SYNTHESIS, PROPERTIES, AND PHASE RELATIONS OF SOLID SOLUTIONS IN ALKALI METAL TITANOSILICATE SYSTEMS

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Abstract
This article presents the synthesis, physicochemical properties, and phase relations of solid solutions in titanosilicate systems containing alkali metal elements. Structural changes, thermal stability, and the interactions between crystalline phases were investigated. Phase characterization was performed using X-ray diffraction (XRD), differential thermal analysis (DTA), and scanning electron microscopy (SEM).

Keywords: Solid solution, titanosilicate, phase diagram, alkali metals, synthesis, XRD, DTA, SEM.

Introduction

Titanosilicates are widely used in industry due to their high mechanical and thermal stability, chemical inertness, and ion-exchange properties. Solid solutions formed in titanosilicate systems with alkali metals (Li, Na, K, Rb, Cs) are considered promising candidates for the development of new functional materials. This study focuses on the phase formation, mechanisms of solid solution formation, and the physicochemical characteristics of these materials.

2. Experimental Methods

2.1. Sample Preparation

Stoichiometric mixtures of TiO_2, SiO_2, and alkali metal carbonates (Na_2CO_3, K_2CO_3, etc.) were thoroughly mixed and calcined in air at 1100–1300°C for 3–5 hours to synthesize the target solid solutions.

2.2. Characterization Techniques

X-ray diffraction (XRD): for phase identification and structural analysis.
Differential thermal analysis (DTA): for studying thermal behavior and phase transitions.
Scanning electron microscopy (SEM): for microstructural examination.

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3. Results

3.1. Phase Formation

In the Na_2O–TiO_2–SiO_2 system, crystalline phases such as Na_2TiSiO_5 and $Na_2TiSi_2O_7$ were detected. At elevated temperatures, these phases underwent solid-state interactions, forming solid solutions.

3.2. Crystal Structure and Modifications

XRD results showed that incorporation of larger alkali metal ions (K^+, Rb^+) caused changes in the unit cell parameters. The expansion of the lattice structure resulted in phase transformations at certain compositions.

3.3. Thermal Behavior

DTA analysis revealed phase transitions in the range of 960–1050°C. Endothermic and exothermic peaks corresponded to crystallization and decomposition processes in the system.

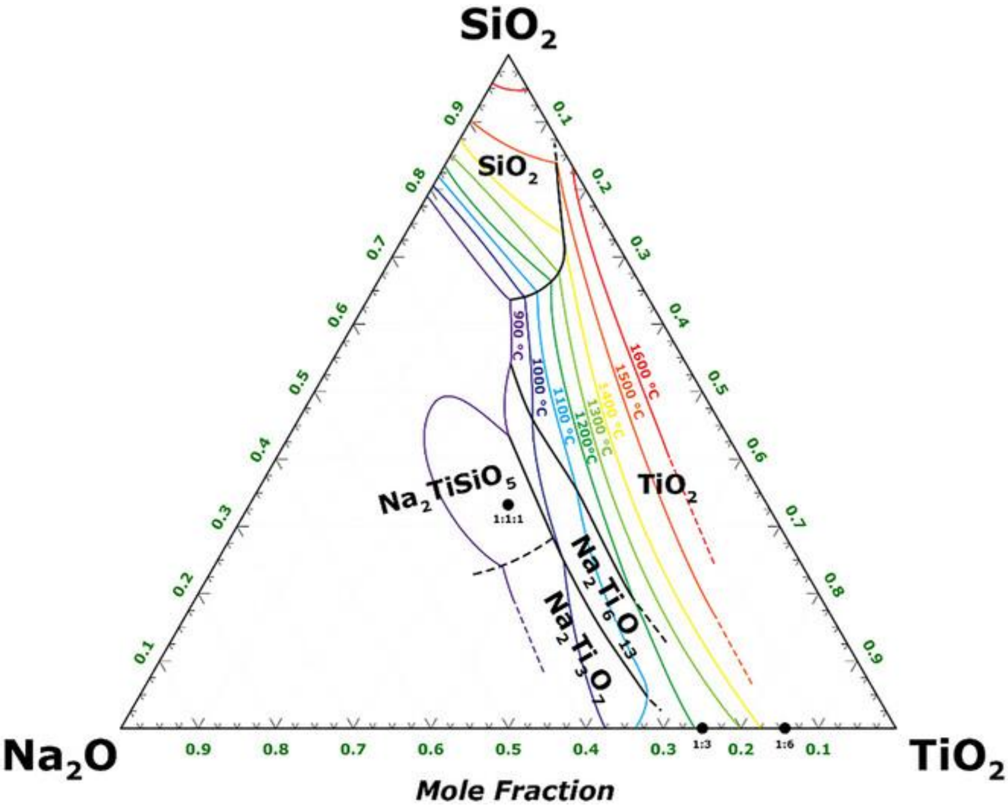

FIGURE 1

The projection of the liquidus of the the Na_2O-TiO_2-SiO_2 system obtained as a result of this study in the temperature range from 900 to 1600 °C.

