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Production of Strontium through a Vacuum Aluminothermic Process

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Abstract
The production of strontium (Sr) metal from its oxide via an aluminothermic process under 1–5 mbar pressure was examined in this study. SrO of 99% purity by weight was used in the experiments. The effects of stoichiometry and the duration of the process on the efficiency ratio of the metallic strontium were examined. The effects of functional additives (BaO, CaO, CaC_2) were also examined. Characterization was carried out using X-ray diffraction spectrometry (XRD), atomic absorption spectrometry (AAS) and flame photometry techniques. The highest Sr efficiency ratio obtained was 96.89% in the experiment conducted at 1250 °C with 300% Al stoichiometry and 300% BaO stoichiometry.

Keywords Strontium · Functional additives · Vacuum aluminothermic reduction

1 Introduction

The element strontium (Sr) is in the second group of the periodic table and is one of the alkaline earth metals. Strontium's atomic number is 38, and it is between calcium and barium in the periodic table. Strontium was discovered by Crawford in 1790, and metallic strontium was isolated by Davy for the first time in 1808 from a strontium carbonate compound obtained from strontian deposits in Scotland. Strontium is more reactive than magnesium and calcium and less reactive than barium. Therefore, it is difficult to keep strontium stable in its metallic form. Strontium easily reacts with H_2O, O_2, N_2, F_2 and S to produce compounds [1, 2].

According to the 2019 United States Geological Survey (USGS) data, 260,000 tons of strontium was produced from mines worldwide in 2019, and the earth's strontium deposits are estimated at over 1 billion tons [3]. Strontium has various application areas in industry today. The thin-film production processes are one example of its use. Physical vapor deposition (PVD) techniques such as magnetron sputtering, thermal evaporation and molecular beam epitaxy (MBE) are the most commonly used methods for the growth of thin films [4–6]. In these applications, strontium is generally used in compound form. Strontium, bismuth, titanium and tantalum thin films are used in ferroelectric and Schottky-based microelectronic data storage applications. Strontium is also used in Sr-hexaferrite form in motors, speakers, high-frequency devices, magnetic recording media and other applications [7, 8]. In the metallurgical industry, strontium is used in metallic form to make compounds. Metallic strontium and its alloys are used as fluxes in the metallurgical industry to purify other metals and to capture gases from electronic tubes. The hardness and the durability of lead and copper are improved by adding very small amounts of strontium to the liquid bath during the production process. Strontium–silicon compounds are used as inoculants in high-quality cast iron production. Strontium is also used as a modifier. Strontium changes the eutectic silicon in hypo- and hypereutectic aluminum/silicon cast alloys from coarse platelet to fine fiber form [9]. It is known that grain refinement is an important method for improving the properties and formability of magnesium alloys. Recent results indicate that Sr can also be potentially effective in the grain refinement of magnesium alloys [10–12]. Since adding pure Sr to magnesium alloys causes serious burning loss, the amount of Sr to use in this process is difficult to adjust.

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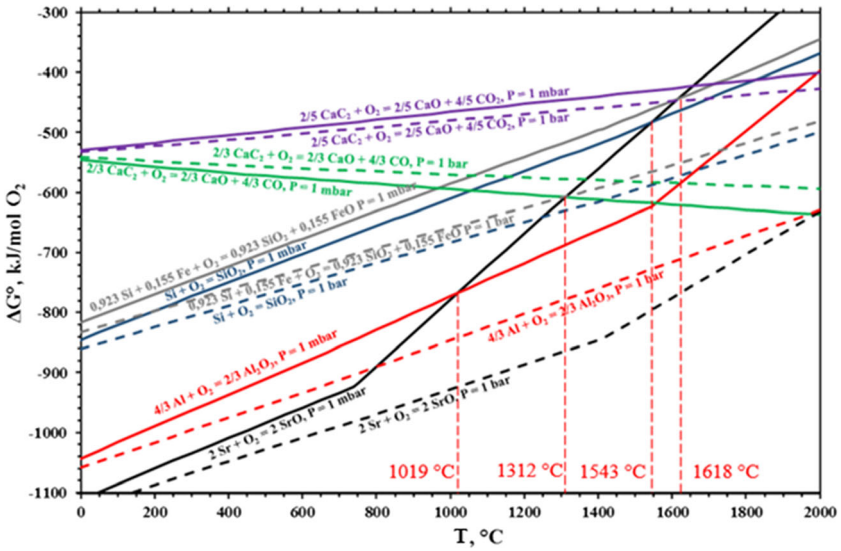

Fig. 1 Sr reduction using aluminum, silicon, ferrosilicon and calcium carbide

Therefore, Sr is frequently used in master alloy form, such as Al-Sr and Mg-Sr master alloys. Recent studies show that adding Al-Sr or Mg-Sr master alloys to magnesium alloys such as AZ31 can effectively refine the grains of magnesium alloys [11]. Zinc–aluminum alloys are generally stronger than most other aluminum alloys, and they have high bearing properties. In spite of their advantages, however, they suffer from shrinkage defects at the lower surface of the castings [13]. Sahoo et al. reported that strontium addition below 0.06% by mass reduced this problem in Zn-27% A1 alloys [14].

Because Sr is a reactive material, the production of Sr is only feasible with carefully controlled processes. Today, the most efficient and feasible means of Sr production is the metallothermic reduction of Sr oxide [15, 16]. In this process, the Sr source is mixed with an excess amount of molten metal, which acts as the main reductant. Sr is dissolved in the molten metal and is then extracted via a vacuum distillation process.

The method investigated in the present study is the vacuum metallothermic reduction process [17]. Timminco Metals (Ontario, Canada) used a method similar to the Pidgeon process to produce strontium [9]. The reaction is endothermic, and the equilibrium is continually and favorably shifted to the right using a high vacuum. The gaseous metal is condensed in a cooler area of the apparatus.

