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DETERMINATION OF ADHESION STAGES OF THE Fe-Ni ORE AT THE FERRONIKELI PLANT IN DRENAS

Purpose. The process of calcine production in rotary kilns at the Ferronikeli plant in Drenas is realized with great difficulties as a result of the formation of large adhesions in the areas of rotary kilns. So far, the removal of load adhesions during the work process inside the rotary kilns is achieved only by their physical removal. The purpose of the paper is to determine the factors in reducing adhesions in the process of calcine production in the areas of rotary kilns, in the Ferronikeli plant in Drenas.

Methodology. Chemical analysis of iron-nickel ore. The determination of the composition of the adhesives was performed with the X-RAY device, in the laboratory of the Ferronikeli plant. Method with the software “Calphad” 2020, at the University of Ljubljana.

Findings. From the composition of iron-nickel ore, the composition of adhesives in rotary kilns, we manage to determine the thermodynamic phases and exothermic and endothermic processes that take place in the process of rotary kilns. From the determinations of the composition of the adhesives in the software “Calphad” 2020, we manage to determine one of the factors that affect the formation of adhesives based on the stages during the process, which do not have a stability, as a result of high moisture content of iron-nickel ores.

Originality. Based on the study at the Ferronikeli plant, shovels were placed on the walls of the rotary kilns in the three areas, the removal of fireclay bricks, the placement of anchored concrete and the project for the placement of two kilns for kiln ore were started.

Practical value. The placement of the shovels has increased the temperature inside the rotary kilns. While the placement of dryers will have an impact on reducing the adhesions inside the rotary kilns and from the economic point of view, we will have a reduction of fuels in the load of the rotary kilns.

Keywords: *Fe-Ni ore, adhesion, Calphad, stages, sub-stages*

Introduction. The new foundry of the new Ferronikeli in Drenas is located near the source “Old Çikatove” 37 km northwest of Prishtina. The production of metallurgy until now in the Republic of Kosovo is oriented in processing ore of local mines such as Golesh and Çikatove, and imported ore (from the year 2007) from: Indonesia, Philippines, Guatemala, Albania, Macedonia, Turkey [1, 2]. The processing is done using the electro-reduction process with the purpose of preparing Fe-Ni as the final product. The fundamental establishments and the equipment of the foundry are: two rotary kilns of the firm “Schmid” Copenhangen, two rotary kilns of the firm “Elkem”-Spikerverket, Oslo and two converters LD of the firm Krupp, Dusseldorf [3].

NewCo Ferronikeli complex in Drenas has turned into an important component of Balfin Group’s entire production rank, since its acquisition by the latter in July 2018. Balfin Group is among the largest privately-owned companies in South-East Europe with large investments varying anywhere from residential and commercial real estate to retail, smelting and mining. As the new leader of NewCo Ferronikeli, Balfin Group is now working in resuming production at the plant and its plans involve the re-establishment of NewCo Ferronikeli as a leading global producer as well as exporter of ferronickel [4, 5].

In the new foundry in the new Ferronikeli in Drenas, the process of Fe-Ni production passes through these stages: processing of Fe-Ni ores and fuels and their delivery to appropriate bunkers where with the aid of conveyor belts they enter the rotary kiln. At a temperature of over 500 °C by means of special equipment, the fuel enters, consisting of heavy-oil and pet-kok, which influence the temperature increase of the rotary kiln (over 700 °C) and the qualitative production of calcine [6]. The calcine produced by the rotary kilns is sent to the electric furnace through special pipes, where the Fe-Ni metal production process and that of some quantity of slag are realized.

The produced metal from the electric furnace is sent to the converter for refining, in which an amount of limestone is introduced, and oxygen is blown into it [7, 8]. After the refining, the metal is poured into special equipment where we have the production of Fe-Ni metal, in the form of granules of size of 15–20 cm, weight of 5–15 gr while the remaining slag in the converter is divided into the metallic part and the non-metallic part, where the metallic part gets back in the process.

In the new foundry of the new Ferronikeli in Drenas during the realization in 2007–2017, these Fe-Ni ores were used: ores from Kosovo (mines: Gllavice and Çikatove), ores from Albania, ores from Guatemala, ores from Indonesia, ores from the Philippines, ores from Turkey and ores from Macedonia [9]. The composition of the ores is oxide and silicate. The annual average of Ni content in the Guatemala, Philippine, Indonesia ore is higher (Ni = 2 %) compared to the Kosovar ore (Ni = 1.5 %) and Albanian ore (0.99 %) [10].

