

5                    HOT ROLLING PROCESS FOR ROLLING ALUMINIUM AND  
                      ALUMINIUM ALLOYS SHEETS

10            The present invention relates to a hot rolling process for rolling aluminium and aluminium alloys sheets.

          The invention also relates to a water-soluble aluminium and aluminium hot rolling oil composition, to a oil-in-water emulsion prepared therewith and to the use of said oil-in-water emulsion in a hot rolling process.

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          The aluminium and aluminium alloys rolling industry expresses the need to maximize the efficiency of their rolled metal manufacturing process. In general terms, this means that there is a wish to operate at higher rolling speeds and to produce more marketable products per operating shift. Additionally, there is also a wish to minimize the number of passes through the mill taken to achieve a given level of reduction. Both these routes require that quality and surface finish be not compromised.

25            The invention thus provides an oil composition for hot rolling mills that affords the following customer benefits:

- a higher reduction ratio : one pass reduction is in most cases achievable;
- 30            - a better rolling ability (i.e. a lower rolling force and a reduced power consumption ) as compared to the rolling ability obtained with oil compositions of the prior art;
- an improved rolled surface finish quality;
- 35            - a shorter emulsion break-in period, which is the time required to obtain the optimal partical size distribution;
- an excellent resistance to corrosion; and

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- a high lubricity (plate-out properties, roll coating).

The invention is effective on any type of hot rolling, be it reversible or not, on breakdown, tandem and combination mills.

Especially, the invention exhibits high reduction and rolling capabilities while providing an excellent strip surface finish when rolling at high speed.

The prior art does not teach or even suggest the instant invention.

Thus, the invention provides a hot rolling process for rolling aluminium and aluminium alloys sheets, comprising applying an effective amount of an oil-in-water emulsion comprising water and a water-soluble oil composition comprising a base stock oil and, based on the total weight of the water-soluble oil composition, from 1 to 80% and preferably from 3 to 30% by weight of di(2-ethylhexyl) adipate ester.

According to one embodiment, the oil composition further comprises, based on the total weight of the composition, from 0,05 to 20%, preferably from 0,1 to 5% by weight of a non-ionic surfactant, such as an ethylene oxide addition polymer. An example of such an ethylene oxide addition polymer is the ethylene oxide addition polymer sold by the company ICI under tradename Hypermer® A60.

According to a further embodiment, the oil composition further comprises, based on the total weight of the composition, from 1 to 30%, preferably from 5 to 20% of oleic acid. It is actually believed that the free oleic acid provides a better surface finish to the aluminium or aluminium alloy strip.

The invention further provides a water-soluble aluminium and aluminium alloys hot rolling oil composition.

The invention further provides a process for preparing the water-soluble oil composition.

The invention further provides an oil-in-water emulsion containing the oil composition and a process for preparing this emulsion.

In addition, the invention provides the use of the water-soluble oil composition of the invention to prepare emulsions intended to be used in a aluminium or aluminium alloy hot rolling process.

5 Finally, the invention provides the use of the emulsion in a hot rolling process.

The invention is now disclosed in more details in the following specification.

10 Figure 1 is a graph showing the applied rolling force versus the number of passes, first, when an emulsion of the prior art and then when an emulsion of the invention are used.

15 Figure 2 is a graph showing the applied net rolling power versus the number of passes, first, when an emulsion of the prior art and then when an emulsion of the invention are used.

20 The water-soluble oil compositions of the invention are neat oil concentrates generally intended to be diluted in water to give oil-in-water emulsions.

The base stock oil is any oil typically used in the field of hot rolling. It can be paraffinic or naphthenic.

