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Research Article

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Study of the Viscosity of a La_2O_3-SiO_2-FeO Slag System

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Abstract: The viscosity and break temperature of La_2O_3–SiO_2-FeO slag was investigated to develop low-Al_2O_3 or Al_2O_3-free slag for the effective recovery of rare-earth metals. When La_2O_3 content is fixed (45, 50 and 55 mass%), the viscosity and break temperature of La_2O_3–SiO_2-FeO slag decrease with an increase in FeO content and a decrease in SiO_2 content. A higher La_2O_3 content in the La_2O_3-SiO_2-FeO ternary slag yields a lower slag viscosity but a higher break temperature. Individual minor components of Al_2O_3, MnO and B_2O_3 does not affect, or decreases slightly the viscosity of La_2O_3–SiO_2-FeO slag, whereas the slag break temperature is reduced so that the reduction ability order is ranked as $B_2O_3 > Al_2O_3 >$ MnO. A small amount of two components Al_2O_3 + MnO and Al_2O_3 + B_2O_3 has little effect on the viscosity of the slag but it has an additive effect on the slag break-temperature reduction.

Keywords: waste rare earth materials, rare earth recovery, break temperature, La_2O_3-SiO_2-FeO slag system

1 Introduction

Rare-earth hydrogen-storage alloys and NdFeB magnets are used in various applications. Large amounts of waste from these material production processes and end-of-life rare-earth materials become significant secondary sources of rare-earth and other valuable metal elements. The development of a cost-effective recycling method is required urgently. Hydrometallurgical methods, pyrometallurgical methods and a combination of the two have been developed to recycle rare-earths. In general, industrial hydrometallurgical extraction proceeds by a hydrochloric-acid selective-dissolution method, such as is used in NdFeB waste treatment. This method dissolves rare-earth oxides by hydrochloric acid preferentially from the oxidizing roasting NdFeB waste, and then separates different rare earths using solvent extraction [1–3]. Despite its high rare-earth recovery and the high-purity rare-earth products that form, hydrometallurgy is unable to collect other valuable elements very well and results in excessive acid consumption. Single pyrometallurgical method can recover rare-earth metals together with valuable metal elements and is more environmentally friendly [4–6]. The combination of pyrometallurgical and hydrometallurgical advantages could be used to recycle rare-earth and valuable elements simultaneously and reduce environmental pollution. A selected suitable slag system for the former pyrometallurgical step is critical to concentrate rare-earth oxides, for the smooth separation of slag-metal and for the efficiency of subsequent hydrometallurgical process.

Many types of flux have been used to recycle rare-earth oxides, such as CaO-CaF_2 [7], CaO-SiO_2-CaF_2 [8], CaO-SiO_2 [9], Al_2O_3-CaO-MgO-SiO_2-(P_2O_5) [10] and SiO_2-Al_2O_3 [11]. However, melting-separation characteristics for slags that are formed by most of the fluxes examined were not good. The purity and added value of the alloy have also been poorly considered. The authors [11] used gas-selective reduction-oxidation to produce high-added-value alloy from the Ni-metal-hydride battery electrode waste and subsequently accomplished excellent slag-metal separation by SiO_2-Al_2O_3 flux addition. This method yielded a ~50 mass% Re_xO_y-containing slag, which contributed to a saving in hydrochloric-acid consumption in the acid-leaching process.

In addition to the high rare-earth-oxides content and appropriate slag-metal separation characteristics for the formed slag, the selection of flux should be considered to ensure that other slag components do not enter the leaching solution or are separated easily and purified even if they enter the leaching solution. Besides, the latter extraction separation of different rare-earth elements cannot be affected by additive slag components. Some studies [12, 13] have indicated that the aluminum ion is an important factor that leads to the emulsification of extractant in the ex-

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traction process of different types of rare-earth elements. Our previous SiO_2-Al_2O_3 flux is effective for single rare-earth elements. So, there is no emulsification problem of the leaching agent results. However, different types of rare-earth elements usually coexist in rare-earth waste materials, and the application of SiO_2-Al_2O_3 flux gets some limitations. It is urgent to develop aluminum-free or low aluminum flux to reduce or prevent emulsification in the latter hydrometallurgical step in addition to separate the slag-metal effectively and recycle other valuable elements in the former pyrometallurgical step.

