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Microstructural and Chemical Characterization of Industrial Ferronickel Produced by the RKEF Process Using SEM–EDS Analysis

Zarife Bajraktari Gashi
University of Chemical Technology and Metallurgy
Janna Mateeva
j.mateeva@uctm.edu

University of Chemical Technology and Metallurgy

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Abstract

Ferronickel (Fe–Ni) is a key ferroalloy used in stainless steel production, predominantly produced from lateritic ores via the rotary kiln–electric furnace (RKEF) process. In this study, the microstructural features and localized chemical composition of industrial ferronickel produced in Kosovo were investigated using scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS).The results reveal a heterogeneous metallic matrix with dendritic morphology, typical of rapid solidification under industrial conditions. SEM observations indicate the presence of non-metallic inclusions embedded within the Fe–Ni matrix. EDS analysis confirms that iron is the dominant element, while nickel is relatively homogeneously distributed, suggesting effective alloying during smelting. Localized oxygen- rich regions are associated with oxide inclusions, likely originating from incomplete slag–metal separation and residual oxides from the reduction process. The presence of carbon is attributed to carbothermic reduction in the rotary kiln stage. The results indicate that reduction reactions are advanced but not fully completed, which is consistent with industrial RKEF operation.

By combining microstructural observations with industrial slag composition and process parameters, this study provides insight into the relationship between process conditions, inclusion formation, and alloy heterogeneity. The findings contribute to improved understanding of metallurgical efficiency and ferronickel quality in large-scale industrial production.

1. INTRODUCTION

Ferronickel (Fe–Ni) is one of the most important ferroalloys in modern extractive metallurgy, serving as the primary source of nickel for the production of stainless and special steels. Industrial processing of lateritic nickel ores is predominantly carried out via the rotary kiln–electric furnace (RKEF) route, where the rotary kiln enables partial reduction of oxide phases, followed by high-temperature smelting in the electric furnace to produce molten ferronickel [1, 2]. In industrial practice, lateritic ores frequently contain significant inherent moisture and are not always subjected to a dedicated drying stage prior to rotary kiln processing. As a result, additional thermal energy is required for moisture evaporation, leading to increased consumption of carbon-based fuels and reductants. This increased energy demand affects not only the overall energy balance of the process but also the reduction kinetics and thermal stability during the pre-reduction stage [3]. Recent studies have demonstrated the complexity of carbothermic reduction mechanisms in lateritic nickel ores, including selective reduction of nickel oxides, nucleation of metallic phases, and slag–metal interactions under industrial conditions [4–6]. These processes strongly influence nickel enrichment efficiency and the final quality of ferronickel, which are highly dependent on temperature regime, reductant distribution, and reaction kinetics during both rotary kiln and electric furnace operation. The metallurgical performance of ferronickel is closely related to its chemical composition and microstructural homogeneity. Residual oxide phases and entrapped slag inclusions can adversely affect downstream refining processes and the quality of stainless steel products [7]. Furthermore, microstructural evolution during solidification governs the distribution of nickel within the iron-rich matrix, influencing alloy uniformity and performance [8].

Advanced characterization techniques, particularly scanning electron microscopy (SEM) combined with energy-dispersive X-ray spectroscopy (EDS), are widely used to investigate microstructural features and local elemental distribution in ferroalloys [9, 10]. These techniques allow identification of metallic matrices, oxide inclusions, and phase heterogeneities formed during large-scale smelting processes. Despite extensive global research, detailed microstructural investigations of industrial ferronickel produced under full-scale RKEF conditions in Southeast Europe remain limited. In particular, previous studies of the ferronickel plant in Drenas have mainly focused on process evaluation and material balance rather than detailed microstructural characterization [7]. The objective of this study is to provide a systematic microstructural and semi-quantitative chemical characterization of industrial ferronickel produced under real operating conditions, with emphasis on the relationship between process parameters, slag composition, and alloy quality.

