

EXPERIMENTAL STUDY OF THE ALUMINOTHERMIC REDUCTION OF CHROMIUM FROM SLAG INTO MOLTEN STEEL

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<https://doi.org/10.37904/metal.2026.5320>

Abstract

The work deals with the issue of chromium recovery from slag systems through its reduction with aluminum. Attention is paid in particular to the behavior of chromium oxides in the melt and their transformation into the metallic phase under appropriately selected conditions. Based on theoretical knowledge and available studies, an experimental procedure was designed to assess the efficiency of the aluminothermic reaction at various process parameters. The aluminothermic reaction was supported by silicon as another additional reducing agent. Experimental work was carried out on a pilot scale with an emphasis on temperature control and optimization of conditions for the course of reduction reactions. Aluminum and silicon were applied as active reducing elements and their effect on the chemical composition of the system and the resulting distribution of chromium was systematically monitored. The obtained findings indicate that the use of aluminum represents a promising approach for the effective use of chromium bound in slag. The work brings new knowledge about the course of aluminothermic reactions in slag melts and suggests the possibilities of their practical application in metallurgy. The work contributes to the circular economy by using waste aluminum to return chromium to the production cycle.

Keywords: Chromium, slag, aluminium, aluminothermic reduction, metallurgy, recycling.

1. INTRODUCTION

Currently, the metallurgical industry is intensively focused on reducing production costs, particularly through the optimization of the energy intensity of processes [1]. However, during the production of molten steel from alloyed chromium waste (hereinafter referred to as Cr waste) in an electric arc furnace (EAF), undesirable oxidation of chromium occurs, resulting in its transfer to the slag in the form of Cr_2O_3 , which leads to losses of this valuable alloying element [2][3][4]. The extent of these losses is significantly influenced by the nature of the input Cr waste. The subsequent reduction of chromium from the slag back into the metal bath is, under EAF conditions, technologically difficult and, at the same time, time- and energy-intensive [5][6]. The issue of chromium oxide reduction has long been the subject of research, with significant attention being paid in particular to aluminothermic processes. For example, plasma-assisted aluminothermy enables the efficient reduction of chromium and iron oxides to the metallic phase without the formation of toxic by-products, thereby representing a promising technology for the environmentally sound recycling of metallurgical waste [7]. However, the high technological and energy demands of these approaches remain a drawback. Further studies focus on the aluminothermic processing of steel slag with the aim of obtaining valuable metal products (e.g. ferrosilicon) and, at the same time, a usable oxide residue, with an emphasis on the use of secondary raw materials, particularly aluminium waste, in the context of the circular economy [8].

Previous research carried out at MATERIÁLOVÝ A METALURGICKÝ VÝZKUM s.r.o. (hereinafter MMV) focused on the reduction of chromium from high-chromium slags, using carbon and silicon as reducing agents,

