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PROCESSES FOR COLD METAL REDUCTION

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This invention relates generally to the cold drawing or reduction of metals both ferrous and non-ferrous, and is directed particularly to improvements in or variations of the kinds of processes dealt with in our copending applications Serial No. 170,130, filed June 24, 1950, on "Method and Solution for Use in Cold Metal Reduction," now abandoned, Serial No. 287,357, filed May 12, 1952, on "Methods for Cold Metal Reduction," and Serial No. 287,358, filed May 12, 1952, for "Cold Metal Reduction," now abandoned.

While applicable, as indicated, broadly to the drawing and reduction of non-ferrous metals, e.g. brass and aluminum, the invention and the particular problems and conditions which it serves, have important application in the cold rolling of ferrous metals including steel, and accordingly the invention will be described typically in this adaptation, with the understanding that the essential conditions maintained at the work metal and pressure member interface may be employed to advantage in many cold drawing or reducing operations whatever may be the specific composition of the metal or the form of the pressure members; dies or rolls.

Considering the general practice followed in the cold drawing of steel, and for example in its reduction to sheet form, the stock to be reduced is advanced between one or a series of metal rolls which apply to the stock extremely high pressures creative of high level temperatures and the necessity for applying to the rolls and work metal, and to their contact interface or interfaces, a fluid which serves the dual purposes of a coolant and, according to past notions, a lubricant for the metal-to-roll contact. Thus the conventional practice has been to apply to the rolls at or near the working interface a circulation of water in which is dispersed or emulsified a water-insoluble material of an oleaginous nature serving as the so-called lubricant. Among the lubricants employed or proposed have been vegetable and animal water insoluble oils and fatty compounds, natural or synthetic palm oil having enjoyed a considerable preference. Thus the insoluble oil has been employed as a lubricant or filming agent with general success, but not without a great many difficulties which are unnecessary to enumerate beyond the observation that their seriousness has occasioned the desire and search for a better filming or lubricating agent capable of greater consistency in its performance under all various operating conditions. In addition to their physical limitations as filming or lubricating agents, the conventional insoluble oils have given rise to a troublesome situation by reason of their tendency to accumulate and become contaminated with mineral oils and greases entering the water coolant from the rolling equipment which the mineral oils lubricate. This contamination has necessitated periodic rejections and replacements of the insoluble oil with fresh batches. Further brief reference may be had to the undesirable surface condition created on the rolled product metal by reason of the insoluble oil film left on it. Further treatments and uses of the metal such as annealing, the application of tin

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plate and the like, have necessitated careful washing or scouring of the sheet to remove residual insoluble oil films.

The methods disclosed in our copending applications referred to above are predicated upon a new concept, the workability of which has been fully established, with respect to the composition of the aqueous coolant and the kind of film to be maintained at the work-pressure roll interface. In those applications we disclosed methods which depart from the conventional use of insoluble additives in the coolant water, by using instead soluble agents, and specifically water-soluble organic sulfates and sulfonates, or mixtures thereof, which create a filming condition different from the conventional insoluble lubricating additives. We view the problem and its best solution basically not as one of lubrication in the ordinary sense, but rather that of maintaining between the work and rolls a dilute aqueous solution film serving essentially as a barrier film in its interposition between the work and the rolls of a filming medium so thin ordinarily as to permit maximum directness of interengagement between the work metal and rolls, while remaining nevertheless as a barrier to their complete engagement in a strict sense.

As disclosed in the aforesaid applications, we have discovered that by the continuous application to the pressure interface of a coolant consisting essentially of a complete solution in water of a small percentage (within the range of $\frac{1}{2}$ to 10% and ordinarily in the neighborhood of 3%) of the water soluble sulfonates and sulfates of fatty compounds such as fatty acids and glycerides, the higher or fatty alcohols and fatty acid esters thereof, we achieve the surprising result that notwithstanding the dilute concentration of the sulfate or sulfonate it has the property of forming a barrier film of such great strength as to resist the extremely high unit pressures applied by the rolls to the work. Such single phase coolant solutions and many additional advantages, unnecessary here to relate in detail, including the facility with which contaminating mineral oil may be separated without disturbing the solution, avoidance of necessity for any special precautions for heating the solution to effect dissolution or dispersion of the added sulfonate or sulfate, have the outstanding advantage that by reason of its dilute concentration and complete water solubility, these coolant additives leave for most purposes no objectionable film on the reduced work, and any residue having to be removed can be washed off very readily.

