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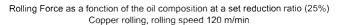
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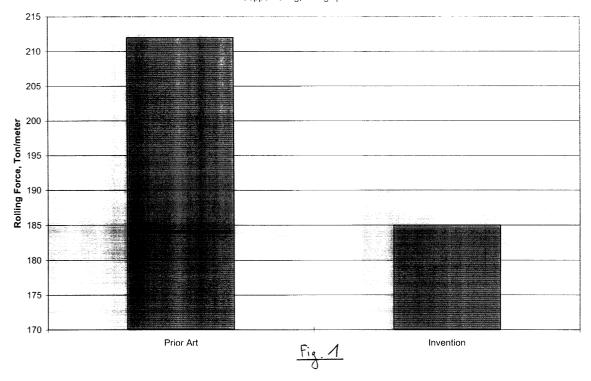
(54) Copper and non ferrous alloys cold rolling oil composition

(57) The present invention relates to a copper and nonferrous alloys cold rolling oil composition comprising a base stock oil and, based on the total weight of the composition, from 1 to 80% by weight of di(2-ethylhexyl)

adipate.

The invention also relates to a process for cold rolling copper and nonferrous alloy sheets and to the use of said oil composition in a copper, copper alloys and nonferrous metals cold rolling process.





Description

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[0001] The present invention relates to a copper and non ferrous alloys cold rolling oil composition and to a process of cold rolling copper and non ferrous alloy sheets.

[0002] The copper and non ferrous alloys rolling industry expresses the need to maximize the efficiency of their rolled metal manufacturing process. In general terms, this means that they wish to operate at higher rolling speeds and to produce more marketable products per operating shift. Additionally, they also 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. Also, there is a wish to roll harder materials, such as special non ferrous alloys (e.g. copper alloys such as bronze) and/or allow higher reduction ratios.

[0003] The invention thus provides an oil composition for copper and alloys thereof cold rolling mills that afford the following customer benefits:

- lower rolling and reduced mill power (this allowing rolling harder non-ferrous alloys and/or allow higher reduction ratios);
- allow one or two pass(es) reduction versus conventional oil lubrication;
- improved rolled surface finish by minimizing the sticking tendency of the strip when performing decoiling after heat treatment.

[0004] The invention is effective on any type of cold rolling, be it reversible or not, of the Sendzimir type (e.g. 1-2, 1-2-3, 1-2-3-4), or of Z-high type or 2-high, 4-high, 6-high mills, be it a reversible mill, a tandem mill, etc..

[0005] Especially, the invention is specially designed for copper and copper alloys for 2-high, 4-high and 12-high mills. The invention provides improved copper strip surface finish by minimizing the sticking tendency after thermal treatment of the rolled coils and excellent rolling capabilities by reducing the rolling force and mill power required when rolling harder copper alloys.

[0006] Thus, the invention provides a copper and copper alloys cold rolling oil composition comprising a base stock oil and, based on the total weight of the composition, from 1 to 80%, preferably from 1 to 30% by weight, of di(2-ethylhexyl) adipate.

[0007] According to one embodiment, the oil composition further comprises an alkyl alkylate ester, in which the alkyl comprises 2 to 8 carbon atoms and the alkylate comprises 14 to 24 carbon atoms, preferably n-butyl, iso-butyl, or tert-butyl stearate, and where the ratio di(2-ethylhexyl) adipate:alkyl alkylate ester is from 1:1 to 20:1.

[0008] According to a further embodiment, the oil composition further comprises a fatty alcohol having from 12 to 18 carbon atoms, preferably from 12 to 14 carbon atoms, most preferably lauryl alcohol.

[0009] The invention also provides a cold rolling process for rolling copper and copper alloys sheets, comprising applying an effective amount of the oil composition of the invention.

[0010] Further, the addition provides a process for the preparation of an oil composition comprising blending the base stock and the other ingredients under stirring or with any mixing device.

[0011] Finally, the invention provides the use of the oil composition of the invention in copper and copper alloys cold rolling process.

[0012] The invention is now disclosed in more details in the following specification, and in reference to the accompanying Figure 1, which 1 is a graph showing the rolling force as a fonction of the oil composition at a set reduction ratio (25%).

[0013] The oil compositions of the invention are neat oils.