25 Paraffinic base oils are made from crude oils that have relatively high alkane contents (high paraffin and isoparaffin contents). Typical crudes are from the Middle East, North Sea, US mid-continent. The manufacturing process requires aromatics removal (usually by solvent extraction) and dewaxing. Paraffinic base oils are  
30 characterized by their good viscosity/temperature characteristics, i.e. high viscosity index, adequate low-temperature properties and good stability. They are often referred to as solvent neutrals, where solvent means that the base oil has been solvent-refined and neutral means  
35 that the oil is of neutral pH. An alternative designation is high viscosity index (HVI) base oil. They are available in full range of viscosities, from light spindle oils to viscous brightstock.

Naphthenic base oils have a naturally low pour point, are wax-free and have excellent solvent power. Solvent extraction and hydrotreatment can be used to reduce the polycyclic aromatic content.

5 A preferred base oil is a mixture of paraffinic and naphthenic oils.

The base oil typically has a viscosity from 7 to 150 cSt at 40°C, and preferably from 20 to 50 cSt at 40°C.

10 The water-soluble oil composition preferably comprises a trialkanolamine ( $C_{2-4}$ ), preferably triethanolamine, the amount of which being such that all bindable trialkanolamine is bound to a part only of the oleic acid. The aim of this embodiment is to ensure that there remains  
15 free oleic acid in the oil composition.

The product of the reaction of the trialkanolamine with oleic acid acts as a surfactant.

The water-soluble oil composition may comprise  
20 classical additives, such as surfactants, coupling agents or cosurfactants, friction reducing agents or lubricity agents, corrosion inhibitors or anti-oxidants, extreme-pressure and anti-wear agents, bactericides and fungicides, anti-foaming agents, anti-rust agents.

25 Examples of anti-foaming agents are silicone based, especially polydimethylsiloxane.

Examples of corrosion inhibitors are hindered phenols and zinc dialkyldithiophosphates (ZDDP).

Examples of extreme-pressure and anti-wear agents are  
30 dilauryl phosphate, didodecyl phosphite, trialkylphosphate such as tri(2-ethylhexyl)phosphate, tricresylphosphate (TCP), zinc dialkyl(or diaryl)dithiophosphates (ZDDP), phospho-sulphurized fatty oils, zinc dialkyldithiocarbamate), mercaptobenzothiazole, sulphurized  
35 fatty oils, sulphurized terpenes, sulphurized oleic acid, alkyl and aryl polysulphides, sulphurized sperm oil, sulphurized mineral oil, sulphur chloride treated fatty oils, chlornaphta xanthate, cetyl chloride, chlorinated

paraffinic oils, chlorinated paraffin wax sulphides, chlorinated paraffin wax, and zinc dialkyl(or diaryl)dithiophosphates (ZDDP), tricresylphosphate (TCP), trixylylphosphate (TXP), dilauryl phosphate, respectively.

5        Examples of corrosion inhibitors or anti-oxidants are radical scavengers such as phenolic antioxidants (sterically hindered), aminic antioxidants, organo-copper salts, hydroperoxides decomposers, butylated hydroxytoluene.

10       Examples of anti-rust agents are amine derivatives of alkenyl succinic anhydride.

      Examples of friction reducing agents/lubricity agents are fatty acids ( $C_{12-20}$ ) like lauric acid, oleic acid, palmitic acid, alkyl( $C_{6-10}$ ) alkylate( $C_{12-20}$ ) esters like di(2-ethyl-hexyl) adipate.

15       Further elements on base oils and additives can be found in "Chemistry And Technology Of Lubricants", R.M. Mortier and S.T. Orszulik, VCH Publisher, Inc, 1992.

20       The following is the content of the water-soluble oil composition of the invention (the percentages are weight percentages based on the total weight of the composition) :

- 0.1-0.5% of trialkyl ( $C_{1-4}$ ) phenol;
- 25       - 0.5-4.0% of trialkyl ( $C_{3-10}$ ) phosphate ester;
- 5-15% of di(2-ethylhexyl) adipate ester;
- 5-15% of organic fatty acid ( $C_{12-20}$ );
- 0.5-2% of 5-carboxy 4-hexyl 2-cyclohexen 1-octanoic acid;
- 30       - 1-3% of alkylene ( $C_{2-6}$ )glycol;
- 0.3-1% of ethoxylated alcohols ( $C_{5-14}$ , comprising 2-10  $CH_2O$  groups);
- 2-5% of trialkanol amine ( $C_{2-4}$ );
- the balance being a mixture of naphthenic and
- 35       paraffinic lube base oils.

