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METHOD OF SINTERING NICKEL POWDER ONTO STAINLESS STEEL

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This invention relates to sintering methods and is particularly concerned with sintering procedures used in connection with metal powder wherein the metal powder is preferably bonded to a strong metal supporting back.

In past disclosures there have been numerous methods set forth by which loose or substantially non-compacted metal powder may be bonded to a strong metal supporting back such as a steel back. Koehring Patents 2,198,253 and 2,198,254 disclose such methods.

In the preferred embodiments disclosed in these patents it is most desirable to have the metal powder layer to be sintered and bonded to the supporting back made up of a combination of metals including a high and a low melting point metal such as copper and tin or nickel and copper, etc., whereby a sintering temperature is selected which is intermediate the melting points of the several metals. By this procedure, as the sintering process proceeds, increments of the lower melting point metal melt and diffuse into the higher melting point metal whereby the metal powders are sintered together and alloyed and are simultaneously bonded to the supporting back without the formation of any noticeable or appreciable quantity of molten constituent being present due to the progressive alloying action of the metals.

Sintering and bonding procedures used with substantially loose non-compacted metal powder become increasingly difficult when a single metal powder is utilized, since bonding of adjacent particles and bonding of said particles to the supporting back must rely upon diffusion alone in view of the fact that the sintering temperature is never elevated to a point in excess of the melting point of the metal powder. For this reason, procedures on single metal powders require relatively high temperatures and relatively longer periods of heating in order to accomplish any useful result.

I have found that when sintering nickel powder to a steel, stainless steel or nickel backing member that temperatures in the order of 2050° F. applied for a period of one hour cause the formation of a bond between the nickel powder and the backing member but that said bond is not of an extremely strong nature and in fact longer sintering periods at higher temperatures are usually required to obtain useful results.

Obviously high temperature sintering, that is to say, sintering at temperatures above 2000° F. is costly and requires special furnace equipment which makes such sintering procedures highly undesirable.

The present invention is directed to a method for sintering together a substantially loose non-compacted single metal powder and for bonding said sintered powder onto a strong metal supporting back whereby the normal sintering temperature may be reduced drastically and wherein the bond ultimately obtained is stronger than that usually obtained by using the higher temperatures.

It is therefore the prime object of the invention to provide a method for bonding and sintering substantially non-compacted metal powder onto a strong metal sup-

porting back wherein the temperature of the operation is reduced and the strength of the resulting bond is increased.

In carrying out this object it is a still further object of the invention to utilize electroless plating on the supporting member and in some cases on the metal powder whereby the plating has a lower melting point than the metal powder or backing member and therefore requires lower temperatures for sintering and bonding, the plate being of such nature that it is completely diffused during the sintering operation.

Specifically, it is a further object of the invention to use as an electroless plate a deposit of a phosphide of the metal powder being sintered whereby the bonding operation either between the metal particles or between the metal particles and the supporting member is accomplished by sintering at a temperature above the melting point of the metal phosphide eutectic whereby extremely strong bonds are obtained at reduced temperatures.

Further objects and advantages of the present invention will be apparent from the following description.

Specifically, one of the most difficult sintering operations to accomplish satisfactorily is the bonding of nickel powder to stainless steel wherein the nickel powder is in the substantially loose non-compacted condition. I have found that this can be readily accomplished through the use of an electroless nickel plate whereby the sintering temperature may be reduced between 300° F. and 350° F. and wherein the bond may be improved many fold.

