UNITED STATES PATENT OFFICE.

BERTHOLD HOFFMANN AND ROBERT SUCHY, OF GRIESHEIM-ON-THE-MAIN, GERMANY, ASSIGNORS TO CHEMISCHE FABRIK GRIESHEIM-ELEKTRON, OF FRANKFORT-ON-THE-MAIN, GERMANY, A CORPORATION OF GERMANY.

PURIFICATION OF MAGNESIUM AND MAGNESIUM ALLOYS.

993,373. Specification of Letters Patent. Patented May 30, 1911.

No Drawing. Application filed September 28, 1910. Serial No. 584,333.

To all whom it may concern:

Be it known that we, BERTHOLD HOFFMANN and ROBERT SUCHY, subjects of the German Emperor, and residents at Griesheim-on-the-Main, Germany, have invented certain new and useful Improvements in the Purification of Magnesium and Magnesium Alloys, of which the following is a specification.

Magnesium and magnesium alloys do not sufficiently resist the influences of the atmosphere because, at their usual degree of purity, they still include metal chlorid which when it comes into contact with the moisture of the air causes efflorescences of magnesia which take place according to Lemoine (see *Chemisches Zentralblatt* 1899 Vol. II page 516) in accordance with the equation:

$$\text{Chlorid} + m\text{Mg} + m\text{H}_2\text{O} = \text{Chlorid} + m\text{MgO} + m\text{H}_2$$

as long as moisture is present and the metal is corroded at the places where such chlorid is present. The presence of said chlorid arises both from the manufacture of the metal and from the usual methods of purification of magnesium and alloys of magnesium hitherto adopted. The metal has been subjected to a process for purification either by fusing it again with carnallite, as indicated by Borchers on page 18 of his *Elektro-Metallurgy* 1901, or by melting with alkali-chlorid as proposed by Deville and Caron (see *Annales de Chimie et de Physique*, 3rd series, Vol. 67, page 340 (1863)). The molten metal has a specific gravity which is near that of the molten chlorid, so that there is no absolute separation of the chlorid and metal. Perfectly pure magnesium, free from chlorid, has already been obtained by distilling the metal (see for instance Abegg, *Handbuch der Anorg. Chemie* II, page 36, 1905, or *Sonstädt Jahresberichte* (1863) page 737) but it is obvious that such distillation on a large scale is not available owing to the impracticability of fulfilling the unusual requirements as regards the material for the vessels, due to the boiling point of the metal which is about 2200° centigrade (see R. Schenk's *Physikal. Chemie der Metalle* 1909, page 4) and owing to the readiness with which the metal combines with the elements of the air. Owing to the valuable properties of pure magnesium and pure magnesium alloys in the metal industry, a process which will remove all traces of chlorid in an economical and advantageous manner is very desirable.

We have discovered that it is possible to readily and completely free magnesium and alloys thereof from all traces of chlorid by passing through the molten metal, hydrogen, or other gas which is inert as regards magnesium. Such gas will drive off every trace of the chlorid at temperatures which are but little higher than the melting point of the chlorid.

The process may be carried out in the following manner but the invention is not limited thereto. A crucible is charged with 8 kilograms of the metal, or alloy, to be purified which may be either magnesium, or a prepared magnesium alloy, or the components of a magnesium alloy. The crucible is then closed by a cover which carries a hydrogen supply pipe extending almost to the bottom of the crucible, and a notch, or a gas outlet pipe. Then a current of hydrogen is passed at a moderate velocity through the fluid metal for 5, or 6, hours at a temperature of from 800° to 900°, centigrade. The hydrogen issuing from the crucible carries away with it the chlorid up to the very last traces thereof. The time required for the purifying operation varies, of course, within certain limits, depending on the temperature, the velocity of the gas through the metal, and the amount of metal treated.

