United States Patent [19]

Katono et al.

[11] **3,915,869**

[45] **Oct. 28, 1975**

[54] **METAL FORMING LUBRICANT**

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[22] Filed: **June 12, 1974**

[21] Appl. No.: **478,820**

Related U.S. Application Data

[63] Continuation of Ser. No. 132,151, April 7, 1971, abandoned, which is a continuation-in-part of Ser. No. 777,742, Nov. 21, 1968, abandoned.

[30] **Foreign Application Priority Data**

Nov. 22, 1967	Japan	42-7462
Oct. 23, 1968	Japan	43-76804
Nov. 13, 1968	Japan	43-82519

[52] **U.S. Cl.** **252/12;** 252/34.7; 252/41; 252/49.5

[51] **Int. Cl.**..... **C10m 7/30;** C10m 7/20; C10m 7/16

[58] **Field of Search** 252/12, 12.2, 12.4, 12.6, 252/34.7, 41, 49.5

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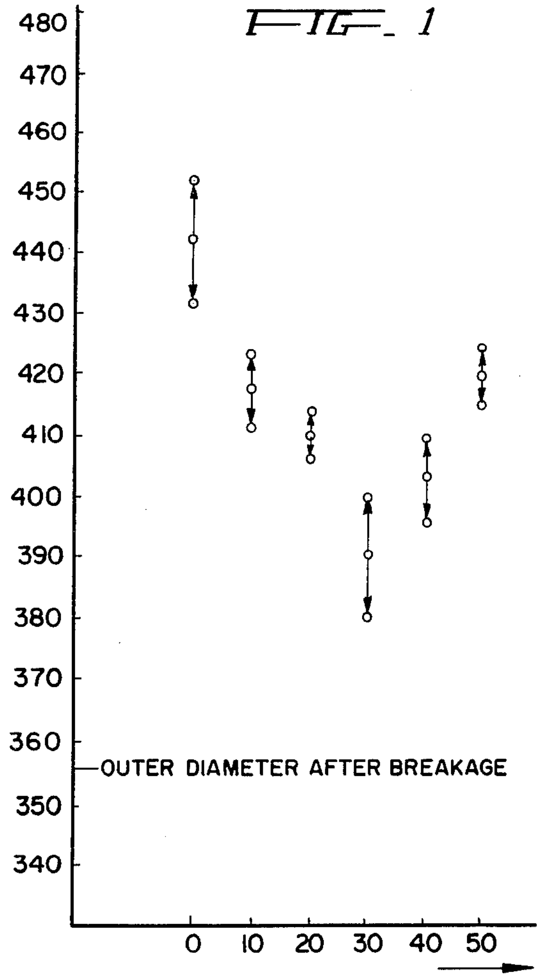
[57] **ABSTRACT**

a. A water-soluble surface-active agent selected from semi-hydrogenated beef tallow potash soap, oleic acid potash soap, oleic acid soda soap, polyoxyethylene lauryl ether, polyoxyethylene sorbitan monooleate, sorbitan trioleate, lauryl amino acetate, and stearyl amino acetate;

b. A water-soluble synthetic resin which is a combination of an oil-modified alkyd resin with a phenol or cresol-novolac-type resin.

1 Claim, 5 Drawing Figures

FIG. 1



WATER SOLUBLE SYNTHETIC RESIN (HITANOL 6080 N) / WATER SOLUBLE SURFACE ACTIVE AGENT (DICE-LEX SOAP) = 50/50

TEST SHEET: SPC-3 480mmø (BANKED DIAMETER) X 0.8mm (THICKNESS)

LUBRICANT: DIE SIDE LUBRICATING

STAMPING CONDITION: PUNCH DIAMETER 200mmø, PUNCHING SPEED 18m/min

FIG. 4

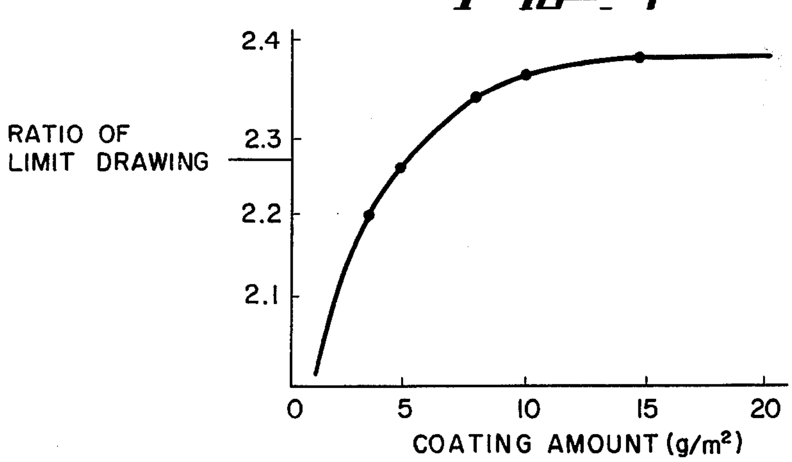
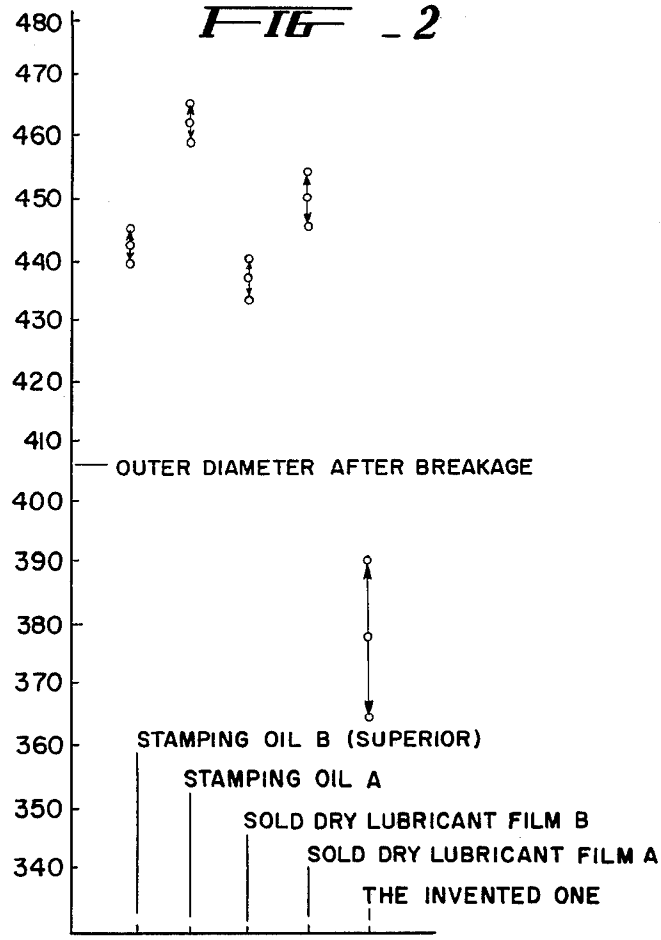


