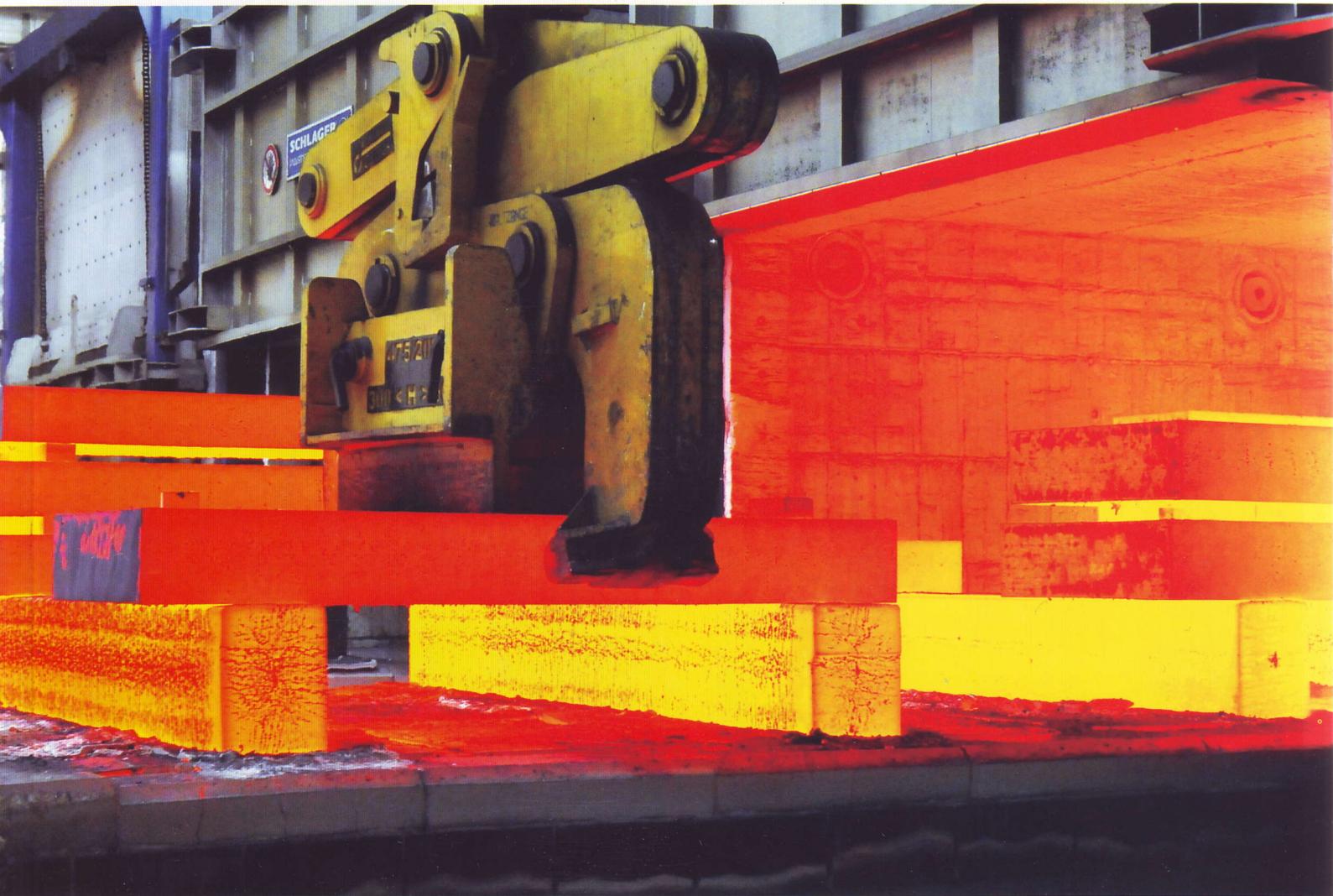


METALL

Fachzeitschrift für Metallurgie
Technik · Wissenschaft · Wirtschaft



Special: Edelstahl

Höherfeste Stahllösungen

Dusts from stainless steel production

Strahlen von Kleinteilen

Stahlmarkt 2013: Nachfragerückgang

In der Diskussion: KrWG

www.metall-web.de

Einzel-Verkaufspreis: 21,00 €

GDMB Verlag GmbH
Paul-Ernst-Straße 10
D-38678 Clausthal-Zellerfeld
PVST · Dt. Post AG · Entgelt bezahlt

 **GDMB**
Verlag GmbH

67. Jahrgang · April 2013

GDMB Verlag GmbH

ISSN 0026-0746

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Characterization of dusts from three types of stainless steel production equipment

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Steelmaking dusts, by-product in steel production process, are considered as hazardous wastes from steel production. On the other hand, these dusts contain significant amount of valuable metals, which could be recovered. Because of its large heterogeneity and anisotropy it is difficult to design a suitable process for its recycling. The first and the most important step for designing an efficient recycling process is thorough material characterization. The aim of this work was to characterize five samples of steelmaking dusts coming from stainless steel production. In particular, two sample of electric furnace (EAF) dust, two samples of argon oxygen decarburization (AOD) converter dust and one sample of ferro-chrome converter (CrK) dust were characterized. Chemical analysis was carried out by Atomic absorption spectroscopy (AAS). For a structural characterization of the samples, the X-ray diffraction phase analysis (XRD) was used. Morphological analysis was carried out by optical microscopy and scanning electron microscopy with semi-quantitative analysis (SEM-EDX) of dust particles. All five samples were characteristic by high content of Zn, Cr, Fe, Ni and Ca. The most common phases identified in the samples were: FeCr_2O_4 , ZnFe_2O_4 , CaO and ZnO. All five samples had 100% of the particles under 55 μm .

