

1

3,398,448

PROCESS FOR COATING STEEL WITH NICKEL
Charles B. Goodrich, Charles E. Manilla, Keith E. Creager, and Harper J. Rudge, Huntington, W. Va., assignors to The International Nickel Company, Inc., New York, N.Y., a corporation of Delaware
No Drawing. Filed Sept. 12, 1966, Ser. No. 578,469
11 Claims. (Cl. 29—420.5)

The present invention relates to a method for nickel coating rectangular-sectioned, rolled steel products resulting from the primary mill and, more particularly, to a method for applying a nickel coating to such a rolled steel primary mill product using nickel powder as the source of nickel. Attempts have been made to provide final steel products such as sheets having a thin nickel coating applied thereto as a direct steel mill product. Thus, it has been recognized that steel sheets and other rolled shapes having applied thereto a thin nickel coating such, for example, as a coating approximately one mil in thickness would be very useful in many applications due to the improved corrosion resistance, improved paint holding qualities, and other advantages which such products would possess. Methods are available for applying nickel coatings upon the finished steel products. Such methods include electroplating, direct application of reduced nickel coatings, etc., to the finished steel. However, such methods are quite expensive and, in fact, the cost of applying such coatings is prohibitively expensive from the commercial viewpoint.

In order to provide the desired final nickel-coated steel mill product, it is evident that very substantial economies could be achieved if a nickel coating of the required properties could be applied to heavy steel rolled shapes such as those resulting from the primary mill. In this way, the primary mill products could be reduced to the final steel mill shape using conventional steel mill practice. It has been found, however, in practice that it is a very difficult matter to apply a nickel coating having satisfactory properties to a heavy, rolled steel shape of the type resulting from the primary rolling mill. For example, attempts have been made to apply heavy, electroplated nickel coatings to rolled steel primary mill products such as billets, blooms, slabs and the like but these attempts have not been completely satisfactory in terms of quality of finished product, excessive costs have been encountered and it has been found that large plant outlays are required in order to carry such schemes into effect.

Although many attempts have been made to overcome the foregoing difficulties and other difficulties, none as far as we are aware was entirely successful when carried into practice commercially on an industrial scale.

It has now been discovered that a satisfactory nickel coating can be applied to rolled steel primary mill products using nickel powder as the source of nickel to produce a coating having satisfactory properties and characteristics on the rolled steel primary mill product which method produces a nickel-coated rolled steel primary mill product satisfactory for reduction to a final steel mill product, e.g., sheet, having a thin, substantially uniform nickel coating thereon.

It is an object of the present invention to provide a method for applying a nickel coating to a face of a rectangular-sectioned, rolled steel primary mill product.

Another object of the invention is to provide a method for applying a nickel coating on a rolled steel primary mill product using nickel powder as the source of nickel.

The invention also contemplates providing an effective method for applying a nickel coating derived from nickel powder upon a rolled steel primary mill product having a rectangular section.

2

A further object of the invention is to provide a method for applying a nickel coating to a rolled steel primary mill product which produces a nickel-coated product susceptible for handling in a steel mill production scheme without substantial modification thereof.

Other objects and advantages will become apparent from the following description.

Generally speaking, the present invention contemplates coating a rolled steel primary mill product having a rectangular section on at least one major face thereof by a process comprising cleaning at least one major face of such steel product to remove mill scale, surface imperfections, etc., applying a thin copper coating upon said cleaned face, applying to said copper-coated face a substantially uniform coating of a fine nickel powder having high purity, covering the powder layer with a nickel or nickel alloy, e.g., a nickel-copper alloy containing up to 60%, by weight, of copper, foil having a thickness of about 0.001 to about 0.006 inch to prevent sticking to a hot platen in pressing, pressing said nickel powder layer under a compressive stress sufficient to prevent lateral shrinkage thereof while heating said nickel powder layer and continuing said heating and pressing until the temperature of said nickel powder layer and the interface between said powder layer and the steel is raised to a point above the recrystallization temperature of the nickel thereby to effect substantial densification of the nickel powder layer and thereafter heating the resulting assembly to a hot rolling temperature in an atmosphere nonoxidizing to nickel and hot rolling the heated assembly to sheet having a substantially completely densified nickel layer metallurgically bonded to a steel base. Preferably, the pressure in the hot pressing operation is maintained for some time, e.g., 1/2 or 1 or 2 or 10 minutes up to 30 or 60 minutes, after the nickel layer has become heated, since layer densification and bonding are time dependent as well as temperature dependent.

