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OPTIMIZE OF ROLLING LUBRICANT BY FOURIER TRANSFORM INFRARED REFELECTION SPECTROSCOPY

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ABSTRACT

Cold rolling manufacturing process, a major issue is roll elastic deformation and its impact on roll load, torque and interaction stresses. As in many systems implying mechanical contact under high loading, a central part is under “sticking friction” (no slip) whereas both extremities do slip to accommodate the material acceleration of the rolled metal sheet. During the cold reduction of steel sheet in a rolling mill, rolling oils are used for lubrication to offer the desired steel surface. Rolling oil formulations consist largely of mineral oils, glycerides and/or synthetic esters, fatty acids, emulsifiers and extracts. These oils are generally used as 3-7% emulsions and will be subjected to high temperature and pressure during use. Evidence was found that the bio composite oils degraded on heating and the spectroscopic investigation indicated that this was due to rearrangement of unsaturated components configuration. In this work bio based composite oil were comparing with synthetic oil and optimize for preeminent performance on rolling operation.

INTRODUCTION

Cold rolling operations associated with the flat processing of steel products involve the use of rolling lubricants. These resources, usually in the form of fatty or mineral oils, applied either neat or in emulsion form, greatly assist the reduction of the strip in that they considerably reduce the rolling force required for deformation. By so exploit, they also (a) make the attainment of acceptable “shape” or flatness in the rolled product greatly easier, (b) lessen roll

wear, (c) reduce roll and strip temperature, and (d) prevent rusting of the reduced strip.

Deprived of knowledge of the effectiveness of the rolling lubricants used, the satisfactory operation of commercial rolling facilities becomes more challenging to achieve. From an economic viewpoint, it is desirable to use the inexpensive material that will provide adequate lubrication in the roll piece. Generally speaking, lubricant consumption choices from 4 to 8 lbs. per ton of steel rolled. From a practical perspective, reduction in the quantity of rolling lubricant castoff is not always feasible because of the “graft out” of the lubricant on the surface of the rolled strip, due to the skimming of the lubricant tanks and because of the additional operations necessary to maintain the aqueous dispersal of the lubricant in a satisfactorily clean situation. Accordingly, it is often easier to achieve economies by shifting the nature of the rolling lubricant rather than by tumbling its consumption.[1]

Although cold rolling lubricants have been extensively used since the early 1930’s, research into their activities was not undertaken to any substantial extent until the late 1940’s. Even then, for a diversity of reasons, the research data was often difficult to interpret or relate to other rolling specifications. Moreover, because of improper assumptions relating to rolling models, computed values of the coefficient of friction on the roll bite were often of questionable accuracy.

The main drive to investigate the poor steel cleanliness phenomenon was initiated by the cold rolling mills supplying coating lines and the automotive industry. Therefore, the initial steel cleanliness problem concerns the rolling oil composition. The most used quality analysis of the rolling oils during the early 1980’s was by measuring the pH, acid value, saponification value, oil content, etc. These indicative methods only give the total change in the amounts of acids and esters contained in the oil. This type of system specific research, limits the use and implementation of the results obtained. Due to the unique demands from each cold rolling mill product, oils are formulated and manufactured according to the type of mill and method of application, resulting in a large range of different products with compositions unknown to the steel industry.

The oil/surface interaction

The properties of the organic residues on the steel surface are important in interpreting the steel surface cleanliness results. The lubricant, usually applied as an emulsion, should form an adequate film in the inlet of the roll bite. This forming of a film on the steel surface is mainly

governed by emulsion properties such as plate-out (homogeneous covering of the steel surface with a thin layer of oil). Plate-out at a given concentration and temperature determines how much oil is delivered to the strip from the emulsion. Tanikawa and his co-worker's¹⁰ attention was focused on organic carbon, one of the surface residues that adversely affected the surface cleanliness of cold rolled steel sheet, and the property alteration behaviour of the rolling oil adherent to the steel in the as rolled condition during annealing was analysed by analytical techniques. Many reports were published that analysed inorganic carbon deposited on the sheets by such techniques as Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS),¹⁴ but organic carbon was not discussed in depth.