In this paper, La_2O_3 was selected as a representative of the rare earth oxides. Acidic SiO_2 does not consume hydrochloric acid in the acid-leaching process. Although FeO can dissolve in the leaching solution, the iron ion is removed easily as a $Fe(OH)_3$ precipitate by adjusting the pH of the leaching solution to ~3–4. SiO_2 and FeO were considered as the main components of slag. The viscosity and break temperature of the La_2O_3-SiO_2-FeO slag system, which are key parameters for effective separation of rare earth oxide-rich slag and valuable metal, were evaluated.

2 Experimental

2.1 Experimental Slag Component Preparation

The based slag system consisted of La_2O_3-SiO_2-FeO ternary slag, which contains a small amount of one or two minor components of Al_2O_3, MnO and B_2O_3. All samples were prepared using analytical reagent grade La_2O_3, SiO_2, Al_2O_3, MnO_2 powder. La_2O_3 (99.95 mass%) was provided by Baogang Rare Earth Science and Technology Co. (Baotou, China). Other analytical reagents were provided by Tianjin Chemical Reagent Co. (Tianjin, China). La_2O_3, SiO_2 and Al_2O_3 reagents were heated in a muffle furnace at 1123 K for 2 h to eliminate adherent moisture and B_2O_3 (99.9 mass%) was dehydrated in vacuum at 393 K for 4 h. MnO was produced by MnO_2 reduction at 1123 K for 2 h in high-purity hydrogen in a tube atmosphere furnace. FeO was prepared by thermal decomposition of $FeC_2O_4{\cdot}2H_2O$ in argon (with a purity of above 99.995 mass%) at 1123 K for 2 h. The final product was confirmed to be a single FeO phase by X-ray diffraction as shown in Figure 1.

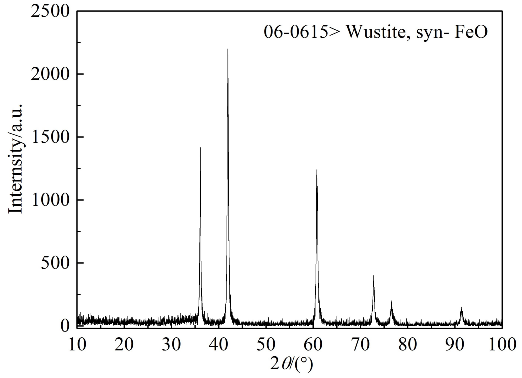

Figure 1: XRD pattern of thermal decomposition product for $FeC_2O_4{\cdot}2H_2O$ in argon at 850°C.

2.2 Viscosity and Break-Temperature Test

The slag viscosity was measured by the internal rotating-cylinder method. The viscosity test principle and instrument were the same as was used in our previous work [14].

According to the designed slag composition, ~160 g of prepared reagents were mixed, briquetted and placed into a molybdenum crucible with a 40-mm diameter and a 75-mm height, and the molybdenum crucible was placed inside the constant-temperature zone of the high-temperature resistance furnace. The temperature of the heating furnace was controlled by a Pt-PtRd thermocouple and the temperature-control accuracy was approximately ± 1 K. The sample was heated to 1823 K and held for 1 h in argon at 0.2 NL/min to obtain a homogeneous melt. The molybdenum spindle was immersed into a liquid slag bath and rotated. The viscosity at a constant temperature of 1823 K was obtained. The viscosity was measured continuously while the slag temperature reduction was controlled at 3 K/min. When the viscosity increased to 6.0 Pa·s, the molybdenum spindle stopped rotating and the furnace was heated again to 1823 K so that the molybdenum spindle was removed from the melted slag. The slag break temperature was identified as the temperature at which there is a significant change in viscosity during the cooling cycle.

Table 1: Constant-Temperature Viscosity and Break Temperature of La_2O_3-SiO_2-FeO Ternary Basic Slag.

