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Density and molar volume of CaO–SiO₂–FeO melts measured by electrostatic levitation aboard the International Space Station

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Abstract

The densities of CaO–SiO₂–FeO melts were measured under microgravity conditions using the Electrostatic Levitation Furnace aboard the International Space Station (ISS-ELF). The nominal FeO contents were 20, 60, and 80 mass%, with CaO and SiO₂ comprising the balance in a 1:1 mass ratio. Density was calculated from the sample mass, while the droplet volume was determined from shadow images recorded during cooling. For all compositions, the density decreased approximately linearly with increasing temperature. At a given temperature, the density increased with increasing nominal FeO content. The maximum relative expanded uncertainty in the density was 3.46%, with a coverage factor of $k = 2$. The measured densities were also converted to molar volumes using the nominal oxide compositions and compared at approximately 1400 °C with literature data and an additive reference. The molar volume decreased from the 20 mass% FeO sample to the 60 mass% FeO sample and changed only slightly between the 60 mass% FeO and 80 mass% FeO samples. The measured molar volumes were generally consistent with the additive-model predictions, although they were slightly larger at higher FeO contents.

Keywords: density; molar volume; CaO–SiO₂–FeO melt; oxide melt; metallurgical slag; electrostatic levitation

1. Introduction

Reliable density data for molten slags are required to analyze fluid flow, heat and mass transport, phase separation, and interfacial phenomena in high-temperature metallurgical processes. The CaO–SiO₂–FeO system is a fundamental subsystem of iron-bearing metallurgical slags, and accurate density data are required for process modeling and thermophysical property databases [1–3].

The density of CaO–SiO₂–FeO and related FeO-bearing silicate melts has been measured using conventional contact methods, including the maximum-bubble-pressure and Archimedean methods [4–7]. However, because these techniques require contact between the melt and crucibles, capillaries, bobs, or other supporting materials, density measurements at high temperatures can be affected by reactions with, or contamination from, them [8, 9]. Therefore, container-less techniques are useful for extending density measurements of melts to higher temperatures [10–14]. In particular, the Electrostatic Levitation Furnace aboard the International Space Station (ISS-ELF) enables stable levitation of oxide droplets under microgravity, which allows density measurements at temperatures where conventional contact methods become difficult to apply [15–19].

In this study, the densities of CaO–SiO₂–FeO melts prepared with nominal FeO contents of 20, 60, and 80 mass% were measured using the ISS-ELF. The temperature dependence of the density was evaluated by linear fitting, and the dependence on FeO content was compared with literature data. The measured densities were also converted to molar volumes using the nominal oxide compositions and compared with literature data and an additive reference constructed by treating the CaO–SiO₂ fraction as a CaO·SiO₂-equivalent component.

2. Methods

Spherical CaO–SiO₂–FeO samples, approximately 2 mm in diameter, were prepared from CaO, SiO₂, and FeO reagent powders. The nominal FeO contents were 20, 60, and 80 mass%, with the balance consisting of CaO and SiO₂ in a 1:1 mass ratio. The samples were designated CS20F, CS60F, and CS80F, respectively. Table 1 lists the nominal compositions and sample masses before and after the ISS-ELF experiments. The powders were weighed according to their nominal compositions, mixed, and melted in an aerodynamic levitation furnace to obtain spherical samples. The samples were mounted

on sample holders and transported to the International Space Station.

The basic configuration and operation of the ISS-ELF were described previously [15]. The sample chamber was evacuated to 500 Pa and backfilled with Ar to a pressure of 2×10^5 Pa. Each spherical sample was electrostatically levitated between three pairs of mutually perpendicular electrodes. The levitated sample was heated and melted using four semiconductor lasers with a wavelength of 980 nm and an output power of 40 W each. The sample temperature was monitored through sapphire windows using a monochromatic pyrometer with a wavelength range of 1.45–1.8 μm . The pyrometer emissivity was fixed at unity throughout the measurements. Shadow images of the droplets were recorded at 60 fps using a CCD camera with ultraviolet backlighting. After the sample was maintained at a high temperature, the laser power was reduced or switched off to initiate cooling. The indicated pyrometer temperatures were corrected using features in the cooling curves, as described in Section 3.1, and the corrected temperatures were used for the density analysis.