OBJECTIVE

The objective with the choice of experimental material, the experiment devised and the analytical technique used, was to determine the influence on steel surface cleanliness of the cold rolled steel with regard to the oxidation stability of the cold rolling oils. The large number of lubricant combinations possible, the number of possible environments and the absence of standardized neat oil evaluation tests for steel surface cleanliness predictions, all add to the uncertainties regarding the use of lubricants on steel surfaces. As will be shown later, the evidence indicates that the poor steel surface cleanliness experience in the cases covered in this dissertation is related to the unsaturated rolling oils. Owing to this fact, an analytical technique was intensively applied and certain aspects of this technique are discussed.

However, only vegetable oil is used in this study, because the poor oxidation stability problem that initiated this investigation occurred after the use of vegetable oils. During the cold rolling process, a high temperature of $\approx 400^{\circ}\text{C}$ is established, as rolling procedure involves continuous forward and backward extrusion of metal, which generates friction between the rolling surface and the strip. Before the usage of cold rolling oil the steel is covered with rust preventative oil. This oil layer serves two purposes, namely:

- (a) Protecting the active, pickled surface from rust formation and,
- (b) Serving as a lubricant during the first stage of the cold reduction process.

Fourier Transform Infrared Spectroscopy

Most of the analysis in this study was carried out on this apparatus, because of its chemical sensitivity. Infrared radiation lies in the Electromagnetic Spectrum between the visible region

and the microwave region. It is measured in units of frequency, wavenumber or wavelength, which are interrelated by the following equations: -

$$F\lambda = c$$

$$v = 1/\lambda$$

$$f = cv$$

Where,

F is frequency in Hz., cycle/second or its multiples

c is the velocity of light in a vacuum

v is the wavelength, f is the wavenumber

The infrared is divided into three regions: Near, Mid and Far. Interest in the NIR has grown in recent years, particularly for process control, raw materials and finished product analysis, especially in the food and feedstuffs industries, where it is routinely used for qualitative analysis 30. The Far infrared is principally concerned with rotational spectra, and crystal lattice vibrations. The Mid infrared is by far the most widely used region as it is involved with molecular vibrations typically found in organic molecules. It provides a wealth of structural information, as well as quantitative data.

When infrared radiation is directed on a sample it is either transmitted or absorbed by the sample or reflected from it. The total incident radiation is I_0 . On placing the detector behind the sample a measurement of radiation occurs. This is the transmitted radiation I . The detector ensures normally measure the reflected/scattered radiation S .

$$T = I/ I_0$$

$$\%T = 100I/ I_0$$

$$\text{Absorbance} = \text{Log} (1/T)$$

$$\text{Absorbance} = \text{Log} (100/\%T)$$

The Fourier transform infrared spectrometer measures a background Interferogram (to give I_0), then a sample Interferogram (to give I) and from this data calculates the percentage transmission of the sample. The total incident radiation is described by this formula:-

$$I_0 = I_R + I_A + I_S + I_T$$

Where;

- I_R = Reflected beam

- I_A = Absorbed beam
- I_S = Scattered beam
- I_T = Transmitted beam

The infrared light beams are schematically shown below.