Sample No.	Slag Composition (mass%)			Constant-Temperature Viscosity (η) (Pa·s)	Break Temperature [K]
	La_2O_3	SiO_2	FeO		
1	45	40	15	0.4598	1727
2	45	35	20	0.2806	1692
3	45	30	25	0.1443	1674
4	50	40	10	1.0447	1797
5	50	35	15	0.3009	1758
6	50	30	20	0.1082	1761
7	50	25	25	0.1376	1715
8	55	30	15	0.1961	1810
9	55	25	20	0.0541	1784
10	55	20	25	0.0811	1780

3 Results and Disscussion

3.1 Viscosity and Break Temperature of La_2O_3-SiO_2-FeO Ternary Slag

The composition, constant-temperature viscosity at 1823 K and break temperature of the La_2O_3-SiO_2-FeO ternary basic slag are given in Table 1. The viscosity curves are shown in Figure 2. For a fixed La_2O_3 mass content, the viscosity at 1823 K and the break temperature is lowered with a decrease in SiO_2 content from 40 to 30 mass% and an increase in FeO content from 15 to 25 mass%. When the La_2O_3 content is 50 mass%, the viscosity-temperature curve of the 10 mass% FeO is supplied to observe better the effect of FeO content change on the break temperature of the slag. For a certain FeO content, the viscosity at 1823 K decreases, but the break temperature of the La_2O_3-SiO_2-FeO ternary slag increases as the La_2O_3 content increases from 45, to 50 to 55 mass% as shown in Table 1.

The decrease in slag viscosity is related closely to a depolymerization of the silicate structure. The free oxygen ions could break the bridged oxygen in the Si-O network structure and simplify the complex network silicate structure to decrease the slag viscosity. The availability of free oxygen ions possibly increases with increasing La_2O_3 content and thus the viscosity of the La_2O_3-SiO_2-FeO slag decreases. Toop and Samis have reported that FeO supplies free oxygen ions in CaO-SiO_2-FeO melts [15]. Moreover, FeO contributes to the formation of a low-melting-point substance, such as fayalite (Fe_2SiO_4). As a result, increasing FeO content decrease the slag viscosity and melting temperature. The lower viscosity and melting temperature of slag as an FeO additive has been already practiced in steelmaking process. The variation behavior of the viscosity and break temperature for the La_2O_3-SiO_2-FeO slag and the CaO-SiO_2-FeO slag is similar with an increase in FeO content. It may be that the basic properties of La_2O_3 and CaO are some similar. However, the reported proof for the similar basic character of La_2O_3 and CaO is scarce at present.

3.2 Effect of Single Minor Component on the Based Slag System Viscosity and Break Temperature

In a $LaNi_5$-type hydrogen-storage alloy, a small amount of Al and Mn are doped to replace Ni to improve the electrochemical properties [16, 17]. In a NdFeB permanent magnet, Co and Al are added to replace Fe to optimize the magnetic performance and corrosion behavior [18, 19]. After gas-selective reduction-oxidation, the active elements Mn, Al, B in the hydrogen-storage alloy and in the NdFeB waste are easily oxidized to MnO, Al_2O_3 and B_2O_3 [11]. They can remain in the rare-earth-oxide slag and may affect the viscosity and break temperature of the La_2O_3-SiO_2-FeO based slag significantly.

The influence of single minor components Al_2O_3, MnO and B_2O_3 on the viscosity and break temperature of the La_2O_3-SiO_2-FeO slag was studied to establish which component has a greater impact on the above values. So that proper viscosities and break temperatures can be obtained by adjusting the slag composition. The high content of rare-earth oxide in the La_2O_3-SiO_2-FeO slag would reduce the acid consumption for the same amount of leaching rare-earth oxides although a high rare-earth-oxide content results in a high break temperature of the slag (shown in Table 1), which is undesirable for slag-metal separation. Therefore, the mass fraction ratio of the main components

