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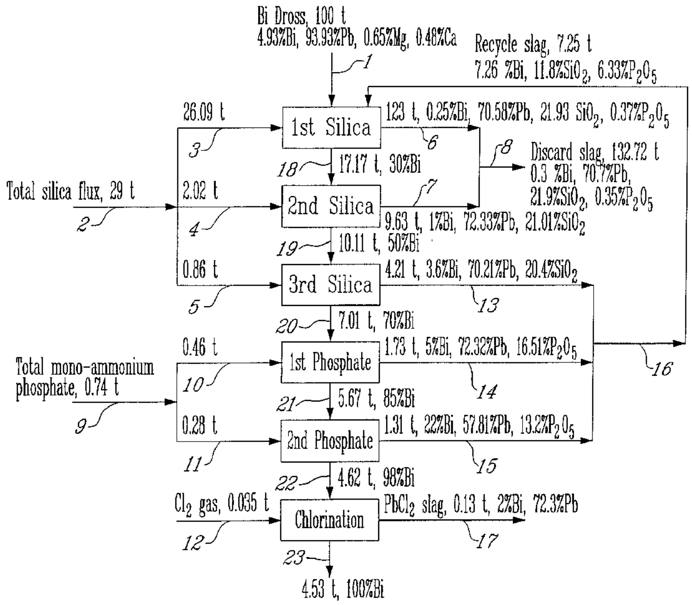
(54) Title: PROCESS FOR BISMUTH RECOVERY



(57) Abstract: A process for recovering bismuth, from lead-bismuth containing material, which is characterized by a series of steps of selective oxidation of lead from said material with the addition of at least one slag forming agent which produces a lead-enriched bismuth-poor slag and an enriched-bismuth product. The slag forming agents are chosen from a group which selectively stabilize Pb while rejecting Bi at the oxygen activity levels required to stabilize the Pb. Typically the process includes a pretreatment process where calcium and magnesium content of the lead-bismuth containing material is reduced and an post-treatment where chlorine is added to remove the residual lead, and obtain a high purity bismuth metal.

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PROCESS FOR BISMUTH RECOVERY

TECHNICAL FIELD

The field of the invention pertains to a process for recovering bismuth from lead-bismuth dross by the selective oxidation of lead with little oxidation of bismuth. The enrichment of bismuth by the present invention minimizes the necessary chlorine gas consumption used for the final lead removal process step needed to attain almost pure bismuth metal (+99%).

BACKGROUND ART

In lead refining, the Kroll-Betterton Process is used to remove bismuth from molten lead bullion. This process utilizes calcium and magnesium metals to react with bismuth to form the solid intermetallic compound, i.e., $CaMg_2Bi_2$, which rises to the surface of the molten lead bullion. The solid debismuthizing dross is subsequently skimmed from the surface of the molten lead bullion and contains from 2 to 15 wt.% Bi, with residual Mg and Ca but predominantly Pb making up the balance.

The debismuthizing dross is heated to form a semi-finished product comprised of a liquid lead-bismuth alloy containing from 2 to 15 % bismuth and a dry slag or crust which is separated. The semi-finished product is often treated to recover bismuth by slagging off the lead. Current industrial practice utilizes sodium nitrate, $NaNO_3$, to increase the bismuth content of the semi-finished product to roughly 30 wt.%. This process is then followed by chlorine gas treatment to remove all the lead remaining entrained in the semi-finished product. Lead chloride is preferentially formed to achieve high purity bismuth metal.

The stringent environmental regulations concerning chlorine and chloride emissions, as well as concerns relating to the lead chloride slag disposal have contributed to interest in increasing the Bi content in the semi-finished product obtained from the debismuthizing dross while reducing chlorine use. A final chlorination step will always be required to remove the final vestiges of Pb from Bi but the treatment of the semi-finished product by chlorine gas requires considerably less chlorine and greatly reduces the amount of lead chloride residue that must be disposed of.

US patents 4,039,322 and 4,039,323 describe a method of upgrading debismuthizing dross by hot vacuum filtering followed by auto ignition of the filter cake. The major problem of the technology is the complexity of the system and sensitivity of the filtering process on the physical and chemical properties of the dross.

