Candidate's Answer Paper

(Examination Paper A - Chemistry)

Claims

1. A process for the preparation of a homogeneous lead-antimony alloy consisting of 1-15% by weight of antimony, the balance being lead and usual impurities; the process comprising quenching the molten alloy to a temperature of less than 200°C with a cooling agent.

2. A process for improving the mechanical strength of a bar of lead-antimony alloy obtainable by the process of claim 1; comprising
   i) heating the alloy bar to a temperature of 135-175°C;
   ii) rolling the bar within this temperature range, reducing the thickness of the bar by 10-20% with each pass; and
   iii) finish rolling the bar at 20-125°C to further reduce the thickness of the bar by at least 10%, at a rate of 1-5% per pass.

3. The process of claim 2 wherein the finish rolling is carried out at about 125°C.

4. The process of claim 2 or claim 3 wherein the thickness is reduced at a rate of 1 to 3% per pass in the finishing rolling.

5. A homogeneous lead-antimony alloy obtainable by the process of any one of claims 1 to 4.

6. A lead-antimony alloy as claimed in claim 5 wherein impurities are present in an amount of not more than 0.05% by weight.

7. An alloy as claimed in claim 5 or claim 6 consisting of 1-5% by weight of antimony.

8. A chemical reactor lining comprising plates of a lead-antimony alloy obtainable by the process of any one of claims 2 to 4.

9. A chemical reactor lining as claimed in claim 8 wherein joints between the plates are filled with a lead-antimony alloy obtainable by the process of claim 1.

10. A chemical reactor comprising a lining as claimed in claim 8 or claim 9.

11. Use of a reactor as claimed in claim 10 wherein the antimony content of the alloy does not exceed 5% by weight, in the production of urea.

12. A homogeneous, solid ternary alloy consisting of 84-89% by weight of lead, 6 to 12% by weight of antimony and 2 to 8% by weight of tin obtainable by quenching the molten alloy to a temperature of less than 200°C with a cooling agent and cooling directly further with the cooling agent.

13. Use of the alloy of claim 12 as a bearing metal.

14. Use of the alloy of any one of claims 5 to 7 in chemical plant construction.

Description

The present invention relates to improvements in or relating to chemical plants, particularly urea production plants; in particular to improvements in the processing of lead alloys for use in chemical plants.

Corrosion in chemical production plants is well known and a number of solutions have been proposed to overcome this problem, but these have often been either very costly or very complicated (or both) and thus difficult to put into practice.

Faced with this problem, in particular in a urea production plant, attempts were initially made to overcome the problem by adjusting the reaction parameters. It soon became evident that any reduction in corrosion was at the expense of uneconomical reduction in yield. Thus the search for a solution was approached rather differently - by selecting a different material for building the reactor. Due consideration for the price was important, as was a preference for a material which would not contaminate the desired reaction product.

Lead has been known for many years as being highly resistant to corrosion from many materials. It is, of course, well known in the lead chamber process for the manufacture of sulphuric acid, as well as the use of lead pipes for drinking water supply.

However, lead is not always suitable because, inter alia, of its inadequate mechanical strength, not to mention its poisonous properties.

It is known, however, that lead alloyed with even small quantities of other metals, such as antimony, tin and/or arsenic, assumes markedly different properties. We have now established that certain Pb-Sb alloys are characterised by very high resistance to corrosion from aggressive media. If such alloys are to be used in the construction of chemical plant this property alone is not sufficient. The alloys need to meet certain mechanical requirements. Document I indicates that certain Pb-Sb alloys have good corrosion resistance and tensile strength, although no details are given. The alloy is described as being suitable for use in water-supply pipes, but no reference to suitability in chemical plant is made. Document II suggests that a lead-antimony alloy comprising 1-8% by weight of antimony has good acid corrosion properties. No evidence of this is provided, but it is shown that the alloy is able to withstand pressures of up to 1.0 MPa.

We have now found a way of manufacturing Pb-Sb alloys with very good chemical resistance and good mechanical properties. These meet our requirements and the material has the potential for a wide variety of uses in chemical plant production. If the inside reactor walls, which are in contact with the reaction mixture, are manufactured from this material, because of their greater strength, a simpler outer construction can be used for the reactor than was hitherto necessary.

Document I and Document II each describe a method for preparing a Pb-Sb alloy, comprising melting the metals together - by dissolving the Sb in molten Pb, or by mixing the molten metals at about 400°C. In each case, the alloy is allowed to cool, following casting, at room-temperature.

As the alloy cools, the components tend to separate giving rise to inhomogeneities in the solid state and thus greatly varying properties depending upon the conditions of manufacture. This also applies near the eutectic, which is known to be 11.1% by weight of Sb. Thus the alloys of the prior art are deficient in the reliability of the properties which is a greater concern in chemical production plant where a high degree of safety and long production life are important factors.

We have surprisingly discovered a special process which achieves these objectives.

In this process, the proportion of Sb should not exceed 15% by weight as above this figure, although hardness improves, the material becomes increasingly brittle and is more likely to contain inhomogeneities. For our purposes, material with less than 1% by weight of Sb does not have the desired properties.