The ferronickel ores used at the Ferronikeli plant in Drenas are inadequate ores regarding their composition of Ni, therefore for the acquisition of Ni from laterite ores high energy spending is required.

In rotary kilns, fuel is used as a source of energy and in turn it is divided in two groups:

- fuels (lignite, coal, biomass, etc.), which make up the charge alongside ferronickel ores;
- fuels (pet-kok, heavy oil), which are put inside the rotary kilns in temperatures above 500 °C.

Since the years 2019–2021, the following ferronickel ores have been used: ores from Kosovo, ores from Albania and ores from Guatemala.

The process of rotary kilns is carried out with enormous difficulty due to adhesions [11]. A. The adhesions on the walls of rotary kilns are formed as a result of an inability to reach adequate temperatures inside the rotary kilns. The zones of the rotary kilns are unable to carry out the process for which they are made, and as a result the adhesion is transmitted from one zone to the next, causing a low prereduction of Ni and Fe in metal. The production process of calcine as a sole product of rotary kilns does not achieve the necessary temperatures, thereby resulting in high energy expenditure in melting the calcine in electric furnaces [10].

Methodology. From the rotary kilns, we have obtained the adhesion sample and afterwards, we have prepared it at the laboratory of the Ferronikeli plant in Drenas.

The rotary kiln was stopped in February of 2019 for the purpose of overhaul, which made it possible to obtain the sample to realize our research. The sample of adhesions was taken in the frying zone, where by looking at it, one is able to deduct that the adhesion is porous.

So far the removal of adhesions from rotary kilns has been done physically, whereas in this paper we will analyze the adhesions through the aid of the software 'Calphad' and therefore analyze the stable phases and subphases of processes inside the composition of adhesions.

The work progress went as follows. The adhesion sample taken from the frying zone in rotary kilns (Fig. 1, *a*), was sent to the laboratory of the Department of Materials and Metallurgy at the Faculty of Geosciences, where we started with the preparation of the sample. Furthermore, we notice the initial preparation of the sample (Fig. 1, *b*). The sample entered the inductive furnace (Fig. 1, *c*) at 10:00, where in thirty minutes, at 10:30, a temperature of 835 °C was reached which caused no visible changes on the sample.

At 10:37, we obtained a temperature of 1020 °C, where it was visible that the lower sample was nearing softening but it was not soft yet. At 10:47, with a temperature of 1260 °C, the softening of the sample started and it is visible that the sample is about to melt completely. At 10:57, with a temperature of 1503 °C, the complete melting of the sample can be observed (Fig. 2).

The sample was melted in the inductive furnace in the laboratory of UIBM and afterwards it was sent to the laboratory of the NewCo Ferronikeli LLC in order for the chemical composition of the sample to be analyzed.

In the following figures we will demonstrate the processes of the preparation of the sample, the determination of the adhesion composition and the analysis of the phases and subphases with the software 'Calphad'.

Initially, we place the sample of adhesion inside the equipment for fragmentation (Fig. 3, *a*) and afterwards we obtain the fragmented parts (Fig. 3, *b*).

Afterwards, the fragmented sample is placed in an appropriate container (Fig. 4, *a*), and turned into a compact mass (Fig. 4, *b*). The next step was grinding of the compact sample (Fig. 4, *c*) and weighing it (Fig. 4, *d*).

The grinding of the sample (Fig. 4, *c*) is an important process as it permits the unification with the Dilitium Tetraborat, and as such the sample and the reagent are placed inside the laboratory furnace (Figs. 5, *a, b*). After a few minutes, the temperature is gradually increased until 1350 °C. After melting of the sample (Fig. 5, *c*) it is shaped into a circular disc (Fig. 5, *d*).


a

b *c*

Fig. 1. The adhesion sample:

a – the general view; b – the cutting process; c – the view in the inductive furnace

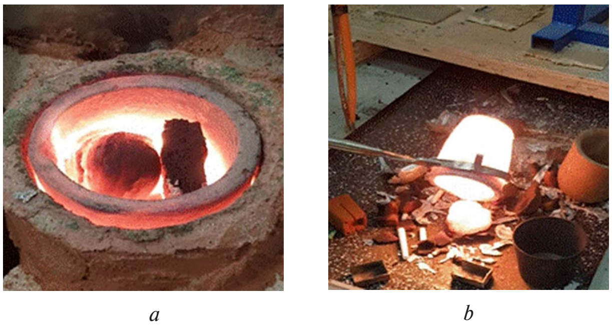

a *b*

Fig. 2. The melting of the adhesion sample (a) and its pouring (b)