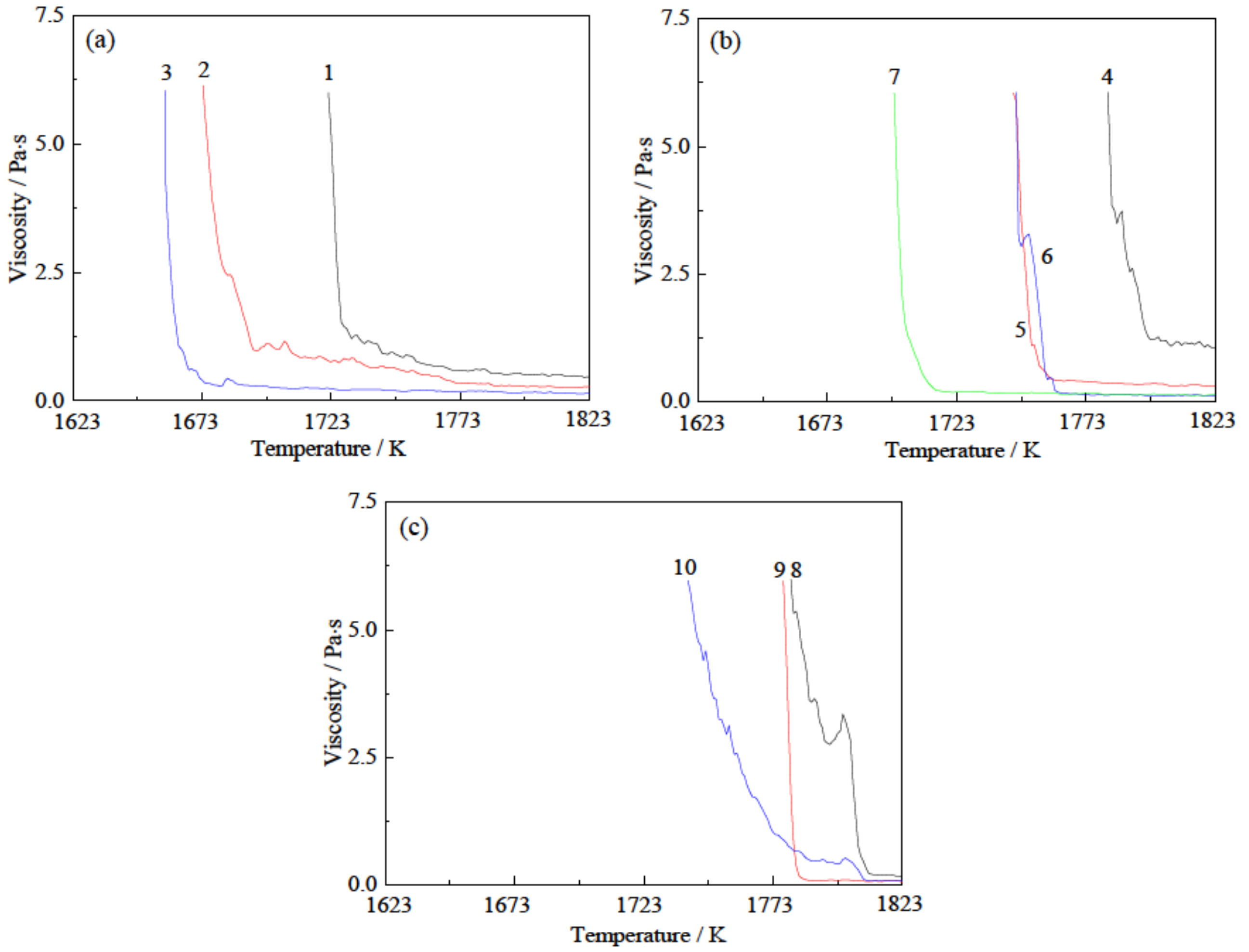

Figure 2: Viscosity curves of La_2O_3-SiO_2-FeO ternary basic slags. (a) 45 mass% La_2O_3-SiO_2-FeO slags, (b) 50 mass% La_2O_3-SiO_2-FeO slags, (c) 55 mass% La_2O_3-SiO_2-FeO slags.

Table 2: Constant-Temperature Viscosity and Break Temperature of La_2O_3-SiO_2-FeO-Al_2O_3 Slag.

