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# Processing of Sb-Pb-Sn-Containing Materials

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## Abstract

During the processing of lead containing products and polymetallic alloys the recovery of tin and antimony from technology of lead production is carried out by oxidation refining of decopperized lead with rich oxides (Sn, Sb ≥ 20%).Tin oxides are melted in a short-drum furnaces to lead bullion (> 96% Pb) and tin-rich (> 20% Sn) slag. The slag is melted in an ore-smelting furnace to obtain a Sn-Pb alloy of next composition, %: 56.1 Sn, 18.2 Pb, 14.6 Sb, 6.9 As, which is refined by vacuum distillation with production of rough tin (Sn ≥ 90%). The additional profit of rough tin obtainment (~310 tons/year), compared with sales of tin slag, is about ~1.3 million $/year.

**Keywords:** lead, tin, antimony, melting, vacuum distillation.

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For the processing of lead-containing industrial products and polymetallic alloys leaching, electrolysis, roasting and melting are widely used. But these technologies have significant disadvantages, such as: high specific consumption of reagents and energy carriers; necessity of a waste water disposal and a complex scheme of electrolytic slime processing; high toxicity of electrolyte; low availability of qualified personnel; large investments and specific financial costs; necessity of a developed scheme of instrumental and remote control; low specific productivity of technological equipment; large losses of metals (lead, gold, silver) and low quality of purification during pyrometallurgical refining. Analysis of modern technological processes of lead, antimony and tin production showed that in comparison with common methods of separation and refining of base metals from the impurities, the vacuum distillation of polymetallic alloys and related industrial products of lead production is technologically acceptable and economically feasible, allowing the obtainment of commercial products of the required quality [1–8].

In the branch "Proizvodstvo splavov cvetnyh metallov" ("Production of non-ferrous metal alloys", PNFMA) of JSC "Uralelectromed" the output of tin and antimony from the technology of lead production was carried out at the first stage through the oxidative refining of lead (~130 tons) (see Figure 1). To do this, the melt was heated up in a refining boiler to ~560 °C and then purged by oxygen and compressed air mixture with ratio of

30:50 $m^3$/h during 1 hour at interval of 2-2.5 hours. During this process the temperature changed within range of 540–860 °C. The purpose of oxidative refining is to obtain rich oxides with a content of antimony and tin of at least 20%. On the surface of the lead bath floats (oxides) of tin, arsenic and antimony in the form of powder, granules or dough-like mass were consistently accumulated with the following composition, %: 45–70 Pb; 15–30 Σ(Sb, Sn, As). The separation of tin, arsenic and antimony during the oxidative refining is only partially possible: the yield of tin oxides was 1.2–1.4 times greater than antimony. At the initial content of antimony and tin in lead within range of 3.5–3.9% rich oxides containing 20–30 % of tin were obtained. The transition of tin to oxides was 76–80% of their total amount.

