June 11, 1940. J. H. DAVIS ET AL 2,203,895

METHOD OF SINTERING POROUS METAL OBJECTS

Filed Jan. 6, 1939

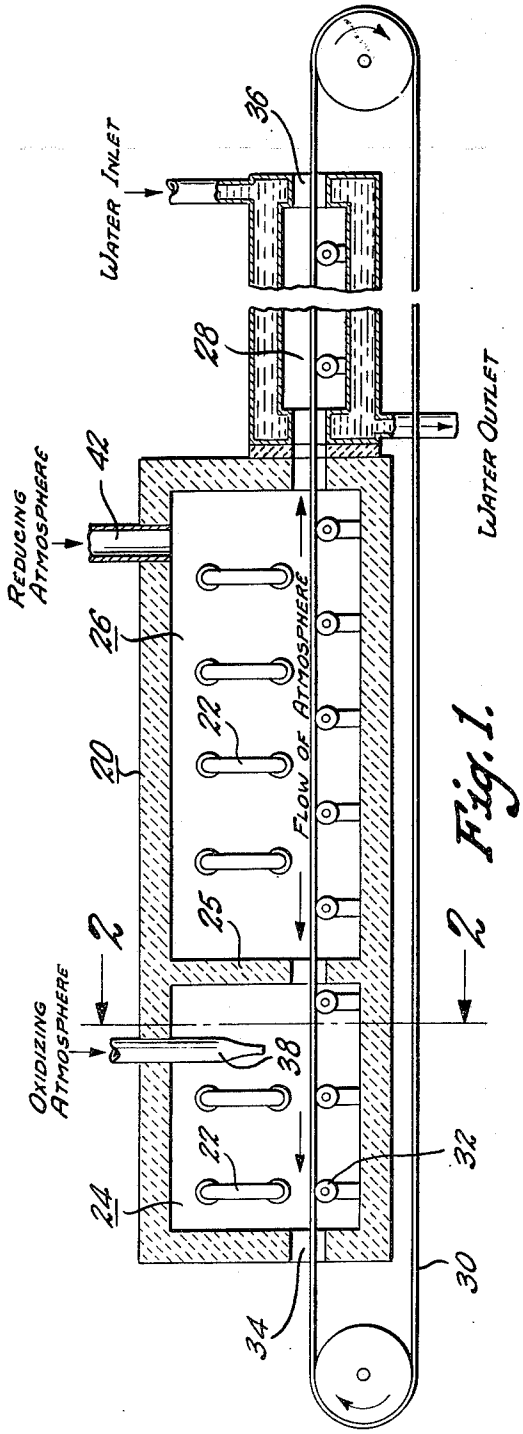



Fig. 1.

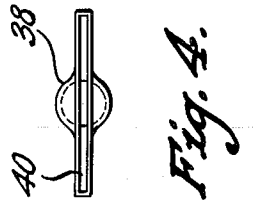



Fig. 4.

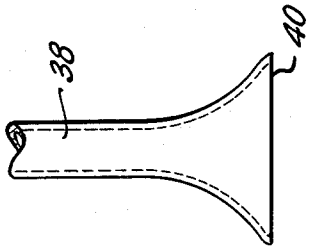



Fig. 3.

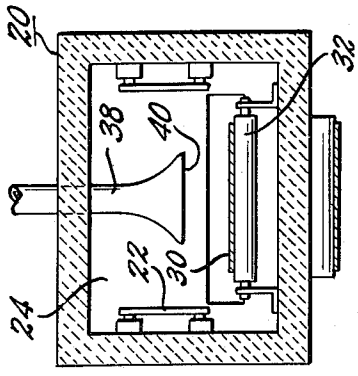



Fig. 2.

James H. Davis
Roland P. Koehring
John T. Marvin
INVENTORS

BY
Spencer Hardman & Fehr
their ATTORNEYS

Patented June 11, 1940 2,203,895

# UNITED STATES PATENT OFFICE

2,203,895

## METHOD OF SINTERING POROUS METAL OBJECTS

James H. Davis, Roland P. Koehring, and John T. Marvin, Dayton, Ohio, assignors to General Motors Corporation, Detroit, Mich., a corporation of Delaware

Application January 6, 1939, Serial No. 249,560

14 Claims. (Cl. 75—22)

This invention relates to an improved method of sintering briquettes made from powdered metal and is particularly concerned with a method of sintering briquettes wherein the appearance and strength of the sintered briquettes are improved.

An object of the invention is to provide a method of sintering briquettes made from powdered metal and including a small quantity of carbonaceous material such as graphite and/or organic compounds, wherein the carbonaceous material is at least partially burned out in an oxidizing atmosphere at substantially the sintering temperature, and then the article is further sintered under reducing conditions for reducing any metal oxides present.