In carrying the invention into practice, it is important that the face of the rectangular-sectioned rolled steel primary mill product (which may be termed a billet, slab or bloom depending upon the parlance employed at a particular steel mill) be cleaned of surface oxides and that mechanical imperfections in the surface to be coated be removed. Conventional operations including pickling, sand blasting, chipping, milling, grinding, etc., may be employed for this purpose. The cleaned face of the rectangular-sectioned, rolled steel primary mill product is then coated with a thin copper coating which may be applied by electrodeposition, galvanic deposition (by displacement of copper from a solution of a copper salt), by painting with a copper paint, spray coating, etc. An electroplated copper coating about 0.2 to about 10 mils thick is very satisfactory. A uniformly distributed layer of nickel powder is then applied to the cleaned and copper-coated face of the rolled steel primary mill product. The powder may be distributed in the dry state and leveled with a doctor blade or it may be applied as a slurry in which case the layer is dried before further processing is conducted. Most advantageously, the dry powder layer is then covered with a thin metal foil which may be folded over or fastened to the sides of the steel shape to hold it in place. This procedure offers the advantage that powder coating can be accomplished at one station and the steel shape bearing the coating on a face can be stored, if desired, and moved to the press at the desired time. The nickel foil becomes an integral part of the final nickel coating and prevents the sticking which can readily occur when the powder coating is contacted with a hot platen during the pressing operation. The metal foil can have the same composition as the alloy to form the coating and, hence, may be nickel, nickel-copper, copper-nickel, stainless steel, etc. Pressure is applied in a

direction normal to the face of the steel product being coated at a level sufficiently high to prevent lateral shrinkage of the nickel powder layer. It is quite important that the pressure be applied to the nickel powder layer during heating thereof in order to prevent lateral shrinkage and cracking of the nickel powder layer. In this connection, it is to be observed that efforts to apply a nickel powder layer having any substantial thickness, e.g., $\frac{1}{64}$ inch or more, to the steel surface by sintering alone are uniformly unsuccessful due to lateral shrinkage and the accompanying cracking of the sintered nickel layer. A pressure of about 500 pounds per square inch (p.s.i.) up to about 30,000 p.s.i. is satisfactory for the foregoing purpose to provide a density of about 40% to about 70% in the nickel layer. Advantageously, a pressure of at least 4,000 p.s.i. is employed since densities of about 50% are then obtained after 30 seconds using a platen heated to 2000° F. in pressing fine carbonyl nickel powder. Simultaneous with the application of pressure to the layer of nickel powder, heat is applied by any convenient means. Most advantageously, heat and pressure are applied by means of an internally heated platen made of a heat resistant alloy mounted in the press die and having a vertical traverse. The temperature of the nickel powder layer and the temperature of the steel face to which it is applied is raised to at least about 750° F. during pressing. A temperature in the range of about 800° F. to about 2000° F. is very satisfactory since this temperature combined with the aforementioned pressure is sufficient to convert the nickel powder layer into a layer of metallic nickel having a density of about 40% to about 70% of theoretical. A nickel layer having the aforementioned density is bonded to the steel face sufficiently strongly to permit handling and there is no necessity for providing elaborate supplementary means for preventing access of air to the nickel layer and the copper-coated steel face upon which it is applied. For best results, the nickel layer is heated to a temperature of at least about 1200° F. so that at least some metallurgical bonding of the nickel layer to the steel is obtained.

It will be appreciated that, upon heating of the steel shape bearing the hot-pressed nickel coating for further rolling, which usually will be conducted at temperatures about 2000° F. to about 2400° F., further densification of the nickel coating and metallurgical bonding thereof to the steel results and the coated steel product may then be rolled in conventional steel mill equipment without encountering spalling, etc., in the nickel coating. Initial passes should be light until complete densification of the nickel layer is accomplished. The bond area should not be subjected to the high tensile stresses produced by heavy drafts until densification of the nickel is complete.

