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(57) Abstract

A lubricant is provided which has a hardness in the range 0.2-10 N/mm at all temperatures in the range 15-40 °C. The lubricant consists of at least one partial ester of a glycol with a fatty acid, e.g. ethylene glycol monolaurate, optionally mixed with a minor amount of a fatty acid such as stearic acid. The lubricant is useful in techniques for converting aluminium sheet to adhesively bonded aluminium structures.
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LUBRICATED METAL WORKPIECE AND METHOD

This invention relates to lubricated metal workpieces, particularly of steel and aluminium, and to a method of using such workpieces to make structures of shaped components.

There is current interest in techniques for producing adhesively bonded structures of shaped aluminium components for use in the automotive industry. Such a technique is described for example in EPA 127343. The technique of converting a coil of aluminium metal sheet into a structure of shaped components for use in the automotive industry may typically involve the following steps:–

- The metal surface is pre-treated to provide a strongly bonded layer thereon which acts as a base for subsequently applied adhesive.

- A lubricant is applied to the treated metal coil. The coil may then be stored or transported, with the lubricant serving to protect the treated metal surface, and is cut up into pieces ready for press-forming.

- The pieces of metal sheet are press-formed into components of desired shape. This and subsequent operations are all performed on an automobile production line.

- Adhesive is applied to selected areas of the shaped components, without first removing the lubricant.

- The components are assembled into the shape of the desired structure, and may be spot welded or otherwise fixed to hold the structure together until the adhesive is cured.
- The adhesive is cured at elevated temperature.
  - The metal surfaces of the structure are subjected to an aqueous alkaline cleaner which removes the lubricant.
  - The structure is painted.
  Alternatively, the press-formed components may be secured together to form the structure by mechanical means, e.g. by rivets or spot-welds, either in addition to or instead of adhesive bonding.
  A lubricant for use in such a technique needs to fulfil several requirements:
  a) The lubricant must, obviously, have suitable lubricating properties for the press-forming operation.
  b) The lubricant should be solid at likely metal storage temperatures in order to prevent stacked sheets from sticking together. Furthermore, a film of lubricant that is liquid or sticky is prone to smear and to pick up dust and dirt.
  c) Since it is not practicable in a production line to remove lubricant prior to application of adhesive, the lubricant needs to be compatible with an adhesive if one is to be used.
  d) After the adhesive has been applied and cured, the lubricant must be readily removable by an aqueous alkaline cleaner of the type conventionally used to prepare metal surfaces for painting.

The lubricants of EPA 227360 are designed to be useful, not only for the technique described above, but also for other forming and shaping operations performed on a variety of metals.

In one aspect, EPA 227360 provides a lubricating composition for press forming consisting of a lubricant dissolved or dispersed in a volatile liquid medium, wherein the lubricant comprises at least one ester of a polyhydric alcohol having two or three
hydroxyl groups of which one or two are esterified with a long chain carboxylic acid and has a melting point above ambient temperature but low enough to permit removal from a metal surface by an aqueous alkaline cleaner.

EPA 227360 mentions that mixtures of esters may be used and may be advantageous; and that the lubricant may contain a minor proportion up to 50% of one or more other lubricating compounds such as long-chain carboxylic acids. The lubricants exemplified are: diethylene glycol monostearate in solution in xylene; and diethylene glycol distearate in solution in xylene.

Although the lubricants described in EPA 227360 are generally successful at meeting requirements c) and d), they are sometimes less successful at meeting requirements a) and b). It is surprisingly found that lubricants of this kind are ineffective, so far as aluminium forming operations are concerned, at temperatures above their liquidus. For good aluminium lubricating properties, in ester lubricants of this kind, it appears necessary that some component be present in the solid state, so that the lubricant is solid or at least mushy or viscous, at the forming temperature which may be as high as 35°C or 40°C or even higher.

It might appear a simple matter to solve this isolated problem by using a different ester with a higher melting point. A difficulty with this strategy is that higher melting esters tend to be relatively hard at low and ambient temperatures, to the extent that they readily spall and flake off metal surface to which they are applied. Metal forming at 15 or 20°C cannot satisfactorily be performed under conditions where the lubricant flakes off the metal workpiece. For use in various parts of the world, there is a need
for a single lubricant system which meets both these high- and low-temperature criteria. It is an object of this invention to meet that need.