A specific example of the operation is as follows. Nickel powder having a mesh size of from 80 to 200 may be bonded to stainless steel, for example, stainless steels having the following designations and compositions:

- #430 SAE (6 to 8% nickel and 16 to 18% chromium)
- #431 SAE (1.25 to 2.5% nickel and 15 to 17% chromium)
- #316 SAE (10 to 14% nickel, 16 to 18% chromium and 2 to 3% molybdenum)

The stainless steel backing member is first prepared for the sintering operation by the deposition of an electroless plate thereon. This is accomplished by first cleaning the stainless steel strip in an alkaline, cleaner, rinsing, dipping in a 25% hydrofluoric acid solution for about 30 seconds, rinsing and then activating the surface by anodizing the strip for two minutes at 20 amperes per square foot current density in a solution containing 32 ounces of nickel-chloride (NiCl_2) and 11 ounces of hydrochloric acid per gallon of water. After this activation the strip is preferably nickel plated for four minutes at a 40 ampere per square foot current density in a solution containing 64 ounces of nickel-chloride (NiCl_2) and 11 ounces of hydrochloric acid per gallon of water at room temperature. The strip is then rinsed and dipped in the electroless nickel bath where it is maintained for about one hour at 190° F.

A satisfactory electroless nickel bath comprises:

- .6 lb. sodium citrate
- .4 lb. ammonium chloride
- 1 oz. sodium hypophosphate
- 5 cc. ammonium hydroxide
- .25 lb. nickel-chloride (NiCl_2)

per gallon of water. At the end of the hour a nickel phosphide coating will have deposited on the stainless steel sheet and the sheet is then ready for the sintering operation.

For this purpose nickel powder of a desired mesh size, for example 80 to 200 mesh, is spread in a uniform substantially non-compacted layer upon the dried sur-

face of the stainless steel sheet which has previously been electroless plated. This sheet with the nickel powder thereon in the desired thickness is then placed in a furnace maintained at about 1700° F. for about one hour. Temperatures of 1650° F. minimum may be used if the time is extended. The furnace atmosphere is preferably dehydrated, incompletely burned natural gas, hydrogen, cracked ammonia, etc. At the end of the hour it will be found that the nickel powder layer is sintered together into a highly porous nickel layer which is strongly bonded to the surface of the strip. It will also be noted that the nickel phosphide which was of microscopic thickness has completely diffused and disappeared.

In the above example, which is given for illustrative purposes only, the nickel plate may be left out of the procedure although stronger bonds appear to be obtained when this secondary plating is used. Also sintering times, temperatures and atmospheres may vary so long as a good bond is obtained. The specific example given for the preparation of the stainless steel sheet is a preferred procedure, variations in concentration, current densities and times may occur within reasonable limits without altering markedly the ultimate result.

In place of stainless steel it is apparent that the bond may be made directly to nickel sheet, carbon steel, chrome iron or other alloy steels by using the same basic procedure. Similarly, the electroless plate may also be an iron phosphide, copper phosphide, silver phosphide, chromium phosphide, etc., by using suitable salts of these various metals in the electroless plating solution. In each instance the plate, being a phosphide, melts at a considerably lower temperature than the pure metal powder and therefore all of these plates may be used in connection with sintering procedures wherein, for example, iron, copper, silver or chromium powders are to be sintered and bonded respectively to a strong metal supporting back. In other words, the electroless plate should be chosen in connection with the metal powder used so that no extraneous metal is present, since it is apparent that if two metals are to be used they can be chosen with different melting points whereby the procedures suggested in the aforementioned Koehring patents may be utilized to promote the bonding and sintering action.

In some cases where an extremely strong porous metal layer is desired, it may be desirable to coat the powder with the electroless plate. In this instance the powder may be coated in much the same manner as the strip. For example, nickel powder that is to be electroless plated with nickel phosphide is preferably washed in an alkaline cleaner, rinsed and then activated in a 5% hydrochloric acid solution at room temperature. After rinsing, this powder is stirred in an electroless nickel bath at 190° F. for one hour. The bath is preferably maintained at a pH of from 8 to 9. At the end of the period the powder is removed from the bath, rinsed and dried, and it will be found that each particle includes a covering or coating of the nickel phosphide. When this powder is used it is apparent that a very strong bond will be obtained although in most instances this secondary coating operation is not necessary.

