L. WALDO.

PROCESS OF REDUCING MAGNESIUM COMPOUNDS.

APPLICATION FILED JULY 31, 1920.

1,379,886. Patented May 31, 1921.

Fig. 1.

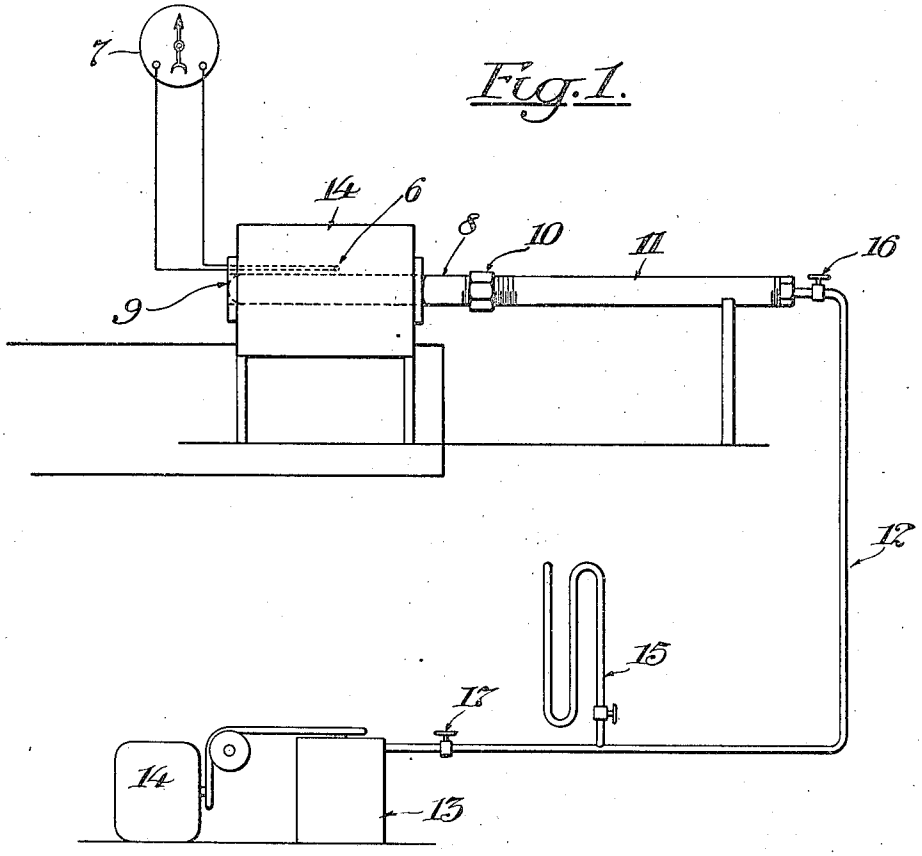
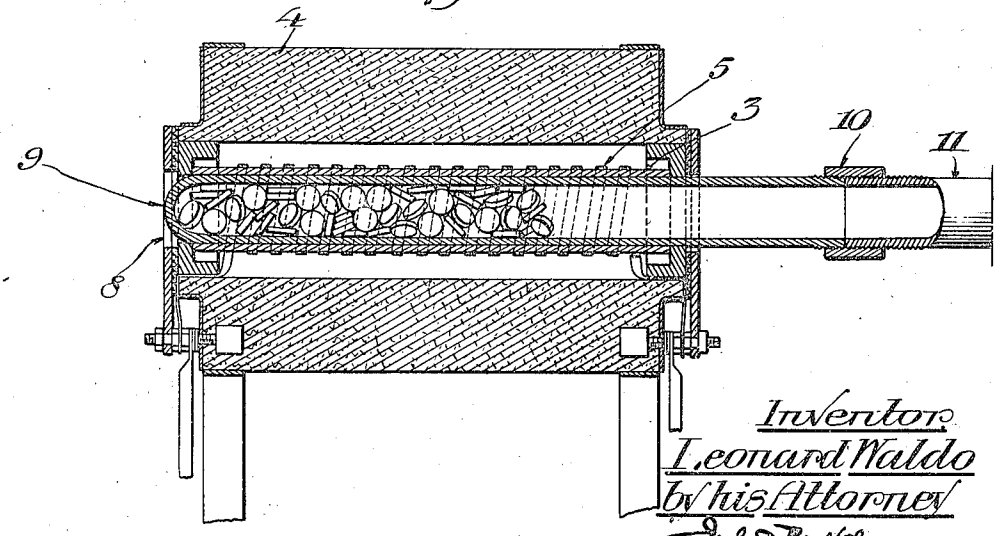


Fig. 2.



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UNITED STATES PATENT OFFICE.

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PROCESS OF REDUCING MAGNESIUM COMPOUNDS.

1,379,886. Specification of Letters Patent. Patented May 31, 1921.

Application filed July 31, 1920. Serial No. 400,554.

To all whom it may concern:

Be it known that I, LEONARD WALDO, a citizen of the United States, and resident of Plainfield, in the county of Union and State of New Jersey, have invented certain new and useful Improvements in Processes of Reducing Magnesium Compounds, of which the following is a specification.

My invention has for its object to provide a simple, efficient and economical method of reducing magnesium compounds to the metal magnesium; and more particularly the reduction of magnesium oxid to magnesium by the use of aluminum.