In one aspect the invention provides lubricated metal, wherein a surface of the metal carries a film of a lubricant which
a) consists essentially of at least one partial ester of a di- or poly-hydroxy compound with a C8 - C18 saturated carboxylic acid optionally in admixture with a minor amount of a long-chain carboxylic acid or ester thereof, and
b) has a hardness in the range 0.2 - 10 N/mm at all temperatures in the range 15 - 30°C, preferably in the range 0.1 - 10 N/mm at all temperatures in the range 15 - 35°C.

In another aspect, the present invention provides a method of making a structure of shaped aluminium components starting from lubricated aluminium metal sheet as defined, comprising the steps:
- forming pieces of the sheet into components,
- bringing the components together in the shape of the desired structure,
- and securing the components together by mechanical and/or adhesive means.

Hardness of the lubricant is measured by a technique whereby a block of the uncoated lubricant is equilibrated at a given temperature and is penetrated by a steel needle. The load on the needle (in Newtons) is measured as a function of its penetration into the lubricant (in millimetres), and an average gradient in N/mm is derived over a 30mm penetration. The test is derived from BS2000 part 59, "penetration of a bituminous materials", with the further adaption that the needle diameter has been increased to 12mm to ensure that the loads are not too small for accurate
measurement. Although forming, e.g. press-forming, of metal sheet is generally performed at temperatures in the range 15 - 30°C or 35°C, in some tropical locations temperatures in the press may rise to 40°C or even 45°C. It is therefore preferred that lubricant films of this invention have specified hardness values at temperatures within the range 15 - 40°C, in the case of particularly preferred lubricants, within the range 15 - 45°C.

If the lubricant film is too hard, it is likely to be brittle and have poor frictional characteristics during forming e.g. press-forming. If the lubricant film is too soft, then again the lubricating characteristics are inferior.

Preferably, the lubricant film has a hardness in the range 0.2 - 5 N/mm at all temperatures within the range specified at which forming e.g. press-forming is likely to take place in different parts of the world. It is surprising that the hardness of the lubricant film has useful predictive value for its lubricating characteristics.

The major component of the lubricant film is a partial ester of a polyhydric alcohol with a long-chain carboxylic acid. Dihydric or trihydric alcohols are suitable, for example ethylene glycol, propylene glycol, diethylene glycol and glycerol. The long chain carboxylic acid is preferably a saturated straight-chain monocarboxylic acid having from 12 to 18 carbon atoms in the chain, such as lauric, palmitic or stearic acid. Mixtures of esters may be used and may be advantageous.

The partial ester or partial esters may be used optionally in admixture with a minor amount of a long-chain carboxylic acid or ester thereof, preferably a saturated straight-chain monocarboxylic acid having from 14 - 20 carbon atoms in the chain, or ester
thereof with a saturated monohydric alcohol. The optional minor component is present in an amount of less than 50% most usually 5 - 20%, by weight on the weight of the mixture. Particularly preferred partial esters are ethylene glycol monolaurate (EGML) and propylene glycol monostearate (PGMS). A particularly preferred fatty acid is stearic acid.

To the best of our belief, there is no single partial ester of commercial purity which meets the above-stated hardness requirement. Suitable lubricants may be achieved in one or both of two ways. The first is by blending two or more components together; thus a mixture of commercial purity EGML with commercial purity PGMS in a weight ratio of 1:3 has the required hardness characteristic.

The other possible way of meeting the required hardness characteristic is to use a purer material. For example; EGML is made by partially esterifying ethylene glycol with "lauric acid", which is a fraction of coconut oil fatty acids and contains more than 30% by weight of fatty acid material which is not lauric acid. Thus:

- EGML from one commercial source had a melting point of 24°C. The "lauric acid" used in its preparation was a mixture of coconut oil fatty acids with an evident high content of unsaturated fatty acids.

- EGML from another commercial source had a melting point of 29°C and was derived from a coconut oil fraction containing more than 30% of fatty acid which was not lauric acid.

- EGML has been prepared specially for us, using lauric acid containing less than 10% of other fatty acid impurities. This EGML has a melting point of 43°C. Unlike the two commercial materials noted above, a film of this EGML does have the hardness
characteristic of this invention.

