

UNITED STATES PATENT OFFICE

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PRODUCTION OF REFINED MAGNESIUM AND
MAGNESIUM ALLOYS

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5 Claims. (Cl. 75—67)

This invention relates to the production of refined magnesium and magnesium alloys.

The present invention is a continuation in part of my application Serial No. 706,558, filed January 13, 1934 for the Production of refined magnesium and magnesium alloys, which has since become Patent No. 2,066,579, dated Jan. 5, 1937.

One object of the present invention is to provide a simple and commercially practicable process for the production of compact metallic magnesium in a substantially chemically pure state.

Another object of my invention is to enable magnesium and magnesium alloys respectively, to be recovered, in a refined state, from waste, such as for example scrap arising from machine working and casting of magnesium or its alloys, and thereby not only to obtain a purer product as a result of my improved process, but also to ensure great saving by increasing the quantity of the recoveries.

The mechanical properties and resistance to corrosion of magnesium and its alloys are adversely affected by the presence of impurities, especially extraneous oxides and other compounds (nitrides, carbides, silicides and the like), and also by other foreign substances such as carbon, even though said impurities be present in very small amounts.

The present invention aims at enabling magnesium being technically pure (and its alloys) to be very extensively refined in the simplest manner. The process is also admirably adapted for the elimination of impurities present during the recovery of magnesium by re-melting magnesium scrap (for example from machine working and from the foundry). Thus, for example in the case of magnesium dross, from which only 30% of the magnesium content could be recovered, as refined metal, by treatment according to known processes, the present invention enables up to 90% of the metal originally present to be recovered as metal free from slag.

For this purpose, it was formerly the practice to re-melt magnesium scrap high in magnesium under a layer of chlorides, especially magnesium chloride. In such case, traces of magnesium chloride remained in the metal and had to be removed by special after-treatment. In order to avoid this necessity, it has been proposed to moisten the metal with only just sufficient of a magnesium chloride melt to effect a cementation of the foreign substances and to assure the recovery of a metallic melt free from magnesium chloride. With this object the scrap metal was to be melted at high temperatures, such as 800°

C. and over, with small quantities of anhydrous magnesium chloride, corresponding approximately to the amount of impurities present. Moreover, the use of saline melts consisting of mixture of magnesium chloride or carnallite ($K Mg Cl_3 + 6H_2O$) with inspissating additions, such as oxides (e. g. of magnesium) or fluorides (of magnesium, calcium, aluminium or other metals) was proposed for the purpose. Subsequently it was proposed to replace these melts by potassium chloride, with additions of fluorspar, the salts primarily separating out during the cooling of the melt then serving as the thickening media.

This group of processes also includes the proposal to refine magnesium by melting the metal in association with alkali chlorides (especially sodium chloride or potassium chloride) or mixtures of alkali chlorides, having melting points substantially higher than that of magnesium. These conditions are fulfilled by mixtures in which the sodium chloride or potassium chloride predominates. After melting, the whole is left to cool down to a temperature at which the salt, or saline mixture, has solidified to form a crust in which the impurities are occluded, whilst the metal is still fluid.

Another group of processes consists in refining the metal by melting it in association with additions which lead to the formation of an alloy to a moderate extent. Zinc, for example, is specified as an addition of this kind in United States specification No. 1,698,647, with the qualification that, as an alloying component, this metal exerts a highly unfavorable influence on the magnesium (page 1, lines 41 to 58). For this reason, the specification recommends that, when magnesium fluoride is used as the main refining agent, a small amount (0.05 to 0.3%) of metallic calcium should be incorporated.

According to the present invention, the magnesium, or material containing same, that is to be refined is melted in association with small additions of a salt of antimony, such as a halide or sulphide, with or without the addition of halides or other heavy metals the cation of which combines or does not combine with the molten material. The salts which I have found suitable for the purpose are:—halides of cadmium, copper, iron, manganese, zinc. In addition, the flux contains preferably a proportion of magnesium halide.

The amount of the additions depends on the quantity and nature of the impurities, 1 to 3 per cents by weight (referred to the total weight

of material under treatment) being usually sufficient.