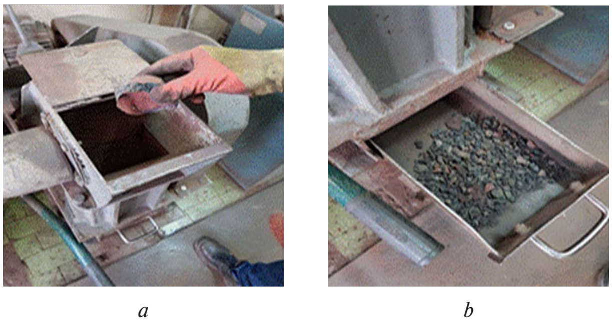
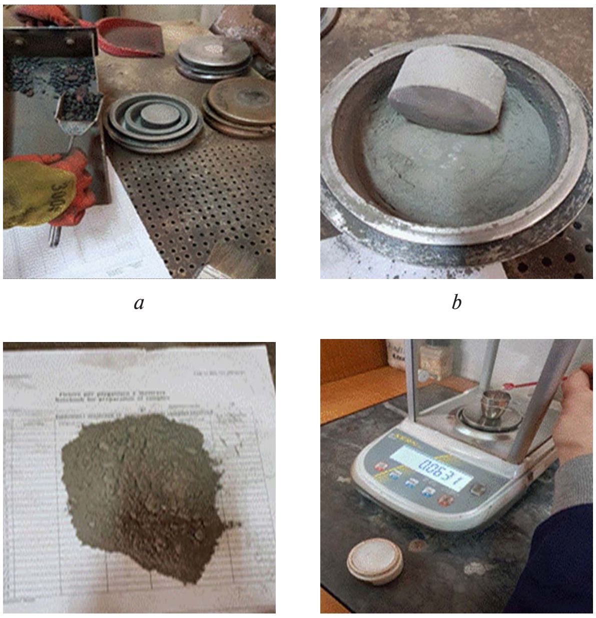

a *b*

Fig. 3. Placing the adhesion sample for fragmentation (a) and girding of the sample (b)

The determination of the adhesion composition was realized using the X-RAY equipment (Fig. 6). As a result, we have obtained the following representation of adhesion (Table 1).

The realization of thermodynamic calculations and the determination of stages and sub-stages of rotary kiln adhesions were realized on the software 'Calphad'. The following are the characteristic of the 'Calphad' software:

- chemical composition in %;
- temperature $T = 0$–1500 °C;
- pressure $P = 100\,000.0$ Pascal;
- substance amount = 1 mol.

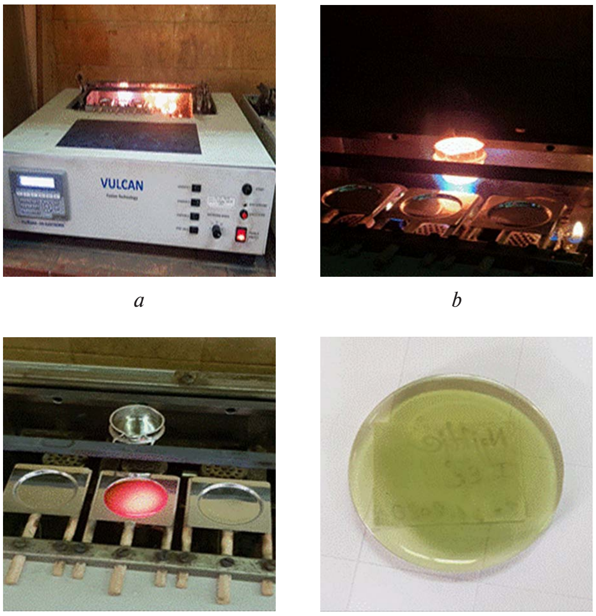


a *b*

c *d*

Fig. 4. An experimental preparation procedure:

a – preparation of the sample; b – preparation of the sample of compact mass; c –grinding of the compact sample; d – weighing of the sample



a *b*

c *d*

Fig. 5. Laboratory testing:

a – laboratory furnace Vulcan Fusion Technology; b – prepared sample together with the reagent; c – view of the molten sample; d – adhesion sample

Using the software 'Calphad' we have determined the following:

- entalphy;
- phase compositions;
- compositions of components and microcomponents;
- mass losses;
- number of moles for elements;
- helmholtz energy;
- construction of phases and elements;
- volume and structure.

