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TECHNICAL NOTE

Crystallization Behavior of Synthetic Blast Furnace Slag by TiO₂[†]

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KEY WORDS: (Blast Furnace Slag) (Crystallization) (TiO₂)

In the previous paper,¹⁾ the crystallization behavior of the synthetic blast furnace slag containing various amounts of TiO₂ as a nucleating catalyst was investigated. The compositions of the slag (basicity, CaO/SiO₂ = 1.225) existed in the limiting region of glass formation. Therefore, the effect of TiO₂ as a nucleating catalyst has not been fully observed previously.

In this present note, the authors report the crystallization behavior of the slag (CaO/SiO₂ = 1.0) containing 10% TiO₂ during heat-treatment. The compositions of the investigated slag are given in Table 1.

Table 1 Chemical composition of the investigated slag.

Composition Wt%						basicity
CaO	SiO ₂	Al ₂ O ₃	MgO	Fe ₂ O ₃	TiO ₂	CaO/SiO ₂
39.96	39.96	14.96	5.08	0.33	0	1.0
35.96	35.96	13.46	4.57	0.30	10	1.0

The slag was prepared from the parent slag,¹⁾ TiO₂ and SiO₂ (reagent grade, respectively) weighted. The thoroughly mixed raw materials were melted in Pt crucible at 1500°C for 1 hr in air, and then quenched into the ice-cooled water. To cause crystallization, the glassy slag was heat-treated at 1000°C under various time schedules in air, and then quenched into the ice-cooled water.

In order to determine the crystallinity α of the heat-treated slag, the amorphous X-ray scattering was measured. The crystallinity α is defined as²⁾⁻⁴⁾

$$\alpha = \frac{I - I_c}{I_g - I_c}$$

where I is the total scattering intensity from the partly crystallized slag of the measurement, I_g the total scattering intensity from glassy part ($\alpha = 0$), and I_c the total

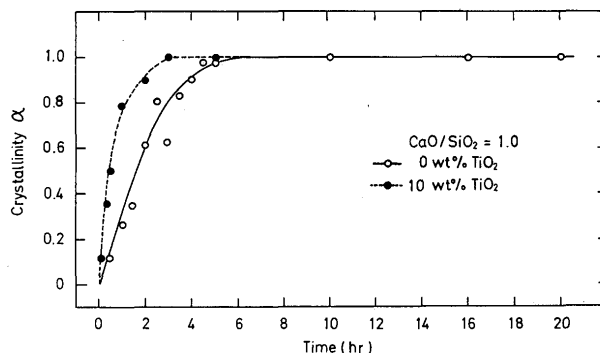


Fig. 1 Crystallinity α of slag during heat-treatment at 1000°C.

scattering intensity from crystalline one ($\alpha = 1$). The amorphous X-ray scattering of each slag was measured by counting of 1000 sec at the fixed angles of $2\theta = 34^\circ$ and 42° , respectively, using CuK α radiation (current and voltage: 35 Kv–15 mA). The angles were chosen that there was considerable amorphous X-ray scattering and there were no diffraction maxima near to those. Fig. 1 shows the crystallinity α of the heat-treated slag. The slag containing 10% TiO₂ was fully crystallized with the heat-treatment of 3 hr, while the measurement over about 6 hr was necessary in the case of the slag not contained TiO₂. This result shows that the crystallization of the slag can be remarkably promoted by addition of TiO₂.

The X-ray diffraction patterns of the slag containing 10% TiO₂ during heat-treatment at 1000°C are shown in Fig. 2. It was recognized that melilite which was a solid solution from gehlenite (2CaO·Al₂O₃·SiO₂) and akermanite (2CaO·MgO·SiO₂) was precipitated from the glassy slag. The diffraction peaks ($2\theta = 22.3^\circ, 30.0^\circ, 31.7^\circ, 34.6^\circ$) due to an unknown crystalline phase appeared besides. It seems that the unknown crystalline phase is a

