

Calcium–aluminothermal production of niobium and mineral composition of the slag

Valeri Gorkunov and Rein Munter*

Department of Chemical Engineering, Tallinn University of Technology, Ehitajate tee 5, 19086 Tallinn, Estonia; valerig47@mail.ru

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Abstract. The process of niobium pentoxide calcium–aluminothermal reduction in pilot plant and full scale conditions was studied at Silmet JSC, Estonia. In the paper the construction and operating parameters of the pilot plant shaft melting furnace and the composition of raw materials mixture as well as of the product (niobium ingot) are described. The optimum ratio of Ca/Al to obtain the best quality niobium ingot was revealed in pilot plant melting tests, and the results of analyses of the mineral and phase composition of the slag are presented. Addition of metallic Ca to the raw materials mixture with 10% excess enabled to reduce the Al content in ingot to 4.4% and increase the Nb content >90%. In the optimum conditions of the reduction process a slag with the composition of $3\text{CaO}\cdot 5\text{Al}_2\text{O}_3$ is formed. Also different niobium containing oxides and metallic phases may be present in the slag. The results obtained enable to optimize the niobium production process and to elaborate an effective protective layer for the graphite facing of the furnace as well as to make some suggestions for possible utilization of the waste slag in the building materials industry.

Key words: rare metals, niobium, metallurgy, alloys production, waste slag.

INTRODUCTION

Silmet JSC is one of the largest companies in Europe in rare elements production, holding the third position in the world in the metallic niobium production capacity. The greatest producer of rare elements in the world is China [1, 2]. The main suppliers of the raw material are Brazil and Nigeria. The products are mainly exported to Japan, the USA, and Europe. At the present time Silmet JSC produces about 20% of the global amount of niobium. The production of master alloys, alloys, and powders containing niobium at Silmet JSC has a clear tendency to grow [1].

* Corresponding author, Rein.Munter@ttu.ee

The basic technology of rare metals manufacture is aluminothermal reduction of metal oxides [3–5]:



At Silmet JSC two reducers, metallic powdered aluminium and chips of metallic calcium are used, and the reduction process corresponds to the following prevailing reaction [6]:



The mechanism of metallothermal reduction processes has not been sufficiently studied yet [7]. These processes are complicated due to the fact that the reagents, for example calcium, may be simultaneously in two different aggregation states, in liquid and in vapour one. As a result of the reduction reaction (2) two products are formed: alloy of niobium with residual aluminium and slag (calcium–aluminium oxides), having usually a quite clear separation or interfacial area (Fig. 1). The melting furnace is made of steel and supplied with a specific graphite facing inside, with a system of ventilation and hermetization, and Ar gas input (Fig. 2). The initial compounds (Nb_2O_5 , Ca, Al) are first mixed in a drum during 15–20 min. Nb_2O_5 and Al are in the form of powder and Ca is used in the form of chips. After mixing the raw mixture is loaded into the shaft furnace. Incendiary is placed on the surface of the mixture, and the furnace is hermetized. After that the space of the furnace is filled up with the inert gas argon. The reaction is initiated by a special ignition facility on the inner side of the upper cover of the furnace. During the melting process the ventilation system is switched on to remove hot gases.

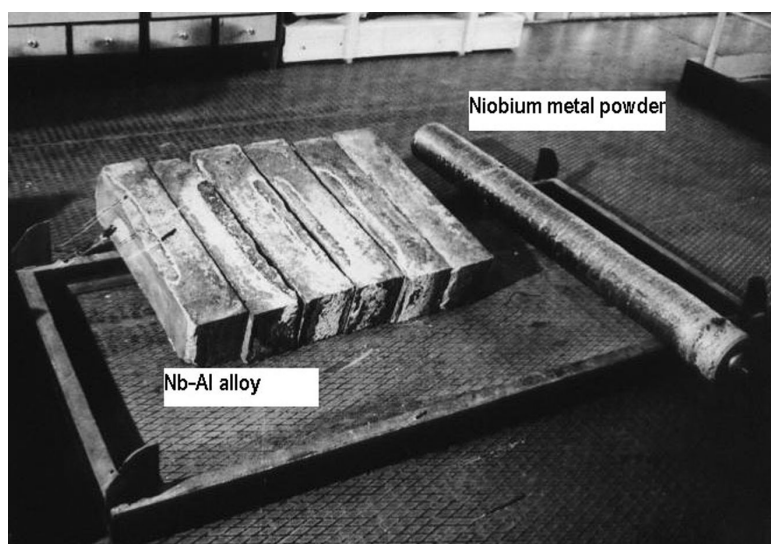


Fig. 1. Niobium–aluminium alloy and remelted niobium powder.