Results and discussion. The differences in stages occur from the solid phase to the liquid one, the liquid phase to the gas stage and vice versa. During these transformations, many large differences occur on the enthalpy value (as well as the internal energy) for a substance. Moreover, these differences are called latent changes of heat.

Fig. 6. Spectrometer (X-RAY Equipment)

Table 1

Representation of adhesion composition from rotary kilns (%)

Fe	MgO	Ni	SiO_2	Al_2O_3	Co	CaO	Cr_2O_3	Cfix	S
25.15	9.70	1.39	46.42	2.16	0.09	2.59	1.54	0.10	0.26

From the graphical representation in Fig. 7, we notice that as a result of the stage transformations of the load in rotary kilns, we have an unstable representation of stages.

As the charge entering the rotary kilns at the plant of Ferronikeli does not undergo the process of drying (the removal of moisture of the ferronickel ore and wet lignite) the processes is realized with difficulty [12].

With the start of the process in rotary kilns, there is a release of volatile matter of fuel which creates large amounts of gasses in the process (300 000 m^3 N/h) [1]. From the graphical representation (Fig. 8), the relation between the temperature and the percentage of Ni shows that above 2 % of Ni achieves a temperature higher than 500 °C, marking the end of the phase and the start of others with a composition of constitu-

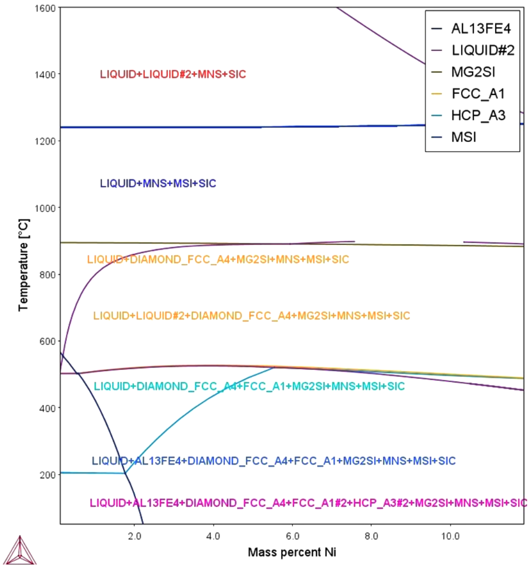


Fig. 7. The calculation of Fe-Ni adhesion stages in rotary kilns on the software 'Calphad' (variation of Ni amount in relation to the temperature)

ents of adhesions of rotary kilns (C, Al, Si, Co, Fe, Ni, etc.).

The results obtained show that the stages (Fig. 7) LIQUID + LIQUID#2 + MNS + SIC are stable with components of adhesive compositions. Furthermore, we notice that MNS ≠ 1 has the percentage of Ni = 1.13 % of a temperature of 1379.52 °C, where at the temperature of 900 °C, the following components were identified: Ca, Cr, Fe, Mg, S, as well as two phases. Its first phase contains Ca, Mg, Fe, Cr, whereas the second phase contains sulfur.

In addition, the representation of the phase SiC ≠ 1 is shown as manifested in two sub-phases with a composition of Si and C.

From the diagram (Fig. 7) we notice the representation of the phase LIQUID + MNS + MSI + SIC with a composition of Ni = 1.227 % of temperature 1082.7 °C as well as a composition of constituents. LIQUID#1 presents a stable phase in the diagram in Fig. 7, with a composition of Al, C, Ca, Co, Fe, FeS, Mg, Ni, NiS, S, and Si.

The phase SiC ≠ 1 with components where two other phases (one with a composition of Si and the other with a composition of C) are shown within the phase SiC ≠ 1. The phase LIQUID + DIAMOND_FCC_A4 + MG2SI + MNS + MSI + + SIC with a composition of Ni = 0.94 % at a temperature of 844.8 °C and other represented components is manifested with a diagram of a stable phase LIQUID ≠ 1 as well as manifested with a phase characterized by a composition of the following components: Si, Al, Ca, Mg, Ni, Fe, Co, Cr, C, NiS, FeS, S. The other phase, DIAMOND_FCC_A4#1, is unstable and consists of components such as: Si, Al, C.