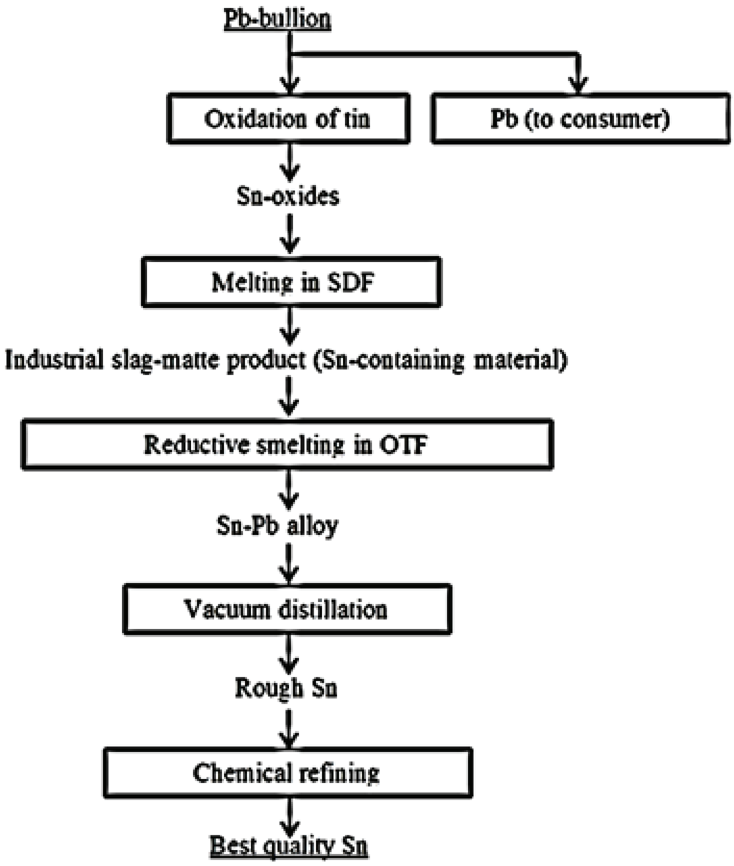


**Figure** 1: Conceptual diagram of production of tin and its alloys. Author's own work.

At the second stage tin oxides (above 30 tons) derived from the oxidative refining of lead were melted in short-drum furnaces (SDF) to produce rough lead with a minimal content of impurities and industrial slag-matte product (ISMP) rich in tin and suitable for further processing (or for sale). Rough lead of a high quality (96.4% of Pb) with an output of 99.3% of the initial metal content and ISMP with ~21% of tin suitable for sale were obtained after melting of charge containing 60% of tin oxides, 24% of soda ash, 14.8% of sand and 1.2% of coke breeze. At the end of the process the melt was released from the SDF and then crude lead and ISMP were drained, cooled and crushed. Technologies of lead refining and oxides melting were developed on an industrial scale and implemented in PNFMA branch of JSC ”Uralelectromed”.

Until recently the ISMP has been successfully realized to consumers as a tin containing material, however, it was obvious that increasing the content of tin in pure metal commodity products is advisable and rational option. For this purpose charge (~74 tons) containing 85% of ISMP and 15% of coke breeze was processed in ore thermal furnace (OTF) for 3 days at the third stage (in an industrial scale) in the presence of liquid slag of the following composition, kg/%: 1400/40 $SiO_2$; 1050/30 CaO; 1050/30 $Na_2CO_3$. The highest temperature (1500 °C) was observed at the area adjacent to the surface of electrodes. The release of melting products from the OTF was carried out from the level of 800–1100 mm. Following products were consistently drained from the OTF:

~ 24.0 tons of slag composed of, % 0.28 Pb, 6.8 Sn, 0.8 Sb, 1.3 As;

~ 19.2 tons of Sn-Pb composed of, %: 18.2 Pb, 56.1 Sn, 14.6 Sb, 6.9 As.

The actual energy consumption was ~62.5 kW per hour and the electrode consumption was ~890 kg (5 pieces). As a result, the average tin content in Sn-Pb alloy was 56.1%, while the maximum content in the middle of the operation was 61.6%. Slag obtained from the OTF with 6.8% of tin can be sold or treated for reducing the tin content to 0.5–1% with further disposal. Tests for production of Sn-Pb alloy were subsequently implemented on an industrial scale.

At the fourth stage vacuum distillation of Sn-Pb alloy (with 55-60% of tin) was carried out in a pilot scale with the purpose to obtain a product with higher consumer properties, namely with a high content of tin (at least 90%). The principal feasibility and practicability of the oxidizing melting of tin slags into Sn-Pb alloy has been confirmed. Production of rough tin by vacuum distillation for determination of possibility of rough tin obtainment with the recovery of individual arsenic, antimony and lead products (sublimates) was tested with 81.2 kg of tin alloy of the following composition, %: 30.6 Pb, 50.4 Sn, 11.1 Sb, 4.3 As, 1.05 Cu. The modes of distillation of tin alloy are given in Table 1.

