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Slutrapport för projekt

Pure Brass: Pb-removal by Vacuum Treatment and Si- removal by Oxidation of Brass

Projektperiod: Juni 2017 till Juni 2019
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Med stöd från

VINNOVA
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FORMAS 

**Strategiska
innovations-
program**

Pure Brass

Pure Brass

Titel på projektet – svenska Pure Brass
Titel på projektet – engelska Pure Brass
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Nyckelord: 5-7 st Lead free brass, Pb-removal, Si-removal, vacuum treatment, novel approach

Förord

This project has been carried out by Swerim and NBG (Nordic Brass Gusum) according to the project plan. The main parts of the project tasks including the modelling, small scale test, and pilot smelting test campaign have been carried out at Swerim. The industrial scale tests on Si-removal has been performed at the NBG plant in Gusum. In addition to the funding support of Energimyndigheten, NBG has also contributed with in-kind activities and funding. The funding of Energimyndigheten and contribution of NBG are highly acknowledged.

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Sammanfattning

Mässing är en legering som i sin basform innehåller koppar och zink i proportionerna 60/40. Utöver dessa tillsätts andra legeringsämnen för att förbättra egenskaperna hos slutprodukten. Exempelvis tillsätts bly (Pb) för att göra mässingen enklare att bearbeta, medan kisel (Si) ger ökad motståndskraft mot abrasion. Många olika länder och organisationer arbetar idag för att eliminera bly i mässing för att minska människors exponering av bly från dricksvatten, eftersom mässing ofta används i vattenkranar och liknande applikationer. I dagsläget innehåller nästan allt mässingsskrot som är i cirkulation bly, vilket skapar svårigheter när det ska återvinnas. Ett annat problem som ibland kan uppstå är att mässingsskrot med högt Si-innehåll blandas med normalt mässingsskrot som har lågt Si-innehåll vid återvinning. Detta skapar en situation där den resulterande smältan har för hög nivå av Si jämfört med den önskade sammansättningen. Det finns i dagsläget ingen etablerad metod för att avlägsna Si. Med fungerande metoder för att rena dessa föroreningar skulle det vara möjligt att uppnå bättre resurseffektivitet, eftersom föroreningarna inte gör att man riskerar att behöva kassera hela smältor. Det huvudsakliga målet för hela projektet är att stärka och förenkla mässingens cirkulära ekonomi genom att skapa metoder för att hantera dessa oönskade legeringsämnen vid återvinning.

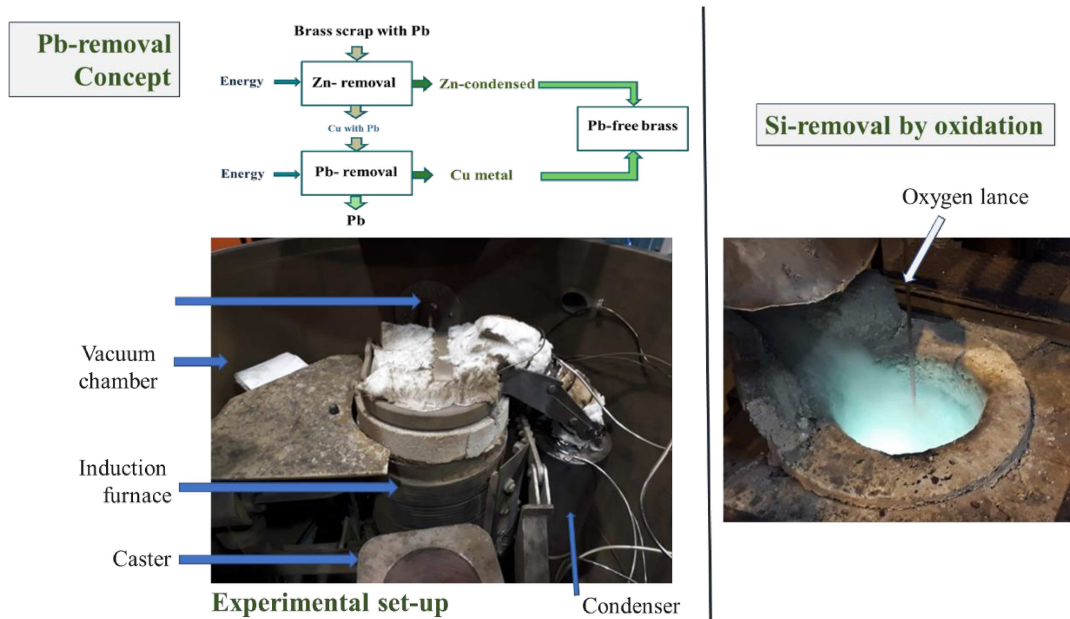
Arbetet för det här projektet var uppdelat i två delar, blyrening och kiselrening. Blyreningen inkluderade en serie försök i pilotskala i Swerims vakuuminduktionsugn. Kiselreningen inkluderade försök i både labb- och pilotskala på Swerim samt försök i industriskala på Nordic Brass Gusum (NBG).