Phase equilibria of the Na_2O-TiO_2-SiO_2 system between 900 and 1600°C in air

Phase equilibria in the the Na_2O-TiO_2-SiO_2 system were studied at temperatures from 900 to 1600 °C in air. New data were obtained on the liquidus at TiO2 saturation at 1500 and 1600 °C. liquids in solid saturation with SiO_2Thio -2, $Na_2Ti_6O_{13}$, Na_2SIO_5, $Na_2Ti_3O_7$, and in double solid saturation SiO_2 + - Thio -2, Thio -2 + h2ti6O13, Na2ti6hours 13 + na2ti3hours 7, na2ti6hours 13 + NA2SIO5, and NA2SIO5 + na2ti3hours 7. The comparison with the phase diagram estimated using the HTOX and FToxid databases was carried out in the temperature range from 1000 to 1600 °C. The data obtained in the course of this study can be used to revise and re-optimize existing phase diagrams and databases containing silica, soda, and titanium dioxide to obtain a more optimal model.

3.4. Microstructure Analysis

SEM images demonstrated that the synthesized materials consisted of fine, uniformly distributed particles with irregular shapes, indicating a consistent synthesis process.

4. Discussion

The formation of solid solutions in alkali titanosilicate systems is strongly influenced by the ionic radius of the alkali metal. Smaller ions (Li^+, Na^+) integrate more easily into the lattice structure, enhancing thermal and structural stability. In contrast, larger ions (Rb^+, Cs^+) may disrupt the crystal lattice and limit phase stability. The phase transitions are highly sensitive to synthesis temperature and initial molar ratios. These results are significant for the development of high-performance ceramic materials, ion-exchange substrates, and catalysts.

5. Conclusion

Solid solutions in titanosilicate systems containing alkali metals can be successfully synthesized at high temperatures. The ionic radius of alkali metals significantly affects the crystal structure and thermal stability of the resulting materials. These materials are promising for use in advanced ceramics, ion-exchange technologies, and catalytic applications.

References

1. 1El-Rehim AF, Wahab EA, Halaka MM, Shaaban KH. Optical properties of SiO2–TiO2–La2O3–Na2O–Y2O3 glasses and a novel process of preparing the parent glass-ceramics. Silicon. 2022; 14(2): 373–384
2. Zulkhan Z8, Dinilla R, Julianton T, Santoso I, Hidayat T. Carbothermic reduction of ilmenite concentrate with addition of sodium carbonate to obtain iron granules and slag with high titanium content. Metals. 2022; 12(6): 963.
3. Zhang YUM9, Yi LA, Wang LN, Chen DSH, Wang VJ, Liu YUH, and others. A new process for extracting iron, titanium, and vanadium from vanadium-containing titanomagnetite: a process of sodium modification and direct reduction. Int J Miner Metall. 2017; 24(5): 504–511.
4. Zhang YUM10, Wang LN, Chen DS, Wang VJ, Liu Yu, Zhao HS, and others. Method of extraction of iron, titanium and vanadium from vanadium-containing titanomagnetite. Int J Miner Metall. 2018; 25(2): 131-144.
5. Glasser F. P.11, Marr J.. Phase relations in the Na2O-TiO2-SiO2 system. J Am Ceram. 1979; 62(1-2): 42-47.

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6. Santoso I12, Taskinen P. Phase equilibria in the $Na2O–SiO2$ system at temperatures from 1173 to 1873 K. Can Metall Q. 2016; 55(2):243-250.
7. Santoso I13, Riihimaki M, Sibarani D, Taskinen P, Hupa L, Peck M. K. and others. The effect of recently discovered solutions of sodium and calcium silicate on phase diagrams relevant to glass ceramics in the Na2O-CaO-SiO2 system. J Eur Ceram. 2022; 42(5): 2449-2463.
8. Santoso I14, Taskinen P, Jokilaakso A, Peck M. K., Lindberg D.. Phase equilibria and behavior of the liquid phase in the K2O-CaO-SiO2 system during biomass gasification in a swirling stream. Fuel. 2020; 265:116894.
9. Hamilton E. H.15, Klick G. U. Properties of silicate glasses with sodium titanium. J Res Nat Bur Std. 1958; 61: 89–94.
10. Vanis M16, Zemann J., Pah L. Measurement of liquidus temperature and direct identification of crystalline phases using a high-temperature microscope. Collection of Scientific papers of the Faculty of Chemistry and Technology of the Russian Academy of Sciences. 1978; 237-242.
11. Kirschen M17, De Capitani K, Millot F, Riffle J. K., Couture J. P. Immiscible silicate liquids in the SiO2–TiO2–Al2O3 system. European Journal of Mineralogy. 1999, 11: 427-440.
12. Kirillova S. A.18, Almyashev V. I., Gusarov V. V. Phase relations in the SiO2-TiO2 system. Russian Chemical Journal. 2011; 56(9): 1464–1471.
13. Ericsson G19, Pelton A.D. Critical evaluation and optimization of thermodynamic properties and phase diagrams of MnO-TiO2, MgO-TiO2, FeO-TiO2, Ti2O3-TiO2, Na2O-TiO2 and K2O-TiO2 systems. Metall Mater Trans B. 1993; 24(5): 795-805.
14. Vu P20, Ericsson G, Pelton A.D. Optimization of thermodynamic properties and phase diagrams of Na2O–SiO2 and K2O–SiO2 systems. J Am Ceram. 1993; 76(8): 2059–2064.