Steelmaking dust is generated as a by-product from steelmaking processes in amount around 10 to 20 kg per ton of produced steel [1]. Due to its chemical and physical properties, steelmaking dust is categorized, according to the US EPA classification, as a hazardous waste [2].

The increasing demand for metals has stimulated the development of new technologies worldwide to treat secondary resources like steelmaking dusts, which can present risks to the environment if managed in an incorrect way [3].

The methods for steelmaking dust processing can be divided into following categories: pyrometallurgical, hydrometallurgical and electrometallurgical processes, or their combination. Hydrometallurgical processing appears to be more perspective in the future mainly from the environmental and economic point of view [4, 5].

One of the major problems regarding steelmaking dusts processing is their heterogeneity in chemical and mineralogical composition. For this reason it is difficult to design "versatile" technology for their processing and every technology must be adjusted to the certain steelmaking dust.

For this reason, the chemical and structural characterization of the material is a very important stage to evaluate the recycling feasibility [6].

Both chemical and mineralogical compositions depend on:

- Steelmaking process – Electric arc furnace (EAF), Argon oxygen decarburization converter (AOD), Ferro-chrome converter, Basic oxygen furnace (BOF),
- The chemical composition of a raw material – steel scrap (almost 100% in EAF)
- Type of produced steel – carbon steel (high Zn content) [7] or stainless steel (high Cr and Ni content) [1].

There are several works [1, 6, 8, 9] concerning with characterization of EAF (electric arc furnace) dust from carbon steel production but only very few papers [1, 3] concerned with steelmaking dust from stainless steel production. This work is intended for filling a gap in the area of characterization steelmaking dust from stainless steel production. Dusts from carbon steel and stainless steel production seem to be very similar at the first sight but they differ significantly. The main difference is in their chemical composition. In Table 1 can be seen the comparison in chemical composition between dust formed during carbon steel production and the one formed in stainless steel production.

As shown by Table 1 the biggest difference between dusts coming from stainless steel and carbon steel production is in zinc content, which is more than three times higher in case of carbon steel production dust. On the other hand, dust coming from stainless steel production contains other valuable metals like Cr, Ni and Mn in significant amounts. These facts

	EAF (stainless steel production)	EAF (carbon steel production)
Compound/Element	[w. %]	[w. %]
CaO	6.59	-
SiO ₂	5.76	-
MgO	4.25	-
MnO	5.88	-
Fe ₂ O ₃	39.56	-
Fe	-	27.23
Cr	10.9	-
Ni	4.1	0
Pb	1.4	1.28
Zn	5.2	17.05
Ca	-	4.42
Cd	-	0.09
Mn	-	1.03

Table 1: Chemical composition of EAF dusts from stainless steel and carbon steel production [1, 10]

Sample	Labeling
Electric furnace dust from production line 1	EAF1
Electric furnace dust from production line 2	EAF2
Argon oxygen decarburization converter dust from production line 1	AOD1
Argon oxygen decarburization converter dust from production line 2	AOD2
Ferro-chrome converter	CrK

Table 2: Labeling of five different steelmaking dusts from stainless steel production

Sample	content [%]									
	Zn	Fe	Ni	Cr	Mn	Pb	Cd	Ca	Mo*	LOI
EAF1	8.10	20.23	1.45	18.16	1.67	0.46	0.48	10.84	0.07	0.67
EAF2	6.40	15.44	2.40	17.19	1.61	0.88	0.38	15.10	0.51	2.93
AOD1	9.75	29.20	0.67	18.56	1.35	0.09	0.19	5.50	0.11	1.35
AOD2	5.20	18.75	2.70	13.39	1.68	0.39	0.15	17.20	1.25	1.30
CrK	7.35	13.94	0.12	22.08	0.24	0.11	0.12	17.40	0.01	2.18

Table 3: Chemical composition of the samples

must be taken into account when designing recycling technology, which must be adopted, to some extent, to the nature of an input material. Therefore, a thorough characterization of the dust is necessary. The aim of this paper is to characterize five samples of steelmaking dust from stainless steel production. Samples of steelmaking dust came from three different steelmaking equipment. The characterization is carried out through chemical, mineralogical, morphological and granulometric analysis.