The first method for producing metallic strontium involved salt electrolysis using strontium chloride. Efforts have been to improve this process to increase the tendency of metallic strontium to migrate into the molten electrolyte, but the results have been unsatisfactory. The most effective method for the production of metallic strontium is metallothermic reduction from strontium oxide. In this process, strontium oxide is reduced using aluminum according to Eq. (1) [18].

$$3SrO + 2Al \rightarrow 3Sr + Al_2O_3 \quad (1)$$

In this study, the production of strontium metal from its oxide was examined. The aim of the study was to investigate the parameters affecting the aluminothermic reduction of SrO using aluminum as the reductant. The charge composition, amount of strontium oxide, functional additives and process duration were considered as variables in the experiments which were carried out in order to obtain high efficiency.

Table 1 Minimum reduction temperature (°C) of Sr using Al, Si, FeSi and CaC_2 at 1 bar and 1 mbar

Reductant	Pressure	
	1 bar	1 mbar
Silicon	2654 °C	1543 °C
Aluminum	1884 °C	1019 °C
FeSi	3050 °C	1618 °C
CaC_2	2102 °C	1312 °C

2 Theoretical Background

Before the vacuum aluminothermic reduction experiments were conducted, a thermochemical simulation was performed to estimate the effect of the precursor composition on the reduction process. Some studies in the literature have examined the change in Gibbs free energy during the formation of strontium. The change in the decomposition of $SrCO_3$ to SrO was examined by Li et al., who determined that delta G values became more negative for Ba, Ca, Sr and Mg carbonates as the temperature

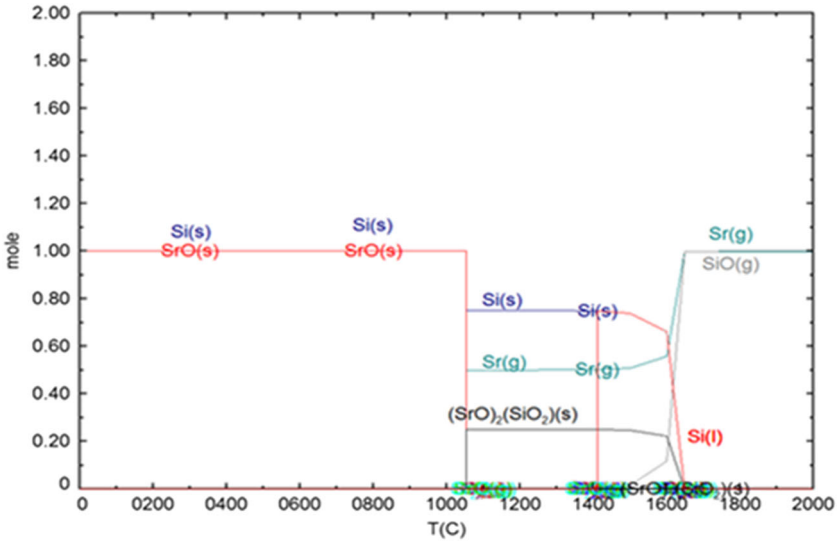

Fig. 2 Silicothermic Sr extraction modeling under 1 mbar atmosphere

increased [19]. In the present study, the transformation parameters of SrO to Sr with increasing temperature were investigated. The effects of possible reductants (Al, Si, FeSi, CaC_2) on the products under 1 bar and 1 mbar pressure were examined using FactSage 6.4 software. Figure 1 and Table 1 show the minimum reduction temperature for the SrO reduction process for different reductants.

According to Fig. 1 and Table 1, Sr reduction is possible at 1 mbar vacuum atmosphere with Si, Al, FeSi and CaC_2 at 1536, 996, 1618 and 1312 °C, respectively. These reduction reactions were also examined without the vacuum atmosphere, and the minimum reduction temperature of the reactions increased to 2654, 1884, 3050 and 2102 °C for the same elements and compounds, respectively.

Figure 2 shows the Sr reduction modeling when silicon is used as reductant under 1 mbar atmosphere. First, strontium is obtained in gas phase at 1050 °C, but there is another strontium oxide phase $(SrO)_2(SiO_2)_{(s)}$ in the mixture at this temperature. The reaction occurs according to Eq. (2). The reduction reaction eliminating the remaining strontium oxide phase starts at 1536 °C. The remaining strontium oxide is reduced at temperatures above 1536 °C according to Eq. (3).

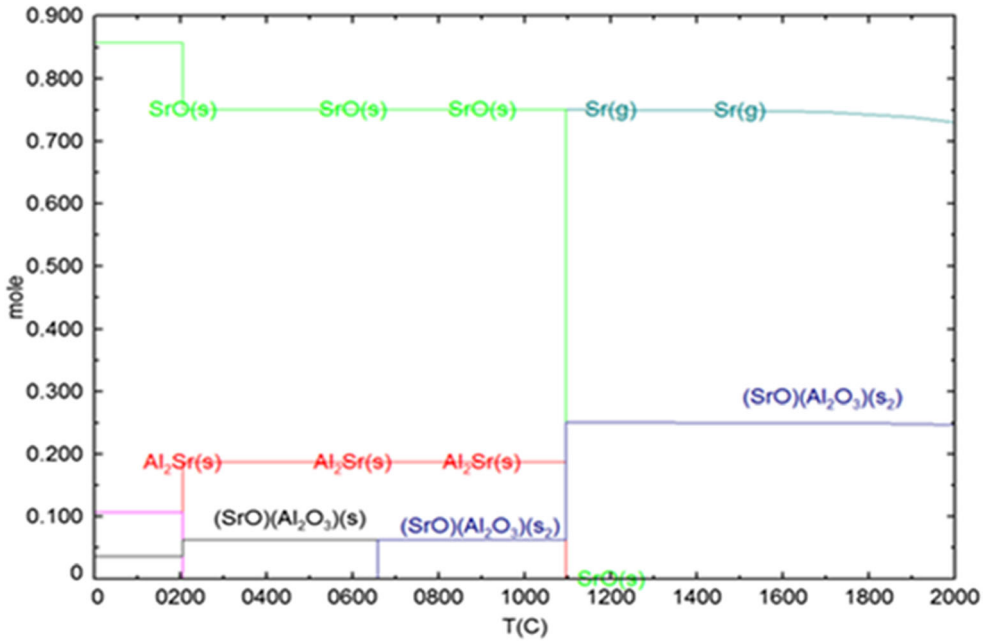

Fig. 3 Probable phases in aluminothermic Sr reduction under 1 mbar atmospheric pressure

