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SCTA AND CERAMICS

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Conventional thermal analysis has since its introduction been used abundantly in studies of classical ceramics (clay products, porcelain, concrete) to establish the optimum process conditions for the fabrication of these materials and to measure their properties and behaviour. As a relatively new technique, however, the main application of SCTA has been in more fundamental studies of what generally is termed as engineering ceramics (structural and functional ceramics).

Besides the application of SCTA in thermogravimetric studies on ceramic materials, this technique has also proved useful in dilatometric studies of the sintering behaviour of ceramic compacts and some examples of this application was presented in this presentation.

By the technique used in these studies, which can be designated as stepwise isothermal dilatometry (SID), the temperature is controlled by the change in length of the sample – which corresponds to the shrinkage due to the densification during the sintering – in the same way as for the thermogravimetric SIA technique and the sintering (densification) thus takes place in characteristic isothermal steps as observed for thermal decompositions. A typical SID-curve obtained for sintering of yttria doped zirconia [1] in the temperature range 850 to 1450°C is shown in Fig. 1.

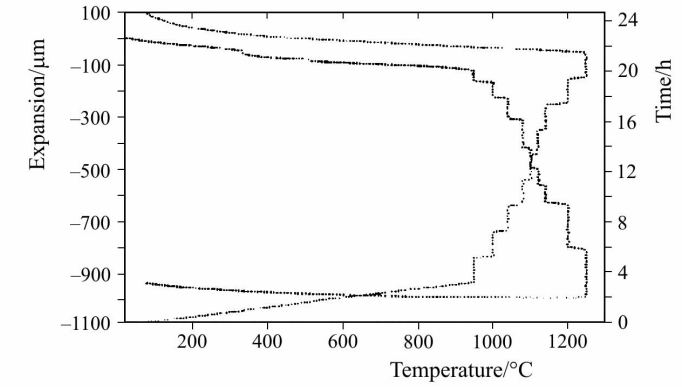

Fig. 1 SID curve for zirconia ceramics (TZ3YA)

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Sintering kinetics

For the first stage in the sintering, formation of necks between the powder particles, it is quite easy to establish both the sintering mechanism and the activation energy from the SID plot [1]. The equation for this stage can generally be expressed as:

$$y=\Delta l/l_0 \tag{1}$$

which by differentation gives:

$$y^{N-1}=(k(T)/N)(y')^{-1} \tag{2}$$

In these equations: l_0 is the initial sample length, $k(T)=A\exp(-E/RT)$ is the Arrhenius constant, n a constant depending on the sintering mechanism and $N=1/n$.

Equation (2) can also be expressed as:

$$(N-1)\ln y=\ln k(T)-\ln N-\ln(y') \tag{3}$$

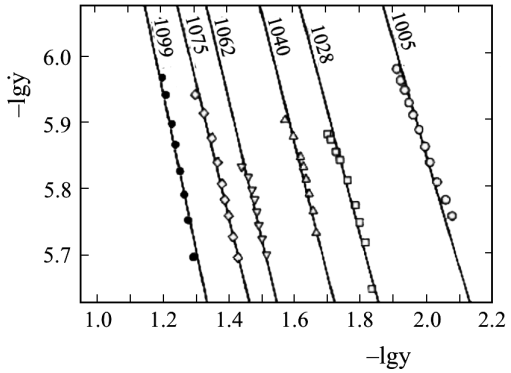

Fig. 2 Shrinkage rate *vs*. shrinkage for isothermal steps obtained from SID on CeO_2 powder-compacts

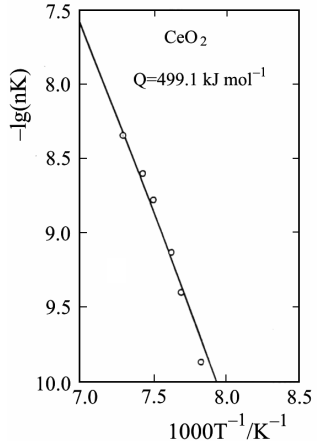

Fig. 3 Arrhenius plot for CeO_2 compacts

and plotting $\ln y'$ *vs*. $\ln y$, which both can be determined from the experimental data, a straight line should be obtained from which the value of N and thus n can be determined from the slope.

Furthermore, when N is known it is also possible to calculate $k(T)$ at different temperatures and the activation energy can then be determined from an Arrhenius plot. Figures 2 and 3 show such plots for the sintering of CeO_2 compacts.

The average value of n was found to be 0.33 indicating that the initial sintering stage for CeO_2 is controlled by grain boundary diffusion of Ce-ions.

Determination of diffusion coefficients

Finally it should be noted that it is also possible to calculate the diffusion coefficients from the Arrhenius equation. From the data presented in Fig. 3 the coefficients found for the Ce-ions (slowest diffusing ions and thus rate controlling) was in the range $8.5 \cdot 10^{-17}$–$3.1 \cdot 10^{-15}$ $cm^2\ s^{-1}$ for the temperature range 1005–1099°C, which are comparable to values obtained by the author in sintering studies of other oxides with the flourite structure, for instance UO_2 [2].

References

1 O. T. Sørensen, J. Thermal Anal., 38 (1992) 213.

2 M. El Sayed Ali and O. T. Sørensen, in 'Thermal Analysis, Proc. 7th Int. Conf. Therm. Anal.' (Ed. B. Miller) John Wiley & Sons, Chichester 1982, p. 344.