MS10.09.05 IN-SITU SYNTHESIS OF CERAMICS AT ELEVATED TEMPERATURES. [1]M.A. Roberts, [1]G. Sankar, [2]G.N. Greaves, 1C.R.A. Catlow, [1]J.M. Thomas, [3]L.M. Colyer, [3]R.H. Jones, [2]E.G. Giulotto, [1]The Royal Institution of G.B., 21, Albemarle Street, London. W1X 4BS, [2]CCLRC Daresbury Laboratory, Daresbury, Warrington. WA4 4AD U.K. [3]Chemistry Department, Keele University, Staffordshire. ST5 5BG U.K.

The formation of ceramic materials from precursor sol-gels and zeolitic systems has been investigated by combined QuEXAFS/XRD and SAXS/WAXS, measured in-situ, under isothermal or ramping temperature regimes upto 1200°C. A new high temperature environmental cell, with the advantage of measuring both fluorescence and transmission EXAFS data, and having a wide (10° - 70° 2Θ) angular range for the XRD, has been designed in order to improve temperature uniformity at 1000°C and sample support for fragile specimens, a similar furnace being used for the SAXS/WAXS measurements. A variety of materials have been studied, including PLZT, LSCF's, cordierite glasses, phase transitions in oxides, nanocrystalline materials, and zeolites as precursors to ceramics.

The crystallisation of PLZT (lead lanthanum zirconate titanate) from an alkoxide sol-gel shows a three stage evolution, an amorphous precursor, a pyrochlore/fluorite intermediate and the final perovskite phase. From the in-situ measurements it is possible to obtain quantitative kinetic information, the activation energy for crystallisation depending on the La concentration.

The combined QuEXAFS/XRD and SAXS/WAXS studies on the collapse of zeolites A and Y has enabled the dilute crystallography of Cd-exchanged sites, followed by Cd K-edge EXAFS, to be directly related to the altering mesostructure defined by the SAXS, on structural change from microporous crystalline to amorphous alumino-silicate as the glass transition is approached, by comparison between the WAXS diffraction profiles. Within the temperature regime investigated zeolite A is found to recrystallise into carnegieite and nepheline, whereas zeolite Y tends to remain amorphous.

MS10.09.06 HIGH TEMPERATURE X-RAY DIFFRACTION STUDY OF THE MELTING SEQUENCE OF THE 2223 PHASE IN THE Bi-Sr-Ca-Cu-O SYSTEM. C. Park, W. Wong-Ng*, L. P. Cook*, R. L. Snyder, P. V. P. S. S. Sastry, A. R. West**, Alfred University, Alfred, NY, *Ceramics Division, National Institute of Standards and Technology, Gaithersburg, MD, **Aberdeen University, Aberdeen, Scotland

Unlike the Pb-doped 2223 ((Bi,Pb):Sr:Ca:Cu) phase, relatively little data has been reported on the characterization and applications of the Pb-free 2223 phase. This is probably due to the difficulty of preparing high purity material. We have successfully prepared a mostly single phase 2223 material. Since melting information is important for processing, the goal of this paper is to use the in-situ high temperature x-ray technique (in combination with the thermal differential analysis (DTA) and quantitative SEM/EDS) to investigate the melting sequence of the 2223 phase.

It was found that the melting sequence of the 2223 phase is similar to the 2212 phase in that the primary crystals are calcium strontium cuprates and calcium strontium oxides. In the Ag-free material, at least two phases were observed during the melting process. At around 890°, the $(Ca,Sr)_2CuO_3$ appeared and at around 950°C, the (Ca,Sr)O phase was formed. When Ag powder was well mixed with the the sample, the melting temperature of the 2223 phase was lowered by about 20°C and at least 3 alkaline-earth phases were observed during melting. At around 870°C $(Ca,Sr)CuO_2$ was observed first followed by the appearance of $(Ca,Sr)_2CuO_3$ at ≈ 890°C and (Ca,Sr)O at about 950°C.

PS10.09.07 KINETICS OF DEHYDRATION OF Ca-MONTMORILLONITE: AN IN SITU SYNCHROTRON X-RAY STUDY. Helen J Bray, Simon A T Redfern, Department of Earth Sciences, University of Cambridge, Downing Street, Cambridge, CB2 3EQ, United Kingdom

The dehydration of Ca-montmorillonite has been studied using time-resolved high-temperature energy-dispersive X-ray diffraction at the Daresbury Synchrotron Radiation Source. Upon heating, water loss from the interlayer region causes changes in the basal spacing of smectites. We have used the d-spacing of the 001 reflection as a measure of the progress of the dehydration reaction. Non-isothermal and isothermal kinetics have been investigated using an ambient pressure heating cell. The position and width of the 001 reflection reflect the average reaction coordinate and spatial heterogeneity of the dehydration process, respectively. We have used these data, in conjunction with in-situ infrared and thermogravimetric studies, to develop a kinetic model for the reversible dehydration of montmorillonite at temperatures up to 450 K. The results point to a change in dehydration mechanism from low to high temperature, as well as demonstrating the variation in equilibrium water content with temperature up to 450 K.

PS10.09.08 REAL-TIME X-RAY POWDER DIFFRACTION EXPERIMENTS ON COCOA BUTTER. H. Schenk, K.F. van Malssen and R. Peschar, Laboratory for Crystallography, Amsterdams Instituut voor Moleculaire Studies, University of Amsterdam, Nieuwe Achtergracht 166, 1018 WV Amsterdam, The Netherlands

The crystallization, melting and re-crystallization behaviour of twelve different cocoa butter (CB) samples has been investigated with a special real-time x-ray powder diffractometer [1]. With this equipment a powder diffraction pattern of CB, that exhibits typical peaks in the d-range 3.0-6.1 Angstrom, can be recorded within one second.

Crystallization experiments carried out as function of cooling rate and crystallization temperature lead to the observation that the beta polymorphic form of CB can not be obtained directly from the melt [2]. Melting trajectories of the beta form of all CB samples have been established and subsequently rationalized in terms of the major chemical components of CB [3]. The occurrence of a memory effect will be discussed on basis of re-crystallization experiments as carried out for the beta form of CB [4].

References

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