2. MATERIALS AND METHODS

2.1. *Industrial process and sampling*

The investigated material consisted of industrial ferronickel (Fe–Ni) samples collected from a full-scale rotary kiln–electric furnace (RKEF) production line processing lateritic nickel ores in Kosovo [7]. The analyzed material represents the final metallic product obtained after electric furnace smelting and subsequent refining in the converter. Samples were collected in granulated form under standard industrial operating conditions. Microstructural and chemical characterization was performed using scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS), widely applied for microstructural investigation and localized elemental analysis in ferroalloys [9, 10]. Granulated ferronickel samples were examined in the as-received condition without chemical etching in order to preserve the original solidification morphology. Prior to analysis, selected particles were mounted and prepared for observation. SEM examinations were carried out under high-vacuum conditions using an accelerating voltage of 15–20 kV. Both secondary electron (SE) and backscattered electron (BSE) imaging modes were employed to distinguish between metallic and oxide phases.

2.2. *Slag sampling and chemical analysis*

To evaluate slag composition and metal losses during ferronickel production, industrial slag samples were also analyzed. A total of 31 slag samples obtained from electric furnace operation were considered. The amount of slag produced during individual operational periods ranged between approximately 788 t and 1502 t [7].The chemical composition of slag samples was determined using X-ray fluorescence (XRF) analysis in an industrial laboratory. The analyzed oxides included SiO_2, MgO, FeO, Al_2O_3, CaO and Cr_2O_3, together with nickel and total iron content. The slag composition is typical of lateritic nickel ore smelting systems and is characterized by a silicate-based matrix. Based on the collected operational data, the average slag composition is presented in Table 1.

Table 1
Average chemical composition of industrial slag (wt.%)

Component	Content (wt.%)
SiO_2	58.9
FeO	15.2
MgO	17.4
Al_2O_3	2.4
CaO	2.38
Cr_2O_3	1.49
Ni	0.083
Fe (total)	11.76

2.3. *Microstructural characterization*

Microstructural characterization of ferronickel samples was performed using SEM in order to evaluate phase morphology, dendritic structures and the presence of non-metallic inclusions.

The observations focused on identifying the metallic Fe–Ni matrix, solidification features, and possible oxide inclusions formed during smelting and cooling. The use of EDS imaging enabled compositional contrast between metallic and oxide phases, facilitating phase identification.

Microstructural characterization of ferronickel samples was performed using SEM in order to evaluate phase morphology, dendritic structures and the presence of non-metallic inclusions. A representative SEM micrograph of the investigated ferronickel is shown in Fig. 1.

Figure 2. SEM micrograph of ferronickel (Fe–Ni) with selected point EDS analysis indicating local elemental composition within the metallic matrix and inclusion regions.

2.4. *Elemental analysis (EDS)*

The local chemical composition of ferronickel samples was determined using EDS analysis attached to the scanning electron microscope. Measurements were performed at multiple selected micro-areas (≥ 10 points) within the metallic matrix to improve statistical representativeness.The analyzed elements included Fe, Ni, O and C. Representative EDS results indicate a metallic region composed mainly of iron (approximately 68.6 wt.%), with nickel present at about 5.5 wt.%. Oxygen (≈ 21 wt.%) and carbon (≈ 4–5 wt.%) were also detected in localized regions, indicating the presence of oxide inclusions and residual carbon (Table 2.).

Table 2
Semi-quantitative EDS analysis of a representative micro-area in industrial ferronickel (Fe–Ni)

Map Sum Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	4.71	0.41	12.92
O	K series	21.16	0.30	43.54
Fe	K series	68.62	0.49	40.45
Ni	K series	5.51	0.35	3.09

The EDS results presented in Table 1 correspond to a localized micro-area within the ferronickel matrix and should therefore be considered semi-quantitative. The analysis indicates that iron is the dominant element, while nickel is present as the principal alloying component.

The relatively high oxygen content suggests the presence of oxide inclusions or residual slag phases entrapped within the metallic matrix. Similarly, the detected carbon is attributed to carbothermic reduction processes and possible surface contamination during sample preparation. It should be noted that EDS analysis is sensitive to the analysed micro-volume and may not represent the bulk chemical composition of the ferroalloys (Fe-Ni).