It is convenient to make the platen of a chromium-containing alloy containing, for example, about 8% to 30% chromium, up to 70% iron, and the balance nickel. These chromium-containing alloy platens may be heated in an oxidizing atmosphere such as air to a temperature in the range of about 1000° F. to 2000° F. to cause formation of an adherent layer of chromium oxide thereon. It is convenient to incorporate heating means such as resistance heaters directly in the platens.

It is highly important in carrying the invention into practice to employ a fine metal powder of high purity so that the nickel powder layer will have a recrystallization temperature below about 1000° F. A satisfactory nickel powder is carbonyl nickel powder having an average particle size of about 3 to about 10 microns. Chemically precipitated nickel powder having a particle size up to about 50 microns may also be employed. It is, of course, to be appreciated that other metals and alloys can be coated upon steel in accordance with the concepts of the present invention by employing suitable alloyed powders or mixed metal powders. Thus, nickel-copper alloys and copper-nickel alloys containing up to 60% copper, nickel-chromium alloys, nickel-chromium-iron alloys, and stain-

less steels can be coated upon steel. In general, the metals and alloys employed as coatings will have melting points exceeding about 2200° F., and will contain, by weight, up to about 60% copper, up to about 35% chromium, up to about 70% iron, and the balance essentially nickel, with the nickel content being at least about 7%. Dispersion-hardening agents such as alumina, thoria, etc., in finely divided form may be included in the initial powder mixture in amounts up to 5% or even 10%, by weight. Iron and chromium powders employed in the initial mixture should be nickel-coated to prevent oxidation.

In order to give those skilled in the art a better understanding of the invention and/or a better appreciation of the advantages of the invention, the following illustrative examples are given:

Example I

A steel slab about 48 inches by about 48 inches by about 4 inches thick is cleaned by sand blasting. After the sand blasting, a substantially flat surface of the steel slab is inspected and mechanical defects are removed by chipping. The steel slab is transferred to a copper plating tank and 1 mil of copper is deposited upon the cleaned flat surface. A press having a capacity of 3,000 tons is fitted with an upper flat platen made of an alloy containing about 15% chromium, 7% iron, and the balance essentially nickel and having dimensions of about 25 inches by about 50 inches on its lower face. The surface of the platen bears a retentive chromium oxide coating and the platen is fitted with internal electrical resistance elements to preheat the platen to about 2000° F. A metal dam is then placed about the upper, clean, copper-coated face of the slab and a layer of fine carbonyl nickel powder weighing about 0.025 p.s.i. is distributed upon the upper surface of the steel slab. A nickel foil about 0.0025 inch thick is then placed over the nickel layer. A pressure of about 2,400 tons is then applied upon the steel slab resulting in a unit pressure of about 4,000 p.s.i. over half the area of the nickel powder layer. Simultaneous with the application of the pressure, further heat is applied through the electrical resistance elements in the platen and the temperature of the nickel powder layers is raised throughout by contact with the platen to a temperature of at least about 800° F. This operation is accomplished in about three minutes. The pressure is then relieved, the upper platen is lifted, and the operation is repeated upon the unpressed remainder of the nickel powder layer. Upon completion of the pressing, it is found that the steel slab bears a nickel coating which is about 50% dense. The coating is adherent and is sufficiently strong to withstand handling. The coated steel slab is then transferred to a heating furnace, is raised to a temperature of about 2000° F. and is then rolled to sheet about 0.250 inch thick. The resulting steel sheet bears a dense, firmly and metallurgically bonded nickel coating about 5 mils thick.