The droplet volume, V , was determined from the shadow images acquired during cooling. Figure 1 shows representative optical and shadow images of a levitated $\text{CaO-SiO}_2\text{-FeO}$ droplet; the shadow image was used for volume determination. Images without appreciable shape oscillations were selected, and droplet contours were extracted. Under microgravity, the gravitational deformation of a molten droplet is negligible. Therefore, the droplet width in each image row was treated as the diameter of a circular cross-section at the corresponding height, and the volume was calculated by numerical integration of these disk-shaped cross-sections:

$$V = \frac{\pi}{4} \sum_i w_i^2 s_x^2 s_y, \quad (1)$$

where w_i is the droplet width in pixels in the image row i , and s_x and s_y are the horizontal and vertical image-scale conversion factors, respectively. The image scales were determined from an image of a reference sphere (2.0 mm diameter) recorded near the sample position. The density, ρ , was calculated from the droplet volume and the sample mass measured after the ISS-ELF experiment using an electronic balance:

$$\rho = \frac{m}{V}. \quad (2)$$

The uncertainty in density was evaluated as described in Section 3.3.

3. Results and discussion

3.1. Temperature dependence of density

Figure 2 shows representative cooling curves obtained during the ISS-ELF density measurements. Recalescence-related inflections were observed for the CS80F and CS60F samples. The liquidus temperatures were calculated using FactSage 8.4 with the FToxid database [20], in which Fe was treated on an FeO-equivalent basis. The effective emissivity was determined such that the indicated temperature at the recalescence-related inflection point agreed with the calculated liquidus temperature. This procedure yielded an effective emissivity of approximately 0.8 for both samples.

For CS20F, no recalescence-related inflection was observed. As shown in Figure 3, the recovered CS20F sample exhibited a smooth glassy appearance, whereas the CS80F and CS60F samples exhibited rougher, opaque surfaces. The glassy appearance was consistent with the absence of a recalescence-related inflection in the cooling curve. Therefore, the effective emissivity estimated from CS80F and CS60F (approximately 0.8) was used to correct the indicated temperatures of CS20F.

Figure 4 shows the temperature dependence of the density of the CaO–SiO₂–FeO melts measured using the ISS-ELF, together with the data reported by Lee and Gaskell [5]. The measurements covered temperature ranges of 1331–1623 °C for CS80F, 1313–1934 °C for CS60F, and 1280–1942 °C for CS20F. The error bars represent the expanded uncertainty with a coverage factor of $k = 2$. For all samples, the density decreased approximately linearly with increasing temperature. At a given temperature, the density increased with increasing nominal FeO content. The data reported by Lee and Gaskell also showed negative temperature coefficients and exhibited the same dependence on FeO content.

The temperature dependence of the density of each sample was determined by fitting the measured data to the following equation:

$$\rho(T) = \rho_L + \frac{d\rho}{dT}(T - T_L), \quad (3)$$

where T_L is the liquidus temperature, ρ_L is the density at T_L , and $d\rho/dT$ is the temperature coefficient of density. The fitted values of ρ_L and $d\rho/dT$, together with T_L

and the analyzed temperature ranges, are summarized in Table 2.

3.2. Composition dependence of density and molar volume

The measured densities were converted to molar volumes using the nominal oxide compositions listed in Table 1. The molar volume was used to compare the present density data with literature data on a common oxide-component basis. The mole fraction of each oxide component, X_i , and the mean formula mass of the melt, $\bar{M}$, were calculated as follows:

$$X_i = n_i / (n_{\text{CaO}} + n_{\text{SiO}_2} + n_{\text{FeO}}) \quad (4)$$

$$\bar{M} = X_{\text{CaO}} M_{\text{CaO}} + X_{\text{SiO}_2} M_{\text{SiO}_2} + X_{\text{FeO}} M_{\text{FeO}}, \quad (5)$$

where n_i and M_i are the amount of substance and the formula mass of the oxide component i , respectively. The molar volume was then calculated as:

$$V_m = \frac{\bar{M}}{\rho}. \quad (6)$$

Figure 5 compares the molar volumes at approximately 1400 °C with literature data [4–7] and an additive reference. For the present samples, the densities at 1400 °C were obtained from the fitted relationships in Eq. (3), and the molar volumes were calculated using Eq. (6).