US Patent 5,234,492 describes an improved process for the partial removal of lead from debismuthizing dross. This process involves preferential oxidation of the lead over bismuth by cupellation to form a slag until the level of bismuth in the upgraded dross reaches 45 -

wt.%, preferably 30-wt.%, and most preferably between 15 to 25-wt.%. The main problem of this described oxidation process is the excessive bismuth oxidation when the bismuth content of the upgraded dross exceeds 30-wt.%. In order to reduce bismuth losses to the slag, the above process is best used up to only 20 wt.% Bi in the upgraded by the process of USP 5,234,492. Consequently, this process still requires a considerable amount of chlorine to complete the lead removal process. For 100 kg of Pb-Bi upgraded dross, 80 kg. of lead remain, at least 27 kg of Cl_2 are required while producing 107 kg of $PbCl_2$ residue. While using enrichment according to the process of the present invention (See Figure 1, Feed to the final Unit Operation, Chlorination) for 100 kg of Pb-Bi less than 1.5 kg of lead remain in the form of upgraded Bi alloy, which requires 0.51 kg. of chlorine, and produces only 2.0 kg of lead chloride. In this case, some fifty times less chlorine is used with some fifty time less lead chloride residue produced.

<u>**DISCLOSURE OF THE INVENTION**</u>

It is the object of this invention to provide an improved process for bismuth recovery and refining from lead-bismuth containing materials, through the selective oxidation of lead without the loss of bismuth through oxidation to the slag.

It is a further object of this invention to significantly reduce the chlorine required in removal of lead from a bismuth-enriched product to obtain a high purity bismuth metal while producing a lead chloride residue.

In accordance with one embodiment of the invention there is provided a process for recovering bismuth, from lead-bismuth containing material, which is characterized by a selective oxidation of lead from said material with the addition of at least one slag forming agent which produces a lead-enriched bismuth-poor slag and an enriched-bismuth product.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1. Illustrates a block diagram and typical mass balance of the process of the invention.

Fig. 2 Illustrates the Bi solubility of slag in equilibrium with Bi-Pb alloy at 950-1000 °C.

DESCRIPTION OF THE PREFERRED EMBODIMENT

The present invention applies to any suitable lead-bismuth containing material such as debismuthizing dross from a Kroll-Betterton process, lead-bismuth alloy and lead electrolytic refining anode slimes which are rich in lead and bismuth etc. For the sake of convenience, the process will be described considering debismuthizing dross obtained from a lead refining process as the raw material.

Although it is not necessary, a pretreatment is preferable to first remove calcium and magnesium from the dross by conventional techniques with the addition of air, oxygen or $NaNO_3$, where $NaNO_3$ addition is a preferred embodiment. The resulting material produced after calcium and magnesium removal is defined as a lead-bismuth semi-finished product, and still considered a lead-bismuth containing material.

The semi-finished product usually contains 2 to 12 wt.% bismuth or higher, but preferably 4 to 7 wt.% Bi, with the balance essentially lead. The semi-finished product is charged to a suitable furnace or vessel where the semi-finished product is melted if necessary and where air or oxygen enriched air is introduced to the charge to react preferentially with lead to form a lead oxide containing slag which is removed either continuously or at desired intervals by tapping, skimming etc. The key feature of the present process is to add specific slag forming agents which, effectively reduce PbO activity in slag during the oxidation process. The slag forming agents effectively lower the oxygen activity at play during the process and hence lower the bismuth losses to the slag.

A slag forming agent is also defined as a flux. These slag forming agents, are not limited to but are typically SiO_2, P_2O_5, B_2O_3 or GeO_2 effectively reduce the PbO chemical activity. SiO_2 and P_2O_5 are preferred embodiments of slag forming agents. The flux also affects the melting point of the slag. Consequently, the operating temperature range of the furnace or vessel can be wide, but must be sufficient to keep the slag fluid. The Table 1 lists some practicable temperature ranges for typical systems of the invention:

Table 1. Feasible operating temperature range (°C)

$PbO-SiO_2$	750 ~ 1000
$PbO-B_2O_3$	600 ~ 1000
$PbO-P_2O_5$	900 ~ 1000
$PbO-B_2O_3-SiO_2$	600 ~ 1000
$PbO-SiO_2-P_2O_5$	750 ~ 1000

Although it is possible to operate the furnace out side the recommended temperature range, from 500 to 1200 °C, for the systems listed in Table 1, it should be noted that too high an operating temperature may increase refractory wear of the vessel and too low a operating temperature may increase the viscos ity of the slag and hence increase the metal loss due to physical entrainment.