The present invention provides [as claim 1]. The molten alloy is prepared by a conventional process, for example that of Document I.

Such a process produces an alloy having excellent chemical resistance to corrosion. However, to reach the strength values required for plant construction, formed bodies (bars) obtained as above are further processed by a method comprising [steps (i)-(iii) of claim 2].

The temperature of the finish-rolling operation is selected depending upon the exact composition and exact requirements to be met by the alloy.

Experiments have shown that the mechanical strength of the alloy is greater the nearer the finish-rolling temperature is to the upper limit of 125°C. Above this temperature, the strength falls below the minimum value acceptable for plant construction (20 MPa).

Furthermore, it has been observed that the alloys for plant construction, particularly in the case of urea production, should preferably not contain more than 5% by weight of Sb as above that time limit the number of cracks which can appear in the finish rolling step rises steeply and weakens the material.

In noting that our alloys which have not undergone the special rolling process have the same chemical resistance as alloys which have been processed in this way there arises the possibility of constructing a reactor with inner walls - which in use come into contact with the reaction mixture - consisting of plates of the hardened alloy with unhardened alloy of the same composition used for joints between plates.

As the appended examples show, a reactor line with plates alone provides an improvement over reactors used hitherto, but the use the alloy in the joints provides yet further advantages. Contact corrosion is avoided and the structure is simpler from the point of view of expansion and contraction of the reactor as temperatures change. As the softer material is more ductile, it can help to prevent leakages between the joints. When the joint is being cast, appropriate steps should be taken to ensure rapid heat dissipation to avoid inhomogeneities. Additional cooling may be appropriate.

The interaction, in the production of urea, between the alloy and the urea, has also been investigated in some detail. We have found that no traces of the alloy constituents could be found in the urea even when left in the reactor for long periods in reaction conditions going well beyond the usual period of contact. This has been observed in respect of both the hardened and unhardened alloys.

This provides the advantage that additional purification of the product is unnecessary. This is particular advantage where the product is to be used in animal feed or other products for human or animal administration.

Apart from the usual impurities (standard quality metals can be used in the present invention), lead and antimony are the sole constituents of the alloy. Impurities should preferably constitute no more than 0.05% by weight of the total.

However, the processes of the present invention are also applicable to other alloys. In particular, a ternary alloy having a composition of 84 to 89% by weight of Pb, 6 to 12% by weight of Sb and 2 to 8% by weight of Sn can be prepared by melting together the metals as described above followed by quenching to the solid state immediately after casting, with further cooling directly by means of the cooling agent.

Such a ternary alloy is suitable for use as a corrosion-resistant slide-bearing metal, for example as an axle-bearing metal for moderate loads and speeds.

The following examples illustrate the above and other aspects of the present invention. In the examples the percentages are by weight relative to the total weight of the alloy, the alloys were all manufactured by melting the components together as described above and in Document I and the resulting formed bodies were manufactured by casting in moulds and quenching to below 200°C.

Notes to Examiner

I hope that support for the novelty and inventive step of the claims is apparent from the description. In summary however,

Claim 1 - The process is not described in DI or DII and the product being homogeneous would appear from the clients letter to have advantages over the prior art non-homogeneous alloys. This also justifies this process step as being distinct from those of claim 2.

Claim 2 - This is clearly a novel process and again gives further advantages not shown in DI or DII.

Claims 3 + 4 - Fall back positions drawn from the examples and supporting text of client's letter

Claims 5 - 7 - Insofar as the products of the processes of claims 1-4 have advantages over the prior art - which makes no mention of homogeneity - the solid alloy must have a different structure. Apart from defining it as "homogeneous" no further details are given in the client's letter. Thus the "obtainable by" form of claim is appropriate.

Claims 8 - 10 and 14 - The client is involved in construction of chemical plant and so a claim to their actual product is thought to be appropriate.

Claim 11 - A claim to the particular use at which the client was aiming.

Claims 12 + 13 - The client has been asked that they be included. It could be argued that the process step in claim 1 which is repeated in claim 12 provides a unifying concept regarding Art. 82. Both alternative alloys could have been included in a single main claim but I felt that this would have confused later claims and thus have been contrary to Art. 84 EPC.

Two part claim format (Rule 29(1)(b)) was not thought to be appropriate in this case in any of the claims because (in part) the word "homogeneous" was used in the early part of the claims as a distinguishing feature and other features were used later in the claim (process steps etc.). Two part form would have resulted in claims which were not clear and concise as required by Art. 84 EPC.

A claim to an alloy of Pb and Sb *per se* (with a disclaimer to the those alloys known from DI and DII) would certainly have lacked an inventive step. Only Pb89 Sb11 and Pb97.5 Sb2.5 are known a binary alloys from DI but as a range of experimental data is given in DII, the disclosure of all alloys in the range Pb92-99, Sb1-8 must be accepted. DI applied to DII could be taken to suggest to the skilled person that he should consider extending the Sb content of the alloy.

Similarly, the broad idea of a ternary alloy of Pb/Sb/Sn is shown in the last 3 examples of DI and use as bearing metal mentioned. This would suggest a broad claim to these ternary alloys would be unallowable.