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METHOD OF MAKING METALLIC BERYLLIUM.

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To all whom it may concern:

Be it known that I, SHELDON J. DICKINSON, a citizen of the United States, and resident of Dundee, in the county of Kane and State of Illinois, have invented a certain new and useful Improvement in Methods of Making Metallic Beryllium, of which the following is a full, clear, concise, and exact description.

My invention relates to a method of producing metallic beryllium (glucinum) by electrolysis. My invention contemplates producing this metal in compact bodies of substantial size, as distinguished from powdered form. So far as I am aware, no one has heretofore been able by electrolysis, or by any other commercially feasible method, to produce metallic beryllium in any form save that of a finely divided powder consisting of oxidized particles of the metal.

I am aware that metallic beryllium has been produced by the electrolysis of a bath consisting of a beryllium salt, for instance, beryllium fluoride, fused with a salt of another element. In the electrolytic methods heretofore employed, the metal collected at the cathode of the cell has been in powdered form, and great difficulty has been experienced in obtaining sufficient of the metal in one piece to determine its characteristics. The reason for the difficulty in securing the metal in compact form is the fact that each particle of the powder obtained becomes coated with a relatively thick oxide film. The density of the film on such a particle is approximately 3, while the density of the unoxidized metal at the center of the particle is 1.86, hence, it is extremely difficult, even upon a laboratory scale, to fuse such particles together. By very tedious and purely laboratory methods, very small bodies of metallic beryllium have been produced from particles of the powder, but so far as I am aware no one has ever been able to devise a commercially feasible method of fusing the oxidized particles of the powder heretofore produced by the electrolysis of compounds of beryllium.

I have discovered that if a bath containing a suitable beryllium compound is subjected to electrolysis, while heated to a temperature higher than the melting point of metallic beryllium (approximately 1285° C.) the metal may be collected in the form of compact beads or bodies of substantial size at the cathode of the cell. The size of the beads or bodies of metallic beryllium recovered by the method of my invention can be varied by the capacity and character of the equipment employed, but in all cases when my method is employed, the metallic beryllium is deposited in compact form, as distinguished from a powder of oxidized minute particles. I have stated that the melting point of beryllium is approximately 1285° C. and I use this temperature as synonymous with the expression "the melting point of beryllium". While I believe that the temperature 1285° C. is within 5° of the melting point of the metal, it may be that the difference between the correct temperature and the temperature given by me is as great as 20°.

Heretofore, chemists seeking to obtain metallic beryllium by electrolysis apparently deemed it necessary to carry on the process of electrolysis at temperatures below the melting point of beryllium. In order to carry on electrolysis at temperatures below the melting point of beryllium, such chemists have endeavored to obtain electrolytic baths which will become fluid at temperatures less than 500° C. In carrying out the method of my invention, I effect electrolysis of the bath at a temperature greater than 1285° C., and provide a bath which will function properly at a temperature above the melting point of metallic beryllium. Other respects in which my method and the product thereof differ from prior methods and the products thereof will hereinafter appear.

In carrying out my method, I prefer to employ a bath composed of beryllium fluoride, and the fluoride of a more positive element having a melting point higher than that of the beryllium fluoride. A very satisfactory bath for my purpose consists of approximately 47 parts of beryllium fluoride to 156 parts of calcium fluoride (propor-

tions by weight). An electrolyte composed of these fluorides becomes fluid when heated to or above 1285° C. The proportions of the constituents of the electrolyte are not absolutely fixed. Thus, I find that a mixture having the composition BeF_2—$2CaF_2$ will serve my purpose. If desired, the electrolyte comprising the hereinbefore mentioned fluorides may also contain dissolved beryllium oxide, in such quantity as may be desired.

If desired, magnesium fluoride may be substituted for the calcium fluoride, the magnesium fluoride having a melting point of approximately 1396° C. With the use of magnesium fluoride, the composition of the bath differs from the bath comprising calcium fluoride, a relatively smaller percentage of magnesium fluoride being required to obtain the correct proportion. It is my opinion that other less commercially feasible compounds may be prepared to carry on my invention.

