Dec. 26, 1961 R. W. SENIFF ETAL 3,014,865

MOLYBDENUM DISULFIDE LUBRICATING COMPOSITION AND METHOD

Filed Feb. 4, 1954

FIG. 1

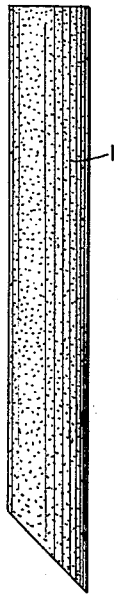
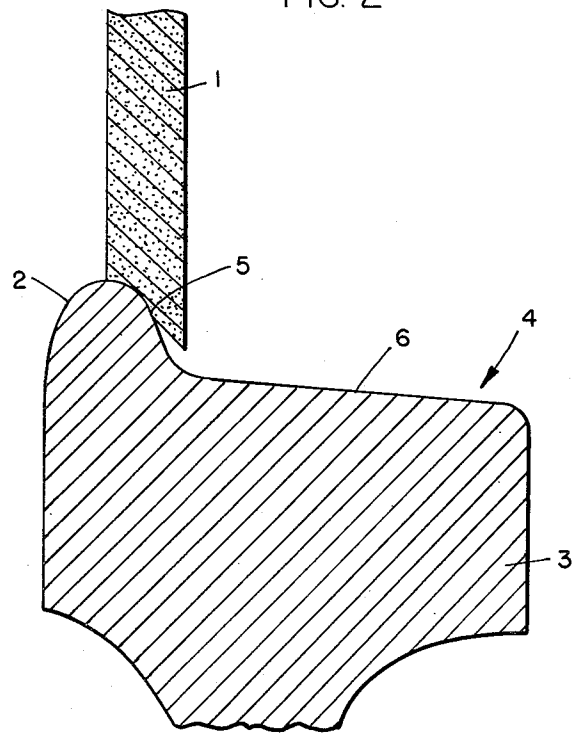


FIG. 2



INVENTORS:
RUSSELL WADE SENIFF
ROLAND NICHOLAS FRACALOSSI

BY Margaret, Johnston, Cook & Root

ATT'YS

United States Patent Office 3,014,865 Patented Dec. 26, 1961

3,014,865
MOLYBDENUM DISULFIDE LUBRICATING COMPOSITION AND METHOD

Russell Wade Seniff, 4319 Barrington Road, and Roland Nicholas Fracalossi, 1508 Ramblewood Road, both of Baltimore, Md.

Filed Feb. 4, 1954, Ser. No. 408,173
2 Claims. (Cl. 252—12)

This invention relates to a lubricating composition and method of preparation and to the lubrication of moving parts. More particularly, the invention relates to a compact lubricating composition containing molybdenum disulfide as the lubricant. The invention finds important application in the lubrication of railway locomotive wheel flanges and the like.

Due to the nature of certain railway operations, i.e., heavy freight operation on curved trackage or switcher service in yards and shops, wheel flanges of cars and locomotives and the rails of the track show high rates of wear. this gives rise to what is called "sharp flanges" on wheels. This condition condemns a wheel for further railway use and, if of wide incidence, naturally involves high maintenance and replacement costs. The wear on the rail necessitates costly rail renewals. The extremely high pressures encountered by wheel flanges against the rails under these conditions call for a lubricant with matching extreme pressure characteristics.

In addition to this, the dust conditions commonly found along railroads favor the use of a dry film lubricant to avoid contamination of the lubricating film and lubricated surfaces by extraneous abrasives inherent in the conditions under which wheel flanges are required to operate. These abrasives include silica sand which is used under the locomotive's wheels to prevent slip of wheels on the rails during periods of heavy traction effort and when braking. They also include extremely hard and abrasive cast iron and steel particles worn from the brake shoes, wheels and rail. These particles—because they are produced by heat resulting from friction—are so hard that they will scratch glass. They cut and wear steel like abrasive or grinding compounds. These abrasive dusts adhere to the conventional lubricating oils commonly used for railroad wheel flange and rail lubrication, and the mixture acts as a grinding compound similar in action to those used for grinding valves of internal combustion engines. It is known that an abrasive will have a much more active grinding or cutting action if mixed with an oil or similar liquid than if it is used as a dry powder. Observations of wheel flanges in actual service indicate that where abrasive dusts are present in relatively high volume, wear occurs at a more rapid rate if the flange is oiled than if no oil is present. A dry lubricant does not collect and retain the abrasives and is, therefore, superior to a "wet" one.