The diagram shows phase MG2SI#1, which is manifested with two sub-phases, the first one composed of Mg and the other of Si. Based on the given equilibrium conditions, the phase LIQUID#1 alongside its components is stable. Moreover, we have sub-phases with composition of elements such as: Si, Al, Ca, Ni, Mg, Fe, Ca, Cr, C, NiS, FeS, S.

From the diagram shown in Fig. 8, it is visible that we have ten stable stages in the calculations of the adhesion of the rotary kilns at the Ferronikeli plant in Drenas. As well as more than thirty sub-stages, which result as a consequence of not reaching the adequate temperatures inside the zones of the rotary kilns (considering that Fe-Ni ore does not go through the drying process before entering the rotary kilns, the average composition reaches 27 % of humidity), where during the stage transformations none of the zones of the rotary kilns is able to finish the process and, as a result, the transformations of the Fe-Ni mineral are carried from one zone to another, where in the rotary kilns at the Ferronikeli plant in Drenas we have only the pre-reduction of Fe-Ni.

Another conclusion derived from the diagram shown in Fig. 8 is that the process realized inside the rotary kilns is an exothermic process, because the energy source is the fuel that makes up the load of the rotary kilns together with Fe-Ni ore. In addition, the calculations help in realizing it is an exothermic process as a result of the differences in fuel – the requests of electric furnaces.

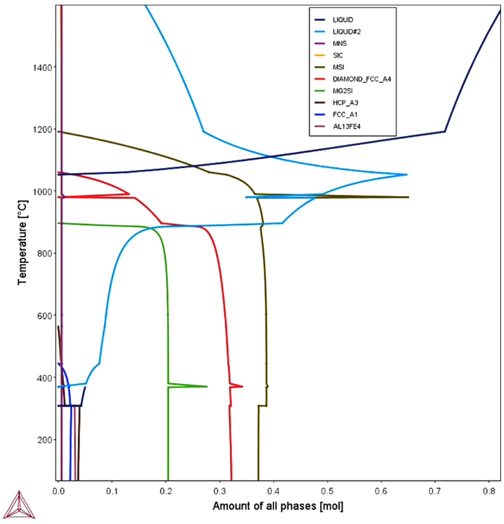


Fig. 8. Calculation of all adhesion stages of Fe-Ni in rotary kilns, using the software Calphad

In addition, from the diagram in Fig. 8, we have obtained ten phases from the calculated parameters on the software 'Calphad'.

Table 2 represents the interpretation of the diagram in Fig. 8 of the stable phase of a temperature of 398.18 °C, characterized by the following data: Gibbs Energy –31049.26044JJ, enthalpy –18672.70979 J. The graphical representations (Table 3) show a clear interpretation of unstable phases, as a result of the high moisture content of the Fe-Ni ore. Out of the three zones of the rotary kiln at the Ferronikeli plant, none of them are able to reach their designated function, due to the adequate temperature of 900–1000 °C not being reached.

Table 3 depicts the liquid phase with a composition of constituents of rotary kiln adhesions (C, Al, Mg, S, etc.). The structure of Ni (Fig. 8), is FFC, the values of the axes being c = 4 · 06 A, a = 2 · 474 A ratio 1 · 64, which is near enough to the ratio 1 · 633 for closest packing [13].

We obtained a negative result of the enthalpy (Fig. 8) which demonstrates that the process in this phase is exothermic, where the energy is released from the reaction inside rotary kilns. Gibbs free energy is a derived quantity that blends together the two great driving forces in chemical and physical processes, namely enthalpy change and entropy change [13]. The obtained negative value of Gibbs Energy (Fig. 8) shows that the entropy and enthalpy changes occur spontaneously.