FIG. 2



TEST SHEET: SPC-3 480mmø (BLANKED DIAMETER) X 0.8mm (THICKNESS)
LUBRICANT: DIE SIDE LUBRICATING
STAMPING CONDITION: PUNCH DIAMETER 200 mm Ø
PUNCHING SPEED 18 m/min

FIG. 5

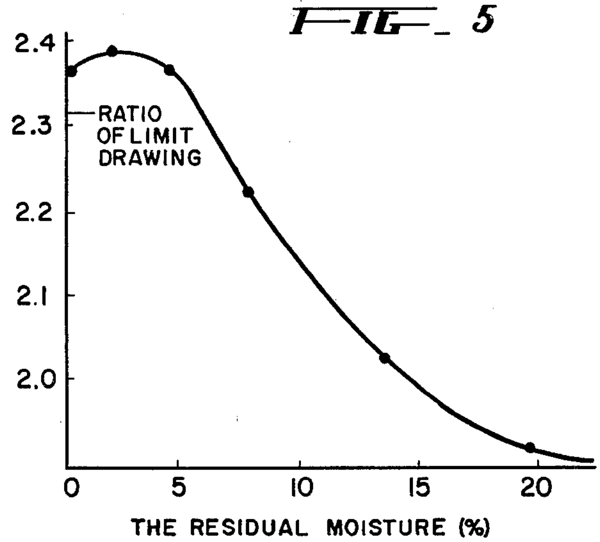
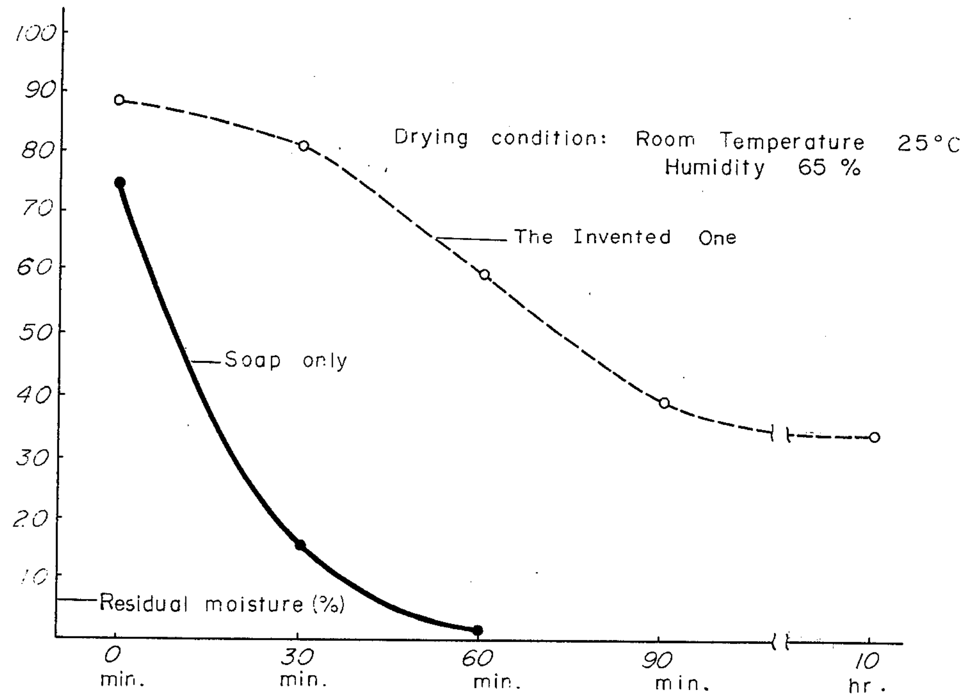


FIG. 3


100
90
80
70
60
50
40
30
20
10
Drying condition: Room Temperature 25°C
Humidity 65 %
The Invented One
Soap only
Residual moisture (%)
0 min.
30 min.
60 min.
90 min.
10 hr.

METAL FORMING LUBRICANT

This is a continuation of application Ser. No. 132,151, filed on Apr. 7, 1971, which application Ser. No. 132, 151 in turn is a continuation-in-part of Serial No. 777,742 filed on Nov. 21, 1968, both now abandoned, by the same inventors in respect of "DRY LUBRICANT AND METHOD OF MANUFACTURING METALS COATED THEREWITH."