Sample No.	Slag Composition (mass%)				Constant-Temperature	Break Temperature
	La_2O_3	SiO_2	FeO	Al_2O_3	Viscosity (η) (Pa·s)	[K]
8	55	30	15	0	0.1961	1810
11	53.9	29.4	14.7	2	0.1812	1800
12	52.8	28.8	14.4	4	0.1913	1748
13	51.7	28.2	14.1	6	0.1476	1719

in the La_2O_3-SiO_2-FeO-Al_2O_3 (MnO, B_2O_3) slag is fixed at 55:30:15.

3.2.1 Effect of Al_2O_3 on La_2O_3-SiO_2-FeO slag viscosity and break temperature

The composition, constant-temperature viscosity at 1823 K and break temperature of the La_2O_3-SiO_2-FeO-Al_2O_3 slag are listed in Table 2. The viscosity curves are shown in Figure 3. The viscosity change at 1823 K is insignificant but the break temperature is lowered substantially. The break temperature of the La_2O_3-SiO_2-FeO slag that is doped with 0, 2, 4 and 6 mass% Al_2O_3 is reduced from 1810 to 1800, 1748 and 1719 K, respectively. This result implies that the dissolution of a small amount of Al_2O_3 into the slag, even from low-cost corundum-crucible corrosion, does not cause melting problems in the slag–metal separa-

Table 3: Constant-Temperature Viscosity and Break Temperature of La_2O_3-SiO_2-FeO-MnO Slag.

Sample No.	Slag Composition (mass%)				Constant-Temperature	Break Temperature
	La_2O_3	SiO_2	FeO	MnO	Viscosity (η) (Pa·s)	[K]
8	55	30	15	0	0.1961	1810
14	52.8	28.8	14.4	4	0.1242	1804
15	51.7	28.2	14.1	6	0.1208	1772
16	50.6	27.6	13.8	8	0.1007	1754

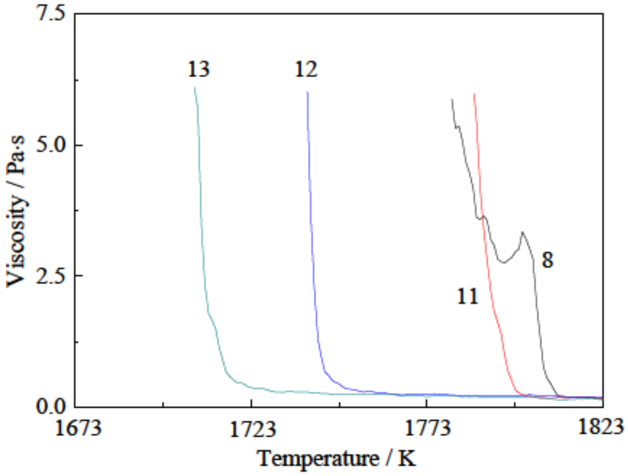

Figure 3: Viscosity curves of La_2O_3-SiO_2-FeO-Al_2O_3 slags.

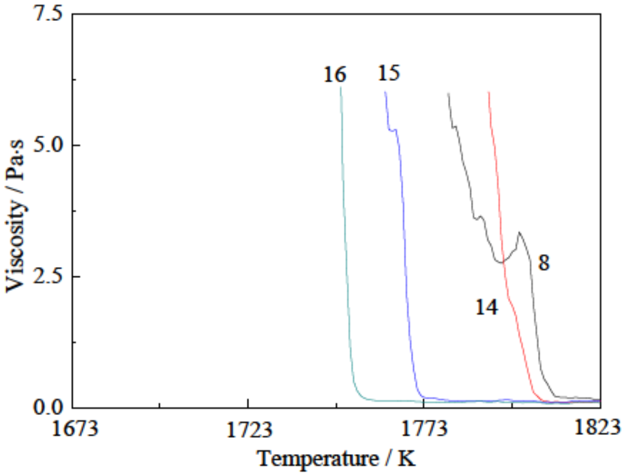

Figure 4: Viscosity curves of La_2O_3-SiO_2-FeO-MnO slags.

tion process. The break temperature decrease with the an increase in Al_2O_3 content from 0 to 14.35 mass% in CaO-7 mass% MgO-SiO_2-20 mass% Fe_tO-Al_2O_3 slags has been reported [20], in which the slag-composition range and ternary basicity ($(W_{CaO} + W_{MgO})/W_{SiO_2} = 1.5$) was approximately same as those in this study ($W_{La_2O_3}/W_{SiO_2} = 1.83$).

