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THERMOANALYTICAL STUDY OF WATER-BASED ALUMINIUM ROLLING FLUIDS COMBINED WITH DIRECT OBSERVATION

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ABSTRACT

During cold-rolling of aluminium sheets the auxiliary lubricant fluids used decompose giving rise to discolouring of the sheet surface. In the present study a combined TGA-DTG-DTA analysis has been used in combination with direct observation of the colour change of carboxylate-based fluids in oxidizing and inert atmosphere to determine the temperature region of decomposition and/or polymerization. Significant effects have been observed depending not only on the type of atmosphere but also on the type of the cationic part of the organic component in these fluids.

INTRODUCTION

Cold rolling of aluminium sheet is conventionally performed using a light mineral oil fraction supplied with suitable oil-soluble polar load-carrying additives. Base oils and additives are required to be volatile at temperatures below 250 °C in order to avoid chemical reactions leading to discolouration caused by a layer of excess lubricant remaining on surface of coiled metal sheet in subsequent heat treatment.

There are a number of drawbacks inherent in hydrocarbon base fluids, such as heavy emission to the environment due to a need for effective ventilation of work areas, severe fire hazard and dissolution of non-volatile lubricants from the rolling equipment leading to dark colouring of sheet following heat treatment.

It has been long recognized that these problems could be overcome by the design of waterbased lubricants, which also would offer improved productivity due to superior cooling capacity.

Water-soluble or water-dispersible lubricant components will not easily be made volatile within the temperature range needed to avoid detrimental chemical reactions in layer deposited on metal surface. Thermoanalytical technique [1-3] provides a tool for the study of influence of chemical and physical parameters on amounts and nature of residues formed in heat treatment.

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In this paper some preliminary experiments are described.

EXPERIMENTAL PROCEDURE

The lubricant fluids used were based on an organic carboxylate (TDF):

a) carboxylate in acidic form (TDF/H)
b) carboxylate as potassium salt (TDF/K)
c) carboxylate in basic form with triethylamine (TDF/TEA).

Reference lubricant fluids (no 1 and 2) were studied with respect to the effect of substrate as described later.

Experiments were carried out in a universal micro-thermo balance METTLER TA1 with a transparent silica furnace permitting direct observation of the colour change of the fluids suspended in Al sheet pans, in addition to recording of the TGA-DTG-DTA curves. The organic components (ca 10 % by weight) were soaked into Al powder in Al crucibles and heated at a linear heating rate (6 K·min^{-1}) to 500 °C both in oxidizing (air) and inert (N_2-SR) atmosphere. The experimental parameters were the following:

TG range - 10 mg (with 1 mg expansion)
DTG range - 20 mg·min^{-1}
DTA range - 100 µV
Reference substance - Al_2O_3
Gas flow rate - ca 4.5 l/h
Mass of specimens - ca 4.5-5 mg lubricant soaked into ca 40-45 mg Al powder

In experiments involving the visual observation of colour changes of the Al surface the organic lubricant (ca 30 mg) was deposited on the Al sheet pans (30 mm in diameter) or sintered Al_2O_3 sample pans. In this case only thermogravimetric analysis was carried out. The TG range was 100 mg. The other parameters were the same as mentioned earlier.

RESULTS AND DISCUSSION

General features

Figures 1-3 show the DTA and DTG curves for the Al powder/fluid specimens and the DTG curves for the fluids suspended on the Al pans, in air and in nitrogen atmosphere, respectively. As is apparent the temperature range for the major decomposition reaction is broadened by 50-150 °C for the Al powder/ fluid specimens as compared to the fluid deposited on Al pans. The colour changes observed in situ (Fig. 1-3) indicate that the occurring weight loss mostly involve decomposition coupled with polymerization rather than evaporation of the original compound. Thus the polymerization (carbonization) resulting in discolouring of cold-rolled Al sheet seems to be correlated to the decomposition of the rolling fluids in the range of 150-250 °C (Fig. 1-3).