      The oil composition is prepared by blending the base oil and the other ingredients, preferably under stirring or

with any mixing device, whilst controlling the temperature so that it does not exceed 50°C, and more preferably, 35°C.

5 An oil-in-water emulsion is prepared by diluting under stirring the oil composition of the invention in water. It is preferred to use deionized water which may previously have been warmed to around 35°C.

10 The emulsion generally comprises water and, based on the total volume of the emulsion, from 0.5 to 30%, preferably from 1 to 15%, by volume, of the oil composition.

15 The aluminium alloys to which the invention applies are any aluminum alloys, including 1000, 2000, 3000, 5000, 6000, 7000 series.

The hot rolling process can be the classical process. The rolled metal temperature is generally around 600-650°C for breakdown mills and around 400-500°C for tandem mills.

20 The process is preferably carried out on a breakdown rolling mill, on a tandem rolling mill or on a finishing rolling mill. The instant oil composition allows a significant reduction of the number of passes. With conventional prior art oils, the number of passes was typically 13. The oil composition of the invention allows  
25 lowering this number to two passes, which is a significant improvement.

30 When the hot rolling process is carried out in a breakdown mill, the emulsion preferably comprises, based on the total volume of the emulsion, from 2 to 4% by volume of the oil composition.

35 When the hot rolling process is carried out in a finishing or tandem mill, the emulsion preferably comprises, based on the total volume of the emulsion, from 5 to 7% by volume of the oil composition.

The following examples illustrate the invention without limiting it. All parts and ratios are given by weight, unless otherwise stated.

### 5      Example

A composition is prepared by mixing the ingredients of Table 1 in the order in which they appear in this table. The temperature is to be maintained at a maximum of 35°C to ensure a complete dissolution and homogeneisation of the ingredients without impairing the properties of the emulsion.

| TABLE 1                                                              |               |
|----------------------------------------------------------------------|---------------|
| Ingredients                                                          | Content (wt%) |
| Base oil (naphthenic, 600 SUS)                                       | 27.00         |
| Base oil (paraffinic, 9 cSt at 40°C)                                 | 44.60         |
| Di-tertiobutyl paracresol (antioxidant)                              | 0.20          |
| Trioctylphosphate ester (extreme pressure agent)                     | 2.00          |
| Di(2-ethylhexyl)adipate ester (lubricity agent)                      | 8.00          |
| Oleic acid (friction reducing agent/lubricity agent)                 | 11.6          |
| 5-carboxy 4-hexyl 2-cyclohexen 1-octanoic acid (corrosion inhibitor) | 1.00          |
| Diethylene glycol (coupling agent)                                   | 2.10          |
| Ethylene oxide addition polymer *                                    | 0.50          |
| Triethanol amine (buffer/cosurfactant)                               | 3.00          |

\* : sold by ICI under the tradename Hypermer® A60

The characteristics of the composition of Table 1 are set out in Table 2.

| TABLE 2                         |          |             |                         |
|---------------------------------|----------|-------------|-------------------------|
| Oil concentrate before dilution | Unit     | Method      | Typical characteristics |
| Colour (ASTM)                   |          | ISO 2049    | L 1.0                   |
| Density at 15°C                 | G/ml     | ASTM D 1298 | 0.8908                  |
| Pour point                      | °C       | ISO 3016    | -27                     |
| Viscosity at 40°C               | CSt      | ASTM D 445  | 32.6                    |
| Neutralization                  | KOH mg/g | ASTM D 974  | 25.6                    |

|                          |          |           |      |
|--------------------------|----------|-----------|------|
| number                   |          |           |      |
| Saponification<br>number | KOH mg/g | ASTM D 94 | 51.6 |
| Total base number        | KOH mg/g | ISO 3771  | 11.5 |