Experimental

Five samples of steelmaking dust produced in Outokumpu Stainless (Tornio, Finland) were analyzed. Description and labeling of the samples are shown in Table 2. These samples were chosen mainly because they represent dusts coming from three the most essential equipment used in stainless steel production.

Methods

Chemical analysis

All five samples were submitted to the chemical analysis by using method AAS on atomic absorption spectrophotometer Varian Spectrophotometer AA20+.

Mineralogical analysis

Mineralogical analysis was carried out through X-Ray diffraction qualitative phase (XRD) analysis on the PANalytical X'Pert PRO MRD X-ray diffractometer using Co K α radiation.

Optical microscopy

The samples were introduced to observation by optical microscopy using Digital microscope MZK 1701. Magnification of 200x was used during the observation. No optical filters were used. These observations allowed not only the appreciation of macromorphology of the samples, but also the color resolution, what is impossible by using SEM microscopy.

Granulometry analysis

Granulometry was determined by Scanning-foto-sedimentograph, Fritsch - GmbH, Analysette.

Scanning electron microscopy - SEM - EDX

Both of morphology of the samples and chemical microanalysis were carried out on scanning electron microscope JEOL JSM-35CF with EDX analyzer LINK ANALYTICAL AN 10/85S.

This observation serves to a closer investigation of chemical composition individual particles and areas in the samples. During the observation, several areas were chosen for chemical analysis in order

to find out the distribution of elements in the samples.

Results and discussion

Chemical analysis

The results of the analysis are documented in Table 3.

It results from the Table 3, that these dusts are typical by a high content of chromium (13 – 23 %), zinc (5 – 10 %) and iron (13 – 30 %). The source of chromium in dusts from stainless steel production is ferro-chrome used as an alloy and zinc in dusts originates from the galvanized iron scrap [7].

In samples EAF2 and AOD2 also Ni content was considerably high when comparing to primary ores where the average Ni content is around 1.45 % [11].

These results show that besides considering these dusts as a hazardous waste they might be also valuable secondary raw materials for chromium, nickel and/or zinc production. However, when designing a recycling process it is necessary to take into account also mineralogical form of the metals.

Mineralogical analysis

The main phases identified in each sample are given in Table 4. Evaluating the results of XRD analysis was carried out using software HighScore.

As it results from the XRD analysis, all five samples are very similar from point of view mineralogical composition. The main phases present in almost every sample are:

- oxides – CaO, ZnO and Fe₂O₃
- ferrites – FeCr₂O₄, ZnFe₂O₄, and Ni_{0.25}Fe_{0.75}Fe₂O₄

From a hydrometallurgical point of view it is very important to know the mineralogical form of the metals in order to set up optimal leaching conditions. As can be seen in Table 4, zinc is present mainly as ZnO and/or ZnFe₂O₄, which is very resistant to any kind of hydrometallurgical treatment.

Sample				
EAF1	EAF2	AOD1	AOD2	CrK
FeCr ₂ O ₄	FeCr ₂ O ₄	FeCr ₂ O ₄	FeCr ₂ O ₄	FeCr ₂ O ₄
ZnFe ₂ O ₄	ZnFe ₂ O ₄	ZnFe ₂ O ₄	ZnFe ₂ O ₄	ZnFe ₂ O ₄
Ni _{0.25} Fe _{0.75} Fe ₂ O ₄	NiFe ₂ O ₄	ZnCr ₂ O ₄	Ni _{0.25} Fe _{0.75} Fe ₂ O ₄	ZnFeCrO ₄
ZnO	CaO	Fe ₃ O ₄	ZnO	Cr ₂ O ₃
CaO	CaCO ₃	Fe ₂ O ₃	CaCO ₃	Fe ₂ O ₃
	MnO ₂	ZnO	Ca(OH) ₂	ZnO
				CaO

Table 4: Main phases identified in each sample

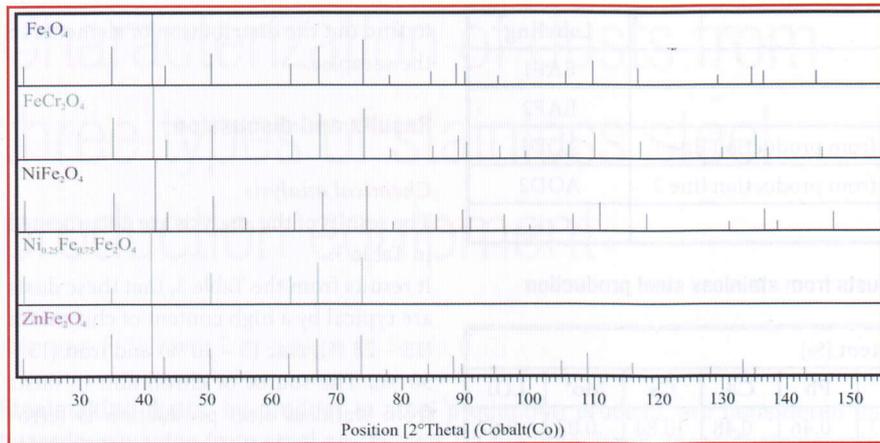


Fig. 1: XRD patterns of five main ferrites present in the samples

Nickel is present as a spinel compound $(NiFe)Fe_2O_4$. Chromium is present in all samples mainly as chromite, $FeCr_2O_4$. The biggest problem regarding XRD analysis of this kind of material is that almost all metals in these samples are present as ferrites, whose XRD patterns are very similar what makes their identification