in an induction melting furnace. The influence of process parameters, including the use of an oxygen-fuel burner, on the efficiency of chromium re-reduction into the metallic phase was experimentally evaluated [9]. The results showed that, with a suitable combination of reducing agents and operating conditions, chromium recovery can be significantly increased and losses to slag reduced. The use of carbon and silicon was primarily motivated by their economic availability; however, the potential for utilising aluminium waste within the circular economy is now also coming to the fore. In metallurgical practice, elements with a higher affinity for oxygen than chromium are used for the reduction of chromium. In particular, carbon, silicon and aluminium. The efficiency of these processes is significantly influenced by the specific surface area of the reducing agent, which accelerates the reaction kinetics. For example, BGH's technology utilises granulated aluminium of 1–2 mm in size with defined dosing whilst simultaneously heating the bath using a combination of electrical energy and an oxygen-fuel burner [1]. When using oxygen as a heat source, it is important to have a higher silicon content in the melt prior to heating, due to its preferential oxidation over chromium [10][11]. From a thermodynamic perspective, the course of chromium reduction from slag is determined primarily by temperature, the chemical composition of the slag and the composition of the metal bath [12]. Nevertheless, there is a significant difference between theoretical equilibrium models and the actual behaviour of the system under industrial conditions, which has led to the formulation of empirical relationships based on the statistical evaluation of operational data. At the same time, it is known that the kinetics of chromium reduction can be significantly influenced by the choice of reducing agent – whilst reduction using silicon and carbon proceeds gradually, the aluminothermic reaction is strongly exothermic and proceeds very intensively once a sufficient temperature is reached. Experimental studies confirm that during aluminothermic reduction, both the degree of chromium reduction and the reaction rate increase significantly with rising temperature, and at temperatures around 1523 K, high efficiency can be achieved in a short time [12]. This mechanism is associated with the formation of metallic chromium and slag rich in Al_2O_3 and represents an effective route for returning chromium to the metallic phase. At a fundamental level, the aluminothermic reduction of Cr_2O_3 is highly exothermic (around -526 kJ/mol) and proceeds rapidly after initiation, with a required molar ratio of $\text{Cr}_2\text{O}_3\text{:Al}$ close to 1:9 for complete reduction. The reaction products include Al_2O_3 and metallic chromium, although in aluminium excess, intermetallic phases such as AlCr_2 may also form. Kinetic analysis indicates that the reaction can follow a first-order chemical control under certain conditions, with activation energies in the range of $\sim 140\text{--}190 \text{ kJ/mol}$ [13]. Temperature and slag basicity were also found to influence the reduction efficiency, with higher values improving reduction extent and the separation between slag and metal. Work [14] observed a similar behaviour for Electric Arc Furnace (EAF) slags where the reduction of Cr_2O_3 using aluminium proceeded rapidly at high temperature (1793K), with a large proportion of chromium being reduced within a few minutes. However, the process was not only based on chemical reaction but also controlled by mass transfer in the slag phase. This indicates that slag properties such as viscosity and fluidity may affect the transport phenomena and the overall reduction efficiency [15].

The present work builds on these findings, with the aim of experimentally elucidating the reduction of chromium oxides from slag into molten steel using granulated aluminium and silicon. The experiments were carried out on a pilot scale with a melt weight of approximately 1000 kg. The main contribution of this work lies in the isolated study of aluminothermic reduction in the slag–steel system and a comprehensive assessment of its influence not only on chromium recovery but also on the behaviour of other elements in the system.

2. EXPERIMENTS

Based on a literature review and previous experimental work carried out at MMV, a series of pilot-scale smelting runs was designed to reduce chromium from slag into molten steel using aluminium as a reducing agent. The experiments were carried out in an atmospheric induction melting furnace with a nominal capacity of 1750 kg. For each melt, 1000 kg of X4CrNiMo16-5-1 grade molten steel was prepared. The chemical composition of this grade was modified for the purposes of the experiment with regard to the potential transferability of the proposed technology to large-scale metallurgical production conditions (see **Table 1**).

Table 1 Chemical composition of grade X4CrNiMo16-5-1: standard and for experiments

		C (wt%)	Mn (wt%)	Si (wt%)	P (wt%)	S (wt%)	Ni (wt%)	Cr (wt%)	Mo (wt%)	V (wt%)	N (ppm)
Standard	min	-	-	-	-	-	4.00	15.00	0.80	-	200
	max	0.06	1.50	0.70	0.040	0.030	6.00	17.00	1.50	-	-
Production at a large steelworks	min	-	0.50	0.30	-	-	4.50	15.00	0.90	-	200
	max	0.03	1.00	0.50	0.025	0.020	5.50	16.50	1.20	0.10	-
Experiments	min	0.15	0.25	0.25	-	-	4.50	15.00	0.90	-	-
	max	0.20	0.35	0.35	0.025	0.020	5.50	16.50	1.20	0.10	-

After the charge had melted and the required temperature had been reached, 45 kg of chromium slag was added to the surface of the melt, corresponding to 4.5 % of the melt's weight. The chemical composition of the slag used is given in **Table 2**. In order to improve its fusibility and achieve homogeneous coverage of the surface, the slag was mechanically processed in advance from its original particle size of approx. 250–300 mm to a fraction of 20–50 mm.