[0014] The base stock oil is any oil typically used in the field of cold rolling. It can be paraffinic or naphthenic, hydrocraked or not.

[0015] 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 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 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.

[0016] 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.

[0017] A preferred base oil is an hydrotreated paraffinic neutral.

[0018] The base oil typically has a viscosity from 1.5 to 20 cSt at 40°C, preferably from 1.5 to 12 cSt at 40°C. Viscosity can be adjusted by using a viscosity adjuster (such as kerosene), if needed.

[0019] The oil may comprise classical additives, such as surfactants, coupling agents or cosurfactants, friction re-

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ducing agents, lubricity agents, corrosion inhibitors or anti-oxidants, extreme-pressure and anti-wear agents, bactericides and fungicides, anti-foaming agents, anti-rust agents, passivating agents.

[0020] Examples of anti-foaming agents are silicone based, especially polydimethylsiloxane.

[0021] Examples of corrosion inhibitors are hindered phenols and zinc dialkyldithiophosphates (ZDDP).

[0022] Examples of extreme-pressure and anti-wear agents are dilauryl phosphate, didodecyl phosphite, trialkyl-phosphate such as tri(2-ethylhexyl)phosphate, tricresylphosphate (TCP), zinc dialkyl(or diaryl)dithiophosphates (ZD-DP), phospho-sulphurized fatty oils, zinc dialkyldithiocarbamate), mercaptobenzothiazole, sulphurized 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.

[0023] 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.

[0024] Examples of anti-rust agents are amine derivative of alkenyl succinic anhydride.

[0025] Examples of passivating agents are benzotriazole, tolyltriazole, toluol triazole, 2-(5-aminopentyl)benzimidazole, Irgamet® 39 (1H benzotriazole 1 methanoimine,N,N, bis (2-ethylhexyl)methyl and Irganox® MD1024.

[0026] Examples of friction reducing agents or lubricity agents are fatty alcohols having a carbon number in the range from 12 to 18, and fatty esters having a carbon number in the range from 12 to 18, like glycerol monooleate.

[0027] Other additives which are efficient in bondary type lubrication can be used.

[0028] Further elements on base oils and additives can be found in "Chemistry And Technology Of Lubricants", R. M. Mortier and S.T. Orszulik, VCH Publishers, Inc, First published in 1992 and in Tribology International Paper, Vol. 30, Number 12, 1997, pages 881-888.

[0029] The copper and copper alloys and non ferrous alloys to which the invention applies are any alloys, including hard alloys, as well as conventional alloys such as brass and bronze.

[0030] The oil composition of the invention can also be used in a process for cold rolling non-ferrous metals like nickel and zinc.

[0031] The cold rolling process is the classical process. The oil temperature is generally maintained at a temperature below 70°C, preferably below 50°C. The process can be carried out on any rolling mill, such as of the Sendzimir type or of the Z-high type or more generally from 2-high to 20-high mills, in tandem, etc.. The instant oil composition allows a significant reduction of the number of passes. With conventional prior art oils, the number of passes is in the range from 3 to 10. The oil composition of the invention allows reducing this number by 1 to 2 passes, which is a significant gain. **[0032]** The following examples illustrate the invention without limiting it. All parts and ratios are given by weight.

Example

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[0033] The following composition of the invention was prepared:

Ingredients	Content (wt%)
Base oil (paraffinic, 9 cSt at 40°C)	86.65
1H-benzotriazole 1-methanoimine N,N, bis (2-ethylhexyl) methyl (metal passivating agent)	0.10
Butylated hydroxytoluene (anti-oxidant)	0.20
tri(2-ethylhexyl)phosphate ester (extreme-pressure agent)	1.00
Amine derivative of alkenyl succinic anhydride (anti-rust agent)	0.05
n-butyl stearate (lubricity agent)	2.00
di(2-ethylhexyl) adipate (lubricity agent)	8.00
Lauryl alcohol (lubricity agent)	2.00

[0034] The following composition of the prior art was prepared:

Ingredients	Content (wt%)
Base oil (paraffinic, 9 cSt at 40°C)	97.40
Lauryl alcohol (lubricity agent)	1.00

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(continued)

Ingredients	Content (wt%)
n-butyl stearate ester (lubricity agent)	1.00
Tri(2-ethylhexyl)phosphate ester (extreme-pressure agent)	0.50
Butylated hydroxytoluene (anti-oxidant)	0.10

[0035] Said invention and prior art compositions were tested according to the following method.