The flux composition and addition rate can be varied over a wide range, but the following factors may be taken into consideration for its optimal control:

Bismuth loss in slag
Refractory wear of the vessel or furnace
Amount of slag and cost of the flux

Furthermore, the flux composition and addition rate may be varied according to the bismuth grade in the lead - bismuth semi-finished product required.

By way of example, when the present process is applied to the treatment in a conventional cupel furnace of a lead-bismuth semi-finished product containing 5 wt.% Bi, a batch operation is preferred. This makes it feasible to use silica flux for to upgrade an initial lead-bismuth semi-finished product, to over 60 % wt. Bi but preferably above 70 wt.% Bi. This assures the utilization of low cost silica flux for the major portion of lead removal.

However, as Bi content increases in lead-bismuth semi-finished product, the bismuth transfer to the slag increases to a point where SiO_2 can no longer be used as a flux without incurring high bismuth losses. It is then preferred to use P_2O_5 containing material. The P_2O_5 flux is added in the form of a phosphate (PO_4) which thermally decomposes to P_2O_5 in the furnace. A preferred embodiment of the phosphate flux being ammonium mono-phosphate. The product produced, after the final addition of the fluxes is defined as the bismuth-enriched product.

A mixture of SiO_2 and P_2O_5 and/or B_2O_5 can also be added to effectively reduce the PbO activity in slag. Removal of lead from bismuth-enriched product, whose concentration of bismuth can attain over 80 wt.% Bi, and preferably more than 90 wt.% Bi, requires much chlorine for the final treatment than the conventional process which has a much heavier lead load to be removed by chlorine.

To further reduce the overall bismuth loss from the process, the slags produced after the final flux addition and secondary silica flux additions, can be recycled back to the first stage of the oxidation process. Figure 1 presents an example of a flowsheet showing typical mass flowrates and compositions of the process streams in an embodiment of the invention.

The flowsheet for the bismuth recovery process of the invention as represented in Fig. 1 includes the following sequential process steps: three silica fluxing unit operations, two phosphate fluxing unit operations and a polishing chlorination unit operation.

Silica fluxing Unit Operations 1, 2 and 3

In stream 1, 100 tonnes of Bi dross raw material (4.93% Bi, 93.93% Pb, 0.65% Mg, 0.48% Ca) enters the first silica fluxing unit operation and is combined with 26.09 tonnes of silica, stream 3. A recycle slag stream 16, of 7.25 tonnes (7.26% Bi, 11.8% SiO_2, 6.33% P_2O_5) is also added in this unit operation. These three streams combine to produce 123 tonnes of slag, stream 6 (0.25% Bi, 70.58% Pb, 21.93% SiO_2, 0.37% P_2O_5) and a product stream 18, of 17.17 tonnes containing 30% Bi.

Stream 18 enters the second silica fluxing unit operation where it is combined with stream 4 of 2.02 tonnes of SiO_2 to produce stream 7, of 9.63 tonnes of slag (1% Bi, 72.33% Pb, 21.01% SiO_2) and a product stream 19, of 10.11 tonnes 50% Bi. Stream 6 and 7 when combined produce stream 8, of 132.72 tonnes of slag to be discarded (0.3% Bi, 70.7% Pb, 21.9% SiO_2, 0.35% P_2O_5).

Stream 19 enters the third silica fluxing unit operation where it is mixed with stream 5, of 0.86 tonnes of SiO_2. The unit operation produces stream 13, 4.21 tonnes of slag (3.6% Bi, 70.21% Pb, 20.4% SiO_2) and a stream 20 containing 7.01 tonnes of 70% Bi. The total SiO_2 flux requirement, stream 2, for the process as described in Fig. 1 is 29 tonnes (the sum of streams 3, 4, and 5).

Phosphate fluxing Unit Operations 1 and 2

0.46 tonnes mono-ammonium phosphate stream 10, enters the first phosphate fluxing unit operation where it is reacts to produce P_2O_5, combine with stream 20. 1.73 tonnes of stream 14 are produced (5% Bi, 72.32% Pb, 16.51%P_2O_5), along with 5.67 tonnes of product stream 21 (85% Bi).

