRUDE RUBBER

The wild rubbers and some grades of plantation rubbers must be washed to remove mechanically mixed impurities, such as sand, wood, stones, fragments of plant tissue, salts, etc. Most of the plantation rubber, however, reaches the factory clean and ready for use. The method of washing depends upon the type of rubber.

The rubber to be washed is placed in tanks of warm water until soft, after which the larger pieces are cut up by knives or powerful shears and torn up on the cracker. The cracker is a massive machine, having two corrugated hardened steel rolls (see fig. 1) which rotate toward each other at different circumferential speeds and thus produce a tearing action. The rubber comes out torn into ragged pieces. During the process a continuous stream of water is allowed to flow down on the rubber. If the rubber is very dirty, it is placed in a churn or beater washer. The churn washer consists of an oval tank in which a large paddle wheel keeps the rubber and water in continuous agitation, while the impurities settle to the bottom. After this the rubber is washed on a washer until it comes out in a continuous uniform sheet having the appearance of crêpe. A washer (see fig. 1) is similar to a cracker, except that the corrugations are finer.

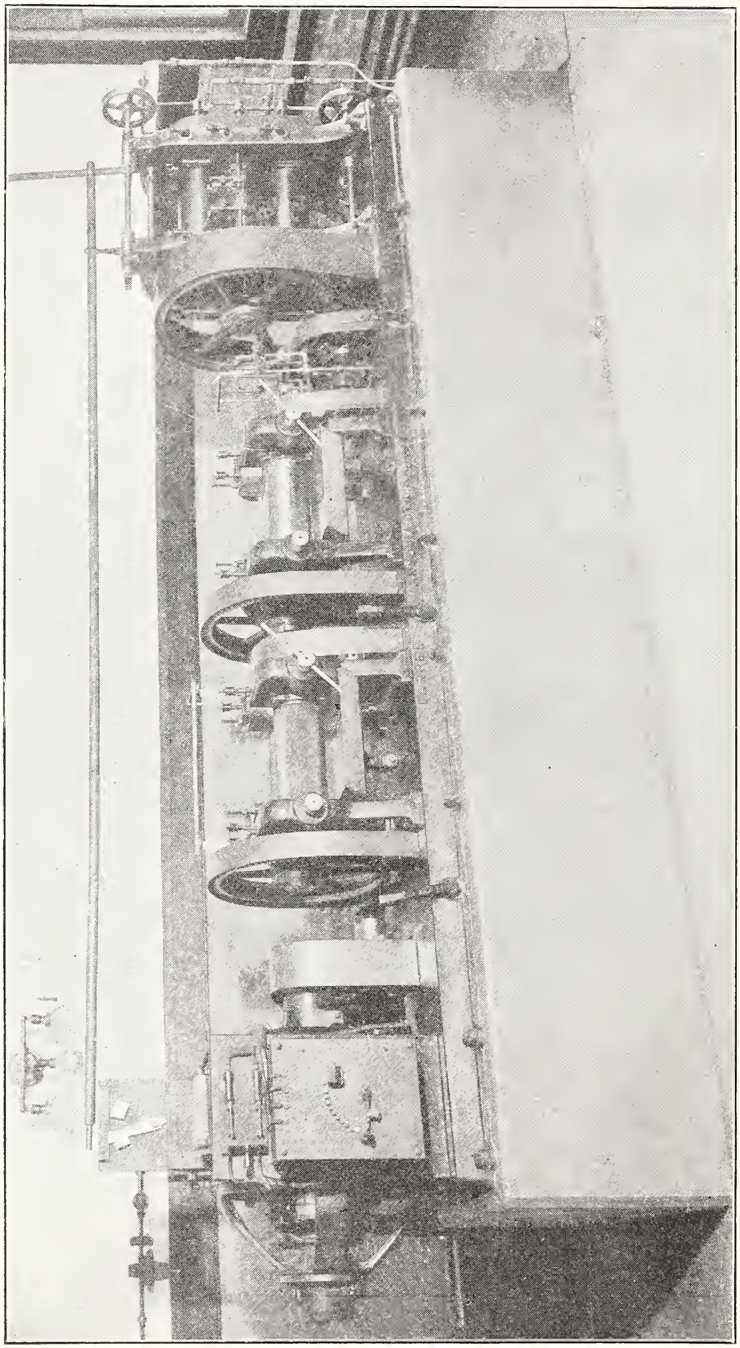

FIG. 1.—*Experimental "cracker," mixing mill, and calender used in preparing rubber compounds of definite composition*

The cracker is used to wash dirt, bark, etc., from the crude rubber; the mixing mill is used for incorporating the compounding ingredients with the rubber, and the calender for sheeting out the rubber compound

2. DRYING

The method of drying depends upon the properties of the various rubbers. Those having sufficient strength are hung in sheets in a large dark room, through which a current of warm air is passed. The lower grades of rubber which are sticky and not strong enough to bear their own weight are dried on horizontal racks. By this method several weeks are required. It has been largely replaced by the more rapid method of vacuum drying in pans. Rubber is also dried under automatically controlled temperature and humidity.

3. COMPOUNDING OR MIXING

The mixing of a rubber compound is carried out on a massive "mill," similar to Figure 1, which consists of two smooth, polished cast-steel rolls which rotate toward each other, the back roll rotating slightly faster than the front one. They are hollow and have steam and cold-water connections for the regulation of temperature as required by different types of rubber compounds. The distance between the rolls is adjustable by means of set screws in the front part of the frame.

The rubber is placed on the rolls and adheres to the slower moving front roll, which is the warmer. At first the rubber has the appearance of a ragged sheet. Pieces which break off are caught in a pan set beneath the rolls and are again added to the mass. The rubber gradually softens and forms a smooth sheet, when it is ready for the addition of the fillers. The operation just described is called "breaking down" the rubber. The fillers are now gradually added. The part which falls into the pan is again added to the mass. During the breaking down of the rubber and the mixing the operator occasionally slits the sheet with a short knife and folds it back on itself as it goes in between the rolls. The sulphur and accelerator are frequently and preferably added after the fillers have been incorporated with the rubber. The mixing is continued until a homogeneous mass is obtained, which is cut off, dusted with talc or soapstone, and sent to the storage room. Recent developments in milling rubber are described by Spear and Moore.[2]

4. CALENDERING

Sheets of any thickness are produced by running the rubber on a calender (see fig. 1) which consists of three adjustable, hollow, smooth, polished steel rolls, similar to those of a mixing machine, set vertically in a massive iron frame and rotated at the same rate of speed. The temperature required for various rubber compounds is maintained by steam and cold-water connections to the rolls.

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[2] Ind. and Eng. Chem., **17**, p. 936; 1925.

The rubber compound before going to the calender must be softened by working on a "warmer," which is generally located in front of the calender and is similar to a mixing machine. The skeleton diagram in Figure 2 shows the method of operation.

The rubber from the warming mill is fed in between the two top rolls, which rotate toward each other. The rubber compound adheres to the middle roll until it is taken up by the running cloth from reel 1 which passes between the second and third rolls, which rotate toward each other. The rubber comes in contact with the winding cloth, which is to keep it from sticking together, and is automatically wound on reel 2. Any desired thickness is obtained by building up the necessary number of layers. This building-up process tends to avoid flaws in the finished sheet.

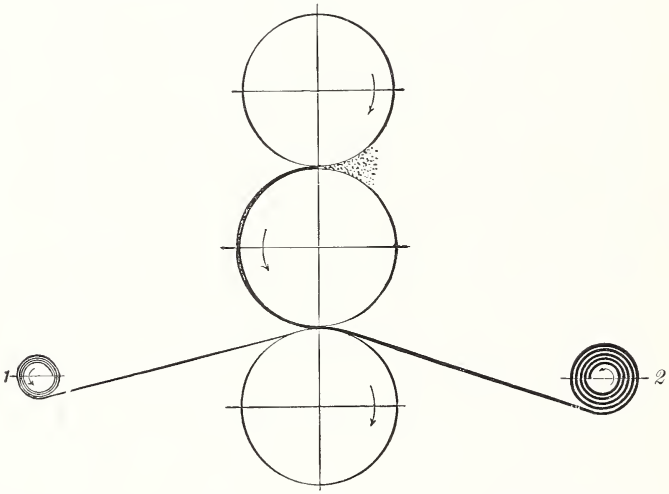

Fig. 2.—*Diagram showing operation of calender rolls illustrated in Figure 1*

5. FRICTIONING

The "friction" is a layer of rubber which acts as an elastic bond to hold together layers of rubber and fabric or layers of fabric. The "frictioning" is carried out on the same type of machine as a calender; in fact, a calender is generally geared so that it can be used for calendering or frictioning. In calendering the rubber is merely sheeted out to a definite thickness, whereas in frictioning it is forced into the meshes of the fabric. The forcing of the rubber into the fabric is accomplished by driving the bottom roll at a slower speed than the middle roll.

6. VULCANIZATION

In 1839 Charles Goodyear found that the physical properties of crude rubber are considerably altered by heating it with sulphur. At about the same time John Hancock found that by immersing rubber in a bath of molten sulphur vulcanization takes place. In 1846 Parkes discovered the cold-cure process, which consists in immersing the rubber in a weak solution of sulphur chloride, or subjecting it to the vapors of sulphur chloride. The solvent is generally naphtha, benzene, carbon bisulphide, or carbon tetrachloride. This method gives only superficial vulcanization, and can be used only on very thin sheets, for the rapidity of the reaction is so great that thick material will be overvulcanized on the surface by the time the interior is vulcanized.

The beneficial changes that are brought about by the process of vulcanization are as follows: The strength, elasticity, and resilience of the rubber are increased, it is less affected by changes of temperature, and it becomes insoluble in the ordinary rubber solvents.

Attempts to explain the process of vulcanization have been made, but the various investigators do not agree. Thus Weber believed that vulcanized rubber is an "addition product" which is formed by chemical combination between sulphur and rubber hydrocarbon, whereas Ostwald believed that it is only a physical combination between rubber and sulphur. It is not the purpose of this circular to attempt to discuss the different theories of vulcanization. The different methods of vulcanization are:

(a) OPEN STEAM CURE

The vulcanizer or heater (see fig. 3), which consists of an insulated cylinder provided with steam and drip connections, is heated by direct steam. The rubber goods are placed in an iron carriage which is run into the heater on tracks, the door is closed, and the steam turned on. The temperature of the heater is controlled automatically.

(b) PRESS CURE

The vulcanizing press (see fig. 4) is used for molded goods. It consists of two or more hollow platens heated by steam with automatic temperature control. They are forced and held together by hydraulic pressure, the rubber being contained in molds placed between the platens.

(c) COLD CURE

"Cold cure" consists in dipping the rubber article in a solution of 1 to 3 per cent of sulphur chloride in carbon bisulphide, carbon tetrachloride, naphtha, or benzene. This method is used only for the manufacture of thin articles.

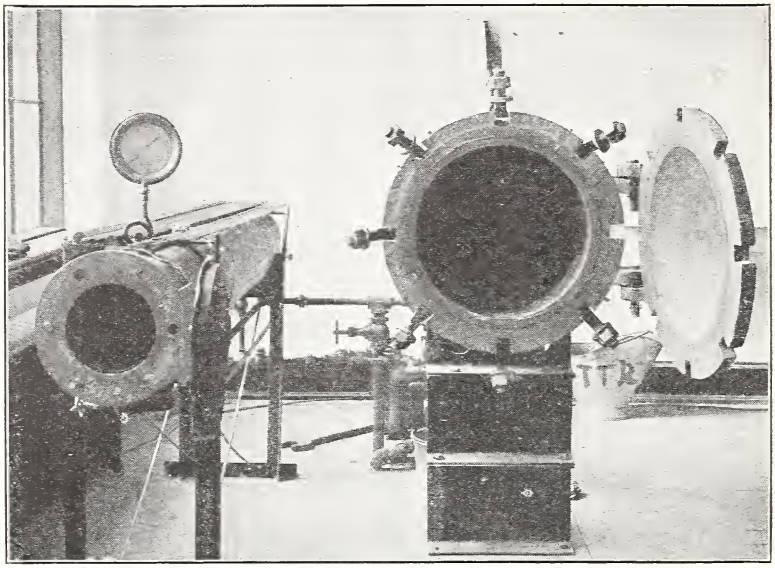

Fig. 3.—*Vulcanizing boilers for the vulcanization of hose, tubing, etc., which are cured in open steam*

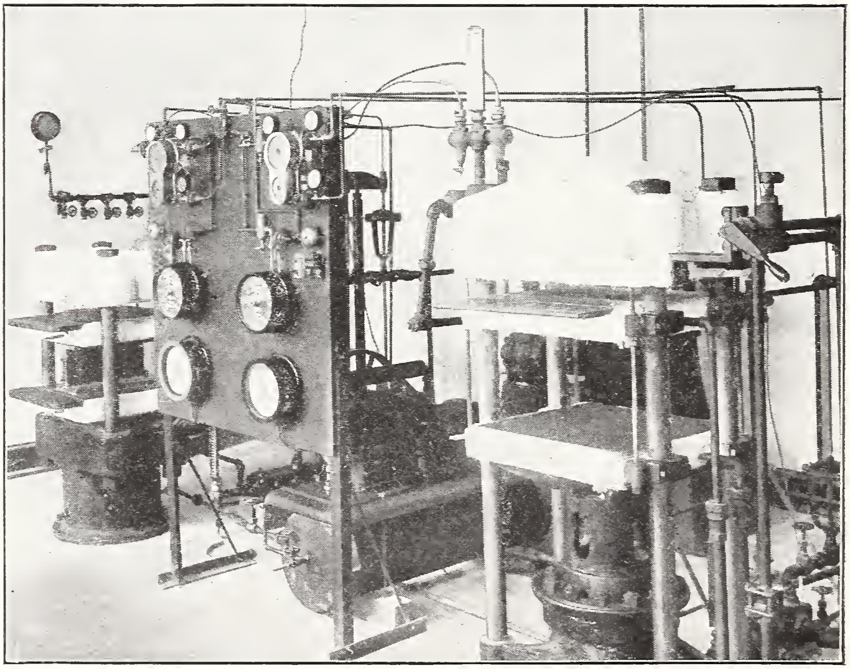

Fig. 4.—*Vulcanizing press equipped with constant temperature control*

This is used for vulcanization of molded articles which are cured under pressure. The temperature and time of vulcanization are controlled automatically

(d) BATH CURE

Vulcanization has been effected by dipping the rubber articles into a bath of molten sulphur.

(e) HOT-AIR CURE

Vulcanization is carried out in large chambers heated by steam in which the air is thoroughly circulated.

(f) VAPOR CURE

This is accomplished by exposure to the vapors of sulphur chloride.

(g) HOT-WATER CURE

This is an old process which is used for certain hard-rubber articles, for thread sheet, and recently it has been adapted to inner tubes. Vulcanization by immersing rubber articles in water at elevated temperature under hydraulic pressure permits close regulation of temperature by circulation of the water, and insures uniform vulcanization.

(h) GAS CURE (PEACHEY PROCESS)

The rubber is vulcanized at room temperature by successive treatments with hydrogen sulphide and sulphur dioxide. An active form of sulphur, which is liberated by the reaction of one gas on the other, vulcanizes the rubber.

7. ELECTRODEPOSITION OF RUBBER

When rubber latex or aqueous solutions of dispersed rubber are subjected to the action of an electric current, the rubber particles, which have a negative charge, are deposited on the anode. At first glance it appears that the deposited rubber would quickly insulate the anode, preventing further action of the current and that a porous deposit of rubber must result if the electrolyte continues to function. Difficulties such as these have apparently been overcome. A process has been devised which deposits the rubber in a homogeneous, coherent form over a considerable range of thickness. Compounding ingredients, including, if desired, sensitive accelerators of vulcanization, may be prepared in colloidal solution, mixed with the colloidal rubber solution and deposited on the anode with the rubber. The procedure essential to electrodeposition consists in the preparation and compounding of the rubber solution, electrodepositing the rubber, drying the deposited article, and vulcanizing. The mechanical operations of milling, calendering, and vulcanization at elevated temperatures which in ordinary mill practice may hinder the development of optimum tensile properties, are avoided in the new method. It is stated that the deposited rubber, when vulcanized, is very resistant to tear and is characterized by high tensile strength. Inner tubes, rubberized fabrics, hot-water bottles, bathing caps, tobacco pouches, rubber bands, surgeons' gloves, and insulated wire are representative articles which have been made by electrodeposition.

IV. MANUFACTURE OF RUBBER GOODS

1. SOLID RUBBER TIRES

Although there are several types of solid tires, the methods of manufacture are essentially the same in most respects. All solid tires consist of a tread or wearing portion, which constitutes the larger part, and a hard rubber base. In some tires there is a strip of rubber to act as a bond between the tread and the base. The base is extruded through a tubing machine and comes out in the proper shape to fit the steel tire rim. The steel rim contains circumferential dovetailed grooves, into which the hard rubber composition is forced. A tubing machine (see fig. 5) contains a screw revolving inside a cylinder. The stock is delivered from a "warming-up" mill directly to the tubing machine. The rubber is fed into the mouth of the cylinder and is forced out through a die which gives it the proper shape. The die is heated by steam to make the rubber more plastic. After leaving the machine the extruded stock passes out on a table where it is cut into proper lengths. The steel rim is painted with rubber cement and the base put on. The tread stock is shaped in the tubing machine, as described above, or built up to the required thickness from calendered sheet which is wound over the hard-rubber base. About 20 tires are vulcanized at once, the molds being stacked one upon the other and closed by hydraulic pressure exerted by a plunger which passes up through the bottom of the steam-heated vulcanizer. The cure is longer for solid than for pneumatic tires, because of the greater thickness of rubber through which the heat must penetrate.

2. CUSHION TIRES

So-called cushion tires are simply solid rubber tires which contain cavities or depressions designed to give greater cushioning than a solid tire of the same general contour. In these tires various means are employed to produce the cavities, but the general methods of manufacture are the same as for solid tires.

3. PNEUMATIC TIRES

The fabrication of pneumatic tires consists in building up the tire structure from various separately made parts which are distinct. The building of the tire consists, therefore, in assembling these various essential parts, called the "bead," "frictioned fabric," "cushion," "breaker strip," "side wall," and "tread."

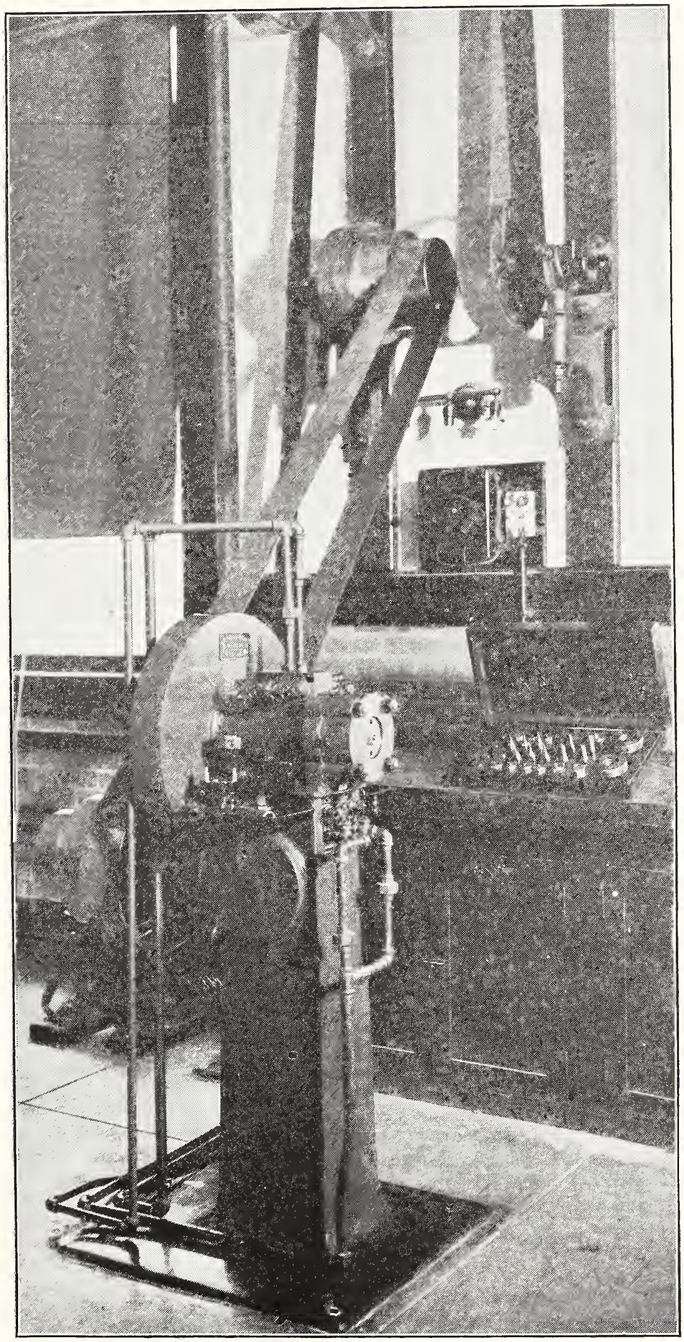

Fig. 5.—*Machine for making seamless rubber tubing by forcing the rubber compound through a die*

The "bead" is the edge of the casing which holds the tire in place on the rim. There are two types of beads, the clincher and the straight side. The clincher bead is usually made of rubber which is run on a tubing machine and partially cured before going into the tire. The straight-side bead is rubber reinforced with strands of steel wire. A coat of cement is applied to the bead and layers of fabric are put on. The beads are now ready for tire building.