Now it is found that under most operating conditions and in the reduction of most metal stocks, the reducing operation proceeds satisfactorily with reduction of the metal smoothly and to exceptionally uniform gauge, due apparently and at least in part to the film strength and capacity of the barrier film to permit such close interengagement between the rolls and the work metal as to avoid slippage and "chatter" which frequently give rise in conventional operations to off-gauge reduction and reject products. However, it has been observed that at times and over intermittent periods, operating conditions, whether by reason of the rolling equipment or work metal or a combination of both effects, may give rise to a degree of slippage between the rolls and work metal, productive of off-gauge or irregularities in the rolled stock.

Our primary object in the present invention is to provide for the correction of such conditions by increasing the traction or frictional co-efficient at the roll-work interface, and in so doing to limit or reduce slippage and chattering effects to the point where uniformly rolled gauges will be restored.

In specific relation to the combined effects of the coolant solution and insoluble added oil, the invention presents a unique film condition and formation at the working

interface. Apparently, the necessity for greater traction may be occasioned by the very high film strength and film continuity of the sulfonate or sulfate solution, a quality of essential importance from the standpoint of assuring maintenance of a pressure-withstanding barrier film, but nevertheless a condition not necessarily conducive to maximum traction. By dispersing within the solution a small percentage of the finely divided insoluble and usually lower film strength traction agent, and often desirably in less concentration than the sulfonate or sulfate, the insoluble oil in effect interrupts in localized areas or spots the continuity of the true solution film and in so doing presents corresponding areas or spots of reduced film strength and increased tractive properties, so that the overall result is to increase adequately the coefficient of friction between the rolls and work metal while retaining the sulfonate or sulfate solution as a coolant. Then instead of completely changing and replacing the coolant as traction requirements vary, it is only necessary to add the insoluble agent to the same coolant solution useable where the higher tractive property and effect are not demanded.

In accordance with the invention, it is found that as slippage occurs at the interface, proper frictional interengagement between the work and rolls may be restored or established by adding to the coolant solution a small percentage of a non-mineral oil, water insoluble additive of an oleaginous character. For this purpose we may employ amounts within $\frac{1}{2}$ to 10% by volume of the solution, and usually in the neighborhood of 3% or less, of a water insoluble additive of which the following are typical: Natural palm oil, synthetic palm oil (e.g. "Palmoshield," produced by the Ironsides Company of Columbus, Ohio), oleic and other fatty acids, lard oil, tallow and waxes. Of these, natural or synthetic palm oil may be regarded as preferred.

As previously indicated, the coolant proper is a dilute aqueous solution of an organic sulfonate or sulfate, and preferably the sulfuric acid reaction products of fatty acids, fatty glycerides, fatty alcohols and their fatty acid esters, each having between 12 to 20 carbon atoms in their molecules.

As an example of water soluble fatty acid and fatty glyceride sulfonates, we may use the sulfonates of ricinoleic acid and its glycerides, obtained from whatever source, but usually from castor oil. In making the material the castor oil preferably is completely sulfonated and then neutralized, as with ammonia, to reduce the cloud point of the oil to as low as around 20° F.

We may employ as the aqueous coolant, a dilute water solution of one or a mixture of water soluble sulphated or sulphonated unsaturated fatty acids, particularly of the ethylenic (oleic) series and of which such monoethylenic acids within the C₁₂ to C₂₀ range as lauroleic, linoleic, palmitoleic, oleic, gadoleic, cetoleic and myristoleic acids are typical. It is to be understood that reference to these acids is intended to be inclusive of their glycerides. Such unsaturated fatty acids or their glycerides may be obtained from any of various sources, including animal fats and fish oils, as well as vegetable fatty materials, e.g. the esterifiable fatty acids of tall oil. The dissolved filming agent may consist essentially entirely, except for impurities, of one or a mixture of the sulphate esters of such acids (and including their glycerides), or in such predominance, say in excess of 50%, in the filming agent as to constitute an essential component forming the body of the barrier film existing on the roll and work surfaces. For some purposes it may be desirable to include with the fatty acid sulphonate, other fatty water soluble compound sulphates or sulphonates in order to impart to the barrier film particular properties identified with such other compounds. Thus the filming agent may contain, for example, amounts of fatty monohydric alcohol sulphate esters. In preparation of the sulphated filming agent, the fatty acid or acids,