Molybdenum disulfide has demonstrated its superior film-strength characteristics under high pressures and its significant tendency to bind tightly to a metal surface upon which it is applied. Both rubbing and high pressure are conducive to forming an even tighter bond since, in this manner, the molybdenum disulfide molecules are more properly oriented in the thin boundary film. This orientation has been shown to increase the metal-lubricant bond.

It is therefore an object of the present invention to utilize the advantageous properties of molybdenum disulfide in the provision of a dry lubricating composition well-suited for application to moving parts and furnishing considerable improvements over prior compositions.

Another object is to provide a compact lubricating composition containing molybdenum disulfide as the lubricant, desirably in the form of a lubricating element.

A specific object is to provide a lubricating composition

in solid stick form for use in lubricating a moving metal surface, particularly a wheel flange such as a railway car or vehicle wheel flange or the like.

Additional objects include the provision of a compact lubricating composition formed of molybdenum disulfide and a binder, satisfying the following requirements:

(1) The composition should be sufficiently hard to furnish a long lasting stick from which only the necessary amount of molybdenum disulfide will rub off. It should be possible to provide a range of hardness to furnish optimum results under different conditions of use.

(2) The binder should not be abrasive or corrosive in the composition.

(3) The composition must not be too brittle, as the molybdenum disulfide lubricant should be removed only in a finely divided state. At the same time, it must be tough enough to withstand the normal surface conditions.

(4) The bidder should not be softened or melted by the heat generated when contacting a moving surface.

(5) The binder should be economical and adapted for combining with the lubricant in a simple and economical process.

A further object of the invention is to provide a method of preparing a lubricating composition having the described characteristics, advantageously in the form of a lubricating element.

A still further object is to provide a method of lubricating a moving surface, particularly a metal wheel flange as employed on metal wheels traveling on metal rails.

These and other objects and advantages of the invntion will become apparent on reference to the following description taken in conjunction with the attached drawing, illustrating a preferred embodiment of the invention, in which

FIGURE 1 is an elevation of a lubricating element according to the invention, and

FIGURE 2 is a fragmentary cross-section of the lubricating element in contact with a railway car wheel flange.

In accordance with the invention, a lubricating composition is provided which consists essentially of molybdenum disulfide and a cured heat-resistant thermosetting phenolic resin. The composition is provided in the form of a compact mixture for use as a dry lubricant. It is desirable to maintain the highest possible proportion of molybdenum disulfide for lubricating purposes, and accordingly, a major proportion. preferably at least about 60% by weight, of the disulfide is preferably provided. A minor binding proportion of the resin is used, and the resin is preferably derived from a mixture of a phenolic casting resin and a curing phenolic molding resin Molybdenum disulfide and the resin constitute the essential ingredients or components, that is, the composition includes no other ingredients materially affecting the basic and novel characteristics of the composition.

The lubricating element of the invention consists essentially of a mixture of molybdenum disulfide and a minor binding proportion of a cured heat-resistant thermosetting phenolic resin in solid stick form. It is particularly well suited for contacting or bearing on moving surfaces, for example, wheel flanges, such as railway car or vehicle wheel flanges, especially locomotive wheel flanges, or the like, which are very difficult to maintain. Such an element is illustrated in FIGURE 1, in which a cylindrical lubricating element, rod or stick 1 of the foregoing composition is represented. One end of the stick is bevelled to approximately 45°, to facilitate proper contact of the element with a wheel flange, for uniform lubrication.

In the practice of the invention, a method is provided for preparing the composition which comprises intimately mixing molybdenum disulfide and a curing phenolic resin, the resin preferably being fluid so that the mixture may

be poured, and curing the mixture. The resin preferably consists of a liquid phenolic casting resin and a powdered curing phenolic molding resin in proportions that provide a fluid mixture with the molybdenum disulfide. It is further preferred to mix the disulfide and a casting resin, then mix the resulting mixture with a molding resin, agitate the second mixture to separate the water therefrom, remove the separated water and cure the resulting mixture. "Casting" as used herein refers to the process which involves pouring a potentially heat-convertible liquid resin into a mold and then hardening it, e.g., by heat. A casting resin is one which can be cast. "Curing" refers to the conversion of a fusible resin to its infusible state, such as by heating or catalytic action. A curing resin is one which can be cured.