The tread is run on a tubing machine or on a calender designed to give the desired shape. The side wall is cut from a calendered sheet. The carcass of the tire is made up of superimposed layers or plies of square-woven or cord fabric which are held together by a rubber compound. Cord fabric (which is used in cord tires) differs from square-woven fabric in that the usual filler strands are omitted except for small soft threads of light yarn which are spaced about one-half inch apart for the purpose of holding the strong cordlike warp threads together during the coating processes, described later. Even these filler threads are sometimes omitted and the warp threads held together by dipping in rubber cement or rubber latex. Several methods or combinations of methods for rubberizing the fabric are used, such as dipping, spreading, frictioning, and coating. One of the common methods is to friction both sides and coat or "skim" at least one side. The fabric to be frictioned is first dried by being passed slowly over steam-heated rolls. While still warm it is passed through the friction calender once or twice, depending upon whether one or both sides are to be frictioned. As it comes from the calender a "liner" is wound up with it to prevent the layers of rubberized fabric from sticking to each other. After being frictioned the fabric is coated or "skimmed"—that is, a thin layer of rubber is applied as the fabric is again passed through the calender. The fabric is now taken to the bias cutter which cuts definite widths at an angle of about 45° with the warp. The machine operates as follows: A row of automatic fingers grasps the edge of the fabric and pulls it forward the required distance. Then a knife drops and cuts off a strip. The fingers release the strip and return to grasp another width of fabric. The fabric is unwound automatically from the roll as it comes from the frictioning calender, and a slack is maintained between the unwinding device and the cutter. The pieces, as they are cut off, drop upon an endless belt which carries them to the operator, who places them between layers of fabric. They are now taken to the splicer, who either splices the bias-cut pieces in continuous lengths which are then wound in rolls, or else makes them up into "bands" of two or more plies of the proper size for the tire to be built.

Fabric tires are practically always built on iron cores having the shape and size of the inside of the tire. The tire-building machine is arranged so that the core can be revolved and the frictioned fabric fed from rolls on adjacent racks. About half of the total number of plies which are to be used in making the tire are put on the core. In doing this, care is taken that no seam shall come over another. Each ply is rolled down smooth before applying the next one. After this has been done the bead is put in place on each side and the remaining plies are put on. The plies of fabric are worked around the bead, and the side wall is put in place. A strip of practically pure rubber, called the "cushion," goes on top of the fabric. On the cushion is placed a "breaker strip" of coarse, loosely woven fabric, and finally the tread is applied. It is good practice to have the work inspected after each operation in order to detect any defects in workmanship which might otherwise be concealed in the finished tire. Fabric tires are vulcanized on the iron cores in molds which have depressions to produce the characteristic design of the tread. The vulcanizer is the same as that used for solid tires.

Cord tires are made of essentially the same component parts as fabric tires and are often constructed on the same tire-building machine. Two other methods of building, however, are used for cord tires. With the band method the plies are made up into groups of bands, each of which usually consists of two plies. These are stretched over the iron core and rolled down in place the same as is done with fabric tires.

With the drum method the whole tire (or practically all of it) is built on a flat drum of a diameter approximating that of the beads of the tire. After the component parts are put together the tire is shaped by an expander, of which there are several types. The tires are cured over an inflated bag which is similar to an inner tube reinforced with two or three layers of fabric. After the molds are on the bags are inflated and the tires cured in the same manner as fabric tires.

4. INNER TUBES

Inner tubes are manufactured in various ways. Sometimes a tubing machine is used, but, in general, rubber sheet which has been made up from about three calendered plies is cut to the desired width and length. This is wrapped several times around a steel tube called a "pole" and the edge rolled down, so as to form a tight seam. It was formerly the common practice to wrap the tube with a strip of cotton cloth wound spirally, which was left in place during the curing process. This wrap is often omitted at the present time or else removed before the tube is vulcanized and other means used to insure a compact tube and joint. The poles are piled in racks and the tubes cured in steam, the method being similar to that described under "plied hose." See Section IV, 5. After they are removed from the vulcanizer the tubes are drawn off the poles by inflating them with compressed air in such a manner as to turn them inside out. The ends if not already tapered during the process of manufacture, are placed on mandrels and forced against a wet, high-speed, circular knife which skives them so that they can be smoothly spliced without much increase in thickness. The tapered ends are roughened on a wire buffing wheel. At the point where the valve is to be inserted the tube is reinforced with a valve patch, which consists of an oval piece of rubber, generally with two plies of fabric inserted. The valve hole is punched and the valve screwed on. The ends are now placed on special sleeves and coated with rubber cement. After the cement has dried they are brushed with a dilute solution of sulphur chloride, and by means of compressed air one end is slipped over the other and strapped down firmly with a rubber strip until properly vulcanized, after which the strip is removed. The tube is then inflated and immersed in water to detect any leaks. The use of a quick-curing cement and a steam-cured splice is said to produce a more reliable tube than the method described above, and the steam cure, though more costly than the cold cure, is meeting with increased favor.

5. PLIED HOSE

(a) MACHINE-MADE HOSE

For hose of small diameter it is usual to form the tube by passing the rubber compound through a tubing machine. The compound is first softened on a warming mill, generally situated near the tubing machine. The tube as it comes from the nozzle of the machine is carried away on an endless belt which is adjusted to run at the proper speed. The desired lengths of tube are cut and talc is blown in to prevent the walls of the tube from sticking together if this has not already been done as the tube emerges from the die. These tubes are placed on steel mandrels by a rather ingenious process.

The mandrel, which is about 52 feet long, is placed on an endless belt and held stationary. One end of the tube having been placed over the mandrel, sufficient air pressure to expand the tube slightly is applied at the other end. The belt is now set in motion, and the tube as it is fed upon the belt floats over the mandrel on a cushion of air.

Frictioned duck for use in making rubber hose is usually cut on the bias from strips 40 inches wide into pieces long enough so that when placed end to end and lapped the resulting strip is just wide enough to produce the necessary number of plies on the hose.

The tube is wiped with a rag moistened with gasoline and the frictioned fabric applied. The frictioned fabric is wrapped on by a machine which consists of three rolls slightly over 50 feet long. The two bottom rolls lie in the same horizontal plane, and the top roll, which is just above and between the other two, can be raised while the pole or mandrel carrying the tube to be wrapped is being placed on the bottom rolls. After the mandrel is in place the top roll is lowered upon it, thus holding it firmly between the three rolls. A rotary motion imparted to the rolls causes the tube to revolve, and the fabric and the rubber cover, which is attached to the fabric, are wrapped on in a few seconds. Before going to the vulcanizer the hose is wrapped with wet cloth. First a long strip is wrapped lengthwise on the hose and over this a narrow strip is wrapped spirally. This is done in a machine similar to that used for wrapping the frictioned fabric and rubber cover around the tube. The narrow strip is held under tension and guided by hand. The hose is now placed on racks set in a carriage and run into the vulcanizer and cured.

After vulcanization the wrapping cloth is wetted and stripped off, and the hose is removed from the mandrel by means of compressed air.

(b) HANDMADE HOSE

For hose of a diameter greater than 1½ inches the tube is usually made from a strip of calendered sheet rubber. The sheet is skived or cut with a tapering cut and wrapped around a mandrel by hand so that the edges overlap. They are pressed flat by means of a small roller to make a perfect seam. The frictioned fabric is cut as above for tubed hose, laid over the rubber tube on the mandrel, and gradually rolled around it by means of a small hand roller until the required number of plies have been made. The rubber cover is made from a sheet of calendered rubber of the proper width to pass around the hose once and form a narrow lap, and put on in the same manner as the fabric except that the lap is carefully rolled down. In suction or high-pressure hose, wire is generally used to strengthen the walls. It is wound on spirally and strips of rubber are used to fill the spaces between the turns of wire. The hose is generally wrapped with a long strip lengthwise and then with a narrow strip wound spirally by rotating the hose on roller bearings. The narrow strip is held under tension and guided by hand. The hose is then finished as described above under tubed hose.

6. BRAIDED HOSE WITH RUBBER TUBE AND COVER

Another type of hose is made by passing a rubber tube of the extruded type, which is distended by moderate air pressure, through a bath of rubber cement, and then to a braiding machine, where the first ply of fabric is braided over fresh cement. The dipping and braiding are repeated until the desired number of plies have been formed. Then the rubber cover is put on with a tubing machine, and the hose is vulcanized in a mold. While being vulcanized the hose is subjected to air pressure from within, which forces the rubber well into the meshes of the loosely braided fabric.

7. COTTON RUBBER-LINED HOSE

In the manufacture of woven cotton hose with a rubber lining the tube is usually made by hand and partially vulcanized in order that it may develop sufficient strength to be drawn through the cover. In the better grades the semicured tube is covered with a thin layer of softer compound, known as the "backing." In the cheaper grades of hose the tube is sometimes merely coated with rubber cement. A long slender rod is passed through the cover and carries with it a stout cord, which is attached to the end of the rubber tube. The tube is now drawn through the cover. The ends are clamped over cones and the hose is filled with steam under pressure. This expands the tube, forces the backing into the fabric, and vulcanizes the rubber. The vulcanization is carried out on an inclined table in order that the condensed steam may not collect in the hose.

8. RUBBER TUBING

Rubber tubing, for which there is a large demand, is made either with a tubing machine, as described on page 24, or from calendered sheet, as described on page 19. Compounded tubing, which is most extensively used for general purposes, is made by the former method. Dies and cores of different sizes are provided. They may be interchanged to produce any diameter and thickness of wall within the machine's capacity.

Pure gum tubing is usually made from calendered sheet, but it is sometimes run on a tubing machine. Its production by the latter method is rather difficult and requires careful supervision.

Tubing is vulcanized in open steam. It is usually coiled in a pan as it comes from the tubing machine, being carefully embedded in talc at the same time. If it is made from a stock which becomes very soft during vulcanization, it has to be placed on mandrels and wrapped, as in the case of tubing made from calendered sheet. It is then vulcanized as described on page 21.

9. RUBBER BELTING

Duck for rubber belting is passed over steam-heated rolls to remove the moisture and is then frictioned, as described under the manufacture of pneumatic tires. The frictioned duck is cut lengthwise into strips of any desired width by an arrangement of adjustable circular knives. The first strip is folded upon itself by being drawn through an opening with flared edges, so that its edges form a butt joint. This folded strip is placed with the joint down upon the next strip, which is similarly folded to form a butt joint on the back of the first strip. In this way the belt is built up with the desired number of plies. Where there is an odd number of plies, the first strip is not folded. The belt then passes between rolls, which press the plies firmly together and at the same time lay and press a narrow strip of rubber over the joint. When the belt is to have a rubber cover, this is calendered on the outside ply of duck before the latter is put on. Rubber-covered belting is necessary only for conveyor and a few other special uses.

The belt is vulcanized while it is stretched and held under heavy pressure between the steam-heated faces of a long hydraulic press. This drives the friction into the meshes of the duck, vulcanizes the rubber, and prevents the belt from stretching excessively when in use.

10. MOLDED RUBBER GOODS

A large variety of rubber goods, such as pump valves, heels, soles, mats, erasers, tiling, etc., are vulcanized in molds under hydraulic pressure. Pieces are cut from the compounded sheet by means of dies of the approximate size of the finished article. The pieces are sometimes trimmed to a definite weight so that they all have the same volume before they are placed in molds and cured.

11. INSULATED WIRE

Rubber has electrical [3] and mechanical properties which render it particularly suitable for insulating electric wires and cables. It is used very extensively for this purpose, although from the standpoint of permanence it leaves much to be desired. Electric conductors of copper, which are almost universally used, must be tinned before the rubber is applied. The purpose of tinning is both to protect the copper from the action of the sulphur used for vulcanization, and also to protect the rubber from the catalytic deteriorating action of the copper.

The rubber coating may be applied to a wire or cable by either of two methods. The more common method is the extrusion or "spewing" of a seamless covering of rubber on the conductor as it is drawn through a machine similar to a tubing machine. The other method is the application of rubber in strips that have been cut from calendered sheets. These strips may be wrapped spirally around the conductor or applied longitudinally. Grooved disks are employed to press down the rubber and make it into a coherent, practically seamless covering.

The strip covering method is particularly well adapted to the making of large cables. Multiple layers of insulation may be applied by either the extrusion or the strip-covering process. Insulation in the unvulcanized state requires careful handling to prevent distortion.

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[3] The electrical properties of soft rubber are discussed in detail in B. S. Tech. Paper No. 299.

Small sizes of wire are generally coiled into large pans with talc while larger sizes are wound in one or more layers on drums. In one modification of the strip-covering process, rubber strips which have a sheet tin backing are employed. The tin forms a protective sheath around the insulated wire or cable and prevents injury during handling and vulcanization. Tape is also used for this purpose, and some types of cable are given a lead sheath by means of a lead press before vulcanization. The tin or lead coverings are stripped off and recovered after vulcanization.

Vulcanization is usually effected by means of steam in large autoclaves. Long cures are given, particularly when the insulated wire is in pans of talc, in order to insure even vulcanization throughout the large mass of material. The recent developments of ultra-accelerators bring continuous vulcanization within the realm of possibility, but as yet no continuous vulcanization process is employed in the industry.

For most uses rubber insulation requires protection against mechanical wear and the action of light and weather. Protection against ozone from corona discharge is also required for high-tension cables. Protection may be afforded by tape or braid that is impregnated with asphaltic or waxy "weatherproofing" materials or varnishes. Metallic armor in tape or wire form is extensively used for interior wiring, while a seamless lead sheath is sometimes employed. Portable conductors for use on garage floors, in mines, and similar places are protected by a tough abrasion-resisting rubber jacket.

12. RUBBER BOOTS AND SHOES

The manufacture of rubber boots and shoes involves a number of operations, which may be referred to briefly as follows:

The rubber, having been compounded in the usual way, is calendered into sheets of the desired thickness, one of the rolls being embossed to produce certain impressions, knurlings, or trade-marks on the sheet. The fabric is thoroughly dried and then rubberized or frictioned on one or both sides.

Fabric and rubber parts are cut by hand or machine according to patterns. Fabric parts are usually stamped by machinery with heavy steel dies, several layers of fabric being stacked together.

The cut parts are assembled and sent to the "making room" where the article is built up in sections on lasts of aluminum or wood which produce the desired shape. The fabric lining parts are first drawn tightly around the last, and then coated with rubber cement in order that the several boot or shoe parts shall adhere firmly when rolled down. The various parts, including insole, filling parts, heel piece, upper, and outsole are applied and rolled on. The boot or shoe is now ready for vulcanization.

Rubber footwear is vulcanized on the lasts which are held on racks, either in air chambers heated by steam coils, or in steam vulcanizers under pressure. About two hours are required for the steam cure and a longer time for the hot-air cure.

After vulcanization, the footwear is removed from the lasts and inspected for defects. It is then sorted according to sizes and types, and packed in cardboard boxes or wooden cases for shipment.

13. RUBBERIZED FABRICS

In the manufacture of rubberized fabrics, the fabric is run slowly over a series of steam-heated coils to dry it thoroughly, after which it is kept in a drying room at a temperature of about 60° C. (140° F.) until it is to be spread or calendered. When the rubber is put on by calendering no solvent is required, and the loss due to evaporation of the solvent is avoided. Calendered fabric is more nearly uniform than spread fabric and it can be made more quickly because no time is required for drying. Calendering, however, requires more skill than spreading.

A spreading machine consists of a horizontal roll supported on an iron frame. A long knife is so supported above the roll that it can be raised or lowered to obtain the desired thickness of rubber on the fabric. The rubber is mixed into a dough with a solvent, generally gasoline. A thin coating of rubber is spread on the fabric by placing the rubber dough in front of the knife as the fabric passes between the knife and the roll. The cloth now passes over steam-heated pipes, where the solvent is evaporated. The spreading is then repeated until the desired thickness of rubber is obtained.

Rubberized fabric can be vulcanized by either the cold or the heat cure. The cold cure is carried out by allowing the rubber to come in contact with a dilute solution of sulphur chloride in carbon bisulphide or carbon tetrachloride. After the cure some magnesium carbonate is dusted on to neutralize any acid that is formed. If the rubber compound contains reactive fillers, such as litharge, lime, magnesia, or zinc oxide, the cold cure must not be used. When a velvety finish is desired, the rubber is dusted with starch before it is vulcanized.

For the heat cure the fabric is dusted with starch or talc and is wound on a steel drum. A sheet of tin foil is sometimes placed between the layers of fabric in order to facilitate the distribution of heat and to insure uniform vulcanization. When the cure is to be carried out in steam, the drum is inclosed in a waterproof bag to keep the fabric dry. When dry heat is used no covering is necessary. In some factories the fabric passes from the spreading machine through the heated chamber at such a rate that the rubber will be properly vulcanized.

14. DIPPED GOODS AND DRUGGISTS' SUNDRIES

Rubber gloves for surgeons and housekeepers, rubber dam, toy balloons, and nursing nipples are among the important rubber articles which may be produced by cold vulcanization. The best results are obtained with clean, dry, high-grade plantation or Pará Hevea rubber. The first operation consists of milling or masticating the rubber between the rolls of a rubber mill for 15 minutes or longer. When pigment colors are used they are mixed with the rubber on the mill after the preliminary mastication. The milled rubber is dissolved in a suitable solvent to produce a cement. An excellent solvent is naphtha of a grade which has a minimum boiling point of 95° C. (approximately 200° F.) and a maximum boiling point of 150° C. (approximately 305° F.). When lighter naphthas are used, the extreme volatility of the solvent tends to produce bubbles and similar defects in the rubber articles.

Approximately 2 gallons of solvent per pound of rubber are used by many manufacturers. The rubber and solvent are put in a drumlike receptacle which is tightly closed to prevent loss of the solvent and to minimize fire hazard, as well as to produce a cement of uniform consistency. The drum is revolved until a uniform solution results, which usually takes place after 20 hours. In the manufacture of transparent goods the cement is transferred to settling tanks and allowed to stand for 24 hours, or until it is sufficiently clear. For other purposes the cement, as a rule, is simply strained.

The forms used for making toy balloons are usually of wood which is coated with shellac and is dipped in glycerol before being dipped in the cement. Other articles are prepared on porcelain or glass forms. The forms are mounted on suitable frames so that many of them can be dipped simultaneously. The number of times the forms are dipped in the cement depends on the concentration of the cement and the desired thickness of the finished article. The forms must be dried between dips and after the final dipping must be thoroughly dried. Any naphtha remaining in the rubber before vulcanization causes a weak product. Excessive humidity also has a serious effect and air conditioning is usually employed in order to insure the uniform production of satisfactory articles. After dipping and drying, beaded edges are produced by rolling the edge of the rubber back on itself.

Vulcanization is effected by dipping the forms into a solution of sulphur chloride or by exposing them to the vapors of sulphur chloride. When the solvent method is used, the bath consists of sulphur chloride dissolved in benzene, carbon tetrachloride or carbon bisulphide. It is necessary that all moisture be removed from the solvent. The concentration of sulphur chloride in the solvent is regulated to produce vulcanization in 15 to 30 seconds, according to the thickness of the rubber. The correct conditions for vulcanization are largely a matter of experience.

When the vapor cure is used, dipped goods are exposed to the vapor of sulphur chloride in an air-tight inclosure at approximately 80° C. (176° F.) for about an hour. The percentage of relative humidity determines the time necessary. All wooden and exposed metal parts of the vulcanizing chamber must be protected with acid-proof paint.

When vulcanized the goods are dusted with talc and stripped from the forms. Talc dust can be removed by tumbling the article in a barrel. They are then tested and packed for shipment. A small cube of ammonium carbonate is frequently placed in the cartons to neutralize any acidity remaining after vulcanization.

Hot-water bottles, ice bags, water and air cushions are either molded or handmade. In the former case the article is vulcanized in a mold under hydraulic pressure. Handmade goods, in which rubberized fabric is sometimes used, are made up from parts cut according to pattern and vulcanized in open steam.

15. RUBBER BANDS AND THREAD

These goods are generally made from new rubber, sulphur, and small amounts of fillers. Rubber bands are made either on a tubing machine or from calendered sheet. The calendered sheet is cut into strips, the edges of which are then cemented together to form tubes. The tubes are cured in open steam or under water. After vulcanization the tubes are cut while wet by revolving cutters. The bands are dried in hot air, inspected, and boxed.