or their mixture with other sulphatable fatty materials, may be treated in accordance with known procedures for production of the water soluble sulphate esters of the fatty compounds. As to neutralization of the sulphated material, ordinarily it will be preferred to use only limited though variable neutralization, as with an alkali metal or ammonium hydroxide, to the extent of neutralizing any free sulfuric acid and of controlling specific properties, such as clarity of the product, which thus is produced as a freely flowing liquid.

Our preferred coolant is a dilute water solution of a water soluble filming agent containing effective concentrations as filming materials, of soluble sulphated higher fatty alcohols. As used herein, including the claims, the term sulphated higher fatty alcohols shall be understood to mean the water soluble sulphate ester or esters of one or a mixture of monohydric fatty alcohols, containing in the aliphatic chain between 12 to 20 carbon atoms, and of which the following are typical: cetyl, stearyl, palmoleyl, oleyl alcohols, and n-tetradecanol. In natural oils, these higher alcohols are understood to occur mainly as esters with fatty acids. For example, sperm oil is reported to be composed mainly of unsaturated higher fatty acids, possibly of the oleic series, esterified with the fatty alcohols, mainly oleyl and cetyl alcohols.

In commercial practice it has been found that these sulphated higher fatty alcohols are outstanding in imparting to the barrier film the required strength and other properties. The invention contemplates the presence in the sulphated solution or entire dissolved filming agent, of the sulphated fatty alcohols in varying concentrations so long as they are present in sufficient amounts to contribute and impart their properties substantially to the barrier film formation. Accordingly, the dissolved sulphonate may consist of the alcohol sulphate, except perhaps for small percentages of other soluble materials or sulphates. On the other hand, it is found that the favorable effectiveness of the sulphated alcohols is sufficiently great that the dissolved filming agent may contain, and for most purposes is preferred to contain substantial quantities of other sulphated or sulphonated (whichever term may be preferred) fatty materials. Thus the dissolved filming agent may comprise a mixture of sulphated fatty alcohols and sulphated fatty acids. Generally it is contemplated that the sulphated fatty alcohol content of the sulphated soluble filming agent, or of a sulphated fatty alcohol-sulphated fatty acid mixture, shall be at least 25% by volume of the agent or mixture.

The coolant lends itself to the use of sulphated natural oils containing the higher fatty alcohols and their glycerides, and to oils containing other sulphatable fatty compounds or glycerides. Outstanding results have been obtained using as the filming agent and within the concentration ranges indicated, the water soluble sulphates of sperm oil, and comprising mainly the sulphate esters and sulphates, respectively, of the higher fatty alcohols and fatty acids of the molecular size herein contemplated and which are present in sperm oil. It is to be understood that in the preparation of the sulphated filming agent, the sperm oil may be treated in accordance with any known procedure for production of the water soluble sulphate esters or their mixture with water soluble sulphated fatty acids present in the oil. The following may be cited as a typical procedure for sulphating a commercial sperm oil containing predominately, it is believed, the esters of higher fatty alcohols and fatty acids.

To 800 grams of bleached commercial sperm oil may be added from 15% to 25% by weight of concentrated (66 Bé.) sulphuric acid, slowly and with constant agitation during the mixing. The temperature of the mixture preferably is maintained substantially above atmospheric temperature and below 150° F., over a period of time that may range from 15 to 90 minutes. After this period the mixture is agitated with around 200 grams of water for about 10 minutes, the aqueous acid solution

is allowed to settle and is drawn off from the bottom of the mixture. The sulphated residual material is washed with around 240 grams of an aqueous sulphate salt solution, for example a 22% solution of ammonium sulphate. Other sulphates such as an alkali metal or aluminum sulphate may be used. The aqueous sulphate liquor is drawn off and this wash is repeated using a fresh ammonium sulphate solution. The sulphated fatty ester material then is finished preferably by treatment with a small amount of an appropriate base, e.g. alkali metal or ammonium hydroxide, the quantity of which may be variable. Ordinarily it is preferred to finish the material by treatment with e.g. ammonium hydroxide solution sufficient to clarify and at least render it free of unneutralized sulfuric acid. The product thus made is a water soluble freely fluid material.