An emulsion is prepared by diluting under stirring the oil composition of Table 1 in deionized water prewarmed to 35°C. The characteristics of the obtained emulsion are given in Table 3.

| TABLE 3                                                               |                     |                         |
|-----------------------------------------------------------------------|---------------------|-------------------------|
| Emulsion                                                              | Method              | Typical characteristics |
| Stability of the 6% (v/v) emulsion(at room temperature, for 20 hours) | Mobil <sup>1)</sup> | 1% cream                |
| pH value of fresh 5% (v/v) emulsion at 20°C                           | ASTM E 70-90        | 8.3                     |

<sup>1)</sup>: The emulsion stability was determined according to the following procedure. 470 ml of distilled water at room temperature or test temperature were measured into a 800-ml beaker. A 50-ml stirrer having four paddles was attached to a stirring motor so that the paddles were positioned 25mm above the bottom of the beaker. A 50-ml dropping funnel was positioned such that the outlet was 15mm from the beaker wall. The stirrer was turned on and the rate adjusted to 1000 rpm. The sample was then heated up to a temperature of 35 ± 1°C. 30 ml of the test oil were added to the dropping funnel. The dropping rate was adjusted such that all the oil was transferred to the water within 120 ± 20 seconds. The stirring was then continued for an additional 60 seconds while the sample temperature was maintained at 35±1°C. The resulting emulsion was poured into a 500-ml graduated cylinder and allow to stand at room temperature

for 20 hours. After 20 hours, the upper layer (yellow cream + oil) was read in volume percent.

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#### EXPERIMENTAL TESTING

A blank is first prepared by diluting a prior art oil composition which has the composition set out in Table 4 :

| TABLE 4                                                      |               |
|--------------------------------------------------------------|---------------|
| Ingredients                                                  | Content (wt%) |
| Paraffinic base oil (25 cSt at 40°C)                         | 48.5          |
| Naphthenic base oil (115 cSt at 40°C)                        | 13.0          |
| Naphthenic base oil (8 cSt at 40°C)                          | 20.0          |
| Ditertiobutyl paracresol                                     | 0.2           |
| Organic fatty acid (C <sub>12</sub> to C <sub>18</sub> )     | 11.6          |
| Dialkylene glycol                                            | 2.1           |
| Trialkanol amine (C <sub>2</sub> to C <sub>4</sub> )         | 2.6           |
| Trialkylphosphate ester (C <sub>3</sub> to C <sub>10</sub> ) | 2.0           |

Two emulsions are prepared by respectively diluting the oil compositions of the invention and of the prior art in dionized water.

Measurements with a Sephy Zetameter show that the Zeta potential of both emulsions is -62mV, which means that these emulsions have a high stability.

Both emulsions are evaluated on an industrial test mill. The rolling conditions were as follows :

- mill type : 2 high
- motor output : 45 kW or 67 kW
- roll diameter : 760 mm
- roll hardness : 58-61 Rockwell C
- maximal width of metal : 685 mm
- typical width of metal : 305 mm
- maximum speed : 30 m/min
- entrance temperature (ingot) : 450°C

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- ingot dimensions : 305 x 610 x 1650 mm
- final thickness : 25,4 mm
- emulsion volume : 400 l
- emulsion temperature : 50°C
- 5        - emulsion concentration : 5%

The following procedure was used with each oil in the rolling tests :

