MS10.01.06 THE LOW TEMPERATURE WAY TO OXIDES VIA ALCOHOLATE PRECURSORS: STRUCTURE, REACTIVITY, MORPHOLOGY. Heiko Thoms, Matthias Epple, Armin Reller, Institute of Inorganic and Applied Chemistry, University of Hamburg, Martin-Luther-King-Platz 6, D - 20146 Hamburg, Germany

Different alcoholates of magnesium and mixed Mg/Si-, Mg/Ti-, Mg/Zr-and Ti/Si-diolates were prepared. A single-crystal X-ray structure analysis was so far only possible for the compound $Mg(OCH_3)_2 \cdot 2\ CH_3OH$ in the orthorhombic space group $Ccca$ with Z = 64 [1]. The main structural elements are isolated heterocubane units with Mg^{2+} and OCH_3^- ions in the corner. All magnesium alcoholates follow the same thermal decomposition pathway to magnesium oxide MgO via alkoxy-hydroxy intermediates, a fact also confirmed by EXAFS. Scanning electron microscopy of the magnesium oxides and mixed oxides prepared by thermal decomposition of the alcoholates at temperatures between 500°C and 600°C shows significant differences in morphology and domain size which are important for many applications of these oxides.

[1] H. Thoms, M. Epple, H. Viebrock, A. Reller, *J. Mater. Chem.,* **5** (1995) 589594

PS10.01.07 GLASS-MICA COMPOSITIONS AS AN ELECTRIC HEATER SHELL. L.V. Chilikanova, T.I. Shishelova, T.V.Sozinova, A.V.Shcherbakov, Irkutsk Technical University, Irkutsk, Russia

Glass-mica compositions are successfully used as shells for electric heaters. The long service life, mechanical and electric strength of these materials depend on the interaction of Ni-Cr alloy with fusible glass and mica components under working conditions. The data of x-ray diffraction and thermography have shown that upon thermal activation of these substances the processes of metal oxidation, muscovite decomposition, and interaction between the decomposition products and glass are hindered as a result of the reinforcing effect of the silicate medium on the heater metal. The reinforcing film on the mica- composition-metal interface is formed due to the dissolution of spinels (Ni-Cr alloy oxidation products) in the softened glass, that leads to the formation of iron silicate. The oxidation of heater metal starts with the diffusion of iron ions and the formation of ferrospinels. The latter, known for a wide range of homogeneity with many oxides, dissolve the protective solid layer of oxides. The water which is released during dehydration and dehydroxylation of mica and fills intercrystallite pores serves as an oxidant. The established physico-chemical interactions between mica, glass and the products of Ni-Cr alloy oxidation make it possible to find optimal conditions for the production and modification of electric heaters in a shell of glass mica-composition.

PS10.01.08 CHARACTERIZATION OF EPITAXIAL ZrO_2 BY X-RAY DIFFRACTOMETRY WITH PARALLEL BEAM GEOMETRY. R. Guinebretière, A. Dauger, O. Masson, B. Soulestin, Laboratoire de Materiaux Ceramiques et Traitements de Surfaces UA CNRS n°320-ENSCI-47 Av. A. Thomas - 87065 Limoges France

The epitaxial growth of tetragonal zirconia produced via a metal-organic precursor route and deposited on the (h00) planes of MgO substrates was characterized with an original X-ray diffractometer. A parallel $CuK\alpha_1$ X-ray radiation incident beam is provided by a rotating anode source through a four reflections monochromator made of two channel-cut germanium single crystals. The flat specimen is fixed at controlled incidence angle on the four circle diffractometer. Data are collected with a Curved Position Sensitive Detector (INEL CPS 120). The CPSD angular calibration procedure first proposed for capillary sample setting has been extended to the case of flat plate samples. A specific specimen positioning procedure is used and the d-spacing measurement accuracy has been checked using NIST Silicon standard sample.

A zirconia precursor sol was first prepared in the zirconium n-propoxide - acetylacetone - n-propanol system, deposited by dip coating on the polished (100) face of a MgO crystal and then heat treated for 1.5 hour at successive temperatures ranging from 600 to 1400°C. A polycristalline thin film of tetragonal zirconia nanosized grains first appeared. Sintering and normal grain growth simultaneously occurred. Then the film began to break up into isolated islands. At high temperature, the size of single grained islands increased rapidly, up to about $1\mu m$ after 1.5 hour at 1400°C, and complete epitaxial orientation with respect to the substrate was observed: $<100>_{zrO2} // <100>_{mgO}$ and $\{100\}_{zrO2} // \{100\}_{mgO}$. The breadth of t-$ZrO_2$ (200) rocking curve demonstrated the very low level of misorientations. The modification of the substrate (200) and (400) rocking curves was related to surface strains. The interface was imaged by transmission electron microscopy on cross sectional thin samples.

PS10.01.09 DETERMINATION OF SiC POLYTYPES DISTRIBUTION WITH HIGH ACCURACY. By Hongchao LIU and Changlin KUO, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai 200050.

Quantitative determination of SiC polytypes distribution is very difficult from traditional X-ray powder diffraction quantitative analysis methods. SiC polytypes seem to form as the result of stacking faults introduced into the parent structures, the main reflections will overlap with those of the parent polytype and the differences will be relative small. So many efforts have been concentrated on this topic(see L. K. Frevel, et al, *J. Mater. Sci.,* 27(1992)1913-1925). However, it was still hard to obtain accurate results of SiC polytypes distribution by these traditional methods for various reasons.

The whole pattern fitting Rietveld method(H. M. Rietveld, *J. Appl. Crystallogr.,* 2(1969)65-71) is introduced to determine SiC polytype distribution. With the scale factors and cell parameters given by the Rietveld program, equation

$$x_j = S_j \cdot \rho_j \cdot V_{oj}^2 \left/ \sum_{k=1}^{n} S_k \cdot \rho_k \cdot V_{oj}^2 \right. \quad \text{, where S,}$$

ρ and V are scale factor, density and unit cell volume of each polytype, can be used to calculated the relative weight percentag-