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Innovative Economical Ecotechnology for Refining Intermediate (Bullion) Lead

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***Abstract*— Raw lead resulted from pyrometallurgical procedures includes a series of trace elements like: Cu, As, Sn, Fe, S, noble metals etc., that reduce to a high extent physical-chemical and technological properties of lead. Therefore, removal of such elements is absolutely necessary to acquire a lead with the purity dictated by quality standards. Purification of lead is achieved by hydrometallurgical procedures (thermal refining) or combined, pyro-electro-metallurgical procedures (thermal copper removal and electrolytic refining).**

Researches made by the group of authors resulted in an innovating technology for the thermal refining of lead, with outstanding performances from technological and cost-effectiveness standpoints, also providing a low pollution level.

***Index Terms*—lead, lead alloys, thermal refining, environmental impact.**

I. INTRODUCTION

This technology involves the design of lead alloys and thermally refined lead using a modernized, integrated alkaline refining. This technology is based on Invention Patent no. A/00454/2009, entitled: "Procedure for elaboration of lead alloys and lead using thermal refining". The issue solved by this invention consists in the design of a procedure and a proper plant that allows achievement of lead and lead alloys in cost-effective and environment-friendly conditions superior to those currently used. The technological flow, used by invention, consists in the following steps: loading, gross copper removal, fine copper removal, alkaline refining, zinc removal, correction and casting, and the plant used comprises: refining bowl, mechanical agitator, transfer pump, casting device and burner.The invention consists in refining methods [1] for various temperatures and in dosage of reagents to acquire an optimum efficiency, as follows:

This procedure, as per invention, incurs the following benefits:

- Much lower thermal and electric power costs.
- Acquiring of a thermally refined lead with minimum 99,975% contents that may be used in various industry branches.
- In case of the design of lead alloys, it allows selective removal or preservation of alloy elements, resulting in low production costs.
- Highly reduced pollution level compared to lead acquired by electrolysis [2]

II. PROCEDURE OF ACCQUIRING OF LEAD

The procedure of acquiring of lead, as per invention, comprises the following steps:

III. LOADING

The refining bowl (1) is loaded with liquid or solid raw lead taken from a revolving kiln, or with metallic lead wastes. When the bowl is full, the metallic bath is cleaned of slag and ashes, and the mechanical agitator is installed (2) (fig. 1). The pilot installation is presented in fig. 2.

IV. ZINC REMOVAL

This operation aims to remove zinc below the limits set forth in the technical specifications for thermally refined lead or for the intended lead alloy.

Reagent used: NaOH.
Reagent requirement:
Temperature: 380-400 ºC.
Duration: 25-35 min.
Method of execution:

- The reagent is applied on the surface of metallic bath, with the agitator turned off;
- After the reagent is melted, the agitator is turned on and refining process begins;
- When this operation is complete, the resulting ash is cleaned using the drilled shovel;
- This operation is repeated until zinc is removed below the threshold set forth in technical specifications (below 0,001%).

V. COPPER REMOVAL

A. Gross removal

Copper is intended to be removed down to the threshold of 0,2 % (if the case).

Reagent used: woodchips.
Reagent requirement: 0,8 Kg/ton.
Temperature: 360-380 ºC.
Duration: 45 min.
Method of execution:

- While the agitator homogenizes the melted mass, woodchips are poured, gradually, in the "eye" formed by agitator, at 5-10 min. intervals;
- When the copper crust accumulates in large quantities, it is collected with a drilled shovel and stored in a metallic container, with the agitator turned off;

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- This operation is intended to remove copper down to the threshold of 0,2% (performed only in case of high copper contents).

B. Fine copper removal

Copper is intended to be removed below the threshold set forth for the thermally refined lead or for the lead alloy intended to be acquired.
Reagent used: a mixture of pyrite and sulfur (30% pyrite and 70% sulfur).
Reagent requirement: 2 Kg/ton.
Temperature: 330-340 ºC.
Duration: 35-45 min.
Method of execution:

- First, the ratio between the Cu contents and Sn contents must be assessed, to determine whether additional quantities of tin are required, in addition to tin already present in the metallic bath. This ratio must meet the following condition:

$$Sn_{required}\ [Kg] = [Kg]\ Pb\ in\ bowl \times [\%]\ Cu \times 1{,}3:100$$

- Only if the case, the quantity of tin is added, determined following checks of Cu/Sn ratio as per formula above;
- The two elements of the reagent are prepared (30% pyrite and 70 % sulfur), by mixing them and acquiring an homogenous mixture;
- This mixture is poured into the refining bowl, in portions of app. 2 Kg, in the "eye" formed by agitator, at 5-10 min. intervals;
- Refining is continued until ashes formed acquire a dusty appearance and accumulate in large quantities;
- Then, the agitator is turned off and the metallic bath is cleaned using the drilled shovel;
- Refining is continued until Cu contents drops below the maximum threshold of 0,0015%.