Table 2 Chemical composition of the slag

	C (wt%)	S (wt%)	Fe (wt%)	FeO (wt%)	Fe₂O₃ (wt%)	MnO (wt%)	Cr₂O₃ (wt%)	V₂O₅ (wt%)	TiO₂ (wt%)
Composition	0.12	0.034	12.95	0.01	0.47	2.97	15.87	0.21	3.12

continued from **Table 2**

CaO (wt%)	SiO₂ (wt%)	P₂O₅ (wt%)	Al₂O₃ (wt%)	MgO (wt%)	CaF₂ (wt%)	Na₂O (wt%)	Zn (wt%)	K₂O (wt%)	BaO (wt%)	Nb₂O₅ (wt%)
24.63	21.27	0.01	8.94	8.72	0.01	0.01	0.01	0.04	0.01	0.47

Based on stoichiometric calculations, it was determined that approximately 3.9 kg of Si or 3.8 kg of Al is required for the complete reduction of chromium from the supplied slag. In the experiment, pure aluminium in the form of granules with a particle size of 1–4 mm was used as the reducing agent.

The reducing agent was dosed onto the surface of the slag whilst an oxygen-fuel burner was in operation. It was then incorporated into the slag mass by intensive mixing. Approximately 5 minutes after the application of the reducing charge, its melting was visually observed, and the first samples of metal and slag were taken for chemical analysis. Sampling and measurement of the melt temperature took place at five-minute intervals for a period of 30 minutes following the application of the first reducing charge.

A total of five experimental melts were carried out. In each melt, a multiple of the theoretically calculated dose of reducing agent was applied sequentially. In the first melt, the amount of reducing agent corresponded to the stoichiometric requirement (3.9 kg Si, or 3.8 kg Al); in the second melt, double the amount was used (7.8 kg Si, or 7.6 kg Al); and in subsequent melts, this ratio was further increased. Metal samples were taken at regular five-minute intervals up to the 30th minute in all experiments.

After the completion of each experimental melt, the melt was poured from the induction furnace into a ladle and subsequently cast from the bottom into a V2A-type mould assembly on an atmospheric casting line under a protective argon atmosphere. The ingot cast in this manner served as the charge for further experiments.

3. RESULTS AND DISCUSSION

Changes in the chromium content in the melt and chromium trioxide in the slag were plotted in **Figure 1** (Cr), **Figure 2** (Cr_2O_3) and summarised in **Figure 3** and **Figure 4**. A quantitative expression of the changes in chromium oxide content in the slag is given in **Table 3**. The results clearly show that the highest reduction efficiency was achieved with a triple dose of the reducing agent, where the efficiency of chromium transfer from the slag to the metal bath reaches up to 86 %.

Table 3 Chromium content in the slag during the series of experiments

Slag samples taken	Time of sampling (min)	Cr ₂ O ₃ content in the slag in individual experiments				
		1.	2.	3.	4.	5.
		(wt%)	(wt%)	(wt%)	(wt%)	(wt%)
1	0	15.9	14.4	9.3	9.8	8.4
2	5	12.9	3.7	1.2	2.7	4.7
3	10	6.4	3.7	1.1	2.5	4.3
4	15	6.6	3.3	1	2.6	4.1
5	20	8.3	3.1	1.2	2.4	4.5
6	25	7.7	3.3	1.3	2.3	4.4
7	30	8.4	2.9	1.3	1.9	4.8
	Units	Experiments				
		1.	2.	3.	4.	5.
Slag weight	[kg]	49.00	47.00	52.00	48.00	49.00
Change in Cr content in slag	[kg]	-2.51	-3.70	-2.85	-2.59	-1.21
Change in Cr content in slag	[%]	-47.17	-79.86	-86.02	-80.61	-42.86