EGML for use as the only ester in the lubricant of this invention preferably has a melting point of at least 35°C. As noted below, such EGML is a mixture, but of a particular kind.

Partial esters such as EGML can have impurities arising from two main sources:-

a) The nominal fatty acid, e.g. lauric acid, is in fact a mixture of saturated long-chain monocarboxylic acids, typically containing more than 30% of acids other than the nominated one. An effect of these contaminating acids is to depress the melting point of the partial ester. For the purposes of this invention, partial esters are preferably derived from fatty acids which are at least 80% pure.

b) The partial ester was derived from a fatty acid mixture which contained ethylenically unsaturated acids. Such impurities make the lubricant less adhesive-compatible and less easy to remove from the metal surface, and are therefore preferably absent or present in amounts below 5% by weight.

Partial esterification of a polyhydric alcohol having n hydroxyl groups is effected by reacting a mixture of 1 mole of the polyhydric alcohol with less than n moles of a monocarboxylic acid. This typically results in a mixture containing a substantial proportion of the unchanged polyhydric alcohol, substantial proportions of the or each possible partial ester, and a substantial proportion of the full ester.

For example, the result of reacting a mixture of 1 mole of a glycol with less than 2 moles of a monocarboxylic acid is a mixture containing substantial proportions of the unchanged glycol, the monoester and the diester. These proportions can be altered to some extent by control over the amount of carboxylic acid used and the reaction conditions, but a mixture always results.
Solvent and unreacted polyhydric alcohol may be readily removed by volatilisation. The remaining material is generally called a partial ester, and the term "partial ester" is herein used in this sense. The partial ester has an acid value intermediate that of the corresponding polyhydric alcohol, and that of the corresponding full ester.

As noted above, the lubricating characteristics of the lubricant film on lubricated metal according to this invention fall off at both excessively high and excessively low temperature. We have developed a test, which is described below, for measuring lubricating characteristics in terms of a frictional coefficient (μ). This frictional coefficient is preferably below about 0.1 at all temperatures within the range of interest, that is to say 15°C up to 30°C or 35°C or 40°C or 45°C. As noted above, the hardness of the lubricant film at any temperature is predictive of its frictional coefficient.

Depending on its intended use, the lubricant may need to be compatible with subsequently applied adhesive. In general, the esters described herein are compatible as a result of being either absorbed or displaced by subsequently applied adhesive without grossly impairing the adhesive bond strength obtainable. By contrast, resinous lubricants and metal soap lubricants are generally not adhesive compatible in this sense.

The lubricant has a melting point above ambient temperature, preferably of at least 30°C. This ensures that the lubricant is present as a solid film on the metal substrate, which avoids problems with smearing and blocking during coiling, decoiling, slitting and cutting. The use of such a lubricant avoids contamination of the metal surface with a
possible adhesive-incompatible oil or contaminant and prevents local build up of lubricant to an undesirably thick layer.

The lubricant melts at a temperature low enough to permit its removal from a metal surface by an aqueous alkaline cleaner, such as is used in automotive production lines to prepare metal parts for painting. The highest practicable temperature for aqueous alkaline cleaners in such circumstances is about 70°C.

Lubricants melting below 70°C and preferably below 65°C can thus always be removed by aqueous alkaline cleaners. Lubricants melting above 70°C may or may not be removable depending on whether they have chemical groups, e.g. hydroxyl groups, which can react with the alkali to assist removal from the metal surface. Thus, for example, a commercially available wax having a melting point of 85°C and an acid number of 135 to 155 by DIN 53402, was found not to be removable by aqueous alkaline cleaners. On the other hand, glycerol mono-stearate, having a melting point of 81°C and two free hydroxyl groups per molecule, is removable by aqueous alkaline cleaners, and falls accordingly within the scope of this invention. A lubricant is deemed removable by aqueous alkaline cleaners if it can be removed by treatment for 2 minutes at 70°C with a 15% by weight aqueous solution of Ridolene 160 (a silicate-based proprietary cleaner marketed by I.C.I. plc.)

