Patented June 26, 1951

2,558,627

UNITED STATES PATENT OFFICE

2,558,627

METHOD FOR THE PRODUCTION OF ZIRCONIUM ALLOYS

Sven Christian Pyk, Nynashamn, Sweden

No Drawing. Application March 4, 1949, Serial No. 79,727. In Sweden March 19, 1948

6 Claims. (Cl. 204—71)

1

Magnesium-zinc-zirconium alloys can be produced by melting a zirconium-zinc alloy in magnesium. By this means, apart from a better yield of zirconium as compared with production by conversion with a flux containing zirconium and magnesium, a more stable product from the point of view of corrosion is obtained.

The alloys of zinc and zirconium referred to above can be produced by the electrolytic precipitation of zirconium from a molten salt containing zirconium with molten zinc as a cathode. As the melting point of the alloy formed rises, however, with an increasing zirconium content, a relatively high temperature must be employed during electrolysis which, for the extraction of alloys which in practice often have a preferably high zirconium content, may eventually exceed the boiling point of zinc. The result of this will be that the zinc will be distilled off whereby the melting point of the alloy formed will be raised still further, and consequently the electrolytic production of alloys with a high zirconium content for the purpose in question is associated with considerable difficulties.

According to the invention the above-mentioned disadvantages can be avoided by interrupting the electrolysis as soon as the zirconium content in the alloy formed has increased to such an extent that the temperature rise thereby entailed would have a prejudicial effect on the electrolysis, and therefore from a practical point of view at least before the melting point of the zirconium alloy being formed rises to the boiling point of the zinc and then distils off zinc from the alloy as the result of subsequent heating. Investigations made have shown that under ordinary conditions the electrolysis may suitably be interrupted when a zirconium content of about 18% has been reached. The subsequent heating, required to increase the zirconium content of the alloy, can either be carried out by directly heating the alloy formed in a vacuum or at normal pressure in a protective gas, or by employing a protective flux, but it is, of course, permissible to first allow the metal to become cold or to cool the latter and heat up the solid and preferably finely crushed alloy.

The employment of additions which lower the solidifying point of the zinc-zirconium alloy may, of course, come into consideration in certain cases and falls within the scope of the invention.

As a fusible salt containing zirconium a mixture of alkali metal halides with a zirconium halide dissolved therein may preferably be employed for the electrolysis, and in certain cases also magnesium chloride. Graphite or zirconium may be employed as the anode, and electrolysis is carried out in a container of suitable refractory and preferably ceramic material such as brick with a current supply through the bottom which is preferably constructed of graphite. Electrolysis can, of course, be carried on continuously by adding zirconium chloride and zinc as required, whereby the alloy can be tapped off progressively.

2

As an example of the invention it may be mentioned that a melt weighing two kilogrammes containing 28% potassium chloride, 22% sodium chloride and 50% zirconium tetrachloride was electrolysed for two hours at about 750° C. with a current of 500 amperes and with two kilogrammes of zinc as a cathode. A metal was hereby obtained containing 15% zirconium and 84.5% zinc. This finely pulverized alloy was then heated up in a high vacuum for two hours up to 700° C., whereupon a metal was obtained containing 35% zirconium and 64% zinc. After heating for a further four hours at 800° C. the zirconium content had increased to 98% and the zinc content had fallen to 0.5%.

In another example a molten salt bath of similar composition but also containing magnesium chloride was electrolysed. The electrolysis was carried out in the same manner. A zirconium zinc-magnesium alloy was formed which contained 12% zirconium, 80% zinc and 8% magnesium. After distilling off the zinc and also a part of the magnesium at the same temperature and for the same length of time as before, a metal was obtained containing 40% zirconium, 45% zinc and 15% magnesium.

The high concentration zirconium alloys may, of course, also be employed for other purposes than for the production of magnesium alloys.

I claim:

1. In the manufacture of zinc-zirconium alloys of high zirconium content, the process which comprises forming a molten salt bath containing a zirconium halide, adding metallic zinc to the bath to form a molten zinc cathode, electrolyzing the salt bath while maintaining the temperature of the bath above the melting point of the resulting cathode alloy but below the boiling point of zinc, recovering the cathode alloy and heating and distilling off zinc therefrom to enrich the alloy with zirconium.

2. The process of claim 1 wherein the salt bath comprises alkali metal chlorides and zirconium chloride.

3. The process of claim 1 wherein the salt bath contains a magnesium salt and the cathode alloy formed is a zinc-zirconium-magnesium alloy.

4. The process of claim 1 followed by adding the cathode alloy to a molten bath of metallic magnesium to form a zinc-zirconium-magnesium alloy.

5. The process of claim 1 wherein the electrolysis is stopped when the cathode metal acquires a zirconium content of about 18 percent.

6. In the manufacture of zinc-zirconium alloys of high zirconium content, the process which comprises forming a molten salt bath comprising alkali metal chlorides and zirconium chloride, heating the bath above the melting point of zinc, forming a molten zinc cathode in the bath, electrolyzing the bath while maintaining the temperature thereof above that of the melting point of the resulting zinc-zirconium alloy and until the temperature has reached substantially the boiling point of zinc, then recovering the zinc-zirconium alloy and distilling zinc therefrom to enrich the alloy with zirconium.

SVEN CHRISTIAN PYK.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,323,042	Honsberg	June 29, 1943

OTHER REFERENCES

U. S. Bureau of Mines Information Circular 7341, Feb. 1946, by W. J. Kroll and A. W. Schlechter, pp. 8, 11, 12, 13, 15.

Compt Rendu, vol. 61 (1865), pp. 109–113, an article by Troost.

The Journal of the Franklin Institute, May 1895, page 376.