To construct the additive reference, a reference curve was calculated assuming volume additivity between FeO and a CaO·SiO₂-equivalent pseudo-component. The CaO–SiO₂ fraction was treated as this pseudo-component, and its volume was estimated using the density of the corresponding binary liquid reported by Lee and Gaskell [5]. The amount of this component, n_{CS} , was defined as

$$n_{\text{CS}} = \frac{n_{\text{CaO}} M_{\text{CaO}} + n_{\text{SiO}_2} M_{\text{SiO}_2}}{M_{\text{CaO} \cdot \text{SiO}_2}}. \quad (7)$$

The additive reference was then expressed on the same oxide-component molar-volume basis as the measured data:

$$V_m^{\text{add}} = \frac{n_{\text{CS}} V_{m, \text{CaO} \cdot \text{SiO}_2} + n_{\text{FeO}} V_{m, \text{FeO}}}{n_{\text{CaO}} + n_{\text{SiO}_2} + n_{\text{FeO}}}, \quad (8)$$

where $V_{m, \text{CaO} \cdot \text{SiO}_2}$ and $V_{m, \text{FeO}}$ are the reference molar volumes used for the CaO·SiO₂-equivalent term and FeO term in the additive reference at 1400 °C, respectively. The CaO·SiO₂-equivalent molar volume was calculated from the CaO·SiO₂ density described

above, whereas the FeO molar volume was calculated from FeO-side density data reported by Hara et al. [7]. This additive reference is a first-order volume additivity estimate on an oxide-component molar-volume basis [21] and does not imply thermodynamic ideality.

For the present samples, the molar volume decreased from CS20F to CS60F and changed only slightly between CS60F and CS80F. The present values were generally consistent with the additive reference, although they were slightly larger at higher FeO contents.

3.3. Uncertainty of density measurement

The uncertainty in the density measurements was evaluated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM) [22]. The uncertainty propagation was based on the volume and density calculations described in Section 2. By substituting the image-scale conversion factors, $s_x = D_{\text{ref}}/P_x$ and $s_y = D_{\text{ref}}/P_y$, into Eqs. (1) and (2), the density can be rewritten directly in terms of the measurement parameters:

$$\rho = \frac{4mP_x^2P_y}{\pi D_{\text{ref}}^3 \sum_i w_i^2}, \quad (9)$$

where m is the sample mass measured after the ISS-ELF experiment; D_{ref} is the measured diameter of the reference sphere; P_x and P_y are the horizontal and vertical pixel diameters of the reference sphere, respectively; and $\sum_i w_i^2$ is the image factor obtained from the droplet contour.

The standard uncertainty contribution of each input quantity was calculated as $u_i = |c_i|u(x_i)$, where the sensitivity coefficient is $c_i = \partial\rho/\partial x_i$. The standard uncertainties originate from the measurement resolution and repeatability. The resolutions of the electronic balance, micrometer used for the reference-sphere diameter measurement, and camera images were treated as rectangular distributions, and their standard uncertainties were obtained by dividing the corresponding scale intervals by $\sqrt{3}$. The repeatability components for all input quantities were evaluated from the standard deviations of the repeated measurements. The repeatability of the image factor was determined by analyzing approximately 100 frames captured under identical conditions. The combined standard uncertainty was then calculated using the root-sum-of-squares method:

$$u_c(\rho) = \sqrt{\sum_i u_i^2(\rho)} . \quad (10)$$

Table 3 presents the uncertainty budget for CS80F, which shows the largest relative expanded uncertainty among the three samples. The combined standard uncertainty was 61.3 kg m⁻³, corresponding to 1.73% of the representative density used for the uncertainty evaluation. With a coverage factor of $k = 2$, the expanded uncertainty was 122.6 kg m⁻³, corresponding to 3.46%.