Table 1: The modes of distillation of tin alloy. Author’s own work.

| Stage | Temperature, °C | Duration, sec | Pressure, Pa |
|---|---|---|---|
| 1 | 600–800 | 900–7200 | 80–60000 |
| 2 | 1100–1300 | 1800–5400 | 0.2–20 |

Following products were received:

– 50,2 kg of rough tin (extraction of Sn > 99%) composed of, %: 94.5–97.1 Sn, 0.024–0.3 Pb, ~1.0 Sb, 0.01–0.03 As, 1.5–1.7 Cu;

– 1.4 kg of 1st distillate with 40.5% of As (extraction – 35.1%);

– 28.2 kg of 2nd distillate (Sb-Pb) wit the content/extraction, %: 5.4–8.0/43–87 As, 57–79/78–99 Pb, 8.5–26.5/25–99 Sb, 1.2–9.1/0.9–8.5 Sn.

On the basis of the conducted researches the balance scheme (see Table 2) and the technological scheme with vacuum distillation of Pb-Sn alloys were made (see Figure 2).

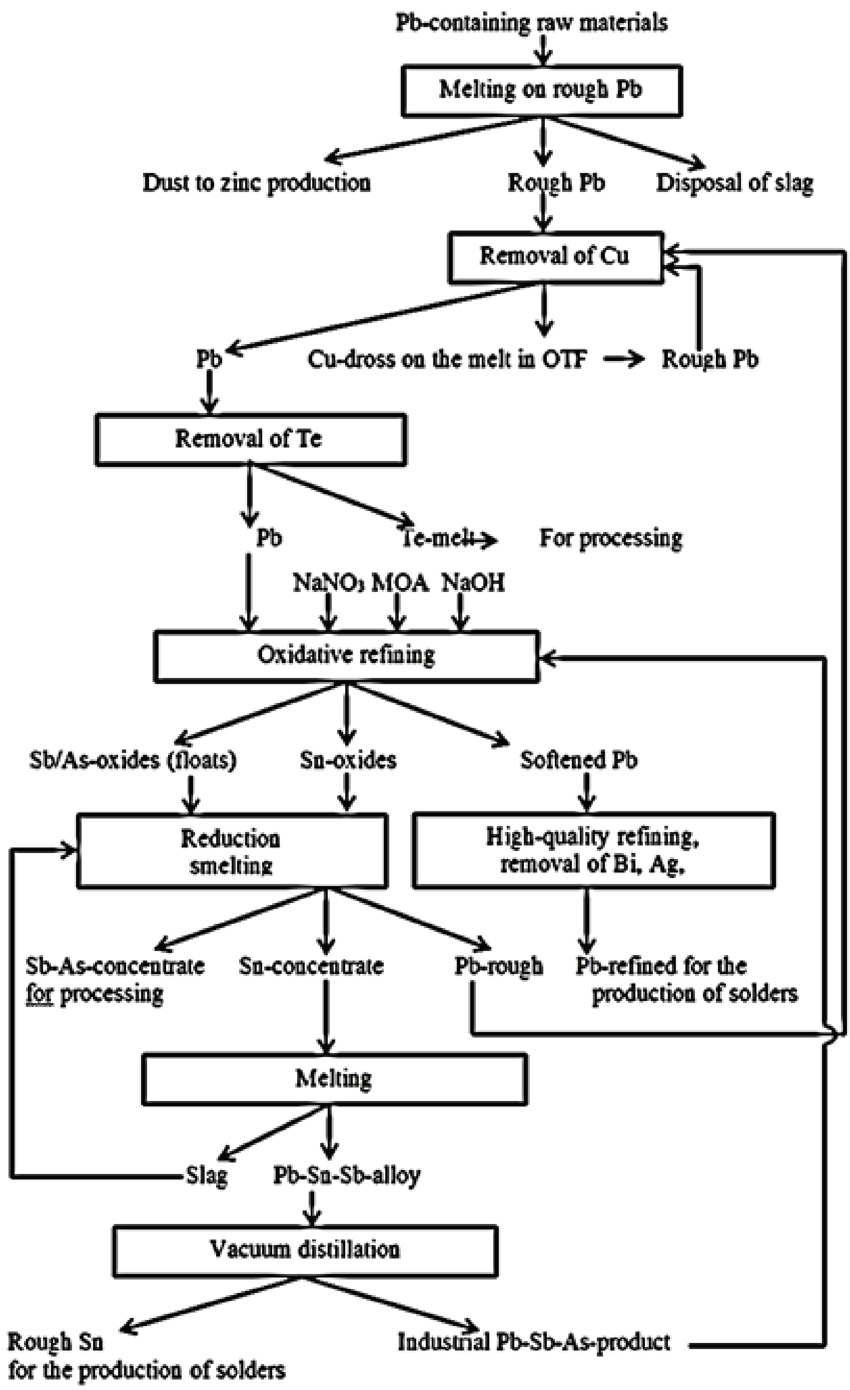


**Figure** 2: Technological scheme of processing of lead-containing material. Author's own work.

As a result of processing of Pb-containing raw materials according to the scheme of oxidative refining from antimony and tin using vacuum distillation, a commercial product with high consumer properties is obtained – tin rough, which can be used for the preparation of branded solders.

Table 2: Contents/distributions of elements during the processing of tin oxides. Author's own work.

| Raw material, industrial product | Content (%) / Distribution (%) | | | |
|---|---|---|---|---|
| | Sn | Sb | As | Pb |
| **Melting on rough Pb** | | | | |
| *Input* | | | | |
| Sn-oxides (1577.2 tons/year) | 21.3/100 | 4.5/100 | 2.6/100 | 60/100 |
| *Output* | | | | |
| Rough Pb (797 tons/year) | 2.78/6.60 | 3.77/42.40 | 0.38/7.32 | 87.2/73.44 |
