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USE OF COLEMANITE IN FERRONICKEL SMELTING

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Abstract

Use of colemanite in metal-slag systems aims primarily to decrease the viscosity of slag and, therefore, achieve better metal-slag separation. Enhanced metal-slag separation is helpful to decrease the number of suspended metal/alloy droplets in slag, i.e. the physical losses. In the literature, successful use of colemanite was reported both in steelmaking and copper matte smelting processes. Ferronickel smelting slags contain nickel in the range of 0.1-0.2% and correspondingly, metal-slag distribution ratio values of nickel are reported even above 200. On the contrary, nickel recoveries are hard to exceed 95%. This can be mostly attributed to the physical losses of nickel due to very high slag volume in ferronickel smelters; for 1 ton of ferronickel, 10-15 tonnes of slag are generated regardless of the type of the laterite, which contains significant quantity of gangue components. The authors thought that use of colemanite could be a solution to decrease physical losses. Therefore, the use of colemanite in ferronickel smelting was investigated in the present work. Laboratory-scale smelting experiments were conducted using calcined and prereduced laterites in a vertical tube furnace under different gas atmospheres. The amount of colemanite added was in the range of 0 - 2.5% of the total charge. The experiments were also performed using ferronickel and slag samples obtained from a ferronickel smelter.

Keywords: *Colemanite; Ferronickel; Smelting*

1. Introduction

Colemanite is one of the fluxes used in iron and steelmaking. In steelmaking, it has been added to the basic oxygen furnaces (BOF) instead of CaF_2; and/or used as a flux in sintermaking before the blast furnace. According to the studies conducted, the following advantages were reported in relation to the use of colemanite in BOF: 1. It decreased the melting temperature of the slag, thus providing higher fluidity, 2. It increased the solubility of lime and magnesia in the slag. 3. No significant damage was reported for the refractories of the converter. On the other hand, one important disadvantage was also noted as reversion of boron from slag to steel under reducing conditions [1-4]. This is primarily due to the slag carryover taking place during tapping and the reversion becomes more severe when the amount of slag carryover is higher than usual. As an example, during one of the investigations, colemanite addition trials resulted in slabs having boron content more than 100 ppm, causing severe cracks on the slab surfaces [5]. Regardless of the slag-free tapping systems and/or thermal cameras installed to the converters, unfortunately, some amount of converter slag went to the ladle during tapping. Therefore, the risk of boron reversion is always a major task in steelmakers' mind; therefore they are very reluctant to use the colemanite. It is indicated as colemanite for the sake of simplicity, actually it is calcined colemanite since calcined form of colemanite can be used in such high temperature vessels due to the presence of chemically bound water in the colemanite structure.

Colemanite has also been used in extractive metallurgy of copper. In reverberatory matte smelting, colemanite and boric acid were added to the furnace and it was reported that matte-slag equilibrium were attained much quickly with the addition of such fluxes [6]. In a more recent laboratory scale work, the addition of colemanite to the matte smelting slags was found to be beneficial to lower copper losses to slag from 0.88% to 0.3-0.4% levels. This addition was also applied industrially and it was indicated that significant fuel economy was obtained with the use of

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colemanite. The money saved was reported to be higher than the cost of the colemanite. According to the results, slag fluidity increased dramatically; on the other hand copper losses to the slag did not decrease. The authors addressed this to the deficiencies of the furnace design [7,8].

The authors of the current study worked on ferronickel smelting from one of the Turkish laterites present in the Central Anatolia, namely Sivrihisar. This work covered two projects [9,10] and a PhD thesis study [11] dealing with primarily characterization [12], calcination [13,14], prereduction [15,16] and smelting [17,18] of Sivrihisar laterites in laboratory scale systems. Ferronickel smelting slags contain nickel in the range of 0.1-0.2% and correspondingly, metal-slag distribution ratio values of nickel are reported even above 200. On the contrary, nickel recoveries are hard to exceed 95%. This can be mostly attributed to the physical losses of nickel due to very high slag volume in ferronickel smelters; for 1 ton of ferronickel, 10-15 tonnes of slag are generated regardless of the type of the laterite, which contains significant quantity of gangue components. Efforts should be made to decrease the viscosity of ferronickel slags. In relation to the viscosity of laterite smelting slags, the readers are referred to the paper which was published by X.M. Lv et al. [19]. The authors of the present work thought that use of colemanite could be a solution to decrease physical losses. Therefore, the use of colemanite in ferronickel smelting was investigated in the present work.