The largest contribution arose from the repeatability of the reference-sphere diameter measurement because D_{ref} enters the volume calculation as a cubic term (D_{ref}^3).

4. Conclusions

The densities of CaO–SiO₂–FeO melts with nominal FeO contents of 20, 60, and 80 mass% were measured using the ISS-ELF. For all samples, the density decreased approximately linearly with increasing temperature. At a given temperature, the density increased with increasing nominal FeO content. These results provide density data for CaO–SiO₂–FeO melts over a high-temperature range where conventional contact methods are difficult to apply, and are expected to serve as reference data for metallurgical process modeling and thermophysical-property databases. The maximum relative expanded uncertainty in the density was 3.46%, with a coverage factor of $k = 2$.

The measured densities were converted to molar volumes using the nominal oxide compositions. At approximately 1400 °C, the molar volume decreased from CS20F to CS60F and changed only slightly between CS60F and CS80F. The present values were generally consistent with the additive reference, although they were slightly larger at higher FeO contents. The additive reference represents a simple volume additivity estimate and does not imply thermodynamic ideality.

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Statements and Declarations

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Competing interests

The authors declare that they have no competing interests.

Author contributions

Y.S.: formal analysis, visualization, and writing—original draft. M.W.: supervision and project administration. T.I., C.K., and Y.W.: investigation and data acquisition using the ISS-ELF. All authors contributed to the discussion of the results, reviewed and edited the manuscript, and approved the final version.

Data availability

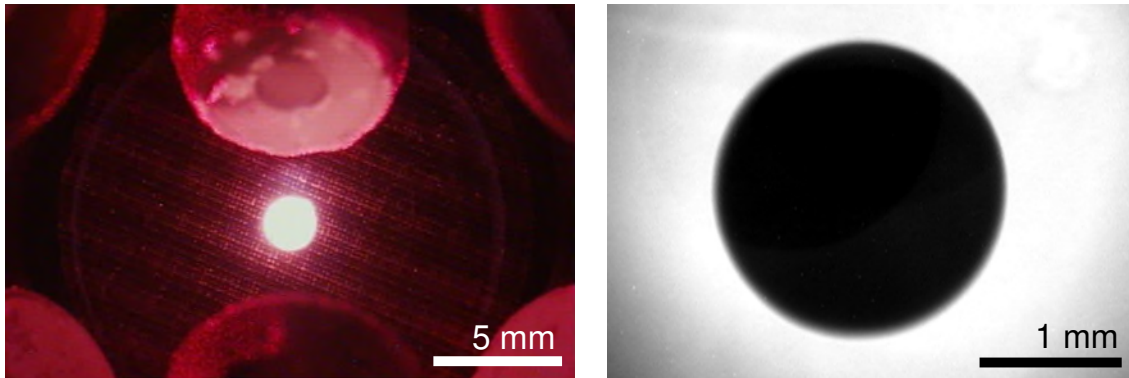
The datasets generated and analyzed during the current study are available from the corresponding author on reasonable request.

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1 **Figures**



2
3 **Fig. 1** Representative optical and shadow images of a levitated CaO–SiO₂–FeO droplet.
4 The shadow image was used for volume evaluation