The method of application of the coolant and film-forming solution, and the addition of an insoluble traction agent will be understood more fully from the following description of a typical adaptation of the invention as applied to the rolling and reduction of sheet metal stock. Throughout the description reference is had to the accompanying drawing illustrating diagrammatically the rolling and coolant circulation equipment.

The sheet metal stock 10 to be reduced is shown to be advanced between a pair of hard metal rolls 11 of the kind used in steel rolling mills and rotatably driven by suitable means (not shown) in the directions indicated so as to simultaneously rotate and advance the work metal 10. Should the peripheral speed of the rolls exceed the linear advancing speed of the work metal 10, slippage occurs between the roll and work metal surfaces at their interfaces 12.

Heat frictionally generated at the interfaces is dissipated, and barrier films assuring smooth and regular reduction of the work metal are maintained at the interfaces, by continuous circulation of say a 1/2 to 3% water solution of the previously described completely water soluble sulphated filming agent against the work and roll surfaces in the vicinity of their interfaces. Typically the dilute aqueous coolant solution 13 is shown to be taken from tank 14 and discharged by pump 15 through line 16 into headers 17 and 18 respectively above and below the work metal 10 and in proximate relation to the rolls 11. The solution is sprayed from the headers through nozzles 19 against the roll and work metal surfaces approaching the interfaces 12, thus maintaining a constant flow of the coolant for heat removal, and assuring the presence at the interfaces of the barrier films. Flowing from the rolls and work metal, the solution enters an appropriate cooler 20 and drains back through line 21 into the tank 14.

As previously indicated, the usual rolling mill equipment gives off mineral lubricating oil or grease which contaminates the aqueous coolant solution. By reason of the coolant itself being a complete solution of the fatty sulphonates, the insoluble mineral oil or grease accumulated by the liquid is present therein free from any association with the dissolved filming agent which could give rise to the condition heretofore recounted where an insoluble vegetable oil lubricant has adhered to prevent independent separation of the mineral oil contaminants. In the present system, the latter may readily be separated from the aqueous solution in any suitable manner, as by continuously or intermittently skimming off the mineral oil scum 22 collecting on the body of liquid in the tank 14.

As previously explained, the invention is primarily concerned with overcoming a condition of slippage at the interfaces 12, which may arise, and sometimes tend to continue only temporarily, because of any of various factors relating to the rolling equipment or the particular stock being reduced. When slippage or chattering is observed to occur we introduce to and uniformly disperse throughout the coolant solution, as by agitation

in the tank 14, a small percentage, say in the neighborhood of 3%, of the water insoluble traction agent, typically palm oil. The traction agent is found to possess, in the presence of the barrier film forming agent, and with the latter acting as a dispersing and emulsifying agent for the insoluble additive, the property of so increasing the traction at the interface as to relieve the slippage, or excessive slippage, so that the reduction is restored to smooth operation and uniformity in the gauge of the rolled sheet.

When abnormal conditions giving rise to the slippage no longer exist, some or all of the added traction agent and any mineral oil or other foreign or insoluble matter accumulating therewith in the coolant, may be separated as by filtration, decantation, or skimming so that the coolant may be restored to its normal composition of essentially a true dilute aqueous solution of the organic sulfonate.

The drawing also illustrates the applicability of the invention to multiple stand sheet steel rolling operations wherein the stock initially is reduced by passage between a first roll pair or stand, and is subsequently further reduced between however many additional stands that may be required ultimately to reduce the work metal to the desired gauge. Merely as illustrative, after passage between the rolls 11, the metal advances between a second stand composed of rolls 23 to which the coolant solution is delivered through headers 24 and 25 which discharge the solution through nozzles 26 to the roll-work metal interfaces.