very difficult. In Fig. 1 are shown theoretical peak positions in XRD pattern of five main ferrites present in the samples: Fe_3O_4 , $FeCr_2O_4$, $NiFe_2O_4$, $Ni_{0.25}Fe_{0.75}Fe_2O_4$ and $ZnFe_2O_4$. As can be seen from the figure above the peaks positions of the ferrites in XRD pattern are very similar and it is very diffi-

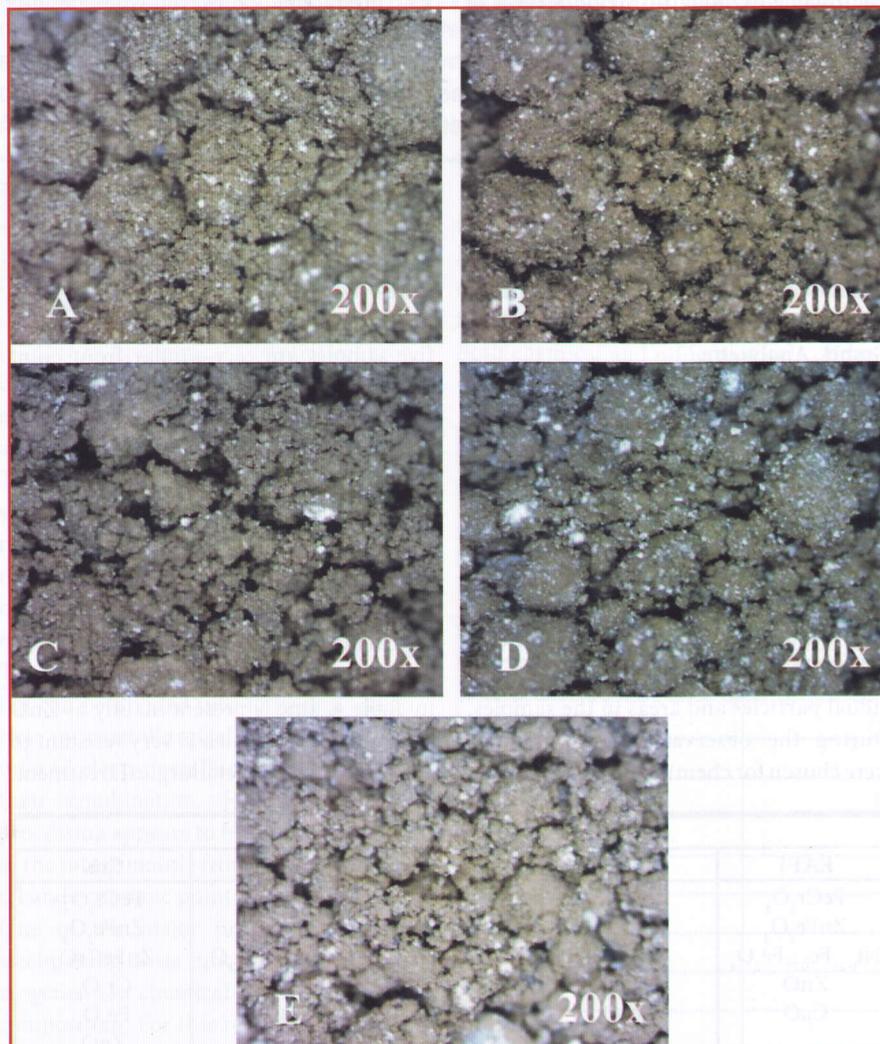


Fig. 2 Morphology of five different dusts from stainless steel production: a) EAF1; b) EAF2; c) AOD1; d) AOD2; e) CrK

cult to distinguish what kind of ferrite is present in the sample. In addition, ferrites present in the real sample very often do not have ideal structure and therefore their peaks can be shifted from the theoretical position.

Others work [3] concerning with characterization of AOD converter dust from stainless steel production also confirms the presence of phases such as: chromite $FeCr_2O_4$, magnetite Fe_3O_4 , hematite Fe_2O_3 and calcite $CaCO_3$.

The results of the XRD analysis show that zinc, iron and chromium are mainly present as ferrites which are very resistant to any kind of treatment. Because of that, it is necessary to use more aggressive conditions (high temperature or stronger leaching reagent) for metals recovery.

Optical microscopy

Results of optical microscopy observation are listed in Fig. 2.

As can be seen from Fig. 2, particles in all five samples have predominantly spherical shape where bigger particles are covered with the smaller ones. The presence of two fractions was also confirmed by granulometry analysis. It is possible to observe small white spots in all samples, which represent CaO , whose presence was confirmed by XRD analysis. The smallest amount of white spots was observed in the sample AOD1 where the content of calcium comparing with the other samples was the lowest. On the other hand the highest amount of white particles was observed in samples AOD2 and CrK where the calcium content was higher than 17 wt.%.