2. Experimental

The laterite ore from Sivrihisar was supplied by META Nickel Cobalt Co. Calcination experiments were conducted in an externally controlled muffle furnace. 700ºC and 40 minutes were determined as the optimum calcination temperature and time, respectively [13, 14]. The chemical composition of the laterite after calcination is shown in Table 1. Ni content was reported to increase from 1.41% to 1.58% together with calcination.

Prereduction experiments were performed using a horizontal tube furnace under different atmospheres as argon, nitrogen and a mixture of N_2-CO_2-CO. The readers are referred to the related reference for more details about the prereduction setup and the results [16].

Table 1. *Chemical composition of the laterite after calcination (%) [11]*

Ni	Co	Fe	SiO_2	MgO	Cr	CaO	Al_2O_3
1.58	0.103	37.1	31.6	1.44	1.38	1.65	3.48
MnO	As	P_2O_5	S	Cu	TiO_2	Zn	K
0.85	0.03	trace	0.05	0.013	0.07	0.03	0.1

Smelting experiments were performed using both a horizontal tube furnace and a vertical tube furnace, but considering certain difficulties of the horizontal one, which are out of scope of this paper, the ones conducted using vertical tube furnace were emphasized more. The smelting system utilizing vertical tube furnace is presented in Figure 1, together with the sample crucible used in the experiments [18]. The same setup was also used in all the runs subjected to colemanite addition. Smelting experiments were conducted under argon (Ar) and 67%CO-33%CO_2 gas mixture, which is compatible with the typical gas composition present in electric arc furnaces used for ferronickel smelting [20].

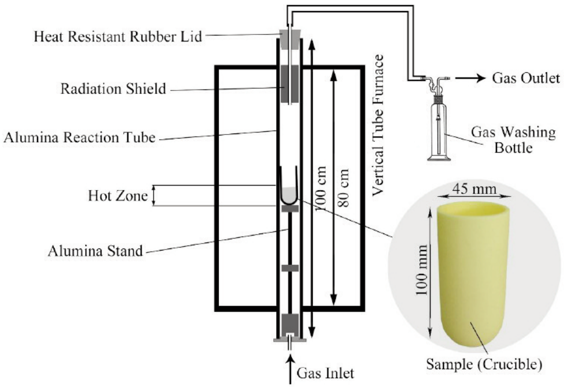

Figure 1. *The experimental setup with a sample crucible [18]*

Ground colemanite having particle size of -75 µm was taken from Eti Mine Works Co. It was subjected to a thermal treatment at 400ºC for 24 hours to ensure the removal of chemically bound water present in its structure. For this purpose, sample was mixed occasionally during that period. The chemical analysis of calcined colemanite - for the sake of simplicity, it is shortly called colemanite in the rest of the paper, is shown in Table 2.

Table 2. *Chemical composition of colemanite in calcined form (%) [11]*

B_2O_3	CaO	$CaCO_3$	SiO_2	Al_2O_3+MgO+SrO
51.7	27.7	8.6	7.9	4.1

Colemanite addition experiments were also conducted using a real metal-slag system established with ferronickel and slag samples supplied from the smelter of Larco. Chemical compositions of ferronickel and slag samples are shown in Table 3 and Table 4, respectively.

Table 3. *Chemical analysis of the industrial ferronickel (%) [11]*

Ni	Co	As	S	C	Cu	P	Si	Al	Fe
14.85	0.466	0.055	0.214	0.052	0.034	0.035	0.005	0.003	Balance

Table 4. *Chemical analysis of the industrial slag (%) [11]*

FeO	SiO_2	Al_2O_3	CaO	Cr_2O_3	MgO	MnO	TiO_2
43.71	33.49	8.83	4.44	4.33	3.72	0.45	0.41

Chemical compositions of metal and slag produced in the experiments were determined by XRF. Wet methods containing two steps were utilized to quantify the physical and chemical losses. In the first step, physical losses were obtained using bromine-methanol technique [11, 21], and afterwards, the atomic absorption spectrophotometry (AAS) was used to measure the dissolved metals. In the second step, the residue from the first step was dissolved in an acid mixture (HNO_3+HCl+HF) and the solution was analyzed by AAS to obtain the chemical losses.