The calendered sheet from which elastic thread is to be cut is wound with a liner upon a hollow drum and vulcanized under water. The vulcanized sheet is unrolled, coated with thin shellac solution, and tightly rewound. The sheet is then cut into threads on a lathe. The threads are boiled with caustic-soda solution to remove the shellac and free sulphur, thoroughly washed, dried, and stored in the dark.

16. SPONGE RUBBER

There are numerous methods of making sponge rubber. From a technical standpoint the best procedure consists in mixing with the rubber a substance, such as ammonium carbonate, which dissociates into ammonia, steam, and carbon dioxide at the temperature of vulcanization. These gases produce the porous structure of the sponge. The red color which commonly characterizes sponge rubber is produced by sulphide of antimony or the calcium lake of paranitraniline red. It is said that the custom in Europe is to use suitable volatile organic compounds to produce porosity. Amyl or butyl acetates are mentioned as suitable compounds. In order to obtain good results, the rubber must be properly plasticized, and softeners consisting of suitable oils and (or) waxes are in common use. Thorough mastication between the rolls of the rubber mill at an elevated temperature is necessary.

A recent method for making rubber sponge employs rubber latex. The latex is coagulated at an elevated temperature in the presence of a suitable vulcanizing agent and compounds which induce porosity during coagulation.

A common type of sponge rubber is represented by the ordinary bath sponge. There are, however, other varieties of sponge rubber and many other possible applications of the product.

A sponge characterized by very fine pores and known as rubber foam may be produced by subjecting highly plastic rubber to nitrogen under several hundred atmospheres pressure. This causes the rubber to swell to several times its original volume when the pressure is released and the heat of vulcanization applied. The final volume of the rubber may be eighteen times the original volume. The reverse of this process, with the same final results, is to apply a partial vacuum to the plastic rubber in order to expand the bubbles of air entrapped during the mastication.

Owing to its cellular structure, rubber foam presents some striking properties. It has an apparent density as low as 0.05, lower than that of any other solid material. If vulcanization is continued to produce a hard rubber or ebonite sponge the apparent density is generally higher, about 0.2 to 0.5 or more. Ebonite sponge can be produced with an apparent density about one-sixth that of cork. It is therefore possible to use sponge rubber in the manufacture of life belts and life rafts, as well as beacons, buoys, and other floating apparatus. Sponge rubber in life belts fits the contour of the body and can be cut or punctured without affecting its properties. Sponge rubber is used to some extent as a substitute for air-filled inner tubes in the tires of motor vehicles.

Other advantages of the cellular structure are the heat and sound insulating qualities it imparts to the rubber. The sponge is therefore of value in making clothing for aeronauts and polar explorers, and in constructing telephone booths.

17. RUBBER CEMENTS

A cement is essentially a solution of unvulcanized rubber in naphtha, benzol, carbon bisulphide, or carbon tetrachloride. Naphtha and benzol are commonly used because they are cheap.

The solubility of vulcanized rubber in ordinary rubber solvents decreases with the degree of vulcanizaton. For all practical purposes vulcanized rubber is useless in the preparation of cement.

Cement usually contains from 6 to 8 per cent of rubber and is made, as already described under the heading "Dipped goods and druggists' sundries."

Self-vulcanizing cements are available which vulcanize at ordinary temperature after the solvent has evaporated. Two-solution cements are also on the market, one containing rubber and an ultra-rapid accelerator of vulcanization, the other containing rubber, sulphur, and an activator of vulcanization, such as zinc oxide. These are mixed in equal volumes before use and the resulting rubber vulcanizes in a few days at room temperature.

In using rubber cement to join rubber surfaces the areas of the junction should be cleaned with gasoline and roughened with sandpaper. Two or three applications of cement should be made to each surface, and each coating should be allowed to dry until tacky before a succeeding coat is applied. The surfaces are then joined and are preferably clamped together for a short time.

Rubber cement is a useful adhesive for other materials than rubber. It is of value in mending toys, books, bric-a-brac, torn fabrics, and for many other purposes.

18. HARD RUBBER

Soft vulcanized rubber contains only a relatively small proportion of sulphur, but hard rubber contains approximately the maximum amount of sulphur which will enter into combination with the rubber hydrocarbon. Except for the fact that the same ingredients are used in making hard rubber as enter into the composition of the more familiar soft rubber, there is little similarity between the two materials. The methods of making hard rubber and its properties and uses differ so much from those of soft rubber that hard rubber manufacture constitutes a separate branch of the industry. Whereas soft rubber goods are usually vulcanized in their finished form, much hard rubber is made in the form of panels, rods, or tubes from which articles are manufactured by machine work in much the same way as articles are made from metal.

In the manufacture of panels the stock is calendered in thin sheets which are plied up to the requisite thickness and covered with sheet tin to afford protection during vulcanization. The "plating" with tin is done by skillful manipulation with a heavy roll on a heated metal table. The use of oil insures the removal of entrapped air from between the tin and the rubber. Vulcanization is carried out in open steam or under water. The heat liberated during the reaction between the rubber and sulphur necessitates slow vulcanization in order to avoid scorching the rubber by superheating in the interior of the mass. Blanks for simple articles, such as combs, are molded between tin sheets and vulcanized under water. The articles are finished by machine work. Articles of less simple shape, such as pipestems or battery jars, are molded in a vulcanizing press. Vulcanization is allowed to proceed, with the aid of an accelerator, until the article is sufficiently cured to retain its shape. It is then removed from the mold and vulcanization is completed in open steam. Molded articles of hard rubber usually require polishing before they are ready for the market.

V. TESTING OF RUBBER

For a number of years this bureau has been investigating rubber products for various branches of the Government service. The purposes are to determine the character of the materials used, how they meet the conditions of service, how they can be improved in quality, and, finally, to develop specifications for the purchase of goods of the desired properties without unnecessarily restricting competition. This investigation could not be made without suitable laboratory tests for measuring the qualities desired. Old tests were adopted or modified, new ones were devised, and apparatus was designed.

Physical tests are made to determine the tensile strength, the permanent set after stretching, the strength of adhesion between plies in built-up fabrics, and the various other physical properties which each specific material possesses.

Chemical analysis can not of itself furnish information on these points until physical tests and actual service have demonstrated what compounds are best adapted to particular needs. In many cases chemical analysis furnishes the only available information as to the nature and uniformity of composition of rubber goods.

Finished rubber products are not in a stable condition, but undergo change when exposed to heat, light, and air. It is therefore desirable to learn, if possible, what is the probable life of a given product in actual service or in storage. Various accelerated aging tests have been devised, but the results obtained by them should be used with caution. When satisfactory aging tests are developed they should be incorporated in specifications.

After the Federal Specifications Board had been established in 1921 it appointed technical committees to prepare specifications for various commodities. The committee on rubber found that there were many different specifications for the same products. This state of affairs was unfortunate, because it increased the cost of the products without any material advantage. The bureau has cooperated with the committee and the board in the preparation of specifications for rubber goods required by the Government, including tires and tubes, mechanical goods, packings, and druggists' sundries. A list of these specifications is given on page 81.

In the following pages the methods and apparatus for making physical tests are described in detail.

1. PHYSICAL TESTING OF SOFT RUBBER

Rubber testing in the present stage of its development is not susceptible of very great refinement as regards measurement. The nature of the material is such that refinement seems of less importance than uniformity of methods, which is absolutely essential when the work of different laboratories is to be compared. A more general interest in this matter would result in a substantial benefit not only to reputable manufacturers and large consumers, but also to the public.

The different properties that have been found desirable in rubber intended for different purposes have naturally given rise to numerous tests, of which the most widely applicable are the various tension tests. These tests in various forms are used to determine the more important physical properties, such as tensile strength, ultimate elongation, elasticity, and reduction in tension when held at a definite elongation.

In the case of pneumatic tires, rubber hose, rubber belting, and other articles which are built up with layers of fabric cemented or frictioned together with rubber, it is customary to determine the friction or adhesion between the plies of fabric. It is also usual to subject hose (particularly fire hose and air hose) to a hydraulic-pressure test in order to detect any imperfections in materials or workmanship.

An important test in the case of steam hose consists in passing steam through a short length of the hose, in order to determine if the tube, the cover, and the friction are of suitable composition to withstand the effects of service conditions. This test lasts for several days, the steam being turned off at night to allow the rubber to cool. A decided hardening or softening of the rubber, or a large decrease in the value of the friction, as a result of steaming, is an indication of inferior quality.

No absolutely reliable test (other than an actual service test) has been devised for rubber steam packing, but in many cases valuable information may be obtained by clamping a piece of the packing between metal plates and subjecting it to the action of steam at a pressure equal to or slightly above that under which it is to be used. A more satisfactory method is to clamp the packing in the form of a gasket between pipe flanges and to apply the desired steam pressure from within. The test should last several days, the steam being turned off at night to see if the joint has a tendency to leak as a result of the cooling effect. This, however, practically constitutes a service test.

The testing of tires is done almost exclusively by manufacturers and large consumers. Manifestly it would be too expensive for the individual consumer or small dealer to sacrifice whole tires for the purpose of securing test pieces. The more progressive manufacturers, however, realize that money expended in thorough and careful testing is more than justified by the increase in efficiency of their product. For detailed information relating to performance tests of tires the reader is referred to the list of bureau publications at the end of this circular.

The accelerated aging of rubber compounds as an indication of their durability in actual service is an important consideration to the manufacturer and consumer. No test which has been suggested appears to duplicate exactly the behavior of all types of rubber compounds during natural aging. Certain of them are of value to the manufacturer in determining the "shelf life" or behavior during storage of compounds of known composition. The results obtained may be misleading when the conditions of vulcanization and composition of the compound are unknown. Accelerated heat tests are of value as an indication of overvulcanization.

Oxidation is commonly considered the chief cause of deterioration. The accelerated aging tests which have been proposed are based upon this assumption. In the Geer test the samples are exposed to a current of circulating air at a temperature of 70° C. The Bierer-Davis test employs oxygen under 300 pounds pressure at 70° C., and that suggested by Marzetti uses oxygen at normal pressure at the temperature of boiling alcohol, approximately 78° C.

The history of rubber compounds in actual service subsequent to lying on the shelf depends not only on their composition and degree of vulcanization but also on the particular environment to which they are subjected. It is evident that aging phenomena present unusual complexities.

Overvulcanized rubber and in the case of some rubber compounds undervulcanized rubber goes to pieces rapidly. It is customary to cure to a point somewhat short of that which gives maximum tensile strength. This is known as the technical cure. The proper choice and use of accelerators determines to a great extent the drop in tensile strength which may accompany overvulcanization. Accelerators which cause a very rapid cure avoid the detrimental effects of overcure when used under conditions which produce small amounts of free sulphur. The effect of high and low free sulphur on the aging properties of rubber compounds has not yet been satisfactorily determined.

Another factor in the decay of rubber compounds is the physical disaggregation of its structure. It is well known that rubber cements lose their consistency or apparent viscosity when exposed to light. A trace of acidity increases the rate of disaggregation materially. It has been shown that free sulphuric acid is present in aged rubber compounds. This is particularly true of compounds containing only rubber and sulphur and may account for the rapid deterioration of such stocks. In general, an acid condition in rubber has a deleterious effect on its aging characteristics and an alkaline condition is favorable to good aging properties. Practically all rubber compounds contain accelerators and the presence of lead or zinc oxides, which are basic, is necessary to activate most of them and cause them to function properly. Consequently an alkaline condition is automatically provided in the manufacture of rubber goods.

(a) COMPARISON OF NATURAL AND ACCELERATED AGING

For this comparison three compounds were chosen. Compound No. 1 has the following composition in percentage by volume:

Smoked sheet	83. 00
Sulphur	1. 47
Diphenylguanidine	. 25
Zinc oxide	. 55
Whiting	14. 73

Cured for 30 minutes at 287° F. (142° C.).

No. 2 compound is a tread compound and contains reclaimed rubber and has the following composition in percentage by volume:

Smoked sheet	60. 41
Hexamethylene-tetramine	. 41
Sulphur	1. 74
Zinc oxide	1. 92
Gas black	10. 75
Mineral rubber	6. 03
Palm oil	3. 80
Reclaimed rubber	14. 94

Cured for 50 minutes at 294° F. (146° C.).

No. 3 compound is very similar in composition to the No. 1 compound except that in No. 3 an inorganic accelerator is used. The composition in percentage by volume is as follows:

Smoked sheet	82. 45
Sulphur	2. 21
Litharge	. 80
Whiting	14. 54

Cured for 15 minutes at 287° F. (142° C.).

Natural aging tests were made on the above stocks under varying conditions as follows: Outdoor exposure on the roof where samples were exposed to all atmospheric conditions, including sunlight. Indoor storage where samples were stored in the dark and aged at room temperature, approximately 75° F., for the same period of time as those outdoors.

Accelerated tests were also made under three different conditions: (*a*) Samples were exposed to a current of air at a temperature of 70° C., (*b*) in an atmosphere of nitrogen at the same temperature, and (*c*) in an atmosphere of oxygen at the same temperature.

The results are given in Figures 6 and 7. Figure 6 shows the results obtained from the three compounds under natural aging conditions. It is evident that No. 1 compound ages quite differently under the two sets of conditions. It deteriorates much faster outdoors than indoors. The difference in aging outdoors and indoors is less noticeable with the No. 2 compound and is still less apparent with the No. 3 compound.

In Figure No. 7 results are given for the three compounds under accelerated aging conditions. Here it is evident that all compounds deteriorate less rapidly in nitrogen than in air and faster in oxygen than in air. The No. 1 compound shows less deterioration under any given condition here than does either of the other two compounds, and No. 2 and No. 3 act similarly under any one given condition.

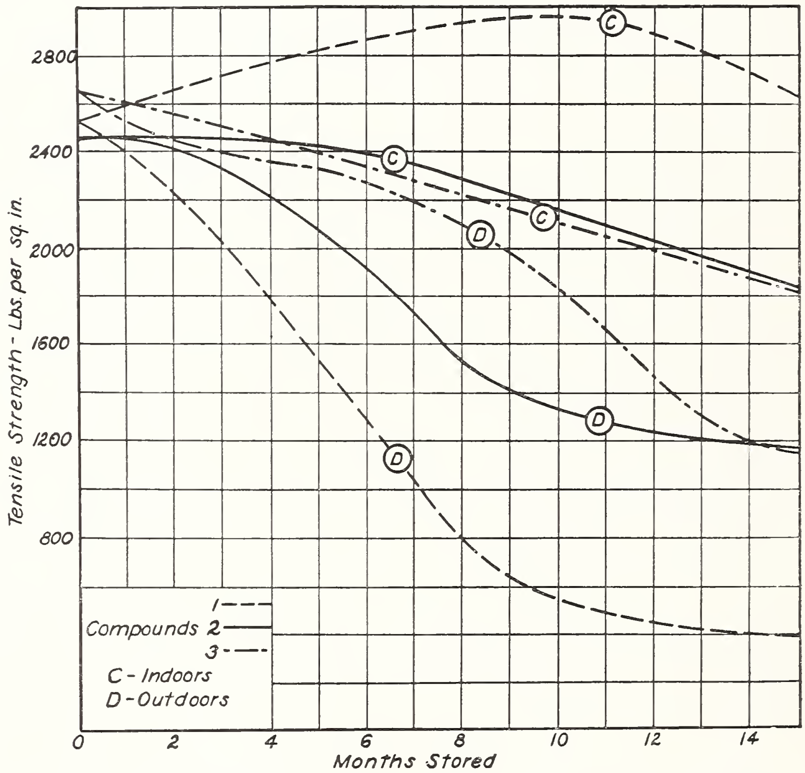

Fig. 6.—*Changes in tensile strength of rubber compounds when exposed to indoor and outdoor conditions*

(b) TENSILE STRENGTH AND ULTIMATE ELONGATION

(1) Separating Rubber from Fabric.—When the material is made up with layers of fabric, as in the case of rubber hose, the first step in preparing specimens for the tension test is to separate the

rubber from the fabric. Unless the frictioning is poor, this will necessitate the use of a solvent. If there is more than one layer of fabric, the easiest way is to remove the first layer along with the rubber. The rubber is then separated from the adjoining layer of fabric with the aid of gasoline blown from a wash bottle. Narrow strips are more easily handled than larger pieces and there is less danger of injuring the rubber. Great pains should be taken during this operation, because any flaw or local imperfection will seriously vitiate the results. The rubber should be allowed to rest for several hours, in order that it may recover from the stretching it has received and that the gasoline may thoroughly evaporate.

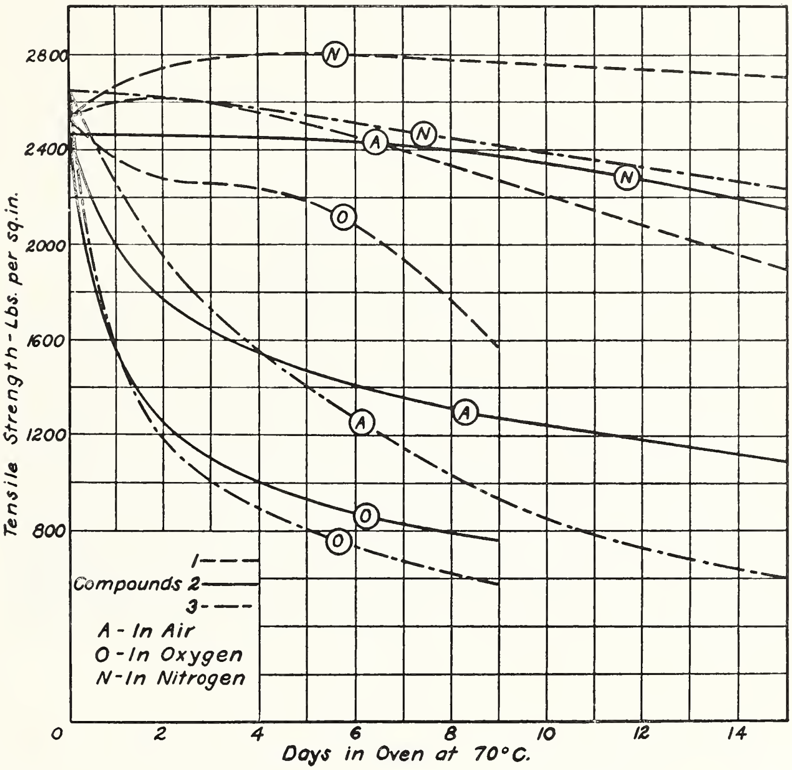

FIG. 7.—*Changes in tensile strength of rubber compounds in air, oxygen, and nitrogen, at 70° C.*

(2) ABRASIVE MACHINE FOR BUFFING THE SURFACE OF RUBBER.—The strips thus removed retain the impressions of the fabric from which they have been separated, and it is necessary to grind the surface smooth in order to measure accurately the thickness of the test pieces. The abrasive wheel shown in Figure 8 was designed at the bureau to accomplish this purpose and has proved very convenient

and satisfactory. The rubber to be buffed is clamped, as shown, to a circular sector which is rotated back and forth in front of a 5 by 1¼ inch emery wheel (No. 16), running at about 3,000 revolutions per minute. An adjusting thumbscrew serves to advance the sector by very small amounts as the buffing proceeds. The face of the wheel should be dressed at intervals as may be required to maintain a good abrasive surface.

(3) Form and Preparation of Test Pieces.—Test pieces are cut with a metal die, which not only saves much time, but also insures uniform width, which it is impossible to obtain if the specimens are cut by hand. An arbor press (fig. 9) is perhaps the most convenient and satisfactory means of forcing the die through the rubber, although some prefer to cut the test pieces by striking the die with a mallet. The operation is facilitated by wetting the rubber or the cutting edges of the die, and the rubber should rest on a smooth and slightly yielding surface which will not injure the cutting edges. A piece of leather is suitable for the purpose. The central portion of the test piece is straight, and the ends are gradually enlarged to prevent tearing in the grips of the testing machine. The width of the contracted section is usually made either one-fourth or one-half inch. Figure 10 gives the dimensions of test pieces which have been found to give satisfactory results. Figure 11 shows the various dies used at this bureau. The one-fourth-inch test piece is to be preferred for

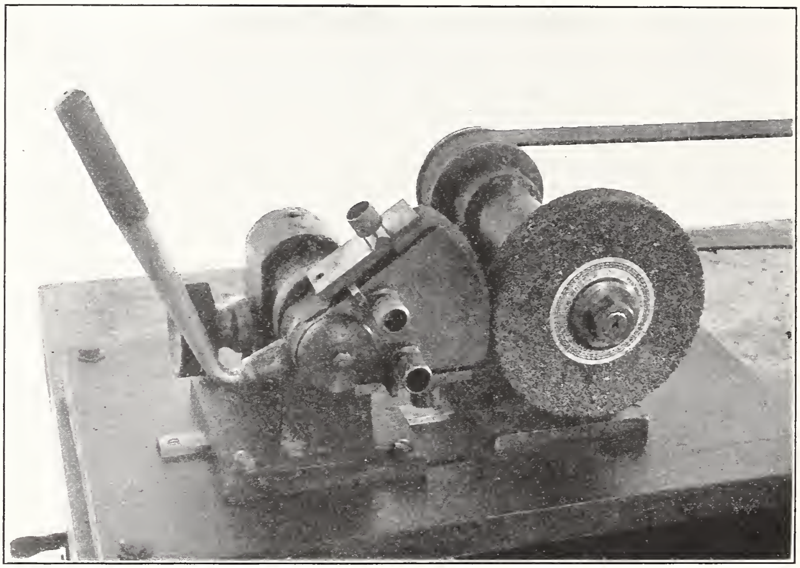

Fig. 8.—*Abrasive wheel for grinding the surface of rubber test pieces to produce a smooth surface*

general use, for with it there is less tendency for the rubber to tear in the grips or across the shoulder where the cross section changes.

