Researches Regarding the Reduction of Oxidation Losses in the Obtaining of Secondary Aluminium Alloys Using Metallic Scraps

CONSTANTIN APOSTOL[1], MIHAI BUTU[1]*, CRISTIAN DOBRESCU[1], ALEXANDRA TACHE[2]
[1]Politehnica University of Bucharest, 313 Splaiul Independentei, 060042, Bucharest, Romania
[2]S.C. AS METAL S.R.L, 104G Berceni Road, 041919, Bucharest, Romania

The paper presents thermodynamic aspects regarding the low temperature processing of small metal waste - aluminum alloy scraps. The activation energies of oxidation reactions of aluminum and some alloying elements with water vapors, carbon dioxide and oxygen present inside the furnace are compared, at the usual and experimentally temperatures (at 580 – 620°C, near the eutectic temperature, when the melt is in the semisolid/pasty state), in the elaborating of EN AB 46100 secondary alloys. The composition of the flue gases is presented at different temperatures and during different technological stages. Using electronic microscopy and EDS analysis, the elemental composition of the impurities resulted from the melt is highlighted.

Keywords: metallic scraps, secondary Al alloys, activation energy, oxidation

Conventional recycling techniques generate hazardous residue that usually requires disposal at a high cost. Aluminium slags contain $MgAl_2O_4$, $Al_2O_3{\cdot}FeO$, calcium oxides, AlN, quartz (SiO_2), α-Al_2O_3, periclase (MgO), spinel (Mg, Si)Al_2O_4, chlorides ($AlCl_3$, NaCl, KCl), fluorides (CaF_2, NaF, AlF_3, Na_3AlF_6 etc.), carbides (Al_4C_3), sulphides (Al_2S_3, $FeSO_3$), phosphors, impurities, apart from metallic aluminium (between 80% and 20%). By washing them with hot water the AlN compound can be transformed into $Al(OH)_3$, which, in reaction with Na and K leads to the removal of chlorides from waste [1, 3, 8, 10, 13 -15].

By processing aluminium and aluminium alloy scraps with the usual techniques, the loss of aluminium can easily reach 50%, traditional recovery procedures becoming ineffective. In paper [2] studies are presented regarding a new aluminium recovery process by treating the black slag, (ABD - Al black dross), a by-product formed during aluminium scraps melting.

Six major phases have been identified: spinel ($MgAl_2O_4$), aluminium nitride (AlN), diaoyudaoite ($NaAl_{11}O_{17}$), aluminium oxynitride (Al_5O_6N), halite (NaCl) and corundum (Al_2O_3). In addition, hibonite compounds ($CaAl_{12}O_{19}$), calcium fluoride (CaF_2), calcite ($CaCO_3$) and sylvite (KCl), are also present as minor constituents. NaCl, KCl and CaF_2 are compounds from the fluxes used to treat the melt in order to protect the metal from the reactive atmosphere in the furnace.

Corundum comes from the reaction of atmospheric oxygen with molten metallic aluminium at high temperatures. As reaction products, we also encounter nitrides due to the presence of nitrogen in the atmosphere of the furnace and due to the high temperatures at which the processes take place. $MgAl_2O_4$ is the result of oxidation of magnesium in aluminium waste, where it is present as an alloying element. The presence of $CaCO_3$ compound can be explained by the reaction of CaO impurities in the raw material with CO/CO_2.

White slag can be reprocessed for the recovery of mechanically entrained aluminium or in compound form, black slag can be, and must be converted into environmentally friendly commercial products [4]. Slag processing can be done by acid solubilization [12].

H.N. Yoshimura et al. and Apeng Li et al. have studied the possibility of using aluminium white slag to obtain a refractory material - High-alumina refractory (HAR), with the main crystalline phase Al_2O_3 and traces of $MgAl_2O_4$ and $CaAl_2O_4$, by replacing the materials currently used. They can be used in the production of ceramics, cement, in the construction of furnaces used in the metallurgy of non-ferrous metals, the machine building industry and the defence industry, extending also in space and nuclear engineering [5, 6, 11].

The production of η-alumina has also been studied to avoid environmental pollution and to obtain a material with special properties. The main advantages of this new material are corrosion resistance, thermal shock resistance, thermal stability and good refractory properties. Another great advantage is the lack of the need to store it, thus avoiding the pollution of the groundwater [7].