1. The AA5182 blocks were lightly scalped, degreased with methyl ethyl ketone and heated to 454°C.
- 10    2. The mill rolls were washed in a dilute solution of sodium hydroxide to remove the roll coating from any previous rolling and then rinsed. The rinse water was checked for residual caustic. A profilometer was used to measure surface roughness.
- 15    3. The rolls were preheated to 77°C using quartz-tube heaters.
4. The coolant sprays were adjusted to give a flow of 200 l/min with a pressure of  $6.9 \cdot 10^4$  Newton/sq. meter (10 psig) on the top spray header, and  $4.1 \cdot 10^5$  Newton/sq. meter (60 psig) on the bottom header.
- 20    5. A 100 cm (4-inch) thick block of AA5182 was given 5 rolling passes using the following nominal pass schedule. Mill speed was 18.3 m/min. All passes were in the east to west rolling direction. The mill gap setpoints were recorded for the first emulsion tested and then duplicated for the remaining emulsions. These settings were 1.27 mm below the nominal desired exit thickness on each pass.
- 25    30    35    6. A 600 mm long piece of metal was sheared from the mid-length of the 25 mm thick piece to be used

later for anodizing. The two remaining pieces were put back into the furnace for reheating.

7. A second block was given the same treatment as indicated in items 5 and 6. Oil concentrate was added to the emulsion to obtain a 7% volume concentration.

8. The two pieces from each of the original blocks were then rolled after reheating at a mill speed of 18.3 m/min using the following nominal pass schedule :

|        |                |
|--------|----------------|
| Pass 6 | 25 mm - 16 mm  |
| Pass 7 | 16 mm - 9.5 mm |
| Pass 8 | 9.5 mm - 5 mm  |

9. After the last pass, two 600 mm long pieces were hot sheared from each of the pieces rolled. This metal was saved for later inspection in the as-rolled and anodized condition. The distance between two marks scribed on the roll was measured on the strip surface following the last pass for use in calculating forward slip.

10. The top work roll was then sampled via caustic extraction of a known area for the later measurement of the aluminium deposited on it.

Figure 1 is a graph showing the applied rolling force (in tons) applied on an aluminium alloy AA5182 versus the number of passes.

As can be seen, an improvement of 2.2% on average is achieved with the emulsion of the invention over the emulsion of the prior art.

Figure 2 is a graph showing the applied net rolling power (total power minus bearing losses, in kW) versus the number of passes.

An improvement of 5.0% on average is achieved with the emulsion of the invention over the emulsion of the prior art.

A pilot anodizing line was used to anodize approximately 150 mm of the length of the 25 mm thick and 5

mm thick pieces (see above point 6) to highlight pickup and surface appearance. The procedure was :

1. Clean for 3 minutes using a non-etching alkaline cleaner.
- 5 2. Rinse twice.
3. Nitric acid desmut for two minutes.
4. Rinse twice.
5. Anodize in 15% sulfuric acid at 15 volts for 10 minutes.
- 10 6. Rinse twice
7. Dry

The results show that the brightness and uniformity of the surface finish of an aluminum or aluminium alloy sheet after rolling with the emulsion of the invention was  
15 similar to the brightness obtained with the emulsion of the prior art.

A roll coating measurement was carried out as follows.

1. A plexiglass, gasketed fixture was attached and sealed to the center of the top work roll. This  
20 fixture includes a reservoir covering an area of 20.26 square centimeters of the roll surface.
2. Fifteen milliliters of 1N sodium hydroxide was syringed into the reservoir where it was permitted to react with the aluminum roll coating on the  
25 roll surface for approximately 5 minutes.
3. The caustic liquor was then extracted via syringe from the reservoir and placed into a sample bottle.
4. Two 15 ml rinses with deionized water were  
30 applied, extracted with a syringe and added to the sample bottle.
5. Total aluminum in the sample was determined via ICP.
6. The roll coating weight was then calculated and  
35 expressed as milligrams of aluminum per square centimeter of roll surface.

The roll coating was found to be better with the invention than with the emulsion of the prior art.

