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METHOD OF PRODUCING ALLOYS OF ALKALI METAL
AND LEAD HAVING A GRAIN FORM AND STRUCTURE
SUITED TO LEAD ALKYL SYNTHESIS
Filed July 9, 1947

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Fig. 1.

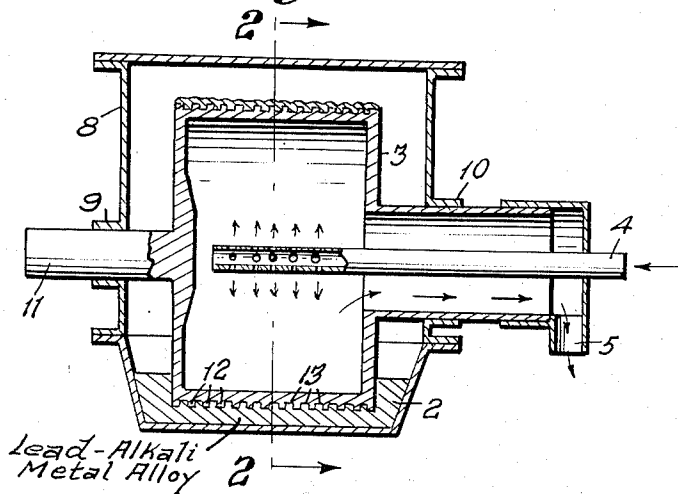


Fig. 3.

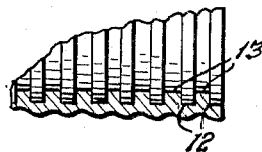


Fig. 2.

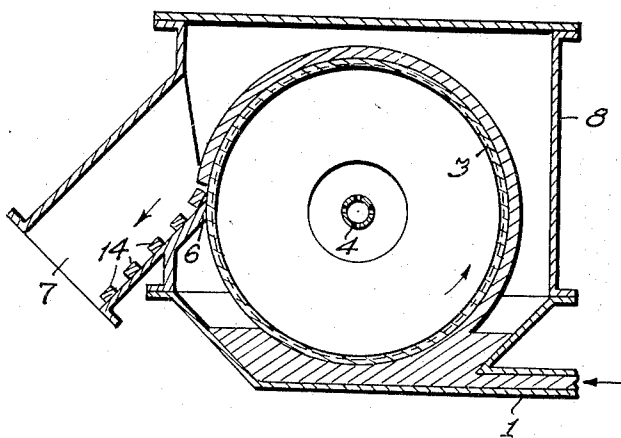


Fig. 4.

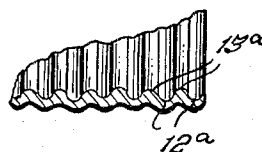
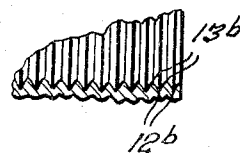


Fig. 5.



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UNITED STATES PATENT OFFICE

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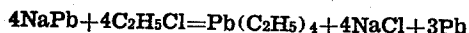
2 Claims. (Cl. 22-200.1)

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During reactions in heterogeneous systems in which a participating, solid reaction component is simultaneously dissolved, the reaction process is often greatly influenced both by the surface area (particle size) and the body structure of said component. It is thus as a rule of great importance for the reaction velocity and the uniformity of such processes in which a metal (by which, in the following, also metal alloys are to be understood) takes part, that the metal has not only a uniform grain size but also a suitable crystal size and crystal arrangement. This is for instance an important condition in the preparation of tetraethyl lead and other lead alkyls from an alloy of sodium and lead.

This alloy generally consists of lead and sodium in amounts proportional to their atomic weights. It is also possible to partly replace sodium by potassium, whereby a better yield is obtained in the synthesis. This alloy composition corresponds to a real chemical compound of lead and alkali metal, having the formula MePb , wherein Me represents an alkali metal selected from a class consisting of sodium and potassium. The sodium alloy has the composition NaPb , containing 10 per cent by weight of sodium.

The reaction in the lead alkyl synthesis for the preparation of e. g. tetraethyl lead proceeds substantially according to the formula:



Since this reaction takes place between solid alloy and liquid or gaseous ethyl chloride, i. e. in a heterogenous system, the form of the alloy will greatly influence the reaction velocity. It has previously been found that dustfree grains of equal size of this alloy are especially suited for carrying-out the highly exothermic synthesis. Grains of the size of 3-5 mm. are considered to be especially suitable. This product has hitherto been obtained by crushing the solidified alloy, which is very brittle, or by vigorously stirring the molten alloy with a simultaneous freezing-out or by a combination of these methods in chilled mills or crushers. This will result in a comminuted product of pieces or grains from which dust and minor particles must first be removed by sifting, since it has been found that these minor grains cause irregularities in the reaction process in question, which are difficult to control.