According to the preferred mode of practising my invention finely-divided magnesium oxid and finely-divided metallic aluminum in their proper molecular proportions are thoroughly and intimately mixed mechanically in any approved manner, such, for example, as by operations employed in the mixing of explosive powders. The mixture is compressed into compact bodies, such as tablets, balls, or the like, by any known or approved means in order to insure a close conglomeration of the particles of the two materials, and also to provide interstices among the assembled bodies. Such bodies are introduced in a retort and subjected to a temperature of about 1100° C. to 1350° C. *in vacuo*, with some such reaction as is indicated in the following equation:

$$3MgO+2Al=Al_2O_3+3Mg.$$

The reaction begins at about 900° C. and its rapidity increases as the temperature rises. Under the condition of intimate contact in the pulverulent state and at high temperature *in vacuo* the oxid of magnesium gives up its oxygen to the contiguous aluminum, forming Al_2O_3 (and possibly other little known oxids of aluminum) and releasing the metallic magnesium in a nascent state which contributes to its immediate volatilization at the high temperature. The vaporized metallic magneisium passes over a vacuum condenser and deposits itself in crystalline form on the condenser surface; the Al_2O_3 (and other oxids, if any) remaining in the retort. The magnesium thus deposited in the condenser is removed therefrom and melted according to known processes of treating magnesium.

The Al_2O_3 is removed from the retort and is reduced to the metal aluminum by any known or approved process, such aluminum being used in subsequent operations for the recovery of magnesium from MgO as previously described.

The magnesium oxid and the aluminum should be very finely comminuted (separately) and most thoroughly mixed, and the two materials should be solidly compressed before subjecting the mixture to the heating operation *in vacuo* above described, as otherwise no substantial reaction will be had. The division of the compacted materials into tablets, balls, or like units, provides throughout the charge within the retort a multiplicity of interstices for the free exit of the magnesium vapor.

The operation can be efficiently conducted in an apparatus of the character illustrated in the drawings; wherein Figure 1 is a diagram of the apparatus, and Fig. 2 is a longitudinal vertical section of an electric resistance furnace and charged retort.

The particular form of electric resistance furnace shown comprises a tube 3 of high melting point metal, such, for example, as chromel or nichrome, supported within a suitable insulating casing 4. Wound about this tube throughout its length is a coil 5, preferably of platinum, which is electrically connected with a source of electric energy of high voltage, whereby the tube can be heated to the desired high temperature. The tube is provided with a thermocouple 6 and the galvanometer 7.

A retort, comprising a metal tube 8 hermetically closed at one end 9, is adapted to be snugly entered in the furnace tube. The other end of the retort is provided with a suitable coupling 10 for one end of a relatively long tube 11 which constitutes a condenser. The outer end of the tube 11 is connected by means of a pipe 12 with an exhausting pump 13, preferably of the continuous oil type, which is geared with and operated by an adjacent motor 14. The pump, when the parts are assembled, is operated to exhaust the retort and condenser and establish therein a high degree of vacuum. The pipe leading to the pump is equipped with a vacuum gage 15 and with suitably-disposed stop-cocks, such as 16, 17.

In practising my invention the retort, charged with the above described bodies comprising MgO and Al, is introduced in the furnace tube. The condenser is then coupled to the retort and the pump is actuated until the desired high degree of vacuum

has been established in the retort and condenser. This done, the cock 16 adjacent the outer end of the condenser is closed; the operation of the pump is stopped, and the current is delivered to the furnace tube and continued until the contents of the retort are subjected to the action of a temperature of about 1350° C. *in vacuo*, which temperature is maintained until the metal Mg has been distilled over into the condenser as above described. This being accomplished the retort, together with the condenser, is withdrawn from the furnace; the retort and condenser are separated from each other by manipulation of the coupling, and the contents of the respective receptacles are removed as hereinbefore mentioned. If desired the exhausting operation of the pump can be continued during the process of reduction.

It is to be understood that the form of apparatus above described is illustrative merely, as any other suitable apparatus capable of effectually performing the steps of my novel process may be employed without departure from the spirit of my invention. It is also to be understood that the details of the process may be varied within the scope of the appended claims.

I claim—

1. A process of reducing a magnesium compound to metallic magnesium, which comprises finely dividing said compound; finely dividing metallic aluminum; intimately mixing the finely divided materials; closely compacting the mixture under pressure, and subjecting the consolidated materials to the action of a high degree of heat *in vacuo*, with the formation and distillation over of the metal magnesium.

2. A process of reducing a magnesium oxid to metallic magnesium, which comprises intimately mixing finely divided magnesium oxid and finely divided aluminum, compressing the mixture into compact bodies, and subjecting the said bodies to the action of a high degree of heat *in vacuo*, with the formation and distillation over of the metal magnesium.

3. A process of reducing a magnesium oxid to metallic magnesium, which comprises intimately mixing finely divided magnesium oxid and finely divided aluminum, compressing the mixture into compact bodies, and subjecting the said bodies to the action of a temperature from about 1100° C. to about 1350° C. *in vacuo*.

Signed at New York, in the county and State of New York, this 30th day of July, A. D. 1920.

LEONARD WALDO.