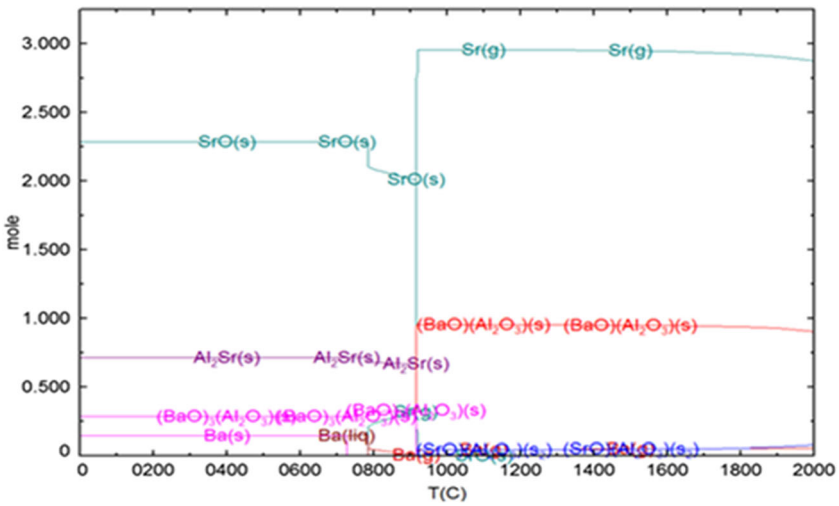

Fig. 4 Effect of BaO on aluminothermic Sr reduction under 1 mbar atmospheric pressure

$$(\mathrm{T}: 1050°C{-}1536°C)\ 4\mathrm{SrO} + 2\mathrm{Si} \rightarrow \mathrm{Sr}_{(g)} + \rightarrow (\mathrm{SrO})_2(\mathrm{SiO}_2)_{(s)} + \mathrm{Si}_{(g)} \quad (2)$$

$$(\mathrm{T}: 1536°C)\ 2\mathrm{SrO} + 2\mathrm{Si} \rightarrow 2\mathrm{Sr}_{(g)} + \mathrm{SiO}_{2(g)}\ \mathrm{Si}_{(1)} \quad (3)$$

Figure 3 presents the simulation of the strontium reduction when aluminum is used as the reductant under 1 mbar atmosphere. During the aluminothermic reduction, Sr is reduced in the form of $Sr_{(g)}$ at 1100 °C. According to Fig. 3, there is a $(SrO)(Al_2O_3)$ phase with $Sr_{(g)}$ phase. The reaction occurs according to Eq. (4).

Based on the simulation results, the silicothermic process would require higher temperatures, and was thus not preferred. Aluminum was used as the main reductant in all experiments. In the Sr reduction experiments with Al, the $(SrO)(Al_2O_3)$ phase was observed. In order to eliminate this phase, the effect of BaO addition was examined in the simulation. The purpose of adding BaO to the mixture is to promote the reaction of Al_2O_3 with BaO to obtain Sr without bonding with Al_2O_3. Figure 4 shows the possible products of Eq. (5). According to Fig. 4, the reduction starts at 950 °C, and BaO reacts with Al_2O_3 above this temperature, resulting in a significant increase in the amount of Sr.

$$7\mathrm{SrO} + 4\mathrm{Al} + \mathrm{BaO} \rightarrow 6\mathrm{Sr}_{(g)} + (\mathrm{BaO})(\mathrm{Al}_2\mathrm{O}_3)_{(s)} + (\mathrm{SrO})(\mathrm{Al}_2\mathrm{O}_3) \quad (5)$$

After examining the effects of BaO addition, the effects of adding CaO were investigated for the same purpose. CaO was added to the mixture in order to obtain Sr gas at lower temperatures and to improve Sr efficiency. Figure 5 shows that Sr passes into the gas phase at 900 °C, and this value is lower than that of the previous experiments under 1 mbar. The reaction occurs according to Eq. (6).

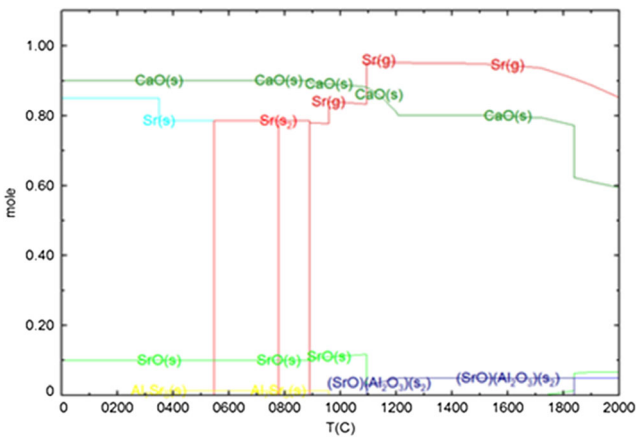

Fig. 5 Effect of CaO on aluminothermic Sr reduction under 1 mbar atmospheric pressure

