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UNITED STATES PATENT OFFICE.

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METHOD OF MOLDING EBONITE COMPOSITION.

No Drawing. Application filed June 14, 1923. Serial No. 645,367.

This invention relates to ebonite or hard rubber compositions and to products made therefrom.

The general object of my invention is to provide an ebonite having high resistance to change of shape under the influence of heat.

A further object of my invention is to provide an ebonite having high resistance to acids, chlorine and the like.

A more specific object is to provide an ebonite mix which may be set-cured in a mold in a very short time so as to hold its shape during further factory processing thereby increasing the output of a given equipment of molds and presses.

A still further object is to provide an improved ebonite mix for vulcanization without form-altering mold pressure.

As a basis for comparing the results obtained by my invention I apply the following test:

An ebonite test piece (½ in. by 3 in. by the thickness of the sheet from which it is cut) is mounted on knife edges 2 in. apart. A third knife edge carrying a load is suspended from the test piece midway between the supporting knife edges. This apparatus is inclosed in an oven whose temperature is gradually raised at the rate of about 1.5° C. per minute to a temperature of 70° C. and thereafter at the rate of 0.2° C. per minute. Deflections of the test piece are noted through a reading telescope by means of an indicator on the telescope frame and a scale carried by the knife edge holding the load. The temperature is raised until the deflection amounts to about 0.5 in. Approximate uniformity in thickness of the test pieces is necessary to avoid errors due to lag of temperature rise in the sample. In order that test pieces of slightly different thicknesses may be tested under the same conditions of stress, the load is adjusted to the cross-sectional area to give a fibre stress of 2500 lbs. per square inch. This load is determined from a curve plotted from the equation:

$$S=\frac{3PL}{2bd^2}$$

where

S = unit stress.
P = load in lbs.
L = length of span in inches.
b = width of test piece in inches.
d = thickness of test piece in inches.

From these observations, a temperature-deflection curve is plotted. This curve shows three distinct regions. 1st, a nearly rectilinear portion; 2nd, a region of rather sharp curvature; and 3rd, a second substantially rectilinear portion where excessive flow takes place. A tangent drawn to the curve in the third region approximately coincides with the last mentioned substantially rectilinear portion and intersects the axis of temperature in the region where yielding becomes substantial, and the point of intersection is taken as the softening point of the ebonite under test.

By this test I find that the commercial ebonites now available have softening points ranging only up to about 155° F. This property of softening at low temperatures causes the effect known as cold flow and offers a serious objection to the use of ebonite in connection with instruments or apparatus where precision is required. This objection is even more pronounced where the ebonite becomes warm during service or where it is subjected to a strain.

I now find that ebonites may be made with softening points ranging, in the test described, as high as 205 ° F., and the products made therefrom possess many distinct advantages over the present commercial articles. These advantages are obtained by incorporating large amounts of pigment, preferably inert pigment, in the rubber mix prior to vulcanization.

Unvulcanized ebonite mixes such as

heretofore used contain rubber, sulphur, organic accelerator and/or inorganic accelerator and, so far as I am aware, an additional amount of pigment not exceeding 25 volumes for each 100 volumes of rubber.

In my ebonite compositions I use from about 35 volumes to as high as 65 volumes of pigment for each 100 volumes of rubber, whereby I obtain, upon vulcanization of the mix, ebonites having very high softening points and better adapted for uses where the ebonite is subject to strain or heat. By the term "volume" as employed in the specification and claims of this application is meant the true or absolute volume as determined in a volumeter or more conveniently by weight using the equation $V=\frac{W}{D}$, where V = volume in cubic centimeters, W = weight in grams, D = specific gravity.

The effect of high amounts of pigment in the softening point of an ebonite is shown by the following examples.

A standard ebonite mix comprising, in parts by weight, rubber—100, sulphur—40 and organic accelerator—2 is mixed with varying amounts of pigment as shown. In the presence of the high amounts of pigment indicated, partial vulcanization may be effected in a mold in 2 minutes at 346° F. to produce a vulcanizate so set or stiffened that it will hold its shape without change while the vulcanization is completed by heating for four hours at 292° F. in open heat. This very brief period of set cure in the mold may be aptly described as "momentary" vulcanization.

Pigment	Volumes per 100 vol. rubber	Softening point of vulcanizate
		° F.
Clay	65	203
	50	192
	30	169
Asbestos	65	191
Powder	50	189
Whiting	65	189
	50	190
Gas black	50	200
	30	177

The above described advantage of quick set in the use of a high amount of pigment in an ebonite is clearly shown by reducing the pigments in the above mixes to the usual 5 to 20 volumes, whereupon the mold cures are found to be soft and full of blow holes after a two minute cure at 346° F., the product being entirely unsuitable for further vulcanization in open heat.

I find the use of clay as the inert pigment especially advantageous not only because of its low cost but because of the very high softening point which results from its use.

A further advantage of my invention is that highly pigmented ebonite mixes show little shrinkage during vulcanization, which is of importance in the manufacture of articles of accurate design.

Another advantage, when the pigments themselves are inert in the presence of sulphuric acid, chlorine and the like, is the increased resistance of the ebonite to these reagents.

My improved ebonite mixes also have the advantage that when articles are molded therefrom without vulcanizing heat and then vulcanized without form-altering or form-maintaining mold pressure they hold, to a high degree, their size, shape or design.

I do not wholly limit myself to the use of the specific pigments nor to the proportions specified above since I find that other pigments such an antimony sulfides lithopone, lime, zinc oxide and the like are more or less serviceable for my purpose when used in large amounts. It is also obvious that a mixture of materials other than sulphur, accelerator and organic fillers may be used to make up the total volume of pigment as may be required by such limitation as availability, cost, specific gravity or color.