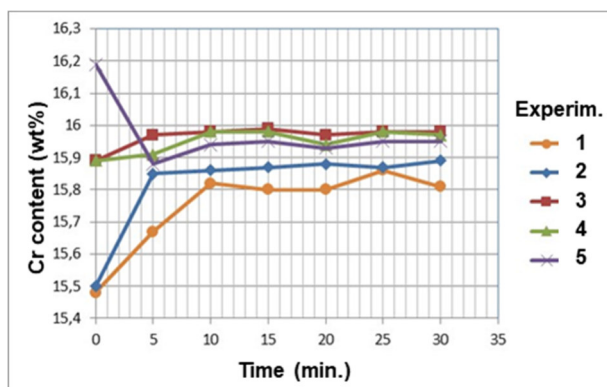


Figure 1 Cr content in melt during individual experiments

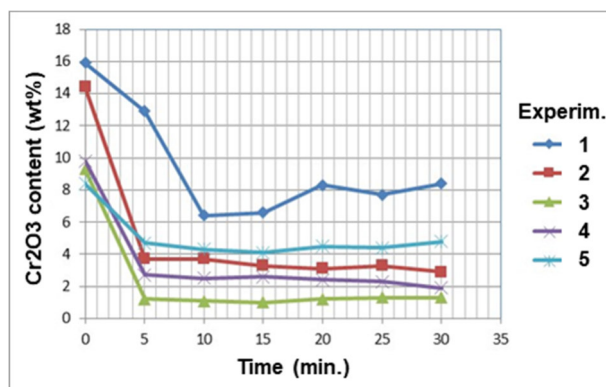


Figure 2 Cr_2O_3 content in slag during individual experiments

It is evident from the curves shown in the graphs that chromium reduction takes place immediately after the application of reducing agents, with the entire process being significantly faster and more intense compared to reduction using carbon and silicon. At the same time, it is evident that even with double and quadruple doses of the reducing agent, the reduction efficiency reaches approximately 80 %, which exceeds the maximum efficiency achieved using C and Si (approx. 62 %).