5

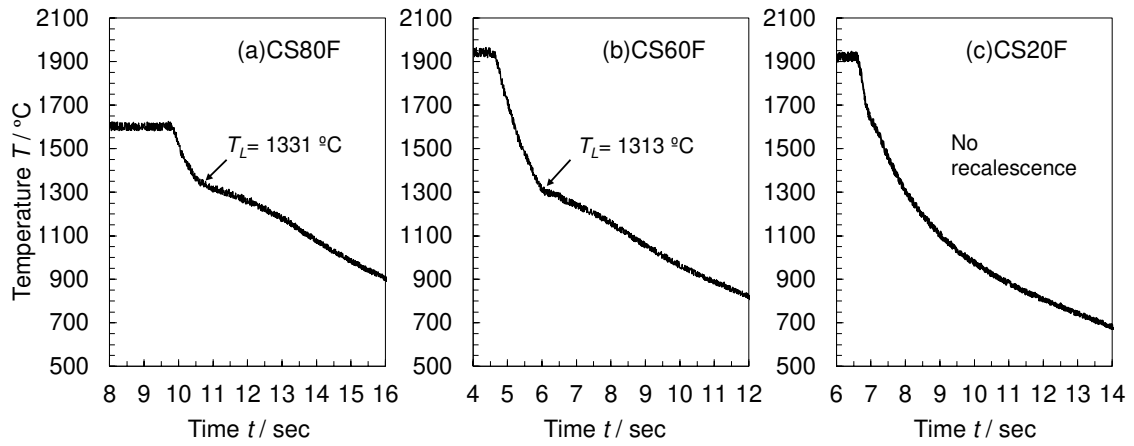


Fig. 2 Representative cooling curves of levitated $\text{CaO-SiO}_2\text{-FeO}$ droplets measured using ISS-ELF. Recalescence-related inflections were observed for (a) CS80F and (b) CS60F and were assigned to the liquidus temperatures calculated using FactSage 8.4 for temperature correction. No recalescence-related inflection was observed for (c) CS20F

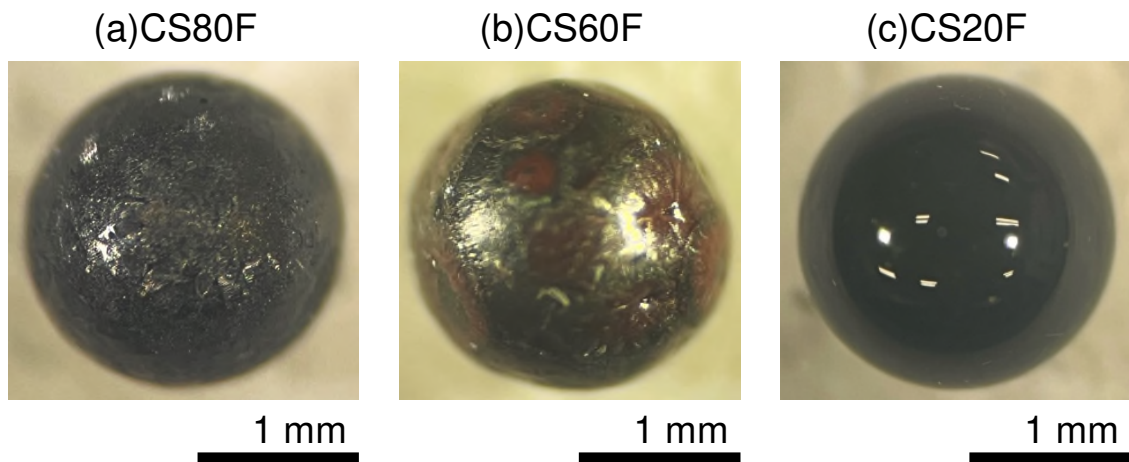
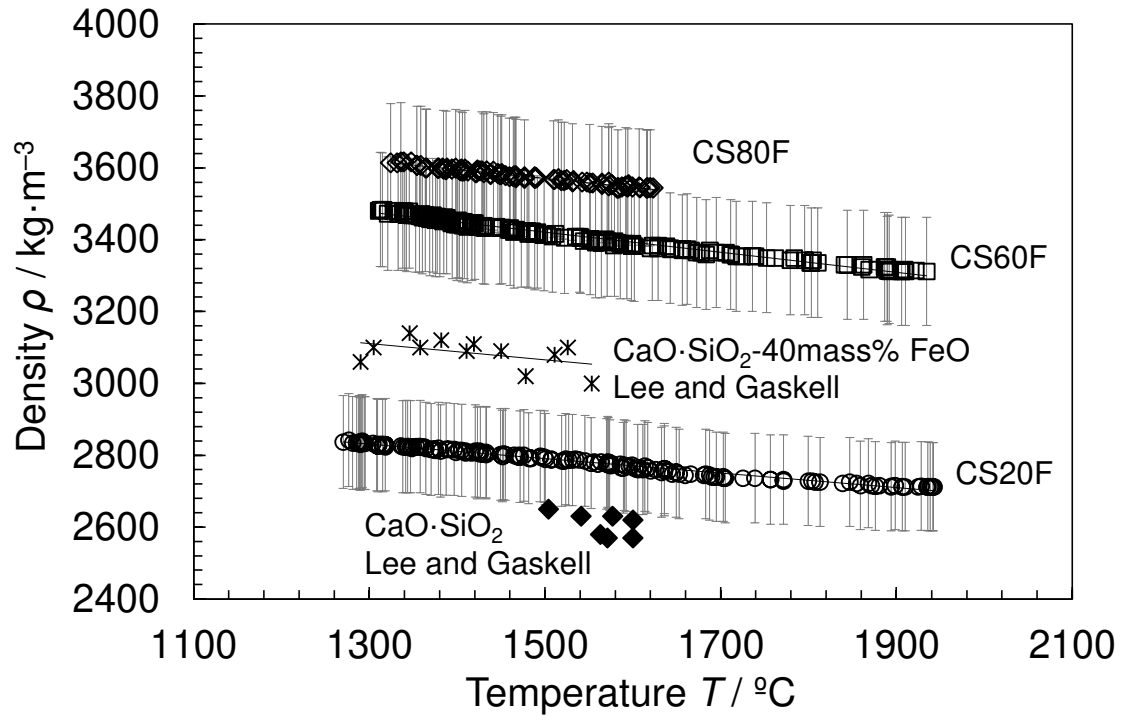