| Sn-concentrate (1279 tons/year) | 24.29/92.46 | 3.12/56.17 | 2.65/82.80 | 16.48/22.28 |
| Dust (112.6 tons/year) | 2.8/0.94 | 0.9/1.43 | 3.6/9.88 | 36.0/4.28 |
| **Processing of Sn-concentrate** | | | | |
| *Input* | | | | |
| Sn-concentrate (1279 tons/year) | 24.29/100 | 3.12/100 | 2.65/100 | 16.48/100 |
| *Output* | | | | |
| Sn-containing alloy (404.2 tons/year) | 51.11/87.2 | 6.81/90.55 | 3.36/52.43 | 38.61/97.07 |
| ISMP (769 tons/year) | 5.31/12.8 | 0.50/9.45 | 2.16/47.57 | 0.83/2.93 |
| **Vacuum distillation of Sn-containing alloy** | | | | |
| *Input* | | | | |
| Sn-containing alloy (404.2 tons/year) | 51.11/100 | 6.81/100 | 3.36/100 | 38.61/100 |
| *Output* | | | | |
| As-distillate (21.3 tons/year) | 0.6/0.05 | 3.4/2.0 | 38.0/45.45 | 37.6/3.91 |
| Sb-Pb-distillate (153.2 tons/year) | 1.68/1.75 | 11.7/91.76 | 3.4/54.1 | 69.03/95.54 |
| Rough Sn (237.4 tons/year) | 94.5/98.2 | 0.8/6.24 | 0.03/0.45 | 0.4/0.55 |

For processing of Pb-Sn alloy a possible scheme was proposed in which temperature and pressure values can be adjusted depending on the requirements for the composition of the vacuum distillation products (see Figure 3).

As a possible option for conducting the technological process, the use of an induction electric furnace SKB-6087 is considered. The use of vacuum distillation allows to obtain rough tin (90–99% Sn) and collective distillate of impurities (90–99 % $\sum$(As, Sb, Pb) of different compositions depending on sublimation temperature (1343–1873 K) at fixed pressure (~1 Pa) and duration (1.5–2 hours) of the process (see Figure 3).