BACKGROUND OF THE INVENTION

The invention relates to a lubricant for coating steel, particularly steel sheets, to prepare the steel for forming operations, particularly deep-drawing.

Deep-drawing of steel sheets is particularly used for making automobile parts, electrical home appliance parts and metal pipes. It is also used on steel for drawing to form metal wires, particularly electric wires.

The lubricant for these purposes must form a film on the metal and must meet a number of requirements.

a. It must have high power lubricating properties.
b. It must be adapted to be readily removed from the metal after forming.
c. It must have good anti-corrosion properties.
d. It must be useful for dry forming of the steel parts.
e. It must have a high resistance against sticking to the different parts involved.
f. It must be well adapted for welding, including spot welding.
g. It must be non-toxic, non-odorous, and non-inflammable.

Present day lubricants meet some of these objectives, but cannot meet them all.

Particularly steel sheets coated with conventional lubricants are hard to dry and require extensive periods for the drying operation. They often are not useful for high-speed stamp- or punch-forming of metals, and if used for these purposes require undue apparatus and processes to be employed.

The present invention, accordingly, has the object to provide for a lubricant for these purposes which will meet all of these requirements and still be easily applicable to the steel, in particular to steel sheets for deep-drawing operations.

SUMMARY OF THE INVENTION

These objects are met by a lubricant which comprises a mixture of a surface-active agent and a water-soluble synthetic resin in relative amounts between 20 and 80 parts of surface-active agent and between 80 and 20 parts of water-soluble resin. Preferably, the lubricant includes 1 to 50 parts of a water-soluble or water-emulsifiable lubricating oil for each 100 parts of the mixture of surface-active agent and water-soluble resin.

The steel is coated with the lubricant after preheating to between 80° and 100°C and subsequent drying so as to reduce its moisture content to below 10% by weight. The coating is thereafter applied to the steel.

The novel features which are considered as characteristic for the invention are set forth in particular in the appended claims. The invention itself, however, both as to its construction and is method of operation, together with additional objects and advantages thereof, will be best understood from the following description of specific embodiments when read in connection with the accompanying drawing.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a diagrammatic illustration of the relationship between the amount of added water-soluble oil in the lubricant of the invention and the outer diameter of a test piece after breakage in flat bottom deep-drawing;

FIG. 2 is a comparative diagram showing on one hand a conventional lubricant, and on the other hand the lubricant of the present invention;

FIG. 3 is a similar comparative diagram showing the drying speed of the lubricant of the invention compared with the drying speed of soap;

FIG. 4 is a diagram illustrating the relationship between the amount of lubricant coated on the steel and the limit of drawing ratio (LDR)(maximum drawing ratio);

FIG. 5 is a diagram illustrating the relationship bewteen the residual moisture on the steel and the limit of drawing ratio (LDR) in a steel coated with the lubricant of the invention.

DETAILED DESCRIPTION OF THE INVENTION AND OF PREFERRED EMBODIMENTS

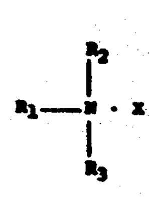
As will be further explained below, the lubricant of the invention is suitable for stamping speeds in excess of 20 m/min. Conventional dry lubricants used for this purpose fail at speeds exceeding 10 m/min. They are only good for rates up to 100 mm/min. Actually, the properties of the lubricant of the invention do improve with higher stamping speeds. The lubricant of the invention, as will be further explained, is suitable for stamping speeds even in excess of 20 m/min.

A wide range of water-soluble surface-active agents may be used in the lubricants of the invention. Reference is made to the comparative tests with different lubricants illustrated in Table 6 below. A broad list of suitable surface-active agents includes the following:

1. Anionic water-soluble surface-active agents: aliphatic acid salts in carboxylic acid soap of the formula $RCOONa(K,NH_4)$ wherein R: C_8–C_{22}. Examples are alkali metal soaps of saturated or unsaturated aliphatic acids, for example oleic acid soda soap, oleic acid potash soap, castor oil soap, beef tallow soda soap, soyabean oil soap.

2. Cationic agents:

aliphatic amine salts:



wherein X is an acid, and R_1, R_2 and R_3 are H or alkyl from C_8 to C_{22}. An example is acetamine (lauryl amine acetate, $C_{12}H_{25}NH_2{\cdot}CH_3COOH$, ["Acetamine 24" made by Kao Soap Company] or stearyl amine acetate ["Acetamine 86" made by the same company]).

3. Nonionic agents:

1. polyoxyethylene alkyl ethers $RO(C_2H_4O)_nH$, for example polyoxyethylene·oleyl ether ("Emulgen 430" of the Kao Soap Company [$C_{18}H_{35}O(C_2H_4O)_nH$])
2. polyoxyethylene sorbitan alkylesters (for example "Emasol 4130" of the same company, a polyoxyethylene sorbitan monooleate)

3. sorbitan alkyl esters, for example "Emazol 430", which is sorbitan trioleate (aromatic group), made by the same company.