Stream 21 is fed to the second phosphate fluxing unit operation where it is reacted with stream 11, containing 0.28 tonnes of mono-ammonium phosphate, to produce 1.31 tonnes of stream 15 (22% Bi, 57.81% Pb, 13.2% P_2O_5) and 4.62 tonnes of stream 22 (98% Bi). The streams 13, 14, and 15 from the third SiO_2 fluxing unit, the first and second phosphate fluxing units respectively are combined to produce recycle slag stream 16 which is returned, as has been previously described, to the first SiO_2 fluxing unit. The total amount of mono-ammonium phosphate added stream 9, is 0.74 tonnes, (the sum of streams 10 and 11).

Chlorination

In the final unit operation of chlorination, stream 22 is reacted with 0.035 tonnes of chlorine gas (stream 12) to produce stream 17, of 0.13 tonnes a $PbCl_2$ slag (2% Bi, 72.3% Pb) and 4.53 tonnes of the very pure Bi product stream 23 (≈100% Bi).

It should be noted that there is a small discrepancy in the total mass flow inputs and outputs represented in Figure 1, because the outputs contain oxygen which was not reported in the inputs.

The chemical equilibrium equation between molten Bi-Pb alloy and molten slag in an oxygen oxidation process may be presented as:

$$(BiO_{1.5})_{slag} + 1.5[Pb]_{alloy} = 1.5\ (PbO)_{slag} + [Bi]_{alloy} \quad (1)$$

The representation of bismuth oxide as $BiO_{1.5}$ instead of Bi_2O_3 in slag is a result of careful thermodynamic study of bismuth oxide in slag (Akira Yazawa, Shigeatsu Nakazawa and Youichi Takeda, Distribution behaviour of various elements in copper smelting systems, Advances in Sulfide Smelting, Edited by H. Y. Sohn, D.B. George and A. D. Zunkel, Vol.1, pp. 99-117, Proceedings of the 1983 International Sulfide Smelting Symposium and the 1983 Extractive and Process Metallurgy Meeting of the

Metallurgical Society of AIME held in San Francisco, California, November 6-9, 1983). The same study has concluded that the removal of lead from molten copper is much more effective with silicate slag than with calcium ferrite slag. However, the removal of bismuth from copper shows little difference between silicate and calcium slag. This suggests that the activity coefficient of $BiO_{1.5}$ in slag is relatively constant with regard to different slag chemistry (acid and basic slag). However, the activity coefficient of PbO can be greatly reduced by addition of acidic compounds such as P_2O_5, SiO_2 (*W.F. Caley and C.R. Masson, Can. Met. Quart., pp.359-365, 1976*) and B_2O_3 (*M. L. Kapoor and M.G.Frohberg, Can. Met. Quart., pp.137-146, 1973*) or any mixture of them.

The unexpected feature of this invention, is that the addition of the suggested fluxes, allows the Pb to be oxidized without excessive oxidation of the Bi or explained differently, the bismuth equilibrium solubility in slag is greatly reduced at the level of oxygen activity now required to perform the preferential oxidation of the lead. This makes it possible to preferentially oxidize lead from bismuth-lead materials to a much higher concentration of Bi than before, with little oxidation of bismuth into slag. The following theoretical analysis is given by way of example.

The equilibrium distribution of bismuth between molten $PbO\text{-}BiO_{1.5}$ slag and Pb-Bi alloy can be represented as:

$$K = \frac{X_{Bi}\gamma_{Bi}(1 - X_{BiO_{1.5}})^{1.5}\gamma_{PbO}{}^{1.5}}{(1 - X_{Bi})^{1.5}\gamma_{Pb}{}^{1.5}X_{BiO_{1.5}}\gamma_{BiO_{1.5}}}$$

Where:

K is the equilibrium constant of reaction (1)

X_{Bi} is the mole fraction of bismuth in bismuth-lead alloy.

γ_{Bi}, γ_{Pb} are the activity coefficients of bismuth and lead in bismuth-lead alloy.

$X_{BiO1.5}$ is the mole fraction of bismuth oxide in $PbO-BiO_{1.5}$ slag.

