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PROCESSING WITH NONSTAINING COM-
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ration of DelawareNo Drawing. Application December 21, 1939,
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This invention relates to operations in working and forming structural solid articles from fusible materials, such as metals, with the aid of volatile oils. More particularly, it is concerned with the utilization in these operations of improved oil compositions.

In the fabrication of ferrous metals, non-ferrous metals, and other fusible materials, the use of oil compositions as a medium between the surfaces of the working apparatus and the article being worked to produce an improved finish on the product plays an important part. For example, the cold rolling of steel, brass, aluminum, copper, or the like, is benefitted to a great extent by having a film of oil between the rolls and the metal piece being shaped. The oil tends to make the surface of the shaped article smoother, to prevent overheating, and to avoid the picking up of particles by the rolls from the material being worked. Similar functions are performed by the oil in metal drawing and die pressing, and die forging operations. In each case there has been considerable difficulty in the removal of decomposition products of the oils from the processed articles after they have been annealed.

Roll oils, drawing compounds, and die swabbing oils which have been in use up until now have the disadvantage of leaving on the annealed articles dark stains. This stain formation evidently results from a thermal decomposition of oil on the surface of the metal article when it is treated at an annealing temperature even in a non-oxidizing atmosphere, but it does not appear to be a function of the carbon content of the oil, since with conventional oil products approximately the same degree of staining is obtained with a paraffinic as with a naphthenic base oil of the same viscosity. There is evidence that the distillation and heat stability characteristics of a lubricant are important factors in the degree of staining left by what seems to be a cracking and polymerizing of the oil during the annealing process at highly elevated temperatures ranging up to about 1750° F.

Conventional oil products now in use as roll oils have certain minimum viscosity and load bearing requirements which usually cannot be successfully met by lubricating oils of viscosities lower than 55-60 Saybolt Universal seconds at 100° F. for the non-ferrous metal industries and 90-100 Saybolt Universal seconds at 100° F. for ferrous industries. Such oil products have been commonly almost 100% petroleum oils in composition but may, on occasion, contain small percentages of oiliness agents to help prevent stick-

ing of the rolls to the processed metal and to provide smoother metal surfaces.

In the usual production of rolled metal shapes, a piece of tough, pliable metal is passed between a train of cylindrical rolls constructed of chilled iron or steel. The rolls are set one above the other with their axes parallel and held in housings so that there is a fixed spaced between them. The upper and lower rolls are so connected as to rotate in opposite directions and the space between these rolls is adjusted so that the rolls exert a high compression on the metal piece passed between them.

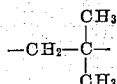
In a drawing operation, a metal bar is compressed and elongated by being drawn through a tapering hole, whereby a smoothly finished product of reduced cross section is formed. To accomplish this operation more efficiently, it is the usual practice to have the piece enter the die coated with a lubricating medium or drawing compound. Many types of compounds have been in use for this purpose, and it has been found that unless the compound gives satisfactory lubrication, the die scratches the work and quickly becomes rough. For this reason, special quality petroleum oils and greases have generally been adopted.

It is an object of this invention to provide non-staining oil compositions to be applied to the surface of the processing apparatus and the material being worked or formed. In a more specific sense, this invention provides oil compositions which have satisfactory viscosity, oiliness, and load bearing characteristics for use in the mechanical forming operations and which leave no objectionable stain on the articles after they have been heated to an elevated temperature in an annealing treatment.

Compositions which are used according to the present invention for a cold working of metals prior to an annealing treatment comprise preferably petroleum distillates of lower boiling range than lubricating oils and a sufficient amount of polybutenes of high molecular weight to confer upon the composition viscosity, consistency, and filming characteristics necessary for any given metal working condition. An oiliness agent such as a fatty ester, a fatty oil, a vegetable oil, or a soap may be added in a minor amount to the composition to give improved oiliness.

The high molecular weight polybutene ingredient is the product obtainable by polymerizing isobutene at a low temperature, for instance below -10° C. and preferably below -40° C. with the aid of a catalyst, such as boron fluoride. In

their commercial production the polybutenes are essentially derived from isobutene, but varying quantities of normal butenes may be present, also, other low molecular weight hydrocarbons which act as diluents. There are a number of variations of this procedure and other iso-olefin reactants, e. g. isopentene, which may be used to form polymers possessing similar physical and chemical properties in general. From their synthesis and analytical determinations, the polymers of iso-olefins are considered to be linear type polymers of a homologous series and be constituted of recurring branched alkene units, viz:



Hence, these polymers may be called appropriately polyalkenes or polyisoalkenes. They have a low iodine number and a hydrogen to carbon ratio of about 2.0, which indicates that on an average no more than one double bond is present in each polymer molecule.