Regarding the effect of the study on the process of the rotary kilns at the Ferronikeli Plant, we can elaborate that the research affected the change in the inner part of the rotary kilns where in June of 2019, the refractory material, which until June of 2019 was chamotte, was changed to anchored thermoconcrete (Fig. 9). Moreover, shovels were placed in the three zones

Table 2

Determination of thermodynamic parameters with 1.39 % Ni composition at temperature 125 °C

Quantities				
Mass percent Ni	1.38571	[Mass percent]		
Temperature	125.02265	[°C]		
System				
Moles	1.00000			
Mass	33.33636	[g]		
Temperature	398.17265	[K]		
Total Gibbs Energy	–31049.2604	[J]		
Enthalpy	–18672.7097	[J]		
Volume	9.20809E–6	[m^3]		
Component	*Mole Fraction*	*Mass Fraction*	*Activity*	*Potential*
C	0.00278	0.00100	6.79013E–10	–69888.2482
S	0.00312	0.00300	1.98668E–54	–4.09367E5
Si	0.59347	0.50000	0.09465	–7805.1059
Mg	0.13716	0.10000	1.51516E–6	–44362.1493
Ca	0.02495	0.03000	2.25396E–12	–88785.0488
Al	0.03707	0.03000	0.00038	–26105.4733
Co	0.00057	0.00100	2.39062E–9	–65721.2818
Cr	0.01923	0.03000	3.27500E–10	–72302.1822
Ni	0.00787	0.01386	1.17969E–14	–1.06174E5
Fe	0.17379	0.29114	7.47636E–11	–77192.4777

Table 3

Stable Phases and Sublattices

LIQUID#1 (Volume fraction – 0.11259, Moles – 06241; Mass – 1.99782)		
Composition		
Component	Mole Fraction	Mass Fraction
Ca	0.34983	0.43802
Si	0.37056	0.32513
Al	0.27845	0.23471
Co	0.00116	0.00214
Ni	2.53598E–7	4.64964E–7
Fe	3.11022E–8	5.42626E–8
Mg	1.81095E–8	1.37503E–8
Cr	5.31017E–12	8.62558E–12
S	3.00000E–12	3.00522E–12
C	1.00000E–12	3.75223E–13
Constitution	*(Al, C, Ca, Co, Cr, Fe, FeS, Mg, Ni, NiS, S, Si)$_1$*	
	Constituent	Site Fraction
Sublattice 1:		
	Si	0.37056
	Ca	0.34983
	Al	0.27845
	Co	0.00116
	Ni	2.53597E–7
	Fe	3.11012E–8
	Mg	1.81095E–8
	Cr	5.31017E–12
	NiS	1.00000E–12
	FeS	1.00000E–12
	S	1.00000E–12
	C	1.00000E–12
AL13FE4#1 (Volume fraction – 0.00832, Moles – 0.00826; Mass – 0.28005)		
Composition		
Component	Mole Fraction	Mass Fraction
Al	0.76078	0.60575
Fe	0.23922	0.39425
Ni	0.00000	0.00000
Cr	0.00000	0.00000
Co	0.00000	0.00000
Ca	0.00000	0.00000
Mg	0.00000	0.00000
Si	0.00000	0.00000
S	0.00000	0.00000
C	0.00000	0.00000
Constitution	$(\mathrm{Al})_{0.63}$ $(\mathrm{Fe})_{0.23}$ $(\mathrm{Al, VA})_{0.14}$	
	Constituent	Site Fraction
Sublattice 1:		
	Al	1.00000
Sublattice 2:		
	Fe	1.00000
Sublattice 3:		
	Al	0.87157
	VA	0.12843
DIAMOND_FCC_A4#1 (Volume fraction – 0.30645, Moles – 8.60703; Mass – 0.40169)		
Composition		
Component	Mole Fraction	Mass Fraction
Si	1.00000	1.00000
Al	1.00000E–12	9.60692E–13
C	1.00000E–12	4.27651E–13
Fe	0.00000	0.00000
Ni	0.00000	0.00000
Cr	0.00000	0.00000
Co	0.00000	0.00000
Ca	0.00000	0.00000
Mg	0.00000	0.00000

of the rotary kilns where their form changes depending on the zone. In addition, adhesions to the walls of the rotary kilns as well as the spending of fuel have both decreased (Figs. 10, 11).

With the placement of shovels inside rotary kilns (as a result of the research in caplhad software) the average pre-reduction rate increased from 46.97 % which was in 2018 to 53.9 % in 2020 [14].

The degree of prereduction is the main factor in the preparation of calcine for the electric furnace. The higher the degree of prereduction is, the lower costs of electricity for the melting of the calcine in electric furnace will be and we will not have difficulties with the acidity of the slag (Fig. 10).

Temperature is an important factor in preparing the calcine as the sole product in rotary kilns. With the placement of the shovels in the areas of the rotary kilns, there was certain improvement in raising the temperature of the calcine but we still need improvement in the preparation of the load for the rotary kilns to reach the temperatures up to 900–1000 °C, signifying the proper temperature for processes in the rotary kilns [14].