Parallel lines are placed on the specimens, and by means of these gauge marks elongation and permanent extension are measured. A stamp, consisting of parallel steel blades, enables one to mark very fine lines with yellow or black ink without cutting the rubber, and in this way much time is saved, and the chance of error very much reduced.

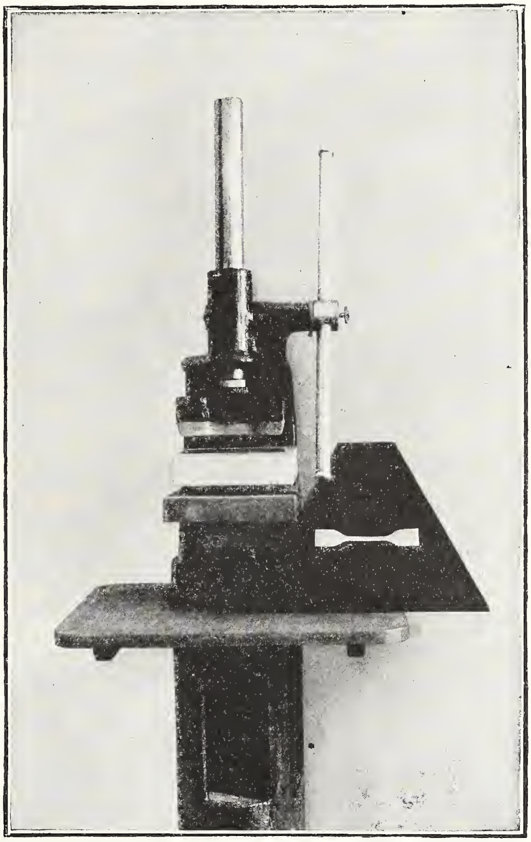

Fig. 9.—*Arbor press and die for cutting rubber test pieces for determining the tensile properties*

In testing pure gum or compounds containing a large percentage of rubber it is not practicable with a machine of convenient length to break test pieces having a gauge length of 2 inches. In such cases shorter test pieces one-fourth inch wide over a gauge length of 1 inch and 1¼ inches wide at the ends are used. Test pieces in the form of a ring will be referred to later on page 60.

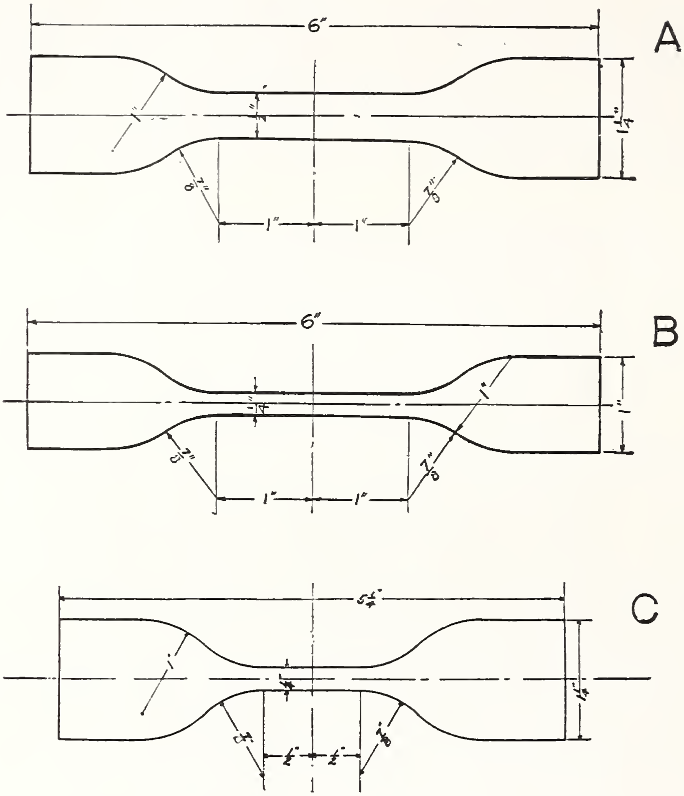

FIG. 10.—*One-fourth and one-half inch test pieces with enlarged ends*

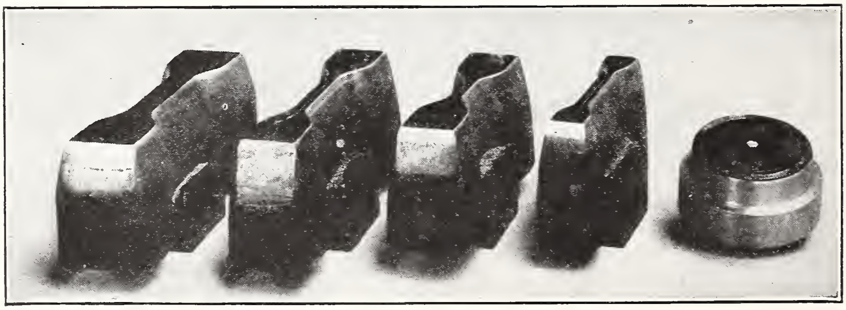

FIG. 11.—*Dies used for cutting test pieces*

(4) Measuring the Thickness of Rubber.—A special form of micrometer caliper (fig. 12, A) provided with enlarged contact surfaces and a ratchet stop is used to some extent for measuring the thickness of rubber and textile materials. The ratchet stop makes it possible to measure each specimen under the same pressure and thus adds to the uniformity of results. A spring micrometer (fig. 12, B) is more generally employed for this purpose. It is exceedingly convenient and very easily read.

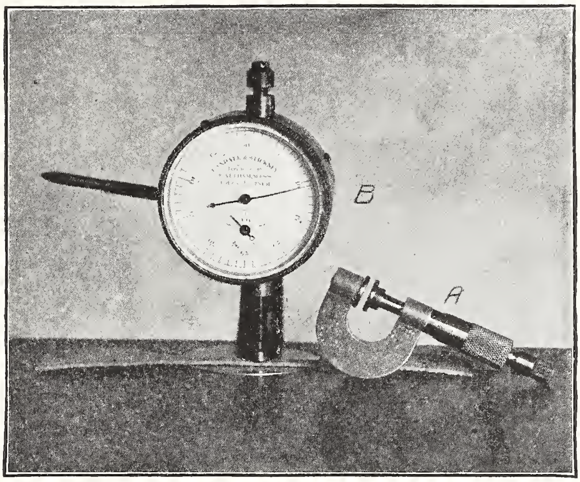

Fig. 12.—*Instruments for measuring the thickness of rubber*

A, micrometer; B, spring micrometer

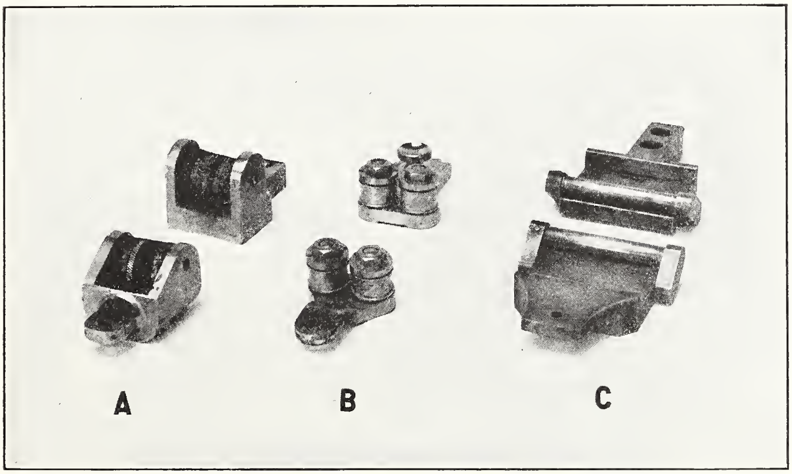

Fig. 13.—*Grips used in making tensile tests of rubber*

A, eccentric disks which tighten automatically as the tension increases; B, spools with ball bearings for testing ring-shaped specimens; C, pivoted link grips for testing very soft stocks

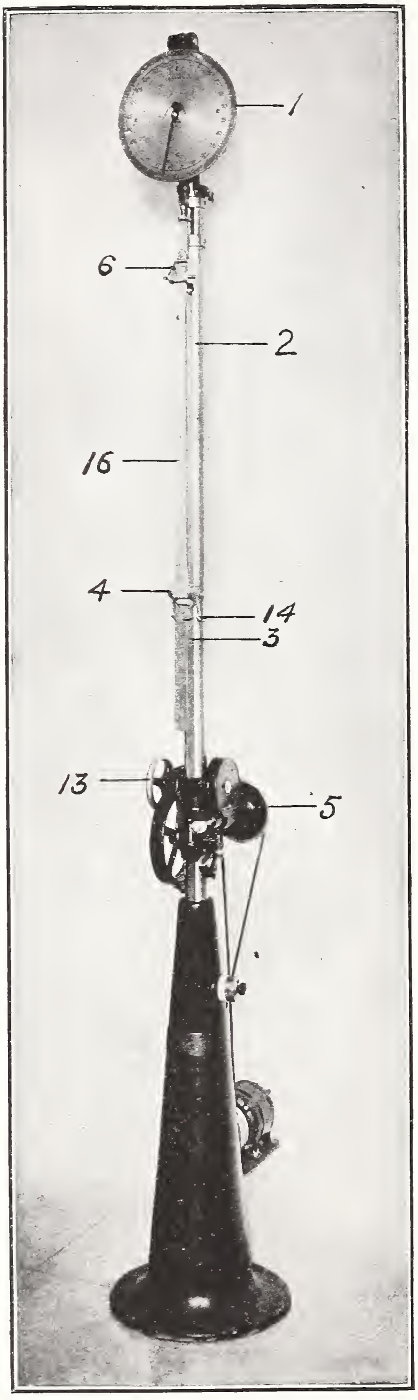

FIG. 14.—*Motor-driven machine for testing the tensile strength of rubber. See Figures 15 and 16 for details*

(5) GRIPS FOR HOLDING TEST PIECES.—In testing rubber one of the greatest difficulties has been to grip the test piece in such a way as to prevent slipping, without at the same time injuring it. Even a very small scratch on the surface of a rubber test piece may cause failure.

In order to prevent slipping of the test piece as its section is gradually reduced under increasing tension, it has been found advisable to provide means for automatically tightening the grip. This is conveniently accomplished by using a number of thin cylindrical disks, with knurled faces, mounted eccentrically on a pin (fig. 13, A), which act independently, thus producing a uniform pressure over the gripping surface and preventing any uneven slipping. The grips shown in Figure 13, C, are especially well adapted for testing soft rubber compounds having a high rubber content.

When it is desired to test specimens of circular form, such as rubber bands, jar rings, or washers, grips of the design shown in Figure 13, B, may be used. Each grip consists of two rollers mounted on ball bearings. The action of these rollers is such that as tension is applied they rotate in opposite directions, thus tending to equalize the stress around the circumference of the test piece.

(6) TESTING MACHINES.—The dynamometer *1* (fig. 14), having a capacity of 125 pounds and graduated to one-fourth

pound, is attached to the upper end of column *2*, which is slotted to receive the rack *3*, carrying the eccentric grip *4* at its upper end. The machine is operated by a one-twelfth horse-power motor, which is belted to the stepped pulley *5*. A worm on the pulley shaft drives a worm wheel which is geared to a spur (not shown) inside of column *2*. The eccentric grip *4* (fig. 15) is attached to the dynamometer through pin connections *7* and *8*. The plate *9* serves to prevent recoil of the springs when a specimen is broken, and acts in the following manner: The rod *10*, which is rigidly attached to column *2*, passes with very little clearance through a hole in the back of plate *9*. The front of plate *9* is slotted to receive the rod *11*, and is supported by the shoulder *12*. As tension is applied to the specimen, plate *9* is free to follow the shoulder *12*, and passes down over the rod *10*, but when the specimen breaks the upward pressure of the shoulder *12* causes the plate to bind on the rod *10*, thus holding the springs under the maximum tension. Hence the pointer of the dynamometer remains at the maximum load when a specimen breaks. The load having been recorded, the upper grip is pulled downward (by means of the hand wheel *13* and hooks *14*, if necessary, fig. 14) to relieve the pressure of the shoulder *12* against plate *9*, and at the

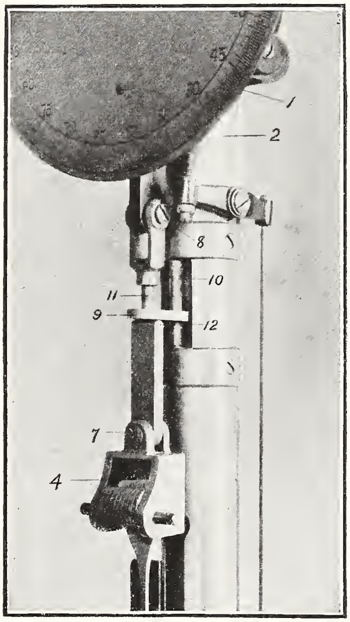

Fig. 15.—*Detail of upper portion of machine shown in Figure 14, illustrating method of attaching grip and device for preventing recoil of spring*

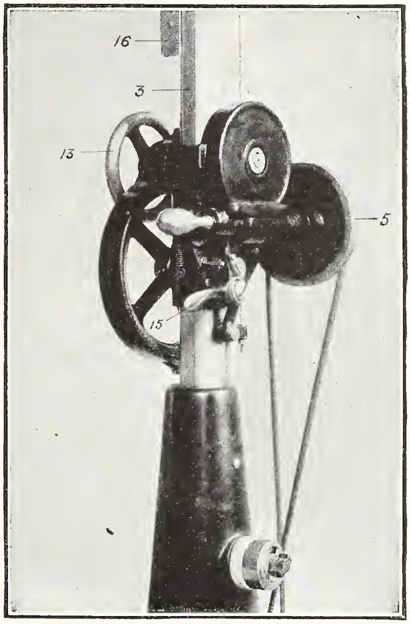

Fig. 16.—*Detail of lower portion of machine shown in Figure 14, illustrating stepped pulley, gear drive, and releasing mechanism*

same time the rear end of the plate is raised to release the rod *10*. The tension of the springs may now be relieved by allowing the grip to rise, and the pointer is returned to zero.

At the end of a test the worm and gear are disengaged by means of the spring clutch *15*, (fig. 16), and the rack is rapidly raised by hand to its initial position.

The stepped pulleys provide for different speeds to meet the requirements of experimental work.

Elongation between gauge marks on the specimen may be measured on the scale *16*, attached to column *2*, but to do this requires the services of two operators. A simpler and equally accurate method is as follows: A wooden scale graduated to one-tenth inch, is held, as shown in Figure 17, in a slightly inclined position at the back of the test piece with its lower end pressed lightly against the rubber just back of the lower gauge mark. As the rubber is stretched the eye follows the scale just back of the upper gauge mark. With a little practice the elongation at break can be measured with a fair degree of accuracy.

The Schopper machine illustrated in Figure 18 is worked by hydraulic power, its operation, briefly stated, being as follows: The rubber test ring is placed over the spools, and the lower spool is geared to the rack in such a way that it is caused to revolve during a test. This motion is transmitted to the top spool by the rubber test ring, the object of rotating the spools being to equalize the tension at all parts of the specimen. As the tension is increased, the weighted lever, to the short arm of which the top spool is attached, is gradually deflected. When the test ring is broken, the lever is held at the point of maximum load by means of a set of pawls. The breaking load is then read from the curved scale and the elongation from the vertical scale just opposite the test ring.

Figure 19 shows a motor-driven machine of 15 pounds' capacity which was designed for testing rubber bands. The load is applied through the steel tape *1*, at the end of which is the grip *2*, carrying spools *4*, similar to those shown in Figure 13, B. The graduated steel tape *3*, attached to grip *2*, with its zero point coinciding with the center of the spools *4*, passes up behind the test specimen and through the column *5* to a reel just behind the spring balance. When the specimen breaks its elongation is determined by the distance between the centers of the spools, as shown by the tape *3*. The plate *6* holds the springs under the maximum tension at break in a way already explained in connection with Figure 15. Stepped pulleys provide for different speeds. When a specimen has been broken the worm and gear are disengaged by means of the lever *7*, and the bottom spools are raised by hand to their initial position. This machine is also provided with eccentric grips which are used for testing straight specimens of low tensile strength.

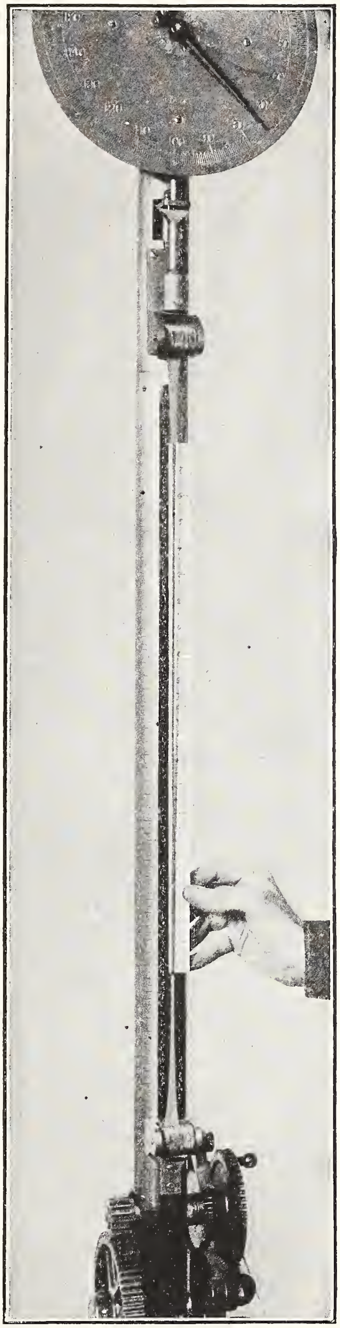

FIG. 17.—*Illustrating method of measuring ultimate elongation*

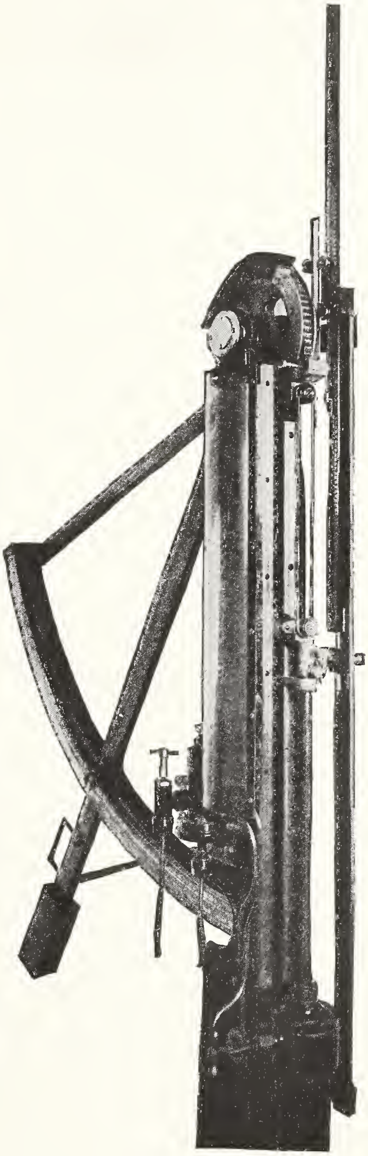

FIG. 18.—*"Schopper" machine for testing the tensile strength of rubber. This machine operates on the principle of a weighted lever*

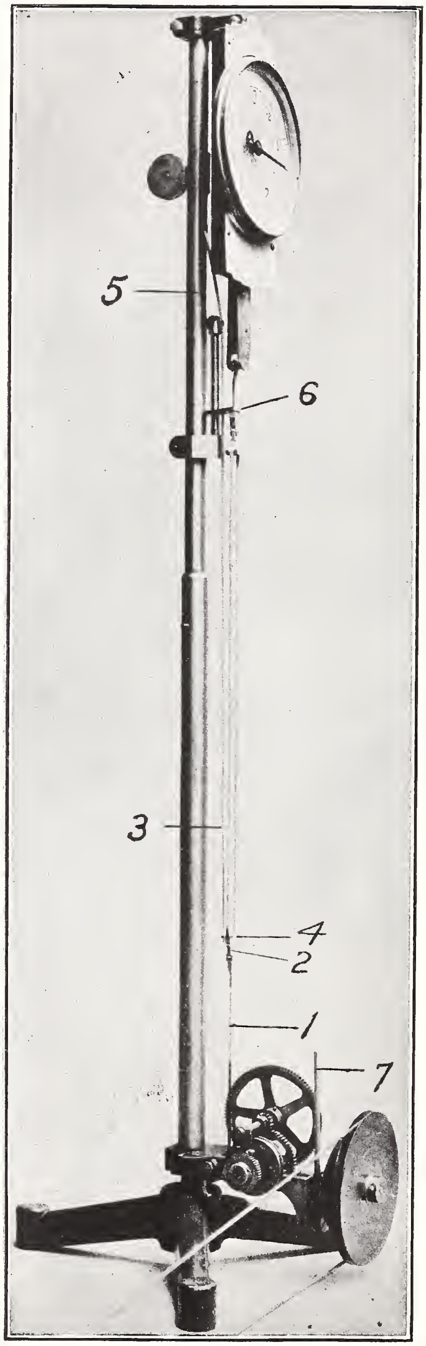

Fig. 19.—*Motor-driven machine for testing rubber bands*

This machine operates on the same principle as the one illustrated in Figure 14

(c) ELASTICITY OR "SET"

"Set" as applied to rubber is measured by the extent to which the material fails to return to its original length after having been stretched. For example, if a test piece is stretched from 2 to 10 inches for 10 minutes and then released, and if its length measured after 10 minutes' rest is 2.4 inches, the "set" under these conditions is 0.4 inch, or 20 per cent.

