Thermal Analysis of Ceramic Materials at High Temperatures

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For the characterization of sintering processes of non-oxide ceramic materials like SiC, Si_3N_4 and AlN experimental investigations at temperatures above 1500°C are necessary.

During sintering at high temperatures chemical reactions, phase and mass changes etc. occur, which can be investigated by different methods of thermal analysis. The most common experimental methods used in this field are the simultaneous thermogravimetric analysis (TGA) and the differential thermal analysis (DTA), ideally coupled with a mass spectrometer (MS) to identify the evolved gases, which can be correlated to the observed mass changes and caloric effects. Dimensional changes like shrinkage are observed by thermodilatometry using pushrod dilatometers, yielding also further information on the densification kinetics. To get a comprehensive understanding of the sintering process, additional information on the phase reactions and constitutional changes during sintering process can be obtained by x-ray diffraction and structural analysis using different methods of electron microscopy.

Examples discussed in the presentation concentrate on the liquid phase sintering of AlN with Y_2O_3 as sintering additive (AlN-Al_2O_3-Y_2O_3 system) and on the liquid phase sintering of SiC (LPSSiC) with AlN and Y_2O_3, (Si-Al-Y-O-N-C system) which is another system of technical importance. After heat-treatment to achieve the equilibrium state, samples in the system AlN–Al_2O_3–Y_2O_3 system are investigated by differential thermal analysis (melting temperatures) in combination with thermogravimetry to study the liquid phase formation in this system. In combination with phase analysis by XRD and structural analysis of the resulting microstructures by SEM and EDX high temperature phase relationships, eutectic temperatures and crystallization paths in the AlN–Al_2O_3–Y_2O_3 system can be discussed. Corresponding investigations on the liquid phase formation in the system SiC–Al_2O_3–Y_2O_3, as a sub-system of the Si-Al-Y-O-N-C system, have been performed.

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[2] R. Neher, M. Herrmann, O. Fabrichnaya, D. Pavlyuchkov, H.J. Seifert, *J. Europ. Ceram. Soc.* **33 (**2013) 2447-55.