Conclusions. From the obtained results using the software 'Calphad' as well as the industrial and laboratory part of the rotary kilns, we can put forth these recommendations:

- a dryer should be inserted at the Ferronikeli plant in Drenas;
- front openings of kilns should be minimized, in order to control the amount of oxygen entering the rotary kilns;
- place shovels on the walls of the rotary kilns in order to continuously mix the charge inside the rotary kilns;
- the return of CO (carbon monoxide) from the electric furnaces into the rotary kilns;
- introduction of the largest amount of dried Kosovo lignite in the load of rotary kilns;
- Fe-Ni granulation should be done after the ore drying process.

Fig. 9. View of shovels inside rotary kilns at Ferronikeli Plant in Drenas (June 2019)

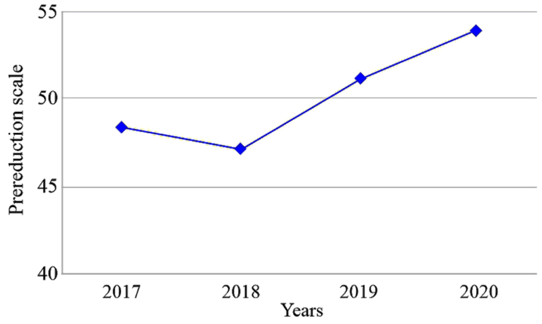


Fig. 10. Prereduction scale (result from the research of adhesion)

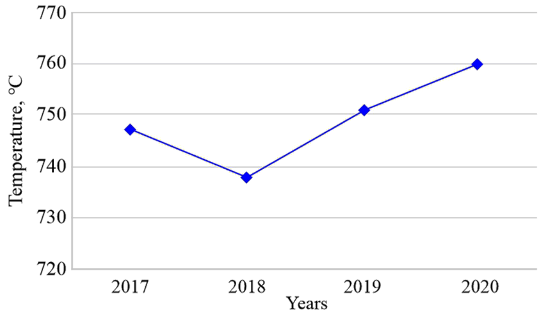


Fig. 11. Representation of the average temperature value of calcine

The previously mentioned conclusions serve as crucial points which we believe will influence the increase in temperatures inside the rotary kilns, an important factor in the metallurgical processes in the rotary kilns.

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Визначення стадій адгезії залізо-нікелевої руди на заводі Ferronikeli в місті Дренас

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Мета. Процес випалу залізо-нікелевої руди в обертових печах на заводі Ferronikeli в м. Дренас проходить із великими труднощами в результаті утворення великих осередків налипання конгломератів кальциту та частинок руди на стінках обертових печей. До теперішнього часу усунення таких налипань у процесі роботи обертових печей досягається тільки шляхом механічного впливу на них. Метою роботи є визначення факторів зниження налипань кальциту у процесі виробництва в обертових печах на заводі Ferronikeli в м Дренас.

Методика. Хімічний аналіз залізо-нікелевої руди. Визначення складу в'яжучих речовин проводилося за допомогою рентгенівського апарату в лабораторії заводу Ferronikeli. Метод із програмним забезпеченням «Calphad» 2020 року, Університет Любляни.

Результати. За складом залізо-нікелевої руди, складом в'яжучих речовин в обертових печах нам вдається визначити термодинамічні фази, а також екзотермічні та ендотермічні процеси, що відбуваються у процесі роботи обертових печей. Із визначень складу клеючих речовин у програмі «Calphad» 2020 нам вдається визначити один із факторів, що впливає на формування клеючих речовин на основі етапів процесу, які не мають стабільності внаслідок високого вмісту вологи в залізо-нікелевих рудах.

Наукова новизна. На підставі дослідження, проведеного на заводі Ferronikeli, на стінах обертових печей у трьох областях були розміщені лопати, було розпочато видалення шамотних вогнетривких цеглин, укладка анкерного бетону та проект розміщення двох печей для обпалювальної руди.

Практична значимість. Розміщення лопат сприяло збільшенню температури всередині обертових печей. Розміщення сушарок вплине на зниження адгезії всередині обертових печей, тоді як з економічної точки зору ми отримаємо зменшення кількості палива в завантаженні обертових печей.

Ключові слова: *залізо-нікелева руда, налипання, Calphad, стадії, підетапи*

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