RECYCLING OF LADLE SLAG IN THE EAF: A WAY TO IMPROVE ENVIRONMENTAL CONDITIONS AND REDUCE VARIABLE COSTS IN THE STEEL PLANTS. THE RESULTS OF STEFANA SPA (ITALY)¹

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Abstract

Environment is becoming more and more a key issue for the Steel Industry. In areas like Europe, where the increasing variable cost of production and the cost of men power are reducing the profit, the additional cost of residues dumping can reduce to zero the result of the steel plant. Only Italian Electric Steel produces more than two million tons of slag per year and other various types of residues as spent refractory, fumes dust, mill scale, etc. The recycling of these by-products permits to achieve three important results: minimizing the exploitation of natural resources, reducing the amount of dumped material, but also the cost of EAF fluxes, something important for the steel production balance. In 2005 has been started up the second Italian industrial plant that permits to convert ladle furnace slag and spent refractory into a final powder product that can be injected into the EAF. The plant is located in the Ospitaletto Works of Stefana SpA, a Steel plant producing about one million tons per year of wire rods and beams. This industrial application shows the sustainability of this particular recycling procedure, cost and process-wise. Important benefits, moreover the reduced cost for slag dumping, is coming from the reduced amount of lime charged into the EAF. **Key words:** Ladle Slag Recycling, Refractory Recycling, Steel Plant Waste Recycling, Foamy Slag

RECICLAGEM DE ESCÓRIA DE PANELA NO FEA: UM MEIO DE MELHORAR AS CONDIÇÕES AMBIENTAIS E REDUZIR CUSTOS VARIÁVEIS NAS SIDERÚRGICAS OS RESULTADOS DE STEFANA SPA (ITÁLIA)

Resumo

O meio ambiente está se tornando cada vez mais um ponto chave para a Siderurgia. Em áreas específicas onde os crescentes custos variáveis de produção e de mão de obra estão reduzindo o lucro no produto final, o custo adicional de descarte de resíduos pode reduzir a zero o resultado da siderúrgica. Somente as aciarias elétricas italianas produzem mais de dois milhões de toneladas de escória por ano e outros tipos variados de resíduos como refratário descartado, pó de fumos, carepas etc. A reciclagem destes subprodutos permite se alcançarem três resultados importantes: minimização da exploração de recursos naturais, redução do volume de material descartado e também do custo de fundentes no FEA, algo importante para o balanco de produção do aco. Em 2005 partiu a segunda planta industrial na Itália que permite converter escória de forno panela e refratário descartado em um produto final em pó que pode ser injetado no FEA. A planta está localizada na Usina de Ospitaletto da Stefana SpA, siderúrgica que produz cerca de um milhão de toneladas anuais de fios-máquina e vigas. Esta aplicação industrial mostra a sustentabilidade deste procedimento de reciclagem em particular, tanto em termos de custo quanto de processo. Benefícios importantes, além do custo reduzido de descarte de escória, estão vindo da redução do volume de cal carregado no FEA.

Palavras-chave: Reciclagem de escória de panela; Reciclagem de refratário; Reciclagem de subprodutos em siderúrgicas; Escória espumante.

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TYPE OF RESIDUES AND WASTES

The majority of the waste material object of this recycling process is the so-called "ladleslag" – the slag that remains in the ladle after the casting process – and all the refractory material of the ladle after its usual life cycle. Both are unstable base-materials, subject to structural changes that convert these materials into very fine powder. For this reason the handling of this waste is difficult and, on another hand, the cost for the disposal or storage is very high due to their chemical composition.



Figure 1. Examples of material that can be recycled: used refractory and ladle slag

TREATMENT METHOD OF RESIDUES AND WASTES

The outcome of the experimental testing in Ferriere Nord and the recycling simulation, as said, lead to the implementation of an industrial-scale prototype plant, in operation since more than three years. The plant is able to treat and inject into the EAF the base mixture made from ladle slag, tundish and ladle refractory, EAF bottom refractory and swept fines. The "metallurgical simulation model" has demonstrated that additional waste material can be added to this recycling mix such as EAF refractory bricks and others. Moreover important implementation in the treatment of these residues was needed in order to speed-up the treatment time and increase the recycling energy efficiency. The design of the plant in Stefana is the natural consequence of the experience in Ferriere Nord and will allow a more complete recycling process





Ladle Slag Treatment

As far as the 90 percent of the material to recycle is ladle slag, it is important to focus the attention on this by-product.