The invention also furnishes a method of lubricating a moving surface which involves contacting the surface with the above lubricant composition, or causing the composition or element to bear on the surface. The method is illustrated in FIGURE 2 of the drawing, wherein the element or stick **1** is in position to lubricate the flange **2** on the rim **3** of a railway car or vehicle wheel **4**. The lubricating element is supported in a suitable holder, not shown, attached to a car member, so that the element contacts the flange **2** with the pressure desired for causing the molybdenum disulfide to rub off of the element and form a suitable coat on the flange. The wheel **4** illustrated is new. In operation, the flange **2** becomes worn on its inner side **5**, and the portion of the wheel tread **6** adjacent the flange becomes worn. It then becomes necessary to turn down or grind the flange and the portion of the tread distant from the flange, so that this portion again forms a continuous straight line with the portion adjacent the flange. Eventually, the wheels must be renewed. Treatment with the stick lubricant has been found to be very effective in reducing the foregoing wear and thus the costs of flanged driving wheels in diesel and steam locomotives.

While pure molybdenum disulfide would of course furnish the best results, it is not necessary that it be entirely pure, but small amounts of impurities are permissible without materially affecting the properties of the composition. Thus, a relatively economical commercial molybdenum disulfide product having the following typical analysis may be employed satisfactorily:

	Percent
MoS_2	90–94
Mineral oil	3–5
Water	3–5
Gangue (mainly SiO_2 and FeS_2)	About 0.7

Finely divided molybdenum disulfide is most desirable. The following representative particle size distribution in the disulfide has been found to be very satisfactory:

		Weight percent
	+200 mesh	5
−200 mesh	+400 mesh	24
−400 mesh	+22 microns	52
−22 microns	+11 microns	12
−11 microns	+5 microns	5
	−5 microns	2

The binder in the composition is preferably a cured heat-resistant thermosetting phenolic resin, preferably obtained by heating a curing phenolic resin to harden it. The curing is effected after mixing with the disulfide. These resins have the advantageous properties of good binding, good mechanical strength and durability, controllable hardness, low friction coefficient and low cost. The phenol-aldehyde resins are preferred, for example, phenol-formaldehyde and phenol-furfural resins, especially the phenol-formaldehyde resins.

While the resin may be in powdered form initially and the lubricant sticks formed by pressure molding, it is much more advantageous to employ a resin in fluid form that can be poured and subsequently cured to form a solid body. The initial resin is most desirably a mixture of liquid and powdered forms. The liquid phenolic casting resins, also referred to as one-stage resins, are prepared in known ways, for example by reacting phenol and excess formaldehyde (i.e., a molar ratio of formaldehyde to phenol greater than 1:1) in the presence of an alkaline catalyst and at a moderately elevated temperature, e.g., at about 90–100° C., as described on pages 12, 13, 18 and 19 of "Experimental Plastics and Synthetic Resins," G. F. D'Alelio (John Wiley & Sons, Inc., N.Y., 1948). They are desirably employed in organic solvent solution, for example, in alcohol or toluene solution. The powdered curing phenolic molding resins, also referred to as two-stage resins, are prepared in known ways, for example, by reacting excess phenol and formaldehyde (i.e., a molar ratio of phenol to formaldehyde more than 1:1) in the presence of an acid catalyst, the reaction proceeding exothermically followed by refluxing, and then treating with hexamethylenetetramine to supply additional formaldeyhde, as described on pages 24 and 26 of the above reference. Both types of resins are believed to polymerize into similar thermosetting end products on curing.

Commercially available resins of these types are the following "Durite" phenol-aldehyde resins (Durite Products Department, The Borden Company, Chemical Division, Philadelphia, Pa.) having the properties indicated:

"Durite" AL–5390 liquid phenolic casting resin:	
Specific viscosity, cp	300–650
Water tolerance (pts. of water to pt. resin)	1.5–4.5
Hot plate cure, sec	100–140
Percent solids content	72
pH	7.70
Specific gravity	1.20
Ash, percent	0.4

"Durite" AD–5094 powdered curing phenolic molding resin:	
Cure time (310° F. hot plate test), sec	55–70
Flow (257° F. glass plate test), mm	20–30
Sieve analysis—	
Max. on 100 mesh	trace
Min. thru 270 mesh, percent	95
Melting point (capillary tube), ° F	169±5
Specific gravity	1.261
Nitrogen content, percent	3.6

The proportions of the lubricating composition ingredients vary with the particular resin or resins used. A major proportion, preferably at least about 60% by weight of molybdenum disulfide (pure basis) is included together with sufficient resin to bind the disulfide. Preferably, liquid and powdered resins are supplied in proportions that will provide a fluid mixture which will flow well enough to be poured, blown, or sucked into a mold. Since it is desired to have a maximum disulfide content, the preferred proportions provide a very viscous mixture.

