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THERMAL EXPANSION BEHAVIOUR — LIMITS AND POSSIBILITIES FOR MATERIALS RESEARCH

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In the field of materials research thermal expansion plays an important role not only in the case of applications at higher temperatures but also in connection with the preparation of the materials themselves.

For porous materials produced by powder metallurgical techniques the sintering process leads to a shrinkage or an expansion of the material during its preparation. From the investigation of the shrinkage or the expansion behaviour new conclusions can be drawn concerning the interpretation of solid or liquid state reactions which allow an improvement of the materials properties.

In the field of materials research the thermal expansion behaviour plays an important role for the application of the materials especially in the case of powder metallurgical samples produced by sintering.

The measurement of the length change in dependence on the temperature results in the characterization of the dimensional properties of the materials under special conditions of application and allows the simulation of technological steps during their production.

Experimental

The concept of the measuring technique is the following. A dilatometer type Netzsch 402 E which gives analogous values for the length change $\Delta l(T)$ is combined with a microcomputer type MPS 4944 (designed by the Academy of Sciences of GDR) which collects the data in a digital form (Δl_i az T_i). Furthermore the microcomputer controls the temperature regime $T(t)$ which was input in a dialogue form before the measurement starts. On a screen the actual state of the experiment can be checked. After finishing the test all the data are transferred in a computer type SM 1420 (USSR) using an on-line connection. Here all the necessary

and wanted calculations and procedures are carried out on the basis of the experimental data and the implemented software. The final results can be discussed on a screen, plotted as a diagram or printed out as a table.

Results

In the following typical examples of the application of a dilatometer in materials research are given.

1. Correction function

The measured values of the length of a sample must be corrected because also the measuring system changes its length in dependence on the temperature and the other experimental conditions (length of the sample, heating rate, atmosphere, etc.) [1].

$$\Delta l_{\text{sample}}(T) = \Delta l_{\text{measure}}(T) - \Delta l_{\text{corr}}(T, l_{\text{sample}}).$$

Figure 1 shows the correction function for a given measuring system (Al_2O_3) for any given length of a sample and for selected temperatures. The correction function was determined using calibration samples of Vacromium of a length of 5, 7, 10, 20, 25, 50 mm. Results of measured sapphire samples (50 mm) were added. For each sample length the experiments were repeated (up to 5 times).

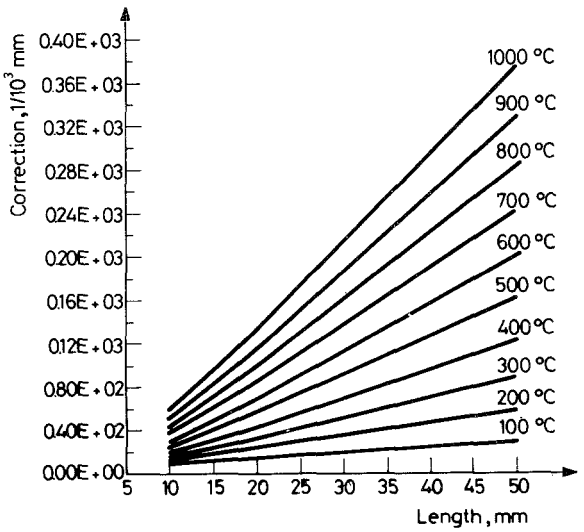

Fig. 1 Correction function: Al_2O_3 system; 5 deg min^{-1}; argon, 3 l h^{-1}, dew point −50 °C

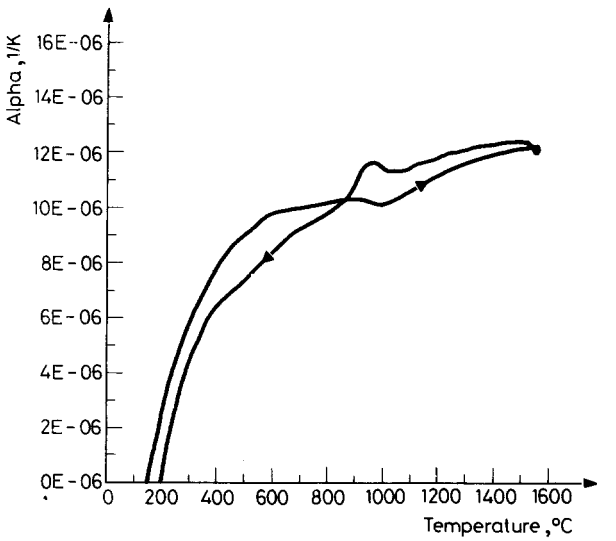

Fig. 2 Thermal expansion coefficient. Sintered ceramics $Al_2O_3 + 11.5\%$ ZrO_2, 10 deg min^{-1}; argon, 3 l h^{-1}, dew point -50 °C

2. Thermal expansion coefficient

The most usual application of a length measurement in dependence on the temperature aims at the determination of the thermal expansion coefficient α. Phase transformations or chemical reactions often lead to a dimensional change of the sample at a definite temperature or within a temperature interval. In Fig. 2 the thermal expansion coefficient α is shown for a sintered ceramics $Al_2O_3 + 11.5$ wt.% ZrO_2.

The results demonstrate the temperature dependence of the ZrO_2 phase transformation and allow for conclusions on the optimization of the processing conditions in order to improve the mechanical properties at higher temperatures [2].