It will be understood that the structural details of the electrolytic cell may vary within wide limits. For this reason, I have not deemed it necessary or desirable to illustrate any details of the cell. I may state, however, that it is somewhat desirable to use a hollowed electrode for the cathode, but this expedient is by no means necessary.

The electrolysis of the bath is carried on at a temperature somewhat higher than 1285° C. The product deposited at the cathode of the cell as a result of the electrolysis of the bath is compact metallic beryllium in the form of beads or bodies of substantial size, the size of the beads or bodies varying with the capacity and structural detail of the cell, as hereinbefore explained. This product differs radically from the products secured by prior electrolytic processes of producing metallic beryllium. The product of my invention is capable of being used commercially for many purposes too numerous to mention. The metallic beryllium produced by prior processes has not been suitable for commercial purposes for reasons hereinbefore explained.

Having thus described my invention, what I claim is new and desire to secure by Letters Patent of the United States is:

1. The method of producing metallic beryllium which consists in subjecting a beryllium salt to electrolysis at a temperature greater than 1285° C.

2. The method of producing metallic beryllium which consists in forming an electrolyte consisting of a beryllium salt and the salt of another element having a higher melting point than the beryllium salt, and subjecting said electrolyte to electrolysis at a temperature greater than 1285° C.

3. The herein described method of producing metallic beryllium by electrolysis which consists in forming an electrolyte consisting of a beryllium salt and a salt of a more positive element, and subjecting the electrolyte to electrolysis at a temperature not less than the melting point of metallic beryllium.

4. The method which consists in obtaining metallic beryllium by the electrolysis of a bath composed of beryllium fluoride and the fluoride of a more positive element.

5. The method of producing beryllium which consists in forming an electrolyte of a beryllium salt and a salt of a more positive element having a higher melting point than the beryllium salt, and subjecting the electrolyte to electrolysis at a temperature of not less than 1285° C.

6. The method which consists in obtaining metallic beryllium by the electrolysis of a bath composed of beryllium fluoride and the fluoride of a more positive element having a higher melting point than the beryllium fluoride.

7. The method of producing beryllium by the electrolysis of a bath composed of beryllium fluoride and calcium fluoride.

8. The method of producing beryllium which consists of subjecting a bath composed of beryllium fluoride and calcium fluoride to electrolysis at a temperature greater than the melting point of beryllium.

9. The method of producing metallic beryllium by the electrolysis of a bath composed of a minor portion of beryllium fluoride, and a major portion of calcium fluoride.

10. The method of producing metallic beryllium by the electrolysis of a bath consisting of approximately 47 parts of beryllium fluoride, and 156 parts of calcium fluoride.

11. The method which consists in the electrolysis of a bath consisting of beryllium fluoride, calcium fluoride and beryllium oxide.

12. The method which consists in subjecting a halogen salt of beryllium to electrolysis at a temperature greater than the melting point of beryllium.

13. The method which consists in forming an electrolyte of a halogen salt of beryllium, and a halogen salt of a more positive metal, and subjecting the electrolyte to electrolysis at a temperature greater than the melting point of beryllium.

14. The method which consists in forming a bath consisting of a halogen salt of beryllium and a halogen salt of another element, which has a higher melting point than the beryllium salt, and subjecting the electrolyte to electrolysis at a temperature greater than the melting point of beryllium.

15. The method which consists in forming a bath consisting of a halogen salt of beryllium, beryllium oxide, and the halogen

salt of another element which has a higher melting point than the beryllium salt, and subjecting the electrolyte to electrolysis at a temperature greater than the melting point of beryllium.

16. The method which consists in subjecting a compound of beryllium to electrolysis at a temperature greater than the melting point of metallic beryllium.

17. The product "compact metallic beryllium".

18. Compact beryllium produced by the electrolysis of a beryllium salt.

19. Electrolytically produced compact beryllium.

In witness whereof, I hereunto subscribe my name this 17 day of September, 1921.

SHELDON J. DICKINSON.

Witnesses:
CHAS. C. WOLAVER,
HANNAH THAMS.