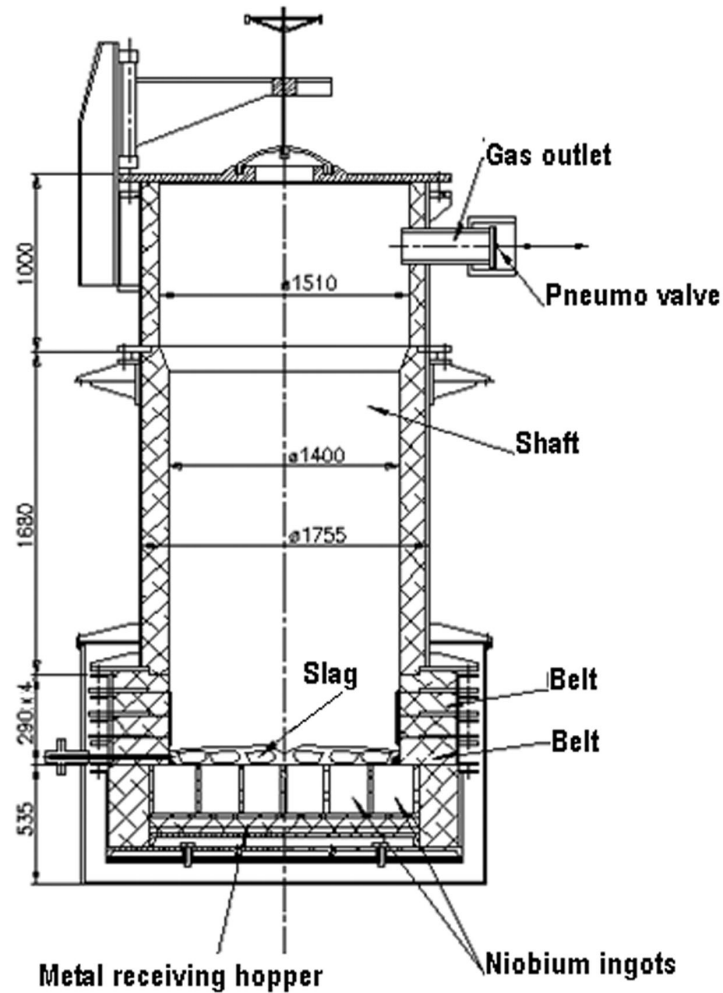


Fig. 2. Melting shaft furnace for rare metals and master alloys manufacture.

The reduction reaction proceeds with a significant temperature gradient (from room temperature to $>2000^{\circ}\text{C}$) in a few minutes ($\sim 5\text{--}6$ min). The furnace is designed taking into consideration the steep increase in temperature and pressure to avoid the breaking of the facing. Liquid metal (niobium) precipitates, passing through the slag layer and fulfills the ground floor of the furnace, the slag on its surface, after 3–4 min of retention, and then flows out into the receiving scoop. The reduction reaction is accompanied by significant gaseous emissions of high temperature. According to the Nb–Al phase diagram in Fig. 3 [8], the temperature of the process reaches almost 2400°C due to the very high specific heating effect.

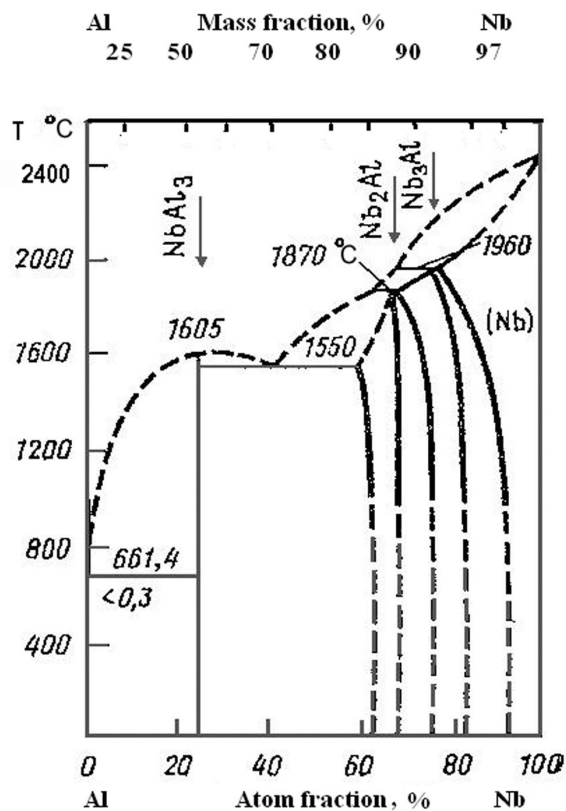


Fig. 3. Phase diagram of the system Nb–Al [8].

In the process in the furnace three metals are involved (melting temperature and standard electrochemical potential are given in parentheses): niobium (2468 °C; 0 V), and the reducers calcium (851 °C; –2.76 V) and aluminium (660 °C; –1.66 V) [7, 9]. The most energetic reducer is calcium, which has the lowest negative electrochemical potential among the known metals.

Niobium ingots must have a high mechanical strength for the next remelting process (refining) in the electronbeam furnace. In the world practice alloys with aluminium content over 20% are prevailing; however, in these conditions intermetallides Nb₂Al and Nb₃Al are formed. Alloys are fragile and easy to crush. At Silmet JSC a different technology leading to the formation of “plastic” alloys resistant to heavy loads and crushing was developed. In these alloys the aluminium content does not exceed 5%. The formation of the “plastic” alloys of niobium with aluminium is possible at high temperatures (up to 2400 °C) (Fig. 3). To increase the temperature of the process, metallic calcium as a reducer is added:



The exothermic heat of this reaction is 510 kJ/mole, and the specific exothermic heat is 106 kJ/g-atom of raw mixture. At the same time the specific exothermic heat of reaction (1) is only 86.1 kJ/g-atom [10]. Changing the ratio of calcium and aluminium makes it possible to increase the exothermic heat of the reduction reaction significantly compared to the effect of reaction (1), and to reduce the melting temperature of the slag (Fig. 4). It is important that differently from aluminium, calcium does not dissolve in niobium and, consequently, does not pollute it. A similar technology with addition of iron as an additional reducer was developed in the USA [10]; however, getting rid of iron compounds in the remelting process is very complicated.