Ladle Slag has an usual composition – for rebar and structural steel – of about 60 percent of CaO plus MgO and about 20 percent of silica and alumina. The analysis of the components of this type of slag, performed for the case of Stefana, indicate that the percentage of Di-calcium silicate is the most important. C2S can be present in the ladle slag in different phases: α , α_{H} ', α ', β , γ . The phase α -C2S is stable at very high temperatures. During the cooling down of the ladle slag it is changing to β -C2S at 630°C, then transforms to γ -C2S at temperature lower than 500°C.

$$\gamma \xrightarrow{1053-1133K}_{963K} \alpha \xrightarrow{1433K}_{H} \alpha_{H}^{2403K} \alpha \xrightarrow{2403K}_{H} \text{ liquid}$$

Figure 3. Experimentally measured phase transition temperatures for C2S polymorphs

The conversion β - γ is accompanied by 10% volume increase (from ~56 to ~62·10⁻⁶ m³/mol) and results in the matrix shattering into powder because of their different crystal structures and density.

The typical temperature of the β - γ transformation for this silicate is well known in literature (The β - γ transformation for di-calcium silicate is starting below 773 Kelvin).

Our treatment method consists in the control the cooling-down curve of the ladle slag and the recovery of the hot powder before it reaches the ambient temperature, when the hydration reaction can easily take place.

One of the problems of the application of this treatment method is that ladle slag is a very good refractory material, due to its analytic composition, and this means that while the surface is cooling down quickly, the internal part of the slag is still hot and far away from the target temperature.

A specific mechanical movement is necessary to homogenize the ladle slag temperature and allow a uniform cooling curve for the slag mass. Ladle slag is vibrated – horizontally and vertically – and revolved in many different steps. Besides that, the atmosphere where the slag is treated is carefully controlled, to avoid as much as possible high rates of humidity and presence of undesired gasses.

The air temperature is also controlled and precisely driven to lower temperatures in order to cool down the slag with the desired curve. Ladle slag temperature is constantly measured to be ready to extract the powder material as soon as it is starting to appear.

The result of this peculiar treatment can be divided in two parts: the shattered fraction and the non-shattered fraction. The first one is collected into special silos to a temperature always higher than hundred degrees Celsius, while the second one is suffering a secondary treatment of crushing in a different section of the Recycling Plant, to which is automatically conveyed.

Ladle, tundish and EAF bottom Refractory Treatment

Due to their analytic composition, the three types of wastes are suffering similar transformation of the crystalline structure; nevertheless the typical transformation time is longer than the case of the ladle slag. As far as these three materials are usually collected and inserted into the recycling process to lower temperature – ladle, tundish and EAF bottom demolition take place when the these three parts are much colder than their

working temperature – the possible interaction with the humidity is certainly higher. This is why the specific mechanical treatment is enforced. Same controls on ambient temperature and gasses are done, same control of the material temperature is performed. The nonpowder fraction is usually higher. The result of this treatment is normally a powder material more rich in MgO respect to the powder material collected in the silo of the ladle slag.

Swept fines and other fine wastes Treatment

This type of residue is already in powder state, so there is no need of special treatment of its crystalline transformation. However the risk of hydration is much higher. This material is usually collected daily in the plant and is transported to the recycling plant where is collected in a specific silo. The silo's atmosphere is controlled as per the other cases. The composition of this material is very flexible, depending on where the sweeping machine has worked that day or depending on specific areas of dust production specially. Nevertheless the quantity of this material is very small respect to the rest of residues to its variability does not affect the final mix.

EAF bricks Treatment

Due to the chemical composition, this material is not performing the natural transformation of crystalline structure. Anyway the high presence of MgO constitutes for the recycling plant an important source of base material. For this reason the EAF bricks are treated in a separate section of the recycling plant, where special crushers are granulating this material to achieve the right grain size.

POWDER MIX COMPOSITION

As far as the chemical composition of the single recycled materials is not variable, on a large-scale average for each plant, the only way to achieve different chemical compositions of the final powder mix is the percentage of each single recycled material in the final powder.