Another object is to provide a method of sintering briquettes made from powdered metal and including a small quantity of carbonaceous material wherein the briquettes are sintered in an atmosphere which will chemically combine with the carbon for at least partially removing the carbon from the briquettes.

A further object is to provide a method of sintering a metal briquette wherein the surface of the briquette, after sintering has the natural color of the metal or metallic alloy which is predominant in the briquette.

A still further object is to provide a method of sintering a powdered metal briquette which includes small quantities of graphite dispersed therethrough, wherein the graphite content adjacent the surface of the sintered briquette may be closely controlled.

In carrying out the above object it is a further object to utilize an oxidizing atmosphere to burn out a portion of the graphite and then utilize a reducing atmosphere to reduce any metal oxides present while the briquette is being further sintered.

Further objects and advantages of the present invention will be apparent from the following description, reference being had to the accompanying drawing wherein a preferred embodiment of the present invention is clearly shown.

In the drawing:

Figure 1 is a view in section of a sintering furnace used in carrying out the hereindescribed invention;

Figure 2 is a view taken on line 2—2 of Figure 1;

Figure 3 is a detailed view of the inlet pipe utilized for distributing the oxidizing atmosphere in the furnace; and

Figure 4 is an end view of the inlet pipe shown in Figure 3.

When sintering metal articles, especially briquettes made from copper powder or mixtures including copper powder as one of the constituents, it has always been a distinct problem to obtain a sintered article in which the metal surface thereof has a bright finish corresponding to the natural color of the metal in the article. In the past, sintered copper containing articles have been dark appearing at the surface thereof due to a deposit of carbon thereon. This carbon is one of the break-down products of residual carbonaceous material used as salicyclic or tannic acid used to form voids or lubricants used in the briquetting of the article. It is conventional practice when mixing metal powders for subsequent briquetting to include a small quantity of a lubricant to prevent excessive wear of the briquetting dies and to facilitate the briquetting of the metal. Common lubricants that are in use at the present time include lubricating oils, fatty acids such as stearic, oleic and palmitic acids, as well as metallic salts of fatty acids such as zinc, copper or aluminum salts thereof. Any of these organic die lubricants volitalize and decompose upon heating and deposit carbon in an amorphous form upon the surface of the article thereby discoloring the same as well as partially filling the pores. This dark finish is very difficult to remove and at best requires additional production steps after the sintering of the article and therefore results in increased production costs.

This invention is directed to a method whereby metallic briquettes containing carbonaceous material may be sintered and removed from the furnace with a bright finish thereon corresponding to the natural color of the metal or metallic alloy therein.

It has also been discovered, when sintering metal briquettes which include graphite dispersed therethrough, that the present method may also be advantageously utilized to control the quantity of the graphite adjacent the surface of the article. The inclusion of graphite in porous metal articles is conventional practice since the graphite acts as an additional die lubricant and is also desirable in articles which are to be used for frictional or anti-frictional purposes such as bearings, etc. It is desirable, however, that a bearing have only a small quantity of graphite adjacent the bearing surface and the present invention provides a means of controlling the graphite content at the surface. In

most cases graphite and another die lubricant are both added to the preliminary metal mix although either one or the other of these lubricants may be used to the exclusion of the other.

Referring to Figure 1, apparatus for carrying out the invention is shown schematically wherein 20 illustrates a furnace having a plurality of heating elements 22 therein. It is to be understood that the furnace may also be heated by any other well known means such as gas burning either at the inside or the outside thereof in a conventional manner. The furnace 20 comprises two chambers 24 and 26 which are separated by an apertured wall 25. The furnace may also be provided with a water jacketed cooling chamber 28. An endless conveyor 30 passes through the furnace and through all of the chambers and is supported by a plurality of rollers 32. The conveyor enters the furnace through an opening 34 and leaves through an opening 36 in the water chamber. Thus, articles placed upon a conveyor move progressively through the furnace passing in order through chamber 24, chamber 26 and chamber 28 at a substantially constant rate of speed. An inlet pipe 38 is disposed within chamber 24 and includes a fan shaped end 40 thereon which comprises the outlet of the pipe 38 and which is disposed directly over the conveyor 30. An oxidizing atmosphere is admitted to the chamber 24 through the pipe 38 and due to the fan shaped outlet 40 on the pipe 38 the gas completely envelopes any articles on the conveyor 30. Air, or other oxygen containing gases, are suitable for the oxidizing atmosphere, although a gas, such as $CO_2$, which will chemically combine with the carbon will accomplish the desired purpose. Thus, as articles pass into chamber 24, they are heated and the carbonaceous material therein decomposes and the carbon is burned out in the oxidizing atmosphere thereby removing the carbon from the surface of the briquette. Simultaneously the oxidizing atmosphere combines with a portion of the graphite thereby removing the graphite from the external surfaces of the briquette and to any desired depth from the surface as controlled by the period of oxidation, the temperature and the effective oxygen content of the oxidizing gas. During this oxidation step some of the metal in the briquette is likewise oxidized to form the corresponding metal oxide. Continued movement of the conveyor carries the articles into the second chamber, namely chamber 26, wherein the articles continue to be heated to finally sinter the metal therein and produce a homogeneous porous metal object. A reducing atmosphere is supplied to chamber 26 through an inlet 42. In this manner any metal oxides which are present in the briquette are reduced in the chamber 26 while the briquette is being sintered. Further movement of the conveyor 30 carries the sintered briquettes into a water cooled chamber 28 and then out of the furnace. The reducing atmosphere may be any of the well known reducing atmospheres, such as hydrogen, carbon monoxide or mixtures thereof, but is preferably natural gas burned in an atmosphere having insufficient oxygen to permit complete combustion thereof. The reducing atmosphere is admitted to the chamber 26 under pressure in excess of atmospheric pressure, and sufficiently high to cause a circulation of a reducing atmosphere toward both the inlet 34 and the outlet 36 of the furnace as shown by the arrows in Figure 1. In this manner no oxidizing atmosphere ever enters the chamber 26 when the furnace is in operation. In some instances, if desired, the atmosphere may be composed of a gas which will chemically combine with the carbon in the carbonaceous material. One of such gases is $CO_2$ which when in contact with hot carbon reacts as follows:

$$CO_2+C\rightarrow 2CO$$

Thus the resulting atmosphere is of a reducing nature thereby only necessitating the use of a single atmosphere source.

It has been found by chemical analysis that the graphite control is quite marked as noted from the following figures taken from analysis of a central bronze bearing having a wall thickness of .250":

| | Per cent |
|---|---|
| Carbon at the surface | 0.0 |
| Carbon .030 from the surface | 1.62 |
| Carbon .060 from the surface | 1.72 |
| Carbon .125 from the surface | 2.04 |

It is believed that in some cases that the removal of the graphite at the surface of articles such as bearings is beneficial, since the graphite combined with the lubricating oil forms a heavy, thick sludge, which has the tendency to decrease the oil flow through the pores of the bearing.

It has further been discovered that the treatment herein described has a marked effect on the strength of the porous metal articles. For example an increase of 20 percent has been noted in the radial strength of articles sintered by the present process over articles sintered by conventional methods wherein only a reducing gas is utilized. One theory explaining this increased strength is the fact that the surface of some of the metal particles is made porous by the oxidation thereof, which porous metal has an activated surface toward alloying with other constituent metals in the briquette and upon reduction of the metal oxide, thereby making the article more homogeneous in character. Furthermore some of the carbon in the briquette is removed and thus permits more ready contact of larger surfaces of adjacent metal particles.

The temperatures maintained in the sintering furnace and the time period of sintering are similar to well known practices when sintering metal briquettes. For example, when sintering briquettes of copper and tin powders a temperature of from 1400° F. to 1600° F. is preferably utilized and a time period of from ten minutes to one-half hour is preferred. The time period that the briquette should remain in the oxidizing zone is best arrived at by experiment according to the ultimate result desired. When sintering small bearings for example, it is sufficient to leave the briquette in the oxidizing zone for about three minutes when using air, during a ten minute total sintering interval to remove the surface carbon deposit and burn out the graphite at the surface thereof. It is manifest that longer periods of sintering can be used with equally successful results and that the times and temperatures can vary considerably according to the ultimate results required since close control of such variables is not critical to the ultimate success of our invention.

The present method produces porous metal articles which are free from deposited carbon on the outer surfaces thereof, and which have a finish corresponding to the natural color of the metal therein. It further produces an article of increased strength over articles of similar con-

stituency made by conventional methods, as well as providing a means for accurately controlling the carbon content adjacent the surfaces of the article.

While the embodiment of the present invention as herein disclosed, constitutes a preferred form, it is to be understood that other forms might be adopted, all coming within the scope of the claims which follow.

What is claimed is as follows:

1. The steps in the method of sintering briquettes made from powdered metal and including a small quantity of carbonaceous material therein, comprising, heating the briquette in an oxidizing atmosphere for a time sufficient to burn out at least a portion of the carbonaceous material, and then heating the briquette in a reducing atmosphere for a time sufficient to reduce any metallic oxide present and for simultaneously sintering the briquette and increasing its strength.

2. The steps in the method of sintering briquettes made from powdered metal including a small quantity of organic material, comprising, heating the briquette under oxidizing conditions for burning out at least a portion of the organic material and then heating the briquette under reducing conditions for reducing any metallic oxide present and for simultaneously sintering the briquette and increasing its strength.