Experimental part

The raw material used in the experiments is aluminium chips, unprocessed, in accordance with national and international standards. This material is classified as conforming SR EN 13920-12 or 13, equivalent to ISRI TEENS (fig. 1) or TELIC (fig. 2) notations and consists of aluminum borings and turnings of one specified alloy (TEENS - the material must be free from oxides, dirt, free iron, stainless steel, magnesium, oils, flammable liquids, moisture and other non-metallic elements), or derived from two or more alloys (TELIC - the material must be clean, uncorroded aluminum borings and turnings of two or more alloys and subject to deductions for fines in excess of 3% through a 20 mesh screen and dirt, free iron, oil, moisture and all other non-metallic items) [18].

This type of material does not always meet moisture or impurity requirements. This paper proposes a high humidity aluminium metallic scraps processing technology, with low values in terms of combustion losses-taking into account the dimensions of the particles.

In this study, it is intended to reintroduce into the industrial circuit small metallic waste (scraps) with high humidity in order to obtain a secondary standardized alloy. The removal of impurities from the melt will be done through fluxing treatment.

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* email: mihaibutu@yahoo.com

Fig. 1. ISRI TEENS

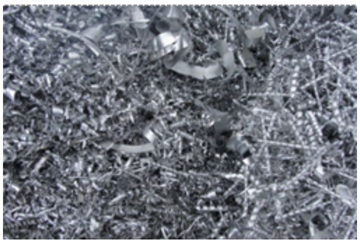
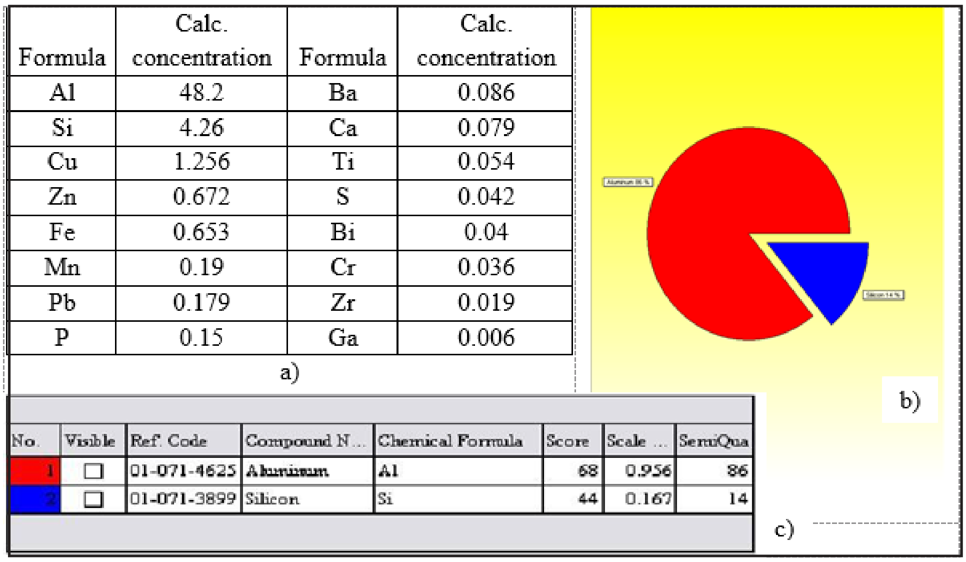


Fig. 2. ISRI TELIC



Formula	Calc. concentration	Formula	Calc. concentration
Al	48.2	Ba	0.086
Si	4.26	Ca	0.079
Cu	1.256	Ti	0.054
Zn	0.672	S	0.042
Fe	0.653	Bi	0.04
Mn	0.19	Cr	0.036
Pb	0.179	Zr	0.019
P	0.15	Ga	0.006

a)

No.	Visible	Ref. Code	Compound N...	Chemical Formula	Score	Scale ...	SemiQua
🟥 1	☐	01-071-4625	Aluminum	Al	68	0.956	86
🟦 2	☐	01-071-3899	Silicon	Si	44	0.167	14

c)

Fig. 3. The aluminium scraps analysis used in experiments a) chemical composition; b) and c) share of the main elements