$\gamma_{BiO1.5}$, γ_{PbO} are the activity coefficients of bismuth oxide and lead oxide in $PbO-BiO_{1.5}$ slag.

The equilibrium constant, K, at 950 °C is estimated to be 16. Based on the readily available activity coefficient of lead and bismuth in lead-bismuth alloy and $\gamma_{BiO1.5} = 0.6$ as reported by Yazawa etc. (Akira Yazawa, Shigeatsu Nakazawa and Youichi Takeda, Distribution behaviour of various elements in copper smelting systems, Advances in Sulfide Smelting, Edited by H. Y. Sohn, D.B. George and A. D. Zunkel, Vol.1, pp. 99-117, Proceedings of the 1983 International Sulfide Smelting Symposium and the 1983 Extractive and Process Metallurgy Meeting of the Metallurgical Society of AIME held in San Francisco, California, November 6-9, 1983), Bi solubility in pure PbO slag was calculated and is shown in Figure 2 by the curve labelled with 'No Flux'. Figure 2 shows Bi solubility in slag over a range less than 5 % wt., which is a zone of particular interest for the present invention.

Test data reported in USP 5,234,492 are also shown in Figure 2. The agreement with the present calculation of the equilibrium constant and the test data reported in US patent 5,234,492 is excellent. This demonstrates the success of the present theoretical analysis.

Incorporating the measured PbO activity coefficient for PbO-SiO_2-P_2O_5 system (W. F. Caley and C. R. Masson, Activities and Oxygen Transport in PbO-SiO_2-P_2O_5 melts, Canadian Metallurgical Quarterly, Vol.15, No.4, 1976, pp. 359-365) into the equilibrium equation enables the prediction of the bismuth solubility in PbO-SiO_2, PbO-P_2O_5 and PbO-SiO_2-P_2O_5 system as shown in Figure 2. Addition of silica or P_2O_5 to PbO slag effectively reduces the bismuth solubility for a given lead-bismuth semi-finished product. Thermodynamically, P_2O_5 is more effective than silica.

The curves represented in Figure 2 from left to right are: 30, No flux (the US Patent 5,234,492); 31, SiO_2 flux (ratio PbO/SiO_2=1); 32, SiO_2 flux (ratio PbO/SiO_2=0.67); 33, P_2O_5 flux (ratio PbO/P_2O_5=3.65) and 34, P_2O_5 flux (ratio PbO/P_2O_5=3.33). From Figure 2, addition of silica to PbO slag enables the preferential oxidation of lead from lead-bismuth semi-finished product to more than 70 wt.% Bi with less than 5 wt.% bismuth leaving in the slag. Presence of P_2O_5 in PbO makes it possible to upgrade the lead-bismuth semi-finished product to more than 95 wt.% Bi (prior to chlorination) while maintaining the % bismuth assay in the slag below 5%.wt., and preferably below 1.0%.wt. and more preferably below 0.5%.wt in the slag leaving the process.

From Figure 2, it is clear that with suitable flux addition, Bi loss to the slag can be reduced sufficiently that the alloy can be enriched in Bi to levels otherwise impossible to attain. It is also clear that in the absence of the flux addition to the oxidation process of lead-bismuth alloy, Bi solubility in slag reaches as high as 4 wt.% for 30 wt.% Bi alloy, but with suitable flux addition, Bi loss to the slag can be reduced, the resulting slag containing less than 5 wt.%, when the alloy has been enriched to 80 to 90 wt.% Bi. In other words, it is now possible to oxidize lead-bismuth alloy to upgrade the lead-bismuth alloy to 80 to 90 wt.% Bi or higher without excessive Bi oxidation into slag and the large bismuth loss.

Many modifications and changes of the features described herein may be made without departing from the spirit and scope of the invention. It is therefore apparent that the proceeding description is by way of illustration of the invention rather than limitation of the invention.

CLAIMS:

1. A process for recovering bismuth, from a lead-bismuth containing material, which is characterized by a series of process steps that include;

   the selective oxidation of lead from said material with the addition of at least one slag forming agent which produces a lead-enriched bismuth-poor slag and an enriched-bismuth product.

2. A process according to claim 1, wherein the said lead-bismuth containing material is pretreated to remove calcium and magnesium impurities.

