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Viscosity measurement of FeO-SiO_2 based slags under controlled oxygen partial pressure

Youngjo Kang[1], Hyunwoo Shin[1], Sang Chul Shim[1], Hye Ran Lee[2]

1. Dong-A University 2 . YoungPoong Corp

1 INTRODUCTION

With the increasing demand for electric vehicles and electronic devices worldwide, nickel compounds have become an important raw material for anode materials in secondary batteries. In nickel dry smelting, pyrite slag composed of FeO-SiO_2 plays an important role. To improve the process efficiency, it is essential to control the physicochemical properties of the slag, especially its viscosity. In this study, a rotational viscometer was used to measure the viscosity of FeO-SiO_2-based slag and a CO/CO_2 gas mixture was used to control the oxygen partial pressure.

2 METHODS

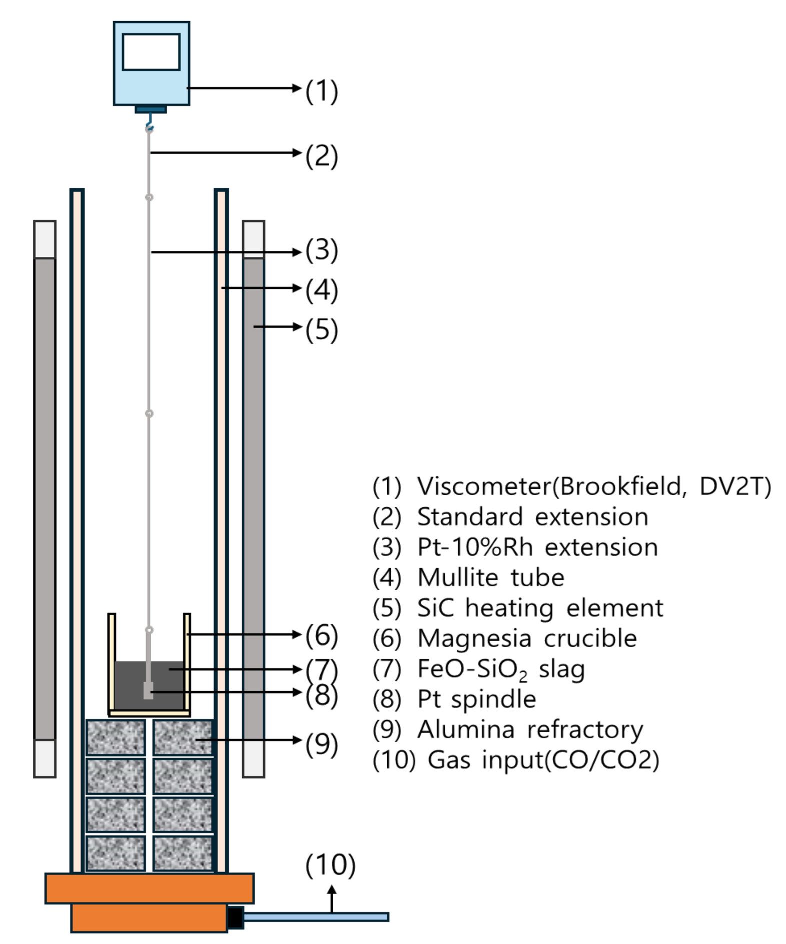

Fig. 1 schematics of the experimental setup

Table. 1 Slag Composition for measurement

No.	FeO	SiO_2	FeO/SiO_2
1	77.3	22.7	3.4
2	72.2	27.8	2.6
3	69.7	30.3	2.3
4	66.7	33.3	2.0
5	61.5	38.5	1.6