Oxides of calcium and aluminium can form series of different compounds in the slag. The average composition of the aluminium–calcium flux (slag) at Silmet JSC is the following: 65–75% Al_2O_3 , 25–30% CaO , and 3–5% Nb_2O_5 [1].

According to equation (2) the prevailing compound of the slag is $3\text{CaO}\cdot 5\text{Al}_2\text{O}_3$. The slags of this type can be classified as highly aluminous, niobium containing, “short”, and non-decomposing. Maintaining this mineral composition of the slag is a very important technological parameter, as it determines the viscosity and surface tension of the slag and has a direct impact on the fulfilment of the reduction process and clear separation of phases. The velocity of liquid niobium sedimentation and its purity (degree of separation between the metallic and slag phases) depend mainly on surface tension between metallic and slag phases.

The aim of the present study was optimization of the Ca/Al ratio in the raw materials mixture to achieve the product of the best quality, analysis of the composition of the product (Nb–Al alloy), and determination of the mineral and phase composition of the waste slag. The last is of interest from the point of view of solving the problem of slag utilization.

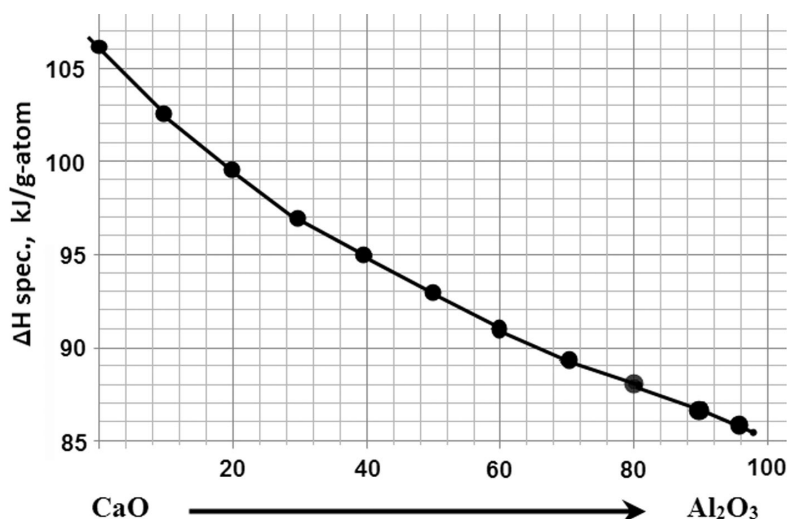


Fig. 4. Specific exothermic heat of the calcium–aluminothermal reduction reaction depending on the ratio of Ca/Al in the raw materials mixture.

EXPERIMENTAL AND ANALYTICAL METHODS

The composition of niobium pentoxide used at Silmet JSC as the initial raw material for the pilot plant and full-scale tests is presented in Table 1. Chemical and granulometric composition of powdered aluminium and metallic calcium chips used in the pilot and full scale meltings is given in Table 2. On the basis of a series of the pilot plant tests the optimum composition of the raw materials mixture was calculated (Table 3). The reducers (Al, Ca) were taken in 110% and 120% excess towards their stoichiometric amounts. Typical composition of slags is given in Table 4. Table 5 presents the composition of the product (Nb–Al alloy). The minimum content of Nb is 94.0% and the maximum content of the main admixture, Al, is 4.5%. This specification is for the standard product of Silmet JSC nowadays. All alloys can also be produced according to the customer’s “recipe”.

Table 1. Composition of niobium pentoxide [1]

Denomination	Content	
	Grade B	Grade C
Nb ₂ O ₅	min 99.2%	min 99.2%
Ta	max 250 ppm	max 200 ppm
Fe	max 1000 ppm	max 1000 ppm
Ti	max 100 ppm	max 100 ppm
Si	max 350 ppm	max 300 ppm
W	max 40 ppm	max 40 ppm
Mo	max 20 ppm	max 20 ppm
P	max 200 ppm	max 200 ppm
Low* at 900 °C	max 0.4%	max 0.15%

* Low – Loss in the weight at 900 °C.

Table 2. Chemical and granulometric composition of aluminium and calcium

Compound	Mass % of admixtures, no higher than						
	O	N	Cu	Si	Fe	Ti	Mo
Al	0.03	0.006	0.02	0.4	0.35	–	–
Ca	0.3	0.015	–	0.008	–	–	–
Ar	0.012	0.008	–	–	–	–	–
	Fractional composition, μm						Volumetric weight, g/cm ³
Al	+220	+140	+100	+71	–71		
	–	2.0	12.7	23.5	61.8	–	1.0

– Not determined.

