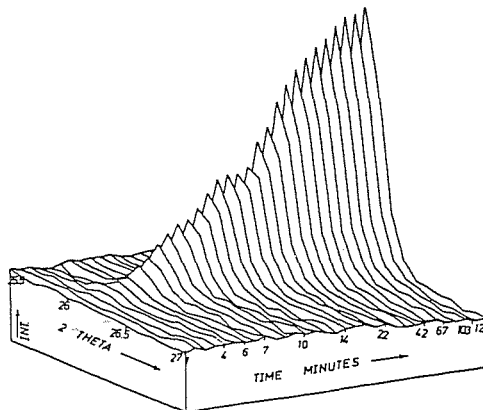


07.9-10 РЕНТГЕНОГРАФИЧЕСКИЕ ИССЛЕДОВАНИЯ КОНТАКТНОГО СЛОЯ СТЕКЛО-СЛЮДА В МИКАЛЕКСЕ. Т.И. Тимшелова, Л.В. Чиликанова, кафедра физики, Иркутский политехнический институт, СССР.

Микалекс - электроизоляционный материал, изготовленный методом горячего прессования смеси порошкообразной слюды мусковит и специального стекла. Материал обладает высокими коррозионной и термическими стойкостями, большой механической прочностью и низкой теплопроводностью. Существенное влияние на формирование необходимого комплекса физико-химических свойств оказывает структура и состав контактного слоя. Этот образуется в процессе растворения пограничных областей кристаллов слюды в расплавленном стекле. Структура и состав переходного слоя исследовались методами микрорентгеноспектрального, рентгеновского, термического фазовых анализов, растровой электронной микроскопии, инфракрасной спектроскопии. Фрактограммы флюоритового и мусковитового микалексов регистрируют слоистое строение композита, неравномерность расположения чешуек слюды в стекле, широкий диапазон их размеров. На поперечных срезах образцов видны отверстия закрытых пор размером до 0,01 мм. Они образуются, очевидно, из-за недостаточных давлений и текучести стекла. Анализ концентрационных кривых показал, что в контактной зоне происходит взаимная диффузия элементов слюды и стекла. Рентгеновским фазовым анализом выявлены фазы, образовавшиеся в процессе спекания. ИК-спектры косвенно подтверждают результаты рентгеновского фазового анализа. В ИК-спектрах мусковитового микалекса обнаружена широкая полоса  $720 \text{ см}^{-1}$ , нехарактерная для ИК-спектров исходных компонентов. Этот факт интерпретируется как частичный разрыв связей  $\text{H}_2\text{-OH}$ .

of transformation by thermal expansion measurement performed on quenched samples.



Time resolved XRD study of the nucleation and growth of h-quartz s.s. from  $\text{Li}_2\text{O-Al}_2\text{O}_3\text{-SiO}_2$  glass at  $800^\circ\text{C}$ .

07.9-11 HIGH TEMPERATURE X-RAY DIFFRACTION STUDIES OF THE TRANSFORMATION KINETICS IN  $\text{Li}_2\text{O-Al}_2\text{O}_3\text{-SiO}_2$  GLASS-CERAMICS. By W. Pannhorst, E. Rodek, U. Schiffner, Schott Glaswerke, Mainz, W. Germany und H. Göbel, P. Chayka, Siemens Research Laboratories, München, W. Germany.

During the production of  $\text{Li}_2\text{O-Al}_2\text{O}_3\text{-SiO}_2$  glass-ceramics two structural transformations occur:

- 1) Primary crystallization of the glass to a high-quartz solid solution containing glass-ceramic and
- 2) a subsequent phase transformation of the high-quartz s.s. crystal phase to a keatite s.s. crystal phase.

In the present study these transformations have been investigated in-situ with a high temperature X-ray powder diffractometer. The diffractometer is equipped with a position sensitive detector which enables to take "snapshots" of the transformation every few seconds where necessary. For both transformations relative integrated peak intensities were measured in isothermal runs to determine the extent of the phase transformation at each time interval.

The kinetics of the glass to h-quartz s.s. transformation were investigated in the range from  $750$  to  $900^\circ\text{C}$ . For glass samples nucleated at  $730^\circ\text{C}$  the h-quartz s.s. phase is first observed between  $750 - 775^\circ\text{C}$ . The time for apparent saturation ranges between approximately 15 min. at  $900^\circ\text{C}$  to over 180 min. at  $775^\circ\text{C}$ . The reaction rates are compared to those obtained for quenched samples analyzed at room temperature using XRD.

The transformation of the h-quartz s.s. phase to keatite s.s. phase was studied between  $900$  and  $1000^\circ\text{C}$ . The time required for apparent saturation of the keatite s.s. phase ranges from about 10 min. at  $1000^\circ\text{C}$  to over 150 min. at  $900^\circ\text{C}$ . The results are compared with those obtained by Pannhorst and Wichelhaus (Glastechn.-Ber. 56 K, 1983, Bd. 2, 572) who have monitored the extent

07.9-12 ON THE CONTROL OF STOICHIOMETRY IN  $\text{SnO}_2$  BY MEANS OF ANIONIC REFLECTIONS. By Yu. A. Rosenberg, I.L. Lunev, L.I. Kleshchinsky, V.M. Kiselev, L.G. Andrievskaya, Inst. of Transp. Engineers, 664074 Irkutsk and N.V. Shokhirev, Inst. of Chem. Kinetics, Sib. Branch of Academy of Sciences, 630090 Novosibirsk, USSR.

The integrated reflecting powers of 111 and 210 Bragg reflections of  $\text{SnO}_2$  pellets have been measured on a double-crystal spectrometer with  $\text{CuK}$  radiation. These reflections are anionic since cations do not contribute to their structure factor. The extinction length of these reflections is much larger than that of the photoelectric absorption and therefore assessing their intensity obeys the kinematical theory formula.

The current anion coordinate in the unit cell has been determined in each sample from the reflection intensity ratio of 210 and 111. The obtained values for different crystals fall within the interval  $0.290 - 0.296$ . The number of anionic vacancies in the studied sample was determined from its structure factor 111 by comparing it to the corresponding standard value with a maximal intensity of this reflection. The results show that the composition of the studied samples changes from  $\text{SnO}_2$  - standard to  $\text{SnO}_{1.78}$ .

The electroconductivity measurements say that with a growing deviation from the stoichiometry the resistance in the samples falls (for extreme samples by 6 orders), which is to say that vacancy mechanism of the electroconductivity in  $\text{SnO}_2$  is substantial.