A further preferred aspect of this invention involves applying the lubricant to the metal in the absence of any volatile solvent or diluent. This avoids the need to evaporate volatile liquid from the lubricant film, and avoids the need to include any surface active agent in the lubricant. It is found that the molten lubricants have satisfactory viscosity for spraying or for application by roller coat. To
ensure rapid solidification of the lubricant film, the metal may be pre-cooled. To ensure good adhesion of a uniform film, the metal may be pre-heated.

The lubricant may be applied to steel or other metals, but is likely to be principally used on aluminium, which term is used to cover the pure metal and alloys in which Al is the major component. The aluminium surface may carry a strongly-bonded inorganic layer, on the top of which the lubricant is present.

Such inorganic non-metallic layers are well known, and may be provided for example as chemical conversion coatings or deposited coatings of the no-rinse type, based on chromium, titanium or zirconium; or may be an anodic oxide layer or a siloxane layer. The metal may be in sheet form. The rate of application of lubricant will depend on the intended use, but may typically be in the range of 1 - 10 g/m², particularly 2 - 4 g/m², for aluminium coil to be formed into adhesively bonded structures.

Reference is directed to the accompanying drawings in which:

- Figure 1 is a schematic view of a strip-draw apparatus used for testing lubricated metal;
- Figure 2 is a perspective view of a modified strip-draw apparatus;
- Figure 3 is a graph of hardness against temperature for films of several lubricants.
- Figures 4, 5 and 7 are Bar Charts showing frictional coefficients of three lubricants at different temperatures and different rates of application.
- Figure 6 is a bar chart showing lubricant residues after cleaning.

A purpose built strip-draw rig was designed and constructed with reference to ASTM 4173-82 for testing sheet metal forming lubricants. The apparatus
is shown in Figures 1 and 2. The die set shown in Figure 1 was designed to simulate material flowing between pressurised binder surfaces containing a draw bead arrangement. The die set of Figure 2 was designed to simulate flow between parallel binder surfaces so as to allow conventional frictional values to be obtained.

Referring to Figures 1 and 2, one die 10 of each tool set is mounted on a load cell 12. The other die 14 of the tool set is mounted on a hydraulic cylinder 16. Flat strips 18, hydraulically pressurised between the two dies, can then be pulled through a particular tool set while the clamp load is measured. The draw load is also measured using a second load cell 20 mounted between a testing machine gripping jaw 22 and a cross head 24. Thus, when used in conjunction with the flat parallel platen set of Figure 2, a conventional frictional value is obtained.

The strip draw rig is designed to be mounted on either a press simulator or a standard tensile testing frame, depending on the variables under investigation.

Lubricated strips of material, 50 mm wide, were placed between the two faces of the flat tool set of Figure 2 and hydraulically pressurised to a particular load. The strips were then drawn through the die set of Figure 1 for a distance of approximately 250 mm, the draw and clamp forces being recorded as a function of time/displacement of the drawn strip. Results presented in the form of a graph (draw force/2) versus clamp load have a slope equal to the conventional friction coefficient.
EXAMPLE 1

The following materials were used for formulating lubricants:

a) Propylene glycol monostearate of commercial purity, melting point 40°C.
b) Ethylene glycol monolaurate of commercial purity, melting point 29°C.
c) Ethylene glycol monolaurate specially made for us using 90% plus pure lauric acid, melting point 43°C.
d) Stearic acid of commercial purity, melting point 70°C.

Various formulations were made and applied by spraying onto aluminium alloy sheets which had been pre-heated to 50°C. By this means, uniform films could be applied at controlled thickness. The hardness of the various lubricants was measured (by BS 2000 Part 49) and the results are recorded on Figure 3:-

1. EGML b) : PGMS a) in a weight ratio 1:3. The hardness at various temperatures is shown by a series of empty circles connected by a solid line. This lubricant material falls within the scope of the invention.

2. EGML c) alone. The hardness is shown by filled upright triangles connected by double lines. This material falls within the scope of the invention.

3. EGML c) : PGMS a) in a weight ratio of 1:3. This is shown by filled circles. This material is in accordance with the invention, but not preferred because the hardness fell off at high temperatures.

4. EGML c) : Stearic acid d) in a weight ratio of 9:1. Two batches were made on separate occasions. The first is shown by filled diamonds connected by a dotted line; the second by filled diamonds connected by a double solid line. Both materials fall within the
scope of the invention.