Table 3. Impact of the ratio of Ca and Al and reducers excess in the raw materials mixture on the reduction process

Ratio Ca/Al	Slag composition Temperature of melting, °C	Reducers equivalent excess, expressed by Al, %	Nb, enraptured to the melt, %	Al in the melt/Nb ₂ O ₅ in the slag, %/%
Al	Al ₂ O ₃ 2050	10	70	5.0/2.8–3.0
		20	94.9	8.3/–
0.45	3CaO·5Al ₂ O ₃ 1720	10	91.2	4.4/2.8–3.0
		20	98.2	7.6/2.0–2.5
0.74	CaO·Al ₂ O ₃ 1600	10	81.1	3.0/8.1–8.3
		20	94.3	6.4/–
1.24	5CaO·3Al ₂ O ₃ 1430	10	73.5	2.0/8.5–9.9
		20	82.4	5.1/–
2.23	3CaO·Al ₂ O ₃ 1539	10	57.8	1.2/–
		20	67.0	3.4/–
Ca	CaO 2570	20	Sintering took place	–/–

Table 4. Typical chemical composition of slags, % mass

Reaction	Chemical composition, % mass						Slag composition	Note
	Al ₂ O ₃	CaO	Nb ₂ O ₅	FeO	SiO ₂ , TiO ₂	C		
7	78	22	2.8–3.0	0.10	0.02	0.05	3CaO·5Al ₂ O ₃	
7	70	30	2.0–2.5	0.07	0.02	0.04	3CaO·5Al ₂ O ₃	
6	64	36	7.8–8.3	0.15	0.02	0.06	CaO·Al ₂ O ₃	
5	53	47	8.5–9.9	0.11	0.02	0.15	5CaO·3Al ₂ O ₃	Bad separation
4	43	57	8.3–10	0.09	0.02	0.19	3CaO·Al ₂ O ₃	No ingot

Table 5. Composition of NbAl alloy [1]

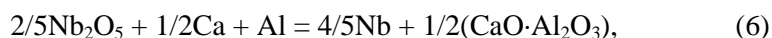
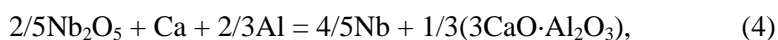
Denomination	Content, %
Nb	min 94.0
N	max 0.25
O	max 0.7
C	max 0.2
Ti	max 0.05
Si	max 0.12
Al	max 4.5
Fe	max 0.5
Ca	max 0.05
Ta	max 0.05
W + Mo	max 0.01

From the results of the pilot tests it was concluded that, for example, for reaction (2), where the slag with the composition of $3\text{CaO}\cdot 5\text{Al}_2\text{O}_3$ was formed, the optimum reducers excess was between 10% and 20%. The produced ingot was compact and “plastic”. The slag samples were taken during the melting tests from different points of the “slag field” and were blanked using a cutting blade. One of the specimen’s surface was polished. Also samples ground to fine-grained powder were analysed. Structural studies of Nb–Al alloys (ingots) were carried out by methods of optical metallography using a microscope NEOPHOT-21 and a scanning electron microscope REM-100U. The chemical composition of slag was determined first by the methods of wet chemical analysis [11]. The slag from the optimum reduction process (below reaction (7)) with the composition of $3\text{CaO}\cdot 5\text{Al}_2\text{O}_3$ (i.e. with a minimum relative amount of Al:Ca) was analysed recently in more detail in the Zeiss Demo Lab in Cambridge, UK [12] as well as in the chemical laboratory of Silmet JSC [13]. The slag sample was polished up to the roughness of $0.05\ \mu\text{m}$. The surface of the polished sample was investigated by the scanning electron microscope ZEISS EVO-50 with an INCA Act X-ray microanalyser in secondary (SE) and backscattered (BE) electron modes at accelerating voltage of 20 kV (Zeiss Demo Lab., Cambridge, UK).

RESULTS AND DISCUSSION

The use of a double reducer Al–Ca in the process of Nb_2O_5 reduction is a positive factor from the point of view of the formation of CaO as it enables to achieve a favourable impact on such important physical parameters of the system as viscosity, density, and surface tension between the metallic and slag phases.

It was established that in the process of Nb_2O_5 reduction with the double reducer Al–Ca the following reactions can proceed leading to the formation of slags with different composition (Tables 3 and 4):



The phase diagram of the system CaO– Al_2O_3 is shown in Fig. 5 [14]. It can be seen that at higher temperatures ($>2000^\circ\text{C}$) Al_2O_3 does not form mixtures with CaO. Starting from 1700°C different solid solutions of Al_2O_3 with CaO are formed ($\text{CaO}\cdot 6\text{Al}_2\text{O}_3$, $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$) and at 1600°C the solid solution $3\text{CaO}\cdot 5\text{Al}_2\text{O}_3$ with the minimum relative amount of CaO– Al_2O_3 is formed. Selection of the optimum ratio of Ca/Al for the reduction process was focused on slag formation with the lower melting temperature. The series of pilot and full-scale melting tests enabled to find the optimum composition of the raw mixture to obtain as much as possible pure niobium ingot with the Al content below 5%