3. A process according to claim 1, wherein the enriched-bismuth product is treated with chlorine to remove residual lead and obtain a high purity bismuth metal while producing a lead chloride residue.

4. A process of claim 1, wherein the lead-bismuth containing materials are selected from a category consisting of debismuthizing dross from a Kroll-Betterton process, lead bismuth alloys and lead electrolytic refining anode slimes.

5. A process according to claim 4, wherein the lead-bismuth containing material is debismuthizing dross from a Kroll-Betterton process with a composition of between for 2 to 15 wt.% Bi, also containing Ca and Mg, and with the majority remaining essentially lead.

6. A process according to claim 2, wherein the lead-bismuth material is pretreated with an oxygen containing reactant selected from air, oxygen and $NaNO_3$.

7. A process according to claim 6, wherein the oxygen containing reactant is $NaNO_3$.

8. A process according to any one of claims 1 to 7, wherein the slag forming agent is selected from a group consisting of SiO_2, P_2O_5, B_2O_3 and GeO_2 or combinations thereof, the slag forming agent promoting selective oxidation of Pb while leaving Bi virtually unreacted.

9. A process according to any one of claims 1 to 8, wherein the slag forming agent is SiO_2.

10. A process according to any one of claims 1 to 8, wherein the slag forming agent is P_2O_5.

11. A process according to claim 10, wherein the slag forming agent is added in the form of ammonium mono phosphate which thermally decomposes to P_2O_5.

12. A process according to any one of claims 1 to 10, wherein said slag contains up to 20 wt.% Bi.

13. A process according to any one of claims 1 to 12, wherein said slag has a composition of 5 wt.% Bi or less.

14. A process according to any one of claims 1 to 9, wherein said enriched bismuth product has a concentration of more than 75 wt.% Bi while maintaining Bi losses at 5 wt.% or less in the slag, when SiO_2 is used as the slag forming agent.

15. A process according to any one of claims 1 to 8, wherein the said enriched-bismuth product can attain a concentration of more than 90 wt.% Bi while maintaining Bi at 5 wt.% or less in the slag, when P_2O_5 is used as the slag forming agent.

16. A process according to any one of claims 1 to 3, wherein the lead chloride residue produced is up to fifty times less then by a Kroll-Betterton process.

17. A process according to claim 1, wherein the temperature of operation is between 500 and 1200 °C.

18. A process according to claim 1, wherein the temperature of operation is between 600 and 1000 °C.

19. A process according to any one of claims 1 to 10, wherein the process is conducted in a cupel furnace or vessel, where the lead-bismuth containing material, the slag forming agent, the oxygen containing reactant, and chlorine can be added, while the lead-enriched bismuth-poor slag, the high purity bismuth metal and lead chloride residue can be removed.