3. Results and Discussion

The smelting experiments conducted to study the outcomes of colemanite addition are presented in Table 5 together with the experimental parameters. The main objective was to decrease slag viscosity and to enhance metal-slag separation and therefore to decrease the physical losses of nickel. There are primarily two sets of experiments: In the first set of experiments, the tests were performed under argon atmosphere (Exps. 1-5); whereas in the second set (Exps. 6-10), the experiments were conducted under a gas mixture of 67%CO-33%CO_2. In the experiments 1 to 7, laterite ore taken from Sivrihisar was the raw material. On the other hand, in the experiments 8 to 10, industrial ferronickel and slag samples supplied from Larco were used.

In the smelting experiments conducted under Ar atmosphere (Exps. 1-5), starting from calcination of Sivrihisar laterite, the following steps were followed till smelting, i.e. the flowsheet given in Figure 2. Weights of ferronickel buttons obtained from the experiments are presented together with their compositions in Table 6.

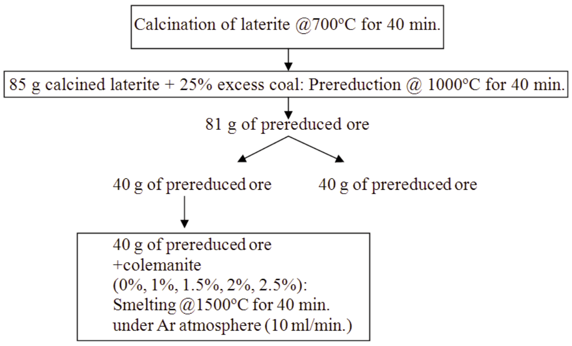

Figure 2. *Flowsheet of the smelting experiments conducted under Ar atmosphere*

According to the results of the experiments conducted under Ar(g) atmosphere, ferronickel weight was reported to decrease with the addition of colemanite. All the runs performed with colemanite addition produced a metal button less than 7.62 g, which was the ferronickel weight obtained from colemanite-free experiment. The decrease of the weight of ferronickel was sharp at first, then after 1 wt.% colemanite addition the weight showed a gradual rise with a final leveling out trend between 2

Table 5. *Smelting experiments conducted to study the effect of colemanite [11]*

Exp. No	Objective	Experimental Parameters					
		Atmosphere	Temp. (°C)	Time (min.)	Excess Coal in Prereduction* (%)	Calcine Input (g)	Colemanite (wt.%)
1	To study the outcomes of colemanite addition in relation to physical losses	Ar (10 ml/min.)	1500	40	25	42.5	0
2			1500	40	25	42.5	1
3			1500	40	25	42.5	1.5
4			1500	40	25	42.5	2
5			1500	40	25	42.5	2.5
6		(20 ml CO +10 ml CO_2)/min.	1480	5	12.5	40	2
7			1500	1	12.5	40	2
8	Smelting of industrial ferronickel and slag samples to analyze the outcomes of colemanite addition		1550	10	No coal, no calcine (50 g powdered slag + 4 g ferronickel filing		0
9			1550	10			1
10			1550	10			2

* Certain parameters: Calcination: @700°C for 40 min., Prereduction: @1000°C for 40 min., Prereduction gas atmosphere: 20%CO_2-10%CO-70%N_2.

Table 6. *Chemical composition of the metal products for exps. 1-5 [11]*

Exp. No.	Flux (%)	Weight of Ferronickel (g)	Contents of Ferronickel (%)					
			Ni	Co	As	S	Cr	Fe
1	0	7.62	8.79	0.57	0.21	0.12	0.09	Balance
2	1	6.47	10.22	0.66	0.19	0.14	0.08	Balance
3	1.5	6.79	9.85	0.64	0.2	0.18	0.1	Balance
4	2	7.19	9.24	0.61	0.2	0.13	0.08	Balance
5	2.5	7.05	9.72	0.58	0.17	0.13	0.1	Balance

