

CHARACTERISATION OF THE AGEING OF COLD ROLLING EMULSIONS BY
DIFFERENTIAL SCANNING CALORIMETRY

B.O.HAGLUND and T.LUKS
Sandvik AB, Coromant Research Center, P.O.Box 42056
S-126 12 Stockholm (Sweden)

ABSTRACT

In cold rolling of steel different oil-water emulsions are used for lubrication and cooling. The emulsions are subjected to ageing in use and different analytical methods are applied to control the function of the emulsions. A method is described in which the change in function is followed by determination of an "Oxidation onset temperature" of the separated oil phase of the emulsion. It is shown that this temperature is successively increased with the time of use.

INTRODUCTION

In cold rolling of strip steel the rolling mills must be lubricated and cooled by applying a liquid. This can be achieved in the form of both oil or an oil-water emulsion. The emulsion has much better cooling capacity than a pure lubricating oil. The emulsion is recirculated in a batch which normally is used several months, and then discarded due to changes in composition and/or function. Several methods are used to check the deterioration of the oil emulsion (ref.1,2). Laboratory methods have also been developed for selection of suitable rolling emulsions (ref. 3,4,5).

METHOD

A new method, based on a Mettler publication (ref. 6), determines the rate of oxidation in the extracted oil phase of the emulsion. When a sample is heated at a constant rate in pure oxygen, an increase in the oxidation rate is suddenly achieved at a certain temperature. This temperature is called the "oxidation onset temperature".

The experiments were done in a Mettler TA 2000C Thermoanalyzer for simultaneous TG-DSC. The sample weight is approximately 10 mg and the heating rate is 5 K/min to a maximum of 230°C.

SAMPLES

The samples are from new and used emulsions and from the original components. The used emulsions are not more than a half year old. The main components of the oil phase in the original sample are mineral oils, esters, tensides and corrosion inhibitors. The oil phase is removed from the oil-water emulsion samples by ether extraction (see Fig. 1).

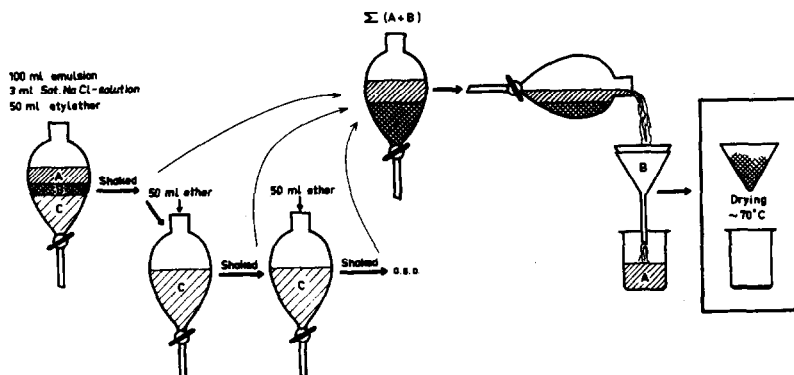


Fig. 1. Isolation of oil and "soaps & dirt" from a cold rolling emulsion through repeated extractions with ether.

The ether phase is labelled A in the figure, the soap & dirt phase is labelled B and the water phase C. (Ref.1)

RESULTS

The experimental results are:

Elapsed time days	Oxidation onset temperature °C
0	164
1/4	173
1	180
3	186
7	190
14	201
31	206
104	204

The reproducibility of the test is good and the range of three subsequent determinations is less than $3-4^{\circ}\text{C}$. Some typical examples of the oxidation curves are shown in Fig.2a-c. The increase of the oxidation onset temperature can clearly be seen. The shape of the curves also changes gradually from a small increase in the slope, of the original (unused) emulsion to a large change of the slope of the used emulsions.

The thermogravimetric curves indicate a higher content of volatile components in the original emulsion.

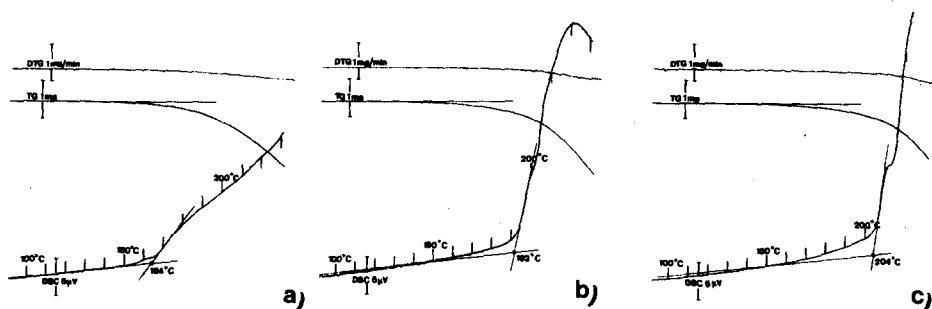


Fig.2. Oxidation stability of the oil phase of oil-water emulsions for cold rolling of steel after different periods of use. (a) From an unused emulsion. (b) After one week. (c) After 3 months.

DISCUSSION

The emulsion contains mineral or vegetable oils with a variable number of double bonds. The unsaturated oils are cheaper, but more sensitive to oxidation than the saturated oils. To improve the oxidation resistance, the unsaturated oils can be exchanged for synthetic esters. Antioxidants can also be added to prevent oxidation, but the lifetime of such additives is restricted, especially at high temperatures.

The high temperature (and possibly high pressure) which the emulsion is subjected to during use may cause a polymerization or a degradation. The composition of the emulsion may also be changed due to a continuous evaporation of the most volatile components.

The fats and fatty oils may also be subjected to a hydrolysis in the emulsion producing fatty acids and glycerol. During this process the conductivity of the emulsion is rapidly increased (ref. 1).

All these reactions will cause a successive change of the composition and properties of the emulsion. It can be seen from the TG-DSC results that there is a rapid increase in the oxidation onset temperature in the early use of the emulsion. The change of the shape of the DSC curve suggests that evaporation causes the rapid change of the emulsions in the early stages of use.

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