This is why, based on the chemical analysis of the various components it is possible to add to the ladle slag powder, which is the base of the recycled product, different percentages of the other materials, in order to achieve the "formula" of the mixture to be fed into the EAF.

A system of vibrant-extractors and independent belts is needed to connect each silo, containing the different recycled materials, to the main transportation belt, on which the ladle slag is conveyed at high temperature before getting to the required screening machine.

A PLC-based automation system controls the composition of the powder mix, and it has tracking functions to determine the stratification composition of the main storage silo, which is the silo where the final mix is charged after the recycling treatment.

In this way, based on the steel grade to produce (quality limitation for each chemical element in the liquid steel), based on the metallurgical model implemented in the automation system (capable to determine the interaction between EAF slag and steel and the possible pick-up of undesired chemical elements), the PLC automatically calculate the right quantity of recycled material that can be added to the EAF slag without compromising the steel quality.

RECYCLING INTO THE ELECTRIC ARC FURNACE

The powder mix is conveyed through a special rubber belt to the storage silo and during this transportation a metallic separator extracts from the mix the iron drops that were contained in the ladle slag and the other iron fraction of the mix. The recovering of iron and

steel is not negligible.

Before the charge to the silo, a screen is calibrating the powder mix grain size. The grain size is determined depending on plant layout, distance between silo and EAF, pressure of the pneumatic transportation, pipeline material and desired wear and other aspects. The material with a higher grain size is automatically recharged into the recycling treatment plant and will suffer granulation or shattering depending on its dimension.

The final stage of the treatment plant is the storage into the daily silo. The process-cycle can be though as a system working on a daily base: the ladle slag and other materials produced in one day are charged in the recycling plant and treated the same day; the powder mix generated the same day is charged into the storage silo the following day. In this way the silo is always charging into the furnace a known powder mix composition, prepared few hours before.



Figure 4. Schematics of the system in operation at Ferriere Nord works in Osoppo, Italy

The final pneumatic transportation drives the powder mix into the EAF via special powder injectors that are the KT Powder Injectors of Techint. The peculiar characteristics of the cooling system of these lances permits their installation submerged into the slag line with maximum operational safety (more than 250 lances in operation worldwide for different type of injections, included the one of Ferriere Nord). The efficiency of the powder mix injection into the slag is thus 100 percent, as the tip of the lance is right into the slag line. Actually, if the powder mix would have been injected with a normal pipe out of the slag line, the EAF Dedusting System would immediately suck the very fines particles, due to the delta pressure between EAF and Fumes duct. The use of the right injector is one of the keys of the success of this recycling process.

BENEFITS ARISING FROM THIS RECYCLING PROCESS

From a chemical point of view, the injection of powder mix, mainly composed of Di-Calcium Silicate, is increasing the amount of the already present C2S of EAF slag, which is characterised by the following mineral composition: olivine, merwinite, β -C2S, C3S, C4AF, C2F, RO phase (solid solution of CaO-FeO-MnO-MgO) and free CaO. The C2S is normally present at EAF slag temperature as "suspended second phase particles".

The addition of other particles coming from the recycled mix has three consequences: a mass effect (lowering FeO, and increasing slag width), the increase of MgO (present in the mix) and the increase of the CO nucleation sites, which leads to a high amount of favourable small gas bubbles in the foamy slag.

The presence of suspended second phase particles has a greater impact on foaming properties than the surface tension decrease and the viscosity increase, which are factors for a good foaming. The "optimum" slag is not completely liquid, but is saturated with respect to C2S and Magnesia-Wustite solid solution, as shown in the Isothermal Stability

Diagrams (dotted lines). This is why the addition of powder recycle mix is improving the EAF slag properties.

The increase of tenor of MgO in the EAF slag is in favour of a lower consumption of the EAF refractory bricks, an additional benefit to be counted.



Figure 5. ISD at 1600°C showing initial slag (•) and three targets (•) for good foaming slags with (a) at 40 %FeO and (b) at 30 %FeO relative to the curve for B3=2.0 and (c) with 30%FeO for B3=1.5.