VI. ALKALINE REFINING

This operation removes highly oxidizing elements: As, Sn, Sb and others. This operation is based on the high affinity to oxygen of these three elements compared to lead, in the order shown above. Impurities are removed successively in this order.

a. Arsenic removal

Previous experiments showed that, if tin removal is started at elevated temperatures (in excess of 580 ºC), arsenic removal occurs concomitantly with the tin removal.

b. Tin removal

Reagent used: modified Na NO_3.
Reagent requirement:

$$Na\ NO_{3\ required}\ [Kg] = [Kg]\ Pb\ in\ bowl \times [\%]\ E \times 1{,}1:100$$

(where E is the Sn contents).
Temperature: 600-620 ºC.
Duration: 40-45 min.
Method of execution:

- The reagent (modified Na NO_3) is poured in portions of app. 2-3 Kg, in the "eye" formed by agitator, at 2-3 min. intervals;
- Agitation continues until the resulting ashes show a dusty appearance. Then the agitator is turned off and the metallic bath is cleaned, proceeding as described above;
- Based on the tin contents, tin removal continues until the tin content reaches the value of Sn < 0,0001%.

c. Antimony removal

Reagent used: modified Na NO_3.
Reagent requirement:

$$Na\ NO_{3\ required}\ [Kg] = [Kg]\ Pb\ in\ bowl \times [\%]\ E \times 1{,}1:100$$

(where E is the Sb contents).
Temperature: 580-600 ºC.
Duration: 35-45 min.
Method of execution:

- The antimony removal operation is similar with the tin removal operation;
- This operation is repeated until antimony is removed below 0,0007%.

VII. CORRECTION

A. In case of thermally refined lead, when all elements have been removed below the threshold values set forth in technical specifications, it is ready for casting, ingoting, and stacking, packaging operations. B. In case of the design of lead alloys, after full or partial refining operations, the alloy is corrected with the primary metals or specific pre-alloys, until the results of analysis fall within technical specification provisions.

VIII. CASTING

When thermally refined lead or the lead alloy is ready for casting, the transfer – casting pump (3) is entered into the metallic bath and the lead is cast on the casting device (4). All temperature corrections are made using the methane burner or light fuel liquid burner CLU (5). The casting is done in ingots (fig. 3)

IX. CONCLUSIONS

Novelty of this technology consists in the following:

a. Innovating refinery method for various temperature ranges and proprietary dosing formula for reagents to achieve optimal efficiency, and also a significant reduction of pollution;
b. Sizing of power and capacity of mechanical agitator and of its blades, based on an invention that allows optimal chemical reactions retaining reagents into the metallic bath;
c. Researches made have shown that the use of a mixture of 30 % pyrite and 70 % sulfur achieves highly superior results from the efficiency and refining type standpoints; also it achieves a significant reduction in toxic gas releases [3];

It generates a low quantity of residues compared to other technical procedures currently available, residues that are not stored in waste heaps but that may be reentered in

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production (by melting in revolving kilns), and therefore environmental impact is significantly lower and since no waste heaps are generated (which would incur additional taxes) production costs are lowered;

d. Thermally refined lead, acquired by this technology, may be used for the manufacture of Pb-Ca accumulators, which are maintenance-free accumulators, with a life span 2-3 longer compared to traditional accumulators, which lowers significantly the environmental impact [4];

e. A thermally refined lead may be achieved that can be used in manufacture of plumbing pipes (recent researches have shown development of residual bacteria when AluPEX pipes are used, and reintroduction of lead plumbing is intended).

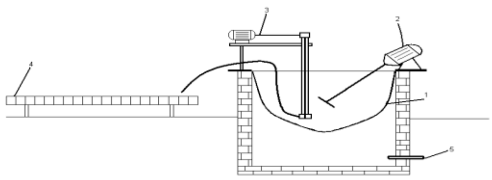

Fig. 1. The plant for execution of the technological process of thermal refining of Pb.

Fig. 2. Refining installation

Fig. 3. The zone of casting in ingots

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AUTHOR'S PROFILE

Adrian Motomancea graduated from the University "Politehnica" of Bucharest, Faculty of Machines Construction Technology, specialty machines-tools, in 1983. Between 1983-1989 he worked as a technical design engineer at the "Titan" Institute of Scientific Research and Technological Engineering for Machine-Tools, Bucharest. He was assistant at the University Polytechnic Bucharest, Mechanics Department between1989-1994 and a lecturer between 1994-1997. He obtained a PhD in Technical Mechanics and Vibrations in 1997 and has been an associate professor starting with 2001.

He has authored 11 specialty books, over 80 articles and presentations in international conferences proceedings and seven patents. He was project manager for more than ten competition-based grants and takes part in a lot of other major national and international projects. He was awarded six gold and silver medals at the Invention Salons of Geneva and Brussels, as well as excellence invention prizes by the Russian Research Ministry and the Slovenian Research Ministry.

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Cristian Apostoloiu graduated from the University "Politehnica" of Bucharest, Faculty of Metallurgy, in 1981. Between 1981-1987 he worked as a technological engineer, coordination of the team activities at SC Neferal SA., Bucharest, one of the greatest Romanian enterprises of non-ferous production. He was Department coordinator between1987-1991 and Production director adviser between 1991-1993. Between 1993-2001 was Technical production director and between 2001-2003, Assistant general director. He became General director in 2003, occupying this position until 20009. From 2009 until now, was general manager of S.C. Prodmed Industrial S.R.L. , a lead production factory.

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Gheorghe Ionescu graduated from the University "Politehnica" of Bucharest, Faculty of Machines Construction Technology, in 1990. Between 1990-2002 he worked as office manager at the S.C. ELCOMP S.A., Bucharest, as Designer of non-standardized equipment and devices. He was technical manager at the S.C. Technosteel Group S.A., Bucharest between 2002-2006, coordinating of design for ball-lead screws, non-standardized equipment and devices. Between 2006-present he was general manager at S.C. Industrial S.R..L. oane of the most important Romanian factory in designing and machine-tools retrofitting. His professional skills focus on design of machine-tools, equipments, and devices.