3.2.2 Effect of MnO on La_2O_3-SiO_2-FeO slag viscosity and break temperature

Table 3 presents the composition, constant-temperature viscosity at 1823 K and break temperature of La_2O_3-SiO_2-FeO-MnO slag. Figure 4 shows the curves of viscosity versus temperature for different MnO contents. An increase in MnO addition results in a small decrease in slag viscosity at 1823 K and the break temperature decreases. When the MnO content increases from 0 to 6 and 8 mass%, the slag break temperature is reduced from 1810 to 1772 and 1754 K, respectively. It has been reported that a 0–3 mass% MnO addition lowers the initial and complete melting temperature of the CaO-Al_2O_3 system mold flux because MnO formed low-melting compounds $CaMnSiO_6$ and Mn_2SiO_4 with CaO and SiO_2 [21].

3.2.3 Effect of B_2O_3 on La_2O_3-SiO_2-FeO slag viscosity and break temperature

The composition, constant-temperature viscosity at 1823 K and break temperature of La_2O_3-SiO_2-FeO-B_2O_3 slag and the viscosity curves are given in Table 4 and Figure 5. The slag viscosity at 1823 K does not change visibly, but the break temperature of the slag is reduced significantly with an increase in B_2O_3 content, which is similar to the viscosity variation with the addition of Al_2O_3 in the slag as shown in Table 2. When B_2O_3 content changes from 0 to 2, 3 and 4mass%, the break temperature of the slag decreases from 1810 to 1731, 1727 and 1697 K, respectively. This finding agrees with many reports [22, 23] although the slag compositions differ.

B_2O_3 is a typical acidic oxide, which behaves as a network former in the current La_2O_3-SiO_2-FeO alkaline slag. Amphoteric oxide Al_2O_3 also tends to behave as a network former in the alkaline slag. Consequently, the same viscosity-change result in the La_2O_3-SiO_2-FeO slag with

Table 4: Constant-Temperature Viscosity and Break Temperature of La_2O_3-SiO_2-FeO–B_2O_3 Slag.

Sample No.	Slag Composition (mass%)				Constant-Temperature Viscosity (η) (Pa·s)	Break Temperature [K]
	La_2O_3	SiO_2	FeO	B_2O_3		
8	55	30	15	0	0.1961	1810
17	53.9	29.4	14.7	2	0.1476	1731
18	53.35	29.1	14.55	3	0.1141	1727
19	52.8	28.8	14.4	4	0.1443	1697

Table 5: Constant-Temperature Viscosity and Break Temperature of La_2O_3-SiO_2-FeO-Al_2O_3-MnO and La_2O_3-SiO_2-FeO-Al_2O_3-B_2O_3 Slags.

Sample No.	Slag Composition (mass%)						Constant-Temperature Viscosity (η) (Pa·s)	Break Temperature [K]
	La_2O_3	SiO_2	FeO	Al_2O_3	MnO	B_2O_3		
13	51.7	28.2	14.1	6	0	0	0.1476	1719
15	51.7	28.2	14.1	0	6	0	0.1208	1772
18	53.35	29.1	14.55	0	0	3	0.1141	1727
20	48.4	26.4	13.2	6	6	0	0.1476	1662
21	50.05	27.3	13.65	6	0	3	0.1544	1585