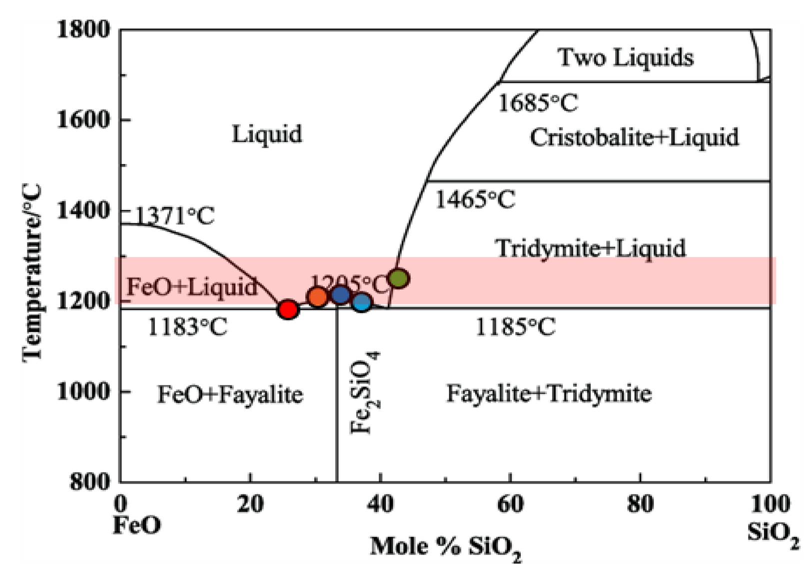

Fig. 2 FeO-SiO_2 phase diagram

- Experiment temperature: 1573K, 1548K, 1523K, 1498K, 1473 K
- Crucible material: MgO

To preserve FeO, the thermodynamic oxygen partial pressures were experimented between Fe/Fe^{2+} and Fe^{2+}/Fe^{3+}, and the optimum oxygen partial pressure was calculated using the reactions presented in (1)-(4). The remaining Fe2+ in molten slag was analyzed using a potentiometric titration method.

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ = -RT\ln K \quad (1) \qquad Fe(l) + \frac{1}{2}O_2(g) = FeO(l) \quad (2)$$

$$2FeO(l) + \frac{1}{2}O_2(g) = Fe_2O_3(l) \quad (3) \qquad CO(g) + \frac{1}{2}O_2(g) = CO_3(g) \quad (4)$$

3 RESULTS

Viscosity of molten FeO-SiO_2 slag

The result of the viscosity measurement of molten FeO-SiO_2 slag are shown as a function of temperature in Fig 3 along with the previous studies.