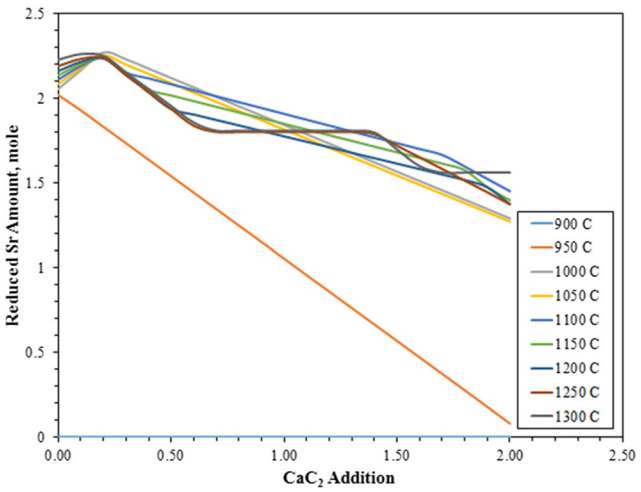

Fig. 6 Effects of CaC_2 on aluminothermic Sr reduction

Table 2 Properties of reactants, reductants and functional additives

Formula	$T_{melting}$(°C)	$T_{boiling}$ (°C)	Density (g/cm^3)	Weight (g)	Form
SrO	2430	3000	4.70	103.62	−100 mesh
BaO	1923	–	5.70	153.33	–
CaO	2572	–	3.37	56.08	–

$$4SrO + 2Al + CaO \rightarrow 3\,Sr_{(g)} + CaO + SrO \cdot Al_2O_3 \quad (6)$$

In the last stage of the thermodynamic simulations, the effects of CaC_2 addition were examined. Figure 6 shows that the reduction of Sr starts at 950 °C, and the amount of Sr formed increases with increasing temperature. The addition of CaC_2 has a positive effect on Sr reduction up to a concentration of 0.5 mol, but over this concentration, CaC_2 reduces the amount of Sr gas formation. The reaction occurs according to Eq. (7). The presence of $CaO_{(g)}$ and $CO_{(g)}$ resulting from the reaction according to Eq. 7 decreases the vacuum value and negatively affects the efficiency.

$$7SrO + 2\,AL + CaC_2 \rightarrow 6Sr_{(g)} + SrAl_2O_4 + 2CO + CaO_{(g)} \quad (7)$$

3 Experimental Studies

In the experiments, Alfa Aesar technical-grade SrO was used as the strontium source. The properties of SrO are given in Table 2. Al powder with 96% purity and grain size of less than 150 μm was used as the reducing agent. BaO and CaO were added to the mixture to react with the undesired aluminum-containing phases. The properties of BaO and CaO are also given in Table 2. ETI Elektrometalurji grade CaC_2 (min. 97.0% CaC_2 by mass) was also used as a reductant for Al reduction.

The experiments were carried out in a cylindrical retort made of Incoloy 800H/HT alloy. The temperature was measured by a 6RhPt-30RhPt (EL-18) thermocouple. To obtain the vacuum atmosphere in the retort, an ILMVAC PK8D two-stage integrated rotary vane pump was used, which can hold a final pressure of 2×10^{-4} mbar. An ILMVAC PIA 100 piezo-electric sensor was used to measure the vacuum pressure. The retort was heated externally using a furnace with a maximum temperature of 1350 °C. The details of the experimental setup can be found in a previous work [16].

Based on the data obtained from the thermodynamic simulations, silicon was not used as the reductant. Instead, aluminum was used as the main reductant. Possible reaction phases are shown in Fig. 3. As can be seen, strontium aluminate and Sr gas phases were observed at 1150 °C. Bonding of strontium oxide with alumina negatively affected Sr formation and decreased efficiency. Accordingly, separation of the SrO from alumina is seen as an important parameter. Therefore, the effects of BaO addition on the elimination of strontium aluminate phase were examined next.

$$(T > 996°C)\;\; 4SrO + 2Al \rightarrow 3Sr_{(g)} + (SrO)(Al_2O_3) \quad (4)$$

In the first experimental series, all reduction experiments were conducted at 1250 °C and under vacuum atmosphere. BaO and Al stoichiometric ratios of 100%, 200% and 300% were selected. SrO, BaO and Al metal powders were mixed according to the stoichiometric ratios in a Turbula mixer for 30 min. BaO was added to the