3. Shrinkage and shrinkage rate of porous materials during sintering $\Delta l/l_0$, $d(\Delta l/l_0)/dt$

Pressed powder samples are compacted by sintering processes in which the pores are partially or completely filled with material according to the temperature dependent mechanisms of material transport. In Fig. 3 the shrinkage behaviour of a typical TiC based hardmetal (TiC–Mo_2C–Ni) is demonstrated. Hardmetals consist of a hard material (carbide) and a ductile binder which are strongly compacted by

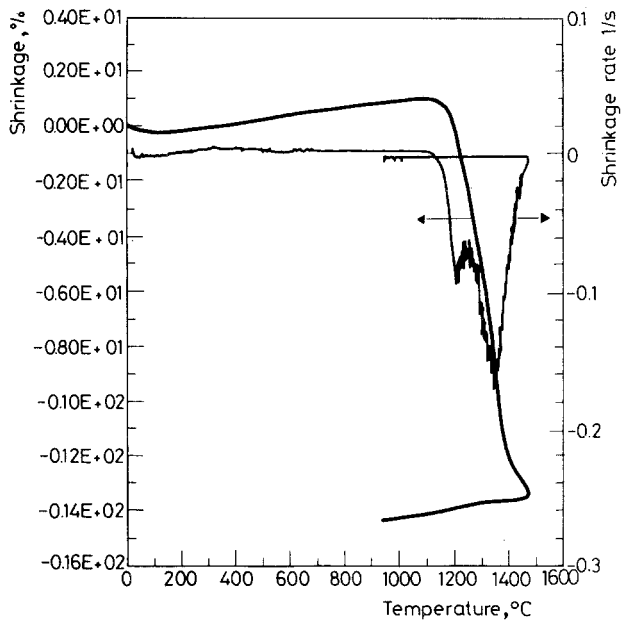

Fig. 3 Shrinkage behaviour of TiC based hardmetal 10 deg min^{-1}; argon, 3 l h^{-1}, dew point −50 °C

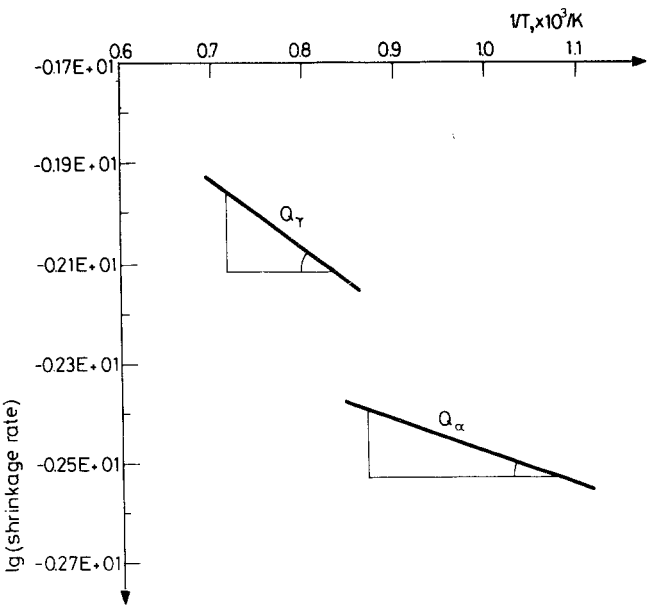

Fig. 4 Arrhenius diagram for sintered iron in the α- and the γ-range. 5 deg min^{-1}; argon, 3 l h^{-1}, dew point −50 °C

liquid phase sintering. The curves of the shrinkage and the shrinkage rate allow the determination of critical temperature ranges (i.e. beginning of the sintering in the binder phase, reduction of oxides of the binder phase and the hard material, formation of a liquid phase, etc.) [3].

Furthermore the influence of critical factors (i.e. atmosphere, admixtures, etc.) on the shrinkage behaviour can be investigated in such experiments. The discussion leads to conclusions on the kinetics of the sintering process, the dominating mechanisms of compaction, the optimization of the technological steps of the sintering process.

4. Arrhenius diagram

Plotting the logarithm of the shrinkage rate versus the reciprocal temperature a term can be calculated from the slope of the curve which has the character of an activation energy. Figure 4 shows the diagram for powder metallurgically compacted iron. The steeper slope in the γ-range compared with the α-range demonstrates that the energy effort for the diffusion controlled sintering process in the range of γ-iron is higher than in the α-range.

References

1 DIN 51 045 Teil 1 Okt. 1976, Bestimmung der Längenänderung fester Körper unter Wärmeeinwirkung.

2 A. Krell, unpublished work (ZFW Dresden) 1987.

3 B. F. Kieback, G. Leitner and K. Pischang, these Proceedings, paper 636.

Zusammenfassung — Auf dem Gebiet der Materialforschung spielt die Messung der thermischen Ausdehnung eine grosse Rolle sowohl für die Anwendung bei hohen Temperaturen als auch im Zusammenhang mit der Präparation des Materials.
Bei porösen Materialien, die nach pulvermetallurgischen Methoden hergestellt werden, ist der Sinterprozess von einem Schrumpfen oder einer Ausdehnung des Materials begleitet. Die Untersuchung dieser Vorgänge liefert neue Ergebnisse zur Deutung von Flüssig- oder Festphasenreaktionen und damit Möglichkeiten zur Verbesserung der Materialeigenschaften.

Резюме — При исследовании различных материалов термическое расширение играет важную роль не только в случае применения более высоких температур, но и в процессе получения самых материалов. Для порошковых материалов, получаемых методом порошковой металлургии, процесс спекания вызывает сжатие или расширение материала. Проведенные исследования процессов сжатия и расширения позволили вывести новые заключения, касающиеся твердотельных реакций и реакций в жидком состоянии, что повволило улучшить свойства получаемых материалов.