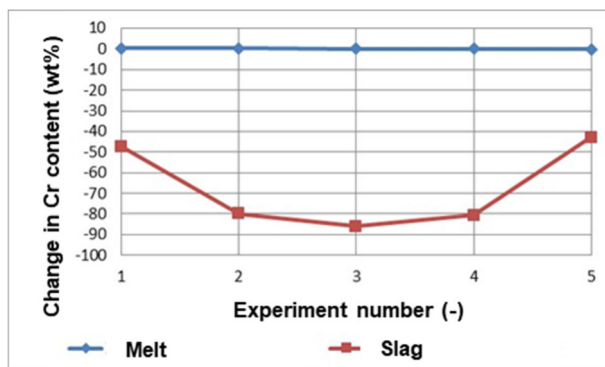


Figure 3 Cr content during individual experiments

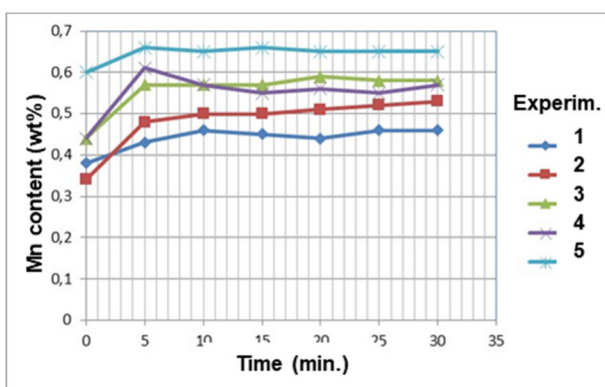


Figure 4 Mn content in the melt during individual experiments

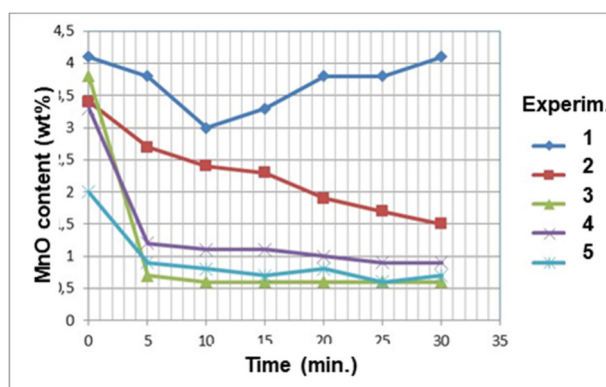


Figure 5 MnO content in the slag during individual experiments

Figure 4 and **Figure 5** show a significant reduction in manganese in the slag immediately after the addition of reducing agents, with the MnO content in the slag falling from the original 3.8 % to approximately 0.7 %. When silicon was used as a reducing agent, its content in the melt increased simultaneously. At the highest dose, the Si content reached a value of up to 3.76 %.

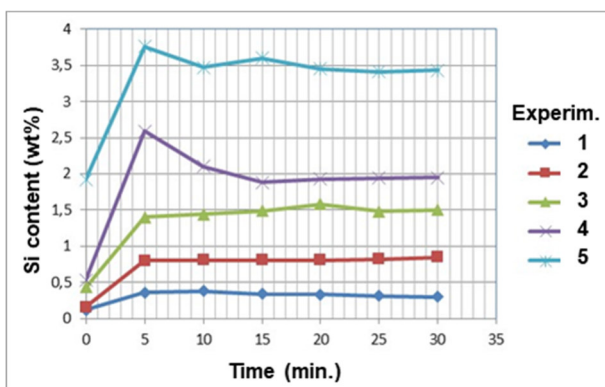


Figure 6 Si content in the melt during the individual experiments

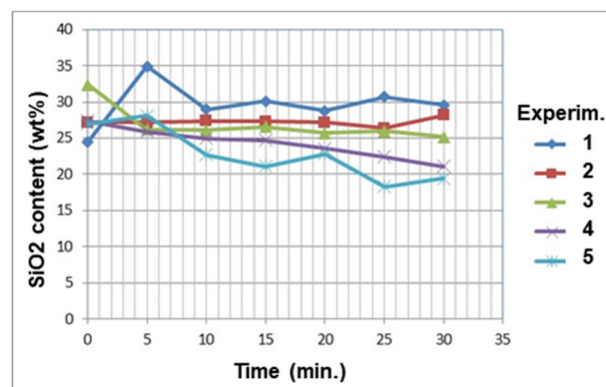


Figure 7 SiO₂ content in the slag during individual experiments

Figure 6 and **Figure 7** show a concurrent increase in the Si content in the metal phase and a decrease in the SiO₂ content in the slag, indicating that part of the silicon was consumed not only for the reduction of oxides but also for the enrichment of the melt. The most significant increase in Si content was recorded in the experiment with the highest dose of reducing agent. When aluminium was used as a reducing agent, its content

in the melt also increased, from 0.042 % at a single dose to 0.96 % at a fivefold dose (see **Figure 8**). In parallel, an increase in the Al_2O_3 content in the slag was also recorded, reaching up to 46.4 % at a fourfold dose (see **Figure 9**). With increasing amounts of reducing additives, a decrease in the nitrogen content in the melt was also observed, falling below the 50 ppm threshold (see **Figure 10**).