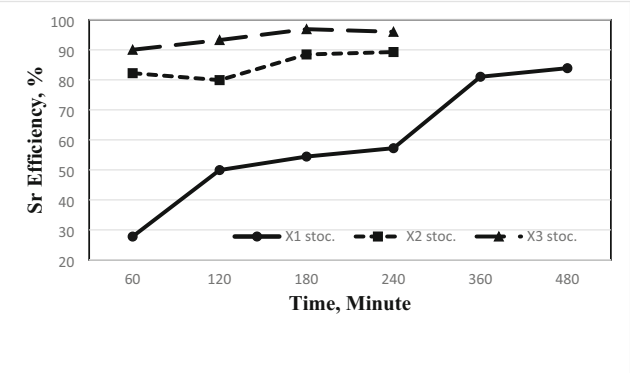

Fig. 7 Effect of BaO addition on Sr reduction efficiency

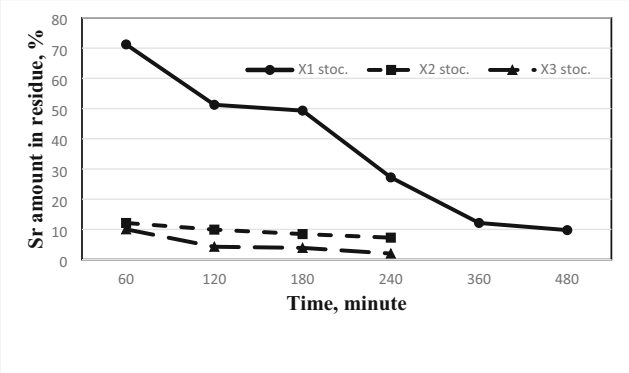

Fig. 8 Sr change in residue with BaO addition

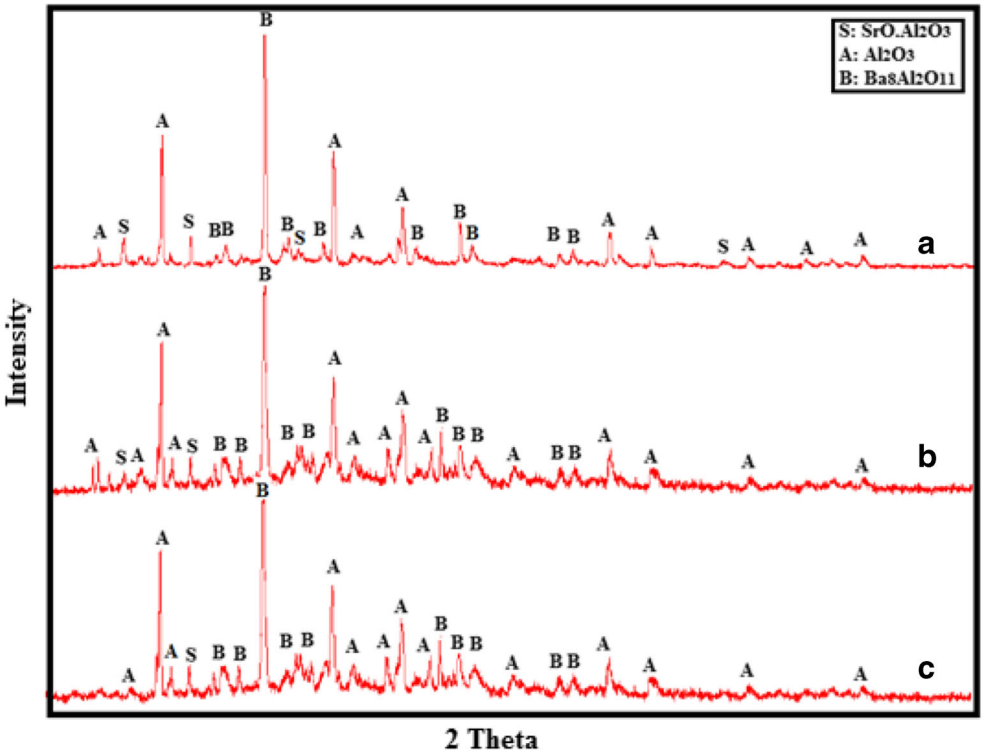

Fig. 9 XRD analysis of the residues in the experiments with addition of BaO (100% stoich., 1 l retort, 1250 °C, 1 mbar) and duration of (**a**) 240 min, (**b**) 360 min, (**c**) 480 min

mixture to react with undesired aluminum-containing phases. The mixture was weighed and briquetted. Briquettes of 2 g were prepared and placed in the Al_2O_3 boats, and the boats were inserted into the retort at room temperature. The retort was then closed and the inside pressure was decreased to 1–5 mbar. In order to obtain strontium as a final product, a cooling zone was mounted on the retort cover. The retort was inserted into the furnace, which was then heated to the required temperature, and the effect of time was examined. The reduction efficiency of the mixtures prepared in 100%, 200% and 300% stoichiometric mixtures were examined by performing experiments for 60, 120, 180 and 240 min. After these experiments, 360- and 480-min experiments with a 100% stoichiometric mixture were performed to determine whether the efficiency of the 100% stoichiometric mixture could be improved by increasing the process duration.

In the experiments with CaO addition, the experiment time was kept constant (120 min). Stoichiometric mixtures of 100%, 200% and 300% were prepared, and the effect of the change in the temperature was examined. These experiments were conducted at 1100 °C, 1150 °C, 1200 °C and 1250 °C. In the experiments where Sr was reduced with aluminum and BaO, the Sr efficiency of the 100% mixtures remained low, and similar results were achieved in the experiments with CaO addition. The effect of time was examined for the 100% stoichiometric series. In this series, experiments were performed for 120, 240, 360 and 480 min at 1250 °C and 1 mbar vacuum pressure.

In the experiments with CaC_2 addition, the change in temperature and the effects of stoichiometry were investigated. In

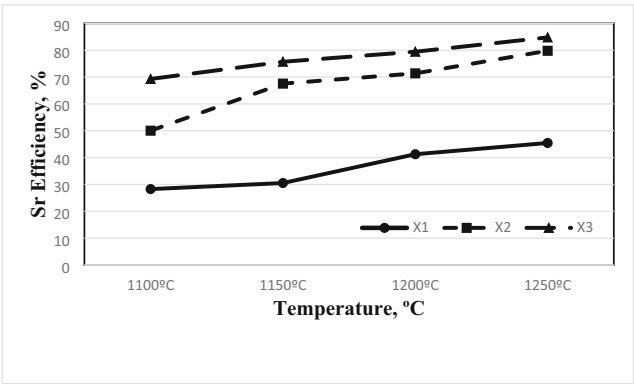

Fig. 10 Effect of CaO addition on Sr efficiency at different temperatures (120 min, 1 l retort, 1 mbar)