5. EGML b) : Stearic acid d) in a weight ratio 9:1. The hardness is shown by open diamonds connected by a solid line. Because the hardness was relatively low at all temperatures, this material does not fall within the scope of the invention.

6. EGML b) : Stearic acid d) in a weight ratio of 3:1. This material, which is not shown in Figure 3, did have a hardness within the scope of the invention.

Lubricants 1 and 5 were further tested in the strip draw rig illustrated in Figures 1 and 2. In each case, tests were performed at four different temperatures, 20°C, 30°C, 40°C and 50°C; and at five different rates of lubricant application ranging from 1 to 6 g/m². The results of these tests are shown in Figure 4 (for lubricant 1) and Figure 5 (for lubricant 5), and Figure 7 (for lubricant 4).

EXAMPLE 2

Lubricant 4 from Example 1 was evaluated. The lubricant consists of 90 wt% EGML (mp 43°C) and 10 wt% stearic acid (mp 70°C). Lubricant 5 from Example 1 was used for comparative purposes.

Experimental Procedure

The experimental work described below was carried out on 1.6 mm gauge 5754 Accomet C pre-treated material.

2.1 Application of Lubricant to Aluminium Sheets

The procedure for lubricant application consisted of pre-heating a reservoir of the new lubricant to 70°C, and applying this onto sheets using air-assisted airless spray nozzles. Lubricant was applied to sheets which were held at both room
temperature (20°C), and preheated to 60°C. These sheets were then placed in stacks. In the case of the pre-heated material, the sheets were placed in a stack when the lubricant had solidified.

2.2 Adhesive Compatibility
The standard test method for adhesive compatibility is to assemble standard lap shear joints with a 10 mm overlap, using lubricated 1.6 mm pretreated coupons and a standard adhesive. A string of six such joints are then exposed to combined stress/humidity testing under a constant load. The time to failure of the first three joints in a set of six joints is then noted. Individual lap shear joints are also exposed to salt spray for given periods of time, and then tested for static strength retention.

Tests were carried out on joints manufactured with the lubricant 4 on their surfaces prior to bonding. Two lubricant weight levels were evaluated, namely 2.0 g/m² and 5.5 g/m².

2.3 Lubricant Softening as a Function of Temperature
The Wax Penetration Test, BS 2000 pt 59, was used to determine the softening response as a function of temperature.

2.4 Strip Draw Evaluation of the Lubricant 4
Lubricated sheets were produced with 3 g/m² of different lubricants via the pre-heated blank route, as indicated in section 2.1. These sheets were guillotined into strips 50 mm wide and then drawn through the strip draw rig, using the described procedure, to allow friction values to be determined at temperatures of 20, 30, 40 and 50°C.
2.5 Press Forming Evaluation of the Lubricant 4

Press forming tests were carried out on a press simulator to evaluate the lubricant. Two distinct trials were used, namely:

(a) Square pan depth to failure.
(b) Pressed dome height to failure.

The above trials were carried out under standard conditions on a 275 mm square tooling without the draw bead sections.

Sheets of AA5754-0 were pressed with 3 g/m² of both lubricants 4 and 5 to allow the comparative performance to be assessed.

2.6 Simulation of Possible Thermal Cycles of Pre-Lubricated Stacks

In order to simulate possible thermal cycles which may be experienced by pre-lubricated material, lubricated stacks were produced by applying the lubricant to pre-heated blanks, as described in section 2.1.

Four stacks were produced containing some thirty sheets, each 500 x 500 mm, with a nominal 3 g/m² lubricant weight. These stacks were heated to four different temperatures in an oven at 30, 35, 40 and 45°C respectively. After removal from the oven, each stack was left to cool with a centrally applied weight of 18.1 kg. After destacking, coupons were removed from a number of adjacent sheets to quantify any lubricant transfer observed.

2.7 Cleaning, Oven Evaporation and Residues

Two distinct tests were carried out in this section, namely different oven bakes followed by a cleaning stage, including no bake, and a typical bonded structure route with the adhesive cure cycle included.