We claim:

1. The process of cold metal reduction that comprises, subjecting the work metal to reduction and simultaneous cooling while moving relatively against a pressure-applying metal surface, by feeding to the work metal and pressure-applying metal surface, interface a dilute aqueous solution consisting essentially of water and under 10% of a filming agent of the class consisting of the sulfonates and sulfates of fatty acids, fatty glycerides and fatty alcohols in which the acid and alcohol molecules each contain 12 to 20 carbon atoms, and fatty acid esters of said alcohols, thereby continuously coating the work at the interface with a film formed by the solution, and incorporating in said solution a dispersed water insoluble oleaginous traction agent, in which the traction agent is of the class consisting of substantially mineral oil-free oils, fats and waxes, acting to interrupt in small localized areas the continuity of the solution interface film and thereby increase the coefficient of friction between the engaged work and pressure-applying metal surfaces.

2. The process of cold metal reduction that comprises, subjecting the work metal to reduction and simultaneous cooling while moving relatively against a pressure-applying metal surface, by feeding to the work metal and pressure-applying surface interface a dilute aqueous solution consisting essentially of water and under 10% of a filming agent of the class consisting of the sulfonates and sulfates of fatty acids, fatty glycerides and fatty alcohols in which the acid and alcohol molecules each contains 12 to 20 carbon atoms, and fatty acid esters of said alcohols, thereby continuously coating the work at the interface with a film formed by the solution, and incorporating in said solution a dispersed water insoluble oleaginous traction agent, in which the traction agent includes palm oil, acting to interrupt in small localized areas the continuity of the solution interface film and thereby increase the coefficient of friction between the engaged work and pressure-applying metal surfaces.

3. The process of cold metal reduction that comprises, subjecting the work metal to reduction and simultaneous cooling while moving between pressure-applying metal rolls, by feeding to the work metal and engaging roll surface interface a dilute aqueous solution consisting essentially of water and under 10% of a filming agent

of the class consisting of the sulfonates and sulfates of fatty acids, fatty glycerides and fatty alcohols in which the acid and alcohol molecules each contains 12 to 20 carbon atoms, and fatty acid esters of said alcohols, thereby continuously coating the work at the interface with a film formed by the solution, continuously circulating said solution between a body thereof and the locus of said interface, and adding to said solution a dispersed water insoluble oleaginous traction agent, in which the traction agent is of the class consisting of substantially mineral oil-free oils, fats and waxes, acting to interrupt in small localized areas the continuity of the solution interface film and thereby increase the coefficient of friction between the engaged roll and work surfaces.

4. The process of cold metal reduction that comprises, subjecting the work metal to reduction and simultaneous cooling while moving between pressure-applying metal rolls, by feeding to the work metal and engaging roll surface interface a dilute aqueous solution consisting essentially of water and under 10% of a filming agent of the class consisting of the sulfonates and sulfates of fatty acids, fatty glycerides and fatty alcohols in which the acid and alcohol molecules each contains 12 to 20 carbon atoms, and fatty acid esters of said alcohols, thereby continuously coating the work at the interface with a film formed by the solution, continuously circulating said solution between a body thereof and the locus of said interface, and adding to said solution a dispersed water insoluble oleaginous traction agent, in which the traction agent includes palm oil, acting to interrupt in small localized areas the continuity of the solution interface film and thereby increase the coefficient of friction between the engaged roll and work surfaces.

5. The process of cold metal reduction that comprises, subjecting the work metal to reduction and simultaneous cooling while moving between pressure-applying metal

rolls, by feeding to the work metal and engaging roll surface interface a dilute aqueous solution consisting essentially of water and under 10% of a filming agent of the class consisting of the sulfonates and sulfates of fatty acids, fatty glycerides and fatty alcohols in which the acid and alcohol molecules each contains 12 to 20 carbon atoms, and fatty acid esters of said alcohols, thereby continuously coating the work at the interface with a film formed by the solution, continuously circulating said solution between a body thereof and the locus of said interface, and adding to said solution a dispersed water insoluble oleaginous traction agent acting to interrupt in small localized areas the continuity of the solution interface film and thereby increase the coefficient of friction between the engaged roll and work surfaces, in which the traction agent when initially incorporated in the solution is of the class consisting of substantially mineral oil-free oils, fats and waxes, and in which the traction agent and mineral oil collected thereby from an extraneous source is separated from the solution.

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