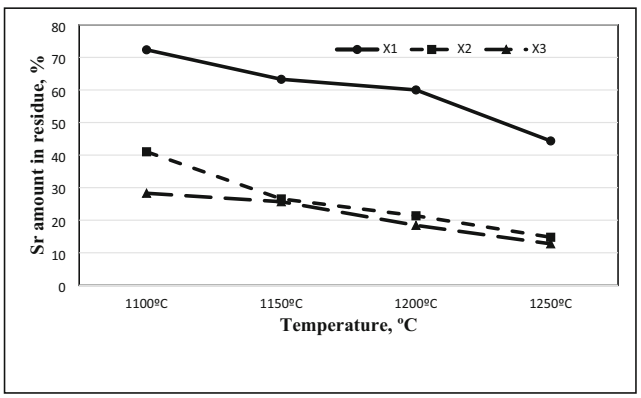

Fig. 11 Effect of CaO addition on Sr quantity in the residue

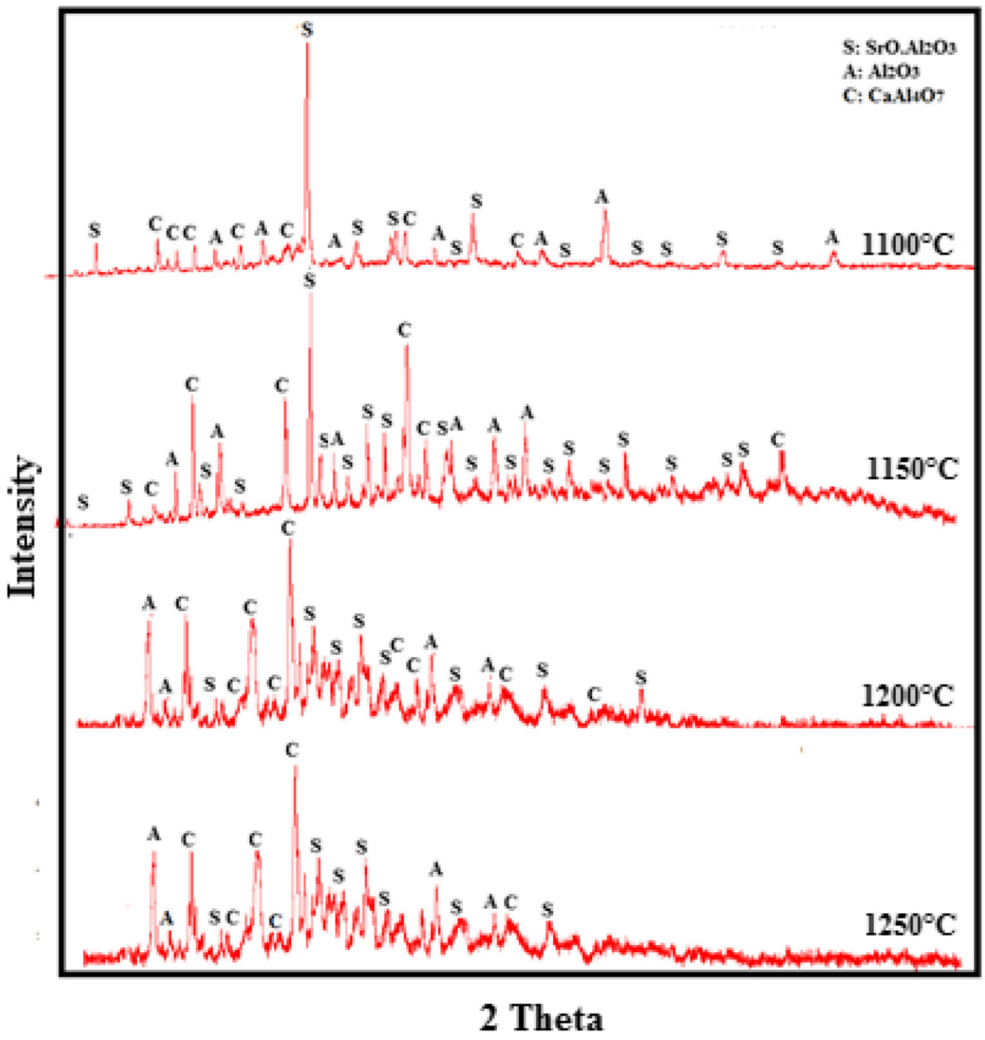

Fig. 12 XRD analysis of the residues in the experiments with addition of CaO (100% stoich., 1 l retort, 1250 °C, 1 mbar)

the first set of experiments with CaC_2 addition, 0%, 25%, 50%, 75%, 100% and 200% stoichiometric mixtures were prepared and were each examined under a vacuum atmosphere at 1100 °C, 1150 °C, 1200 °C and 1250 °C. According to the results of the experiments, the best Sr efficiency was observed with a 50% stoichiometric mixture at 1250 °C. The experiment with the 50% stoichiometric mixture was repeated at 1250 °C, vacuum pressure of 1 mbar, and

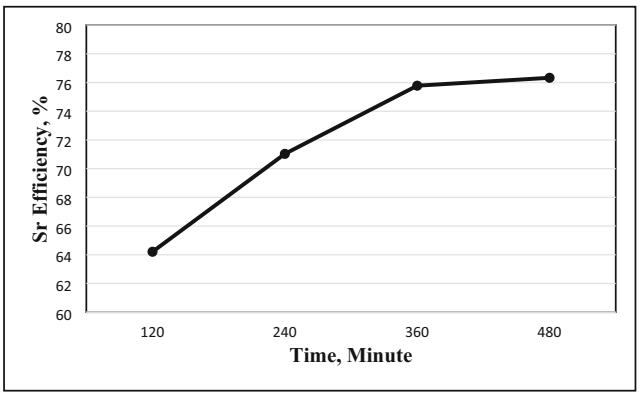

Fig. 13 Effect of CaO addition on the Sr efficiency at ranging time (1250 °C, 1 l retort, 1 mbar, 120, 240, 360, 480 min)