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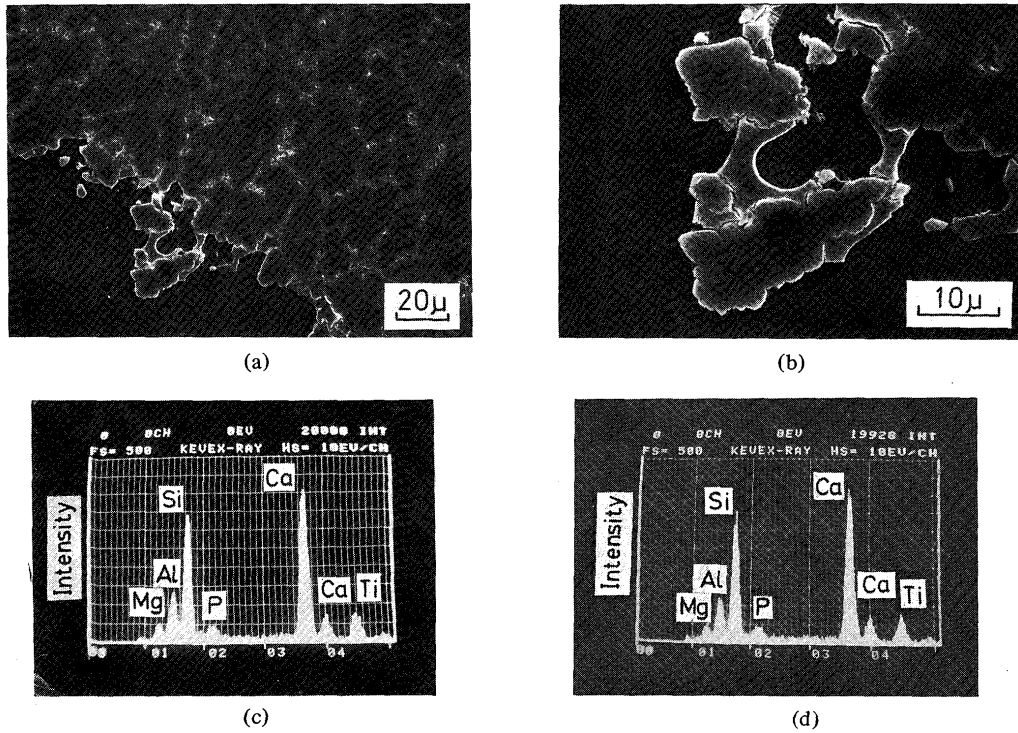


Photo. 1 Scanning electron microscopy analysis of slag containing 10% TiO₂ heat-treated at 1000°C for 30 min.
(a), (b) : surface textures
(c) : analysis of surface crystalline shown in Photo. 1 (b).
(d) : analysis of glassy matrix shown in Photo. 1 (b).

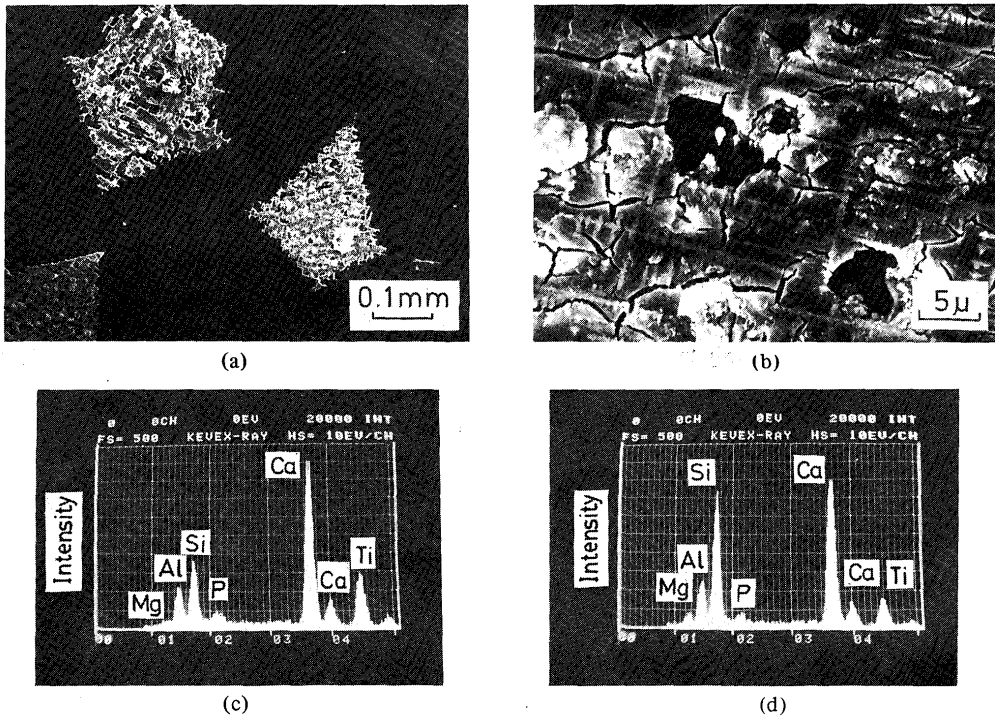


Photo. 2 Scanning electron microscopy analysis of slag containing 10% TiO₂ heat-treated at 1000°C for 30 min.
(a), (b) : Bulk textures
(c) : analysis of bulk star-like crystalline shown in Photo. 2 (a).
(d) : analysis of glassy matrix shown in Photo. 2 (a).