Special attention must be paid to the fluxes addition through the KT Injection System. The recycled powder is added in the EAF slag continuously, and not all together, as per lime addition via buckets. This permits to the slag to control in a better way the basicity index. The presence of "icebergs" of lime is eliminated.

One advantage of this recycling process is the important recovery of iron from all these byproducts. Usually, subcontractors that each plant uses for the slag handling recover the iron contained in the slag, but not always this iron is coming back to where it has been produced. The recycling approach indicated permits to maintain this resource inside the plant.

The environmental situation of the plant is certainly an important item to be analysed. This recycling plant permits to reduce to the minimum the powder dust present in the plant atmosphere, as there aren't anymore areas where dust powder is left at open air. The recycling plant is completely closed and it is equipped with a suction filter and bag-house. The dust here collected is obviously recharged into the recycling process. The powder fractions of the recycled material – as tundish and ladle or pots skulls – are the only output of the recycling plant, which is not stored into the daily silo. The difference of the skulls coming out of the recycling plant and the normal skulls produced in the steel plant is that they are clean and ready to be transported to the scrap-yard, where they can be recharged into the scrap buckets. Even in case these skulls have so big dimensions that must be cut, they do not have slag incrustations, so this cutting operation is faster and saver for the worker. The internal recycling of the wastes and residues listed above produce a lower cost for the disposal of such materials, whose amount is variable depending on the regional legislation, local taxes, disposal sites availability, the associated transportation cost and so on. In Italy, as per many Countries of the U.E., the rules are becoming stricter year by year, so the recycling approach is not only an opportunity, but also a mandatory perspective for the steel production survival.

METALLURGICAL SIMULATION MODEL

The cooperation between Techint and the Italian University Politecnico di Milano, Mechanical Department, has produced as result a versatile and flexible proprietary metallurgical model to calculate the possible application of this recycling process to other steel plants. The large-scale application of this process is a real opportunity. The ideal use of the ladle slag recycling in all European steel plants will possibly reduce of about 30% the utilization of natural lime stones, an ideal perspective environmentally-wise, considering the additional cost benefits for the steel producers. The model is able to calculate the fluxes changes of input and output material in the EAF when the mix recycling is applied. Particularly it calculates the quantity of lime which is possible to save into charge of the EAF.

While the lime is constituted for about 90% by CaO, which is completely able to react in the EAF, the CaO charged with recycling powder mix, although the high rate, isn't entirely available to react because it's already partly committed to form complex compounds.

Therefore it's calculated the active CaO as CaO contained in the powder mix that is able to react:

Where:

CaO $_{\mbox{\scriptsize FREE}}$ is the CaO contained in the powder mix not alloyed to form complex compounds.

CaO _{ALLOYED REAGENT}: is the part of CaO that, although is alloyed to form compounds, can react in the EAF.

In order to find the quantity of free CaO, the formation of compounds in powder mix is simulated basing theories about interaction energy between cation and anion and oxygen potential.

The theoretical approach, that is essential to achieve results of complete validity, applicable not only at specific case, is managed evaluating the chemical reactions that can take place at specific temperature and with the elements contained in the powder mix.

For a qualitative idea about which reactions can take place easily into melt mass, are analysed the free energy values. In fact, the free energy variation measures the tendency of isothermal and isobaric process to develop spontaneously providing work. In fact, we can write:

$$W = (\Delta H - T\Delta S) = \Delta G$$

The calculation of free energy reaction variation is managed utilizing the ΔG of compounds formation.

$$\Delta G_{\text{reaction}} = \Delta G_{\text{products}} - \Delta G_{\text{reagents}}$$

Through use of free energy and its variation in a chemical reaction at constant temperature it's possible to determine the reaction equilibrium. In fact, we can calculate the equilibrium constant K according to:

$$lnK(T) = \frac{-\Delta G}{RT}$$
$$K(T) = e^{\frac{-\Delta G}{RT}}$$

The equilibrium constant is, for definition, the ratio between activity of products and reagents, elevated at own stoichiometric coefficient:

$$K = \frac{\prod_{i=1}^{n} a_{products}^{x}}{\prod_{i=1}^{n} a_{reagents}^{y}}$$

In order to understand how the reactions proceed into melt slag, it's useful to calculate the difference between oxygen potentials concerned oxygen activity of slag and oxygen activity of examined reaction. The oxygen activity indicates the oxygen quantity available in order to the reactions take place. The oxygen activity referred to every reaction represent the oxygen quantity necessary to take place the particular reaction. The greater is the difference between oxygen potential, the better is the realization of reaction.

$$\Delta \mu_{O2} = \mu_{O_2} - \mu'_{O_2} = RT \ln(a_O) - RT \ln(a_{O'})$$

The calculation of reactions oxygen activity is obtainable by thermodynamic equilibrium equations, knowing elements activities, equilibrium constants and imposing to products activities a unitary value.