De viktigaste resultaten från projektet Pure Brass kan sammanfattas enligt följande:

- En ny metod för att avlägsna Pb från mässing via vakuumbehandling har utvecklats och demonstrerats i en 30 kg-skala vakuumugn. Det kan konstateras att:
 - Pb kunde reduceras från 0.68% till 0.26%
 - Majoriteten av Pb och all Zn kan avlägsnas i fast fas och under smältning
 - Zn avlägsnas före Pb
 - Väldigt lågt tryck (<10 mbar) krävs för att få bra Pb separation
 - Processen kan kontrolleras med hjälp av mätningar av temperatur och tryck hos avgaserna
- Det är möjligt att delvis avlägsna Si från en mässingssmälta genom tillsatt av rökgasstoff innehållande ZnO. Det kan konstateras att:
 - Si-rening kan förbättras genom tillsatt av Na_2CO_3 som flussmedel
 - I labbskala är det svårt att få en bra gränsyta mellan mässingssmältan och tillsatt ZnO/ Na_2CO_3

- Ett möjligt alternativ kan vara att injicera ZnO-stoft/ Na_2CO_3 , men detta måste undersökas
- Det är möjligt att avlägsna en betydande andel Si från en mässingsmälta genom att injicera luft genom en lans, dock ger detta upphov till att antal praktiska problem:
 - Lansen måste ha en keramisk beläggning för att undvika slitage
 - Lansen måste bytas ut regelbundet på grund av påbyggnad kring lansspetsen
 - I industriell skala är det svårt att hålla tillräckligt högt gasflöde genom lansen
 - Oavsett skala leder processen till ökad bildning av slagg och damm stoft

För nästa step, NBG har avsikt att vidareutveckla den utvecklade Pb-reningsteknik genom ett antal verifieringsförsök hos Swerim (direkt uppdrag)



Summary

Brass is an alloy containing in its base form a 60/40 ratio of copper and zinc. In addition to this, other elements are added to improve the properties of the end product. For example, lead (Pb) is added to increase machinability and silicon (Si) is added for improving abrasion resistance. Various countries and organizations are working towards removing lead in brass to minimize human exposure to lead from drinking water, as brass is widely used in water taps. Currently, almost all circulating brass scrap contains lead, which causes difficulties during recycling. Also, sometimes the high Si-content brass scrap (up to 3%Si) is mixed with normal low Si brass scrap when recycling, this will create a situation where the Si-content becomes too high comparing to the target Si-level. There is currently no developed method for silicon removal. With working methods for purifying these elements, it would be possible to achieve a greater resource efficiency as the impurities will not cause a problem that risks ruining heats. The primary goal of the project is to strengthen and simplify the circular economy of brass by developing methods for handling these unwanted alloying elements during recycling.

The work for this project was split into two main parts, lead removal and silicon removal. The lead removal included a series of lab scale tests in Swerims vacuum induction furnace. Silicon removal included both lab and pilot scale testing at Swerim as well as industrial plant trials at Nordic Brass Gusum (NBG).

The major results from the Pure Brass project are summarised as follows:

- A novel method for Pb-removal by vacuum treatment has been developed and demonstrated in 30 kg scale. It was found that:
 - Pb could be reduced from 0.68% to 0.26%
 - The majority of Pb and all Zn can be removed in the solid phase and during melting
 - Zn is removed before Pb
 - Very low pressure (<10 mbar) is necessary to achieve good Pb separation
 - The process can be controlled using readings for the temperature and pressure of the off-gas
- It is possible to partially remove Si from molten brass by addition of gas flue dust containing ZnO
 - Si removal is improved by addition of Na₂CO₃ as flux
 - It is difficult to achieve good interface area between the molten brass and added ZnO/Na₂CO₃ in small laboratory scale
 - Injection of the ZnO-dust/Na₂CO₃ is possible option, but need to be tested in large scale

- It is possible to remove a significant portion of Si from molten brass by injecting air through a lance, but there are some practical problems to be solved
 - The lance must have a ceramic coating to avoid rapid wear
 - The lance must be replaced regularly due to build-up around the lance tip
 - In industrial scale it is difficult to inject enough oxygen through a lance
 - Regardless of scale the processes lead to increased slag and dust formation

For the next step, NBG has the intention to carry out additional tests at Swerim to further verify the developed vacuum treatment technology for Pb-removal from brass via direct contract project with Swerim.

1. Introduction

The Pure Brass project is a co-operation project of Swerim and Nordic Brass Gusum (NBG) aiming to develop a new method for brass scrap based production of Pb-free brass and a practical approach for Si-removal in brass. NBG is the major brass producer in Northern Europe with an annual production of about 30 kton. Most of the materials used for the NBG brass production are recycled brass scrap, end of use brass scrap and copper scrap; only 10% of the ingoing material is virgin zinc. The background of the two major tasks are described in the following. The technical experiments and main test results will be summarized based on the main complete technical report given in Appendix I.