The hydrogen or other inert gas can, if desired, be passed over the fluid metal which may be kept in motion by any suitable means. The gas may also be drawn over, or through, the bath at a reduced pressure and thus effect a removal of the chlorid at a temperature lower than that employed when the operation is carried out under normal pressure, or higher pressures obtained by any suitable means may be employed. Furthermore we have found that the time within which the purifying process is carried out and the temperature at which it takes place can be reduced considerably if in lieu of dry hydrogen or other indifferent gas a wet gas

is employed. In this case the action of the dry gas by which the chlorids contained in the metal are vaporized, is assisted by the chemical action of the water on the chlorids according to the equations:

$$MgCl_2 + H_2O \rightleftarrows MgO + 2HCl$$

and

$$MgO + MgCl_2 = Mg_2OCl_2.$$

Therefore from the chlorid are formed hydrochloric acid, according to the first equation, which acid is carried off by the hydrogen, and also oxychlorid of magnesium, according to the second equation, which floats on the metal and can easily be removed. In this way one and the same mass of metal is purified by wet gas within 1 to 2 hours and at a temperature of only 700 to 800° centigrade, while with dry gas the process requires 5 to 6 hours and a temperature of 800 to 900° C. Of course wet gas can also be employed at higher temperatures when the time of purifying is shortened. It is also a matter of course that the time within which the purification takes place depends on the velocity of the passage of the gas, on the quantity of the metal to be purified and on the degree of wetness of the gas. In carrying out the process care has to be taken that no considerable drops of water come into the fluid gas, because otherwise the reaction $Mg + H_2O = MgO + H_2$ would take place with great vehemence and the consequence would be a loss of metal.

In employing wet gas the process may be carried out as follows: The purified dry hydrogen is led through water or steam or a spray of water is admitted to it and before its entrance into the cover of the crucible the thus wetted hydrogen is again sufficiently heated to vaporize again any condensation products of the water. It is sufficient to conduct the wet hydrogen over the surface of the liquid metal if by a stirrer or the like care is taken that always new chlorid containing metal is forwarded to the surface. The hydrogen which leaves the crucible may be purified from the substances it carries with it, by any suitable means, as for instance by washing it with water and then used again. By this continuous circulation the expenditure of the gas is very small.

We claim:

1. The herein described process of purifying magnesium and its alloys from chlorids which consists in bringing a current of an indifferent gas into contact with the molten metal at a temperature above the vaporizing point of the chlorid.

2. The herein described process of purifying magnesium and its alloys from chlorids which consists in bringing a current of hydrogen into contact with the molten metal at a temperature above the vaporizing point of the chlorid.

3. The herein described process of purifying magnesium and its alloys from chlorids which consists in bringing a current of an indifferent gas into contact with the molten metal at a temperature above the vaporizing point of the chlorid and stirring the metal during the passage of the current.

4. The herein described process of purifying magnesium and its alloys from chlorids which consists in bringing a current of hydrogen into contact with the molten metal at a temperature above the vaporizing point of the chlorid and stirring the metal during the passage of the current.

5. The herein described process of purifying magnesium and its alloys from chlorids which consists in bringing a current of a wet indifferent gas into contact with the molten metal at a temperature above the vaporizing point of the chlorid.

6. The herein described process of purifying magnesium and its alloys from chlorids which consists in bringing a current of wet hydrogen into contact with the molten metal at a temperature above the vaporizing point of the chlorid.

7. The herein described process of purifying magnesium and its alloys from chlorids which consists in bringing a current of a wet indifferent gas into contact with the molten metal at a temperature above the vaporizing point of the chlorid and stirring the metal during the passage of the current.

8. The herein described process of purifying magnesium and its alloys from chlorids which consists in bringing a current of wet hydrogen into contact with the molten metal at a temperature above the vaporizing point of the chlorid and stirring the metal during the passage of the current.

In testimony whereof we have signed our names to this specification in the presence of two subscribing witnesses.

BERTHOLD HOFFMANN.
ROBERT SUCHY.

Witnesses for both applicants:
FRANZ HASSLASHER,
ERWIN DIPPEL.