The set, together with the ultimate elongation and tensile strength, is an indication of whether the compound has been properly vulcanized. For a given compound high set indicates undervulcanization.

(1) Machine for Testing Elasticity or "Set."—For merely stretching rubber to determine its elasticity or recovery after a definite elongation, without reference to the tension applied, the apparatus shown in Figure 20 is used, in which six specimens may be tested at once. The spools, *1*, are free to slide on the shaft, *2*, and are slotted to engage pins, *3* (not shown), which act as clutches. The movable grips are attached to three-fourth inch strips of leather-belt lacing which pass through clamps, *4*, and then to the spools, *1*. The action of these clamps is similar to that of an ordinary letterpress, and, with a one-half by three-fourth inch bearing plate, a moderate twist of the knurled head is sufficient

to prevent any slip of the belt lacing when under tension. The operation of the apparatus is as follows:

The specimens being in the grips, one of the spools is moved along the shaft until it engages the corresponding pin, and the shaft is revolved until the desired elongation measured between gauge marks on the specimen is secured. The clamp is tightened to hold the specimen in this position, and the spool is shifted back so as to disengage the pin. The operation is repeated with each of the specimens in turn. Each specimen is released after a specified length of time (usually 1 or 10 minutes), and after an equal interval of rest the permanent extension or set is measured with a scale graduated to 0.01 inch.

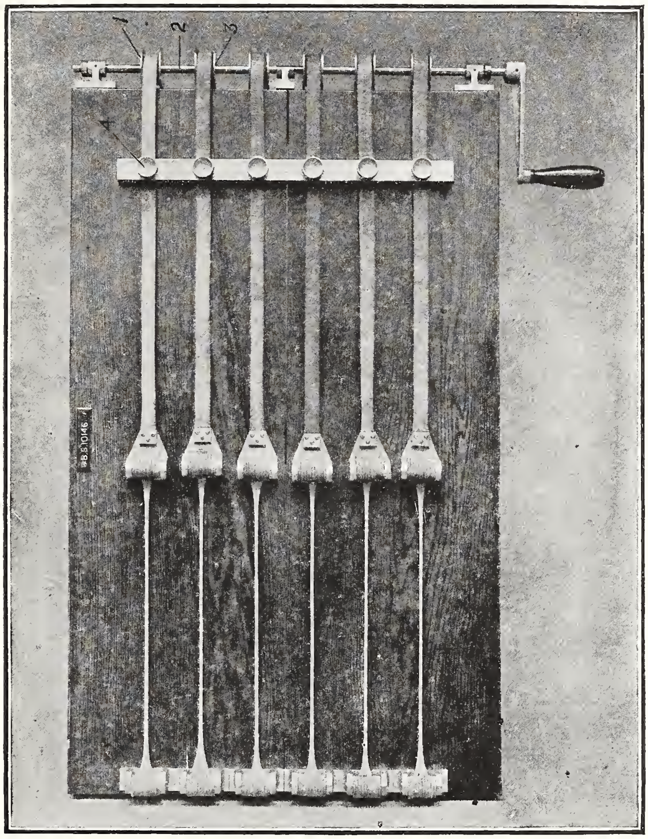

Fig. 20.—*Apparatus for stretching rubber to determine its elasticity*

(d) REDUCTION IN TENSION WHEN RUBBER IS HELD AT A DEFINITE ELONGATION

When rubber is stretched in a testing machine the extension gradually increases with the applied tension, as is the case with other materials, but if the machine is stopped at any point so that the test piece is held fixed under a constant elongation, the tension in the test piece will gradually decrease.

The determination of this decrease in tension under specified conditions constitutes a test which is thought to be indicative of the quality of rubber and the extent of vulcanization.

(1) Machine for Testing Reduction in Tension.—The apparatus shown in Figure 21, which has a capacity of four test pieces, is used in conducting this test. The spring balances, graduated to 0.2 pound, are provided with live and dead pointers, which show the maximum tension as well as the tension at any time during the test. The lower grips are counterbalanced by weights, which are suspended from cords passing over pulleys just back of the spring balances. In this way each grip is held in an accessible position and is prevented from falling should the specimen break. The apparatus is used in the same manner as that described for measuring set.

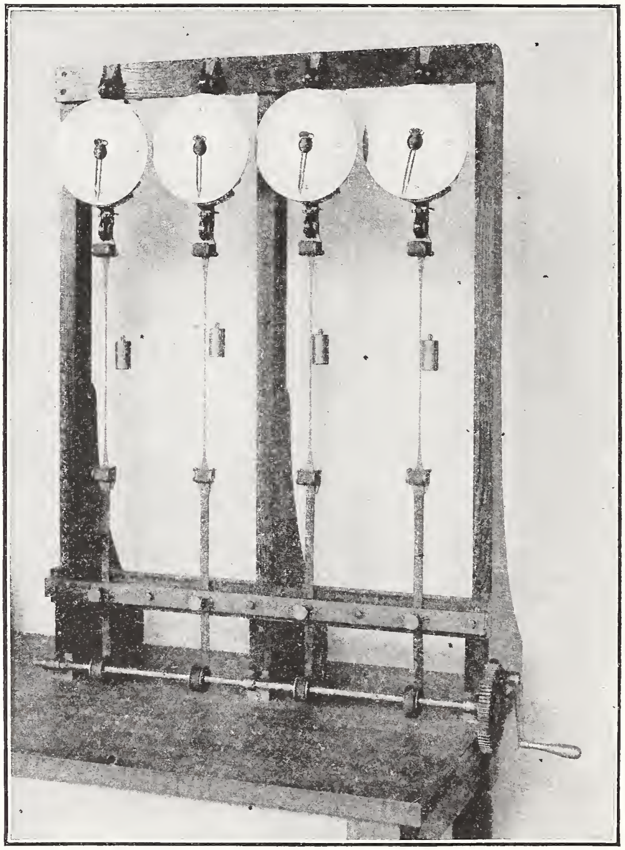

Fig. 21.—*Apparatus for determining the reduction in tension when rubber is stretched and held at a definite elongation*

(e) CONDITIONS AFFECTING THE RESULTS OF TENSION TESTS

In the absence of uniform methods of testing, it is found that results obtained in different laboratories sometimes show marked discrepancies which are due to the varying conditions under which the tests are made.

(1) Influence of Speed on Tensile Strength and Ultimate Elongation.—The speed at which rubber is stretched probably affects the results to a less extent than is often supposed, though doubtless different rubbers are not equally affected.

Table 2 shows the results obtained in a comparative test of four compounds.

Table 2.—*Tensile strength and ultimate elongation as affected by rate of stretching*

[Gauge length = 2 inches]

Compound	Rate of stretch	Tensile strength	Ultimate elongation
	In./min.	*Lbs./in.*^{2}	*Per cent*
G-18	5	2,495	605
	25	2,690	635
	45	2,720	635
M-1	5	1,900	465
	25	1,940	500
	45	1,970	490
B-1	5	375	340
	25	430	360
	45	465	375
Gy-1	5	340	105
	25	390	115
	45	430	120

These results would seem to indicate a general tendency toward higher values for both tensile strength and ultimate elongation as the speed of stretching increases.

(2) Influence of Temperature on Strength, Elongation, and Elasticity.—It is generally recognized that the physical properties of rubber are affected by changes in temperature, though, of course, to a less extent after vulcanization than before.

Figure 22 shows the results of tests at 50, 70, and 90° F. In each case the room was maintained at the specified temperature for three hours before the tests were made. It will be noticed that the rubbers are not all affected to the same extent by equal differences in temperature, but that there is a marked tendency in each case toward decreased strength, decreased set (increased elasticity), and increased elongations as the temperature is raised. It will be noted further that in nearly every case greater differences occur between 50 and 70° than between 70 and 90°.

The set in each case was measured after one-minute stretch and one-minute rest. Nos. 1 and 2 were stretched 350 per cent, Nos. 3 and 4, 300 per cent, and No. 6, 250 per cent.

(3) Influence of Cross Section on Tensile Strength and Ultimate Elongation.—Tensile strength and ultimate elongation are theoretically independent of sectional area, but, as in other materials, there is a tendency for small test pieces of rubber to develop higher unit values than large ones. Complete data on this subject are not at hand, but it is thought that test pieces one-fourth and one-half inch wide will show, in general, but little difference in ultimate elongation, but an appreciable difference in tensile strength, in the case of high-grade compounds.

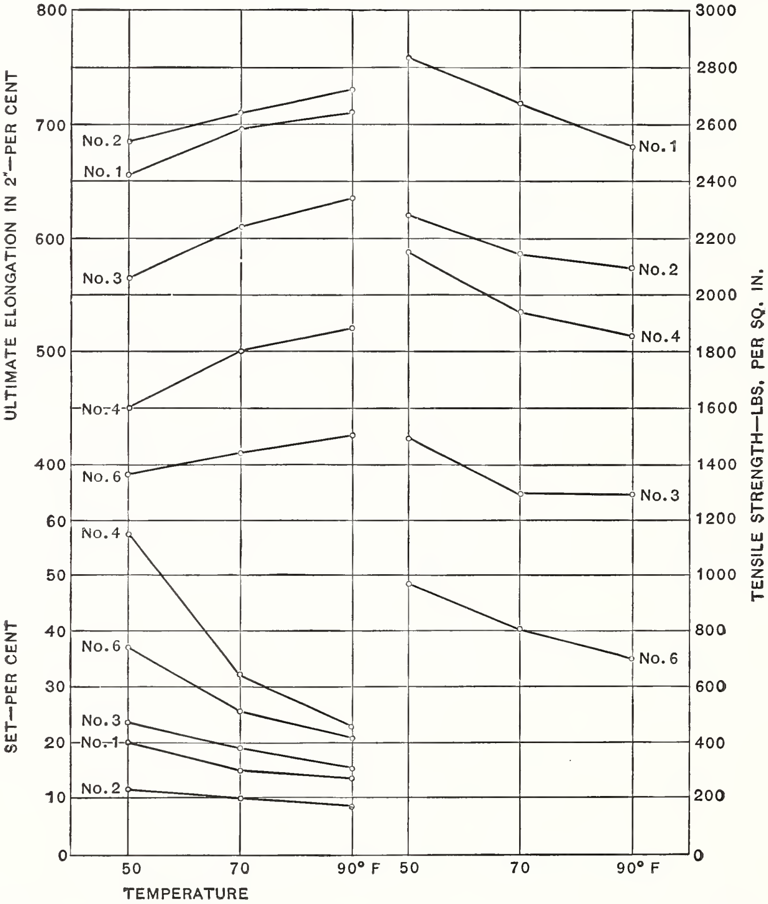

Fig. 22.—*Chart showing influence of temperature on the elasticity, tensile strength, and ultimate elongation of rubber*

Comparative results obtained with test pieces one-fourth and one-half inch wide are shown in Table 3. These rubbers represent commercial compounds. The test pieces were cut from the tubes and covers of plied hose, and the impression left by the fabric was carefully removed with an abrasive wheel.

TABLE 3.—*Influence of cross section upon the tensile strength and ultimate elongation of rubber*

Compound	Width of specimen	Tensile strength	Ultimate elongation
	Inch	*Lbs./in.*^{2}	*Per cent*
M-4 cover	¼	1,565	525
	½	1,455	515
M-3 tube	¼	2,160	580
	½	1,955	570
G-19 tube	¼	1,025	350
	½	955	340
G-19 cover	¼	735	255
	½	690	250
M-5 tube	¼	2,490	615
	½	2,060	575

TABLE 4.—*Influence of direction in which specimens are cut on tensile properties*

Sample No.	Direction	Tensile strength[1]	Ultimate elongation	Set[1] after 300 per cent elongation for 1 minute with 1 minute rest
		Lbs./in.^{2}	*Per cent*	*Per cent*
1	Longitudinal	2,730	630	11.2
	Transverse	2,575	640	7.3
2	Longitudinal	2,070	640	6.0
	Transverse	2,030	670	5.0
3	Longitudinal	1,200	480	22.1
	Transverse	1,260	555	16.3
4	Longitudinal	1,850	410	34.0
	Transverse	1,700	460	24.0
5	Longitudinal	690	320	----------
	Transverse	510	280	----------
6	Longitudinal	880	315	34.3
	Transverse	690	315	25.9

[1] The set and tensile strength were determined with different test pieces.

(4) INFLUENCE OF THE DIRECTION IN WHICH SPECIMENS ARE CUT ON STRENGTH, ELONGATION, AND ELASTICITY.—The tensile properties of sheet rubber are not the same in all directions, as will be shown later in connection with comparative tests of straight and ring-shaped test pieces. There is a tendency for specimens cut longitudinally, or in the direction in which the rubber has passed through the calender, to show greater strength and (at least for the better grades of compounds) less elongation than specimens cut transversely or across the sheet. The recovery, however, is greater in the transverse direction. Table 4 illustrates this.

No. 3 shows slightly greater strength transversely and No. 5 greater elongation longitudinally, while No. 6 shows the same elongation in each direction. The exception noted in No. 3 is attributed to experimental errors or to small defects in the specimens which escaped detection. It may be, however, that low-grade rubbers, such as Nos. 5 and 6, are not more extensible transversely than longitudinally.

(5) Influence of "Backing" on the Tensile Strength and Elasticity of Hose Lining.—In the case of cotton rubber-lined hose the "backing" which is used to cement the lining to the fabric, if not removed, affects to a greater or less extent the values obtained for tensile strength and elasticity or "set."

If the backing has an ultimate elongation greater than that of the lining, it must necessarily increase the tensile strength which is figured from the breaking load and the measured cross section of the rubber lining. The observed value for "set" which is obtained as described on page 52, is greater or less than the true value, according as the lining is more or less elastic than the backing. If the elasticity of the backing differs very much from that of the lining, it will be found that the broken test pieces have a tendency to curl up like a watch spring, with the backing on the inside or outside according as its elasticity is greater or less than that of the lining. It is customary to test hose linings with the backing removed.

In Table 5 are recorded the results of tests that were made to determine the effect of backing on the tensile properties of hose linings. For these tests the backing was removed with an abrasive wheel. Differences in the values of tensile strength in A and B may be attributed partly to errors in measuring the thickness of the specimens that were tested without removing the backing. In the case of No. 1, however, it is thought that the thickness was determined without appreciable error. The adhesion between tube and backing being weak, it was possible to separate the two by hand, and the eight specimens thus secured showed a maximum variation in thickness of only 0''.003. The average thickness was used in computing the strength of specimens tested with backing (A). It is seen that in this case the backing very materially increased the strength of the test pieces.

Table 5.—*Effect of backing on the tensile properties of fire-hose linings*

Sample [1]	Set	Tensile strength	Ultimate elongation
	Per cent	*Lbs./in.*^{2}	*Per cent*
1, A	[2] 24.5	3,080	590
1, B	[2] 23.0	2,535	575
2, A	[2] 17.5	3,015	625
2, B	[2] 18.5	2,790	635
3, A	[2] 17.5	3,025	645
3, B	[2] 19.0	2,615	610
4, A	[2] 20.5	2,705	650
4, B	[2] 19.0	2,720	645
5, A	[2] 24.0	2,795	580
5, B	[2] 23.5	2,300	585
6, A	[3] 20.5	2,555	535
6, B	[3] 22.5	2,410	540
7, A	[2] 21.0	2,095	550
7, B	[2] 17.5	2,335	590
8, A	[3] 21.5	1,190	535
8, B	[3] 20.5	1,190	535

[1] A, tested without removing backing; B, tested with backing removed.
[2] Measured after 400 per cent elongation for 10 minutes, with 10 minutes' rest.
[3] Measured after 350 per cent elongation for 10 minutes, with 10 minutes' rest.

(6) Influence of Previous Stretching on Strength, Elongation and Elasticity.—Test pieces that have been used to determine set usually show greater strength and ultimate elongation than are obtained with test pieces that have not been previously stretched.

Table 6.—*Influence of repeated stretch on tensile strength and ultimate elongation*

Sample No.	Tensile strength		Ultimate elongation	
	Single stretch	Repeated stretch	Single stretch	Repeated stretch
	Lbs./in.	*Lbs./in.*	*Per cent*	*Per cent*
1	2, 470	2, 610	645	765
2	1, 740	1, 960	665	780
3	990	1, 180	510	645
4	1, 710	1, 790	460	555
5	750	790	430	440
6	930	920	375	465

Table 6 gives the strength and ultimate elongation obtained in testing six samples of rubber, first, with a single stretch, and second, by repeated stretching, beginning with 200 per cent and increasing each stretch by 100 per cent until failure.

In testing rubber as described above, it is found that if during the latter part of the test the increments in extension for successive stretches are small (say, 25 per cent of the original length), a point is reached where the rubber breaks down, and beyond which it fails on a subsequent stretch at a tension less than the maximum which it has already withstood.

The elasticity or set after a definite elongation is usually greater if the rubber has been previously stretched than if determined in the usual way. This is illustrated by the results shown in Table 7; those marked "Repeated stretch" show the set after repeated stretching, beginning with 100 per cent and increasing 100 per cent for each subsequent stretch. Those marked "Single stretch" were obtained in the usual way, each specimen being stretched but once. In each case the set was measured from the original gauge marks after one-minute stretch and one-minute rest, the tabulated results being the average of the values obtained in testing a number of specimens.

Table 7.—*Influence of repeated stretch on the elasticity of rubber*

Sample No.	Method of testing	Set (in per cent) after being stretched—				
		100 per cent	200 per cent	300 per cent	400 per cent	500 per cent
1	Repeated stretch	1. 0	4. 5	9. 5	16. 0	25. 0
	Single stretch			11. 7	19. 8	29. 0
2	Repeated stretch	1. 8	4. 0	7. 7	13. 7	21. 2
	Single stretch			8. 0	14. 7	21. 5
3	Repeated stretch	3. 7	9. 0	17. 7	27. 0	37. 0
	Single stretch			21. 7	34. 0	47. 0
4	Repeated stretch	4. 0	12. 3	28. 7	48. 7	
	Single stretch		14. 3	33. 0	56. 0	
5	Repeated stretch	8. 1	19. 4	34. 0		
	Single stretch		19. 3	33. 0		
6	Repeated stretch	4. 3	16. 3	34. 0		
	Single stretch		17. 0	35. 3		

It will be noted that the effect of previous stretching is very marked in the case of Nos. 1, 3, and 4; that it is very slight in the case of Nos. 2 and 6; and that in the case of No. 5 the set is slightly increased by previous stretching.

(7) Influence of the Form of Test Piece on the Results of Tension Tests.—There is a wide difference of opinion in regard to the relative merits of the straight and ring-shaped test pieces. The ring, which is highly recommended by some, undoubtedly possesses certain advantages as regards convenience in testing, and uniform results may be obtained with this form of test piece.

Ring test pieces, however, do not show the full tensile strength of rubber, on account of the uneven distribution of stress over the cross section. This fact is evident from a simple analysis, and may be verified by comparative tests with straight and ring-shaped test pieces, provided the straight test pieces are sufficiently enlarged at the ends to prevent failure in the grips, and provided further that the change in width is not made too abruptly.