[0036] The test mill was a non-reversing single stand 2-high rolling mill with coiler and decoiler designed for 30 mm wide sheets, which can take up to 0.6 mm thick strips of around 1,000 m length. The rolls had a width of 100 mm and a diameter of 95 mm, and the composition of their steel is Z85VCD8-3 (which is used for certain Sendzimir mills).

[0037] Pure copper coils having an initial thickness of 1.5 mm were rolled at a rolling speed of 120 m/min applying a reduction ratio of 25% per pass.

[0038] The rolling force was recorded for the oil composition of the invention and the prior art.

[0039] The results are shown on Figure 1.

[0040] A 15% reduction in rolling load is obtained with the oil composition of the invention, which is a significant gain.

Claims

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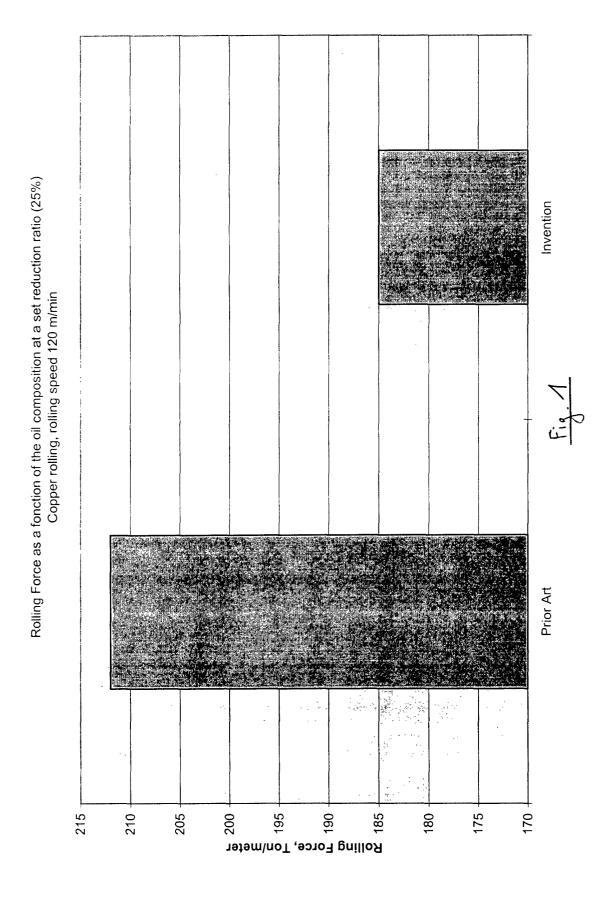
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- 1. Copper and copper alloys cold rolling oil composition comprising a base stock oil and, based on the total weight of the composition, from 1 to 80% by weight of di (2-ethylhexyl) adipate.
- Oil composition according to claim 1, comprising, based on the total weight of the composition, from 1 to 30% by weight of di(2-ethylhexyl) adipate.
 - 3. Oil composition according to claim 1 or 2, further comprising an alkyl alkylate ester, in which the alkyl comprises 2 to 8 carbon atoms and the alkylate comprises 14 to 24 carbon atoms, and where the ratio di(2-ethylhexyl) adipate: alkyl alkylate is from 1:1 to 20:1.
 - **4.** Oil composition according to claim 3, in which the alkyl alkylate ester is n-butyl, iso-butyl or tert-butyl stearate.
 - 5. Oil composition according to any one of claims 1 to 4, further comprising a fatty alcohol having from 12 to 18 carbon atoms, and preferably from 12 to 14 carbon atoms.
 - **6.** Oil composition according to claim 5, in which the fatty alcohol is lauryl alcohol.
- 7. Oil composition according to any one of claims 1 to 6, in which the base stock oil has a viscosity comprised between 1.5 to 20 cSt at 40°C, and preferably between 1.5 to 12 cst at 40°C.
 - **8.** Process for the preparation of an oil composition according to any one of claims 1 to 7, comprising blending the base stock and the other ingredients under stirring or with any mixing device.
 - **9.** Cold rolling process for rolling copper and copper alloy sheets, comprising applying an effective amount of the oil composition according to any one of claims 1 to 7.
 - 10. Use of the oil composition according to any one of claims 1 to 7 in copper and copper alloys cold rolling process.
 - 11. Use of the oil composition according to any one of claims 1 to 7 in a non-ferrous metals cold rolling process.