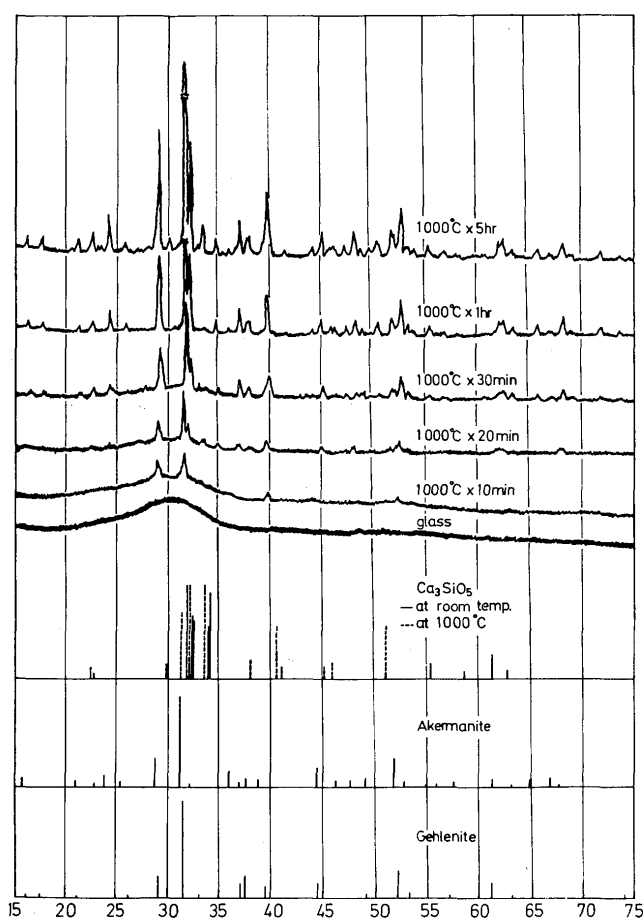


Fig. 2 X-ray diffraction patterns of slag containing 10% TiO_2 during heat-treatment at 1000°C .

Ca_3SiO_5 -like one containing Al_2O_3 and TiO_2 . The diffraction sites and intensities of gehlenite⁵⁾, akermanite⁵⁾ and Ca_3SiO_5 ⁶⁾ are shown in Fig. 2.

Photographs 1 and 2 show the surface and bulk textures of the slag containing 10% TiO_2 , which was heat-treated at 1000°C for 30 min, observed by means of scanning electron microscopy. The surface of the slag was polished by Al_2O_3 powder and then etched in 1% HF acid for 10–30 sec. The surface (Photo. 1 (a) and (b)) and bulk (Photo. 2 (a) and (b)) crystallizations took place from the glassy matrix at the same time. As shown by X-ray energy

dispersion analysis (Photo. 1 (c) and (d)), the difference of the distribution of chemical elements between the surface crystalline and the resided glassy matrix was hardly detected. This surface crystalline might be melilite. On the other hand, the large star-like precipitates, which were aggregated of small dendrites, were formed in the bulk glassy matrix. As shown by X-ray energy dispersion analysis (Photo. 2 (c) and (d)), the amount of Si and Mg in the bulk crystalline decreased in comparison with those of the glassy matrix, while the amount of Ti increased. In view of the X-ray diffraction patterns (Fig. 2), it can be concluded that the bulk crystalline phase is composed from Ca_3SiO_5 -like phase containing Al_2O_3 and TiO_2 .

Maurer⁷⁾ investigated the crystallization of glass containing TiO_2 by means of light scattering. He elucidated that the glass contained a TiO_2 rich emulsion phase, which is known as the two-liquid phase separation, before heat-treatment, and the emulsion phase acted as heterogeneous nuclei in the crystallization of the glass. In this work, the observation of the optical microscope showed partly the less acid-resistant glassy matrix by etching of HF acid. This result suggests that the inhomogeneity of the components can exist in the glassy slag.

The crystallization mechanism of the glassy slag containing TiO_2 will be made clear by carrying out the transmission electron microscope examination or the small angle scattering measurement.

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