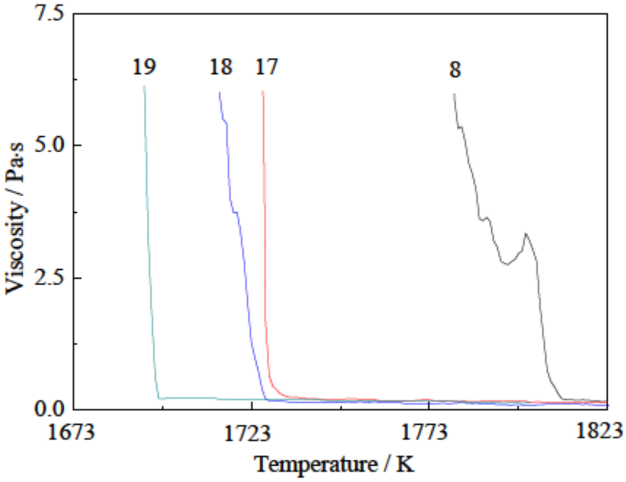

Figure 5: Viscosity curves of La_2O_3-SiO_2-FeO-B_2O_3 slags.

B_2O_3 and Al_2O_3 addition appears as expected. B_2O_3 is used often in metallurgy as a fluxing agent. The reasons for reducing the break temperature of the La_2O_3-SiO_2-FeO slag by B_2O_3 additive could be as follows: (1) The melting point of B_2O_3 is inherently low (723 K). (2) The slag crystallization maybe suppressed by B_2O_3 addition [24]. (3) B_2O_3 reacts with some oxides to form a eutectic of low melting temperature, such as LaB_3O_6 (melting point 1414 K) [25] or FeB_2O_4 (melting point 1223 K) [26], which decreases the break temperature.

The decrease in constant-temperature viscosity at 1823 K by single minor component Al_2O_3, MnO and B_2O_3 is not obvious, and varies from 0.1961 to 0.1007 Pa·s. However, all three components enlarge the temperature range of the low viscosity and obviously decrease the break temperature of the La_2O_3-SiO_2-FeO based slag. The break-temperature reduction ability of the single components is ranked as B_2O_3>Al_2O_3>MnO. For example, when the content of any of the three components in the slag is 4mass%, B_2O_3, Al_2O_3 and MnO lower the break temperature of the La_2O_3-SiO_2-FeO based slag by 113, 62 and 6 K, respectively. It has been reported that using some B_2O_3 to substitute for Al_2O_3 as a fluxing agent decreased the melting temperature of the CaO-based refining flux [27].

3.3 Effect of Complex Components Al_2O_3+MnO and Al_2O_3+B_2O_3 on La_2O_3-SiO_2-FeO slag viscosity and break temperature

After AB_5-type hydrogen-storage alloy and NdFeB waste were selectively oxidized and reduced, Al_2O_3+MnO and Al_2O_3+B_2O_3 could coexist in the rare-earth oxide slag as minor components, respectively. The effect of two components Al_2O_3+MnO and Al_2O_3+B_2O_3, which is close to the real rare-earth waste situation, on the viscosity and break temperature of the La_2O_3-SiO_2-FeO slag was investigated. Here, the Al_2O_3, MnO and B_2O_3 compositions were selected to be 6, 6 and 3 mass%, respectively.

As shown in Table 5 and Figure 6, the coexistence of 6 mass% Al_2O_3 + 6 mass% MnO or 6 mass% Al_2O_3 + 3 mass% B_2O_3 has no effect on the slag viscosity, whereas they continue to reduce the break temperature to 1662 or 1585 K. For the La_2O_3-SiO_2-FeO-6 mass% Al_2O_3-6 mass%

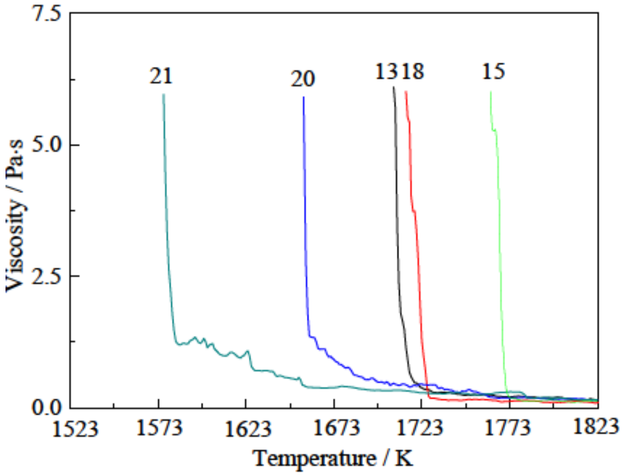

Figure 6: Viscosity curves of La_2O_3-SiO_2-FeO-Al_2O_3-MnO and La_2O_3-SiO_2-FeO-Al_2O_3-B_2O_3 slags.