A convenient way of describing high molecular weight compounds of the polyalkene type utilizable in the present invention is to refer to them descriptively as polymeric homologous compounds characterized by structures of long chains of saturated carbon atoms, a number of which are attached to a recurring organic radical by a carbon to carbon bond. Polymeric homologous compounds according to the definition by Staudinger in "Zeitschrift für Angewandte Chemie," vol. 42 (1929) page 69, are those which are formed by polymerization of specific elementary molecules, and which differ from one another only by the degree of polymerization, otherwise having the same molecular structure. In this definition the term "polymerization" is used in a strict sense to mean the union of like molecules without splitting off any groups. As noted above, the molecules involved herein contain an unsaturated aliphatic radical and an arrangement which predisposes the molecules to unite into a long chain of carbon atoms, all of which with a possible exception of a terminal pair of carbon atoms are saturated and with alternate individual or pairs of carbon atoms in the chain linked to alkyl radicals. Such polymeric homologous compounds, as typified by the polyalkenes of high molecular weight, when heated to temperatures above 300° C. undergo disintegration by scission of the carbon to carbon bonds.

A polybutene product varies in consistency from viscous liquids to tough, elastic solids as the average molecular weight of the polymers increases from about 1000 to 200,000 or higher. The transition from liquid to solid consistency occurs when the average molecular weight of the polymers is in the neighborhood of 27,000 determined by the viscosity method. Beginning at this point of transition the polymers also acquire a pronounced stringiness. It has been found that for more satisfactory results, the polybutenes with molecular weights ranging above about 27,000 are preferred, since they make an oil composition more resistant to shock, diminish the throwing off by centrifugal force, and increase the load bearing capacity of the oil composition.

Practically any desired increase of viscosity in an oil composition can be obtained by varying the molecular weight and proportion of the polymer incorporated. Accordingly, a petroleum distillate base used for the composition may be selected advantageously from a class of petroleum

oils which are more resistant to thermal decomposition and more volatile, and therefore more completely change to a gaseous state at any given temperature than the type of oils which have hitherto been necessarily employed to comply with requirements on viscosities. Tests have shown that to guard against objectionable residue from destructive decomposition of the oil base which tends to occur in some of the higher temperature forming operations and annealing treatments, it is important to restrict the maximum boiling point of a base oil to below a distillation point corresponding to 750° F. at 760 mm. mercury pressure.

In accordance with the present invention, it is possible to use as the base stock a naphtha boiling in the range from about 300° F. to 500° F., a kerosene fraction boiling from 350° F. to 600° F., or a mineral seal oil boiling from about 450° F. to 620° F. or other distillates with final boiling points as high as 750° F. The minimum distillation point is advisably above about 300 F. mainly as a safeguard against fire hazards, since as a safety measure the composition is generally required to have a minimum flash point no lower than 100° F. The petroleum oil distillate used as the base for the composition may come from any type of crude and be produced by any of the well known methods of refining, because, in general, such light fractions all are refractory to decomposition by heat to a high degree.

For the sake of illustration, the following formulae are given as illustrating typical compositions of non-staining roll oils:

I

Ingredients	Percent by weight
Petroleum naphtha distillate (boiling from 307-396° F.)	96
Polybutenes 50,000-60,000 molecular weight	3
Isopropyl esters of oxidized wax acids	1

This composition has a viscosity of 103 Saybolt Universal seconds at 100° F. and a boiling range of 317° F. to 434° F. at atmospheric pressure.

II

Ingredients	Percent by weight
Mineral seal oil (boiling from 480-600° F.)	98.5
Polybutenes 50,000-60,000 molecular weight	0.5
Isopropyl esters of oxidized wax acids	1.0

This composition has a viscosity of 99 Saybolt Universal seconds at 100° F. and a boiling range of 510-620° F. at atmospheric pressure.

Thus, satisfactory non-staining roll oils are compounded to contain above 90% of a volatile, non-viscous petroleum base oil and about 0.5 to 10% of the viscous polybutenes, which give the blend a viscosity ranging from 55 to about 300 Saybolt Universal seconds at 100° F.

