No. 778,270. PATENTED DEC. 27, 1904.

I. L. ROBERTS.

PRODUCTION OF METALLIC MAGNESIUM.

APPLICATION FILED MAY 31, 1904.

Fig. 2

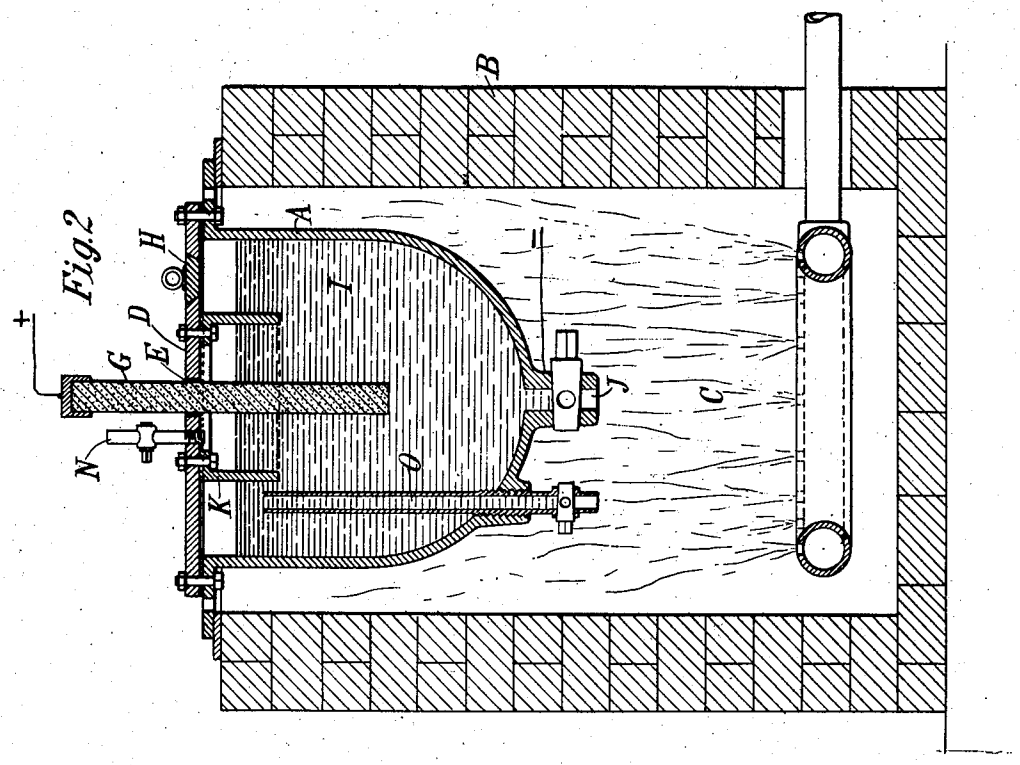
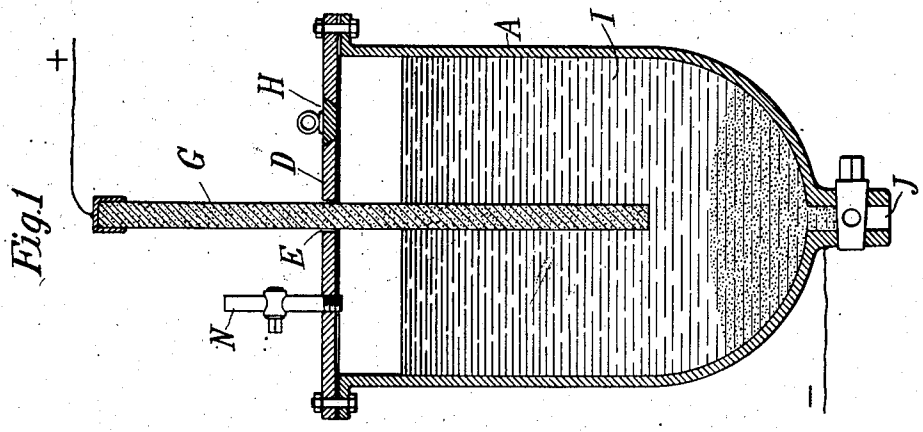


Fig. 1



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UNITED STATES PATENT OFFICE.

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PRODUCTION OF METALLIC MAGNESIUM.

SPECIFICATION forming part of Letters Patent No. 778,270, dated December 27, 1904.

Application filed May 31, 1904. Serial No. 210,393.

To all whom it may concern:

Be it known that I, ISAIAH L. ROBERTS, a citizen of the United States, residing at New York, county of Kings, and State of New York, have invented certain new and useful Improvements in the Production of Metallic Magnesium, of which the following is a specification, reference being had to the drawings accompanying and forming part of the same.

My invention relates to the production of metallic magnesium by electrolysis. Heretofore it has been common to electrolyze a fused bath containing the chlorids of magnesium and other substances; but this method is uneconomical, because it is necessary, before preparing the bath, to heat the magnesium chlorid to drive off the water carried by it. In this heating a considerable part of the chlorid itself is volatilized, and so lost. Volatization of the magnesium chlorid also continues during electrolysis. I have discovered, however, that if the fluorid of magnesium be added to the bath or electrolyte and the whole electrolyzed metallic magnesium will be obtained with little or no loss of the salts by volatilization.