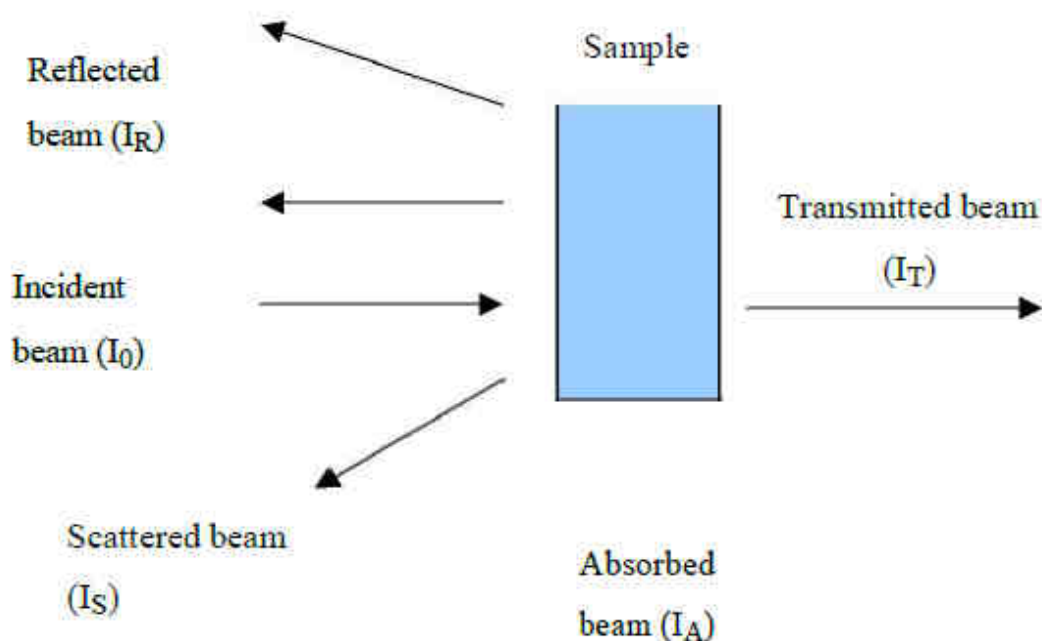


Figure 1: Schematic representation of the infrared light beams

Virtually all-infrared spectrometers that are commercially available use the Fourier Transform process. In the case of Perkin-Elmer infrared spectrometer (which we are currently using) an Interferogram of the instrument background is measured and transformed to give a single beam energy spectrum, then the Interferogram of the sample is measured and transformed. This is then ratioed with the background spectrum to give a recognizable transmittance spectrum as the data shown on the spectrometer screen or hard copy device. The schematic FT-instrumentation is shown below where:

- The Source generates light across the spectrum of interest
- Beamsplitter sends the light into two directions at right angles.
- The sample absorbs all the different wavelengths characteristic of its spectrum
- The detector collects the radiation that passes through the sample and
- An electrical signal is put out and send directly to the computer.

One task of the spectroscopists is to obtain information about the molecular structure of an unknown molecule, by deducing how the observed absorption bands in the spectrum relate to the possible vibrational modes of the molecule.

Assignment of infrared bands of rolling oils

The assignments of the characteristic infrared bands of base oil and ester components of rolling oil are shown in Table 5.1 below

Table 1 IR vibrational assignments of rolling oil

Composition of Rolling oil	Band position (cm ⁻¹)	Intensity	Assignment
Base oil - Aliphatic Components	3100-3000	M	C-H stretch adjoining C=C
	3000-2800	S	CH and CH stretching
	1460	M	CH and CH deformation
	1378	M	CH deformation
	720	W	CH rocking in long chains
	Base oil - Aromatic Components	3100-300	Sh
1605		W	Aromatic ring stretching
			Out of plane vibration of single hydrogen on aromatic ring
865		S	
815		S	Out of plane vibration of two adjacent hydrogens
750		S	Out of plane vibration of four adjacent hydrogens
Esters	3520	w/m	Free -O-H Stretching of hydrogen bonded to O-H group, overlap with H ₂ O
	2500-3300	M	
	3400-2600	M	O-H stretch of an acid
	3470	W	Overtone of C=O ester stretch
	3008	M	C-H stretch of unsaturated acid component

	3000-2800	S	CH and CH stretch overlap with base oil
	1745	Vs	C=O stretch of an ester
	1710	Vs	C=O stretch of an acid
	1656	W	C=C stretch of unsaturated acid component
	1460	M	CH and CH deformation, overlap with base oil
	1378	W	CH deformation, overlap with base oil
	1170	S	C-O stretch of an ester group
	1120-900	m/w	Other less intense absorptions of ester.
	720	w/m	CH rocking in long chains

Rolling oil with polyunsaturated fatty acids

Atmospheric oxidation is the most important cause of deterioration in lubricants ³⁵. This oxidative rancidity is accelerated by exposure to heat, light, humidity and the presence of trace metals. The intense rolling of steel with the oil used as lubricant causes an oxidizing thermal degradation with formation of decomposition products, and change in physical properties such as colour and odour. When oxygen travels through oil, it is absorbed by the oil and reacts mainly with the C=C double bond, thus the polyunsaturated components of the oil oxidize much faster than saturated ones. Spectroscopic methods can be employed to evaluate deterioration in oils subjected to heat. Fourier transform infrared spectroscopy (FTIR) provides a quick and accurate way of evaluating thermal degradation in these lubricants.