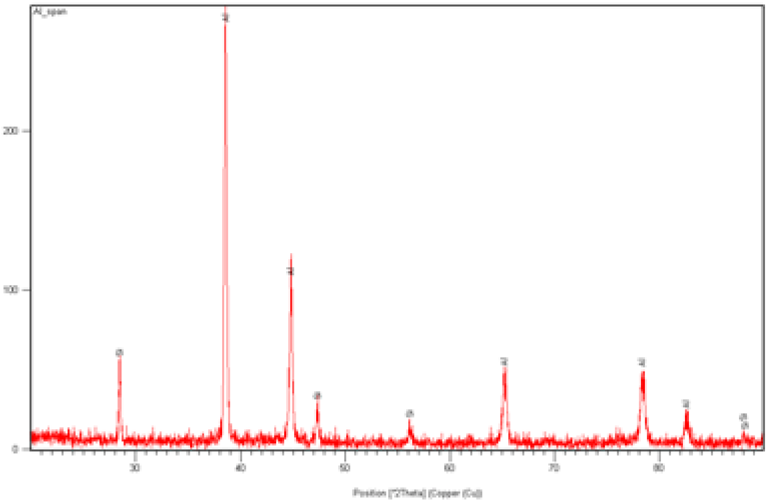


Fig. 4. Diffractogram of high-weight compounds in the analysed chord

The aluminium scraps used in the experiments were analysed by diffraction (fig. 3,4) to highlight its composition. Since the humidity in the material is very high, the results obtained (in percent) are not conclusive.

By combustion of methane gas in room-type furnaces, the flue gases come in direct contact with the raw material for preheating, heating and melting. Following the reactions that take place [16] mainly, the following reaction products are produced: CO, CO_2, H_2 and H_2O. These gases, together with combustion oxygen, interact with the materials that form the charge, leading to the formation of a significant amount of slag, i.e. significant losses through oxidation.

In this article, the authors propose the processing of aluminium alloy castings to obtain low-temperature secondary alloy EN AB 46100 to reduce batch/load oxidation.

The choice of temperature was made based on the data from speciality literature (table 1) [17] but also of its own experiments.

Table 1

EUTECTIC TEMPERATURE OF SOME ALLOYS Al-Si-Cu [17]

Alloy	Si	Cu	Fe	Mg	Zn	Rest, total	Al	T Eutectic, °C
AA 319.0	5.5 – 6.5 %	3.0 – 4.0 %	<= 1.0 %	<= 0.1 %	<= 1,0 %	<= 1.60 %	85.8 – 89.9 %	562.3 (±2,3)
AA 332.0	8.8 – 10.5 %	2.0 – 4.0 %	<= 1.2 %	0,50 – 1.5 %	<= 1,0 %	<= 0.50 %	80.1 – 88.7 %	557.4 (±6)
AA 333.0	8.0 – 10 %	3.0 – 4.0 %	<= 1.0 %	0.05 – 0.50 %	<= 1.0 %	<= 0.50 %	81.8 – 89 %	560.5 (±4)
AA 383.0	9.5 – 11.5 %	2.0 – 3.0 %	<= 1.3 %	<= 0.10 %	<= 3.0 %	<= 0.50 %	79.7 – 88.5 %	566.3 (±2.2)
AA 384.0	10.5 – 12 %	3.0 – 4.5 %	<= 1.3 %	<= 0.10 %	<= 3.0 %	<= 0.50 %	77.3 – 86.5 %	561.3 (±3.2)

Table 2
THE CHEMICAL COMPOSITION OF THE FLUE GASES DURING THE EXPERIMENTS

T-Gas	783.1	574.2	483.2	680.4	580.1	690.7	760.5	787.2	770	821.6	°C
T-Amb	38	32.4	37.6	39.1	37.9	31.4	37.4	38.1	30.9	31.7	°C
O_2	5.7	16.4	20.4	15.1	19.4	18.1	15.1	10.1	21	4.7	%
CO_2	8.9	2.7	0.3	3.4	0.9	1.7	3.4	6.3	0	9.5	%
Losses	37.7	79.1	99.9	75.5	99.9	99.9	85.2	50.7	-	37.8	%
Effic	62.3	20.9	0.1	24.5	0.1	0.1	14.8	49.3	-	62.2	%
Exc.Air	1.37	4.57	35	3.56	13.13	7.24	3.56	1.93	-	1.29	
CO/m^3	2	1265	266	687	923	586	299	656	210	839	mg
NO/m^3	9	3	0	4	0	0	4	5	7	8	mg
NOx/m^3	14	4	0	6	0	0	6	8	10	12	mg
SO_2/m^3	0	2267	206	1896	1973	549	189	369	149	194	mg
Stage	1	2	3	4	5	6	7	8	9	10	

Remarks:

1 - Burners on + closed door before loading;
2 - Burners off + open door + loading;
3 - Burners off + closed door after loading;
4 - Burners on + open door + mixing;
5 - Burners off + closed door after loading;
6 - Burners on + open door + mixing;
7 - Burners on + closed door after loading;
8 - Burners on + open door + mixing after loading;
9 - Burners on + additional oxygen + closed door;
10 - Burners on + closed door.