Table 3
Industrial production parameters of ferronickel obtained from electric furnace and converter operation

Parameter	**Period 1**	**Period 2**	**Period 3**
Nickel production (t)	21.59	624.72	5043.90
Nickel production (t/day)	21.59	20.15	13.78
Ni content in ferronickel (%)	17.69	18.07	18.51
Nickel recovery (%)	100.09	98.17	86.49
Energy consumption (MWh)	1204.63	35997.46	318188.84
Specific energy consumption (MWh/t Ni)	55.81	57.62	63.08

*Values above 100% may reflect uncertainties in industrial mass balance calculations and sampling variability

The industrial data presented in Table 2 indicate a progressive increase in specific energy consumption from 55.81 to 63.08 MWh/t Ni across the analyzed periods. This trend is accompanied by a decrease in

nickel recovery from 98.17% to 86.49%, suggesting reduced process efficiency under later operating conditions.

The increase in energy consumption may be associated with variations in ore quality, particularly moisture content and nickel grade, as well as changes in slag composition and furnace operating conditions. Higher FeO content in slag can indicate incomplete reduction, which is consistent with the oxygen-rich regions observed in the SEM–EDS analysis.

The nickel content in ferronickel remains relatively stable (17.7–18.5 wt.%), indicating that alloy grade control is maintained despite fluctuations in process efficiency. However, the observed decline in recovery and increase in energy demand suggest that optimization of reduction conditions and slag chemistry could significantly improve overall metallurgical performance.

The SEM micrograph (Fig. 1) reveals a dendritic metallic structure typical of rapidly solidified ferronickel produced by the RKEF process. The heterogeneous morphology reflects non-equilibrium solidification conditions during tapping and granulation.

EDS point analysis (Fig. 2) confirms that the metallic matrix is primarily composed of iron with nickel distributed as the principal alloying element. However, the presence of oxygen detected in the analyzed micro-areas suggests the existence of oxide inclusions.

Elemental mapping (Fig. 3) provides further insight into phase distribution. Iron and nickel are relatively uniformly distributed within the metallic matrix, indicating effective alloy formation. In contrast, oxygen is localized in discrete regions, confirming the presence of oxide inclusions. Carbon appears in localized zones, which may be associated with residual carbon from carbothermic reduction or surface contamination.

The presence of oxygen-rich inclusions is consistent with the industrial slag composition and the observed decrease in nickel recovery (Table 3), suggesting incomplete slag–metal separation and partial reduction of iron oxides during smelting.

3. DISCUSSION

The SEM micrograph (Fig. 1) reveals a dendritic metallic structure typical of rapidly solidified ferronickel produced by the RKEF process. The heterogeneous morphology reflects non-equilibrium solidification conditions during tapping and granulation.

EDS point analysis (Fig. 2) confirms that the metallic matrix is primarily composed of iron with nickel distributed as the principal alloying element. However, the presence of oxygen detected in the analyzed micro-areas suggests the existence of oxide inclusions.

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The presence of oxygen-rich inclusions is consistent with the industrial slag composition and the observed decrease in nickel recovery (Table 2), suggesting incomplete slag–metal separation and partial reduction of iron oxides during smelting.

4. CONCLUSIONS

Based on the obtained results, the following conclusions can be drawn:

Industrial ferronickel produced via the RKEF process exhibits a heterogeneous dendritic microstructure typical of rapid solidification. SEM–EDS analysis confirms that iron is the dominant element, with nickel relatively uniformly distributed within the metallic matrix. Oxygen-rich inclusions indicate incomplete slag–metal separation and partial reduction, consistent with industrial slag composition.The increase in specific energy consumption and decrease in nickel recovery suggest reduced process efficiency under certain operating conditions. The study demonstrates that microstructural characteristics are directly influenced by process parameters, highlighting the importance of optimizing reduction conditions and slag chemistry.

Declarations

Funding

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Author Contribution

Z.B.-G. conceptualized the study and supervised the research. J.M. (corresponding author) designed the methodology, conducted the experimental work, performed the SEM–EDS analysis, and prepared the figures. Z.B.-G. analyzed and interpreted the data. M.K.A. wrote the main manuscript text. All authors reviewed, revised, and approved the final version of the manuscript.