Table 7. *Chemical composition of the slags for exps. 1-5 [11]*

Exp. No.	Flux (%)	Weight of Slag (g)	Contents of Slag (%)						
			FeO	SiO_2	Al_2O_3	CaO	Cr_2O_3	MgO	MnO
1	0	29.94	39.56	36.91	15.39	2.78	2.4	1.55	1.14
2	1	32.25	43.67	34.02	14.2	3.31	1.95	1.39	1.15
3	1.5	32.49	41.9	34.22	15.72	3.32	2.01	1.38	1.17
4	2	32.41	40.45	34.51	16.62	3.48	2.07	1.42	1.15
5	2.5	33.09	40.2	34.19	16.71	3.84	2.13	1.49	1.16

and 2.5 wt.% colemanite additions. As it can be seen from Figure 3, just the opposite (symmetrical to x-axis) behavior was noted for variation of the nickel content of the metal (i.e. ferronickel grade). The reason for this behavior was explained in one of the earlier publications of the current authors. After the runs performed with the use of excess coal (0-18 wt.%), the similar behavior was obtained. It was indicated that nickel was much easily reduced than iron at higher coal additions, so nearly the same amount of nickel entered to ferronickel in all runs, proven by analysis and calculations. Therefore, once a decrease in ferronickel weight is reported, its direct consequence is an increase in ferronickel grade since the nickel weight remains nearly constant [18]. The results of the experiments conducted in this laboratory-scale system imply that colemanite addition did not provide beneficial outcomes since ferronickel weight was found to decrease with this addition.

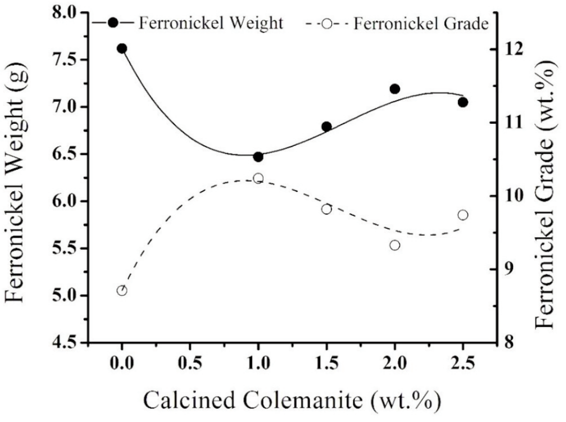

Figure 3. *Change in the weight and the grade of the ferronickel buttons with colemanite addition*

Variations of physical losses with colemanite addition are shown in Figure 4. Both the amounts of entrapped Ni and Co were reported to increase first

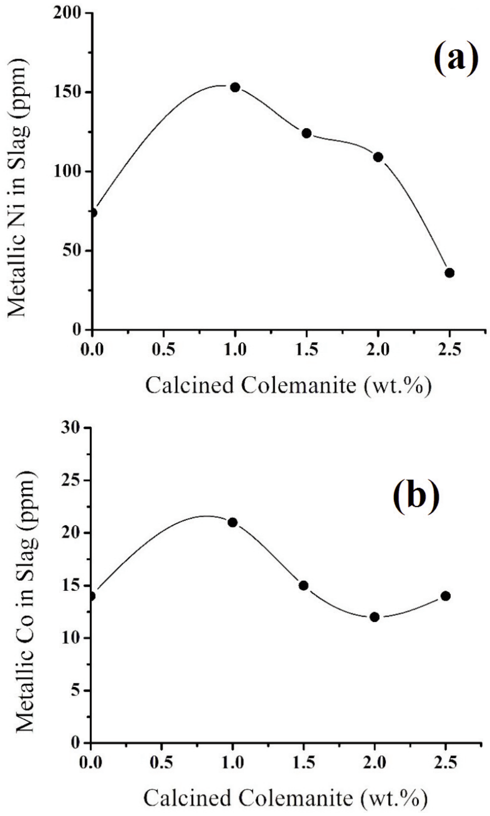

Figure 4. *Variation of physical losses (a) Ni, (b) Co with fluxing of colemanite*

with colemanite addition and therefore showed a peak at 1 wt.% colemanite fluxing. Beyond 1 wt.% colemanite addition, physical loss of Ni showed a continuously decreasing trend, whereas that of Co presented a gradual decrease followed by something like a steady-state behavior. Actually, the only difference between the behavior of Ni and Co was seemingly coming from the experiment with 2.5 wt.% colemanite fluxing. The analysis conducted to determine physical loss of Co for 2.5 wt.% colemanite addition was repeated but the same result was reported. This result might be attributed to an experimental error or a problem in charge preparation.