1.1 Background Pb-removal

Due to its good corrosive resistant properties brass is widely used in plumbing applications. Many countries and organizations are working towards removing lead in brass to minimize human exposure to lead from drinking water, e.g. the strictest legal requirement is 0.25 % lead in brass in the USA. The industry has in response to this taken the approach of choosing lead free brass (0.1 %) to be able to sell products in all regions. Currently, almost all circulating brass scrap contains lead, and if the market for lead free brass continues to grow, it will soon outgrow the capacity for lead free production from scrap if no method for lead removal is found.

In a previous national project, **ADLEAF**, funded by Vinnova and the Nordic brass industry, a two-steps distillation concept based on vacuum technique as illustrated in Figure 1 has been developed by Swerim and NBG. The concept has been demonstrated by extensive experimental test works in laboratory scale, up to 1 kg scale.

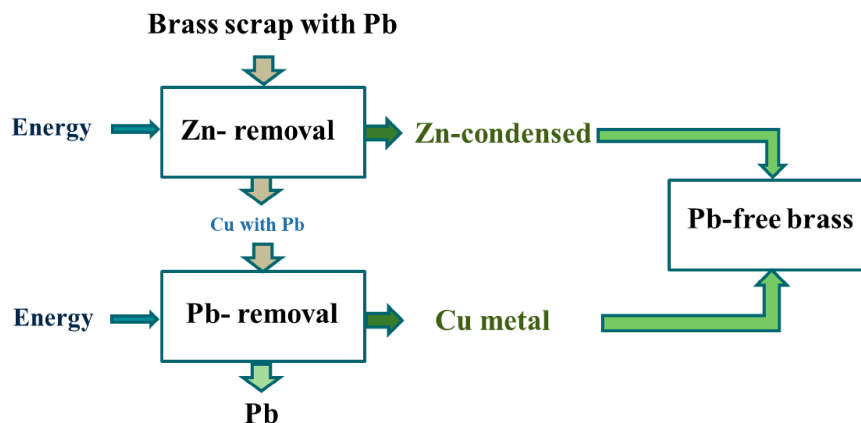


Figure 1 The Pb-removal concept developed in the ADLEAF project

In this project the developed concept will be verified, further developed and demonstrated in a semi-pilot scale vacuum induction furnace with a capacity for treatment of 20-30 kg brass sample. This makes it possible to have much better control of the material balance of the process, especially the evaporated Zn and Pb, and thereby provide more reliable conclusions. It is also easier to identify the most

important technical issues and challenges of the concept when operating in a bigger scale.

1.2 Background Si-removal

Silicon alloyed scrap is sometimes a problem for scrap based brass production. The circulating scrap between the brass manufacturer and brass users is generally well sorted and with known chemistry; however, the end of use scrap from society is less defined. This creates problems when silicon alloyed brass scrap (qualities with 0.7 or 3%Si) ends up in the wrong heat, as most brass alloys have a silicon content limit in the order of 0.02%. Currently there is no method for removing silicon, thus there is a risk of ruining brass heats if silicon alloyed brass scrap is added by mistake. The heat must then be casted, and the resulting scrap will either be slowly diluted or used for making silicon alloyed brass later.

The main obstacle for removing silicon is that there is no liquid slag in brass production, which sets limitations on the effectiveness of the Si refining. As the melting temperature for brass is relatively low (about 950°C) a solid powder is used to prevent oxidation instead. Therefore, if the silicon is removed by oxidation there is no slag layer that can entrap the oxidic inclusions. Trying to remove inclusions without a liquid-liquid interface is a difficult mission. By developing a liquid slag that works for brass the capabilities of the brass smelting process could be increased. In this project, these capabilities will be used to enable silicon removal from brass, for which there is currently no developed method. Methods for Si-removal by oxidisation, including using of ZnO-rich dust generated at NBG smelter with and without soda, and by air injection have been investigated in this project.

1.3 Objectives

The main objectives of the project are to strengthen and simplify the circular economy of brass by creating methods for handling unwanted alloying elements during recycling. This is achieved by the following sub-goals:

- Developing and demonstrating a method for separating lead from brass scrap in semi pilot scale.
- Developing a practical approach for Si-removal including slag design by experimental tests in laboratory, semi-pilot and industrial scale.

2. Summary of the major project activities and results

The most important tasks performed in the project and their major results are summarized in the following subsections.

2.1 Pb-removal by vacuum treatment

2.1.1 Experimental set-up and condenser design

The experimental set-up for the vacuum treatment trials is shown in Figure 2. The furnace itself is enclosed within a bigger vacuum container. The lid of the furnace has been modified with a channel for the created off-gas, which leads to a condenser

system with the purpose of capturing evaporated Zn and Pb. The rest of the gas is then sent to the vacuum container outlet.

Extensive efforts have been made to improve the condenser system connected to the induction furnace lid. In the initial tests there were problems due to Zn leakage to the vacuum chamber and condensing on various surfaces. This has also led to short circuit in the initial trials. These trials thus had to be terminated earlier than the planned schedule. See also Figure 3.

In the end, after a number of modifications of the furnace – condenser system, a well performing system has been developed and established for stable operation thereby making it possible to verify the concept shown in Figure 1. The improved furnace - condenser design/system could be seen in the later trials where almost all evaporated Zn from brass could be collected in the Zn-condenser. This means that Zn-leakage has been minimized and thus it was possible to operate the tests without short circuit.

