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# A DSC STUDY OF THE DEWAXING OF CEMENTED CARBIDE POWDER

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## ABSTRACT

To press cemented carbide powders to compacts different lubricants are used to lower the friction coefficient (1). These lubricants must be almost completely removed in a "dewaxing" process before sintering. Normally dewaxing is performed by heating the compacts in a suitable furnace atmosphere.

Several reactions occur during the dewaxing. The furnace atmosphere is also an important parameter. By a careful control of the temperature cycle the lubricant can be removed at a suitable rate without damaging the compacts.

A DSC study has been made to determine the specific heat capacity during the different stages of the dewaxing process. An example is given of the extraction of polyethylene glycol in hydrogen-containing atmospheres.

## INTRODUCTION

In the production of cemented carbide tool tips, wear parts etc. powder metallurgical methods are used. These methods imply i.a. pressing of milled powder consisting of different metal carbides and binder metal to compacted blanks. In order to lower the friction in the pressing dies lubricants are generally added to the powder mixture (1). These lubricants must be carefully removed before the sintering process as they otherwise may decompose to carbon or carbon-containing residues, which may severely influence the carbon balance of the final sintered carbide piece.

The lubricant is removed during the dewax process by generally heating the compacts at a low rate to a moderate temperature which depends on what kind of lubricant was used. Dewaxing occurs in a suitable atmosphere or in vacuum to protect the remaining powder compact from oxidation. Some of the protective gases, e.g. hydrogen, participate actively in the decomposition of the lubricant.

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Reactions occur which may absorb or evolve appreciable amounts of heat. The heating rate may thus be retarded or accelerated by such reactions in comparison to what can be calculated from the conventional transient temperature equations. It has thus been found necessary to make a quantitative analysis of the heat exchange in the different periods of the dewax process.

## MATERIALS

The cemented carbide powders tested were spray dried and contained 3 % by weight of polyethylene glycol (Peg). A part of each of the powder batches was dewaxed in flowing hydrogen at approximately 400°C for 1 hour. Two different grades of cemented carbide were tested, see Table 1.

As standard materials pure crystalline alpha alumina and tungsten carbide were used.

Table 1

Specification of powders used in dewaxing studies

| Powder | Composition (w/o) | | | | Lubricant |
|---|---|---|---|---|---|
| | Ni | Co | WC | Cr | Molecular weight |
| 1-0 | - | 11.0 | 89.0 | - | Dewaxed |
| 1-P | - | 11.0 | 89.0 | - | Peg 4000 |
| 2-0 | 15.3 | 13.2 | 70.0 | 1.5 | Dewaxed |
| 2-P | 15.3 | 13.2 | 70.0 | 1.5 | Peg 1500+4000 |

## METHOD

### Instrument and conditions

The experiments were performed in a Mettler TA 2000C Thermoanalyzer for simultaneous TG and DSC. The sample weight ranged between 200 and 500 mg and the weight of standard samples were approximately 150 mg for the alumina and 400-800 mg for the tungsten carbide. As reference for DSC an empty crucible was used. All crucibles were of platinum with a perforated lid.

The experiments were performed in a 5 % hydrogen and 95 % argon gas mixture. The gas flow-rate was 50-60 ml/min (NTP). The entire thermobalance was evacuated to less than 1 mbar before each experiment and then filled with the gas mixture to atmospheric pressure.

## Experiments

The experiments were made according to a Mettler programme (2) for determination of specific heat capacity at constant pressure, $c_p$. The sample crucible and an empty reference crucible were placed on the DSC holder and a trace of dH/dt against time was recorded. A blank test with an empty sample crucible was then made to obtain the base line for the system. The difference in signal due to the absorption of heat in the two experiments may be written

$$\frac{dH}{dt} = mc_p \cdot \frac{dT_p}{dt}$$

where m is the mass of sample in g

$c_p$ is the specific heat capacity in J/g,K

$\frac{dT_p}{dt}$ is the programmed rate of temperature increase in K/min

The procedure was then repeated with a standard substance (alumina or tungsten carbide) and another value for the absorption of heat of the standard substance was obtained. The specific heat capacity of the sample was then calculated from the ratio between the dH/dt signals from the sample run and the standard run and from known values of $c_p$ for the standard substances (3,4).

The specific heat capacities were determined as averages over intervals of 10.0°C from 95°C to 305°C.

## RESULTS AND DISCUSSION

The results of the measurements are given in Fig. 1, where also calculated $c_p$ values for WC according to (4) are shown. It is obvious that the higher the content of binder phase (Ni, Co or Cr) the higher is also the specific heat capacity. The presence of a lubricant also increases the specific heat capacity, as also does an increase in temperature.

It can also be seen that at higher temperatures, above 240-250°C, the curves representing material with and without lubricant tend to coincide for each of the two grades. This can be understood from the fact that the lubricant disappears due to decomposition in this temperature range. This tendency is most pronounced for sample 2-P.

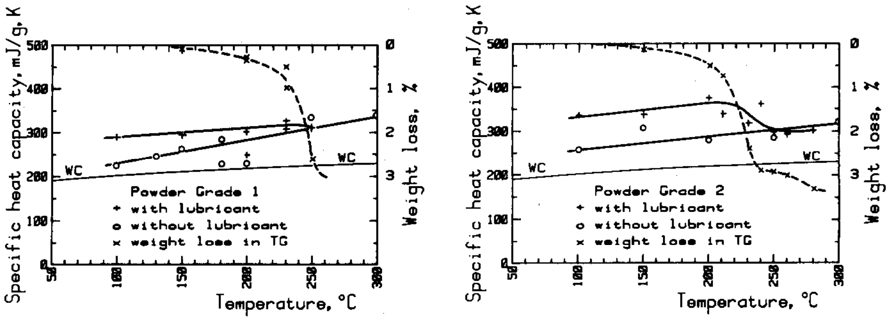

Fig. 1 Specific heat capacities of two powder grades with and without lubricant as function of temperature. Thermogravimetric curves are also shown.

The curves can be interpreted according to the following formula:

$$c_p^{comp}(T) = x_{WC} \cdot c_p^{WC}(T) + (1-x_{WC}) \cdot c_p^{bind}(T) +$$

$$+ y_{lubr} \cdot c_p^{lubr}(T)$$

where

| | |
|---|---|
| $c_p^{comp}(T)$ | is the specific heat capacity of the composite cemented carbide powder at temperature T |
| $c_p^{WC}(T)$ | is the same of tungsten carbide |
| $c_p^{bind}(T)$ | is the same of binder metal forming elements |
| $c_p^{lubr}(T)$ | is the same of the lubricant |
| $x_{WC}$ | is the weight fraction tungsten carbide of the powder |
| $y_{lubr}$ | is the excess weight fraction of the remaining lubricant if the total cemented carbide weight is taken as 1.00 |

## REFERENCES

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