Chemical losses of Ni and Co showed a similar behavior encountered as in the case of physical losses. The variations of dissolved Ni and Co in slag with colemanite addition are illustrated in Figure 5. There are peaks at 1 wt.% colemanite addition for both Ni and Co, followed by decreasing trends. This time, dissolved Ni and Co content values found for 2.5 wt.% colemanite addition are not compatible with the expected trend so there might be certain mistakes in relation to that experiment, as indicated before. Apart from this questionable item, it can be easily concluded that both the physical and the chemical losses are seemingly decreasing beyond 1 wt.% colemanite addition. The trends given in Figure 4 and Figure 5 mostly resemble to the one given for ferronickel grade (i.e. Figure 3). This can be regarded as a proof of the fact that both the physical and chemical losses of Ni and Co can be controlled mainly by the grade of ferronickel.

Having completed the first set of experiments under Ar(g) atmosphere, the next step was to study the outcomes of colemanite addition in the same system under 67%CO-33%CO_2 atmosphere, which is more or less the same as the one present in electric arc furnaces used for ferronickel smelting [20]. Smelting experiments conducted under CO/CO_2 atmosphere (i.e. Exps. 6-7) were performed following the similar flowsheet given in Figure 2, except for the furnace atmosphere in smelting. Weight of ferronickel button obtained from the experiment with 2 wt.% colemanite addition is illustrated together with the chemical composition in Table 8. Correspondingly, physical and chemical losses Ni and Co for the same experiment are illustrated in Table 9. It was reported that the results were not significantly different than the ones conducted under Ar(g) atmosphere. Therefore, the same conclusions can be drawn for the experiments conducted under CO/CO_2 atmosphere.

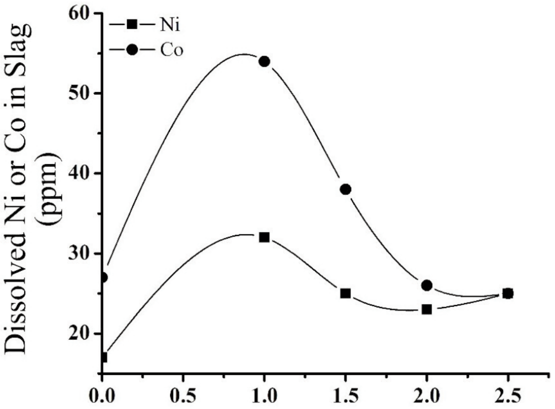

Figure 5. Changes in chemical losses of nickel and cobalt with fluxing of colemanite

***Table 8.** The results of exp. 7 performed with fluxing of 2 wt.% colemanite*

Exp. No.	Weight of Ferronickel (g)	Contents of Ferronickel (wt.%)					
		Ni	Co	As	S	Cr	Fe
7	4.6	14.06	0.94	0.3	0.09	0.08	Balance

***Table 9.** Physical and chemical losses of Ni and Co for exp. 7*

Exp. No.	Entrapped Metal in Slag (wt.%)		Dissolved Metal in Slag (wt.%)	
	Ni	Co	Ni	Co
7	0.0074	0.002	0.0013	0.0058

In an effort to answer the question whether the colemanite addition reduces the melting temperature of the slag or not, exp.6 was performed. The authors previously found that 1490°C was the melting temperature of the slag, which was colemanite-free [11]. Therefore, exp.6 was performed at 1480°C with 2 wt. % colemanite addition. On the other hand, it was found that the sample did not melt completely and that colemanite fluxing did not drop the melting temperature even by 10°C.

In metal-slag systems, entrapped metal droplets in slag settle down according to the Stokes' law of settling, as follows:

$$v_{droplet} = \frac{2}{9}\frac{g(\rho_{metal}-\rho_{slag})}{\mu_{slag}} \cdot r_{droplet}^{2} \qquad (1)$$

where $v_{droplet}$ is the velocity of metal droplet, g=9.81 m/s^2, ρ_{metal} and ρ_{slag} are the densities of metal and slag, respectively; μ_{slag} is the viscosity of slag and $r_{droplet}$ is the radius of metal droplet.