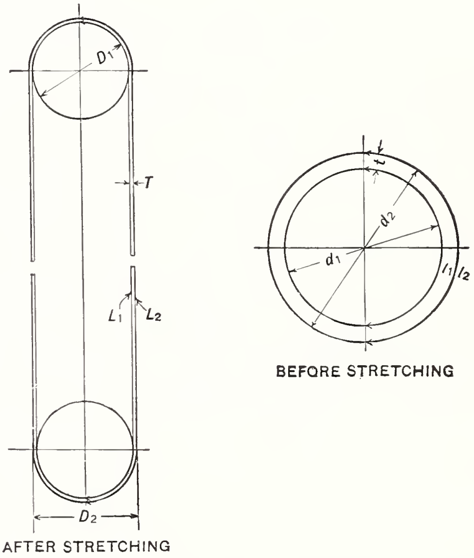

Fig. 23.—*Ring test piece before and after being stretched*

Assuming for simplicity that the extensibility of rubber is the same in all directions, it will be seen by reference to Figure 23, which represents a ring test piece before and after extension, that,

If E_1 = per cent elongation of the inside surface at breaking point (automatically measured), then

$$E_1 = \frac{L_1 - l_1}{l_1} \cdot 100 \qquad (1)$$

If E_2 = per cent elongation of the outside surface when specimen breaks, then

$$E_2=\frac{L_2-l_2}{l_2}\cdot 100 \tag{2}$$

$$L_2=L_1+\pi/2\ (D_2-D_1)=L_1+\pi T \tag{3}$$

$$T=t\sqrt{\frac{l_1}{L_1}}\text{ approximately} \tag{4}$$

(assuming that the volume of rubber is constant).

From equations 1, 2, 3, and 4 we have

$$E_2=E_1\cdot\frac{l_1}{l_2}\cdot\frac{L_1+\pi t\sqrt{\frac{l_1}{L_1}}-l_2}{L_1-l_1} \tag{5}$$

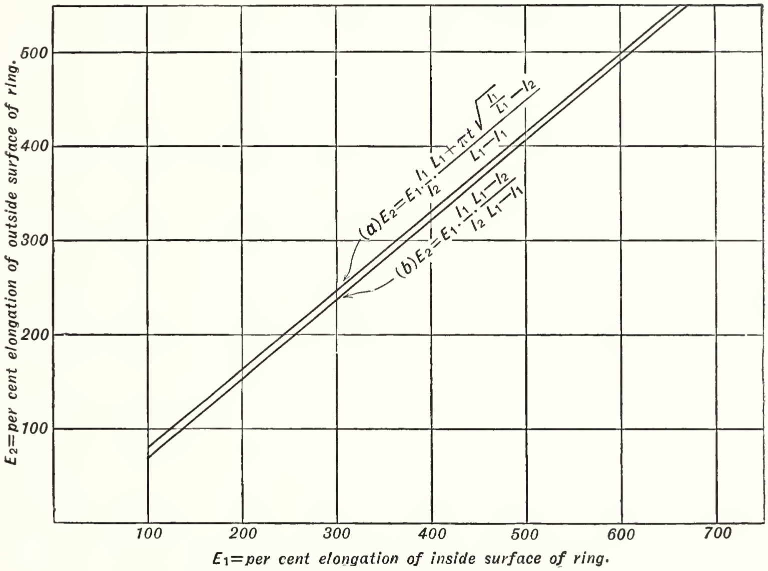

FIG. 24.—*Relation between the elongation at the inside and outside of a ring*

which is represented graphically in Figure 24 (*a*) for the usual size of ring in which $l_1=70$ mm and $t=4$ mm.

This relation is practically a linear one, and $E_2=0.83\ E_1$ approximately. Figure 24 (*b*) shows the slight error introduced by neglecting the term $\pi t\sqrt{\frac{l_1}{L_1}}$

Now, since the percentage of elongation at the outside surface of the ring is less than at the inside surface, the tensile stress must also be less at the outside than at the inside surface. From equation (5) it follows that the decrease in the percentage of elongation is approximately uniform from the inside to the outside of the ring, this relation being shown in Figure 25 for $E_1=600$ per cent and $l_1=70$ mm.

The relation between stress and elongation being practically a linear one for values of elongation near the breaking point, the decrease in tensile stress must also be fairly uniform from the inside to the outside of the ring at the time of failure. This is illustrated graphically in Figure 25, in which the values for tensile stress were taken from the stress-strain diagram shown in Figure 26, No. 1.

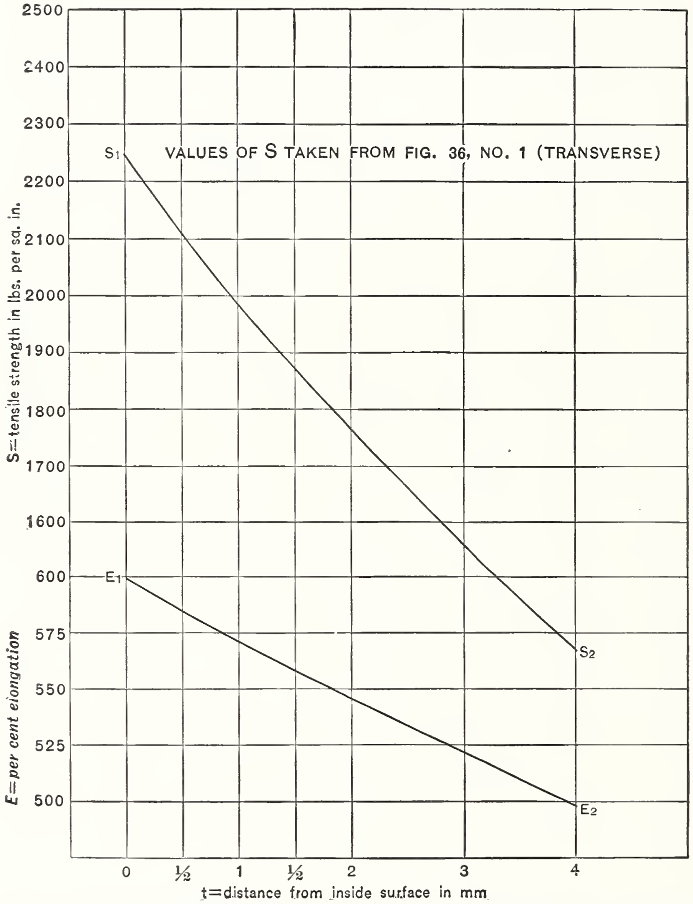

FIG. 25.—*Variation in elongation and tensile strength from the inside to the outside of a ring*

If S_1 = the stress at the inside surface of the ring at failure, or true tensile strength of the rubber corresponding to E_1, and S_2 = the stress at the outside surface, corresponding to E_2, and S = the average stress over the cross section of the ring, which is the value for tensile strength obtained by the ring method, we have

$$S=\frac{\text{Breaking load}}{\text{Area of section}}=\frac{S_1+S_2}{2}=\frac{S_1}{2}\left(1+\frac{S_2}{S_1}\right) \text{ approximately.} \qquad (6)$$

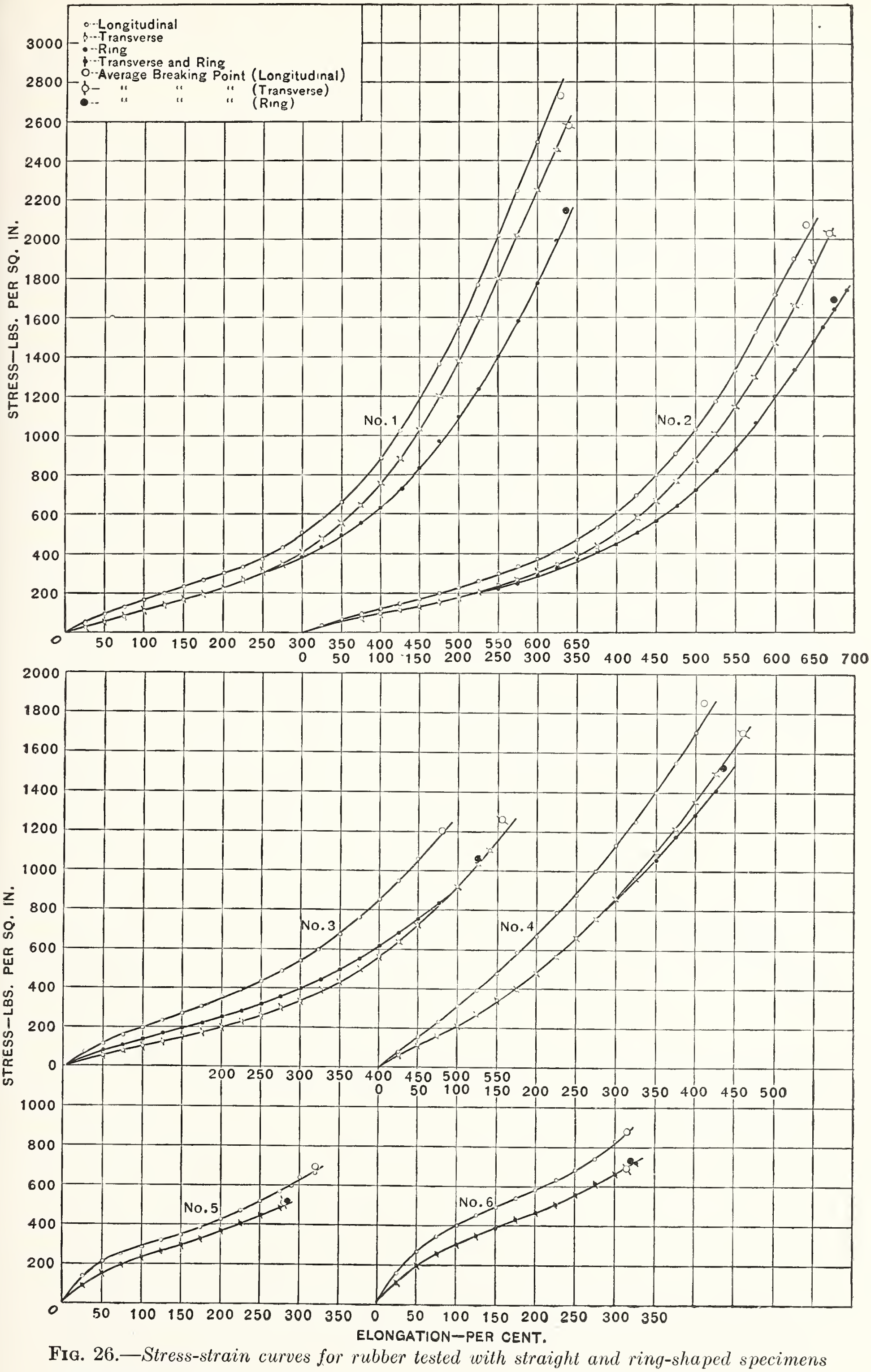

FIG. 26.—*Stress-strain curves for rubber tested with straight and ring-shaped specimens*

Now, since the ratio $\frac{S_2}{S_1}$ varies for different rubbers, S does not bear a constant ratio to, and, therefore, can not be taken as a measure of tensile strength. Elongation, however, is measured at the inside surface of the ring and, therefore, represents the maximum extension of the rubber around the inside of the ring. The average elongation over the cross section of the ring is,

$$E = 1/2\ (E_1 + E_2)\ \text{approximately.} \qquad (7)$$

If the extensibility of rubber were the same in all directions, values of S and E obtained from equations (6) and (7) would, theoretically, give a point lying very near the stress-strain curve for the same rubber tested in the form of a straight specimen. This, however, is not the case, as has been already pointed out, and as may be seen from Figure 26.

The difference between S and S_1 is greater for high-grade rubbers than for compounds of poor quality, as may be seen by reference to Figure 26, which represents stress-strain curves plotted from the results of tests on straight and ring specimens. Table 8 shows values for tensile strength and ultimate elongation obtained for the same rubbers by the two methods.

The ring test piece obviously does not give a true stress-strain curve on account of the varying stress over its cross section.

TABLE 8.—*Relative tensile strength and elongation of rubber tested with straight and ring-shaped specimens*

Specimens	Tensile strength, in pounds per square inch, for rubber compounds Nos. 1 to 6					
	1	2	3	4	5	6
Straight specimens:						
Longitudinal (L) [1]	2,730	2,070	1,200	1,850	690	880
Transverse (T)	2,575	2,030	1,260	1,700	510	690
Ring specimens (R)	2,140	1,690	1,060	1,520	510	730
R/L	.78	.82	.88	.82	.74	.83
R/T	.83	.83	.84	.89	1.00	1.06
	Ultimate elongation, in per cent					
Straight specimens:						
Longitudinal	630	640	480	410	320	315
Transverse	640	670	555	460	280	315
Ring specimens	635	675	525	435	285	320

[1] Longitudinal indicates the direction in which the rubber has been passed through the calender rolls.

Straight specimens were cut both longitudinally and transversely, with reference to the direction in which the rubber had been passed through the calender rolls. They were tested with the machine shown in Figure 14, and the ring specimens were tested with the machine shown in Figure 18. In each case the specimens were stretched at the rate of about 8 inches per minute. A number of test pieces, both straight and ring-shaped, showed, particularly in the case of No. 3, abnormally low tensile strength and elongation on account of small holes or particles of grit at the point of rupture. Such specimens are not included in the results tabulated above, each of which represents the average of from 5 to 15 tests.

A line was drawn across each of the ring specimens to indicate the longitudinal direction, and the point of failure was noted. There was a tendency for the rings to rupture along this line, thus indicating that the sheets were strongest longitudinally, or in the direction of rolling. This difference in strength is shown by the straight test pieces, except in the case of compound No. 3. It is seen from Figure 26 that the curve for transverse specimens lies below that for longitudinal specimens, thus showing that a given stress will produce a greater elongation if applied transversely than if applied longitudinally. It is to be expected, therefore, that the elongation of a ring will be less than that for a transverse straight specimen. The natural variation in rubber, however, is often sufficient to obscure small differences in elongation, which are due to the methods of testing.

In the case of Nos. 5 and 6, the curves for the ring specimens almost coincide with those for the transverse straight specimens, and the tensile strengths of these rubbers when tested by the two methods are seen to agree fairly well. It is to be noted, however, that for the higher-grade rubbers the difference in tensile strength by the two methods is very marked. Although the difference is not great, there is a tendency for the transverse specimens to show a greater ultimate elongation than the longitudinal specimens, notwithstanding the greater stength shown in the latter case.

(*f*) "FRICTION" TEST

The "friction" or adhesion between the plies of fabric, or between the fabric and rubber parts, is of great importance; in fact, the life of pneumatic tires, belting, hose, etc., depends in a great measure upon the efficiency of this adhesion. For a description of detailed test methods see Bureau Circular No. 232.

Friction is preferably determined autographically with the machine illustrated in Figure 27, which records the tension required to cause a definite rate of separation between the two parts considered.

The machine is operated by a one-eighth-horsepower worm-geared shunt motor which is belted to a stepped pulley. A worm on the pulley shaft drives a worm wheel which is geared to a spur inside of the vertical steel column. This spur drives a steel rack, to the upper end of which is attached the movable grip. From the top of the machine is suspended a spring which carries at its lower end a fork or suitable grip for holding the test piece. Between the spring and fork is a pencil holder, in front of which is a drum carrying a paper chart on which the record is drawn.

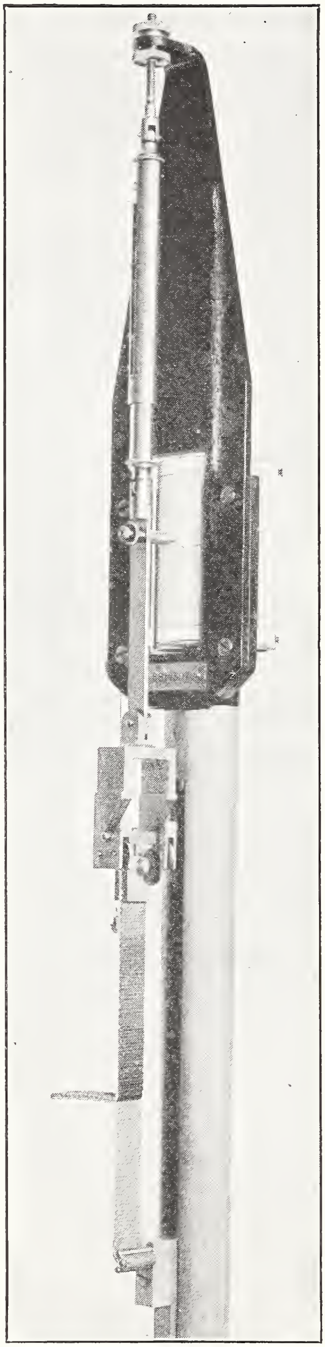

FIG. 27.—*Autographic friction machine for measuring and recording the adhesion between plies of fabric*

The machine is constructed so that a spring suitable for the grade of material being tested can be used in it. The drum is driven by a cord which passes over a small guide pulley and thence to a spool on the spur-gear shaft. The surface speed of the drum is the same as the rate of separation of the plies of fabric being tested.

In the case of rubber hose a 1-inch section is fitted over a mandrel and placed in the fork suspended from the top of the machine, and the detached end of the fabric is secured to the lower grip as shown.

In the case of rubber belting a strip 1 inch wide containing two plies is used. The plies are separated for a short distance and the ends secured in the two grips, the upper grip being a clamp held in the fork above mentioned.

The method formerly used, but which is rapidly going out of use, is illustrated in Figure 28 and consists in measuring the rate of separation produced by the action of a dead weight. The results of this test are influenced by the temperature conditions, the rate of stripping caused by a given weight being greater at high than at low temperatures. Also, the rate of stripping is greater if the mandrel fits snugly in the ring than if the ring is allowed to sag over a loose mandrel. The variation in friction, however, in the same hose is often such as to obscure these influences, unless observations are made under conditions which differ greatly.

In connection with this test, attention may be called to a point which, though generally recognized, is sometimes lost sight of in the interpretation of results.

It has been observed that no stripping is produced by increasing the weight up to a certain point, after which the rate of stripping increases gradually at first, and then more rapidly, with small increments in weight, until finally a very small increase in weight causes a large change in the rate of stripping. The general behavior is illustrated graphically in Figure 29, in which each point represents the average of a number of tests on a very uniformly frictioned hose.

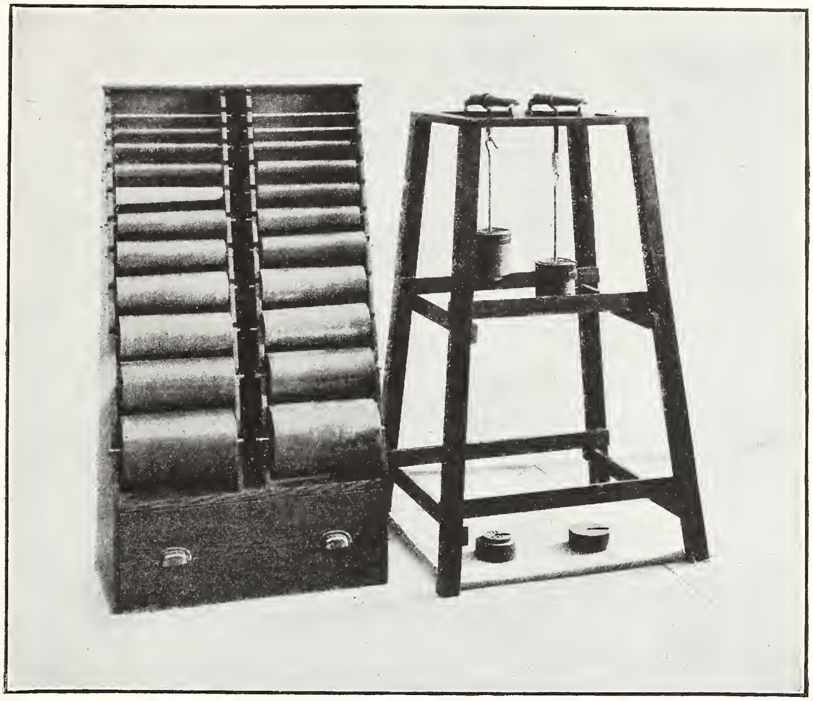

Fig. 28.—*Apparatus for testing the "friction" of rubber hose by the dead-weight method*

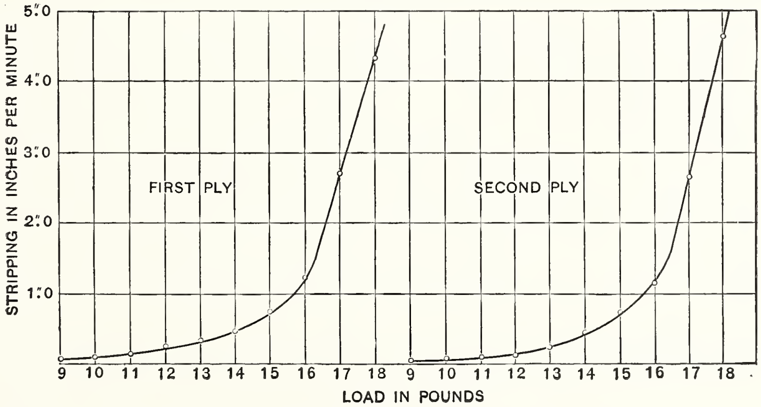

Fig. 29.—*"Friction" test of rubber hose. Curves showing rate of stripping under different loads*

As a result of this behavior, an air hose, for example, which is required to show a rate of stripping not exceeding 6 inches in 10 minutes under 25 pounds, might be regarded as of very inferior quality if it stripped, say, 20 inches in 10 minutes, whereas the same hose would probably show little or no stripping under 20 pounds and come within the required limit under 22 pounds.