Economic efficiency of processing of lead containing raw materials using vacuum distillation of Pb-Sn-alloy (50.4% Sn, 30.6% Pb, 11.1% Sb, 4.3% As) is due to the production

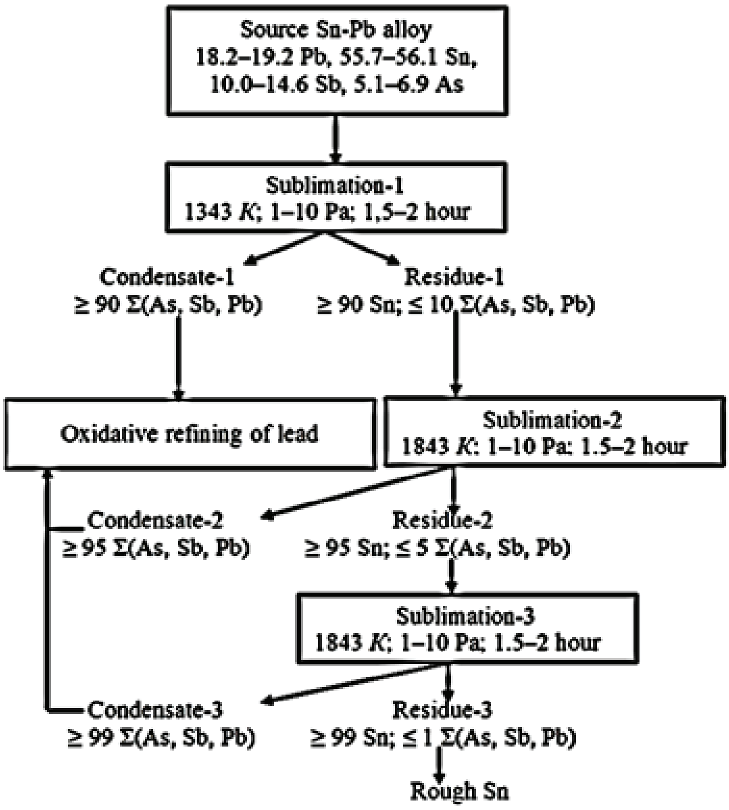


**Figure** 3: Schematic diagram of vacuum distillation of Sn-Pb alloy. Author's own work.

of a purer and therefore more expensive product – rough tin (Sn $\geq$ 90 %). Despite the additional costs (~1.1 million $) for a complex of vacuum distillation equipment for the removal of impurities the increase in additional profit from the obtainment of rough tin (~310 tons/year) in comparison with sales of Sn-concentrate (slag), is about ~1.3 million $/year, which can ensure the return of investments during one year.

## References

[1] Berman, A. (1985). *Total Pressure Measurements in Vacuum Technology*. New York: Academic Press, p. 380.

[2] Winkler, O. and Bakish, R. (1971). *Vacuum Metallurgy*. Amsterdam: Elsevier, p. 237.

[3] Jia, G. B., Yang, B. and Liu, D. C. (2013). Deeply Removing Lead from Pb-Sn Alloy with Vacuum Distillation. *Transactions of Nonferrous Metals Society of China*, vol. 23, issue 6, pp. 1822–1831.

[4] Wang, A., *et al*. (2014). Process Optimization for Vacuum Distillation of Sn-Sb Alloy by Response Surface Methodology. *Vacuum*, vol. 109, pp. 127–134.

[5] Dai, Y. N. (2009). *Vacuum Metallurgy of Nonferrous Metal*s. Beijing: Metallurgical Industry Press, p. 72.

[6] Yang, B., *et al*. (2015). Recycling of Metals from Waste Sn-based Alloys by Vacuum Separation. *Transactions of Nonferrous Metals Society of China,* vol. 25, issue 4, pp. 1315–1324.

[7] Liu, D. C., *et al*. (2012). Research on the Removal of Impurities from Crude Nickel by Vacuum Distillation. *Physics Procedia*, vol. 32, pp. 363–371.

[8] Dai, Y. N. and Yang, B. (2000). *Non-ferrous Metals and Vacuum Metallurgy.* Beijing: Metallurgical Industry Press, p. 40.