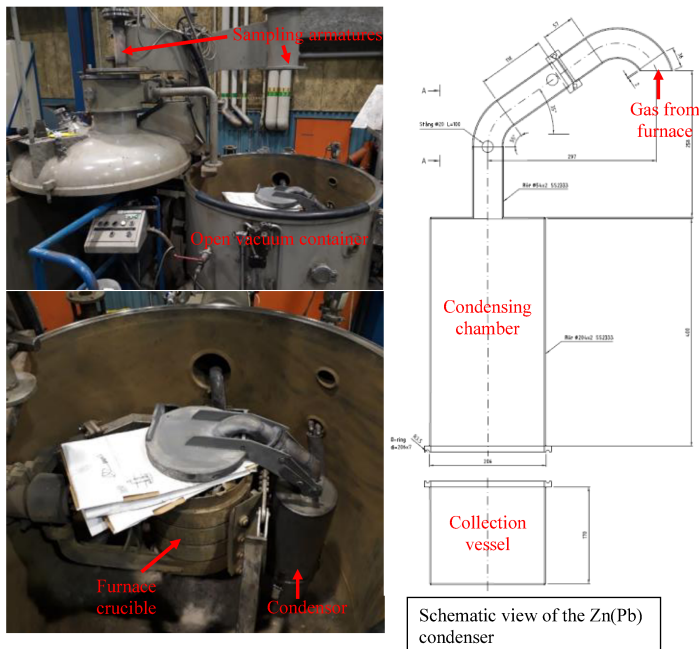


Figure 2. The experimental set-up of the Swerim vacuum induction furnace system



Figure 3 Zn deposits and coatings due to escaped vapour

2.1.2 The main test results

The chemical analyses for the samples from vacuum testing are shown in Table 1. In all cases, the Cu content increased while both Pb and Zn decreased. It is also evident that it is possible to remove all Zn and a significant part of Pb in the solid phase. As is shown for the tests where sufficiently low pressure condition has been achieved (PBV3-6), it became possible to completely separate Zn from the Cu-melt.

The best Pb-removal degree was achieved in the PBV6 test, about 65%, as this test could be operated for the whole test time as planned without any operational problems. A novel and practical approach to efficiently remove Pb has been developed and confirmed though more tests are needed to repeat the test results.

The analysis of the condensed material from tests PBV4-6 is shown in Table 2. The main component was Zn, as expected, with a few percent Pb as well as several other minor elements. These confirmed the removal of zinc and lead.

Table 1 Chemical analysis of the brass/Cu-alloy samples during and after vacuum treatment

Sample	Pb (%)	Cu (%)	Zn (%)
Initial mix (PBV1&PBV3-6)	0.68	90.52	8.80
PBV1-1	0.63	90.58	7.5
PBV1-2	0.71	93.2	4.7
Initial mix (PBV2)	0.46	93.51	6.03
PBV2-1	0.14	96.46	1.12
PBV2-2	0.29	96.50	1
PBV2-3	0.23	95.34	0.91
PBV2-4	0.29	96.70	0.84
PBV2-5	0.25	96.64	0.81
PBV3-1*	0.48	99.09	0
PBV4-1*	0.42	99.43	0
PBV5-1*	0.39	99.41	0
PBV6-1* (0 min)	0.27	99.59	0
PBV6-2 (40 min)	0.25	99.59	0
PBV6-3 (80 min)	0.24	99.59	0
PBV6-4 (120 min)	0.26	99.59	0

*Uses the same initial mix as PBV1, 0 min = when it was melted

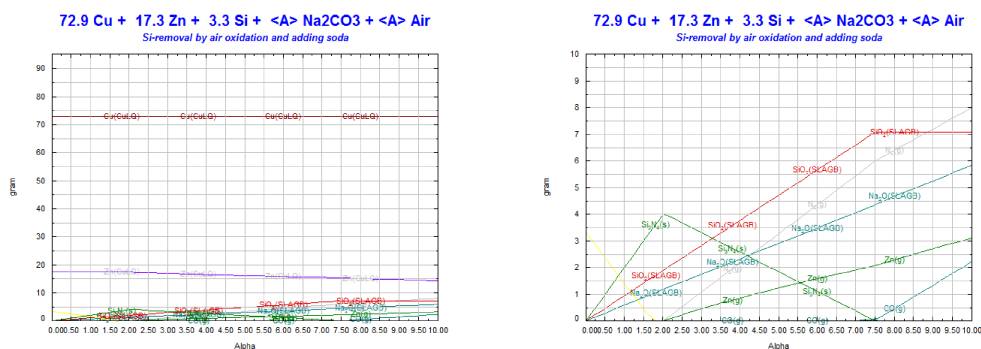
Table 2 Chemical analysis of the collected Zn in the condenser