Products for use as drawing compounds in mechanical forming of seamless tubes, rods, and wires, and other structural shapes may vary widely in consistency, but are generally required to be more viscous than roll oils and even semi-fluid. Furthermore, they may frequently require as ingredients materials which impart additional oiliness or film strength characteristics.

A typical non-staining drawing compound of semi-fluid consistency contains 15.3% of polybutenes having average molecular weights of 50,-

000-60,000; 83.7% of mineral seal oil boiling in the range of 480° F. to 620° F. and 1% of an oiliness agent. In general, satisfactory drawing compounds may contain from about 5% to about 30% of high molecular weight polybutenes.

Although the decided improvement in the described metal working operations through the use of the high molecular weight polybutenes is most noteworthy in that they can be compounded with volatile oils to form a product that leaves no stain on the finished product after heat treatment, it is difficult to ascertain exactly the manner in which certain other advantages are obtained. They stay in place on the working surface of the die or roll to which they are applied under the operating conditions when subjected to a high pressure and high temperature conditions encountered as in a drop forge die. The stability of such compositions to oxidation well may be another factor. They act efficiently to clean the die and to instantly release the die pressure after it has been applied because they readily form gases at the temperatures developed in the compression step. Furthermore, the manner in which they leave the operating die or rolls in a clean condition and protect such tools is also greatly in their favor.

Just as the compositions provided by this invention possess great merit in the metal fabrication processes for obtaining smooth and clean finished products, they are also useful in other industries in which fusible materials are worked and molded, as for example, in the glass and resin industry, because similar blends may be used for swabbing metal molds with avoidance of the formation of varnish-like deposits which are caused by thermal decomposition of conventional petroleum oils hitherto used as mold swabbing oils.

In the foregoing description, the illustrative examples are not intended to limit this invention, as there may be a relatively wide variation in the kinds and proportions of ingredients used in the oil composition, also in the manner in which these compositions are applied to articles being formed and to tools or apparatus used in processing the articles.

I claim:

1. In operations of mechanical working and forming of shaped structural articles from fusible solid materials, the step which comprises bringing between surfaces of the article being processed and the shaping apparatus that tends to contact with said article a non-staining oil composition comprising high molecular weight polymeric homologous compounds characterized by struc-

tures of long chains of saturated carbon atoms, of which a number have a carbon to carbon link to a recurring organic radical.

2. A step as described in claim 1, in which said high molecular weight polymeric compounds consist in polyalkenes having an average molecular weight above 1,000.

3. In operations of cold working metal pieces, the step of applying to the surface of the piece being worked a non-staining oil composition comprising a volatile petroleum oil distillate having a maximum boiling point of not more than about 750° F. and a minor proportion of polyalkenes having an average molecular weight above 1,000.

4. A step as described in claim 3, in which said oil composition comprises a petroleum oil distillate boiling in the range of 300° F. to 750° F., and a sufficient quantity of polybutenes to give the composition a viscosity above about 55 Saybolt seconds at 100° F.

5. A method of improving the finish of a shaped metal article which is produced by cold rolling and a subsequent annealing treatment, which comprises wetting the surfaces of the article as it is being cold rolled with an oil composition containing high molecular weight polybutenes and a petroleum distillate boiling below 750° F.

6. A method of improving the finish of a shaped metal article produced by die drawing, which comprises coating a metal piece as it is being drawn with a non-staining drawing compound containing a major proportion of a petroleum distillate boiling below 750° F. and from 5% to 30% of high molecular weight polybutenes.

7. In the art of cold rolling metals in which the rolled metals are substantially annealed at elevated temperatures, the step of applying to the surface of the metal being rolled a non-staining oil composition comprising above 90% of a petroleum distillate boiling substantially in the range of 300 to 620° F. blended with a sufficient proportion of viscid polybutenes having an average molecular weight above 27,000 to give the blend a viscosity ranging from 55 to 300 Saybolt Universal seconds at 100° F.

8. In the art of molding and die casting fusible solid materials, the method of improving the finish of the shaped article, which comprises the step of applying to the die a non-staining oil composition comprising a petroleum distillate boiling substantially in the range of 300° F. to 750° F. blended with a minor proportion of viscid polybutenes in sufficient amount to give the oil a viscosity above 55 Saybolt Universal seconds at 100° F.

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