ELEMENTS ACTIVITIES

The slag, in accordance with slag theories, is an ionic solution of cations and oxygen anions.

In case of a regular solution, the activity coefficient of general component i, in a multicomponents system, can write as:

$$\mathsf{RT} \ln \gamma_i = \sum_j \alpha_{ij} X_j^2 + \sum_j \sum_k (\alpha_{ij} + \alpha_{ik} - \alpha_{jk}) \cdot X_j \cdot X_k$$
 with: $(i \neq j \neq k)$

Where Xi is the cation fraction and α_{ij} is the energy of interaction between cations.

	Ca ²⁺	Mg ²⁺	Al ³⁺	Mn ²⁺	Si ⁴⁺	P⁵⁺	Fe ²⁺	Fe ³⁺
Ca ²⁺		-100,42	-154,81	-92,05	-133,89	-251,04	-31,38	-95,81
Mg ²⁺	-100,42		-71,13	61,92	-66,94	-37,66	33,47	-2,93
Al ³⁺	-154,81	-71,13		-83,68	-127,61	-261,5	-41	-161,08
Mn ²⁺	-92,05	61,92	-83,68		-75,31	-84,94	7,11	-56,48
Si ⁴⁺	-133,89	-66,94	-127,61	-75,31		83,68	-41,84	32,64
P ⁵⁺	-251,04	-37,66	-261,5	-84,94	83,68		-31,38	14,64
Fe ²⁺	-31,38	33,47	-41	7,11	-41,84	-31,38		-18,66
Fe ³⁺	-95.81	-2.93	-161.08	-56.48	32.64	14.64	-18.66	

Through following expression:

$$\gamma_i = e^{RT \ln \gamma_i}$$

It's possible to obtain the coefficient values of every component. Known the coefficients it's possible to find the activities only multiplying the coefficients.

Known the coefficients it's possible to find the activities only multiplying the coefficient whit cations concentrations:

$$\mathbf{a}_{i} = \gamma_{i} \cdot \mathbf{X}_{i}$$

where Xi is cationic fraction that is calculable by slag chemical analysis.

OXYGEN ACTIVITY

The calculation of oxygen activity contained in the slag is very important in the steelmaking process. In fact, generally, all process characterized by oxidation-reduction reactions, are regulated by oxygen activity. FeO activity is main factor that influences oxygen potential of slag. Considering FeO formation reaction:

$$Fe + \frac{1}{2}O_2 = FeO$$

we can calculate oxygen activity:

$$\mathbf{a}_{o} = \frac{\mathbf{a}_{FeO}}{\mathbf{K}_{Fe-FeO} \cdot \mathbf{a}_{Fe}}$$

where Fe activity is considered unitary.

K is the equilibrium constant of FeO reaction and can calculated by formation energy:

$$K(T) = e^{\left(\frac{-\Delta G(T)_{reazione}}{RT}\right)}$$

where ΔG of reaction is calculated by:

$$\Delta G(T)_{reaction} = G(T)_{products formation} - G(T)_{reagents formation}$$

FeO activity is calculated by following expression:

$$a_{\text{FeO}} = 2,308 \cdot X_{\text{FeO}}$$

where X_{FeO} is FeO molar fraction.

All terms of expression are known and we can calculate oxygen activity of slag.

Now, after we calculated elements and oxygen activity it's possible to calculate for every reaction the part of total energy that it is the work that the system can develop. Through this percentage fraction we can calculate also the percentage of moles of every compound.

If the quantities of reagents aren't enough, the real quantity of products is which is obtained by the saturation of limiting chemical species.