For the first series of tests, pretreated
strips of aluminium were lubricated with lubricant 5 (3.4 g/m²) and lubricant 4 (3.8 g/m²), and given the following treatment:

a) 20 minutes at 170°C
b) 20 minutes at 180°C
c) 20 minutes at 190°C
d) 20 minutes at 200°C
e) No oven bake.

All strips were then cleaned in stirred 20 g/litre solutions of Chemkleen CK165 at a temperature of 60°C for 3 minutes. After drying, organic contamination on the strips was measured, as carbon, by analysis at 600°C.

For the second series of tests, clean sheets of aluminium were coated with lubricant 4 and lubricant 5 at a coating weight of approximately 4.5 g/m². The sheets were then subjected to a cumulative oven-bake and alkali-clean cycle. This consisted of:

a) 10 mins at 145°C
b) 20 mins at 190°C
c) 20 mins at 190°C
d) 30 secs alkali clean, (stirred 2.5% w/w Ridolene 336 at 60°C).

Final coat weights were measured after the cleaning stage using gravimetric determination.

3. RESULTS

3.1 Application of the Lubricant to the Sheets

Satisfactory results were obtained by spraying the lubricant 4 onto sheets held at both room temperature, approximately 20°C, and sheets pre-heated to 60°C. The lubricant solidified upon contact with the sheets held at ambient temperature. However, the latter condition allowed the lubricant to remain liquid on the sheets for a short time period.
The lubricant itself passed through the spray nozzles without any additional problems to those encountered with the lubricant 5.

3.2 Adhesive Compatibility

The results of stress-humidity testing on joints produced with lubricant 4 on their surfaces are presented in Table 1. This Table shows a good strength retention after 20 weeks salt spray, and a testing duration in excess of 100 days during stress/humidity with a 5 MPa applied stress.

3.3 Lubricant Softening as a Function of Temperature

The results of the Wax Penetration Testing carried out on lubricant 4 are presented in Figure 3.

3.4 Strip Draw Evaluation of the Lubricant 4

Table 2 shows the comparative performance of the lubricants 4 and 5 over the measured temperature range for a given lubricant weight of 3 g/m². These figures show an improved performance of the lubricant 4 at temperatures of 30, 40 and 50°C. They also indicate a similar performance at 20°C.

3.5 Press Forming Evaluation of the Lubricant 4

The results of the press forming trials are shown in Table 3. This Table indicates that both lubricants give a similar performance during stretch forming, but an improved performance is obtained during square pan forming with the lubricant 4. The values quoted are the average of five tests in each case. The tests themselves were carried out in ambient conditions of around 22-24°C.
3.6 Simulation of Possible Thermal Cycles of Pre-Lubricated Stacks

Stacks of lubricated sheets, having lubricant 4 on their surface, which were heated to 30, 35 and 40°C and subsequently cooled with a centrally applied weight showed no evidence of de-stacking problems or lubricant transfer between adjacent sheets. The corresponding stack heated to 45°C was more difficult to separate, showing clear evidence of a "patchy" appearance, and slight lubricant transfer between adjacent sheets. With the lubricant 5, a similar effect was seen at a temperature of 35°C.

3.7 Cleaning, Oven Evaporation and Residues

The results of the different oven bakes followed by a cleaning stage, including no bake followed by a cleaning stage, are given in Figure 6. This figure shows that high oven bakes contribute to the surface cleanliness.

The results of the oven evaporation trials show that lubricant 4 evaporates almost totally compared to lubricant 5, 0.03 g/m² and 0.35 g/m² residue respectively. Final coat weights were also measured after the final alkali clean using gravimetric determination. The results of both lubricants fell to between -0.01 and -0.03 g/m² indicating that the cleaning stage is removing all of the residues.

4 DISCUSSION

4.1 Application of the Lubricant to the Sheets

No difficulties were experienced with the spray application of lubricant 4. Adherence to the metal surface was improved by pre-heating the sheets, although a satisfactory appearance was obtained with room temperature metal.
4.2 Adhesive Compatibility

The results of the stress/humidity testing show that joints manufactured with lubricant 4 at a level of 2 g/m² are still on test, with 190 days achieved to date for all stress levels tested.

Salt spray data after 20 weeks exposure shows excellent strength retention at both lubricant weight levels, out performing the lubricant 5.