In a calculation based study, it was found that colemanite addition was beneficial to decrease the melting temperature and the viscosity of fayalite-type slags [8]. So, in the present work it was anticipated to have a decrease in viscosity of ferronickel slags and therefore to have a better slag-metal separation lessening the physical losses but the findings revealed that colemanite fluxing did not create a positive outcome in the system under consideration. In relation to this implication, the effect of colemanite addition on slag composition was further analyzed. It was found that the interaction of the alumina crucible and slag increased with the introduction of colemanite. A slag having no porosity was obtained when there was no colemanite addition. On the contrary, shrinkage porosities were observed when 2.5% colemanite fluxing was conducted (Figure 6). Also, the rise of alumina content of the slags demonstrated the increased interaction of alumina crucible with slag when colemanite was added. It was indicated in the literature that small amount of Al_2O_3 (3-5 wt.%) was effective to reduce the liquidus temperatures of ferronickel slags [22-24], on the other hand Al_2O_3 content of the slags were higher than 12 wt.% in colemanite added runs. Therefore, the positive effect of colemanite addition might be cancelled out partly with the increase in alumina content of the slag.

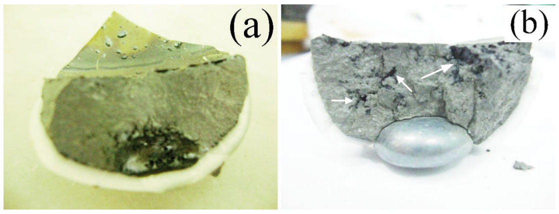

Figure 6. *Cutaway view of the metal & slag with (a) no fluxing and (b) 2.5% colemanite fluxing [11]*

Another negative influence might have arisen from evaporation of boron oxide present in colemanite. The standard Gibbs free energy change of evaporation of B_2O_3 is given as follows:

$$B_2O_3(liquid) = B_2O_3(gas) \quad \Delta G^o = 389.45 - 0.1685T(kJ) \qquad (2)$$

The variation of partial pressure of B_2O_3 (gas) in equilibrium with pure B_2O_3 (liquid) with temperature was constructed in Figure 7.

As it can be seen from Figure 7, evaporation of B_2O_3 increases drastically above the temperature of 1400°C. In fact, after the runs performed with colemanite addition (all of them were performed above 1400°C), needle-like and white colored B_2O_3 structures were observed on the upper parts of the furnace tube. It indicates that evaporation of B_2O_3 took place during the experiments. Moreover, this evaporation might cause foaming of the slag.

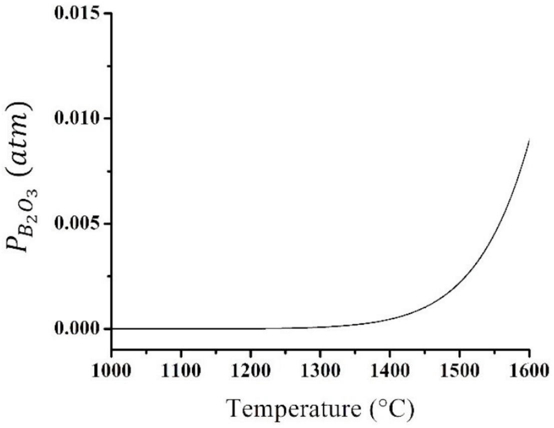

Figure 7. *Change of equilibrium $P_{B_2O_3}$ with temperature*

The experiments performed starting with Sivrihisar lateriate ore reflected the behavior obtained from laboratory-scale systems, including mainly calcination, prereduction and smelting of the ore. It was thought that it was a good idea to repeat the smelting experiments with the use of industrial ferronickel and slag samples supplied by a Greek ferronickel company, Larco. Among the ferronickel producers, Larco was preferred since the Greek and the Turkish laterites are similar to each other in regard to the chemical and mineralogical compositions of the ores [25,26].