In testing fire hose, the central portion of the lining is separated from the jacket for a short distance at one end. The detached end of the jacket is clamped in a stationary grip, and the weight is suspended from the rubber lining.

The "friction" between the plies of duck in rubber belting is sometimes tested by applying the load in a direction at right angles to the plane of separation, as in the case of "plied" hose. As shown in Figure 30, A, this is done by cutting the belt about halfway through along parallel lines 1 inch apart. The belt rests on horizontal supports just outside of the strip which has been cut, and the weight is suspended from the detached end of the duck. Figure 30, B, illustrates a method by which a 1-inch strip is held in a fixed clamp with the weight suspended from the end of a detached layer of duck. In testing by this method it will be found that the angle of separation varies somewhat according to the thickness of the strip and that the results are thereby influenced to some extent. The difficulty may be avoided by subdividing the 1-inch section into strips of two plies each, as shown in Figure 30, C, and very satisfactory results are obtained in this way. It is found that for a given weight the rate of stripping is decidedly greater by methods B and C than by method A. Figure 31 shows graphically the results obtained by the three methods.

A marked difference is often found in the friction between different plies of the same hose, or other articles of plied construction, as well as at different points along the same ply. Uniformity in the friction is desirable.

(g) HYDRAULIC-PRESSURE TEST

The pressure test as usually made consists simply in subjecting the hose to water pressure created by a force pump of any convenient type. The coupling at the free end is closed with a plug, and the pump connection is made with a reducing coupling. By using two clamps at each end it is possible to make a joint which is tight even under high pressure. It is necessary to provide a check valve to protect the pressure gauge against shock when a hose bursts. A pet cock must be provided to release the air as the hose is being filled.

Requirements of specifications as regards the pressure test vary according to the kind of hose, but, as a rule, the test is made not with the view of developing the ultimate strength of the hose but rather to detect defects in workmanship, which are usually noticeable at a pressure well below that necessary to rupture the hose.

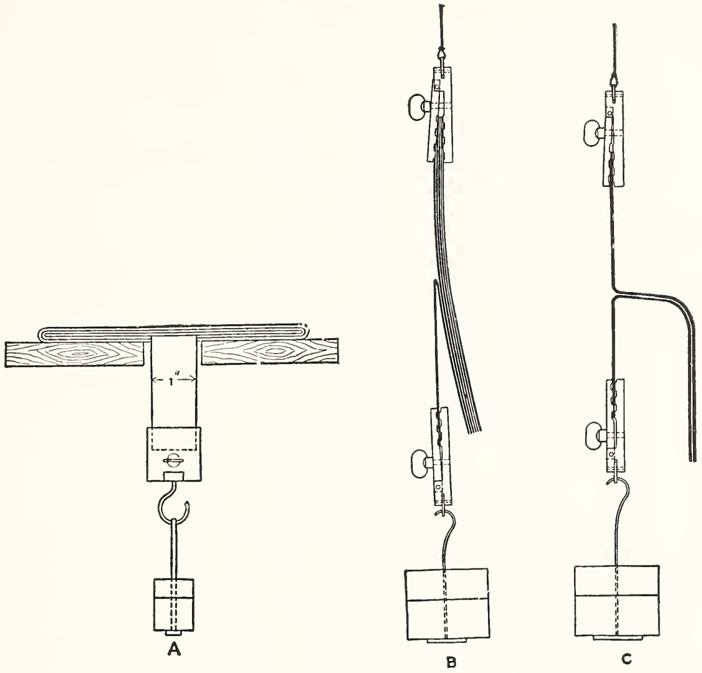

FIG. 30.—*Illustrating methods of testing the "friction" of rubber belting by the application of dead weights*

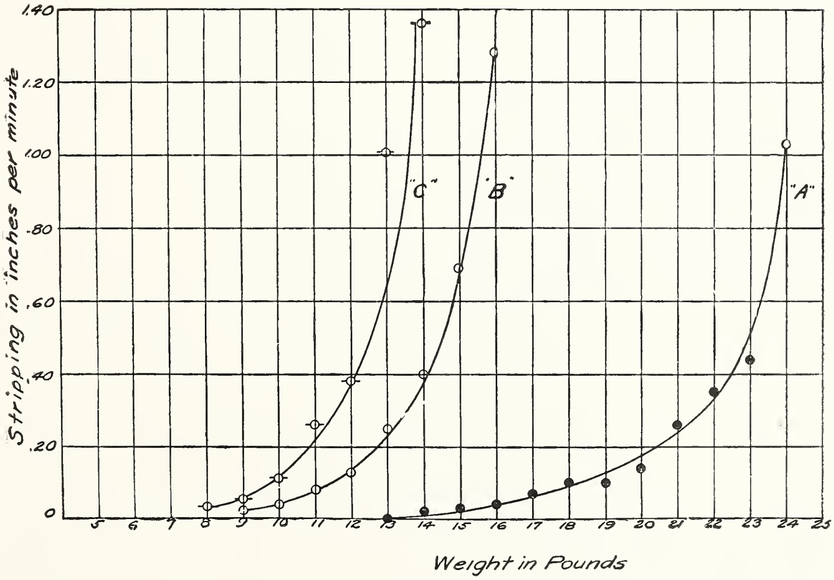

FIG. 31.—*Friction test of rubber belting*

Curves showing rate of stripping when tested by the methods illustrated in Figure 30

In the case of fire hose it is usual to specify a certain pressure when the hose is lying straight or when bent to the arc of a circle of given radius, and the hose must stand a specified pressure when doubled upon itself. A full 50-foot length must not show excessive expansion, elongation, warping, or twist under pressure, and any twist must be in a direction tending to tighten the couplings.

(h) STEAMING TEST

Figure 32 illustrates a method of testing steam hose. The header *1* is provided with six outlets, each of which is controlled by a one-half inch globe valve. The header *2*, which is connected to a steam trap *3*, is similarly provided with inlets and controlling valves. The hose to be tested is cut into lengths that will just fit between the connections on the headers, the bottom connections being made with unions. Steam passes through a regulating valve (not shown) into the header *1* and thence through the hose to the header *2*, from which the condensation is carried to the steam trap.

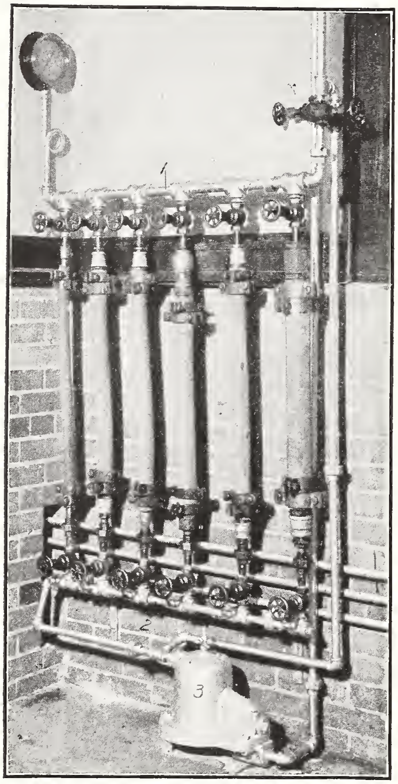

Fig. 32.—*Six samples of steam hose connected up for steaming test*

(i) TESTING THE RUBBER INSULATION OF WIRE

The mechanical tests that are usually specified for the rubber insulation of wire are the same as those already described in connection with tension tests.

The method of preparing test pieces, however, is not always the same, but depends upon the size of the conductor and the character of the insulation. (See Federal Specifications Board Specification No. 59, Bureau of Standards Circular No. 232.)

(j) COMPARATIVE TESTS OF MACHINE AND HANDMADE TUBES

A question is sometimes raised as to whether the tube of a hose has been made in a tubing machine or from calendered sheet. Calendered tubes are often specified, and since it is sometimes impossible to determine by inspection if a tube is machinemade or handmade, a test that could be depended upon in all cases to distinguish between the two kinds of tubes would serve a useful purpose.

From the nature of the case a chemical analysis could not determine this point, because a compound might be calendered or run through a tubing machine without in any way altering its composition. The mechanical properties of a compound, however, are influenced to a greater or less extent by the method of manufacture.

Comparative results for elasticity or "set," tensile strength, and ultimate elongation are given in Table 9. Each of the three compounds was made into two tubes of the same size, one from calendered sheet and the other made in a tubing machine. The conditions of vulcanization were the same for each pair of tubes, so that any difference in their physical properties is fairly attributable to the effects of the tubing machine and calender rolls.

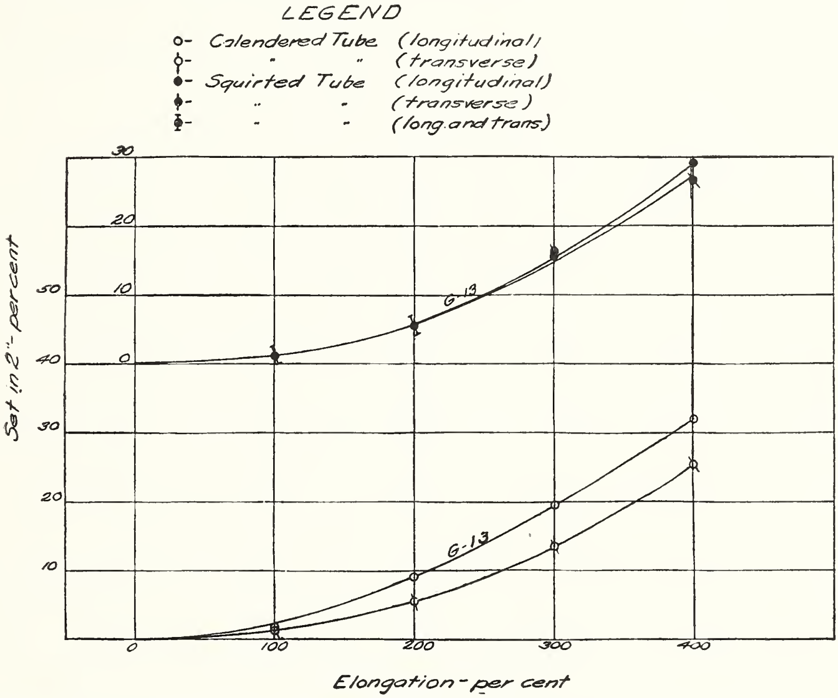

FIG. 33.—*Curves showing the elasticity of a "squirted" tube as compared with a calendered tube, tested in the longitudinal and transverse directions*

In comparing the results it is interesting to note that the squirted tubes show practically the same set when tested longitudinally and transversely, whereas the calendered stocks are much more elastic in the transverse direction, as has been pointed out in connection with tests previously described. This is more clearly illustrated in Figure 33, which shows graphically the relation between set and elongation for test pieces cut longitudinally and transversely from the compound G–13. In the same way Figure 34 shows stress-strain curves for G–13. Similar tests of the other two compounds could not be made for lack of material.

TABLE 9.—*Relative tensile properties of "squirted" and calendered tubes when tested in the longitudinal and transverse directions*

Sample No.	Method of manufacture	Set [1] after one-minute stretch and one-minute rest		Tensile strength		Ultimate elongation	
		Longitudinal	Transverse	Longitudinal	Transverse	Longitudinal	Transverse
		Per cent	*Per cent*	*Lbs./in.*	*Lbs./in.*	*Per cent*	*Per cent*
V-14	Calendered	11. 7	10. 5	520	530	275	290
	Squirted	12. 5	12. 5	460	460	240	235
V-16	Calendered	24. 7	20. 0	1, 450	1, 380	530	535
	Squirted	23. 0	21. 8	1, 310	1, 290	525	505
G-13	Calendered	26. 0	18. 5	1, 110	1, 295	455	510
	Squirted	22. 5	21. 5	1, 130	1, 330	470	510

[1] V-14 was stretched 200 per cent; V-16 and G-13, 350 per cent.

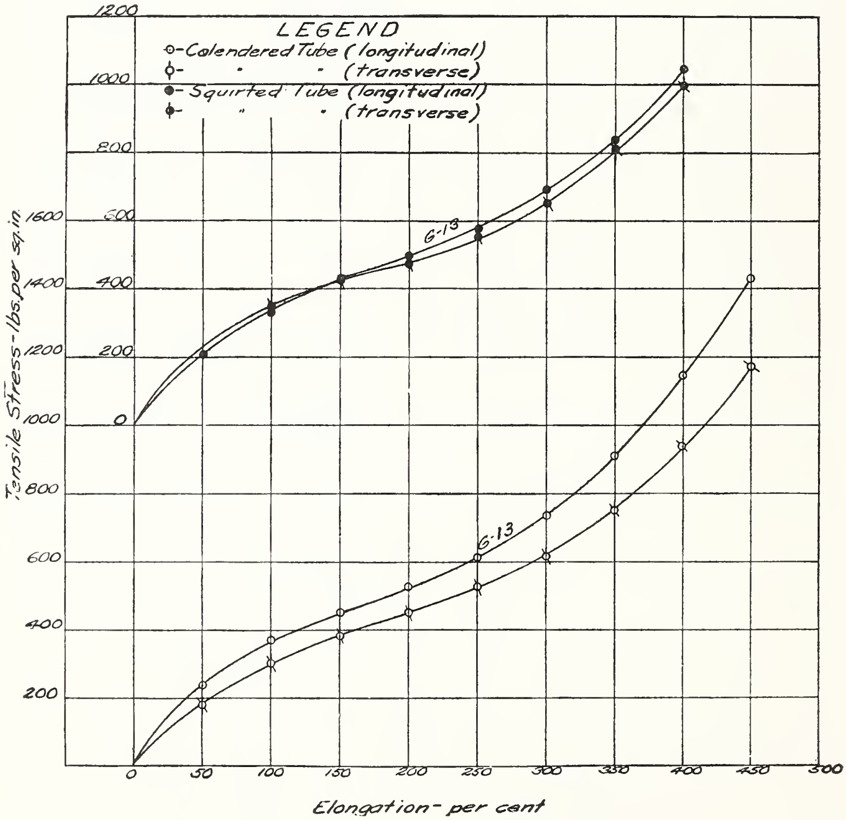

FIG. 34.—*Stress-strain curves for a "squirted" and a calendered tube tested in the longitudinal and transverse directions*

(k) TESTING OF RUBBER BANDS

(1) Under One-fourth Inch in Width.—In the testing of rubber bands a different method is employed from that used in the testing of other types of rubber goods. Owing to the size of the specimens, it is impracticable to obtain accurately the cross-sectional area, consequently the tensile strength is not computed in pounds per square inch, as is customary in other goods.

Double-spool grips similar to the grips illustrated in Figure 13, B, but smaller, are used. The test machine used for this work is shown in Figure 19, and a description may be found on page 50.

Tensile strength and ultimate elongation.—The tensile strength is measured in pounds and the elongation is expressed in percentage of the original length.

$$\text{Ultimate elongation, per cent} = \frac{(L - L_1)\ 100}{L_1}$$

where

L = one half the distance around spools at time of failure.
L_1 = initial inside length of band, measured flat (one-half total length).

(2) Bands One-fourth Inch in Width or Over.—The testing of bands one-fourth inch or more in width requires the use of the grips shown in Figure 13, B, and the tests are made on the machine usually employed for determining the tensile strength of rubber (fig. 14).

Tensile strength and ultimate elongation.—In bands of this size it is practicable to measure the width and thickness. The tensile strength is calculated in pounds per square inch as follows:

$$\text{Tensile strength in pounds per square inch} = \frac{B}{2\ wt}$$

where

B = breaking load in pounds.
w = width of band in inches.
t = thickness of band in inches.

Elongation is measured and expressed in the same way as described above for bands under one-fourth inch in width.

Jar rings are tested in the same way as rubber bands. Owing to the shape of the material, the internal length L_1 becomes one-half the internal circumference, thus:

$$L_1 = \frac{\pi D}{2}$$

where

D = internal diameter in inches.

(l) ABRASIVE RESISTANCE OF RUBBER COMPOUNDS

A great deal of work has been done in the past few years in an attempt to develop laboratory machines which would measure accurately the resistance to abrasion of a rubber compound. Many different types have been tried, several of which when used to compare stocks of similar compositions give a good measure of the relative resistance to abrasion. In spite of the large amount of work done, however, there is no machine which has been generally accepted as a standard.

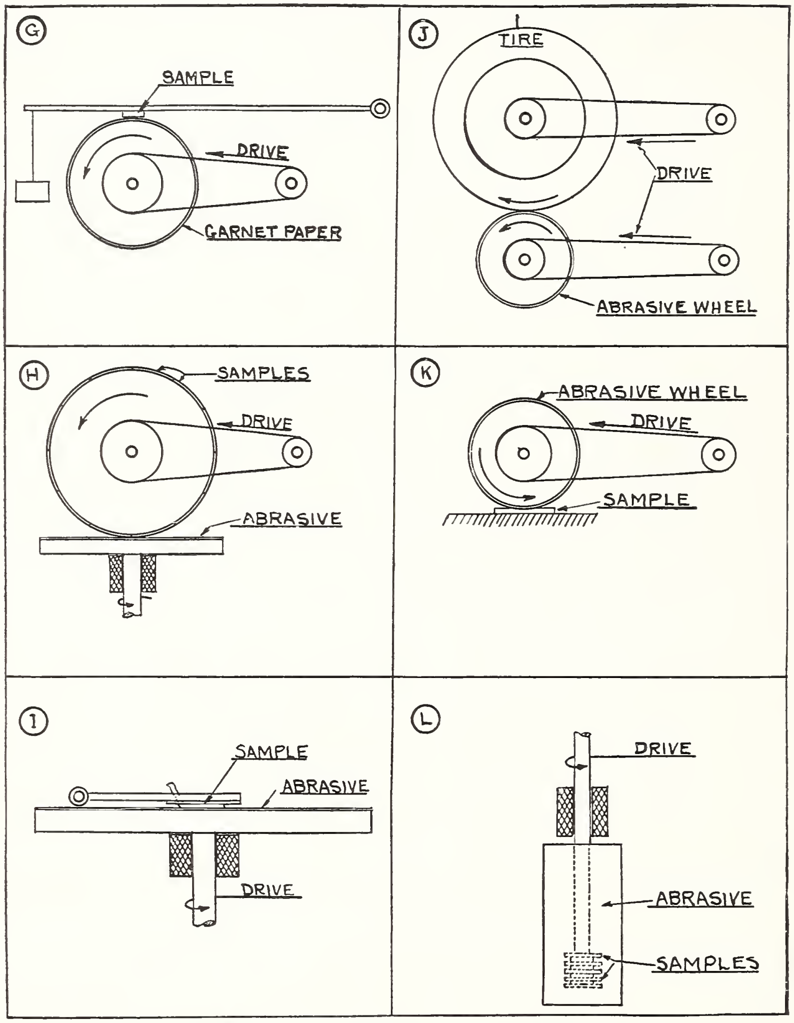

Fig. 35.—*Methods of operation of abrasion test machines*

Some of the different types which have been developed are illustrated in Figure 35.

These machines may be briefly described as follows:

Machine G.—Samples 1 inch square and one-fourth inch thick are secured to a weighted arm and pressed against the face of an abrasive wheel 6 inches in diameter, which is surfaced with garnet paper.

Machine H.[4]—Twelve samples each 2 by 3⅞ inches by ¼ inch are fastened around the circumference of a flat-faced wheel approximately 15 inches in diameter. This wheel rests upon the surface of a carborundum disk the axis of which is at right angles to that of the wheel, and thus the wheel and disk constitute a friction drive by means of which the samples drive the abrasive disk against a constant resistance produced by a friction brake.

Machine I.[5]—This machine consists essentially of a revolving table 30 inches in diameter, the surface of which is composed of crushed feldspar held in place with Portland cement. The samples 2 by 5 inches by ¼ inch are held in grips which are swivelled so that they move under their weight. The samples bear against the surface of the table and are abraded as the table revolves.

Machine J.—In this test a complete tire is used. It is mounted on a rim, inflated, and pressed against the face of a carborundum wheel. The tire and the abrasive wheel are driven in the same direction where in contact, but at different speeds. The tire is run until the tread wears through.