Preferred anionic water-soluble surface-active agents are saturated aliphatic acid salts having 8 to 22 carbon atoms. An example is a mixture of capric acid ($C_9H_{19}\cdot COOH$), lauric acid ($C_{11}H_{31}COOH$), myristic acid ($C_{13}H_{27}COOH$), palmitic acid ($C_{15}H_{31}COOH$), stearic acid ($C_{17}H_{35}COOH$), and a mixture of unsaturated aliphatic acids having 8 to 22 carbon atoms, oleic acid [$CH_3(CH_2)_7CH{=}CH(CH_2)_7COOH$], linolic acid [$CH_3(CH_2)_4CH{=}CHCH_2CH{=}CH(CH_2)_7COOH$], linolenic acid [$CH_3(CH_2)CH{=}CH(CH_2)CH{=}CH(CH_2)CH{=}CH(CH_2)_7COOH$], and ricinoleic acid [$CH_3(CH_2)_5CH(OH)CH_2CH{=}CH(CH_2)_7COOH$].

The water-soluble synthetic resins may be the following:

1. Resins which generally come under the term "phenolic resins" such as cresol-modified novolac-type resins;

2. alkyd resins such as linseed oil, coconut oil, or castor oil modified alkyd resins;

3. phenolic-alkyd resin combinations such as linseed oil-modified alkyd resins which are combined (further modified) with a cresol or phenol novolac resin.

The resins must be water-soluble and should also be soluble in combination with the surface-active agent and the water-soluble oil.

As has been indicated, preferably a third component is included in the combination. This is a water-soluble or water-emulsifiable oil, by which term is understood an oil which is dispersed in water by emulsification.

The amounts of the three components are as follows: The mixture between surface-active agent and water-soluble synthetic resin should comprise 20 to 80 parts of the surface active agent and 80 to 20 parts of the water-soluble resin.

To 100 parts of this mixture, if desired, there may then be added 1 to 50 parts of the water-soluble oil. All parts to be understood by weight.

If the water-soluble surface-active agent is present in excess of 80%, and a corresponding reduction of the water-soluble resin is effected, the results regarding adhesion to the steel sheet are generally poor. Conversely, if the water-soluble resin exceeds 80 parts, and the surface-active agent is present in correspondingly lower amounts, the resultant mixture becomes too sticky and the resistance to sticking and blocking is decreased.

If the amount of the water-soluble oil is below 1 part, the oil would have no appreciable effect. On the other hand, if the oil exceeds 50 parts of the mixture of the mixture of surface-active agent and resin, it will be difficult to obtain uniformity of the composition because of coagulation.

The optimum ratios of amounts are about as follows:

water-soluble surface-active agent	30–70 parts
water-soluble synthetic resin	70–30 parts
water-soluble oil	5–40 parts

The lubricant is applied in an aqueous solution of a water content of between 5 and 60% by weight. Again, if the amount of water is too small, the solution becomes sticky and no uniform coating will be obtained. On the other hand, if too much water is added, the drying is too slow and a film of suitable thickness is hard to obtain.

In general, inclusion of the oil as a third component is definitely preferred because of the increased lubrication and the resulting improvement of the forming and drawing operation.

In applying the lubricant of the invention at a ratio, for instance, of 10 g/m², it was found that if the moisture content was to be reduced below 5%, as is desirable in some cases, it was necessary to apply a hot-air blast of a temperature of 150°C at a speed of application of 5 m/sec for a period of 35 seconds in case of a sheet of 0.8 mm thickness. If the sheet had a thickness of 3.2 mm, the application of the hot-air blast was for 60 seconds. Accordingly, if the conveyor moved, for example, at a speed of 60 m/min, a drying zone of more than 60 m in length would be required on which length no guiding roll should be provided. This obviously would result in difficulties with the apparatus. Besides, more difficulties would occur with respect to the tracking of the sheet. The cost of equipment, in any case, would be excessively high.

To eliminate these objections it was found that the temperature of the hot-air blast should be increased. With a temperature of the hot-air blast above 200°C, however, bubbles were generated on the lubricant-coated surface. This reduced the commercial grade of the coated steel, and also had an adverse physcial and chemical effect on the properties of the lubricating film. All this applies for sheets as well as for metal tubing and wire.

The lubricant of the present invention is slow-drying. FIG. 3 illustrates a comparison of the lubricant of the invention with a water-soluble surface-active agent used alone in the form of a soap. As can be seen, the lubricant of the invention retains approximately 40% of the original moisture after a period of 90 minutes. Thereafter, and during a period of about 16 hours, it still retains 35% of the original moisture. Soap alone retains only 15% after 30 minutes and approximately 2% after a period of 60 minutes.

Preferably the coating of the present invention is applied in an amount of 3 to 20 g/m². Most preferably the amount is between 10 and 20 g/m². The residual moisture is thus dried to an amount of less than 10% of the initial moisture, and preferably to an amount below 5%.

The reduction of the moisture is best obtained by rapid drying with a hot-air blast. The physical and chemical properties of the lubricant are thereby preserved at their maximum value while the moisture is reduced.

The coating is effected by subjecting the metal sheet, metal strip or wire to a preceding cleansing with a degreasing solution and then to a heat treatment at a temperature of 80°–100°C. The preheating treatment may be effected by a high-pressure steam blast on the order of 2 to 15 kg/cm² directed against the surface of the steel sheet or, alternatively, the sheet may be immersed in hot water at a temperature above 90°C.

Table 1 illustrates the heating times required to heat a steel sheet up to 100°C for various thicknesses and various methods of applying heat.

TABLE 1

Time for heating steel sheet to 100°C by different heating methods.

Method of heating	Thickness of sheet (mm)	Heating time
high-pressure	0.8	within 1 sec.
steam; 2 kg/cm^2	3.2	within 1 sec.
high-pressure	0.8	within 1 sec.
steam; 15 kg/cm^2	3.2	within 1 sec.
immersion into hot	0.8	10 sec.
water 100°C	3.2	35 sec.
placing in furnace	0.8	15 sec.
at 570°C	3.2	43 sec.
15 cm distant	0.8	120 sec.
from an infrared radium lamp (250W)	3.2	450 sec.