MnO slag, the reduction in break temperature is 57 and 110 K compared with La_2O_3-SiO_2-FeO-6 mass% Al_2O_3 and La_2O_3-SiO_2-FeO-6 mass% MnO slags. For La_2O_3-SiO_2-FeO-6 mass% Al_2O_3-3 mass% B_2O_3 slag, the reduction level is 134 and 142 K compared with La_2O_3-SiO_2-FeO-6 mass% Al_2O_3 and La_2O_3-SiO_2-FeO-3 mass% B_2O_3 slags, respectively. It can be concluded that the melting speed of the slag is increased by the coexistence of Al_2O_3+MnO or Al_2O_3+B_2O_3 because of the low break temperature, and the improved stability of the slag viscosity varies with temperature, which is favorable for slag-metal separation. The inflection point of the viscosity curves becomes unapparent.

For slag-metal separation, the critical point was the selection of a suitable slag system that fulfils demands such as a low viscosity, and a moderate or low break temperature. In general, the break temperature of the slag is lower by 150 K or more than the melting temperature of the metal. The melting temperature of the recycled metal is expected to reach up to 1773 K, so the break temperature of the slag is best below 1623 K. In this experiment, the low viscosity meets the slag-metal separation requirement for all La_2O_3-SiO_2-FeO slag systems. However, the break temperatures of the La_2O_3-SiO_2-FeO based slag and the slag doped with single Al_2O_3, MnO and B_2O_3 are high, although these oxides could reduce the break temperature of the La_2O_3-SiO_2-FeO based slag to different extents. Two residual components 6 mass% Al_2O_3+6 mass% MnO decrease the break temperature of the slag to close to 1623 K, and the break temperature decreases to below 1623 K when the Al_2O_3 and B_2O_3 contents in the slag are 6 and 3mass% as shown in Table 5. Rare-earth waste often yields two or more minor components in the slag. As a result, the efficient separation of valuable metals and rich rare-earth oxide-containing slag must be achieved using the developed La_2O_3-SiO_2-FeO slag system and simultaneously the slag composition does not affect or affects only slightly the subsequent extraction of rare-earth elements.

4 Conclusions

In this study, the viscosity and break temperature of La_2O_3-SiO_2-FeO ternary slag and the variation as affected by one or two minor components Al_2O_3, MnO and B_2O_3 were investigated. The following conclusions were made:

(1) For 45, 50 or 55 mass% La_2O_3, the viscosity at 1823 K and the break temperature of La_2O_3-SiO_2-FeO ternary slag decreased with an increase in FeO content from 15 to 25 mass% and a decrease in SiO_2 content from 40 to 30 mass%. When the FeO content was fixed at 15, 20 or 25 mass%, the slag viscosity decreased, but the break temperature of the La_2O_3-SiO_2-FeO ternary slag increased with an increase in La_2O_3 content from 45 to 55mass%.
(2) With an increase in individual minor components Al_2O_3 (0–6 mass%), MnO (0–8 mass%) and B_2O_3 (0–4 mass%), the constant-temperature viscosity at 1823 K of the La_2O_3-SiO_2-FeO based slag decreased slightly or remained unchanged, but the break temperature of the slag was reduced significantly. B_2O_3 was most influential, followed by Al_2O_3 and MnO.
(3) The coexistence of Al_2O_3+MnO or Al_2O_3+B_2O_3 reduces the break temperature of the La_2O_3-SiO_2-FeO slag significantly compared with single Al_2O_3, MnO and B_2O_3 and the slag still maintains a low-viscosity feature, which benefits the slag-metal separation.

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