I claim:

1. The method of producing ebonite which comprises mixing rubber, a vulcanizing agent and sufficient pigment substantially to stiffen the mix after a momentary vulcanization, partially vulcanizing the mix in a mold, removing it from the mold and thereafter subjecting it to further vulcanization.

2. The method of producing ebonite which comprises mixing rubber, a vulcanizing agent and substantially 35 to 65 parts by volume of inert pigment to each 100 parts by volume of rubber, partially vulcanizing the mix in a mold, removing it from the mold and thereafter subjecting it to further vulcanization.

3. The method of producing ebonite which comprises mixing rubber, a vulcanizing agent and substantially 35 to 65 parts by volume of clay to each 100 parts of volume of rubber forming and momentarily setting the mix in a mold, removing it from the mold and thereafter subjecting it to further vulcanization.

4. The method of making hard rubber articles which comprises providing a quick-setting hard rubber compound, heating the compound in a confining mold until it has received a preliminary set, removing the compound from the confining mold while the compound is still in a partly cured condition, and thereafter completing the vulcanization without any confining mold.

5. The method of making articles of ebonite which comprises providing a quick-setting hard rubber compound, subjecting the compound in a mold to heat and pressure until it has received a preliminary set, re-

moving the compound from the mold while it is still in a partly vulcanized condition, and thereafter completing the vulcanization in open heat.

6. The method of making hard rubber articles, which consists in providing a quick setting hard rubber compound, pressing the same under pressure in a heated mold until the same has received a preliminary set, removing the same from the mold while the same is still in a partly cured condition, and placing upon supporting means for vulcanizing.

7. The method of making hard rubber articles, which consists in providing a quick setting hard rubber compound, pressing the same under high pressure in a heated mold until the same has received a preliminary set, removing the same from the mold while the same is still in a partly cured condition, and placing the same upon supporting means for vulcanizing.

8. The method of making hard rubber articles which consists in providing a hard rubber compound with an accelerator, said accelerator being sufficient in amount to give a preliminary set to the compound when subjected to high pressure in a heated mold, so that the molded compound can be removed from the mold in a partly cured condition to be placed upon supporting means for vulcanizing.

9. The method of making hard rubber articles, which consists in providing a hard rubber compound with an accelerator, said accelerator being sufficient in amount to give a preliminary set to the compound when subjected to high pressure in a heated mold, so that the molded compound can be removed from the mold in a partly cured condition in approximately one minute, to be placed upon supporting means for vulcanizing.

10. The method of making hard rubber articles, which consists in providing a hard compound with an accelerator, said accelerator being sufficient in amount to give a preliminary set to the compound, in a short period of time, when subjected to high pressure in a heated mold, so that the molded compound can be removed from the mold in a partly cured condition in a short period of time to be placed upon supporting means for vulcanizing.

11. The method of making hard rubber articles, which consists in providing a hard rubber compound with an accelerator, said accelerator being sufficient in amount to give a preliminary set to the compound, in approximately one minute, when subjected to high pressure in a heated mold, so that the molded compound can be removed from the mold in a partly cured condition, in approximately one minute to be placed upon supporting means for vulcanizing.

In witness whereof I have hereunto set my hand this 13th day of June, 1923.

HAROLD GRAY.

CERTIFICATE OF CORRECTION.

Patent No. 1,691,347. Granted November 13, 1928, to

HAROLD GRAY.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, line 117, strike out the period and insert a comma; line 23, strike out the period and insert a colon; line 84, after the word "sulfides" insert a comma; page 3, line 24, claim 8, before the word "which" insert a comma, and line 44, claim 10, after the word "hard" insert the word "rubber"; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 9th day of April, A. D. 1929.

(Seal)

M. J. Moore,
Acting Commissioner of Patents.

moving the compound from the mold while it is still in a partly vulcanized condition, and thereafter completing the vulcanization in open heat.

6. The method of making hard rubber articles, which consists in providing a quick setting hard rubber compound, pressing the same under pressure in a heated mold until the same has received a preliminary set, removing the same from the mold while the same is still in a partly cured condition, and placing upon supporting means for vulcanizing.

7. The method of making hard rubber articles, which consists in providing a quick setting hard rubber compound, pressing the same under high pressure in a heated mold until the same has received a preliminary set, removing the same from the mold while the same is still in a partly cured condition, and placing the same upon supporting means for vulcanizing.

8. The method of making hard rubber articles which consists in providing a hard rubber compound with an accelerator, said accelerator being sufficient in amount to give a preliminary set to the compound when subjected to high pressure in a heated mold, so that the molded compound can be removed from the mold in a partly cured condition to be placed upon supporting means for vulcanizing.

9. The method of making hard rubber articles, which consists in providing a hard rubber compound with an accelerator, said accelerator being sufficient in amount to give a preliminary set to the compound when subjected to high pressure in a heated mold, so that the molded compound can be removed from the mold in a partly cured condition in approximately one minute, to be placed upon supporting means for vulcanizing.

10. The method of making hard rubber articles, which consists in providing a hard compound with an accelerator, said accelerator being sufficient in amount to give a preliminary set to the compound, in a short period of time, when subjected to high pressure in a heated mold, so that the molded compound can be removed from the mold in a partly cured condition in a short period of time to be placed upon supporting means for vulcanizing.

11. The method of making hard rubber articles, which consists in providing a hard rubber compound with an accelerator, said accelerator being sufficient in amount to give a preliminary set to the compound, in approximately one minute, when subjected to high pressure in a heated mold, so that the molded compound can be removed from the mold in a partly cured condition, in approximately one minute to be placed upon supporting means for vulcanizing.

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