The calculation of CaO_{ALLOYED REAGENT} is conducted, in first approximation, considering following reaction in the EAF:

 $2(3CaO.SiO_2)_{mix} + SiO_2 (EAF)^{=}$ $2(2CaO.SiO_2) + 2CaO + SiO_2 = 3(2CaO.SiO_2)$

Through simulation of compounds formation in EAF slag it's calculated quantity of CaO necessary at saturation of SiO₂ and Al₂O₃. This simulation is the same for LF slag; the differences are about chemical composition, especially FeO content, and consequently about compounds formation.

Part of CaO necessary is charged with CaO active contained in powder mix, while residual part should be charged through lime and dololime.

The simulation model is able to:

- Simulate compounds formation in LF slag from chemical composition of material.
- Simulate feasibility of powder mix recycling in EAF and estimate the quantity of lime which is possible to save.

EXPERIMENTAL ANALYSES

Some analyses of powder and not powder samples of LF slag at Stefana SpA have been done, with collaboration of Politecnico di Milano University, to better understand chemical composition of LF slag.

Two types of samples during a significant number of heats have been taken. The first sample has been taken directly in ladle, on melt slag during the temperature measure; the second has been taken on cold slag after ladle empting. Every sample have been sifted to divide the fine part from the gross part. On every sample have been done three analyses: morphologic, chemical and about compounds.

Morphologic analysis

Following images show morphologic differences between powder part (pictures (a)) and not powder part (pictures (b)) of samples analysed with SEM.



Figure 6. Powder X-ray diffraction (XRD) analysis of ladle slag fines showing the ladle slag – samples of Stefana – powder fraction, analyzed by the Politecnico di Milano, Mechanical Dept.

Chemical analysis

The chemical analyses are done with SEM. In the following table the average analysis of powder, not powder and mixed samples are showed.

	powder	mixed	not powder
CaO	50,8%	46,9%	50,2%
SiO2	18,5%	20,8%	19,8%
AI2O3	13,6%	17,2%	17,5%
MgO	3,5%	4,2%	4,0%

Compounds analysis:

It is used a probe, EBSD, assembled on SEM, to analyse compounds in samples. The average results obtained for analysed samples are showed in below table:

compounds analysis	%
3CaO.AI2O3	34%
CaO.Al2O3.2SiO2	24%
3CaO.SiO2	11%
2CaO.SiO2	8%
2FeO.MgO	1%
None	14%

Comparing the model results (in the table below) with average analysis of compounds in slag samples we can to note that they are very similar.

model results	%
3CaO.AI2O3	32%
CaO.Al2O3.2SiO2	17%
3CaO.SiO2	11%
2CaO.SiO2	7%
2FeO.MgO	1%
2MgO.SiO2	6%

RESULTS

In the case of Ferriere Nord different recycling rates have been used, depending on the steel grade. For reinforced concrete steel – the 70 percent of plant production – the recycling rate has been fixed to 1800 kg per heat, while for the other steel grades the recycling rate was dropped to 1200 kg per heat. In this way Ferriere Nord has been able, since January 2003 to recycle the 100 percent of the indicated by-products. The injection of this amount of recycled material has allowed the plant to reduce the normal lime in charge of about 1000 to 1200 kg per heat, saving about the 30 percent of the cost of the lime, over the benefit arising from the non-disposal of the indicated wastes.

The metallurgical simulation with the data of Stefana indicates that it is possible to recycle with a rate of about 1600 kg per heat the powder mix into the EAF slag, saving at least 800 kg of lime per heat, which means a 15 percent of the lime cost, over the benefit arising from the non-disposal of the indicated wastes (for the case of Stefana the complete list of wastes is recycled, so a higher benefit is arising from the non-disposal). The simulation model for Stefana indicates that the total investment cost will be paid back in less than three years.

Ferriere Nord has demonstrated during last years the industrial sustainability of the ladle slag recycling. Plant environment has been improved, not jeopardizing the profitability of the steel production. This Company has clearly marked a milestone for the European Steel Industry: the future of the so-called "electrical steel" must consider as compulsory the reduction of plant wastes, which are an internal resource to save variable production costs.

Stefana has introduced the "full-recycling concept" for the wastes of the liquid steel production.

This plant will have the possibility to reduce the outputs of the liquid steel production to three items: liquid steel / EAF Slag / Dedusting System fines.

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