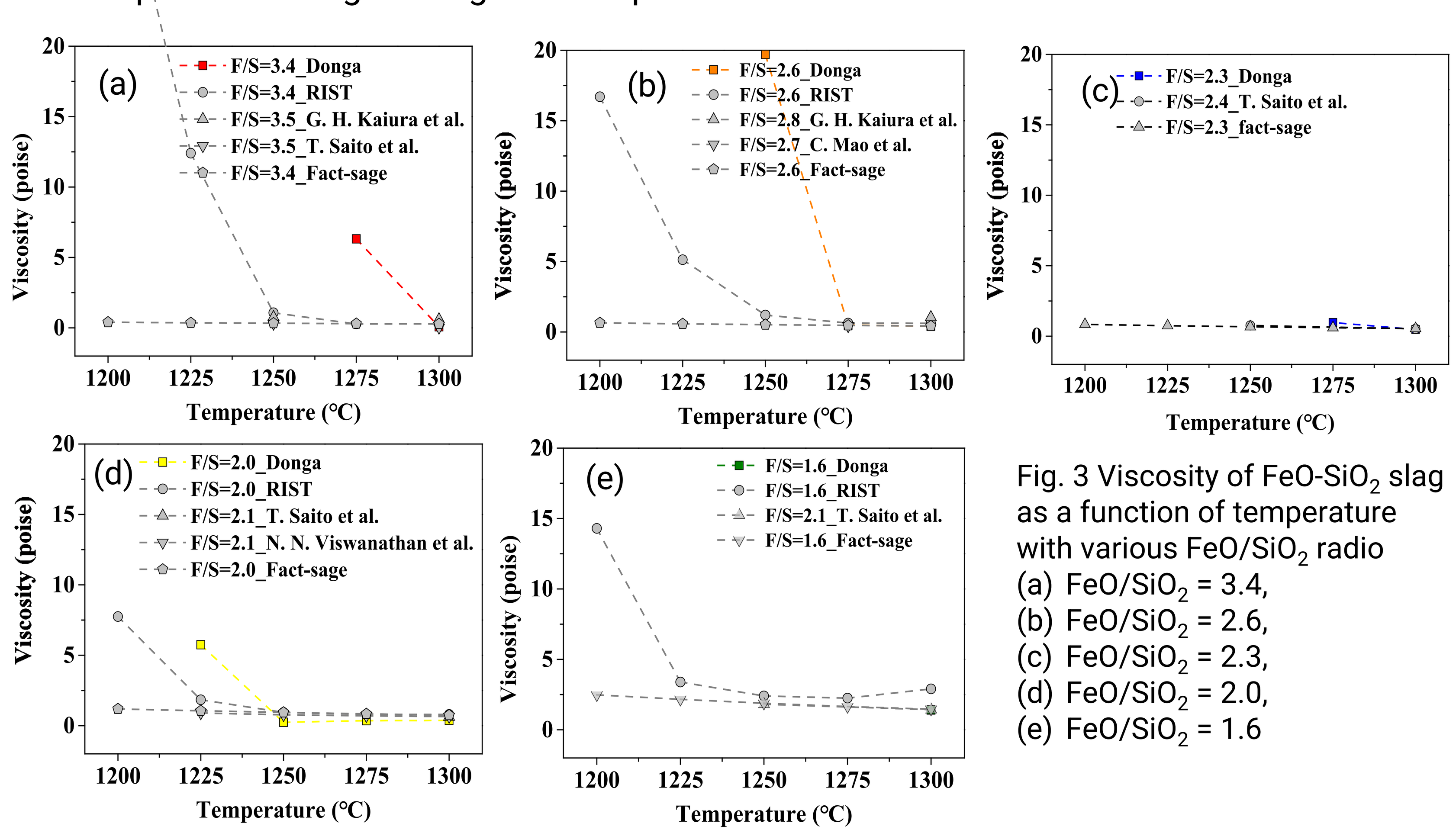

Fig. 3 Viscosity of FeO-SiO_2 slag as a function of temperature with various FeO/SiO_2 radio
(a) FeO/SiO_2 = 3.4,
(b) FeO/SiO_2 = 2.6,
(c) FeO/SiO_2 = 2.3,
(d) FeO/SiO_2 = 2.0,
(e) FeO/SiO_2 = 1.6

- The viscosity increased as the temperature increased, and its temperature dependency appeared to more significant than the previous researches.
- As the FeO content (wt%) increased, the viscosity decreased except for FeO/SiO_2=2.0.

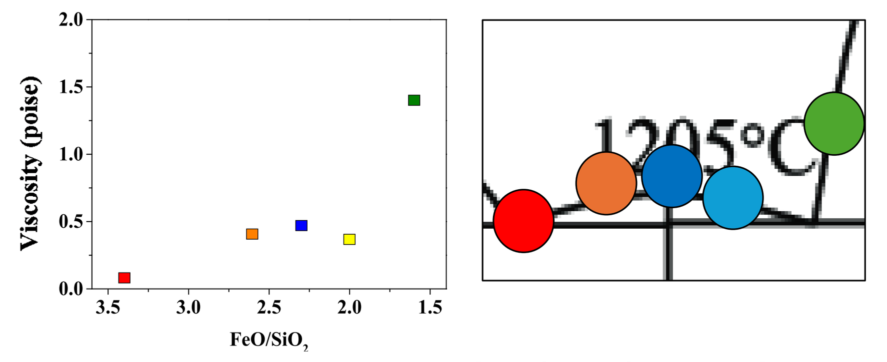

Fig. 4 Measured viscosity of molten slag as a function of FeO/SiO2 at 1573K and liquidus line in the FeO-SiO_2 system

- When the viscosity is plotted as a function of temperature at 1573 K (Fig. 4), the viscosity of FeO-SiO2 slag shows similar tendency with liquidus temperature.
- This shows that the viscosity of the FeO-SiO2 slag is dependent on the tendency to precipitate at the temperature close to liquidus temperature

Composition change during viscosity measurement

- When the FeO/SiO_2 ratio is high, the degree of FeO oxidation reached about 20%, and when the ratio is low, the degree of FeO oxidation was no more than about 10%.
- MgO concentration appeared to be 4-7 wt%, which may be caused by erosion from crucible.
- These compositional variations must be taken into account in measuring the slag viscosity .

Table. 2 Composition change during viscosity measurement

FeO/SiO_2	FeO	SiO_2	Fe_2O_3/Fe_3O_4	MgO	FeO/SiO_2
3.4	59.83	18.46	17.25/25.01	4.47	3.2
2.6	54.58	26.34	16.55/24.00	2.53	2.1
2.3	54.39	28.76	10.02/14.53	6.82	1.9
2.0	57.78	30.49	6.81/9.86	4.94	1.9
1.6	52.73	36.56	7.59/11.0	3.12	1.4