Based on the data in table 1 but also on our own experiments (EN AB 46100, 10.75% Si, 1.81% Cu, 1.34% Zn, 0.93% Fe, T_{eut} = 568°C) it has been chosen that the working temperature, the temperature of the semisolid/pasty metal material in the furnace, should fall within the limits of 580 ÷ 620°C.

The experiment took place at SC AS Metal SRL, on a 16-tonne furnace equipped with the oxy-fuel system. Flue gas samples were taken at different points in the process with DELTA 2000-IV apparatus, as can be seen from table 2.

Low-temperature processing reduces losses of oxidation for both aluminium and other metallic elements in the scraps composition by incorporating it into the semi-solid/pasty melt. The subsequent overheating of the melt leads to an advanced assimilation of the scraps into the molten metal mass.

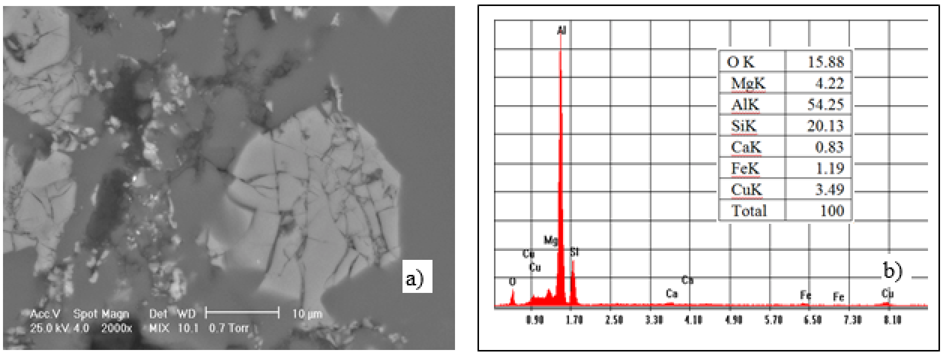
After the loading of each batch, the volatiles and the moisture in the chips/scraps evaporated after the contact with the hot bath and the remainder of the material was homogenized with the melt resulting in a viscous bath having a temperature of about 620°C.

Result and discussions

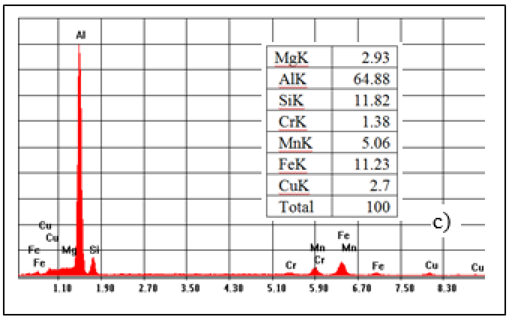
From the thermodynamic studies of the reactions that can occur between the metal bath/chips and the reaction products between methane gas and oxygen, given the chemical composition of the secondary alloy studied, we have selected the following reactions as very probable.

$$2Al + 3H_2O(g) = Al_2O_3 + 3H_2(g) \quad (1)$$
$$2Al + 3CO_2(g) = Al_2O_3 + 3CO(g) \quad (2)$$
$$2Al + 1.5O_2(g) = Al_2O_3 \quad (3)$$
$$2Mg + O_2(g) = 2MgO \quad (4)$$
$$2Mn + O_2(g) = 2MnO \quad (5)$$

Another category of reactions that may occur are those with the formation of complex compounds, between the aluminum oxides and the metal oxides in groups 1 and 2 of the Mendeleev Table (spinel), as can be deduced from the electron microscopy analysis and the EDS analysis presented in figure 5.