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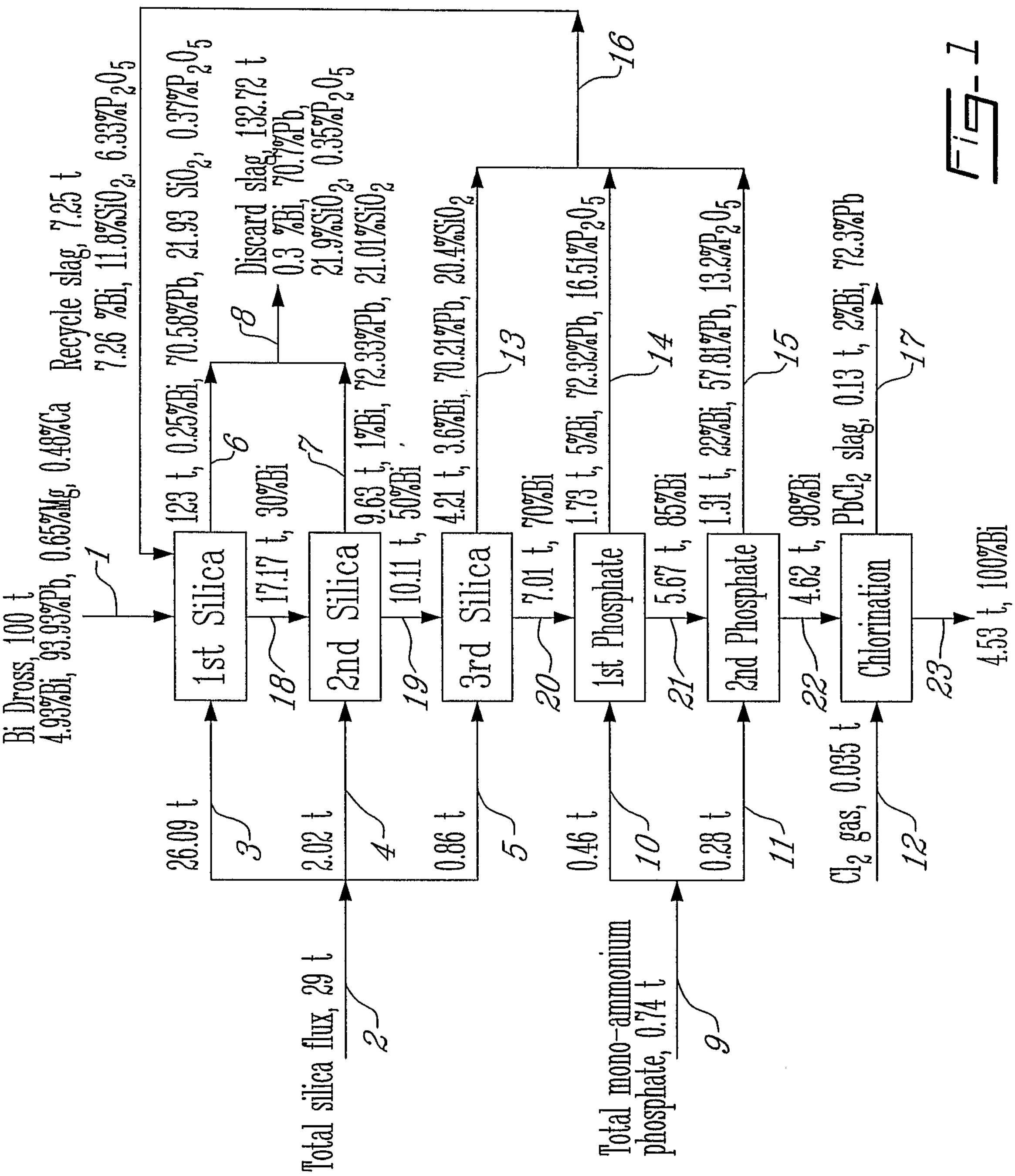
Bi Dross, 100 t
4.93%Bi, 93.93%Pb, 0.65%Mg, 0.48%Ca
Recycle slag, 7.25 t
7.26 %Bi, 11.8%SiO2, 6.33%P2O5
123 t, 0.25%Bi, 70.58%Pb, 21.93 SiO2, 0.37%P2O5
Discard slag, 132.72 t
0.3 %Bi, 70.7%Pb,
21.9%SiO2, 0.35%P2O5
9.63 t, 1%Bi, 72.33%Pb, 21.01%SiO2
4.21 t, 3.6%Bi, 70.21%Pb, 20.4%SiO2
1.73 t, 5%Bi, 72.32%Pb, 16.51%P2O5
1.31 t, 22%Bi, 57.81%Pb, 13.2%P2O5
PbCl2 slag, 0.13 t, 2%Bi, 72.3%Pb
1st Silica
2nd Silica
3rd Silica
1st Phosphate
2nd Phosphate
Chlorination
17.17 t, 30%Bi
10.11 t, 50%Bi
7.01 t, 70%Bi
5.67 t, 85%Bi
4.62 t, 98%Bi
4.53 t, 100%Bi