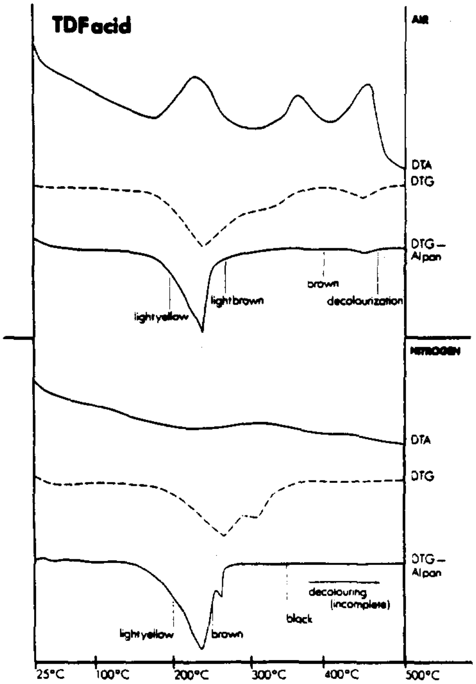

Fig. 1

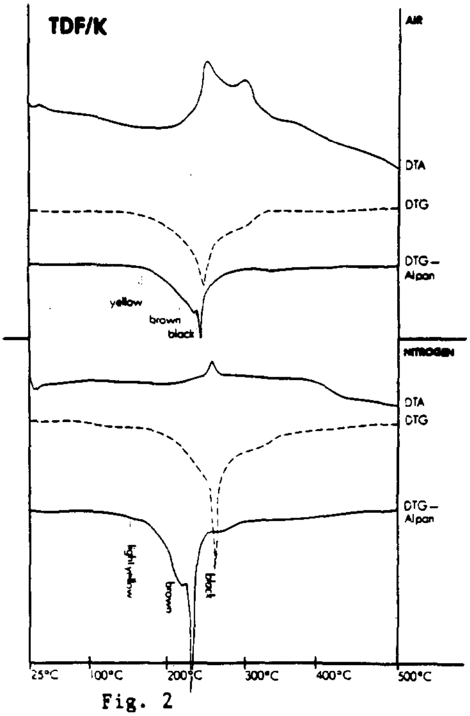

Fig. 2

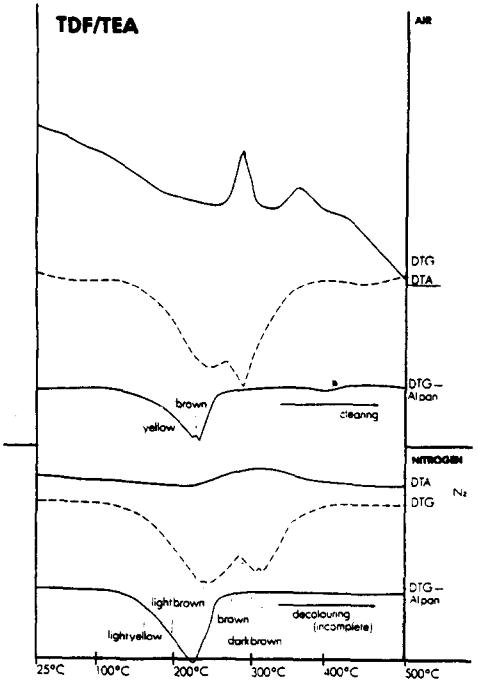

Fig. 3

Fig. 1-3. DTG and DTA curves for Al powders and DTG curves for Al pans obtained in air and in nitrogen atmosphere during reactions involving deposits of (1) TDF acid, (2) TDF/K and (3) TDF/TEA.

The difference in the decomposition path in both air and nitrogen for the fluid soaked in Al powder and deposited on an Al pan, respectively, underlines the importance of considering the role of "micro-atmosphere" generated in the course of decomposition when comparing the results of thermal analysis with the behaviour of the rolling fluids in service. Even so some specific effects can be distinguished.

Effect of atmosphere

The exothermal DTA peaks during the degradation in air are probably mostly due to the oxidation of the volatile hydrocarbon products of decomposition. In nitrogen only weak exothermal signals are obtained which may be related to the heat of polymerization. Only in oxidizing atmosphere full decolorization can be achieved below 500 ^{o}C for TDF/H and TDF/TEA due to the minor oxidative step at about 350-400 ^{o}C (Fig. 1, 3)

Compositional effects

The salt form (TDF/K, Fig. 2) shows the strongest tendency to carbonization regardless of atmosphere, starting already at about 150 ^{o}C. Unlike the acid and the TEA form no decolouring for TDF/K is obtained below 500 ^{o}C.

Effect of substrate

Comparative studies of reference rolling fluids on Al_2O_3 (sintered α-alumina) and Al pans in oxidizing atmosphere showed the decolorization to occur on the Al surface only. The Al surface, probably covered by an amorphous oxide film, thus seems to possess a catalytic activity for oxidative degradation of carbonization deposits. This oxide film may be destroyed in the presence of alkali formed by decomposition of TDF/K. This might explain the pronounced discoloring effect of the potassium salt of TDF (Fig. 2).

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