O K	15.88
MgK	4.22
AlK	54.25
SiK	20.13
CaK	0.83
FeK	1.19
CuK	3.49
Total	100



MgK	2.93
AlK	64.88
SiK	11.82
CrK	1.38
MnK	5.06
FeK	11.23
CuK	2.7
Total	100

Fig. 5. Electron microscopy analysis (a) and EDS analysis with the chemical composition of the filiform compounds (b) and fragmented gray compounds (c)

$2Al_2O_3 + 2Mg + O_2(g) = 2MgAl_2O_4(ia)$ (6)
$Ca + 2Al + 2O_2(g) = CaAl_2O_4(ia)$ (7)
$Ca + 2Al + 4CO_2(g) = CaAl_2O_4(ia) + 4CO(g)$ (8)
$Mg + 2Al + 4CO_2(g) = MgAl_2O_4(ia) + 4CO(g)$ (9)

Activation energies of the reactions

The dependencies of the speed constants of the temperature reactions (1) ÷ (5) are represented in figures 6-10 and can be calculated with Arrhenius's equation:

$$K=A\exp(-Q/RT) \qquad (10)$$

where:

A represents the frequency factor;
Q is the activation energy of the reaction, kJ/mol;
R is the universal gas constant;
T is the absolute temperature, K.
By logarithm, it results:

$$\ln K = \ln A+(1/T)(-Q/R) \qquad (11)$$

The variation of the speed constant parameters in relation to temperature for each reaction is shown in tables (3-7).

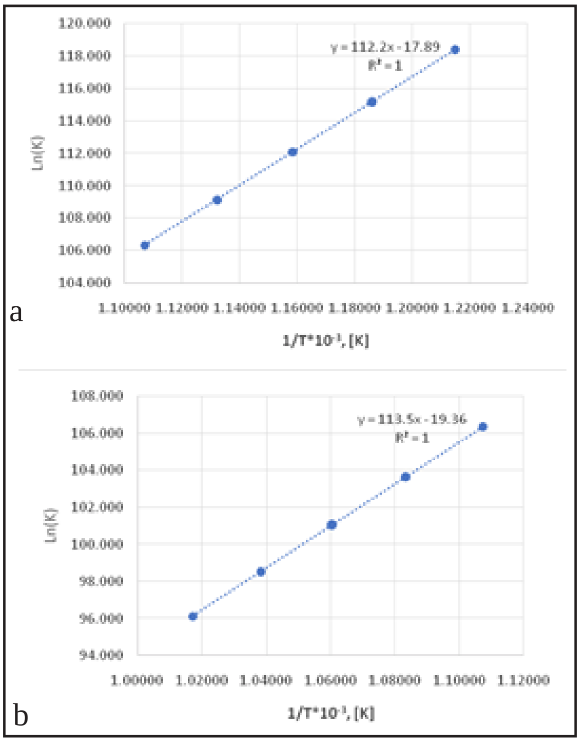


Fig. 6. Dependence of the speed constants of the temperature reaction 1 for the two intervals: a) 823.15 - 903.15 K; b) 903.15 - 983.15 K

T, K	ΔG, kJ	k	ln k	1/T, K^{-1}	1/T*10^{-3}, K^{-1}
823.150	-810.404	2.692E+051	118.422	0.00121485	1.21485
843.150	-807.409	1.058E+050	115.186	0.00118603	1.18603
863.150	-804.428	4.843E+048	112.102	0.00115855	1.15855
883.150	-801.461	2.553E+047	109.159	0.00113231	1.13231
903.150	-798.505	1.535E+046	106.348	0.00110724	1.10724
923.150	-795.561	1.045E+045	103.660	0.00108325	1.08325
943.150	-792.402	7.752E+043	101.059	0.00106028	1.06028
963.150	-789.023	6.234E+042	98.539	0.00103826	1.03826
983.150	-785.655	5.562E+041	96.122	0.00101714	1.01714

Table 3
VARIATION OF THERMODYNAMIC PARAMETERS BY TEMPERATURE FOR REACTION 1

T, K	ΔG, kJ	k	ln k	1/T, K^{-1}	1/T*10^{-3}, K^{-1}
823.150	-823.859	6.023E+049	114.622	0.00121485	1.21485
843.150	-823.896	3.464E+048	111.767	0.00118603	1.18603
863.150	-823.948	2.274E+047	109.043	0.00115855	1.15855
883.150	-824.018	1.688E+046	106.443	0.00113231	1.13231
903.150	-824.105	1.406E+045	103.957	0.00110724	1.10724
923.150	-824.212	1.304E+044	101.579	0.00108325	1.08325
943.150	-845.717	1.299E+043	99.273	0.00106028	1.06028
963.150	-845.760	1.383E+042	97.033	0.00103826	1.03826
983.150	-845.803	1.613E+041	94.884	0.00101714	1.01714