Sample	Zn	Pb
PBV4-Zn	97.87	1.41
PBV5-Zn	96.35	1.94
PBV6-Zn	98.97	0.93

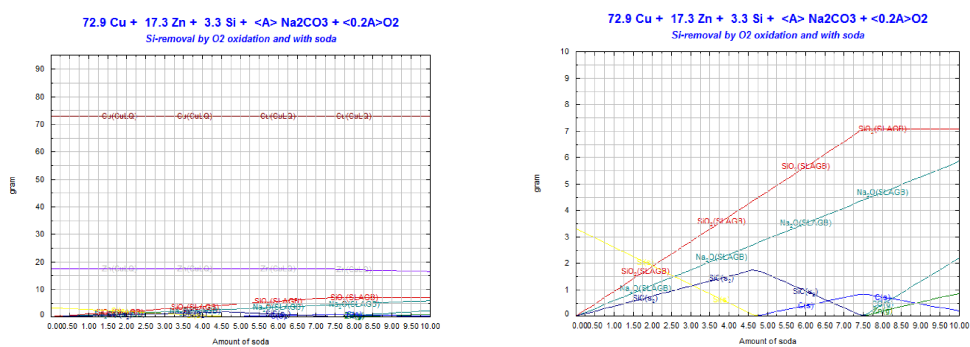
2.2 Si-removal by oxidation

2.2.1 Si-removal by oxidation, thermodynamic modelling

Extensive works on thermodynamic modelling of Si-removal from brass by oxidation using air and O₂ has been performed and some results are shown in the figure below. It is shown that Si-content could easily be oxidized to SiO₂ without oxidation of Zn in the brass. The air and O₂ have been used in the modelling and soda has been added as slag former. Temperature used is about 1050°C. There could be some zinc loss if the temperature is too high, see Figure 4.



Air Oxidation and soda addition



O₂ Oxidation and soda addition

Figure 4 Si-removal by air or pure O₂ oxidation with soda addition

2.2.2 The slag system

Several slag systems have been considered. It was found that the SiO₂-Na₂O based slag as shown in Figure 5 will be the most applicable one for the Si-removal purpose. The melting temperature of the slag should not be higher than 1050°C.

For the experimental tests done at Swerim this slag system has been applied.

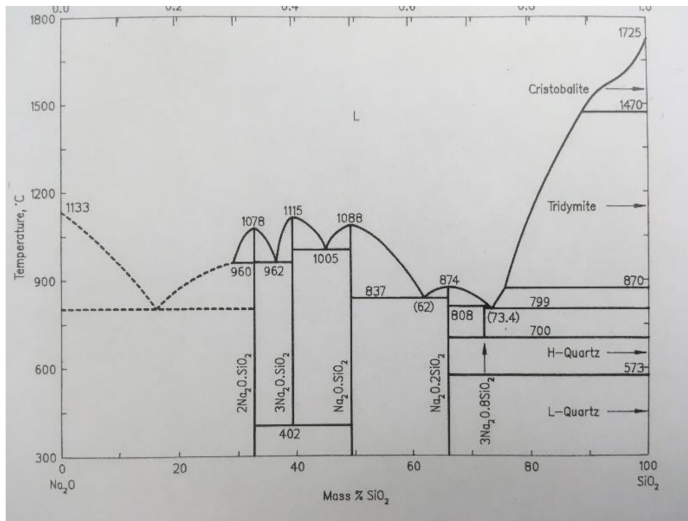


Figure 5 Phase diagram of the $\text{Na}_2\text{O-SiO}_2$ system

2.2.3 The Tamman furnace tests on Si-removal

The experimental set-up of Tamman furnace tests are shown in Figure 6.

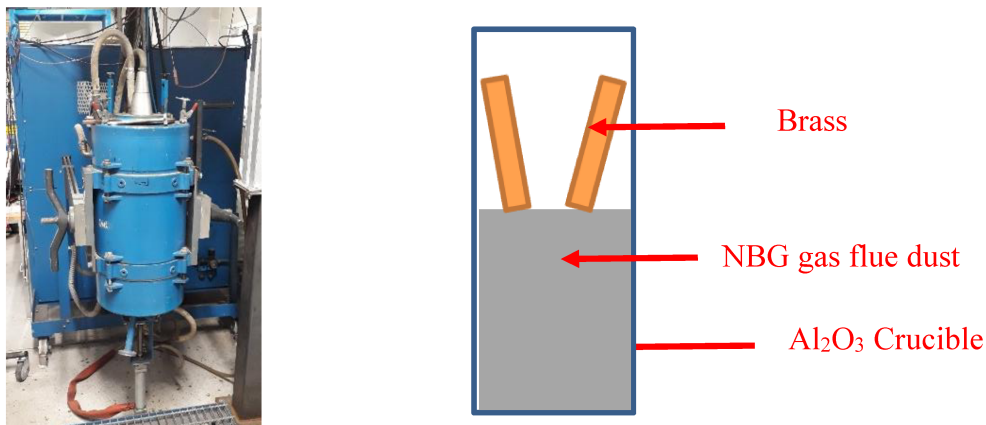


Figure 6 Tamman furnace and crucible contents at test start

Three tests have been performed using NBG dust containing ZnO as oxidation agent and soda as slag former. The aim was to make ZnO reduced by Si and be recovered to the brass, while Si oxidized and removed to the slag forming a low melting point $\text{Na}_2\text{O-SiO}_2$ slag.

