

Determination of scandium in minerals and sea-water. Tsunenobu Shigematsu, Masayuki Tabushi, Yasuharu Nishikawa, Keizo Hiraki, Shiro Goda and Reiko Inoue. *Nippon Kagaku Zasshi*, **84**, 336 (1963), in Japanese.—Sc in several minerals and sea-water samples was determined by the spectrophotometric method with 5,7-dichloroxine. The procedure is as follows: 0.5 ~ 1g. of mineral sample is treated with HF and dissolved in HCl. 100 ~ 200 mg. of Ca^{2+} as a carrier and oxalic acid solution are added and pH of the solution is adjusted to 1.8. After standing over night, precipitate is ignited and the residue is dissolved in HCl. Sc is separated by extracting at pH 1.5 with 10 ml. of 0.2 M TTA-benzene followed by backextracting with 20 ml. of 1 N HCl. After Fe and other interfering elements is pre-extracted at pH 3 as 5,7-dichloroxinate, Sc chelate is successively extracted at pH 9.5 with 10 ml. of chloroform. Absorbances are measured at 397 $\text{m}\mu$ against the reagent blank. Sc in sea-water is concentrated also by coprecipitating with calcium oxalate, using each 200ml. of 2 N oxalic acid for 20 l. of sample. Several mineral samples such as bauxite, garnet, lepidolite, fluorspar etc. were analysed, and the Sc content of 10^{-3} ~ 10^{-4} % was obtained. Sc in sea-water collected at offshore Misaki-cho gave the order of 10^{-3} $\mu\text{g./l.}$

Inorganic Chemistry

Effect of grain size of petalite on the properties of petalite ceramics. Megumi Tashiro and Toshio Maki. *Yogyo Kyokai Shi*, **71**, 65 (1963), in Japanese.—In the previous paper the authors have reported that addition of a small amount of pulverized lithia-containing glass to the powdered natural petalite increased the firing range of their pressed mixture, improving modulus of rupture and thermal shock resistance of their sintered body (*J. Ceram. Assoc. Japan*, **70**, 8 (1962)). In the present study the effects of fineness of the petalite powder on the above behaviors were investigated. 12 kinds of batches comprising 10 % by weight of the pulverized glass (finer than 325 mesh; 10.7 Li_2O , 8.9 MgO , 8.9 Al_2O_3 , 71.5 % SiO_2 by weight) and 90 % of each one or two of the powdered petalites of different fineness (40~80, 80~200, 200~325, >325 mesh) were prepared, pressed into the bars of 5×5×50 mm, and fired at 1250~1280 °C until their porosity reached almost zero. The batches consisting of no glass frit were also prepared for the comparison. The results obtained are summarized as follows: (1) With decreasing the grain size of petalite powders, the firing range and the firing shrinkage of the pressed body increased. (2) With decreasing the grain size of petalite powders, the modulus of rupture and the heat shock resistance of the sintered body were improved. (3) For the body comprising petalite powder finer than 200 mesh, water-quenching from 1000 °C increased the modulus of rupture of the sintered body. For the body comprising the petalite grains coarser than 80 mesh, however, the reversed effect was observed. (4) With decreasing the grain size of petalite powders, the thermal expansion coefficient of the sintered body decreased, reaching almost zero. From above results, it was concluded that the grain size of petalite powder for making petalite-ceramics should be below 200 mesh.

Hot pressing of a mixture of petalite powder and pulverized lithia containing glass. Toshio Maki and Megumi Tashiro. *Yogyo, Kyokai Shi*, **71**, 196 (1963), in

Japanese.—The authors have shown that addition of a small amount of powdered frits of lithia containing glass to naturally occurring petalite powders increases the firing range of their mixture, improving the modulus of rupture and thermal shock resistance of its sintered product (*J. Ceram. Assoc. Japan*, **70**, 8 (1962) and **71**, 65 (1963)). The sintering method so far used in the authors' experiments was the conventional one, viz. cold-pressing followed by high-temperature sintering. In the present study, a hot-pressing technique has been applied to the same mixture for the fabrication of the product of the higher density and mechanical strength. The glass used has the composition of 10.7% Li₂O, 8.9% MgO, 8.9% Al₂O₃, 71.5% SiO₂ by weight. It was added in powder form to petalite powders in a weight ratio of 10:90. The mixture (20 g), after ball-milled to a fineness over 325 mesh, was introduced into a graphite mold with an inside diameter of 30 mm, and hot-pressed in the temperature range of 1,000 to 1,150 °C under pressures varying from 50 to 200 kg/cm² for 60 min. The determinations of bulk density modulus of rupture and thermal shock resistance were made on the hot-pressed bodies prepared under these conditions. The results obtained are as follows: The optimum firing temperature required to produce a dense petalite-glass body was markedly lowered by 150 °C in hot-pressing than in the conventional method. The body hot-pressed under the pressure of 150 kg/cm² at 1,150 °C for 60 min has a bulk density of 2.40 and a modulus of rupture of about 1,000 kg/cm². This body however, has a drawback of being darkened in color by carbon as a result of the use of graphite mold at the higher temperature. The darkening was little for the body hot-pressed below 1,100 °C under the pressure less than 150 kg/cm². The body hot-pressed at 1,100 °C under the pressure of 150 kg/cm² has a density of 2.25 and a modulus of rupture of 800 kg/cm², the latter being higher by about 300 kg/cm² than that of the body having the same density but prepared by the conventional firing method. It did not show any change in modulus of rupture after taken out from the furnace at 700 °C and dropped into water.