With the high-pressure steam heat, the necessary temperature increase can be obtained with a simple and inexpensive apparatus, provided that steam of sufficiently high pressure is used.

The immersion in hot water at a temperature of 100°C requires somewhat longer times and the introduction into a furnace requires a rather long time. By infrared radium lamp, the time of application is rather excessive.

After completion of the cleansing and preheating step the lubricant is applied by coating. This may be done by air spray or by other methods such as rollers or immersion.

The viscosity of the lubricant should be such as to permit application within 15–45 seconds with a Ford Cup No. 4, depending on the amount of lubricant applied and the speed of the conveyor belt.

For application the lubricant should be at a temperature between room temperature (25°C) and 50°C, but it may also be applied at high temperatures up to 90°C, depending on circumstances.

If the application is by immersion, the sheet may then be passed through squeeze rollers to adjust the amount of coating. If the application is by immersion at a temperature of about 90°C, it is also possible to omit the entire preheating step.

The coated sheet then is preferably dried by a hot-air blast.

Table 2 shows the drying time for coating a dry metal sheet with the lubricant of the invention at an amount of 10 g/m^2 and a residual moisture content of 5%. The lubricant employed in these and other tests was that of Example 1, to be described below.

TABLE 2

Relationship between drying conditions and drying time

Hot blast	temp. of sheet before coating		drying time
100°C	room temperature	(25°C)	30 sec.
wire velocity 5 m/sec		100°C	10 sec.
185°C	room temperature	(25°C)	14 sec.
wire velocity 5 m/sec.		100°C	4 sec.

The lubricant of the invention may be used on any kind of steel sheet or steel of other forms. However, a sheet steel of a thickness below 3.2 mm is preferred. Preferably, the coating of this type of sheet steel is effected at a conveyor line speed below 100 m/min. The optimum concentration of the aqueous solution applied

is between 25 and 60% by weight. Preferably, the amount applied of the lubricant is about 1 to 30 g/m^2 on the dry sheet.

The hot blast for drying is preferably applied at a temperature between 100° and 200°C and a blast velocity of 2 to 20 m/sec. The residual moisture should in any case be below 10% and preferably be below 5%.

If the water-soluble synthetic resin used in the lubricant of the invention contains an amine group, a separate anti-corrosion agent may not be necessary. Otherwise, it is preferred to add about 0.05 to 5% of anti-corrosion agent, this amount being relative to the total amount of lubricant.

The following example will further illustrate the invention.

In these examples, as well as in the tables mentioned before, the following materials were employed:

The water-soluble resin in the above tables and in all examples, except Example 5, was a linseed oil-modified alkyd resin which was combined (further modified) with a cresol novolac resin. The particular commercial product is identified as "Hitanol 6080N". This is a product of the Hitachi Company, Ltd. of Japan. In Example 5 a similar resin was used, except that the novolac-type resin was a phenol resin. This product is identified as "Hitanol 311" of the same company.

The alkyd resin is the conventional reaction product of a polybasic acid and polyhydric alcohol. In the examples the polybasic acid was phthalic anhydride and the alcohol was glycerol.

Instead of the linseed oil modification a tung oil or castor oil modification could also be resorted to.

The water-soluble surface-active agent in all above tables and all examples was beef tallow soda soap. However, in Table 6, included in Example 4, comparisons are shown with other water-soluble surface-active agents which show that similar results can be obtained with other surface-active materials.

Except where otherwise indicated, the beef tallow soda soap used in the above tables and the examples is the product commercially available under the tradename "Dice-Lex" and is a product of the Nippon Oil and Fat Company, Ltd. of Japan.

The water-soluble oil used in the above tables and in the examples, except as otherwise indicated, was a mineral base cutting oil which is incorporated in the water by emulsification. Its product identification is "JIS K-2241 WI". Table 3, which forms part of Example 1, shows the use of different types of water-soluble oils which, however, all come under the above general description of mineral oil base water-soluble or water-emulsifiable cutting oils. Where nothing else is indicated, the product employed was the product available under the tradename "Griton 114" from the Toho Chemical Industries Company, Ltd. of Japan.

EXAMPLE 1

18.6 parts of beef tallow soda soap as identified above, 18.6 parts of a water-soluble phenol-modified linseed-modified alkyd resin, and 6.9 of water-soluble oil were dissolved in 55.9 parts of water at a temperature of 60°–80°C. The water-soluble phenol alkyd resin had a pH of 9.0, a viscosity of 4.10 (poise) and a reddish-brown color. The water-soluble oil had a density of 0.93, a contents of non-volatiles of 93% and also a reddish-brown color.

The steel sheet was a sheet of 460 mm width (blank diameter) and 0.8 mm thickness. It was first cleansed and degreased. The lubricant was then applied and, after application, was subjected to drying for 6 minutes at a temperature of 120°C. A well-dried film of high anti-stick properties was thus obtained. The sheet itself :onsisted of cold-rolled steel. The flat-bottom deep-drawing in the following test was effected with a die punch of 200 mm diameter - 5R and an opposed die of 203 mm diameter - 10R. After carrying out the drawing operation on the coated steel sheet, the outer diameter, after breakage, was measured to determine the lubricating property of the deep-drawing operation.

The test results appear from the following Table 3.

EXAMPLE 2

20 parts of beef tallow soda soap, 20 parts of water-soluble phenol alkyd resin, and 12.9 parts of water-soluble oil were dissolved in 60 parts of hot water. The solution was applied to the die side of cold rolled steel (SPC-3, 480 mm inner diameter, and 0.8 mm thickness).