Some preliminary experiments conducted with Larco ferronickel and slag showed that slag had a melting temperature below 1450°C and that melting of ferronickel did not take place at 1500°C. Therefore, 1550°C was selected as the smelting temperature for the experiments investigating the effect of colemanite addition. Three experiments were performed (Exps. 8-10). In each run, 4 g of ferronickel fines were mixed with 50 g of slag. At the end of experiment 8, it was reported that ferronickel weight was 3.12 g. In the experiments 9 and 10, 1 and 2 wt. % colemanite was added to the charge, respectively (Table 10). Results of the experiments revealed that ferronickel weight decreased with the addition of colemanite. Therefore, it seems that the colemanite addition was not helpful to enhance the metal-slag separation for the system under consideration.

Table 10. *Ferronickel weights obtained from the experiments performed with smelting of industrial ferronickel and slag samples*

Exp. No	8	9	10
Flux (wt.%)	0	1	2
Weight of Ferronickel (g)	3.12	3.03	3.04

4. Conclusions

According to the results of the experiments conducted, the following conclusions were drawn for the metal-slag system under consideration:

- Regardless of the type of the furnace atmosphere (Ar or CO/CO_2), colemanite addition caused a decrease in ferronickel weight. Just the opposite behavior was reported for ferronickel grade. Both the physical and chemical losses of Ni and Co showed the same trends with ferronickel grade. The physical and chemical losses of Ni and Co were reported to decrease beyond 1 wt.% colemanite addition. However, ferronickel grade was also found to show a decrease beyond this point. Therefore, there was no significant positive effect of colemanite addition.

• Colemanite addition was not effective to reduce the melting temperature of the slag. This might be attributed to the increased alumina content of the slag caused by higher extent of interaction between the alumina crucible and the slag as a result of colemanite addition. Therefore, the effect of colemanite might be cancelled out partly. Evaporation of B_2O_3 present in colemanite was noted as another important event possibly influencing the process.

• Ferronickel weight was reported to decrease with colemanite addition in the smelting experiments conducted with the use of industrial ferronickel and slag samples. Therefore, colemanite fluxing is seemingly not suitable for the system under consideration. In the extent of the current laboratory-scale study, alumina crucibles were used. On the other hand, industrial ferronickel smelters are mostly lined with magnesia refractories [27]. Therefore, the colemanite addition trials in a real industrial smelting furnace may produce different results. This might be a kind of future study for researchers.

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UPOTREBA KOLEMANITA KOD PROIZVODNJE FERONIKAL LEGURE

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Apstrakt

Kolemanit se prvenstveno upotrebljava u sistemima metal-šljaka da bi se smanjila viskoznost šljake, a samim tim da bi se postiglo i bolje odvajanje metala i šljake. Poboljšano odvajanje metala i šljake doprinosi smanjenju broja suspendovanih kapljica metala/legure u šljaci, odnosno, smanjenju fizičkih gubitaka. U literaturi se primeri uspešne upotrebe kolemanita mogu naći kod proizvodnje čelika, kao i kod postupka topljenja na bakrenac. Šljaka koja nastaje tokom proizvodnje feronikal legure sadrži nikl u opsegu od 0,1-0,2%, i u skladu sa tim, odnos raspodele metala i šljake za nikl iznosi preko 200. S druge strane, količina dobijenog nikla ne prelazi 95%. Ovo se može objasniti fizičkim gubitkom nikla zbog velike količine šljake koja nastaje prilikom topljenja – kod proizvodnje 1 tone feronikla, dobije se 10-15 tona šljake bez obzira na vrstu laterita koji sadrži značajne količine jalovine. Autori su razmatrali upotrebu kolemanita kao rešenje za umanjenje fizičkog gubitka. Stoga, upotreba kolemanita kod topljenja feronikal legure je ispitivana u ovom radu. Prilikom eksperimenata topljenja, koji su urađeni u laboratoriji, korišćen je žareni i umanjeni laterit, kao i vertikalna cevna peć uz prisustvo različitih gasova. Količina kolemanita koji je dodavan je iznosila od 0-2,5% od ukupne količine punjenja. Tokom eksperimenta su takođe korišćeni uzorci feronikla i šljake dobijeni iz topionice.

Ključne reči: *Kolemanit; Feronikal; Topljenje.*