Chemical durability of glass-ceramics. Meguni Tashiro, Sumio Sakka and Tokuji Yamamoto. *Bull. Inst. Chem. Res., Kyoto Univ.*, **41**, 197 (1963).

Glass-ceramics catalyzed with zirconia. Megumi Tashiro and Masamichi Wada. "Advances in Glass Technology," part 2 (Papers from the sixth international congress on glass) p. 18 (Plenum press, 1963), in English.—The compositional search has been made for the glasses in the Li₂O - Al₂O₃ - SiO₂ system, especially in a region near the spodumene composition, which by the aid of small additions of ZrO₂ and P₂O₅ can be converted by heat treatment into the polycrystalline materials with good thermal shock resistance. It was found that: (1) With increasing amount of P₂O₅, the solubility of ZrO₂ in glass at melting temperatures increases. (2) Addition of too much Li₂O in replacement of Al₂O₃ obstructs the uniform crystallization of the glass in the process of heat-treatment, resulting in deformation, cracking, or swelling of the specimens. (3) Replacement of SiO₂ with Al₂O₃ decreases the viscosity and increases the solubility of ZrO₂ at melting temperature. The best composition was 65 SiO₂, 30 Al₂O₃, 5 Li₂O, 1 K₂O, 3 P₂O₅, 4 ZrO₂ by weight ratio. The linear thermal expansion coefficient of its glass after crystallization was 15×10^{-7} (20–500 °C).

The chemically machinable photosensitive glasses of the $\text{Li}_2\text{O-K}_2\text{O-ZnO}_2$ system. Naohiro Soga, Sumio Sakka and Megumi Tashiro. "Advances in Glass Technology," part 2, (Papers from the sixth international congress on glass) p. 34 (Plenum press, 1963), in English.—The chemically machinable photosensitive glasses in which crystals of lithium orthosilicate ($2\text{Li}_2\text{O} \cdot \text{SiO}_2$) precipitate in the reheating stage have been studied. The glass composition region investigated was 15 to 25 Li_2O , 1 to 5 K_2O , 10 to 20 ZnO , and 60 to 70 SiO_2 in mole %. Au and CeO_2 were added in all cases as a nucleating agent and a photosensitizer, respectively. A characteristic feature of this type of glass was found to be the high dissolution velocity of its U. V.-exposed opal area into the HF solution; for example, the value for the glass of the composition, 17 $\text{Li}_2\text{O} \cdot 3 \text{K}_2\text{O} \cdot 15 \text{ZnO} \cdot 65 \text{SiO}_2 \cdot 0.005 \text{Au} \cdot 0.01 \text{CeO}_2$ (mole %), which showed the best result, was 0.02 mm/min. into 5% HF solution at 18 °C. The portion of this glass remaining after etching could be converted, by a U. V. exposure and reheating up to 700 °C into a fully crystallized material with good electric properties ($\tan \delta = 22 \times 10^{-4}$, 1 Mc).

The electron spin resonance spectra of the mononegative ions of *o*-, *m*-, and *p*-terphenyl. Hiroaki Nishiguchi, Yasuto Nakai, Kazuo Nakamura, Yasuo Deguchi and Hideo Takaki. *Rev. Phys. Chem. Japan*, **32**, 57 (1963).

Magnetic study of Fe_3Si and Fe_5Si_3 by Mössbauer effect. Teruya Shinjo, Yoji Nakamura and Naomoto Shikazono. *J. Phys. Soc. Japan*, **18**, 797 (1963). Mössbauer measurements were made on ferromagnetic Fe_3Si and Fe_5Si_3 to investigate the magnetic structure. In the ordered crystal of Fe_3Si , two internal fields were found to be 320 kOe and 205 kOe at liquid nitrogen temperature, corresponding to the two crystallographically different sites, Fe (I) and Fe (II). Since Fe (II) has a small magnetic moment, a low internal field and a large isomer shift, its electronic structure may be fairly modified by a formation of covalent bond with Si atom. On the other hand, the situation of Fe (I) seems to be little changed from that of Fe atom in pure iron. Fe_5Si_3 also has two different sites and two internal fields of 230 kOe and 130 kOe at liquid nitrogen temperature. Both kinds of Fe seem to be considerably different from Fe in pure iron.

Role of hydrogen atoms in palladium. Takashi Tsuchida. *J. Phys. Soc. Japan*, **18**, 1016 (1963).—The Hall coefficient and magnetic susceptibility of the hydrides of palladium silver alloys are measured as a function of hydrogen content in order to clarify the role of hydrogen in palladium. From the relation between the magnetic property and the hydrogen content, it is concluded that a valence electron of hydrogen plays a similar role in the hydride to that of a valence electron of silver atom in palladium-silver alloys. In diamagnetic region, that is, in higher content region of β -phase, the Hall coefficient considerably decreases with increasing hydrogen content, indicating that a valence electron of a hydrogen atom takes a part in conduction.

Electron spin resonance hyperfine spectra of di-panisyl nitric oxide. Kisaburo Umemoto, Yasuo Deguchi and Hideo Takaki. *Bull. Chem. Soc. Japan*, **36**, 560 (1963).