It is manifest that alloy powders, such as stainless steel powder, may be bonded and sintered by the same procedures. In this instance the electroless plate may be either nickel or chromium and after the sintering operation no extraneous metal will be detectable since the electroless plate comprises one of the metals of the alloy.

From the foregoing it is apparent that I have provided a method by which single metal powders may be sintered and bonded to metal supporting surfaces without the introduction of extraneous metals used for bonding purposes and wherein the effective temperatures used during the bonding operation may be reduced and while the strength of the bond may be greatly increased. This objective has been gained through the use of electroless

plates wherein a metal derivative of the metal to be sintered, preferably a phosphide, is deposited on the surface of the strong metal support which deposit has a melting point well below the melting point of the metal per se. In all cases the melting point of this deposit is equal to or above the melting point of the metal phosphorus eutectic and in some cases the deposit per se may be the eutectic composition which in the case of nickel contains in the order of 11.4% phosphorus.

The procedures set forth herein are illustrative of a satisfactory approach to the problem. It is apparent that the surface of the metal sheet to which the metal powder is to be bonded may be prepared in various other ways than those set forth. However, it is preferable to activate the surface so that the electroless plate may be satisfactorily deposited thereon. This particular phase of the process forms no part of the invention which is specifically directed to the bonding of a highly porous metal layer to a strong metal supporting member. It is also pointed out that electroless plates per se are well known in the art. For example, immersion coatings of this nature are disclosed in many articles, one of which, "Nickel Immersion Coatings" by W. A. Wesley, may be found in the July 1950 issue of "Plating." In my copending application S. N. 528,899 filed concurrently herewith I set forth a specific use for the process wherein a strong porous nickel element is desirable.

While the forms of embodiment of the invention as herein disclosed constitute preferred forms, it is to be understood that other forms might be adopted, as may come within the scope of the claims which follow.

What is claimed is as follows:

1. In a method for sintering and bonding a layer of nickel powder in the substantially non-compacted condition onto the surface of a stainless steel supporting back, the steps comprising; depositing a uniform layer of electroless nickel phosphide coextensively onto the surface of the steel, distributing a layer of nickel powder onto said surface, and then heating the assembly to a temperature of about 1650° F. for a period of about one hour under suitable atmospheric conditions to cause substantially complete diffusion of said metal phosphide into both said stainless steel and said nickel whereby the particles of the nickel powder are bonded together and are strongly bonded to the surface of the stainless steel back.

2. In a method for sintering and bonding a layer of nickel powder in the substantially non-compacted condition onto the surface of a stainless steel supporting back, the steps comprising; activating the surface of the stainless steel surface, depositing a uniform layer of electroless nickel phosphide coextensively onto the surface of the steel, distributing a layer of nickel powder onto said plated surface, and then heating the assembly to a temperature of about 1650° F. for a period of about one hour under suitable atmospheric conditions to cause substantially complete diffusion of said metal phosphide into said stainless steel and said nickel whereby the particles of the nickel powder are bonded together and are strongly bonded to the surface of the stainless steel back.

3. In a method for sintering and bonding a layer of nickel powder in the substantially non-compacted condition onto the surface of a stainless steel support, the steps comprising; activating the surface of the stainless steel strip, nickel plating said surface, depositing a uniform layer electroless plate onto the nickel plated surface of said stainless steel support which electroless plate consists of nickel phosphide, distributing a layer of nickel powder onto the plated surface of the support, and then heating the assembly to a temperature in the order of 1700° F. for a period of about one hour under non-oxidizing conditions for causing substantially complete diffusion of said nickel phosphide into the support and into the particles of said nickel powder whereby the nickel

powder is bonded together and is strongly bonded to the surface of the support.

4. The method as claimed in claim 1 wherein the nickel powder is electroless plated with a layer of nickel phosphide prior to its distribution on the plated surface of the back. 5

5. The method as claimed in claim 3 wherein the nickel powder is electroless plated with nickel phosphide prior to its distribution on the plated surface of the support.

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