The melting point of the selected salt or saline mixture should preferably be lower than that of magnesium, but the specific gravity higher than that of the molten metal. If such salts be strewn over the metal during the melting down process, they melt sooner than the metal and envelop it, thereby protecting it against oxidation. This behavior has a specially advantageous effect in the case of magnesium alloys, their melting points being lower than that of the metal. When the metal is stirred, with a further addition of the salts, after fusion has taken place, the higher specific gravity of the saline melt causes it to subside quickly after absorption of the impurities, so that, after settling, the refined metal can be poured off. Nevertheless the fused metal remains protected against oxidation by a super-natant thin film of the saline melt.

In order to obtain intimate mixing, it is advisable to melt the saline mixtures beforehand and to crush the melt for use.

Examples of appropriate salt mixtures are the following:

- (1) 30% Sb_2S_3 + 70% MgCl_2
- (2) 10% SbCl_3 + 90% MgCl_2
- (3) 15% SbCl_3 + 85% CuCl_2
- (4) 10% SbCl_3 + 40% CuCl_2 + 50% MgCl_2
- (5) 40% Sb_2S_3 + 60% CuCl_2
- (6) 20% Sb_2S_3 + 30% CuCl_2 + 50% MgCl_2
- (7) 10% SbCl_3 + 90% CdCl_2
- (8) 10% SbCl_3 + 60% CdCl_2 + 30% MgCl_2
- (9) 40% Sb_2S_3 + 60% FeCl_3
- (10) 30% Sb_2S_3 + 35% FeCl_3 + 35% MgCl_2
- (11) 10% SbCl_3 + 30% FeCl_3 + 60% MgCl_2
- (12) 15% SbCl_3 + 85% ZnCl_2
- (13) 10% SbCl_3 + 50% ZnCl_2 + 40% MgCl_2
- (14) 40% Sb_2S_3 + 60% ZnCl_2
- (15) 30% Sb_2S_3 + 40% ZnCl_2 + 30% MgCl_2
- (16) 35% Sb_2S_3 + 65% CdCl_2
- (17) 20% Sb_2S_3 + 40% CdCl_2 + 40% MgCl_2
- (18) 10% SbCl_3 + 40% FeCl_3 + 50% ZnCl_2
- (19) 10% SbCl_3 + 30% FeCl_3 +
30% ZnCl_2 + 30% MgCl_2
- (20) 30% Sb_2S_3 + 35% FeCl_3 + 35% ZnCl_2
- (21) 20% Sb_2S_3 + 30% FeCl_3 +
30% ZnCl_2 + 20% MgCl_2

The mixtures numbered 8, 11, 18, 20 and 21 have proved in practice to be of particular efficacy.

I use the term magnesium in the following claims to include not only crude or technically pure magnesium metal but also magnesium alloys and magnesium waste arising for example from machine working or casting of magnesium or its alloys.

I claim:—

1. The method of producing refined magnesium, which comprises melting impure magnesium with a fluid flux containing as purifying agents a mixture of antimony sulphide, chloride of iron, zinc chloride and magnesium chloride, agitating the molten metal with the flux, allowing the metal and the flux to segregate from each other, and separating the metal from the flux.

2. The method of producing refined magnesium, which comprises melting impure magnesium with a fluid flux consisting of 40 per cent of antimony trisulphide, 20 per cent of iron chloride, 10 per cent of zinc chloride and 30 per cent of magnesium chloride, agitating the molten metal with the flux, allowing the metal and the flux to segregate from each other, and separating the metal from the flux.

3. The method of refining magnesium, which comprises melting impure magnesium with a fluid flux containing as the main purifier a salt of antimony selected from the group consisting of halides and sulfides, and further containing a salt selected from the group of halides of cadmium, copper, zinc and magnesium, agitating the molten metal with the flux, allowing the metal and the flux to segregate from each other and separating the metal from the flux.

4. The method of refining magnesium, which comprises melting impure magnesium with a fluid flux containing as the main purifier a salt of antimony selected from the group consisting of halides and sulfides, and further containing a halide of iron, agitating the molten metal with the flux, allowing the metal and the flux to segregate from each other and separating the metal from the flux.

5. The method of refining magnesium, which comprises melting impure magnesium with a fluid flux containing as the main purifier a salt of antimony selected from the group consisting of halides and sulfides, further containing a salt selected from the group of halides of cadmium, copper, zinc and magnesium, and further containing a halide of iron, agitating the molten metal with the flux, allowing the metal and the flux to segregate from each other and separating the metal from the flux.

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