The tests listed below were effected with a 200 mm flat-bottom drawing and a stamping speed of 18 m/min. and and outer diamter after breakage of 360–390 mm. A stamping oil as used in the prior art gave a diameter of 440–445 mm.

TABLE 3

Test No.	Surfactant "Dice-Lex"	Resin "Hitanol 6080N"	Water-soluble oil	Outer diameter after breakage (mm)	Remarks (all tests)
1	18.6 parts	18.6 parts	6.9 parts "Griton 113"	406	coating condition:
2	"	"	6.9 parts "Griton 114"	405	coating on die
3	"	"	6.9 parts "Griton 120"	409	side;
4	"	"	6.9 parts "Griton 1000"	411	drying
5	"	"	6.9 parts "Sorton C"	409	condition:
control: superior stamping oil B (one side)				440	120°C, 6 min.

The so-called superior stamping oil B was a commercial product used for control purposes, which had a viscosity of 660.

As can be seen from the test, where the die side of the sheet blank was lubricated with this latter oil, the outer diameter after breakage was 440 mm.

As distinguished with the lubricant of the invention (Tests 1–5), the outer diameter after breakage was 405–411 mm. The lubricant in these cases was also applied to the die side of the steel sheet.

With reference to the drawings, FIG. 1 shows that the outer diameter, after breakage, with a lubricant wherein the beef tallow soda soap and the water-soluble phenol alkyd resin were present at a ratio of 50:50 and the amount of water-soluble oil was varied as indicated, the different figures for the outer diameter after breakage were obtained. The low figure with a 30% water-soluble oil addition is particularly remarkable.

TABLE 4

ubricant	Film thickness (μ) measured value	Film thickness (μ) average value	Flat-bottom deep-drawing (200 mm φ)* outer diameter after breakage (mm)	Average value
.ccording to xample 2	7		drawn	
	9	9	drawn	very well
	10		drawn	
ommercial rippable nyl loride oating	13		467	
	15	16	470	466
	20		460	
	33		466	
	37	37	466	466
	41		465	

his table shows the outer diameters at breakage upon die drawing of circular ane steel sheets. The lower the number the better are the results obtained. tamping speed 18 m/min of flat-bottom drawing. iameter after breakage D = 360 to 390 mm.

Table 4 shows different test results prepared at the stated test conditions with an aluminum-killed steel and shows specifically comparative results with the lubricant of the invention and a strippable vinyl chloride coating.

Table 5 shows similar tests comparing the lubricant of the invention with a commercial dry lubricant film product B, a superior stamping oil No. 660 and a machine oil. The aluminum-killed steel sheet in this case was identified as SPC-1 of Japanese industrial Standard. The stamping conditions were as given above.

TABLE 5

Comparison of LDR (limit drawing ratio).

Lubricant	steel sheet SPC - 3	steel sheet SPC - 1
according to Ex. 2	2.375	2.300
commercial dry lubricant film B	2.300	2.225
Stamping Oil B	2.225	2.175
Stamping Oil A	2.000	1.970

Note:
commercial dry lubricant film B (fatty acid salt)
Stamping oil A (machine oil viscosity 120)
Stamping oil B (machine oil viscosity 660)

EXAMPLE 3

16 parts of beef tallow soda soap, 24 parts of water-soluble phenol alkyd resin, and 10 parts of water-soluble oil were dissolved in water as in Example 2. This solution, as in Example 2, was applied on the die side of cold rolled steel sheet (SPC-3, 480 mm blanked diameter and 0.8 mm thickness). With the same test as in Example 2, the results with the lubricant according to the present invention were a diameter after breakage of D = 370–380 mm, while prior-art stamping oil B gave D = 440 mm to 445 mm, stamping oil A gave D = 460 to

465 mm, commercial dry lubricant B gave D = 435 to 440 mm, and commercial dry lubricant A gave D = 445 to 455 mm. FIG. 2 shows these values in graph form which indicate that the present invention is by far superior to the conventional lubricants in flat bottom deep drawing.

EXAMPLE 4

30 parts of each of the soaps in Table 6 were added to 15 parts of a phenol alkyd resin and 5 parts of a water-soluble oil. Table 4 shows the test results of flat bottom deep drawing obtained when these lubricants dissolved in 50 parts of warm water were coated on the die side of cold rolled steel sheet similar to that in Example 1, followed by drying.

TABLE 6

Soaps	Outer diameter after breakage (mm)
semi-hydrogenated beef tallow potash soal (anionic)	360
oleic acid potash soap (anionic)	405
polyoxyethylene lauryl ether (non-ionic)	380
polyoxyethylene sorbitan monooleate (non-ionic)	405
sorbiton trioleate (non-ionic)	410
lauryl amino acetate, stearyl amino acetate (cationic)	410
oleic acid soda soap (anionic)	400
control: stamping oil B	400

EXAMPLE 5

The water-soluble synthetic resin identified in Table 7 was added to 25 parts of semi-hydrogenated beef tallow soda soap and 7.0 parts of water-soluble oil. Table 3 shows the test results of flat bottom deep drawing which were obtained by using a lubricant dissolved as in Example 1 in 50 parts of warm water.