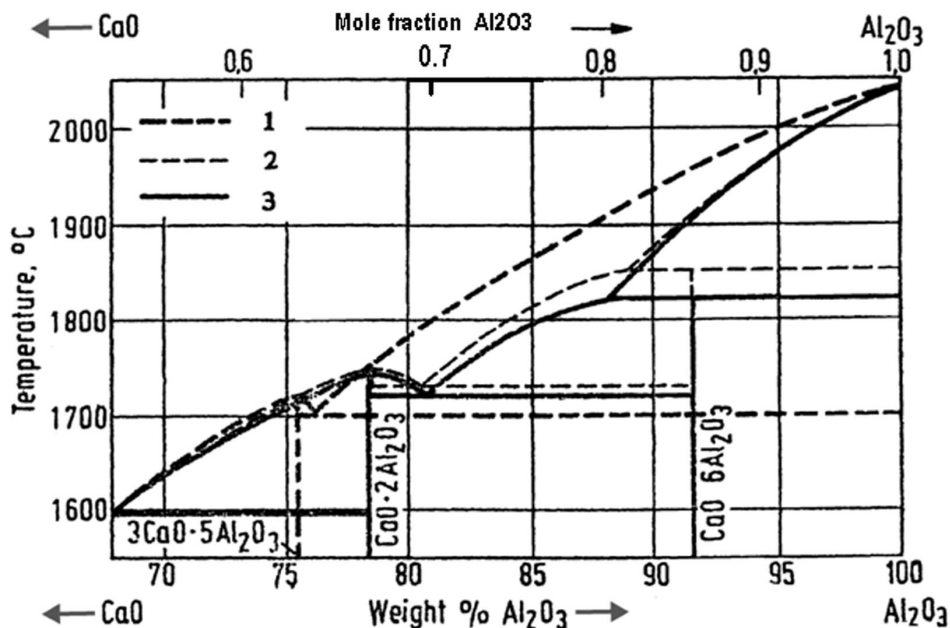


Fig. 5. Phase diagram of the system CaO–Al₂O₃ [14]: 1 – Rankin & Right (1909); 2 – Filonenko & Lavrov (1950); 3 – Visni (1954).

and slag with the minimum residual content of Nb₂O₅ (Table 3). For the reduction of 1 mole of niobium pentoxide 200 kg of Ca were taken, which is equivalent to 90 kg of Al. From Table 3 it can be concluded that these requirements are fulfilled the best at the ratio of Ca/Al = 0.45 in the raw materials mixture and at the reducers excess of 10%. Under these conditions the content of aluminium in the Nb–Al alloy is minimum – 4.4% with the content of residual Nb₂O₅ in the slag 2.8–3.0%. The slag composition corresponds to the formula 3CaO·5Al₂O₃ with the melting temperature of 1720°C (second row in Table 3).

It was found that from the reactions (4–7) in Table 4 the desirable one is the 7th reaction with 10% excess of the reducers, where the consumption of calcium was optimal, a good separation of metallic and slag phases took place, the content of the residual Nb₂O₅ was minimal, and the produced ingot was of very good quality, compact and plastic. It was established that addition of metallic Ca to the raw materials mixture with 10% excess enabled to reduce the Al content in the ingot to 5% or less and increase the Nb content (Fig. 6). Figure 6 demonstrates that the maximum Nb yield (~94%) can be achieved when using 20% Al excess in the raw materials mixture at the Ca/Al ratio around 0.5. However, in these conditions also the content of residual Al in the ingot will be higher (~6–7%). To reduce the residual Al content to 5%, it is necessary to increase the Ca/Al ratio over 0.5. Thus, the optimization of the melting process means achievement of the maximum content of Nb and minimum content of Al in the ingot by varying two parameters – the Al excess in the raw mixture and the Ca/Al ratio.

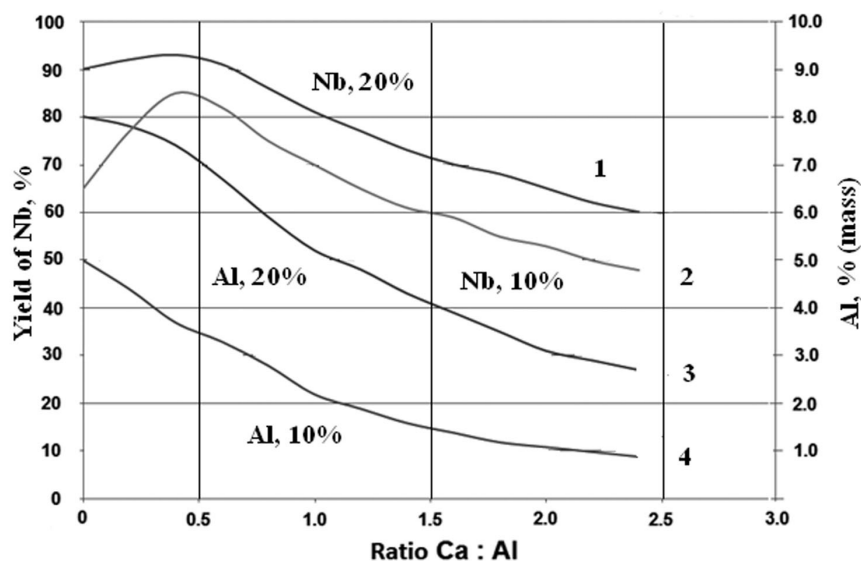


Fig. 6. Yield of niobium in the ingot (%) and content of residual aluminium (%), depending on the ratio of Ca/Al in the raw materials mixture: 1, 2 – enrapture of Nb to the melt at 20% and 10% excess of Al in the raw materials mixture, respectively; 3, 4 – content of residual aluminium in the ingot, at 20% and 10% of its excess in the raw materials mixture, respectively.