When employing the Durite resins described above, the liquid resin may be mixed alone with the disulfide, about 35–40% by weight of the liquid resin preferably being mixed with about 65–60% of the impure disulfide product previously described. It is preferred to mix both liquid and powdered resins with disulfide. At least about 55% of the impure disulfide product is then mixed with about 20–40% of the liquid resin and, correspondingly, about 15–5% of the powdered resin. Greater amounts of liquid resin and lesser amounts of powdered resin are used when the liquid resin has thickened, as occurs on aging. It will be understood that the relative proportions of the essential ingredients will be different in the cured composition, due to the extraneous substances present in the starting materials. As previously set forth, the cured composition should contain at least about 60% of the disulfide, on a pure basis.

It has been found that a lubricating stick best suited for the intended purpose results from the use of an amount

of the powdered resin above a minimum of about 15% by weight based on the solids content of the liquid resin. A maximum amount of powdered resin of about 125% on the solids content of the liquid resin is preferred, since powdered resin increases the volume considerably and the highest possible volume of disulfide is sought. At the same time, the liquid resin is employed in an amount sufficient to furnish a fluid mixture. While liquid resin alone can be used, the use of as much powdered resin as possible has been found to reduce the porosity of the product and settling of the disulfide and to increase the yield of satisfactory elements while considerably reducing the curing time. Other phenolic resins may be used as binders to provide the intended results and in accordance with the above principles.

The lubricating composition is prepared in a preferred method by incrementally adding the disulfide to a liquid phenolic casting resin while agitating, continuing agitation until the mixture is smooth and lump-free, incrementally adding a powdered curing phenolic molding resin to the mixture while agitating, agitating until the second mixture is smooth, agitating to separate water and removing the water, and curing the mixture by heating at moderately elevated or superatmospheric temperatures, for example, in the range of about 150° F. to 250° F., over an extended period of time. A lower temperature within the range, e.g., 150–170° F., is maintained for a major portion of the heating period, and then progressively higher temperatures are maintained for relatively short periods of time. It is important not to attempt to cure too rapidly, for the water must be removed slowly during the initial stages. Lubricating elements or sticks of greater hardness are produced at either higher temperatures or extended curing times; conversely, hardness is reduced by lowering the temperatures or curing times. The cured elements are cooled slowly to atmospheric temperature, at which time they are ready for use.

The following examples are furnished to assist in providing a complete understanding of the invention, but it is to be understood that the invention is not limited thereto nor to the specific compositions, proportions and procedures set forth therein, which are given only for purposes of illustration.

Example 1

Molybdenum disulfide, having the composition described above, is added to Durite AL–5390 liquid phenolic resin in small increments with constant, slow stirring to prevent the formation of small lumps of disulfide powder. The proportions provide about 60–65% of the disulfide and about 35–40% of Durite in the mixture, the exact amounts being regulated to provide a viscous mixture having sufficient flow to permit pouring or sucking into molds. The final mix has a smooth texture. The disulfide particles are readily wetted by the resin and remain in suspension.

The viscous mix is poured into a glass tube for curing and placed in an oven. The temperature of the oven is raised slowly, over a period of about two hours, to 150° F. The following temperatures (oven) are maintained for the periods of time indicated, and the product is then allowed to cool to room temperature in the oven with the heat turned off:

150° F. for 84 hours
160° F. for 2 hours
170° F. for 2 hours
180° F. for 15 hours
190° F. for 3 hours
210° F. for 3 hours
230° F. for 3 hours
250° F. for 8 hours
Total curing time, 120 hours

Lubricating sticks prepared in this manner may be removed from the tubes by pushing them out, there being a slight contraction of the sticks and no adhesion to the glass. The sticks were found to be sufficiently hard for use in lubricating railroad locomotive wheel flanges but possessed an appreciable porosity which caused some of the sticks to crack during curing, so that the yield amounted to about 90%. There was also some settling of the disulfide, resulting in an accumulation of binder at the top surfaces.