4.3 Lubricant Softening as a Function of Temperature

Results of the Wax Penetration test, presented in Figure 3, show the hardness improvement over the temperature range examined. Earlier work had suggested that the hardness value should be maintained between 0.1 and 1.0 N/mm on the vertical logarithmic axis. Significantly exceeding the higher hardness values at lower temperatures will produce a wax which is brittle and has limited value in terms of press die forming. At the higher temperatures, hardness values less than 0.1 N/mm correspond to the melting range of the wax. Figure 3 shows that the hardness of lubricants falls to 0.1 N/mm around 41-42°C, corresponding to its melting range. This is approximately 10-12°C higher than lubricant 5. Thus the upper melting range has been significantly increased without producing a brittle wax at the lower temperature range.

4.4 Strip Draw Evaluation of the Lubricant 4

In terms of frictional performance versus temperature for a lubricant weight of 3.0 g/m², Table 2, the lubricant 4 demonstrated a significant improvement in the frictional coefficient at 30, 40 and 50°C, whilst having a similar performance at 20°C.
4.5 Press Forming Evaluation of the Lubricant 4

The results of the press forming evaluation indicate that the lubricant 4 formulation has an equal performance during stretch forming, but a somewhat better performance during square pan pressing. This should give an advantage to components such as door rings and door inner pressings, where deep corner features are required having small corner sweep radii. Thus the performance improvement will be beneficial.

4.6 Simulation of Possible Thermal Cycles of Pre-Lubricated Stacks

Results of thermal cycles applied to pre-lubricated stacks have shown no evidence of lubricant transfer at 40°C, and only slight evidence of lubricant transfer when the stack was heated to 45°C and then cooled. Thus, problems of lubricant transfer with the lubricant 4 will now become apparent at temperatures between 40 and 45°C. This performance is much better than the lubricant 5 where no evidence of transfer was visible when the stack was heated to 30°C, but evidence was visible when the stack was heated to 35°C. Hence a 10°C temperature improvement in terms of handling performance has been achieved.

4.7 Cleaning, Oven Evaporation and Residues

Trials to assess the cleaning of the surface after either various oven bakes or no bake, Figure 6, have shown that Chemkleen solution does clean the surfaces reasonably effectively. High oven bake conditions, 190°C and 200°C, definitely contribute to surface cleanliness in the case of lubricant 4, which evaporates very cleanly from the aluminium surfaces.

Trials to determine the relative evaporation and remaining residues of lubricant 4 versus lubricant 5 have shown that the lubricant 4 evaporates almost
completely prior to the cleaning stage. Further, lower levels of carbon residuals are obtained.

Table 1: Stress/Humidity and Salt Spray Data for the Lubricant 4

<table>
<thead>
<tr>
<th>Lubricant 4 Weight g/m²</th>
<th>Stress/Humidity Days to Failure</th>
<th>Salt Spray Results (MPa)</th>
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<tr>
<td></td>
<td>3 MPa</td>
<td>4 MPa</td>
</tr>
<tr>
<td>2.0</td>
<td>190+</td>
<td>190+</td>
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<td>5.5</td>
<td>190+</td>
<td>190+</td>
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Table 2

<table>
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<tr>
<th>Friction Value\Temp</th>
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<th>30°</th>
<th>40°</th>
<th>50°</th>
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<tr>
<td>Lubricant 4</td>
<td>0.017</td>
<td>0.012</td>
<td>0.046</td>
<td>0.069</td>
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<tr>
<td>Lubricant 5</td>
<td>0.020</td>
<td>0.036</td>
<td>0.096</td>
<td>0.107</td>
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</table>

Table 3: Effect of Lubricant on Press Formability

<table>
<thead>
<tr>
<th>Lubricant</th>
<th>Coat Weight (g/m²)</th>
<th>Square Pan Depth mm</th>
<th>Dome Height mm</th>
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<tr>
<td>5</td>
<td>3.0</td>
<td>59.1</td>
<td>51.5</td>
</tr>
<tr>
<td>4</td>
<td>3.0</td>
<td>74.3</td>
<td>52.0</td>
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CLAIMS

1. Lubricated metal, wherein a surface of the metal carries a film of a lubricant which
   a) consists essentially of at least one partial ester of a di- or poly-hydroxy compound with a C8 - C18 saturated carboxylic acid optionally in admixture with a minor amount of a long-chain carboxylic acid or ester thereof, and
   b) has a hardness in the range 0.1 - 10 N/mm at all temperatures in the range 15 - 35°C.