Oxidation of rolling oils and polyunsaturated fatty acids

The oxidative deterioration of polyunsaturated fatty acids in cold rolling oils has been monitored by Fourier transform infrared spectroscopy (FTIR). The thermal oxidation of polyunsaturated fatty acid is a free radical chain reaction, in which hydroperoxides are generally recognized as the primary major product ³⁶. The hydroperoxides of the polyunsaturated fatty acids are easily decomposed into a very complex mixture of secondary products with the decrease in saturation. The oxidative advance of unsaturated fatty acids during heating was studied by the determination of non-saturation at different temperatures and heating times.

Cis – trans rearrangement

A comparison between the unsaturated acid (oleic acid) and the saturated acid (palmitic acid) was necessary to confirm the formation of the trans isomer. This comparison is shown in figures 2. The IR spectra of oleic acid shows the C-H band at 965 cm⁻¹, which is characteristic of the

trans isomer of the unsaturated non-conjugated C=C double bond. The same band was observed in vegetable-based rolling oils. No significant changes were seen with the saturated acid (palmitic acid, Fig.2). The results for stearic acid, myristic acid and lauric acid are not shown, as they are similar to that of the palmitic acid.

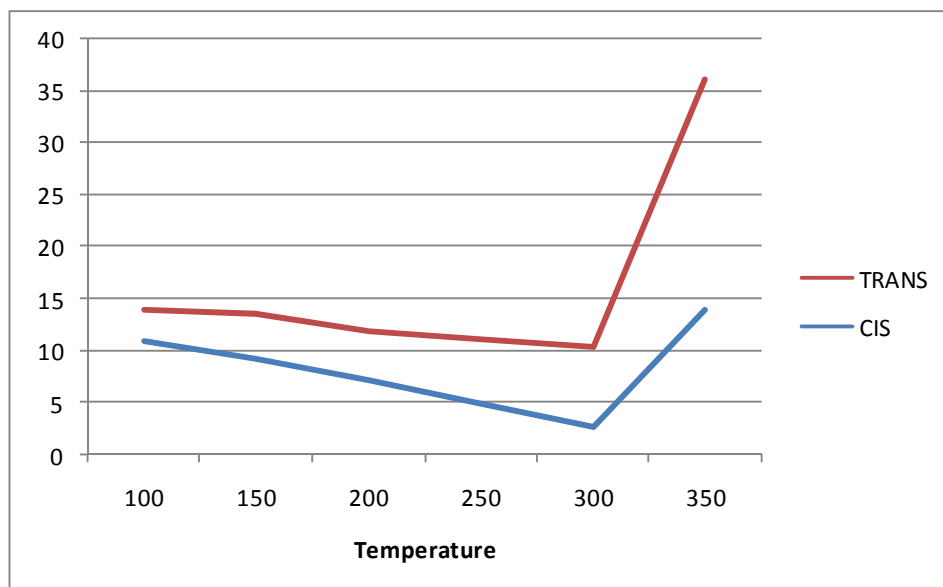


Figure 2 Comparisons of CIS and TRANS Configuration

The cis isomer of the unsaturated double bond compound can be seen for the vegetable-based rolling oil and the oleic acid, at ambient temperature. The results show that the cold rolling process causes a decrease in the cis isomer and an increase in the trans isomer. In all the oils studied, degradation becomes considerable after 150°C. A significant decrease in cis isomer, approximately 70%, is apparent at high temperature. The reason for the thermal and oxidative instability of the vegetable-based rolling oils is the unsaturated double bond in the fatty acid.

CONCLUSION

The cis-trans isomer observed in the rolling oil was due to changes in the unsaturated fatty acids. It can be said that when an unsaturated rolling oil is subjected to a heating process, the initial nature of the oil changes significantly and the decrease in unsaturated compound and increase in the trans isomer is evidence of the transformation occurring in the essential polyunsaturated fatty acids which the oil initially contains, thus indicating a decrease in the oil's performance capacity.

- (a) If the process currently used on the cold rolling mill remains the same, the poor steel surface cleanliness of the steel produced on that line must be taken as characteristic of rolling oil

used on that line.

- (b) The determination of unsaturated compounds in rolling oils makes it possible to classify them and evaluate their oxidative deterioration which is directly related with the degradation of polyunsaturated fatty acids in the oils.

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