Machine K.—In this apparatus a sample approximately 2 by 3 inches by ¼ inch is pressed against the face of an abrasive wheel.

Machine L.[6]—In this machine a series of disks of the rubber samples are held on a spindle and caused to revolve in a cylindrical vessel containing a granular abrasive.

2. THE PHYSICAL TESTING OF HARD RUBBER

The methods of testing soft rubber are not, in general, applicable to hard rubber because of the difference in the properties and uses of the two materials. Much of the testing of soft rubber relates to the state of cure and the aging characteristics. But, in the case of hard rubber, aging is a negligible factor, and there is wide latitude in the proper cure, with little danger of overvulcanization. Furthermore, hard-rubber articles are seldom used where they are exposed to mechanical wear, consequently abrasion and stress-strain tests designed to show the wearing qualities of soft rubber have no place in the examination of hard rubber.

(a) TENSILE STRENGTH

The tensile properties of hard rubber resemble those of a metal. The machines used for the mechanical testing of soft rubber are not suited to measuring the strength of hard rubber because of its high tensile strength and relatively slight ultimate elongation. At the Bureau of Standards a small universal screw-testing machine is used

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[4] An apparatus for measuring the relative wear of sole leather, B. S. Tech. Paper No. 147.

[5] "An abrasion machine," issued by New Jersey Zinc Co., Feb. 1, 1923.

[6] W. W. Evans, Proc. Am. Soc. Testing Materials, 1923, p. 517.

for tensile tests of hard rubber. The tensile strength is indicated by the load on the beam and the elongation is measured with a short scale clipped behind marks on the test strip.

Various methods have been used for the preparation of dumb-bell shaped test strips. Some investigators recommend that they be machined from panels of hard rubber. Others vulcanize test strips in molds of the appropriate shape. A yet simpler method, to which there is no apparent objection, is to cut test strips from a panel, which has been softened by gentle heat, with the use of the same die that is employed for cutting strips from soft-rubber sheets.

(b) IMPACT TESTS

Since many of the failures of hard rubber in service are due to mechanical fracture various impact tests have been devised to determine the resistance of hard rubber to shock. The test machines are designed to deliver gradually increasing blows to a rubber strip of definite dimensions until it is fractured. In one type of machine there is a pendulum which swings with a regularly increasing arc, and in another a weight which is dropped from heights increased in regular increments.

(c) COLD FLOW TEST

Hard-rubber members under heavy load, such as panels carrying heavy switches, are apt to sag and become distorted by slow flowing of the rubber. This property is measured by placing a weight on a strip of hard rubber supported horizontally between two points and observing the deflection after a given time at a fixed temperature.

(d) OTHER TESTS

Various other tests are applied to hard rubber to determine its fitness for specific purposes. The details of these have not been standardized. There is a variety of electrical tests, including the determination of dielectric constant, power factor, surface and volume resistivity, and dielectric strength. These electrical tests, while of considerable importance, are beyond the scope of this publication but are described in a Technologic Paper (in preparation).

3. CHEMICAL ANALYSIS OF RUBBER

A detailed description of the methods employed in the analysis of rubber may be found in Circular 232, United States Government general specification No. 59 for Rubber Goods (Methods of Physical Tests and Chemical Analyses), issued by this bureau.

No satisfactory method for the direct determination of rubber has been found to date. The amount of rubber is determined by the difference between 100 per cent and the sum of the percentages of the other constituents. The methods employed are intended to ascertain the amounts of all other constituents present in a rubber compound. When the sample is comparatively simple in composition and contains a large amount of high-grade new rubber, the analysis presents few difficulties. If it is quite complex in character and contains inorganic and organic fillers, of which there is a great variety, as well as mixtures of crude and reclaimed rubbers, a good approximation of the exact amount of rubber used is all that can be obtained. There is no method of determining the amounts of various crude rubbers if more than one is present, and no method of determining the presence or amount of reclaimed rubber. However, if the analysis indicates the presence of small amounts of a variety of fillers and the tensile strength and other physical characteristics are not what may be expected from a similar properly vulcanized compound known to contain only new crude rubber of standard quality, the analyst is justified in suspecting the presence of reclaim.

Many rubber technologists believe that the analysis of a rubber compound as an indication of its quality has lost its significance. It is true that before the general use of organic accelerators more information could be obtained from the character and amount of acetone soluble material than is possible at present. The amount of combined sulphur when accelerators of vulcanization have been used furnishes little information about the degree of cure. However, an experienced rubber technologist may obtain much valuable information from the results of an analysis. It is surprising how frequently the results of an analysis complement the physical test data.

The determination of the amount of acetone soluble material is important in both crude and vulcanized rubber. The rubber hydrocarbon is insoluble in acetone and alcohol, but acetone is commonly used in rubber analysis because it is an excellent solvent of other organic materials which may be present.

The acetone extract from vulcanized rubber contains rubber resins, oxidized rubber, free sulphur, mineral oils, waxes, and certain softeners added during milling to facilitate mixing of the various materials used in the rubber compound. The acetone partially extracts bituminous materials and vulcanized oils if present. The percentage of extracted or free sulphur is determined and deducted from the percentage of total extract. The corrected value may be helpful in determining the quality of rubber used. The better grades of Hevea rubber contain not more than 5 per cent of acetone soluble material. A high acetone extract does not necessarily indicate the absence of high-grade Hevea rubber nor the presence of reclaimed rubber.

Insulating compounds frequently contain paraffin or ceresin. The maximum amount permitted by specifications is 4 per cent, but as much as this is rarely present in rubber insulating compounds. The amount of waxy hydrocarbons is determined as a correction of the value for acetone extract, as well as to learn whether a compound complies with specifications.

After extracting the rubber with acetone it is extracted in the same apparatus with chloroform, which removes nearly all of the remaining bituminous material that was not extracted by the acetone. Chloroform may also dissolve some of the rubber, particularly when it has been lightly cured. If vulcanized oil substitutes are present, the rubber is further extracted with alcoholic potassium hydroxide. Potassium, and not sodium hydroxide, is used because the potassium soaps which are formed are more soluble than the corresponding sodium soaps.

The free sulphur represents for the most part sulphur which did not combine with the rubber during vulcanization. Acetone may also remove organic sulphur compounds formed during vulcanization or present as residues or decomposition products of organic accelerators. The effect of large or small amounts of free sulphur on the deterioration of rubber compounds has not been conclusively determined.

The total sulphur represents sulphur which is in chemical combination with the rubber, uncombined or free sulphur, sulphur in mineral compounding ingredients such as barium sulphate, in vulcanized oil substitutes, and in residues of certain organic accelerators. The amount of sulphur in combination with the rubber was formerly considered significant as a measure of the degree of vulcanization. There is no relation between combined sulphur and degree of cure when organic accelerators are used. It was formerly the custom to use 6 or even 8 per cent of sulphur, based on the amount of rubber, to effect vulcanization. With the organic accelerators of vulcanization now universally used, 3 per cent or less of sulphur is sufficient to produce vulcanized rubber of the same or better physical properties.

The ash is the residue after ignition. It consists principally of the nonvolatile mineral compounding ingredients. As the amount of rubber is determined by the difference between 100 per cent and the sum of the percentages of all of the other constituents, it is necessary to know as closely as possible the percentage of mineral matter that was used in compounding. In practically no case is the ash left after burning off the rubber the same in composition and weight as the mineral matter compounded with the rubber. Any sulphur in the ash consists of sulphur from certain mineral compounding ingredients and also part of the sulphur that was combined with the rubber and which during ignition entered into combination with one or another of the mineral compounding materials. The amount of sulphur in the ash, except that present as barium sulphate which is used in compounding and is not changed during the ignition, must therefore be determined and subtracted from the weight of ash found. When a rubber compound contains carbonates or other mineral fillers which would be decomposed and changed in weight when the rubber is burned off, a solvent for vulcanized rubber is used for their determination. Turpentine, terebene, cymene, anisole, phenetole, cresol, aniline, salol, and many other chemical compounds have been suggested as solvents. For the most part these solvents produce solutions which filter slowly or do not bring about a perfect separation of the mineral material and rubber. Kerosene has been used with some success. An effective solvent for vulcanized rubber has been obtained by mixing 300° mineral seal oils from different crude petroleums. The mixed oils are passed through a column of fuller's earth until water white. Vulcanized soft rubber goods are dissolved by this mixture at 150° to 155° C., at which temperature even magnesium carbonate is not decomposed. Two hours or more is usually necessary to dissolve the rubber to a clear solution. When cooled and diluted with benzene and petroleum ether, the solution can be filtered readily from the unchanged mineral matter. When using oil as a solvent the rubber is at first depolymerized and dispersed through the liquid, holding the fine-grained mineral fillers in suspension. While the solution is at this apparently homogeneous, turbid stage it is easy to effect the separation of the rubber and rubber-compounding ingredients from any fabric or fibrous material that may be present. If the heating is continued until the solution becomes almost clear, this separation is not so easy nor so complete. The solvent method is of great value in the analysis of asbestos rubber packings and rubberized fabrics as well as of insulation and other compounds which may contain decomposable fillers.

The specific gravities of the various constituents of vulcanized rubber differ greatly. The percentages of the nonrubber constituents found by analysis are usually expressed by weight, but the amount of rubber is reported by volume. If two compounds contain the same percentage of rubber by weight, but one is made with denser fillers than the other, the compounds will not have the same specific gravity. It will be apparent that the compound of the higher specific gravity contains a greater percentage of rubber by volume. The present tendency in writing specifications is to state the minimum permissible percentage of rubber by volume, because experience and laboratory tests show that the quality of the product depends upon this rather than upon the percentage by weight.

Special methods have been devised for the determination of carbon black, glue, cellulose, antimony trisulphide, and barium carbonate.

It has been stated above that sulphur may be present in mineral compounding ingredients. There can be no objection to such sulphur provided that the mineral containing it has no injurious effect on the rubber. Barytes is such a substance and is permitted in practically all compounds. Barium carbonate is sometimes used and must be taken into account when correcting for sulphur as barytes, if mineral compounding ingredients are determined by the ignition method. The amount of barium carbonate can be determined readily in fillers isolated by the rubber solvent described above.

The determination of the amount of rubber present in a vulcanized compound is an important though difficult matter. If a convenient and accurate method for the direct determination of rubber were available, it would greatly lessen the burden that now falls upon the rubber analyst.

VI. APPENDIX

1. STANDARD PROCEDURE FOR TESTS AND ANALYSES

A standard procedure for the physical testing and chemical analysis of rubber has been adopted by the Federal Specifications Board for the use of the Government departments. This procedure, which is based on the work of the Bureau of Standards, has been promulgated as United States Government general specifications No. 59 for rubber goods (Methods of Physical Tests and Chemical Analysis). It has been published as Circular No. 232 of the Bureau of Standards.

A standard procedure for physical and chemical tests of textile materials has been promulgated by the Federal Specifications Board as United States Government general specifications No. 345 for textile materials (Methods of Physical and Chemical Tests). It has been published as Circular No. 293 of the Bureau of Standards.

2. SPECIFICATIONS FOR RUBBER GOODS PROMULGATED BY THE FEDERAL SPECIFICATIONS BOARD

The following specifications have been officially adopted and promulgated by the Federal Specifications Board as United States Government master specifications for the mandatory use of all departments and independent establishments of the Government.

Specifications bearing only a Federal Specifications Board number are in mimeograph form and can be obtained upon request from the Federal Specifications Board, Bureau of Standards, Washington, D. C.

Specifications bearing Bureau of Standards circular numbers or other bureau publication numbers in addition to the Federal Specifications Board number are in printed form and must be purchased from the Superintendent of Documents. In requesting specifications from the Superintendent of Documents the bureau publication number must be stated.

Do not send purchase orders to Bureau of Standards or Federal Specifications Board. *Address the request, accompanied by cash or money order, to the Superintendent of Documents, Government Printing Office, Washington, D. C.*

The price of each printed specification is 5 cents per copy, unless otherwise noted. Postage stamps will not be accepted in payment for Government publications.

Commodity	Federal Specifications Board No.	Bureau of Standards Circular No.
Aprons, surgeons' rubber	215a	C247
Bags, Politzer	218	C222
Bandages, rubber	219	C219
Bands, rubber	64a	C284
Bottle, hot-water, cloth-inserted	220a	C249
Bottles, hot-water, rubber	221a	C248
Catheters, rubber	222	C225
Cement, rubber, for medical rubber goods	223	--------
Cots, rubber finger	224	C226
Cushions, ring, cloth-inserted	226a	C254
Cushions, ring, rubber	227a	C246
Dam, rubber	228	C218
Dredging sleeves	42	--------
Gaskets, asbestos-copper, corrugated	93	--------
Gaskets, asbestos metallic cloth	97b	C242
Gloves, rubber, for electrical workers (for use in connection with apparatus or circuits not exceeding 3,000 volts to ground)	312	C271
Gloves, rubber, surgeons'	229	C217
Hose, air-brake and signal, and gaskets	43	--------
Hose, chemical	47	--------
Hose, divers'	44b	C289
Hose, fire, cotton rubber-lined (couplings and gaskets)	38b	C114
Hose, gas	40b	C290
Hose, gasoline, rubber-metal	136a	C269
Hose, oil suction and discharge	63b	C209
Hose, pneumatic (*a*) hose, rock drill; (*b*) hose, pneumatic tool	41b	C307
Hose, spray	45	--------
Hose, steam	49b	C268
Hose, tender (corrugated)	46c	C288
Hose, water suction (smooth bore)	50a	C292
Hose, water and wash deck	48b	C291
Ice bag, helmet shaped	216	C228
Ice bag, rubber	217	C227
Matting, rubber	382	C306
Matting, rubber, for use around electrical apparatus or circuits not exceeding 3,000 volts to ground	414	C312
Packing, asbestos high-pressure rod	103a	C238
Packing, asbestos metallic cloth sheet	94b	C243
Packing, asbestos sheet, compressed	96b	C241
Packing, asbestos valve stem	95	--------
Packing, diaphragm	98a	C240
Packing, fabric condenser tube	99	--------
Packing, fiber, for lubricating and fuel oil	100	--------
Packing, flax	101b	C239
Packing, low-pressure spiral gland	104a	C237
Packing, metallic (fixed ring type)	105	--------
Packing, metallic, flexible	102	--------
Packing, metallic, plastic	106	--------
Packing, semimetallic	107	--------
Packing, metallic, solid (floating ring type)	108	--------
Packing rings for boiler blow-off valves	109	--------
Packing, rubber, cloth insertion	110a	C236
Packing, rubber, wire insertion	112a	C234
Packing, rubber, and gaskets (molded, sheet, and strip)	111a	C235
Packing, Tucks	113a	C233
Pads, surgical operating	230a	C245
Pillowcases, rubber	231	C224
Pillows, air, rubber	232a	C252
Rings, rubber jar	51	--------
Rubber-covered wires and cables for ordinary purposes	65	--------
Rubber goods, general specifications for (methods of physical tests and chemical analyses) (price, 10 cents))	59a	C232
Sheeting, rubber	233a	C253
Stoppers, rubber	383	C308
Syringe, cloth-inserted, fountain	234a	C250
Syringe, rubber, fountain	235a	C251
Tape, friction	291	C229
Tape, rubber insulating	292	C230
Tips for crutches, rubber	225	C223
Tires, pneumatic, solid and inner tubes	3c	C115
Tube, colon	236	C221
Tube, stomach or lavage	237	C220
Tubing, rubber	39b	C305
Valves, rubber	114a	C244

3. PUBLICATIONS OF THE BUREAU OF STANDARDS RELATING TO RUBBER

Most of the publications can be bought from the Superintendent of Documents, Government Printing Office, Washington, D. C. Some of them are out of print. In each of the larger cities are one or more libraries to which bureau publications are sent when issued.

Scientific Paper No. 174, The Determination of Total Sulphur in India Rubber, by C. E. Waters and J. B. Tuttle. Also in J. Ind. Eng. Chem., **3**, p. 734; 1911.

Technologic Paper No. 35, Combustion Method for the Direct Determination of Rubber, by L. G. Wesson.

Technologic Paper No. 45, A Study of Some Recent Methods for the Determination of Total Sulphur in Rubber, by J. B. Tuttle and A. Isaacs. Also in J. Wash. Acad., **5**, pp. 235–236; 1915; J. Ind. Eng. Chem., **7**, pp. 658–663; 1915.

Technologic Paper No. 64, Determination of Barium Carbonate and Barium Sulphate in Vulcanized Rubber Goods, J. B. Tuttle. Also in J. Ind. Eng. Chem., **8**, pp. 324–326; 1916.

Technologic Paper No. 133, Tests of Flexible Gas Tubing, R. S. McBride and Walter M. Berry.

Technologic Paper No. 136, Determination of Free Carbon in Rubber Goods, A. H. Smith and S. W. Epstein. Also in J. Ind. Eng. Chem., **11**, pp. 33–36; 1919.

Technologic Paper No. 145, Direct Determination of India Rubber by the Nitrosite Method, J. B. Tuttle and Louis Yurow. Also in India Rubber World, **57**, pp. 17–18; 1917.

Technologic Paper No. 154, Determination of Cellulose in Rubber Goods, S. W. Epstein and R. L. Moore. Also in Rubber Age and Tire News, **6**, pp. 289–293; 1920.

Technologic Paper 162, Extraction of Rubber Goods, S. W. Epstein and B. L. Gonyo. Also in Rubber Age and Tire News, **6**, pp. 445–447; 1920.

Technologic Paper 213, Power Losses in Automobile Tires, by W. L. Holt and P. L. Wormeley.

Technologic Paper No. 240, Dynamometer Tests of Automobile Tires, by W. L. Holt and P. L. Wormeley.

Technologic Paper No. 283, Effect of Tire Resistance on Fuel Consumption, by W. L. Holt and P. L. Wormeley.

Technologic Paper No. 299, Dielectric Constant, Power Factor, and Resistivity of Rubber and Gutta-Percha, by H. L. Curtis and A. T. McPherson.

Technologic Paper No. 294, Wearing Quality of Tire Treads as Influenced by Reclaimed Rubber, by W. L. Holt and P. L. Wormeley.

Technologic Paper No. 318, Endurance Tests of Tires, by W. L. Holt and P. L. Wormeley.

Effect of Heat Generated during Stressing upon the Tensile Properties of Rubber, by C. E. Boone and J. R. Newman, Ind. and Eng. Chem., **18**, No. 5, p. 539; May, 1926.

The Sampling of Rubber Goods, J. B. Tuttle, J. Ind. Eng. Chem., **5**, p. 618; 1913.

An improved Extraction Apparatus, T. B. Ford, J. Am. Chem. Soc., **34**, pp. 552–553; 1912.

Detection of Glue in Rubber Goods, S. W. Epstein and W. E. Lange, India Rubber World, **61**, pp. 216–217; 1920.

Determination of Antimony in Rubber Goods, S. Collier, M. Levin, and J. A. Scherrer, Rubber Age and Tire News, **8**, pp. 104–105; 1920; India Rubber J., **64**, p. 580; 1921; Rubber Eng. Production, **2**, pp. 6–13; 1921.

An Improved Method for the Determination of Total Sulphur in Rubber Goods, Rubber Age and Tire News, **9**, pp. 47–48; 1921.

Determination of Rubber and Inorganic Materials in Soft Rubber Goods, by R. T. Mease and N. P. Hanna, Ind. and Eng. Chem., **17**, pp. 2, 161; 1925.

Circular No. 320, Puncture Sealing Compounds for Pneumatic Tires.

The work of the Bureau of Standards on rubber, C. E. Waters, J. Chem. Education, **3**, pp. 291–295; 1926.

Circular No. 327, Selection and Care of Garden Hose.

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Rubber, Its Production, Chemistry, and Synthesis, A. C. Dubosc and A. Luttringer; Charles Griffin & Co. (Ltd.), London; 1918.

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Rubber Machinery, H. C. Pearson; India Rubber World Pub. Co., New York; 1920.

Pneumatic Tires, H. C. Pearson; India Rubber World Pub. Co., New York; 1922.

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5. PERIODICALS

Devoted solely to the interests of the rubber industry.

India Rubber World.
The Rubber Age.
India Rubber Journal.
Bulletin of the Rubber Growers' Association.
Gummi-Zeitung.
Le Caoutchouc et la Gutta-Percha.
Kautschuk.
India Rubber Review.
Archief voor de Rubbercultuur.
Revue Générale du Caoutchouc.
Transactions of the Institution of Rubber Industry.

Having abstracts or occasional articles on rubber.

Industrial and Engineering Chemistry.
Chemical Abstracts.
Journal of the Society of Chemical Industry.
The Analyst.
Kolloid Zeitschrift.
Automotive Industries.
Journal of the Society of Automotive Engineers.
Journal of the American Chemical Society.
Malayan Agricultural Journal.

WASHINGTON, May 2, 1927.