Example 2

Molybdenum disulfide, as described above, is added in small increments to Durite AL–5390, aged one month from the date of manufacture, with constant, slow stirring. Agitation is continued until the mix is smooth and lump-free. Durite AD–5094 powdered phenolic resin is then added in small increments, preferably by light sprinkling of the powder with continuous stirring, and agitation is continued until the mixture has a smooth texture. The proportions of the ingredients in this mixture are 62% of the disulfide, 24% Durite AL–5390, and 14% Durite AD–5094. Agitation is then increased and continued until water bleeds out of the mixture and accumulates on the surface. Stirring is discontinued, and the water is poured off. The mix is quite viscous but capable of being poured.

The mixture is placed in cylindrical glass molds, and curing is started within 12 hours after filling the molds, to avoid excessive settling of the disulfide. The molds are placed in an oven, heated to the starting temperature over a period of about 2 hours, and heated at the following temperatures and for the periods of time indicated:

170° F. for 60 hours
190° F. for 2 hours
210° F. for 2 hours
250° F. for 8 hours
Total curing time, 72 hours

With the oven heat off, the sticks are allowed to cool in the oven to room temperature.

It is possible to extend the curing time at lower temperatures, e.g., 200° F., and produce sticks of similar hardness and strength. Sticks of greater or lesser hardness are produced by heating either at higher or lower temperatures, respectively, or longer or shorter curing times.

Sticks prepared in this manner were found on removal to be only very slightly porous. They were hard yet flexible enough to give slightly under bending stress. A thin film of resin coated the outer surfaces, giving them a clean, smooth and slightly polished appearance. The yield was about 100%, cracking was eliminated, the disulfide was more uniformly distributed, and the overall properties of the sticks were improved over those prepared according to the method of Example 1.

It was also found that the viscous mixture could be charged to and cured in any suitable tube, such as paper, cardboard, aluminum foil, and the like, and allowed to remain in the tube when used. The tube serves as a protective container for shipping and may be labeled on its outer surface.

Example 3

The procedure of Example 2 is repeated except that the proportions of the disulfide and resins are 61% of disulfide, 34% of Durite AL–5390 and 5% of Durite AD–5094, the Durite AL–5390 liquid resin being aged two and one-half months from the date of manufacture. The liquid resin may be used within a period of three months from the date of manufacture. Due to the increase in viscosity resulting from aging, less powdered resin and more liquid resin is used as the liquid resin becomes older to supply a mixture having suitable viscous flow characteristics. There are obtained lubricating elements corresponding in properties and utility to those prepared according to Example 2.

The invention provides a lubricating composition and element eminently suited for dry lubricant applications,

such as in the very troublesome maintenance of wheel flanges. The composition supplies a lubricant superior in its high pressure and binding characteristics and has the necessary hardness, toughness and resistance to heat while being economically manufactured. A very advantageous method is furnished, and there is no necessity for high pressure operations. A moving metal surface is lubricated by contacting the surface with a compact mixture according to the invention, to minimize the problems encountered in heavy duty operations.

The invention is claimed as follows:

1. A car wheel flange lubricating element consisting essentially of a cured mixture of, in percentages by weight, at least about 55% of molybdenum disulfide having a purity of 90–94%, about 20–40% of a liquid one-stage phenol-aldehyde resin having a solids content of 72%, and about 15–5% of a two-stage phenol-aldehyde resin, in solid stick form.

2. The method of preparing a car wheel flange lubricating element which comprises intimately mixing ingredients consisting essentially of, in percentages by weight, at least about 55% of finely divided molybdenum disulfide having a purity of 90–94%, about 20–40% of a liquid one-stage phenol-aldehyde resin having a solids content of 72%, and about 15–5% of a powdered two-stage phenol-aldehyde resin, each of said resins being selected from the group consisting of phenol-formaldehyde and phenol-furfural resins, the proportions of said resins being selected to provide a fluid mixture, separating water from the resulting mixture, and curing the resulting mixture in the form of a solid stick.

References Cited in the file of this patent

UNITED STATES PATENTS

1,054,265	Baekeland	Feb. 25, 1913
2,156,803	Cooper et al.	May 2, 1939
2,361,211	Kalischer	Oct. 24, 1944
2,367,946	Kaercher	Jan. 23, 1945
2,466,642	Larsen	Apr. 5, 1949
2,600,321	Pyle	June 10, 1952
2,686,155	Willis	Aug. 10, 1954