Example II

Two steel plates 10 inches by 20 inches by $\frac{3}{4}$ inch thick were cleaned on a major face and copper in the thickness range of about 1 to about 5 mils was electroplated thereon. A layer of fine carbonyl nickel powder about $\frac{1}{4}$ inch thick was placed upon the upper copper-coated face of each plate and was covered with a nickel foil about 0.0025 inch thick. The edges of the foil were folded over to permit handling. A 23-inch square ingot of an alloy containing about 29.5% copper, about 2.8% aluminum, about 0.5% titanium, and the balance essentially nickel preheated to about 2100° F. was placed individually on the nickel-powder coated surface of each plate while the plate was held upon the anvil of a 3,000-ton press. After one minute, a pressure of about 13,000 p.s.i. (1,250 tons load) was applied and held for thirty seconds. The pressure was then released and the plates were air cooled. The density of the powder layer in each case was about 70% of theoretical and the nickel foil

5

was bonded thereto. The plates were heated to about 2100° F. for one hour and hot rolled in six passes to 0.125 inch thick sheet without reheating. Each sheet was sheared to 12 inches wide, annealed, pickled and cold rolled to 0.056 inch with no difficulties.

It will be appreciated that impact pressure equipment may be used for hot pressing by applying static pressure until the powder-steel interface approaches the die temperature and then using impact pressure, such as hammer blows, to achieve the desired pressures for hot pressing.

Furthermore, it will be appreciated that pressures required for conducting the coating operation in accordance with the invention are readily attained in conventional equipment such as forging presses which are actuated hydraulically or otherwise. As previously noted herein, hot pressing can be accomplished stepwise so that higher unit pressures are obtained.

It will also be appreciated that the initial thickness of the nickel layer applied to the steel slab, billet or bloom will be governed by the thickness of the nickel coating desired on the final rolled steel product having regard for the amount of reduction to be encountered during the subsequent rolling operations.

It is to be understood that the term "primary mill" used herein is employed in the sense described at page 681 of the standard handbook "The Making, Shaping and Treating of Steel," edited by Camp and Francis, Sixth Edition, 1951, published by the United States Steel Company. Furthermore, the term "rolled steel primary mill product" used herein is employed to describe the rolled steel product of such a mill. As pointed out in the said handbook, such products are usually described as "blooms," "billets" or "slabs."

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention, as those skilled in the art will readily understand. Such modifications and variations are considered to be within the purview and scope of the invention and the appended claims.

We claim:

1. The process for producing a metal-coated steel sheet which comprises cleaning a substantially flat face of a steel primary mill shape, copper coating said cleaned steel face, applying a layer of powder containing at least one metal having a melting point at least about 2200° F. upon said cleaned and copper-coated steel face, covering said

6

metal powder layer with a metal foil having a composition matching that of said metal, hot pressing said metal powder layer upon said steel face to achieve substantial densification of said powder layer and adherence of said powder layer to both said foil and said steel shape, thereafter heating said steel shape to a hot rolling temperature and hot rolling said metal coated steel shape to sheet having upon a face thereof a substantially completely densified metal coating metallurgically bonded to a steel base.

2. The method according to claim 1 wherein said metal and said foil are nickel.

3. The process according to claim 1 wherein said copper coating is about 0.2 to about 10 mils thick.

4. The process according to claim 2 wherein the nickel foil has a thickness of about 0.001 to about 0.006 inch.

5. The process according to claim 1 wherein hot pressing is conducted by means of a heated platen applied substantially normal to said nickel powder layer.

6. The process according to claim 2 wherein said hot pressing operation is conducted at a pressure of about 500 to about 30,000 pounds per square inch.

7. The process according to claim 6 wherein the hot pressing pressure is at least about 4,000 pounds per square inch.

8. The process according to claim 2 wherein the nickel layer is heated during hot pressing to a temperature of about 750° F. to about 2000° F.

9. The process according to claim 8 wherein the nickel layer is heated during hot pressing to a temperature of at least about 1200° F.

10. The process according to claim 8 wherein the pressure in the hot pressing operation is continued for about 30 seconds to about an hour.

11. The process according to claim 1 wherein heating to hot rolling temperature is conducted in an atmosphere nonoxidizing to said metal.

References Cited

UNITED STATES PATENTS

2,241,094	5/1941	Marvin.	
2,289,311	7/1942	Wellman	29—420.5
2,289,658	7/1942	Koehring.	
2,372,607	3/1945	Schwartzkopf	75—208
3,142,560	7/1964	Storchheim	29—420.5 X
3,316,625	5/1967	Flint et al.	29—420.5

JOHN F. CAMPBELL, *Primary Examiner.*

PAUL M. COHEN, *Assistant Examiner.*