TABLE 7

Water-soluble synthetic resin	outer diameter after breakage (mm)
phenol resin ("Hitanol 311" of Hitachi Company, Ltd.)	395
alkyd resin (water sol "TD-125" of Nippon Reichhold Company)	392
melamine resin series (super bechamin "TD-95" of Nippon Reichhold)	405
methoxymethylated nylon of the nylon series sold by Teikoku Chemical Company, Ltd.)	402
vinyl acetate (Sekisui Kagaku K.K.)	407

EXAMPLE 6

Similarly as in Example 2, 12.9 parts each of the water-soluble cutting oils in Table 8 were added to 20 parts of beef tallow soda soap and 20 parts of phenol alkyd resin. Table 8 shows the test results as in Example 1 in which flat bottom deep drawing was carried out using the lubricant dissolved in 50 parts of warm water.

TABLE 8

Water-soluble oil	outer diameter after breakage (mm)
mineral oil emulsion (trade name "Swallow Cut No.4" of Maruzen, Sekiyu Co. Ltd.)	403
mineral oil emulsion (trade name "Pio Cut WX" of Daikyo Sekiyu Co., Ltd.)	405

EXAMPLE 7

Table 9 shows the results of stamping tests of cold rolled steel sheet of 0.8 mm thickness coated with the lubricant of Example 1 which was subjected, respectively, to hot-blast drying, a preheating process, and combined preheating and hot-blast drying.

TABLE 9

preheating process	coating process airless spray	drying process hot blast 5 m/sec.	cooling process: cold-air blast for 5 sec after coating	Stamping properties residual moisture (%)	LDR	height of plate after stretch forming*	Remarks
none	10 g/m²	150°C 5 sec.		25%	1.88	49.0	
"	"	150°C 40 sec.		below 5%	2.39	62.1	
"	"	300°C 5 sec.		7%	2.25	58.0	too many bubbles on surface
degreased liquid 100°C, 1 sec.	"	none	after 5 sec.	31%	1.90	48.6	
" 10 sec.	"	"	after 5 sec.	24%	1.86	49.2	
hot water 100°C, 1 sec.	"	150°C 5 sec.	"	22%	1.92	49.3	
hot water 100°C 10 sec.	10 g/m²	150°C 5 sec.	after 5 sec.	below 5%	2.36	62.4	better
high-pressure steam 6 kg/cm² 1 sec.	"	150°C 5 sec.	"	below 5%	2.38	62.3	best

Note: residual moisture = $(W-W_o)/W$ W = weight of film/unit area W_o = perfectly dry weight/unit area
*the larger the number, the better is the result obtained

Table 10 shows the results obtained by coating the above lubricant on hot rolled steel sheet of 3.2 mm thickness and preheating to dry it, and otherwise similar conditions.

TABLE 10

Preheating process	coating process airless spray	drying process hot blast 5 m/sec.	cooling process cold draw 5 m/sec.	residual moisture (%)	Stamping properties LDR	height of stretch forming (mm)	Remarks
none	10 g/m²	150°C 5 sec.		29%	1.73	49.7	
"	"	40°C 5 sec.		17%	1.82	50.5	
"	"	300°C 5 sec.		20%	1.74	50.1	too many bubbles on the surface
degreased liquid 100°C 1 sec.	"	none	after 5 sec.	31%	1.72	50.0	
degreased liquid 100°C 10 sec.	"	"	"	26%	1.70	49.8	
hot water 100°C 1 sec.	"	150°C 5 sec.	"	21%	1.75	50.3	
hot water 100°C 10 sec.	"	"	"	9%	1.88	64.0	
hot water 100°C 40 sec.	"	"	"	below 5%	2.03	68.2	good drawing results, but excessive time required
high-pressure steam 6kg/cm² 1 sec.	"	"	"	"	2.02	68.4	

In connection with Table 9 and Table 10, the values of LDR were obtained under the following conditions: punch diameter: 200 mm, radius of die shoulder: 5 mm, punching speed: 18 m/min. The height of stretch forming was obtained under the following conditions: spherical punch of radius 75 mm, die: 160 mm in diameter, bead: 190 mm in diameter and punch speed: 100 mm/min.

According to the method of the present invention, as is evident from Table 9 and Table 10, the residual moisture can be reduced to less than 5% within 5 seconds, thereby obtaining excellent results in stamping such as LDR or height of stretch forming.

Table 11 shows tests of the residual moisture of coated film and the fraction of rusted area in % after exposing steel sheet coated with the lubricant of the present invention to room air.

TABLE 11

Exposure time	Residual moisture (%) 3	7	15	30
45 days	0%	1%	8%	12%
90 days	16%	20%	21%	56%

Note: cold rolled steel sheet was used.
Test conditions:
temperature: 18 – 30°C
relative humidity: 40 – 83%
coating amount: 10 g/m²

Table 12 shows the results of similarly performed tests as to the residual moisture therein and the resistance to sticking.

TABLE 12

Exposure time	Residual moisture (%) 3	7	15	30
20°C 50% relative humidity 3 hours	*	*	O	X
35°C 5% relative humidity 30 days	O	O	X	X

Appreciating standards: * = excellent, O = medium, X = poor

coating amount: 10 g/m² load: 2 kg/cm²

As shown in Table 11 and Table 12, it is advantageous to reduce the residual moisture below 5%, both from the test results on rusting and the resistance to sticking. Furthermore, even if the lubricant of the present invention is coated on steel sheet with water-soluble skin pass oil, there is only little effect obtained. That is to say, when the lubricant of this invention was coated in an amount of 10 g/m² on cleansed steel sheet by the method of the invention, the LDR was 2.39. When the steel sheet was coated with the commercial product "Multi-luble 50 A" (trade name) in an amount of 0.2 g/m², the limit of the drawing ratio was 2.36.