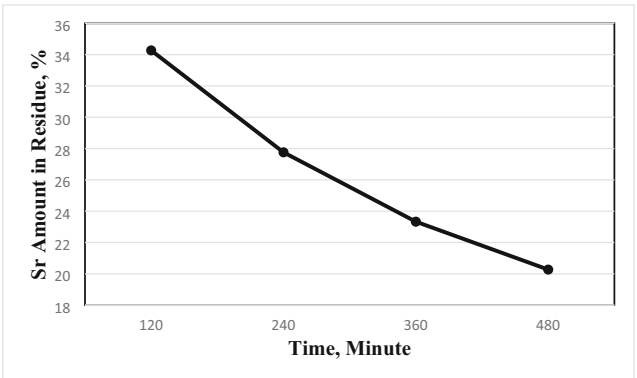

Fig. 14 Effect of CaO addition on the amount of Sr in residue with changing time (1250 °C, 1 l retort, 1 mbar, 120, 240, 360, 480 min)

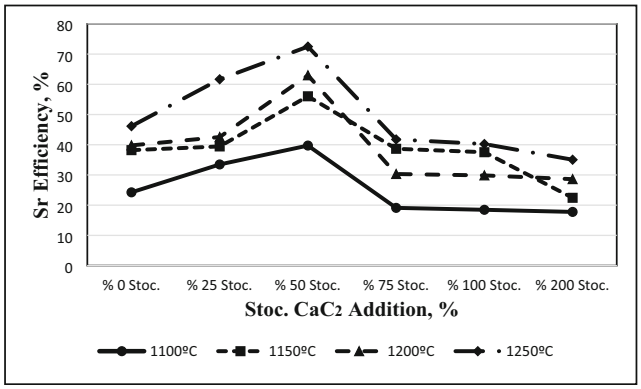

Fig. 15 Effect of CaC_2 addition on Sr efficiency with varying time and Al addition

experiment times of 240, 360 and 480 min in order to determine whether the Sr efficiency could be improved by increasing the experiment duration.

At the end of the reduction experiments, the retort was left in the furnace under vacuum atmosphere and cooled to room temperature. The cover was then opened, and the residue left in the boat was weighed and analyzed chemically. The degree of Sr metal efficiency was calculated as

$$\text{Sr Yield}, \% = 100\text{–}[(W_t \times \%Sr_t)/(W_0 \times \%Sr_0)] \times 100 \quad (8)$$

where W_t is the weight of the residue at time t, % Sr_t is the weight percent of the strontium in the residue at time t, W_0 is the weight of the briquette, and % Sr_0 is the weight percentage of the strontium of the briquette. The Sr recoveries cannot be calculated from crown Sr, because there is not enough Sr in the condensation zone of the retort for analysis.

4 Results and Discussion

The effects of BaO addition were examined during the first stage of the Sr reduction experiments. In the BaO addition experiments, the effect of time and change in the aluminum

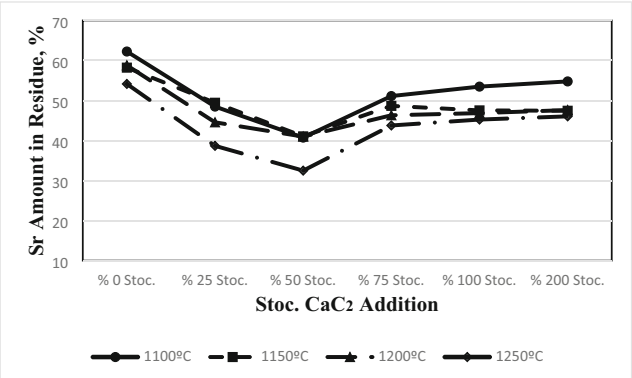

Fig. 16 Amount of Sr in residue with CaC_2 addition with varying temperature

stoichiometric ratio were examined. With a 100% stoichiometric mixture, strontium efficiency of 56.1% was obtained, while 96.89% efficiency was obtained in a 300% stoichiometric mixture after 4 h. The results of the experiments carried out at 1250 °C for 60, 120, 180, 240, 360 and 480 min are shown in Figs. 7 and 8. It is observed that the efficiency of strontium metal increases with increasing experiment time. The Sr concentration in the residue decreased with increasing time. According to Fig. 8, the strontium concentration in the residue of the 100% stoichiometric mixture was determined to be 72.4% after 60 min and 83.89% after 480 min. Figure 9 presents X-ray diffraction (XRD) graphs for the 100% stoichiometric mixtures in experiments with durations of 240, 360 and 480 min. It can be seen from the graph that the $SrO{\cdot}Al_2O_3$ phase is largely eliminated in the experiment carried out for 480 min with 100% stoichiometric BaO and Al. The XRD graph indicates that the amount of aluminum bound to strontium decreased with an increase in the $Ba_8Al_2O_{11}$ phase formation, and only one remaining $SrO{\cdot}Al_2O_3$ peak was observed. It was found that when the experiment time was increased, the reaction proceeded in a more efficient manner.

In the experiments where CaO was used as an additive, the effect of temperature was examined with a constant experiment duration (120 min) for 100%, 200% and 300% stoichiometric ratios. In the experiments conducted with a 100% stoichiometric Al·CaO mixture, Sr efficiency of 27.07% was obtained at 1100 °C, and the efficiency increased to 54.2% when the temperature was increased to 1250 °C. The highest Sr efficiency of 83.43% was observed with a 300% stoichiometric mixture at 1250 °C and 1 mbar vacuum atmosphere. Figure 10 presents the effect of CaO addition on Sr efficiency at various temperatures, while Fig. 11 presents the change in the amount of strontium in the residue. The XRD plots of the residue obtained after the experiments are given in Fig. 12. In Fig. 12, the effect of temperature is presented for a 100% stoichiometric mixture with CaO addition. High $SrO{\cdot}Al_2O_3$ peaks are found in the residue at 1100 °C, and these peaks decrease in height as the temperature increases. The results of these experiments are presented in Fig. 13, and the amounts of Sr in the residue are presented in Fig. 14. In this experimental series, Sr efficiency increased from 64.2% to 76.6%, and the amount of Sr in the residue decreased from 34.33% to 21.56%, when the experiment duration was increased from 120 min to 480 min.