4 DISCUSSION

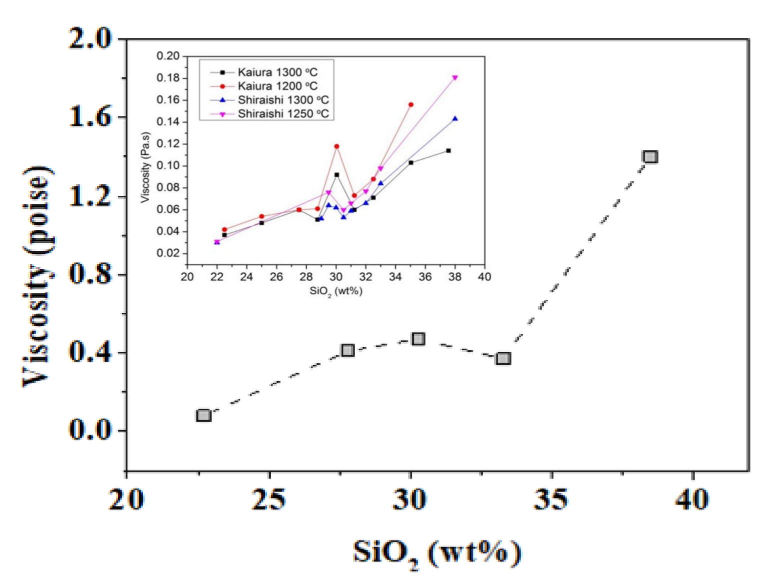

Fig. 5 Viscosity of FeO-SiO_2 slag as a function of SiO_2 concentration at 1573K

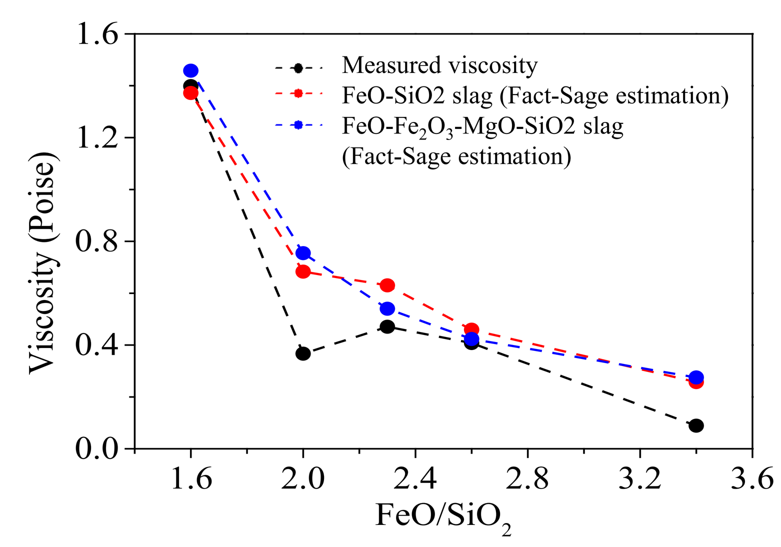

Fig. 6 Fact-Sage 8.2 Viscosity Estimation Model vs. Current Experimentally Measured Viscosity

- To determine the effect of composition on the viscosity of the molten slag, the viscosity measured at 1573 K is plotted against SiO_2 concentration in Fig 5. The viscosity reaches a peak at about 30 wt% SiO_2 concentration, decreases slightly at FeO/SiO_2=2.0, and then increases sharply at SiO_2 concentrations above 34 wt% and low FeO/SiO_2 ratios. A similar trend is observed in past experiments, as shown in Fig 5.
- The viscosity was predicted using the thermodynamics-based simulation software Fact-Sage 8.2 (Fig. 6).
- The results of the predicted viscosity at the targeted composition showed similarity in terms of the decrease compared to the actual viscosity, which is presumed to be due to the emphasis on the effect of each oxide on the viscosity at that composition.
- The predicted viscosity result of the analyzed composition showed a continuous decrease, unlike the actual viscosity, and the decrease was completely curved, unlike the previously predicted target composition, which reflected the effect of MgO on viscosity in that composition.

5 CONCLUSION

- The viscosity of molten FeO-SiO_2 slag was measured between 1573 and 1473 K
- The composition of the slag after the measurement, especially the Fe balance, was analyzed using a potentiometric titrator and ICP-OES.
- The viscosity measurements showed that the molten FeO-SiO_2 slag has a fairly low viscosity value and that the viscosity value increases rapidly with decreasing temperature.
- This can be attributed to the change in composition due to oxidation of Fe^{2+} and dissolution of MgO, which inevitably occurs during the experiment.
- The optimal CO/CO_2 ratio for each temperature was calculated, but oxidation of FeO could not be completely prevented.

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