The strenghtening of the metallic niobium ingot and the increase in its plasticity in pilot plant and full scale meltings proceeded due to the formation of the solid solution of aluminium in niobium and diminishing of the content of the fragile phase of intermetallides. The microstrength of the ingot was $600 \pm 100 \text{ kg/mm}^2$. The structure of the alloy with 4.5% aluminium content in the field of X-rays is shown in Fig. 7 (a, b). The alloy contains a solid solution of aluminium in niobium with an insignificant amount of intermetallides Nb_3Al . The main mass of the ingot has a uniform structure corresponding to the solid solution. Introduction of the results of the pilot plant melting tests into practice led to excellent parameters of the industrial Nb–Al alloys with mass up to 2000 kg (Table 6).

Addition of metallic Ca into the raw materials mixture led to the formation of different calcium aluminates ($n\text{CaO} \cdot m\text{Al}_2\text{O}_3$). A typical picture of freshly formed slag is shown in Fig. 8. The main mass of the slag is made up of $3\text{CaO} \cdot 5\text{Al}_2\text{O}_3$. Energy Dispersive Spectrometric (EDS) analysis [12] from the area (Fig. 9) showed the presence of carbon, oxygen, aluminium, calcium, and niobium in the slag in the concentrations presented in Table 7. Metallic niobium was diffused over the slag sample volume in the form of small particles (diameter 1–5 μm). As the oxygen distribution does not coincide with the distribution of Nb particles, it was concluded that Nb was in the form of pure element. The proportions of the Ca and Al content corresponded to the proportions of their oxides in the slag with the composition of $3\text{CaO} \cdot 5\text{Al}_2\text{O}_3$. The sample volume was porous with pore diameter of about 20 μm . The phase composition of the sample was determined

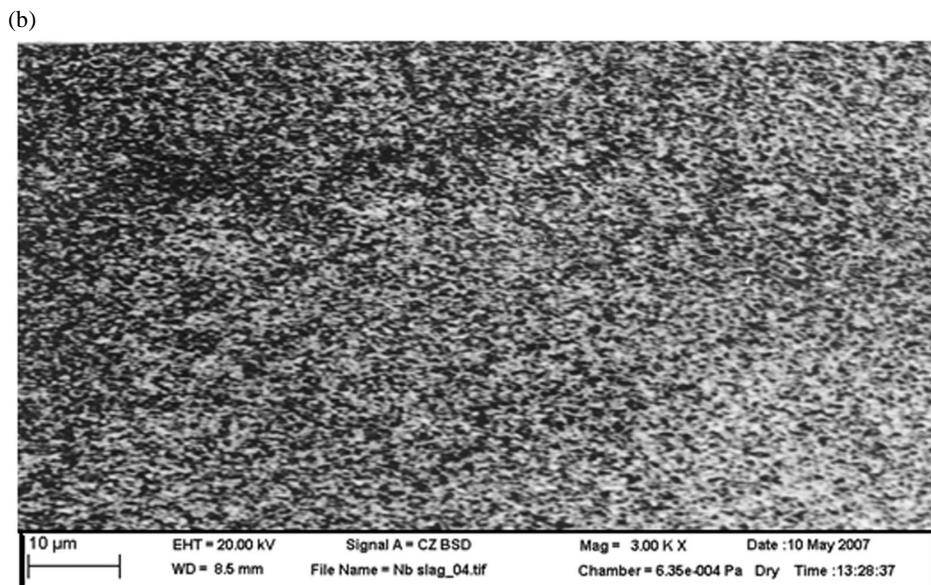
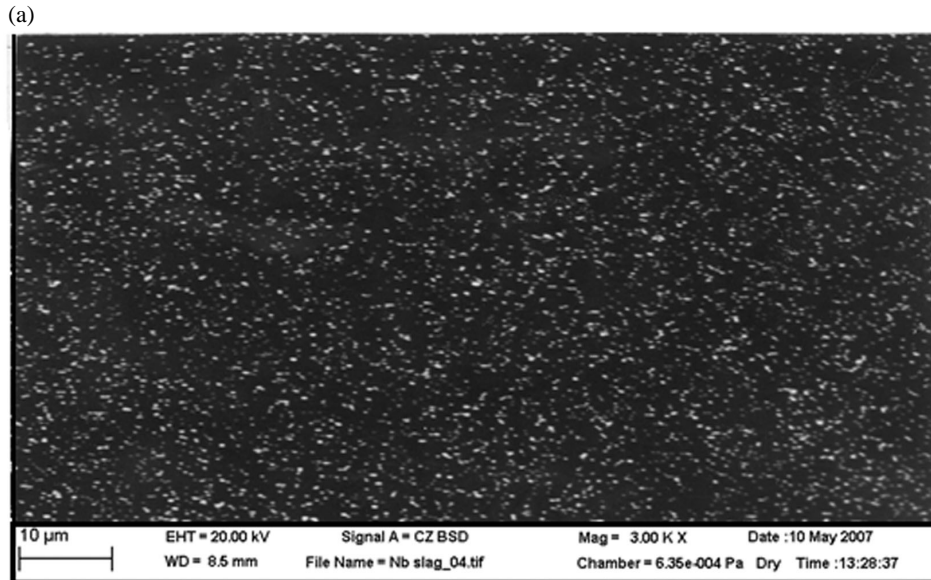