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Figures

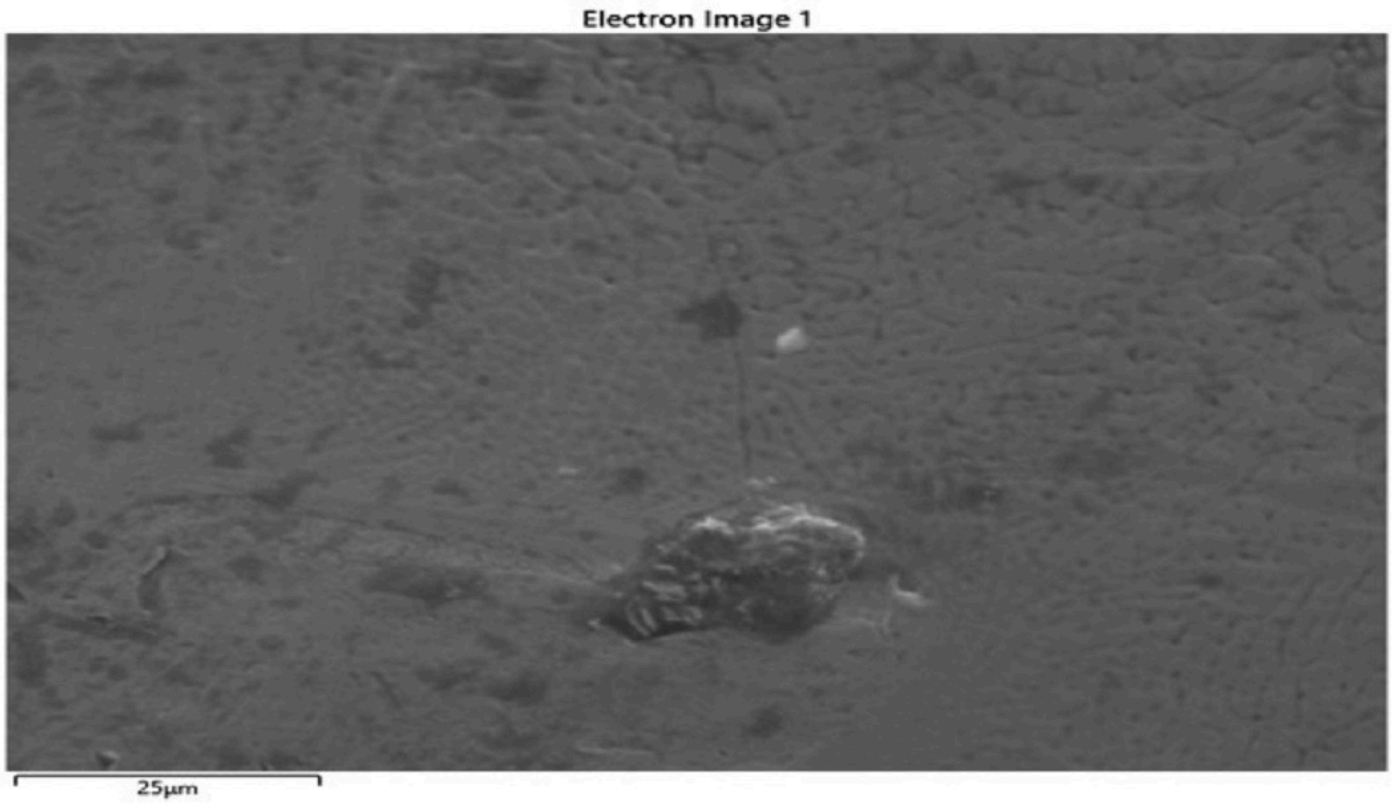


Figure 1

SEM micrograph of industrial ferronickel (Fe–Ni) showing dendritic metallic microstructure and heterogeneous phase distribution.



Figure 2

SEM micrograph with selected analysis point indicating the location of EDS measurement within the ferronickel matrix.