3. The steps in the method of sintering briquettes made from powdered metal including a small quantity of organic material, comprising, initially heating the briquette in an oxidizing atmosphere for burning at least a portion of the organic material and then continuing to heat the briquette in a reducing atmosphere for reducing any metallic oxide present and for simultaneously sintering the briquette.

4. The steps in the method of sintering briquettes made from powdered metal which include a small quantity of organic material and a small quantity of graphite uniformly dispersed therethrough, comprising, heating the briquette in an oxidizing atmosphere for burning out the organic material and for burning at least a portion of the graphite adjacent the surfaces of the briquette, and then heating the briquette under reducing conditions for reducing any metallic oxide present and for simultaneously sintering the briquette and increasing its strength.

5. The steps in the method of sintering briquettes made from powdered metal and residual organic material comprising the steps of heating the briquette in an oxidizing atmosphere for burning out at least a portion of the residual organic material present, and then changing the atmosphere to a reducing atmosphere while maintaining the temperature condition of the briquette to finally sinter the briquette and to reduce any oxide coating thereon for causing the briquette to have a bright finish corresponding to the natural color of the metal therein, and for increasing the strength thereof.

6. The steps in the method of sintering briquettes made from powdered metal including a small quantity of graphite uniformly dispersed therethrough, comprising the steps of oxidizing the briquette at a sufficiently high temperature to substantially remove the graphite from at least the surface of the briquette, and then heating the briquette under reducing conditions for reducing any metal oxides present and for sintering the briquette and increasing its strength.

7. The steps in the method of sintering metal briquettes comprising providing a sintering furnace having at least two connecting chambers therein, progressively passing metal briquettes containing carbonaceous material through said chambers, introducing oxidizing gas into a first of said chambers in sufficient quantities to molecularly combine with said carbon for forming a gas, supplying reducing gas in a second of said chambers at a pressure sufficient to prevent ingress of any extraneous gases into said second chamber, said reducing gas reducing the metal oxide in the briquette while the briquette is being sintered for increasing the strength and improving the appearance of the briquette.

8. The steps in the method of sintering powdered metal briquettes including small quantities of carbonaceous material comprising passing the briquettes at a substantially constant speed through a sintering furnace having at least two connecting chambers therein, introducing oxidizing gas into one of said chambers in sufficient quantity to molecularly combine with said carbon in the briquettes, introducing reducing gas in the second of said chambers at a pressure sufficient to cause reducing gas to flow countercurrent to the movement of said briquettes through said chambers whereby the reducing gas prevents the ingress of any extraneous gases into said reduction chamber said reducing gas reducing any metallic oxide present in the briquette while the briquette is being sintered for increasing the strength and improving the appearance of the briquette.

9. The steps in the method of sintering briquettes made from powdered metal which includes small quantities of carbonaceous material comprising heating the briquette at a temperature sufficient to cause the briquette to sinter into a strong homogeneous porous metal article and progressively introducing an oxidizing gas and then a reducing gas into the heating chamber for burning at least a portion of the carbonaceous material and reducing any metallic oxide present and for increasing the strength and improving the appearance of the briquette, and then cooling the sintered briquette under non-oxidizing conditions.

10. The steps in the method of controlling the graphite content at the surface of a metal briquette comprising, sintering the briquette in an oxidizing atmosphere for a time sufficient to burn a desired quantity of graphite adjacent the surface of the briquette and then changing the atmosphere to a reducing gas while continuing the sintering for reducing any metal oxides present and increasing its strength.

11. The method as disclosed in claim 1 wherein the oxidizing step is carried out in an atmosphere of air.

12. The method as disclosed in claim 1 wherein the oxidizing step is carried out in an atmosphere of carbon dioxide.

13. The step in the method of sintering cuprous articles made from powdered metal which include a small quantity of carbonaceous material, comprising the steps of; providing an article made from powdered metals which contains a substantial percentage of copper, heating the article under oxidizing conditions at a temperature below the melting point of copper and for a time sufficient to burn out at least a portion of the carbonaceous material and partially oxidize the copper, then continuing to heat the article under reducing conditions at a temperature below the melting point of copper for reducing the copper

oxide and for completing the sintering and simultaneously increasing the strength of the article.

14. The steps in the method of heating briquettes made from a mixture of powdered copper and tin which includes a small quantity of carbonaceous material, comprising the steps of: heating the briquette under oxidizing conditions at a temperature below the melting point of copper and above the melting point of tin for a time sufficient to burn out at least a portion of the carbonaceous material, and then continuing to heat the briquette at a temperature below the melting point of copper and above the melting point of tin under reducing conditions for reducing any metal oxides present and for simultaneously completely sintering the briquette and increasing its strength.

JAMES H. DAVIS.
ROLAND P. KOEHRING.
JOHN T. MARVIN.