Table 4
VARIATION OF THERMODYNAMIC PARAMETERS BY TEMPERATURE FOR REACTION 2

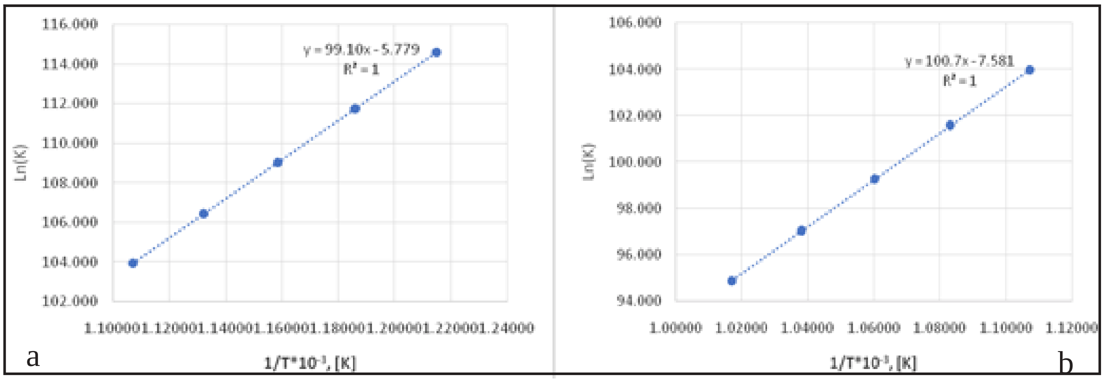


Fig. 7. Dependence of the speed constants of the temperature reaction 2 for the two intervals: a) 823.15 - 903.15 K; b) 903.15 - 983.15 K

Table 5

VARIATION OF THERMODYNAMIC PARAMETERS BY TEMPERATURE FOR REACTION 3

T, K	ΔG, kJ	k	ln k	1/T, K^{-1}	1/T*10^{-3}, K^{-1}
823.150	-1417.438	8.992E+089	207.126	0.00121485	1.21485
843.150	-1411.220	2.722E+087	201.326	0.00118603	1.18603
863.150	-1405.006	1.079E+085	195.795	0.00115855	1.15855
883.150	-1398.794	5.492E+082	190.515	0.00113231	1.13231
903.150	-1392.585	3.535E+080	185.469	0.00110724	1.10724
923.150	-1386.378	2.832E+078	180.642	0.00108325	1.08325
943.150	-1379.947	2.705E+076	175.992	0.00106028	1.06028
963.150	-1373.286	3.047E+074	171.505	0.00103826	1.03826
983.150	-1366.629	4.120E+072	167.202	0.00101714	1.01714

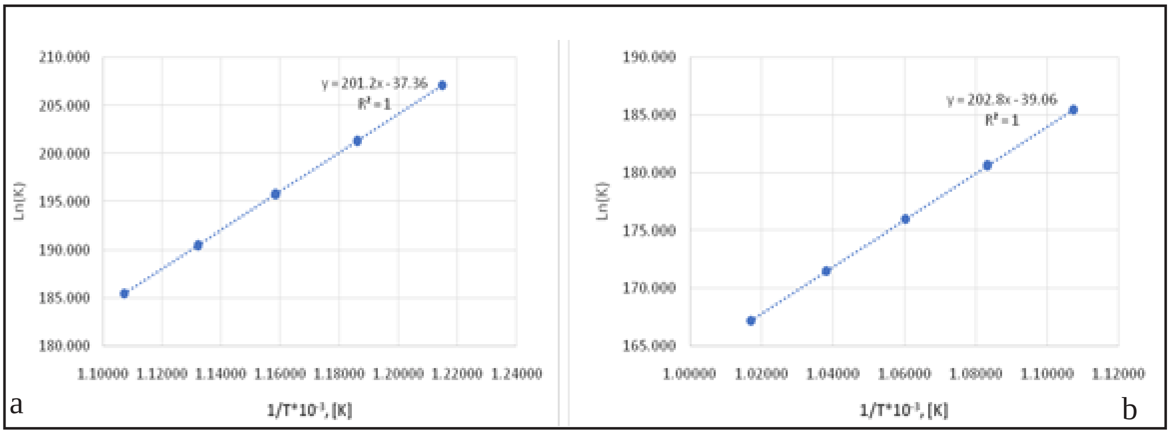


Fig. 8. Dependence of the speed constants of the temperature reaction 3 for the two intervals: a) 823.15 - 903.15 K; b) 903.15 - 983.15 K