Fig. 7. Microstructure of the Nb–Al alloy at the aluminium content of 4.5%. (a) Al in the field of X-ray. (b) Nb in the field of X-ray.

in the chemical laboratory of Silmet JSC [13]. According to the quality certificate No. 0012870 issued by the factory, the content of Al_2O_3 in the slag was 68% and that of CaO 24%. This composition corresponds to the molar ratio of $\text{Al}_2\text{O}_3/\text{CaO} = 1.56$, which is quite close to the theoretical ratio of these oxides (1.67) in the slag of the composition $3\text{CaO}\cdot 5\text{Al}_2\text{O}_3$.

Table 6. Results of the industrial meltings of Nb–Al alloys with aluminium content <4.5%

Reducer excess in raw materials mixture, %			Yield of Nb in alloy, %	Chemical composition of alloy, % mass				
Al	Ca	Equivalent excess by Al, %		Nb	Al	O	N	C
4	15	5.8	92.2	95.6	3.4	0.3	0.09	0.013
4	21	6.8	93.3	94.7	3.8	0.4	0.09	0.020
4	30	8.3	94.2	95.0	3.8	0.4	0.12	0.020
4	32	8.8	94.4	94.7	4.2	0.08	0.08	0.017
4.4	33	9.0	94.6	94.7	4.4	0.07	0.12	0.015
5	25	8.3	94.6	94.7	4.1	0.08	0.09	0.015
8.2	9.5	8.4	94.5	94.5	4.4	0.08	0.11	0.020

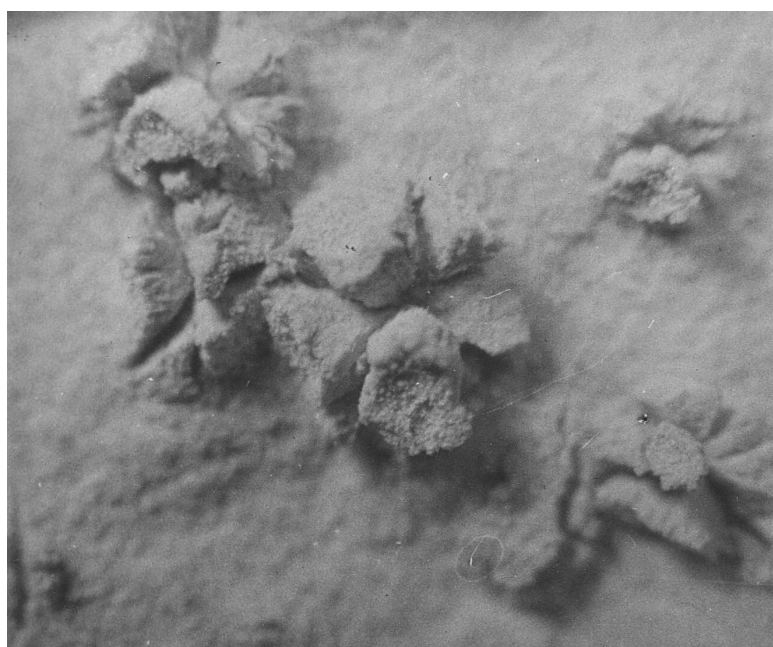


Fig. 8. Niobium slag with a distinguishable clear interfacial area: the “blossom” is $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$ and $\text{CaO}\cdot \text{Al}_2\text{O}_3$; the rest is $3\text{CaO}\cdot 5\text{Al}_2\text{O}_3$.

Table 7. Elementary composition of the slag from reaction (7)

Element	Weight %	Atomic %
C	3.59	6.78
O	35.16	49.77
Al	35.30	29.63
Ca	23.35	13.19
Nb	2.60	0.63
Total	100.00	100.00

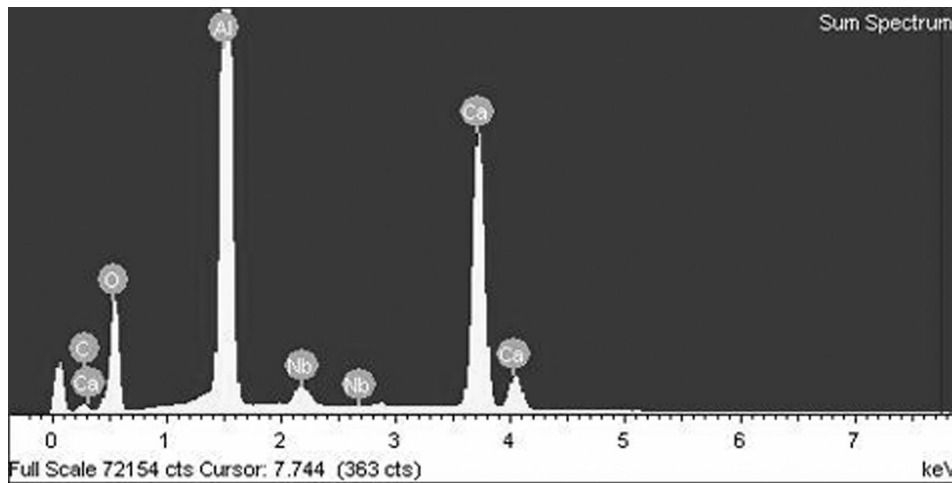
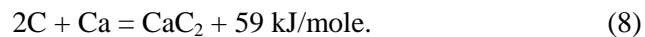
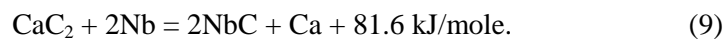