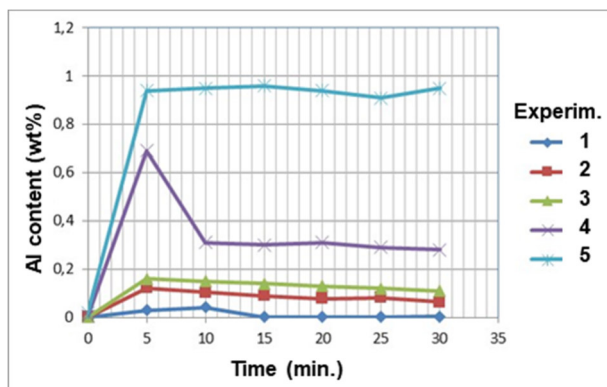


Figure 8 Al content in the melt during individual experiments

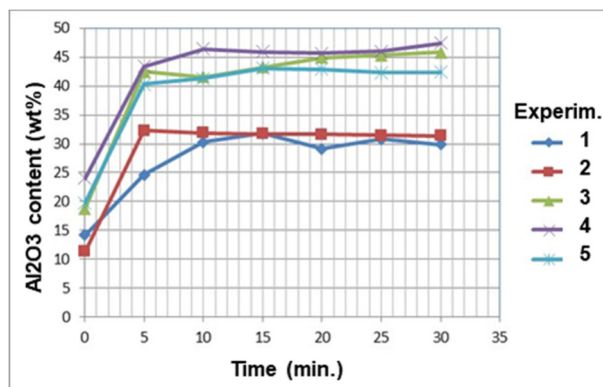


Figure 9 Al_2O_3 content in the slag during individual experiments

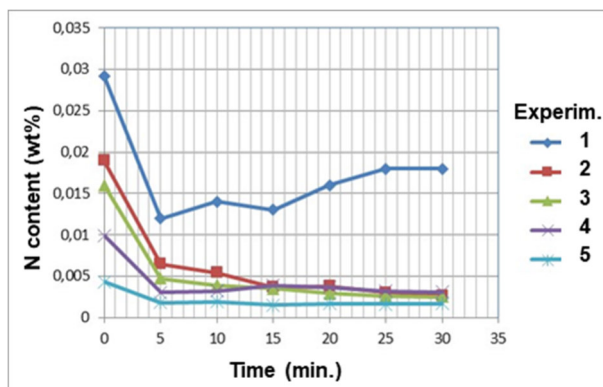


Figure 10 N content in the melt during the individual experiments

4. CONCLUSION

A technology for the reduction of chromium from slag into molten steel was designed and experimentally verified under the conditions of MATERIÁLOVÝ A METALURGICKÝ VÝZKUM s.r.o. The design was conceived with an emphasis on its applicability in industrial production conditions, particularly in large metallurgical plants. Experimental verification was carried out on X4CrNiMo16-5-1 grade steel with a modified chemical composition to reflect the operating conditions of metallurgical companies. The results obtained show that the use of aluminium and silicon as reducing agents demonstrates significantly higher efficiency compared to the combination of carbon and silicon. Within the scope of this work, a reduction efficiency of up to 86 % for chromium from slag into the metal bath was achieved, which represents a high level of efficiency. The repeated use of a single batch of raw material for all experiments slightly affected the initial conditions of the experiments, particularly the chemical composition of the melt, which also influenced the chemical composition of the slag after melting. The results can be evaluated in terms of trends. At the same time, it was confirmed that to achieve high reduction efficiency, it is necessary to apply an increased amount of reducing additives, specifically approximately three times the stoichiometrically calculated dose required for complete chromium reduction. This finding highlights the significance of kinetic and transport limitations in the actual process, which cannot be fully captured by theoretical calculations alone.

ACKNOWLEDGEMENTS

This paper was produced within the framework of the Institutional support for long-term and conceptual development of a research organisation in 2026, provided by the Ministry of Industry and Trade of the Czech Republic.

This work was supported by the European Just Transition Fund within the Operational Programme Just Transition under the Ministry of the Environment of the Czech Republic, project CirkArena No. CZ.10.03.01/00/22_003/0000045.

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