As the next step of the experiments, the effect of temperature on strontium efficiency was examined for mixtures with different CaC_2-Al stoichiometries. According to Fig. 15, maximum Sr efficiency of 74% was obtained for the 50% stoichiometric CaC_2 experiment at 1250 °C. The CaC_2 addition improved Sr efficiency up to a 50% stoichiometric value, but beyond this value, the addition of CaC_2 reduced the Sr efficiency dramatically. The experiments showed that CaC_2 does not have any effect on $SrAl_2O_4$ over 1 mol concentration.

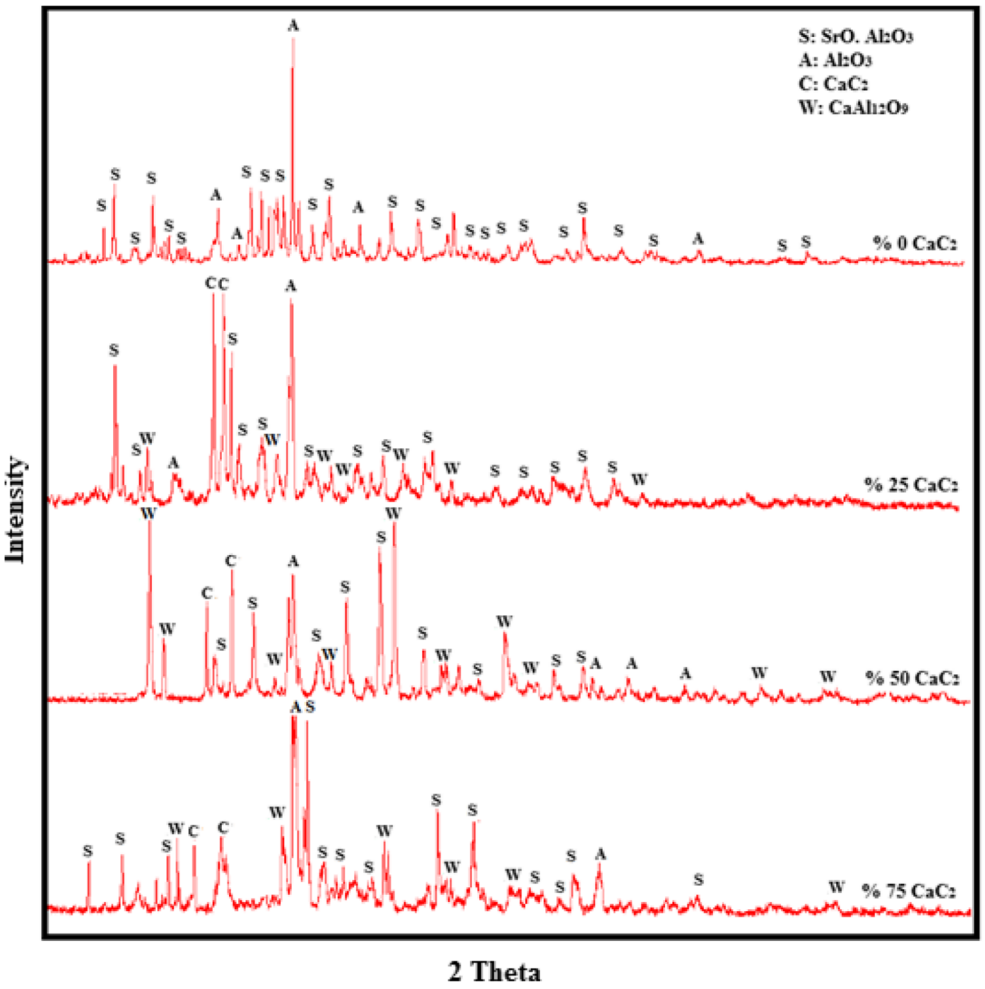

Fig. 17 XRD analysis of the residues in the experiments with CaC_2 addition (0, 25, 50, 75% stoich., 1 l retort, 1250 °C, 1 mbar)

Figure 16 presents the Sr amount in the residue. The XRD analysis of the residues indicates that the amount of $SrO{\cdot}Al_2O_3$ in the residue decreases up to a 50% stoichiometric CaC_2 mixture, but after this point, the amount of $SrO{\cdot}Al_2O_3$ in the residue increases dramatically (Fig. 17).

5 Conclusion

Strontium oxide was subjected to reduction processes with Al, BaO, CaO and CaC_2 under an average pressure of 2 mbar at temperatures of 1100 °C, 1150 °C, 1200 °C and 1250 °C for durations of 60, 120, 180 and 240 min (in 1 l retort). It was found that the addition of BaO is essential for high efficiency in strontium reduction. The temperature must be above 1150 °C, and 1250 °C is sufficient to achieve high efficiency. The maximum efficiency of 96.89% was obtained with the addition of 300% stoichiometric Al and BaO and a duration of 240 min. It was observed that Sr efficiency increased with increasing experiment time and an increasing stoichiometric ratio of Al and BaO. The best results are compared in Table 3.

Table 3 Comparison of functional additives with respect to Sr yield (1250 °C, 2 mbar, 240 min)

Type of functional additive	BaO	CaO	CaC_2
Sr yield, %	96.89	83.43	74.00
Stoichiometry, %	300	300	50

Compliance with Ethical Standards

Conflict of Interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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