Fig. 9. The EDS spectrum of the slag sample with the composition of $3\text{CaO}\cdot 5\text{Al}_2\text{O}_3$.

It was also established that in the case of the melting processes according to reactions (4–7) erosion and destruction of the graphite facing of the furnace took place; the higher was the excess of calcium; the more intensive were these processes:



At the stage of higher temperatures calcium carbide reacts with metallic niobium according to reaction (9) and “pollutes” it with niobium carbides:



Niobium carbides (NbC and Nb_2C) are very stable (the melting temperature of NbC is 3480°C [10]), and their removal in the process of remelting is almost impossible.

CONCLUSIONS

The process of Nb_2O_5 calcium–aluminothermal reduction with the aim of metallic niobium production was studied in a shaft melting furnace at Silmet JSC in pilot plant and industrial conditions. The process proceeds in the atmosphere of the inert gas Ar in a few minutes (~5–6 min). Differently from the traditional aluminothermal technology here another reducer, metallic calcium, was added to the raw materials mixture. The ratio of Ca/Al as well as the reducers excess were optimized to achieve a good separation of the slag and metallic phases and a good, “plastic” ingot. It was found that addition of metallic Ca to the raw materials mixture with 10% excess at the ratio of $\text{Ca}/\text{Al} = 0.45$ enabled to reduce

the Al content in the ingot to 4.4%, to increase the Nb content over 90%, and to reduce the residual Nb₂O₅ content in the slag to 2.8–3.0%. Introduction of the results of the pilot plant melting tests into practice led to excellent parameters of the industrial NbAl alloys with the mass up to 2000 kg.

It was found that in optimum conditions of the reduction process (reaction (7)), characterized by a good separation of metallic and slag phases, the minimal content of the residual niobium oxides in the slag, and a very good quality, compact and plastic niobium ingot with the aluminium content below 5% were achieved. The slag with the composition of 3CaO·5Al₂O₃ was formed. The slags that contain calcium aluminates can be considered as a possible source of raw material for the production of fast-setting and very strong cements [15]. Erosion and destruction of the graphite facing of the furnace taking place during the reduction process is an urgent problem, which has to be solved in the nearest future.

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Niobiumi tootmine kaltsium-alumotermilisel meetodil ja šlakide mineraalkoostis

Valeri Gorkunov ja Rein Munter

1990. aastatel tootis niobiumi märkimisväärses kogustes Sillamäe metallurgiakombinaat (nüüd AS Silmet) Ida-Virumaal Eestis. Erinevalt traditsioonilisest alumotermilisest protsessist hakati seal niobiumpentoksiidi taandamiseks kasutama peale pulbrilise alumiiniumi ka metalset kaltsiumi, et tõsta protsessi temperatuuri ja saada suurema puhtusastme ning mehaanilise tugevusega (nn plastset) toodet (Al-i sisaldus alla 5%). Kahe taandaja kasutamine mõjutab kogu protsessi füüsikalisi-keemilisi parameetreid ja ka tekkiva šlaki koostist. Artiklis on kirjeldatud Sillamäe tehase niobiumisulatuse katsehju konstruktsiooni, toorsegu ja toote koostist. On leitud Ca/Al-i optimaalne suhe ja määratud šlaki mineraloogiline ning faasikoostis. On välja selgitatud, et metalse Ca lisamine toorsegusse 10%-lises ülehulgas Ca/Al-i suhte 0,45 juures võimaldab vähendada tootes jääkalumiiniumi sisaldust kuni 4,4%-ni ja tõsta Nb sisaldust üle 90%. Protsessi optimaalsetes tingimustes moodustab šlaki põhimassi $3\text{CaO} \cdot 5\text{Al}_2\text{O}_3$. Šlaki koostist on kontrollitud üksikute elementide (Nb, Ca, Al, O, C) sisalduse kaudu, mis on määratud skaneeriva elektronmikroskoobiga ZEISS EVO-50. Sama šlaki-proovi faasikoostis on määratud tehase AS Silmet keemialaboris ja see vastab šlaki põhimassi eeldatud koostisele (68% Al_2O_3 , 24% CaO). Pilootkatsete tulemused võimaldavad optimeerida niobiumitootmise protsessi ja välja töötada efektiivset kaitsekihti sulatusahju grafiitvooderdise kaitseks. Kaltsium-alumiinaatse koostisega šlakke võib käsitleda võimaliku tooraineallikana kiirelt tarduvate ja suure tugevusega tsementide valmistamiseks.