CLAIMS

5           1. Hot rolling process for rolling aluminium and  
aluminium alloys sheets, comprising applying an effective  
amount of an oil-in-water emulsion comprising water and a  
water-soluble oil composition comprising a base stock oil  
and, based on the total weight of the water-soluble oil  
10 composition, from 1 to 80% by weight of di(2-ethylhexyl)  
adipate ester.

          2. Hot rolling process according to claim 1, wherein  
the water-soluble oil composition further comprises, based  
15 on the total weight of the composition, from 0,05 to 20%,  
preferably from 0,1 to 5% by weight of a non-ionic  
surfactant, preferably an ethylene oxide addition polymer.

          3. Hot rolling process according to claim 1 or 2,  
20 wherein the water-soluble oil composition comprises, based  
on the total weight of the composition, from 3 to 30% by  
weight of di(2-ethylhexyl) adipate ester.

          4. Hot rolling process according to any one of claims  
25 1 to 3, wherein the water-soluble oil composition further  
comprises, based on the total weight of the composition,  
from 1 to 30%, preferably from 5 to 20% by weight of oleic  
acid.

30           5. Hot rolling process according to any one of claims  
1 to 4, wherein the water-soluble oil composition further  
comprises a trialkanolamine ( $C_{2-4}$ ), preferably  
triethanolamine, in such an amount that all bindable  
trialkanolamine is bound to a part of the oleic acid.

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          6. Hot rolling process according to any one of claims  
1 to 5, wherein the oil-in-water emulsion comprises water

and from 0.5 to 30%, preferably from 1 to 15% (v/v) of said water-soluble oil composition.

7. Hot rolling process according to any one of claims 1 to 6, wherein the hot rolling process is carried out in a breakdown mill and the emulsion comprises, based on the total volume of the emulsion, from 2 to 4% by volume of said water-soluble oil composition.

8. Hot rolling process according to claim 11, wherein the hot rolling process is carried out in a finishing or tandem mill and the emulsion comprises, based on the total volume of the emulsion, from 5 to 7% by volume of said water-soluble oil composition.

9. Water-soluble aluminium and aluminium alloys hot rolling oil composition comprising, in weight percentages based on the total weight of the composition :

- 0.1-0.5% of trialkyl (C<sub>1-4</sub>) phenol;
- 0.5-4.0% of trialkyl (C<sub>3-10</sub>) phosphate ester;
- 5-15% of di(2-ethylhexyl) adipate ester;
- 5-15% of organic fatty acid (C<sub>12-20</sub>);
- 0.5-2% of 5-carboxy 4-hexyl 2-cyclohexen 1-octanoic acid ;
- 1-3% of alkylene (C<sub>2-6</sub>) glycol;
- 0.3-1% of ethoxylated alcohols (C<sub>5-14</sub>, comprising 2-10 CH<sub>2</sub>O groups);
- 2-5% of trialkanol amine (C<sub>2-4</sub>);
- the balance being a mixture of naphthenic and paraffinic lube base oils.

10. Water-soluble oil composition according to claim 9, in which the base stock oil has a viscosity comprised between 7 and 150 cSt at 40°C, preferably between 20 and 50 cSt at 40°C.

11. Process for the preparation of a water-soluble oil composition according to claim 9 or 10, comprising

blending the base stock and the other ingredients under stirring or with any mixing device.

5       12. Oil-in-water emulsion comprising water and from 0.5 to 30%, preferably from 1 to 15% (v/v) of the water-soluble oil composition according to claim 9 or 10.

10       13. Process for the preparation of an oil-in-water emulsion according to claim 12, comprising diluting the oil composition in water under stirring.

15       14. Use of the water-soluble oil composition according to claims 9 or 10 to prepare emulsions intended to be used in a aluminium or aluminium alloy hot rolling process.

      15. Use of the water-in-oil emulsion of claim 12 in a hot rolling process.