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EUROPEAN SEARCH REPORT

Application Number EP 00 40 0350

	DOCUMENTS CONSID	ERED TO BE RELEVAN	<u> </u>	
Category	Citation of document with i of relevant pass	ndication, where appropriate, sages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.CI.7)
X	DD 127 620 A (ANDRE 5 October 1977 (197 * page 4, paragraph 1; example 8 *			C10M129/72 C10M111/02 C10M169/04 //(C10M111/02, 101:02,
X	DATABASE WPI Section Ch, Week 19 Derwent Publication Class E17, AN 1999- XP002141643 & JP 10 298578 A (N 10 November 1998 (1 * abstract *	s Ltd., London, GB; 041018 IPPON OIL CO LTD),	1,2,5-11	
X	19 March 1991 (1991 * column 5, line 45 * column 6, line 55	- column 6, line 8 *		
X A	4 June 1991 (1991-0 * column 1, line 12 * column 2, line 62		*	TECHNICAL FIELDS SEARCHED (Int.CI.7)
X	EP 0 332 433 A (GRA 13 September 1989 (* column 3, line 14		* 1,2,7-11	
A	US 3 840 462 A (GER 8 October 1974 (197 * column 1, line 58		* 1-11	
A	23 May 1989 (1989-0 * column 1, line 66	- column 2, line 19 - line 30; table 1 *	*	
		-/		
	The present search report has	•		
Place of search Date of completion of the				Examiner
X : parti Y : parti docu A : tech	THE HAGUE ATEGORY OF CITED DOCUMENTS icularly relevant if taken alone icularly relevant if combined with anotument of the same category nological background—written disclosure	E : earlier paten after the fillin her D : document ci L : document cit	nciple underlying the i it document, but publi	shed on, or

EPO FORM 1503 03.82 (P04C01)



EUROPEAN SEARCH REPORT

Application Number EP 00 40 0350

Category	Citation of document with income of relevant passa	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.CI.7)		
Α	EP 0 315 873 A (IDEM 17 May 1989 (1989-05 * page 2, line 41 - * page 3, line 35 -	ITSU KOSAN CO) -17) line 45 *	1-11		
				TECHNICAL F SEARCHED	ELDS (Int.Cl.7)
	The present search report has be	een drawn up for all claims Date of completion of the search		Examiner	
	THE HAGUE	3 July 2000	Rot	saert, L	
X : part Y : part docu A : tech O : non	ATEGORY OF CITED DOCUMENTS icularly relevant if taken alone icularly relevant if combined with another ment of the same category inclogical background—written disclosure mediate document	T: theory or princip E: earlier patent de after the filling de or D: document cited L: document cited	ole underlying the in ocument, but public ate in the application for other reasons	nvention shed on, or	

EPO FORM 1503 03.82 (P04C01)

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 00 40 0350

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

03-07-2000

Patent document cited in search repo		Publication date	Patent family member(s)	Publication date
DD 127620	Α	05-10-1977	NONE	
JP 10298578	Α	10-11-1998	NONE	
US 5001013	Α	19-03-1991	NONE	
US 5021172	Α	04-06-1991	US 5132032 A	21-07-199
EP 0332433	А	13-09-1989	US 4882077 A AU 3074589 A BR 8901073 A JP 1263194 A NZ 228067 A	21-11-198 14-09-198 04-09-199 19-10-198 26-04-199
US 3840462	Α	08-10-1974	AT 323306 B	10-07-197
US 4832860	Α	23-05-1989	JP 2021477 C JP 7045674 B JP 62201994 A KR 9004530 B	19-02-199 17-05-199 05-09-198 29-06-199
EP 0315873	Α	17-05-1989	JP 1123897 A JP 2501217 B DE 3867433 A KR 9310573 B US 4828731 A	16-05-198 29-05-199 13-02-199 28-10-199 09-05-198

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82