Granulometry analysis

Cumulative and distribution curves of the particle size in individual samples are illustrated in Fig. 3.

The results of granulometric analysis showed that all samples have a very similar granulometry, where 100 % of the particles are below 55 μm . From the distributive curves of all five samples can be clearly seen that there are two major fractions in size range 0 to 22 μm and 28 to 47 μm for the samples EAF1 and EAF2. For the samples AOD1 and AOD2 it was 8 to 22 μm and 28 to 47 μm . For the sample CrK the size ranges were 0 to 22 μm and 27 to 47 μm .

If chemical composition and granulometry analysis are compared a correlation between Ca content and quantity of the size fraction 0 to 22 μm could be found. In the samples where the Ca content was the highest (sam-

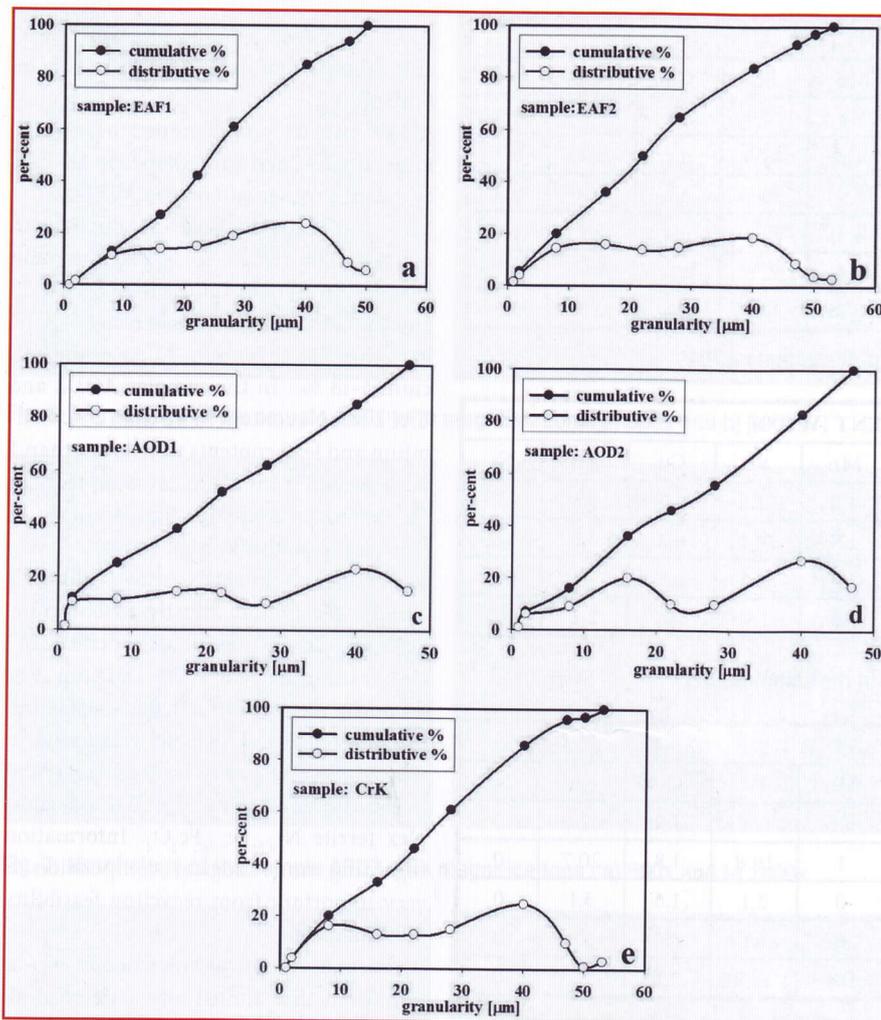


Fig. 3: Granulometry of the samples: Cumulative and distribution curve a) EAF1; b) EAF2; c) AOD1; d) AOD2; e) CrK

AREA	CONTENT [WT. %]									
	Mg	Si	Ca	Cr	Mn	Fe	Cu	Zn	Ni	
EAF1-1	5	7.3	15.7	9.7	5.5	29.2	2.5	17	2	
EAF1-2	1.2	1.4	1.5	5.7	0.4	84.8	1.4	2.5	0.3	
EAF1-3	2.9	19.8	17.7	4.8	6.8	29.2	1.9	12.2	0.2	
EAF1-4	0.9	9.5	35.3	4.6	2.3	38.5	1.9	5.4	0.2	
EAF1-5	3.3	4	2	12	3.2	53.2	2.2	17.7	0.8	