2. Lubricated metal as claimed in claim 1, wherein the partial ester is ethylene glycol monolaurate and/or propylene glycol monostearate.

3. Lubricated metal as claimed in claim 1 or claim 2, wherein the partial ester is ethylene glycol monolaurate having a melting point of at least 35°C.

4. Lubricated metal as claimed in any one of claims 1 to 3, wherein the film of lubricant has a hardness in the range 0.2-5 N/mm at all temperatures within the range specified.

5. Lubricated metal as claimed in any one of claims 1 to 4, wherein the film of lubricant has a hardness within the range specified at all temperatures in the range 15 - 40°C.

6. Lubricated metal as claimed in any one of claims 1 to 5, wherein the metal is sheet.

7. Lubricated metal as claimed in any one of claims 1 to 6, wherein the metal is aluminium.

8. Lubricated metal as claimed in claim 7, wherein the aluminium carries a strongly-bonded artificial inorganic surface layer on top of which the film of lubricant is present.
9. A method of making the lubricated metal claimed in any one of claims 1 to 8, which method comprises applying the lubricant to the metal in the absence of any volatile solvent or diluent.

10. A method as claimed in claim 9, wherein the lubricant is applied by roller coating.

11. A method of making a structure of shaped aluminium components starting from lubricated aluminium metal sheet according to claim 6 or claim 7 comprising the steps:
   - forming pieces of the sheet into components,
   - bringing the components together in the shape of the desired structure,
   - and securing the components together by mechanical and/or adhesive means.

12. A method as claimed in claim 11, comprising the steps:
   - forming pieces of the sheet into components,
   - applying adhesive to the components,
   - bringing the components together in the shape of the desired structure, and
   - curing the adhesive.

13. A method as claimed in claim 11 or claim 12, comprising the additional steps of subjecting the structure to the action of an aqueous alkaline cleaner, and thereafter painting the structure.

14. Lubricated metal as claimed in any one of claims 1 to 8, wherein the lubricant consists of 80-95 wt% of ethylene glycol monolaurate having a melting point of at least 35°C and 5-20 wt% of stearic acid.
Fig. 4  BAR CHART SHOWING THE RELATIONSHIP BETWEEN FRICTIONAL COEFFICIENT (μ), LUBRICANT WEIGHT AND TEMPERATURE FOR LUBRICANT 1.

Fig. 5  BAR CHART SHOWING THE RELATIONSHIP BETWEEN FRICTIONAL COEFFICIENT (μ), LUBRICANT WEIGHT AND TEMPERATURE FOR LUBRICANT 5.
Fig. 6: LUBRICANT RESIDUES AFTER DIFFERENT BAKES FOLLOWED BY CLEANING

LUBRICANT RESIDUES AFTER CLEANING
(3 MINS IN 20g/l CHEMKLEEN 165 AT 60°C)

mg/M² AS CARBON

5

LUBRICANT

4

170°C  180°C  190°C  200°C  NO BAKE
20 MINUTES OVEN BAKE
Figure 7: Bar Chart Showing the Relationship Between Frictional Coefficient, Lubricant Weight and Temperature for the Lubricant 4.
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

IPC 5 C10M105/40 C10M169/04 //C10N40/20, C10N70/00, C10N80/00,
(C10M169/04, 105:40, 129:40, 129:70)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 C10M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>EP,A,0 276 568 (ALCAN INTERNATIONAL LIMITED) 3 August 1988 see page 5, line 35 - line 38 see page 8; example 3 see claim 12 ---</td>
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Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

* Special categories of cited documents:
  * "A" document defining the general state of the art which is not considered to be of particular relevance
  * "E" earlier document but published on or after the international filing date
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* X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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* & document member of the same patent family

Date of the actual completion of the international search: 30 November 1993

Date of mailing of the international search report: 27.12.93

Name and mailing address of the ISA

European Patent Office, P.B. 3418 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo NL,
Fax (+ 31-70) 340-3016

Authorized officer

Hilgenga, K
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