The composition of the electrolyte which I prefer to use is as follows: one hundred and twenty parts of lithium fluorid, eighty parts of potassium chlorid, forty parts of sodium chlorid, ten parts of ammonium chlorid, ten parts of magnesium chlorid, eighty parts of magnesium fluorid, and twenty parts of magnesium oxid or carbonate.

The above mixture becomes liquid at a dull-red temperature, and if then it is electrolyzed by direct current metallic magnesium will be liberated at the cathode and chlorin and oxygen at the anode.

The composition above described may be varied considerably and good results still be obtained—that is, certain of its constituents may be omitted or others substituted or their proportions varied, so long as magnesium fluorid and a chlorid of the alkali group are used. For example, the bath may contain simply sodium or potassium chlorid and magnesium fluorid. The advantage of using a certain amount of the lithium salt resides in the fact that it lowers the specific gravity of the bath, causing the liberated magnesium to sink readily to the bottom, where it is protected from the air and the electrolytic gases, also for the reason that the lithium salt lowers the fusing-point of the bath. It helps to absorb the oxid or carbonate of magnesium when either is present, but whether by solution or suspension I am unable to say. The ammonium and magnesium chlorids also lower the specific gravity and fusing-point of the bath; but they are very volatile, and only limited quantities thereof should be used; otherwise considerable waste may result from their being more or less volatilized before the bath is completely fused. Only so much of either should be used as will stand a dull-red heat slightly above the melting temperature of magnesium. The oxid or carbonate mentioned is not essential; but if added in such amounts as will be dissolved or suspended in the bath more or less of the oxid or carbonate will be reduced along with the fluorid and chlorid. It should be observed, however, that the oxid and carbonate of magnesium raise the specific gravity of the mixture and the quantity of either in a bath or apparatus in which the metallic magnesium is to be removed from the bottom should not be so great as to cause the magnesium to rise instead of sink. It will therefore be seen that the essential constituents of the bath are magnesium fluorid and a chlorid of an alkali. Others of the substances named assist either as electrolytes or solvents or by lowering the specific gravity or melting-point of the bath. Hence when only the heavy alkali chlorids are used with the magnesium fluorid or, stated more generally, whenever the liberated metal floats on the bath the metal should be protected by a suitable screen from the air and anode gases. The specific gravity of the bath should therefore, to obtain the best results, be regulated to permit the metal to rise readily to the surface or sink readily to the bottom.

In the accompanying drawings I have shown convenient apparatus for use in practicing my method.

Figure 1 shows in section an apparatus for

use with a light bath, wherein the free metal sinks to the bottom; and Fig. 2 shows an apparatus adapted for a heavy bath, in which the metal rises to the surface.

Referring to Fig. 1, A indicates an iron pot or crucible set in a suitable furnace B, heated by a gas-flame from the burner C. The pot is covered by a slab of slate D, having an aperture E for the insertion of the carbon anode G. The cover is also provided with another opening closed by a lid H, through which additions to or removals from the bath may be made. A draw-off cock J is provided at the bottom of the pot to permit withdrawal of the metal and other contents when desired. For this purpose the gas-burner is removed and a vessel set under the cock to receive the material.

For heavy baths the apparatus shown in Fig. 2 should be used. This is substantially like the former except for the following: A draw-off pipe O is provided extending up to the floating metal, so that the magnesium may be drawn off without removing any of the bath. The cover D should be securely fastened to the pot, as by bolts, as shown, preferably with an asbestos gasket between the two, to secure an air-tight joint. Depending from the lid is a ring K, projecting into the bath I. The magnesium will rise along the sides of the pot and be retained in the annular space outside the ring K, whence it may be withdrawn through the pipe O. A bottom cock J may also be provided, so that the apparatus may also be used for light baths, if desired. The screen K should be made of non-conducting material, as otherwise electrolysis would occur there, and the anode gases would not all be confined inside the screen. Porcelain is very effective for this purpose. The anode G should fit tightly, as I find that the less air admitted to the pot the better in either case. The air seems to accelerate or assist volatilization more than does an atmosphere of the electrolytic gases. Escape-cocks, as N, may be provided to permit the gases to escape when necessary. It may be noted that a cover for the pot is essential where a heavy bath is used, but may be dispensed with in the other case. In the latter instance, however, it is advantageous, since it tends to prevent volatilization and retains the heat of the bath.

The types of apparatus described are of course merely convenient forms for practicing my invention, which is a process, and therefore independent of any particular apparatus or mechanism.

What I claim is—

1. The method of producing metallic magnesium, which consists in electrolyzing a fused bath of magnesium fluorid containing a chlorid of an alkali, as set forth.

2. The method of producing metallic magnesium, which consists in electrolyzing a fused bath containing magnesium fluorid, a chlorid of an alkali metal, and a lithium salt, as set forth.

3. The method of producing metallic magnesium, which consists in electrolyzing a fused bath containing magnesium fluorid and two or more alkali-metal chlorids, as set forth.

4. The method of producing metallic magnesium, which consists in electrolyzing a fused bath containing fluorid and chlorid of magnesium, and one or more chlorids of the alkali-metal group, as set forth.

5. The method of producing metallic magnesium, which consists in electrolyzing a fused bath containing magnesium fluorid and another compound of magnesium, and one or more chlorids of the alkali-metal group, as set forth.

6. The method of producing metallic magnesium, which consists in fusing a mixture of magnesium fluorid and chlorids of the alkali-metal groups, and electrolyzing the fused mixture in an atmosphere composed of the gases produced by the electrolysis, as set forth.

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Witnesses:
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