Table 5: Chemical composition of certain areas in the sample EAF1

AREA	CONTENT [WT. %]									
	Mg	Si	Ca	Cr	Mn	Fe	Cu	Zn	Ni	
EAF2-1	1.9	6.2	29.2	12.9	3.9	25.7	3	9.4	2.8	
EAF2-2	1.3	6.1	2.8	8.9	3.2	62	2.4	5.6	4.5	
EAF2-3	12	39.3	28.5	0.9	0.5	13.4	1.4	1.5	0.2	
EAF2-4	3.3	16.3	25.9	5.7	9	22.8	2.5	4.1	3.1	
EAF2-5	1.3	8	10.8	8.9	2.7	39	3.4	8.4	13.6	
EAF2-6	1.7	5.1	46.5	9.4	2.5	18.1	2.3	6.8	2.1	

Table 6: Chemical composition of certain areas in the sample EAF2

ples EAF2, AOD2, CrK, - over 15%) the size fraction 0 to 22 µm was clearly visible in dis-

tributive curves (Fig. 3b,d,e). On the other side in samples where the Ca content was the

lowest (EAF1 and AOD1) the size fraction 0 to 22 µm was hardly visible in distributive curves (Fig. 3a,c). From these results, it concludes that Ca (white spots in Fig. 2) is represented mostly by the size fraction 0 to 22 µm.

These results of granulometric analysis are important in designing conditions for hydrometallurgical treatment.

Scanning electron microscopy - SEM - EDX

Fig. 4 represents the sample EAF1 where the chosen areas contain mainly Fe, Ca and Zn (Table 5). In the area of EAF1-3 was also relatively high content of Si (19.8 %), what can indicate a local presence of Si phases.

The areas in the sample EAF2 (Fig. 5) are by their chemical composition similar to the areas in the sample EAF1. However, this sample is characteristic by significantly higher content of Ca (area EAF2-6), what is also confirmed by the chemical analysis of the sample listed in Table 1. According to the stoichiometry composition, Ca present in the area EAF2-6 can be present as Ca(OH)₂, which was also found by the XRD analysis (Table 4). One area with high content of Si (area EAF2-3) was also present in the sample.

The areas in the sample AOD1 (Fig. 6 and Table 7) are characteristic by a high content of Fe (in the area AOD1-1 over 90 %), Cr (over 20 % in the area AOD1-5) and Ca (36.6 % in the area AOD1-6).

The highest content of Ca was found in the areas of the sample AOD2 (Fig. 7 and Table 8) that correspond with the chemical composition in the Table 3. The highest content 93.6 % of Ca was found in the area AOD2-4. Other significant metals in the sample AOD2 were Fe, Cr and Zn.

Particle CrK-1 in the sample CrK (Fig. 8 and Table 9) contains almost 43 % of Zn and area of the particle CrK-5 more than 43 % of Zn. It results from the Table 9 that both particles have very similar chemical composition. Area CrK-4 contains 64 % of iron what is value close to the stoichiometric amount of Fe in Fe₂O₃. This iron oxide was identified also by XRD analysis. Area CrK-3 is typical by the highest amount of Ca. However, this high amount of Ca was expected as the CrK sample, according to Table 3, has the highest content of Ca from all five samples.

Conclusion

Five samples of steelmaking dusts from stainless steel production were investigat-

AREA	CONTENT [WT. %]								
	Mg	Si	Ca	Cr	Mn	Fe	Cu	Zn	Ni
AOD1-1	0	0.7	0.4	2.7	0.9	90.4	n.a	1.6	0.2
AOD1-2	1.7	1.4	9	12.2	4.2	48.5	n.a	2.1	0.7
AOD1-3	1.5	0.9	24	10.2	3.9	39.5	n.a	2.8	0.5
AOD1-4	2	1.7	2.8	12.2	4.4	50.7	n.a	3.1	1.1
AOD1-5	1.3	1	1.2	20.5	5.4	62.6	n.a	2.1	1.2
AOD1-6	1	1.2	36.6	9	3.7	33.7	n.a	2.1	0.5

Table 7: Chemical composition of certain areas in the sample AOD1

AREA	CONTENT [WT. %]								
	Mg	Si	Ca	Cr	Mn	Fe	Cu	Zn	Ni
AOD2-1	2.6	1.3	33.7	12	4.8	31.5	n.a	9.5	3
AOD2-2	0.6	0.5	13.8	3.7	1.5	73.1	n.a	3.9	2.2
AOD2-3	1.2	0.5	37.1	11.7	6.8	30.1	n.a	7.6	2.8
AOD2-4	0.5	0.5	93.6	0.7	0.3	1.9	n.a	2.1	0

Table 8: Chemical composition of certain areas in the sample AOD1

AREA	CONTENT [WT. %]								
	Mg	Si	Ca	Cr	Mn	Fe	Cu	Zn	Ni
CrK-1	2.5	8.6	12.5	12.7	0.5	17.4	1.9	42.9	0
CrK-2	4.6	3.1	30.9	20.3	1	16.8	1.8	20.7	0
CrK-3	1.2	0.9	88.7	2	0	2.1	1.6	3.1	0
CrK-4	0.7	0.5	7.3	19.3	0	64	3.4	3.9	0
CrK-5	1.2	3.5	10.1	19.7	0.4	18.5	2.8	43.3	0

