CHARACTERIZATION OF DRAWING LUBRICANTS BY THERMAL ANALYSIS

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INTRODUCTION

Fine metal wires are manufactured by drawing from coarser rolled wire in drawing dies. The cross section is gradually reduced when the wire is plastically deformed during its passage through drawing die (Figure 1). To lower the friction coefficient between the wire and drawing die and thus to lower the heat evolved different kinds of lubricants are used (Figure 2). Due to the high temperature in the contact area the lubricant may break down due to oxidation or thermal degradation.





LUBRICANT

Figure 1. Dry drawing of wire. A = wire, B = die, C = lubricant

Figure 2. Enlarged view of the contact zone

CHARACTERIZATION PRINCIPLE

The principle of screening different kinds of lubricants is based on oxidation at high temperature in pure oxygen and has been described earlier (1). The determinations have been made in a simultaneous TG-DSC apparatus, Mettler TA 2000C.

The TG curves generally show a rapid weight loss over a certain temperature due to release of gaseous oxidation products.

The DSC curves often contain details already at relatively low temperatures, due to e.g. partial melting or decomposition.

DTG has also been used in some tests to indicate the temperature at which the maximum rate of decomposition occurs.

MATERIALS

A number of different commercial dry lubricants for wire drawing has been investigated. For each lubricant a sample has also been scraped from the wire after passage through the drawing die.

Lubricants are often mixtures containing sodium stearate, calcium stearate or molybdenum disulphide together with saturated fatty acids.

One of the commercial lubricants also contained boric acid or sodium borate. For comparison pure samples of sodium stearate, calcium stearate and sodium tetraborate were investigated.

TEST SAMPLES

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Lubricant of Ca-type
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" " Ca-Na-type
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" Ca-Mo-type
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" borate-type
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Pure calcium stearate Pure sodium stearate Pure sodium tetraborate

TEST CONDITIONS

Equipment	: Mettler Thermoanalyzer TA 2000C
Atmosphere	: O ₂ 100 ml/min STP
Crucible holder	: DSC-1
Crucibles	: Pt 0.15 ml
TG sensitivity	: 10 mg f.s.d
DTG "	: 10 mg/min f.s.d
DSC "	: 50 _/ uV f.s.d
Heating rate	: 5 [°] C/min
Max temperature	: 260 [°] C
Chart speed	: 40 cm/h

TEST RESULTS

1. Calcium and sodium stearates (Figures 3 and 4)

A loss of water occurs below 100[°]C and several endothermic peaks are visible for both samples below 140[°]C. Melting of calcium stearate starts at 180[°]C. The oxidation of sodium stearate becomes rapid at 175[°]C which is also valid for calcium stearate. However once the calcium stearate has melted the oxidation proceeds slowly.

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TG



Figure 3. Thermal analysis of calcium stearate

 $\sum_{j=1}^{2} 10^{-1}$

Figure 4. Thermal analysis of sodium stearate

2. Lubricant of Ca type (Figure 5)

The oxidation curve is similar to the pure calcium stearate but the low temperature peaks below 130° C are not resolved. Rapid oxidation occurs over 200° C which is faster than the oxidation of pure calcium stearate.

The lubricant that has passed the drawing die has a very similar curve but the melting of the stearate is not visible and the rapid oxidation starts already at 200° C.



Figure 5. Thermal analysis of lubricant of Ca type. Full line = before, dashed line = after passage of die.

3. Lubricant of Ca-type containing MoS₂ (Figure 6)

The oxidation curve is very similar to that of the lubricant of Ca-type. The same is true for the lubricant that has passed through the die. It seems that the MoS_2 has little influence on the low temperature oxidation behaviour.



Figure 6. Thermal analysis of lubricant of Ca-type containing MoS_2 . Full line = before, dashed line = after passage of die.

4. Lubricant of Na-Ca type (Figure 7)

The DSC curve indicates strong similarities to that of sodium stearate. An endothermic peak is observed at 120[°]C which is between the corresponding peaks of sodium and calcium stearates. Rapid oxidation starts at 190[°]C in the unused lubricant.



Figure 7. Thermal analysis of lubricant of Na-Ca-type. Full line = before, dashed line = after passage of die.

5. <u>Pure sodium tetraborate and a lubricant containing sodium</u> tetraborate (Figure 8 and 9)

Sodium tetraborate decahydrate, $Na_2B_4O_7$. 10H₂O, gives off its crystal water in several steps from $60^{\circ}C$ to $450^{\circ}C$. The dehydration is strongly endothermic, which is seen from the DSC curve.

A lubricant containing sodium tetraborate shows also a series of endothermic peaks up to 200° C. It is claimed by the manufacturer that these reactions will decrease the drawing die temperature, which seems plausible. The start of rapid oxidation does not occur until 230° C or a higher temperature is reached.

After passage of the drawing die the lubricant has decomposed in such a way that almost all endothermic peaks have disappeared. In this case rapid oxidation starts at a normal temperature, 190⁰C.



Figure 8. Thermal analysis of sodium tetraborate decahydrate



Figure 9. Thermal analysis of lubricant containing sodium tetraborate Full line = before passage Dashed line = after passage

CONCLUSIONS

Thermal analysis is a valuable tool for characterization of lubricants for wire drawing with respect to their composition and oxidation behaviour. Positive correlations were also found to their performance in practice.

REFERENCE

1 B.O. Haglund and T. Luks, Characterization of the ageing of cold rolling emulsions by differential scanning calorimetry, Thermochimica Acta, 72 (1984) 51-54