EXAMPLE 8

Table 13 shows comparative tests of cold drawing properties obtained in following two methods: 50 parts of water were added to the mixture of 23 parts of Dice-Lex which is beef tallow soda soap and 27 parts of Hitanol 6080 N (water-soluble phenol alkyd resin). The mixture was heated to 70°C and coated on carbon steel tube STKM44 (42.7 mm in outer diameter and 4.5 mm in thickness). The tube was drawn to a reduction in area of 20%, 30% and 40%, respectively, and the drawing loads were measured. As control, values were obtained by using zinc phosphate and fatty acid soap. The present invention got nearly equal results with those of the above-mentioned lubricants which were obtained in substantially more complicated processes.

TABLE 13

	Ratios of reduction in area		
	20%	30%	40%
method of the invention	11.0 t	14.3 t	17.6 t
lubricant: zinc phosphate and fatty acid soap	10.5 t	12.3 t	15.2 t

t = metric tons

EXAMPLE 9

7.0 parts of water-soluble oil were mixed with 21.5 parts of beef tallow soda soap and 21.5 parts of water soluble phenol alkyd resin. 50 parts of water at 20°C were then added. The solution formed was heated to 70°C, and the same steel tube as in Example 8 was drawn to reduction in area of 20%, 30% and 40%, and the drawing loads were measured. As described in Example 8, the values thus obtained were compared with those obtained by using zinc phosphate and fatty acid soap lubricant. Table 14 shows the comparison tests.

TABLE 14

	Ratios of reduction in area		
	20%	30%	40%
method of the invention	10.4 t	13.0 t	15.0 t
lubricant: zinc phosphate and fatty acid soap	10.5 t	12.3 t	15.2 t

As can be seen, with addition of water-soluble oil, results superior to those of conventional methods were obtained. The lubricant of the invention does not require the conventional zinc phosphate treatment (80°C), water-washing thereafter, neutralizing treatment (80°C) and fatty acid soap treatment (75°C). That is, the lubricant of the invention is treated at 20°–40°C and this step of treatment therefore is very simple. After drawing even if being left for 1 month, no corrosion is noted, and if washed in alkali after drawing, metallic glaze remained on the surface. Roughness of surface was less than 1 micron in H max.

EXAMPLE 10

A mixture was prepared by adding, as described in Example 8, 50 parts of water to the solution of 30 parts of beef tallow soda soap and 20 parts of water-soluble phenol alkyd resin. With this solution cold drawing was performed on stainless steel tube SUS 27, 35 mm in outer diameter and 3.8 mm in thickness by reduction in area of 20% and 30%, and measuring the drawing loads. These tests were compared with those by conventional methods, as shown in Table 15.

TABLE 15

	Ratios of reduction in area	
	20%	30%
method of the invention	14.1 t	17.7 t
lubricant: zinc phosphate and fatty acid soap	13.5 t	15.9 t

EXAMPLE 11

7.0 parts of water-soluble oil were added to 21.5 parts of beef tallow soda soap and 21.5 parts of water-soluble phenol alkyd resin. Thereto 50 parts of water were adde as described in each of the examples. The same steel tube as described in Example 11 was coated and then drawn to reduction in area of 20% and 30%. The measured drawing load was compared with those necessary with conventional methods as stated above. The results appear from Table 16.

TABLE 16

	ratios of reduction in area	
	20%	30%
method of the invention	12.7 t	15.4 t
lubricant: zinc phosphate and fatty acid soap	13.5 t	15.9 t

This case confirms the superiority to the conventional method. The same excellent results were also obtained in Examples 11 and 12 as in Examples 9 and 10.

EXAMPLE 12

7.0 parts of water-soluble oil were added to 21.5 parts of beef tallow soda soap and 21.5 parts of water-soluble phenol alkyd resin. Thereto 50 parts of water were added. The lubricant was used for coating mild steel wire, electric copper wire and electric aluminum wire, each being 1.54 mm in diameter, and each then drawn to a reduction in area of 20%. The drawing load was compared with those necessary with the conventional lubricant, as shown in Table 17.

TABLE 17

	method of the invention	conventional method
mild steel wire	43 kg	44 kg
electric copper wire	28 kg	33 kg
electric aluminum wire	12 kg	15 kg

EXAMPLE 13

18.6 parts of beef tallow soda soap and 18.6 parts of water-soluble phenol alkyd resin were added to 60 parts of hot water at temperatures of 60°–80°C under the same condition as in Example 1 (Griton 114). Flat bottom drawing test with the lubricant was 425 mm in the outer diameter after breakage.

Without further analysis, the foregoing will so fully reveal the gist of the present invention that others can by applying current knowledge readily adapt it for various applications without omitting features that, from the standpoint of prior art, fairly constitute essential characteristics of the generic or specific aspects of this invention and, therefore, such adaptations should and are intended to be comprehended within the meaning and range of equivalence of the following claims.

What is claimed as new and desired to be protected by Letters Patent is set forth in the appended

We claim:

1. A dry lubricant for coating steel to prepare it for deep drawing and molding operations, the lubricant comprising a mixture of a surface-active agent selected from the group consisting of semi-hydrogenated beef tallow potash soap, oleic acid potash soap, oleic acid soda soap, polyoxyethylene lauryl ether, polyoxyethylene sorbitan monooleate, sorbitan trioleate, lauryl amino acetate, and stearyl amino acetate and a water-soluble synthetic resin which is a combination of an oil-modified alkyd resin with a phenol or cresol-novolac-type resin, the two components being present in relative amounts between 20 and 80% of the said surface-active agent and between 80 and 20% of the said synthetic resin.

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