Table 9: Chemical composition of certain areas in the sample CrK

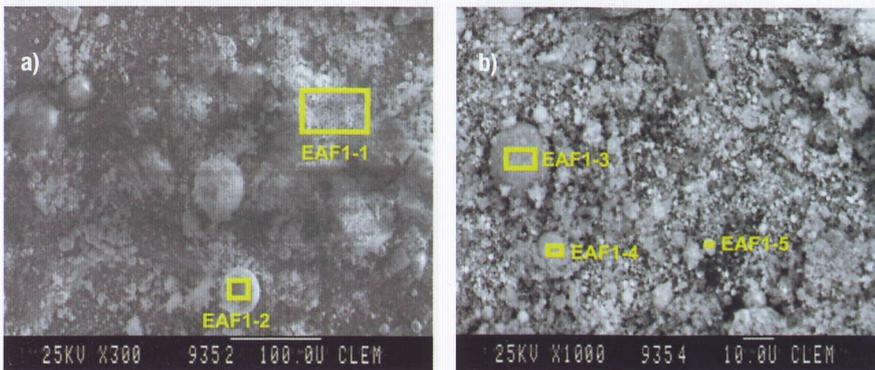


Fig. 4: Morphology of the sample EAF1 with magnifications: a) 300x and b) 1000x

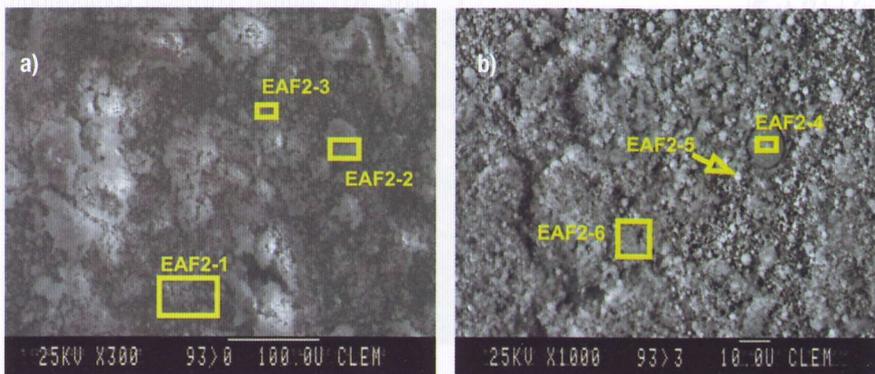


Fig. 5: Morphology of the sample EAF2 with magnifications: a) 300x and b) 1000x

ed in this paper. The samples were delivered from Outokumpu Stainless (Tornio, Finland).

All five samples were submitted to chemical, mineralogical and granulometric analysis as well as to observation by SEM-EDX.

Chemical analysis was carried out by AAS method. The results show that the main elements in all five samples were Fe, Cr, Zn and Ca. Content of Zn was in range 5-10 %, iron 13-30 %, chromium 13-23 % and calcium 5-18 %. In the samples AOD2 and EAF2 was Ni content more than 2 %. Cadmium and lead contents were lower than 1 %. It results from chemical analysis than Cr, Fe and Zn content is significant for its recovery from this kind of waste.

Mineralogical analysis shows that Cr and Fe are present mostly as chromite, $FeCr_2O_4$. Zn is present as zincite, ZnO and franklinite, $ZnFe_2O_4$. Calcium was found in the form of calcite, $CaCO_3$ and calcium oxide CaO. Nickel was found in the sample EAF2 as a ferrite $NiFe_2O_4$ and in the samples EAF1 and AOD2 as a complex ferrite $Ni_{0.25}Fe_{0.75}Fe_2O_4$. Information regarding mineralogical composition is very important from recycling feasibility point of view.

By means of optical microscopy it was found out that almost all particles in all five samples have spherical shape. Also it was observed that bigger brown particles (consisted mainly of Fe, Zn, Cr) are covered with smaller white particles (consisted of Ca). The size range of the bigger particles was from 27 μm to 47 μm and size range for the smaller particles was 0 to 22 μm . Granulometry of all five samples was very similar where 100 % of the particles were below 55 μm .

SEM-EDX confirmed the spherical shape of most particles in all samples. This observation shows that selected particles consist of more than only one phase. The main elements in most spherical particles were Fe, Ca and Zn. In the sample EAF2, there was also a small particle (EAF2-5 in Fig. 5) with Ni content 13.6 %. Small spherical particles in the sample CrK (CrK-1 and CrK-4 in Fig. 8) contain more than 42 % of Zn. Rectangular particle in the sample AOD2 (AOD2-4 in Fig.7) contains 93.6 % Ca.

Results of the characterization showed that steelmaking dusts coming from stainless steel production could be used as secondary raw materials for production of metals like Cr, Ni and Zn as their contents in these dusts are comparable or even higher than in primary ores.