The test results are shown in Table 3. As shown, the Si-removal was quite poor. Only Test PBT2 has shown some indication on notable Si-removal. It was concluded that Tamman furnace was too small and not able to provide the needed kinetic conditions to force the NBG dust (ZnO) to react with Si in the molten brass. This is because the dust is very light and will float on the surface of the brass melt. It is difficult to get consistent test result due to the factors mentioned above.

Table 3 Chemical analysis of Tamman furnace metal samples

Sample	Si (%)	Cu (%)	Zn (%)
CW709R	0.70	65.4	31.8
PBT1	0.71	65.5	31.4
CW709R	0.70	65.40	31.8
PBT2	0.59	65.7	31.4
CW724R	3.60	78.00	18.5
PBT3	3.62	76.6	19.3

2.2.4 The pilot tests on Si-removal at Swerim

Based on the poor test results it was decided to test the Si-oxidation tests in a bigger scale using the 150 kg induction furnace at Swerim. If the test results are promising the developed technique will be further verified in the industrial tests.

Two oxidation tests have been performed:

- Oxidation using a mixture of ZnO (NBG dust) and soda as described in the Tamman furnace tests (see previous Chapter)
- Oxidation using a single air lance

2.2.4.1 Induction furnace trials using NBG ZnO-rich dust

A total of four tests were performed in Swerims induction furnace, a schematic of which is displayed in Figure 7.

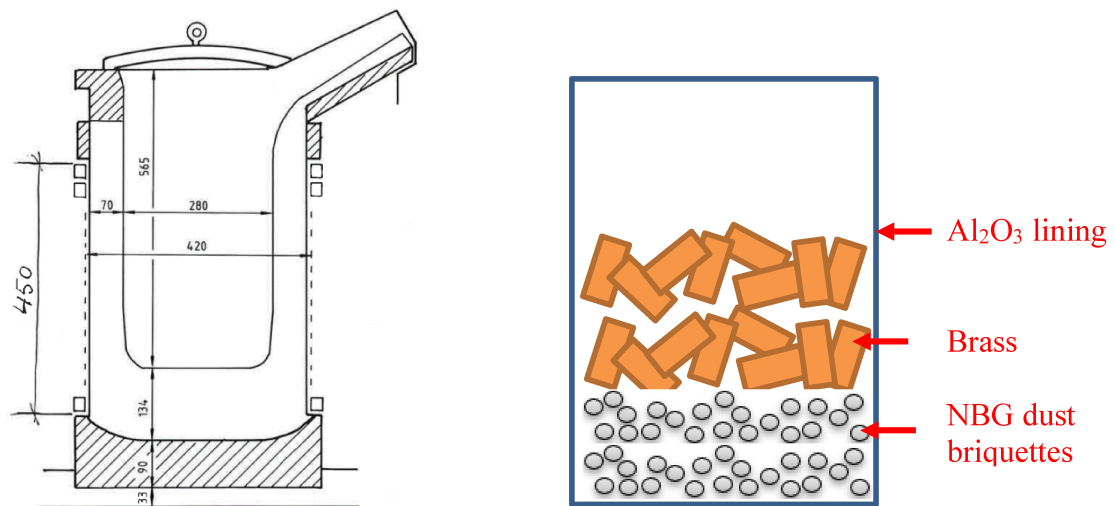


Figure 7 Induction furnace used for the tests, schematic and charging of material

The test plan and the main test results are summarized in Tables 4 and 5.

Table 4 Test plan and recipes for induction furnace trials

Test	Brass (type)	Brass (kg)	Briquettes (type)	Briquettes (kg)
PBI1	CW709R	100	NBG Dust	6.5
PBI2	CW709R	100	NBG Dust + soda	6.5
PBI3	CW724R	94	NBG Dust	6.1
PBI4	CW724R	98.5	NBG Dust + soda	6.4

Table 5 The major component and Si-content in the investigated brass qualities and the test results after treatment using high ZnO dust from NBG

Sample	Si (%)	Cu (%)	Zn (%)
CW709R*	0.70	65.40	31.80
PBI1-4**	0.65	70.18	26.92
CW709R*	0.70	65.40	31.80
PBI2-1	0.60	65.86	31.69
PBI2-2	0.54	67.85	29.65
PBI2-3	0.47	68.59	28.96
PBI2-4**	0.40	69.56	27.86
CW724R*	3.60	78.00	18.50
PBI3-4**	3.57	77.17	18.89
CW724R*	3.60	78.00	18.50
PBI4-4**	3.07	76.60	19.89

*Initial analysis of the brass quality **Final sample after 2h treatment at 1100°C

As shown, it seems that the mixture of soda and the used NBG dust can substantially reduce the Si-content, probably due to more "liquid slag" fraction because of soda addition. For the 2nd test, PBI2, almost half of Si in the brass has been removed. The test results were however not that consistent and it was still difficult to estimate the overall efficiency of the used oxidizing agent, which in turn made it difficult to add a correct amount of soda. The same phenomena of the light ZnO dust fraction (in form of briquettes) quickly floating to the top as soon as brass is melted, was also observed in these trials. A low oxidation efficiency, meaning a large amount of reagent (NBG dust and soda) has to be added, leads to a high slag amount. Due to these uncertainties, it was decided to test oxidation using air as oxidising agent.