Fig. 3 Optical images of the recovered $\text{CaO-SiO}_2\text{-FeO}$ samples after ISS-ELF measurements: (a) CS80F, (b) CS60F, and (c) CS20F. The CS20F sample showed a smooth glassy appearance, whereas CS80F and CS60F showed rougher opaque surfaces

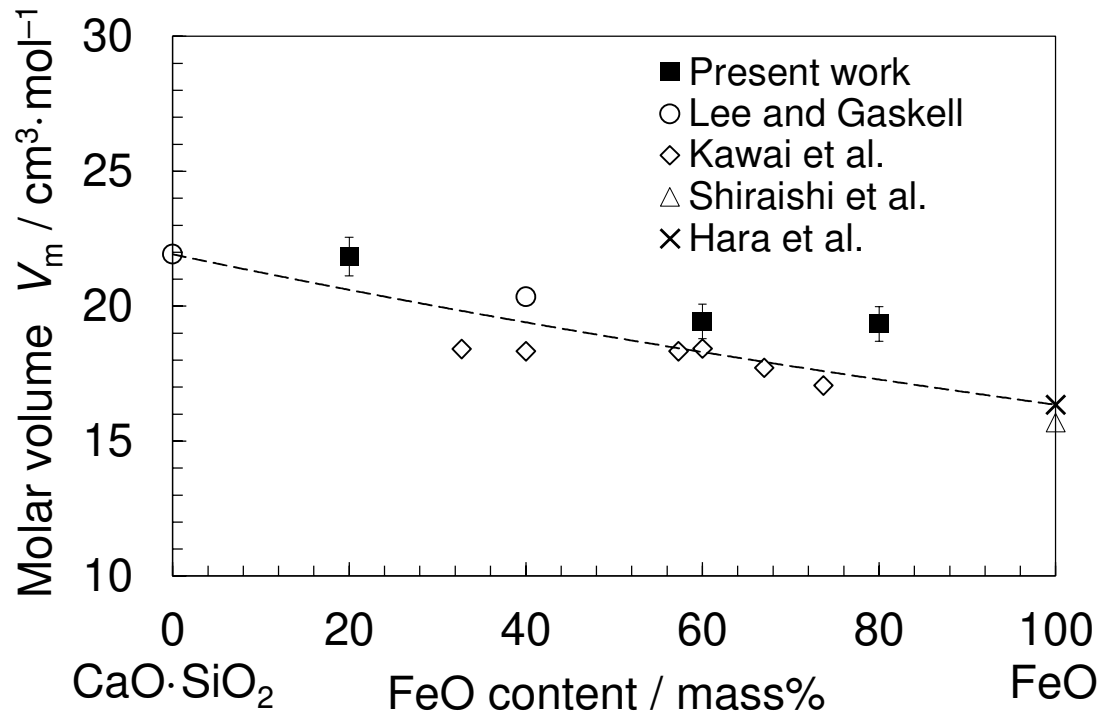
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2