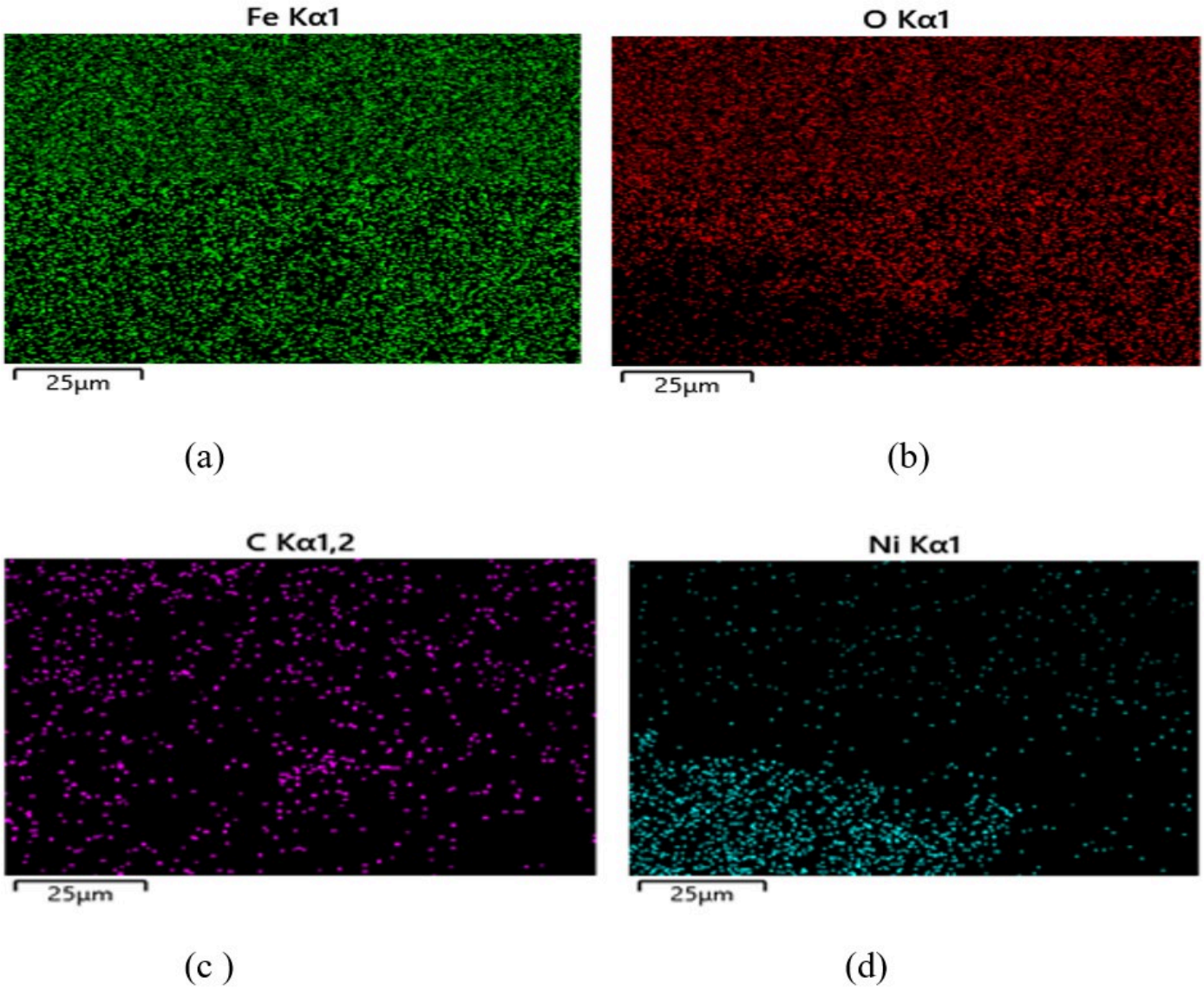


Figure 3

EDS elemental mapping of ferronickel (Fe–Ni): (a) Fe distribution, (b) O distribution, (c) C distribution, and (d) Ni distribution.