It has now been found, however, that properties favourable in said respects can be obtained by allowing the alloy, after melting, to harden on a cooled roller of particular shape which is

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partly immersed into the alloy bath or in some other suitable manner is kept in direct contact with it, whereby the alloy is supplied to the surface of the roller and, after hardening, continually scraped off from it during the rotation of the roller.

According to the present invention the adhesion of the molten alloy to the roller is facilitated and the size of the separated alloy flakes limited by providing the roller surface with grooves and ridges or similar elevations. If the grooves etc. are placed mutually equidistant and arranged at right angles to the axis of rotation of the roller, weakenings are produced in the solidifying alloy layer, which weakenings cause said layer, when being scraped off, to loosen into strips of equal width which, in turn, on account of the brittleness of the alloy and the crystal growth at right angles to the surface of the layer, are automatically broken up at the grain boundaries into pieces of an even size.

The adhesion of the alloy to the roller can also be improved by coating the surface of the roller with a metal or alloy of lower melting-point than the alloy separated on the roller by cooling. Tin or lead has thus proved to be especially suitable as a coating on the roller surface for the present purpose.

The device may be made closed, so that the process can be conducted in an atmosphere of neutral character. In this case the device is provided with suitable gas-tight feeding and discharging arrangements.

By producing the alloy grains according to the methods of the present invention, due regard is taken not only to the above mentioned known fact that an equal grain-size is favourable, but also to the fact that both the crystal size and the crystal arrangement have an essential influence on the reaction process. Another advantage is that a product cooled down close to the temperature of the cooling medium is directly obtained, whereby a further cooling of the easily oxidized alloy is not needed, contrary to previously mentioned known methods, where such an extra measure has proven necessary.

On the accompanying drawing is schematically shown an example of a suitable arrangement for carrying out the invention.

Fig. I is a vertical section through the apparatus and Fig. II a section at right angles to Fig. I. Figures III-V show details of suitable forms of the roller surface on a larger scale.

The molten alloy is introduced through pipe 1 and forms a bath 2 at the bottom of the appara-

tus, in which the rotating roller 3 is partly immersed. The roller is cooled on the inside by means of a liquid cooling medium which is introduced through a central pipe 4, the inner end of which is provided with a number of small openings through which the cooling medium is sprayed in various directions against the roller inside, so that the whole inside is kept cooled. The cooling medium leaves the interior of the roller by the outlet 5. When the roller rotates, the molten alloy solidifies on the outside of the roller while filling out the grooves and at the same time forming an integral layer over the ridges. When the alloy layer, which is thus kept under a constant cooling, reaches the scraper 6 the alloy layer is detached from the roller and is simultaneously automatically broken up into fragments which drop down through the spout 7. It will be noted from Fig. II of the drawing that the layer of alloy is bent by the scraper through a rather sharp angle as it leaves the collecting drum and this causes it to break up since it is very brittle.

In order to protect the alloy from atmospheric influence, the roller is enclosed in a gas-tight chamber 8 and supported in gas-tight bearings 9 and 10. The chamber is kept filled with a gas which is inert to the alloy. The rotation of the roller is effected by the driven shaft 11. The spout 7 is provided with a suitable sluicing device to enable the alloy fragments to be removed from the chamber without admittance of the outer atmosphere.

Figs. III, IV and V show on an enlarged scale various modifications of the roller surface which have been found suitable. From these figures the various forms which the solidified metal layer assumes will be readily apparent. The form with square grooves shown in Fig. III has proven particularly useful for effecting the desired dividing of the lead-sodium alloy into uniform fragments according to the invention.

I claim:

1. In the process of forming small particles of lead-alkali metal alloy of uniform size and having crystals of relatively high purity and a size which makes the particles particularly suitable for use in the preparation of lead alkyls, the steps which comprise forming a molten bath of a lead-alkali metal alloy having a composition corresponding substantially to the formula MePb , wherein Me represents an alkali metal selected from a group consisting of sodium and potassium, which when in the solid state is very brittle and which when solidified on a cooled surface forms a crystal growth perpendicular to the surface, passing through said bath a cylindrical surface cooled below the solidification point of said alloy, said surface being provided with equally spaced parallel peripheral ridges with intermediate depressions, whereby a solidified brittle layer of metal with a crystal growth at right angles to the cooled surface is formed on said surface, scraping said solidified metal layer from said surface and bending it simultaneously through a sharp angle, whereby it is simultaneously and automatically broken along the depressions and crystal boundaries into particles of substantially uniform size.

2. The process of claim 1 wherein the steps are performed in an inert atmosphere.

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