2.2.4.2 Induction furnace trials using air injection

Figure 8 shows the simple set-up for air injection into the brass melt. The idea is to oxidise Si in brass to SiO₂ by air.



Figure 8 Induction furnace in operation during air injection tests

A lot of efforts have been made to develop a suitable lance that could sustain the high temperature for >4 hours. Figure 9 shows the lance developed and later used in the industrial scale tests.



Figure 9 Improved lance design

Test results

Table 6 shows the main test results. As shown, for the 2nd test PBL2, about 50% of Si in the brass has been removed. For the 1st test, the blowing time was much shorter due to troubles with the air lance, therefore only limited Si-removal occurred.

Table 6 Chemical analysis of samples from induction furnace trials using air injection

Sample	Si (%)	Cu (%)	Zn (%)
CW724R	3.60	78.00	18.50
PBL1-1	3.53	76.60	19.46
PBL1-2	3.48	77.17	18.95
CW709R	0.70	65.40	31.80
PBL2-1	0.58	66.88	30.28
PBL2-2	0.50	68.32	29.27
PBL2-3	0.50	67.14	29.39
PBL2-4	0.45	67.83	28.57
PBL2-5	0.44	68.35	27.54
PBL2-6	0.38	69.43	26.82
PBL2-7	0.40	69.19	26.80
PBL2-8	0.34	69.46	26.86

Figure 10 shows the Si-content over the air injection time. Based on the promising results shown in the figure, it was decided to use the same approach for the industrial trials.

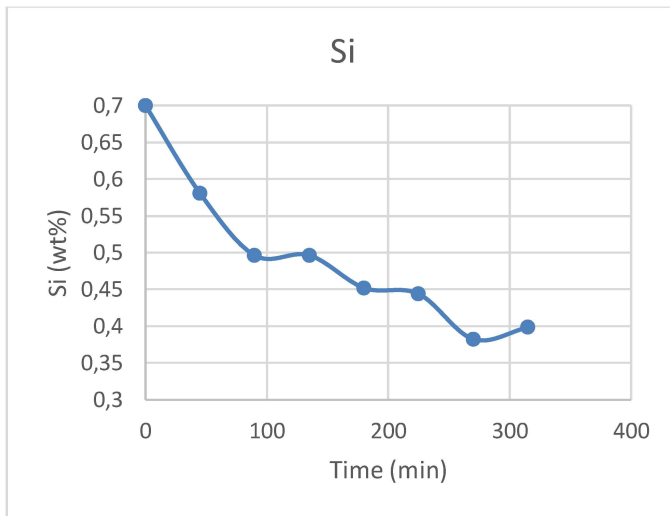


Figure 10 Si content during induction furnace tests with air injection

2.2.5 Industrial scale trials using air injection

For the industrial scale testing, a cart was constructed to hold the lance used for oxygen injection. This enabled easy removal for sampling, deslagging and replacement of the lance. The lances used were so-called "Shinto" pipes, which consist of stainless steel with a ceramic coating of alumina. Figure 11 shows the test setup.

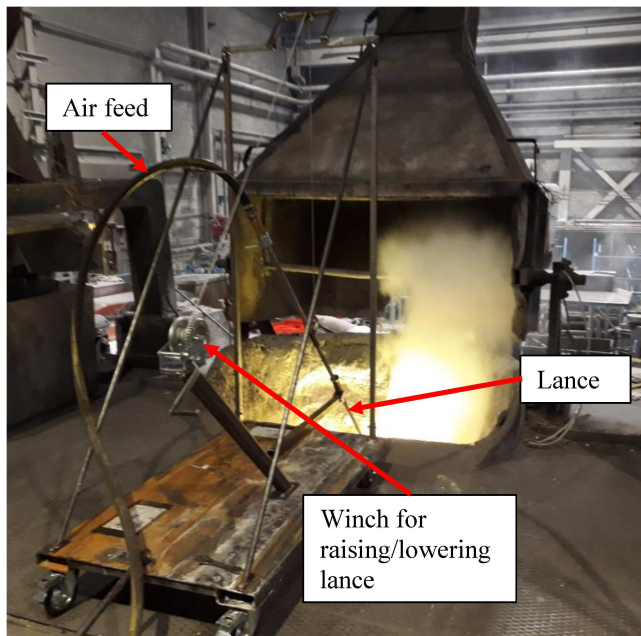


Figure 11 Industrial scale test setup

The test campaign took place over a period of two and half days during day-time shifts. During this time, air blowing took place periodically. The starting point of the test was a brass melt of approximately 10 tons with a composition of 64% Cu, 33 % Zn, 1.7% Pb and 0.7% Si. After initial sampling and temperature measurement, the lance was lowered into the melt, with an initial air flow of ca. 180 NI/min. Blowing proceeded for 60 minutes during which the temperature was measured with 20 minutes intervals. Then, the lance was lifted out of the melt and either shortened or replaced.