3 **Fig. 4** Temperature dependence of the density of CaO-SiO₂-FeO melts measured using
 4 the ISS-ELF, together with the density data reported by Lee and Gaskell [5]

1



2

3 **Fig. 5** Molar volume of CaO–SiO₂–FeO melts at approximately 1400 °C as a function of
4 FeO content, together with literature data for related CaO–SiO₂–FeO compositions [4–7].
5 The dashed line represents an additive reference calculated by treating the CaO–SiO₂
6 fraction as a CaO·SiO₂-equivalent component

Tables

Table 1. Nominal compositions of the CaO–SiO₂–FeO samples and their masses before and after the ISS-ELF experiments.

Sample	CaO	SiO ₂	FeO	Sample mass	
	[mass%/mol%]	[mass%/mol%]	[mass%/mol%]	Before [mg]	After [mg]
CS80F	10 / 12.2	10 / 11.4	80 / 76.4	18.5	16.2
CS60F	20 / 23.4	20 / 21.8	60 / 54.8	16.1	15.4
CS20F	40 / 43.0	40 / 40.2	20 / 16.8	14.1	12.8

Table 2. Density at the liquidus temperature and temperature coefficients of molten CaO–SiO₂–FeO melts.

Sample	Liquidus temp. T_L [°C]	Density ρ_L , [kg·m ⁻³]	Temp. coefficient $d\rho/dT$, [kg·m ⁻³ °C ⁻¹]	Temp. range [°C]
CS80F	1331	3612	-0.2458	1331–1623
CS60F	1313	3473	-0.2796	1313–1934
CS20F	1280	2836	-0.2021	1280–1942

1

Table 3. Uncertainty budget for the density measurement of CS80F.

Source of uncertainty	Quantity	Standard uncertainty, $u(x_i)$	Sensitivity coefficient, c_i	Uncertainty contribution, $u_i(\rho)$
Resolution of electronic balance	m	2.89×10^{-8} kg	2.19×10^8 m^{-3}	6.31 kg m^{-3}
Repeatability of mass measurement	m	1.00×10^{-7} kg	2.19×10^8 m^{-3}	21.9 kg m^{-3}
Resolution of reference-sphere diameter	D_{ref}	2.89×10^{-6} m	-5.31×10^6 kg m^{-4}	15.3 kg m^{-3}
Repeatability of reference-sphere diameter	D_{ref}	1.00×10^{-5} m	-5.31×10^6 kg m^{-4}	53.1 kg m^{-3}
Horizontal image resolution	P_x	0.289 px	30.2 kg m^{-3} px^{-1}	8.72 kg m^{-3}
Repeatability of horizontal-diameter detection	P_x	0.302 px	30.2 kg m^{-3} px^{-1}	9.12 kg m^{-3}
Vertical image resolution	P_y	0.289 px	13.5 kg m^{-3} px^{-1}	3.91 kg m^{-3}
Repeatability of vertical- diameter detection	P_y	0.175 px	13.5 kg m^{-3} px^{-1}	2.37 kg m^{-3}
Repeatability of volume evaluation	$\sum_i w_i^2$	$5.89 \times 10^3 \text{ px}^2$	-3.38×10^{-4} $\text{kg m}^{-3} \text{ px}^{-2}$	1.99 kg m^{-3}
Combined standard uncertainty, $u_c(\rho)$ 61.3 kg m^{-3} (1.73%)				
Expanded uncertainty, $U(\rho)$, $k = 2$ 122.6 kg m^{-3} (3.46%)				

2

3