Table 6

VARIATION OF THERMODYNAMIC PARAMETERS BY TEMPERATURE FOR REACTION 4

T, K	ΔG, kJ	k	ln k	1/T, K^{-1}	1/T*10^{-3}, K^{-1}
823.150	-1025.971	1.290E+065	149.922	0.00121485	1.21485
843.150	-1021.719	2.007E+063	145.760	0.00118603	1.18603
863.150	-1017.469	3.790E+061	141.790	0.00115855	1.15855
883.150	-1013.220	8.566E+059	138.000	0.00113231	1.13231
903.150	-1008.973	2.290E+058	134.379	0.00110724	1.10724
923.150	-1004.723	7.162E+056	130.914	0.00108325	1.08325
943.150	-1000.109	2.476E+055	127.549	0.00106028	1.06028
963.150	-995.493	9.845E+053	124.324	0.00103826	1.03826
983.150	-990.877	4.462E+052	121.230	0.00101714	1.01714

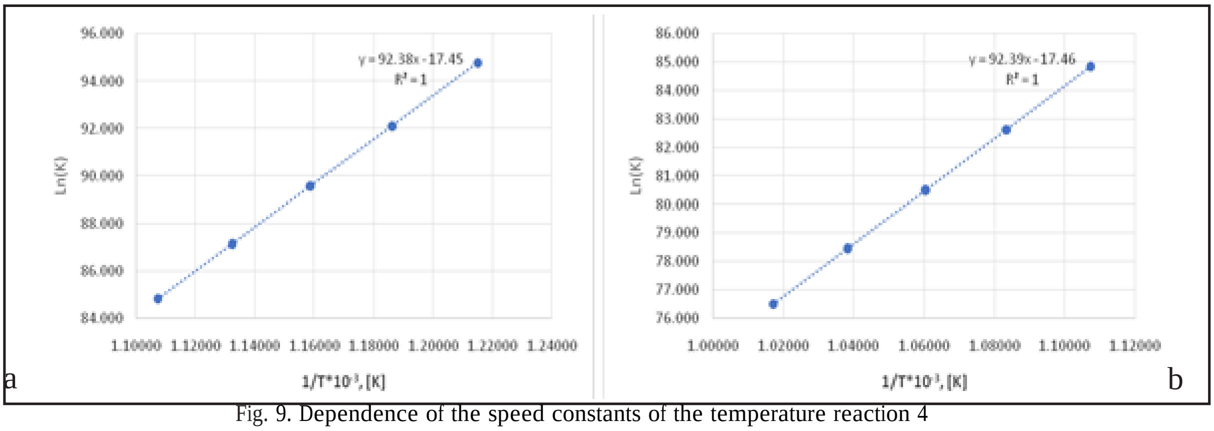


Fig. 9. Dependence of the speed constants of the temperature reaction 4 for the two intervals: a) 823.15 - 903.15 K; b) 903.15 - 983.15 K

Table 7

VARIATION OF THERMODYNAMIC PARAMETERS BY TEMPERATURE FOR REACTION 5

T, K	ΔG, kJ	k	ln k	$1/T, K^{-1}$	$1/T*10^{-3}, K^{-1}$
823.150	-648.604	1.452E+041	94.779	0.00121485	1.21485
843.150	-645.702	1.013E+040	92.117	0.00118603	1.18603
863.150	-642.800	8.001E+038	89.578	0.00115855	1.15855
883.150	-639.899	7.088E+037	87.154	0.00113231	1.13231
903.150	-636.998	6.991E+036	84.838	0.00110724	1.10724
923.150	-634.097	7.624E+035	82.622	0.00108325	1.08325
943.150	-631.197	9.132E+034	80.500	0.00106028	1.06028
963.150	-628.296	1.195E+034	78.466	0.00103826	1.03826
983.150	-625.379	1.695E+033	76.513	0.00101714	1.01714