Test results

No Si-removal could be observed in the industrial trials, see Figure 12. On the contrary, the Si-content slightly increased over the blowing time. The test results could not be explained from a thermodynamic point of view. It is most likely the slight increase of the Si-content is due to oxidation of Zn by air (about 10% loss of Zn), thereby a reduced total amount of brass and hence a slightly increased Si-content.

The poor oxidation of Si in the industrial trials was probably due to the poor injection of the air, which lead to inadequate stirring as compared to the pilot test trials since the furnace size is much bigger. It is believed that the injected O₂ reacted quickly with Zn (30%Zn in the brass), forming ZnO that floats quickly to the top “slag” without any chance to meet the dissolved Si in the melt, thereby no Si-oxidation occurred and thus no Si-removal.

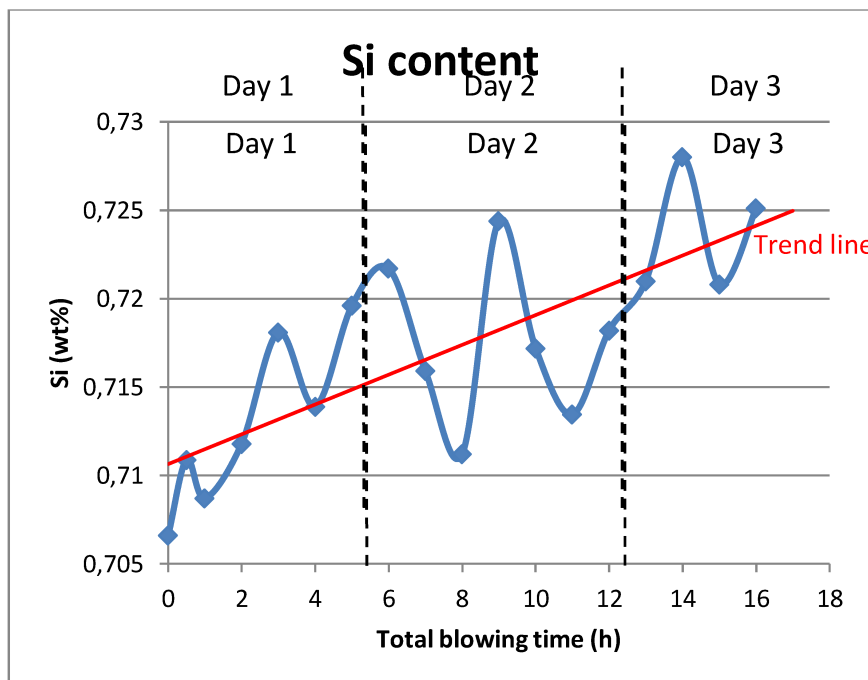


Figure 12 Si content as a function of total blowing time

3. Concluding summary

Based on the obtained test results, several conclusions can be drawn.

- A novel method for Pb-removal by vacuum treatment has been developed and demonstrated in 30 kg scale. It was found that:
 - Pb could be reduced from 0.68% to 0.26%
 - The majority of Pb and all Zn can be removed in the solid phase and during melting
 - Zn is removed before Pb
 - Very low pressure (<10 mbar) is necessary to achieve good separation
 - The process can be controlled using readings for the temperature and pressure of the off-gas
- It is possible to partially remove Si from molten brass by addition of gas flue dust containing ZnO
 - Si removal is improved by addition of Na_2CO_3 as flux
 - It is difficult to achieve good interface area between the molten brass and added $\text{ZnO}/\text{Na}_2\text{CO}_3$ in small laboratory scale
 - Injection of the ZnO -dust/ Na_2CO_3 is possible option, but need to be tested in large scale
- It is possible to remove a significant portion of Si from molten brass by injecting air through a lance, but there are some practical problems
 - The lance must have a ceramic coating to avoid rapid wear
 - The lance must be replaced regularly due to build-up around the lance tip
 - In industrial scale it is difficult to inject enough oxygen through a lance
 - Regardless of scale the processes lead to increased slag and dust formation

For the next step, NBG has the intention to carry out additional tests at Swerim to further verify the developed vacuum treatment technology for Pb-removal from brass via direct contract project with Swerim.

Appendix I Pb-removal by vacuum treatment and Si-removal by oxidation of Brass – final report of the “Pure Brass” Project, MEF19021K, a Swerim report Confidential

Publikationslista

Ingen publikation förutom interna rapporter.

Projektkommunikation

NBG har avsikt att skydda den utvecklade metoden för Pb-rening som har varit huvudfokus för projektet därmed ingen föredrag eller publikation.

Referenser

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Bilagor

- Detaljerade information angående ritningar, fördjupade metodbeskrivningar, resultattabeller etc finns i Appendix (den konfidentiella version av rapporten) som skickas in separat
- Administrativ bilaga