26.09 t
2.02 t
0.86 t
0.46 t
0.28 t
Cl2 gas, 0.035 t
Total silica flux, 29 t
Total mono-ammonium phosphate, 0.74 t
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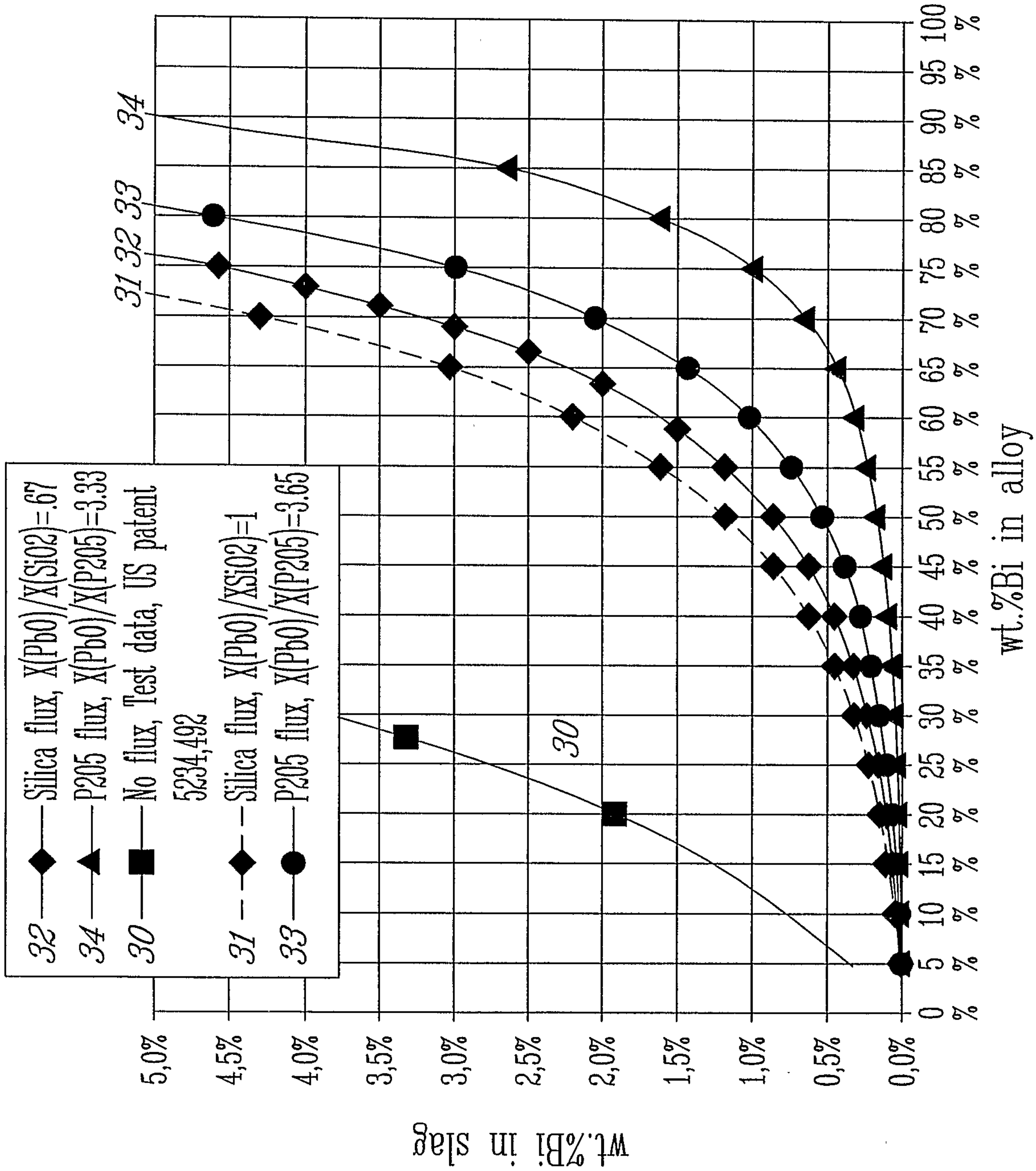
Fig-1

2/2

Fig-2


32 —◆— Silica flux, X(PbO)/X(SiO2)=.67
34 —▲— P205 flux, X(PbO)/X(P205)=3.33
30 —■— No flux, Test data, US patent 5234,492
31 - -◆- - Silica flux, X(PbO)/XSiO2)=1
33 —●— P205 flux, X(PbO)/X(P205)=3.65
31 32 33 34 30
wt.%Bi in slag
5,0% 4,5% 4,0% 3,5% 3,0% 2,5% 2,0% 1,5% 1,0% 0,5% 0,0%
wt.%Bi in alloy
0 % 5 % 10 % 15 % 20 % 25 % 30 % 35 % 40 % 45 % 50 % 55 % 60 % 65 % 70 % 75 % 80 % 85 % 90 % 95 % 100 %