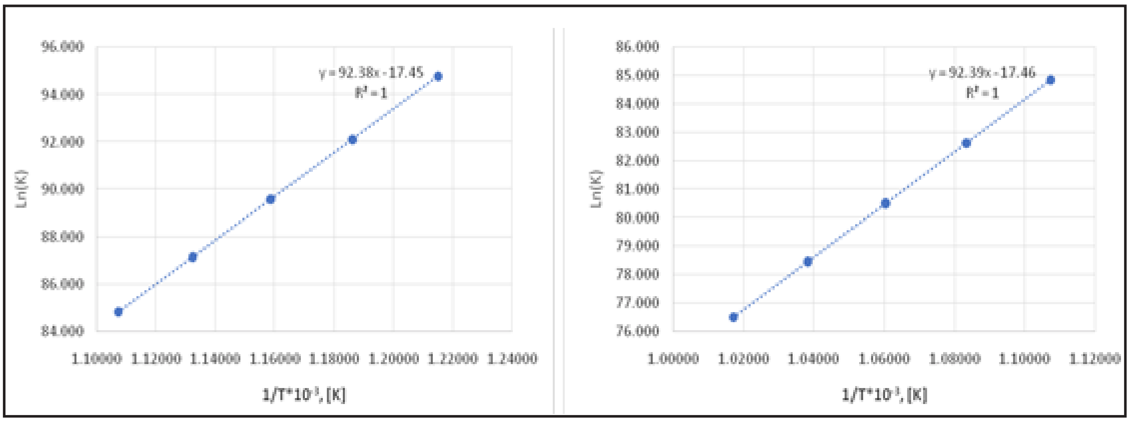


Fig. 10. Dependence of the speed constants of the temperature reaction 5
for the two intervals: a) 823.15 - 903.15 K; b) 903.15 - 983.15 K

Table 8

VARIATION OF ACTIVATION ENERGIES (Q) FOR THE REACTIONS (1) ÷ (5)

Reaction	Temperature, K	ln A	A	-Q/R	Q, kJ/mol
1	823.15 - 903.15	112.2	4.720283	- 17.89	0.148746
	903.15 - 983.15	113.5	4.731802	- 19.36	0.160968
2	823.15 - 903.15	99.1	4.596129	- 5.779	0.048049
	903.15 - 983.15	100.7	4.612145	- 7.581	0.063032
3	823.15 - 903.15	201.2	5.304299	- 37.36	0.310630
	903.15 - 983.15	202.8	5.312220	- 39.06	0.324764
4	823.15 - 903.15	144.4	4.972587	- 25.55	0.212435
	903.15 - 983.15	146.0	4,.983606	- 27.29	0.226902
5	823.15 - 903.15	92.38	4.525911	- 17.45	0.145088
	903.15 - 983.15	92.39	4.526018	- 17.46	0.145171

For reactions (1) ÷ (5), for temperature ranges, 823.15 - 903.15 K (550 - 630°C) and 903.15 - 983.15 K (630 - 710°C), result in the following values for the frequency factors (A) and activation energies (Q) (table 8).

Conclusions

The processing of small metallic waste at high temperatures leads to significant losses of both aluminum as well as the metals that are part of the secondary alloys. The losses are even higher as the quality of the materials used is lower.

The proposed technology consists of processing the small waste at low temperatures (580 ÷ 620°C / 853.15 ÷ 893.15 K for the EN AB 46100 alloy), temperatures near the eutectic temperature of the said alloy.

Using an intermediate step in the technological process -oxygen only feeds, stage after loading the furnace with a new amount of waste, leads to the reduction of natural gas consumption by burning volatile substances from pre-heating waste composition.

For reactions (1) ÷ (5), values of free enthalpy (Gibbs energy) have very high values in absolute value, which means that all these reactions take place from left to right (for example, at a temperature of 610°C / 883.15 K: DG_1 = - 801.461 kJ, DG_2 = - 824.018 kJ and DG_3 = - 1398.794 kJ).

The reaction activation energy (Q) exhibits higher values for the 630 ÷ 710°C / 903.15 - 983.15 K temperature range, than the 550 ÷ 630°C / 823.15 - 903.15 K interval. The phenomenon that occurs and leads to an increase in activation energy is oxidation of the scrap surface, a phenomenon which subsequently leads to an increase in the amount of slag resulting in the production of secondary alloys with aluminium base, using small waste.

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