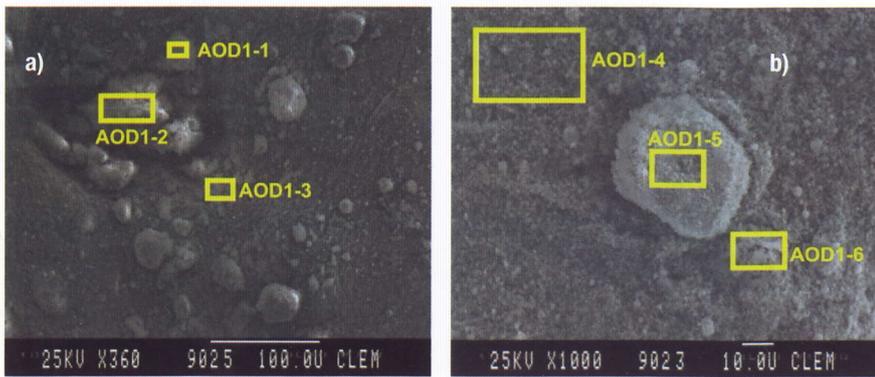


Fig. 6: Morphology of the sample AOD1 with magnifications: a) 360x and b) 1000x

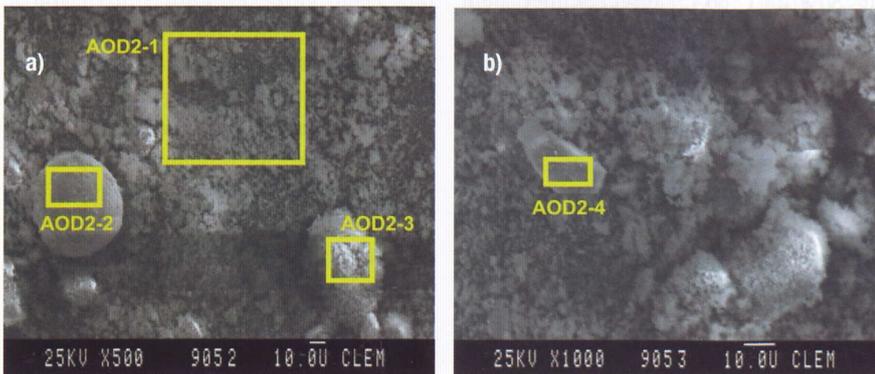


Fig. 7: Morphology of the sample AOD2 with magnifications : a) 500x and b) 1000x

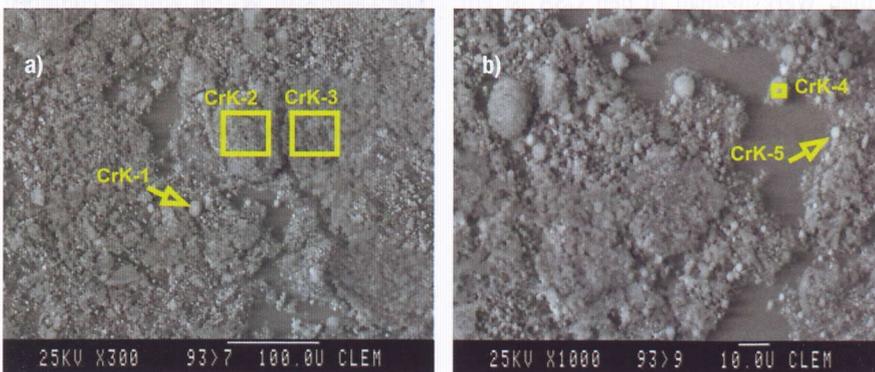


Fig. 8: Morphology of the sample CrK with magnifications : a) 300x and b) 1000x

Acknowledgements

This work was supported by Ministry of Education of the Slovak republic under grant VEGA MŠ SR 1/0123/11 and project No. APVV-20-013405. This contribution is also the result of the project implementa-

tion Research excellence centre on earth sources, extraction and treatment supported by the Research & Development Operational Programme funded by the ERDF, ITMS number: 26220120017. This work has been done in the METDUST project of the ELEMET research program

funded by FIMECC Oy. The financial support of TEKES, Outokumpu Oyj, Outotek Oyj and New Boliden Oyj (Kokkola) is gratefully acknowledged.

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Netzentgeltbefreiung sichert Stromnetz und Wettbewerbsfähigkeit

7. März 2013 – Die Entscheidung des Oberlandesgerichts Düsseldorf zur Netzentgeltbefreiung zwingt, so die Wirtschaftsvereinigung Metalle (WVM), den Gesetzgeber, handwerkliche Fehler in der Gesetzgebung zu bereinigen. Hier sei dringend eine Kor-

rektur formaler Fehler notwendig, die nicht zu Lasten der Industrie gehen dürfe. Der Beitrag der Industrie für die Stabilität der Stromnetze müsse wieder ins Gesetz aufgenommen werden, forderte WVM-Präsident Oliver Bell. Er begrüßte, dass die Bundes-

regierung bereits eine neue Regelung vorbereite. Die zuvor rechtlich einwandfreie Regelung zeigte, dass die Netz stabilisierende Leistung des industriellen Stromverbrauchs zum Gelingen der Energiewende beitrage.