United States Patent [15] 3,653,884
Davies et al. [45] Apr. 4, 1972

[54] PROCESS FOR THE CONTINUOUS PRODUCTION OF A STRIP FROM POWDERED METAL

[72] Inventors: **Idwal Davies**, Killay, Swansea; **Alan G. Harris**, Tycoch, Swansea, both of Wales

[73] Assignee: **The British Iron and Steel Research Association**, London, England

[22] Filed: **Mar. 13, 1969**

[21] Appl. No.: **807,101**

[30] **Foreign Application Priority Data**

Mar. 14, 1968 Great Britain 12,484/68

[52] **U.S. Cl.** 75/214
[51] **Int. Cl.** **B22f 1/00**
[58] **Field of Search** 75/200, 214

[56] **References Cited**

UNITED STATES PATENTS

3,115,698 12/1963 St. Pierre ....75/200 X
3,335,000 8/1967 Bliss ....75/214 X

Primary Examiner—Carl D. Quarforth
Assistant Examiner—R. L. Tate
Attorney—Sughrue, Rothwell, Mion, Zinn and Macpeak

[57] **ABSTRACT**

A process is provided for rolling metal strip, particularly iron or iron alloy strip, directly from powdered metal using a technique in which a self-supporting metal powder/binder strip is formed from a slurry containing the metal and binder composition, together with a hygroscopic substance on a support surface and the resultant strip is compacted by rolling and then sintered. Typical hygroscopic substances are polyhydroxy compounds such as glycerol and polyalkylene glycols and their use is found to improve the mechanical properties of the final strip.

7 Claims, No Drawings

A PROCESS FOR THE CONTINUOUS PRODUCTION OF A STRIP FROM POWDERED METAL

CROSS-REFERENCES TO RELATED APPLICATIONS

U.S. application Pat. No. 683,983 filed Nov. 17, 1967.

BACKGROUND OF THE INVENTION

This invention relates to a process for the production of metal strip from powdered metal which is particularly, but not exclusively, applicable to the production of steel strip.

In our co-pending U.S. application Pat. No. 683,983 filed Nov. 17, 1967, and in the article entitled "Thin Strip Steel from Powder," published in "Powder Metallurgy" 1968, Vol. 11, No. 22, there is described a process for the production of strip from powdered metal.

This process basically comprises the steps of forming a slurry comprising a suspension of a metal powder in a binder composition, depositing on a support surface, such as a metal band or drum, a coating of the slurry to form, after any necessary drying, a self-supporting metal powder/binder strip, removing the strip from the support surface and rolling it to effect compaction and thereafter sintering the compacted strip in a reducing atmosphere at a temperature below the melting point of the metal. Normally the strip obtained after sintering is subjected to a further rolling and then a further sintering and finally a planishing or temper rolling.

Water-based binder compositions are preferred for a variety of reasons including economy and absence of fire hazard and film-forming cellulose derivatives have been found to generally meet the requirements of suitable film-forming materials for formulation of the binder compositions.

As explained in our above-mentioned patent application, the metal powder/binder strip obtained by deposition and drying of the slurry should possess sufficient strength and flexibility to withstand mechanical handling before the first compaction step in which the metal particles are cold-welded and inter-locked. The preferred binders are film-forming, water-soluble cellulose ethers, particularly those containing methyl groups, e.g. methyl cellulose, since metal powder/binder strip of good strength and flexibility can be formed from binder compositions based on these cellulose ethers. In order that the process should be attractive commercially as an acceptable method of producing bulk steel strip, such as blackplate, rather than simply a process for the production of more expensive strip products such as stainless steel strip or very thin steel foil, it is necessary to increase the line speed at which the process is operated to the order of several hundreds of feet per minute. To achieve line speeds of this order, it is necessary to use slurries having viscosities of about 1,000–5,000 centipoises. Unfortunately, it is found that when the viscosity of the slurry is reduced significantly below 50,000 centipoises, the dried metal powder/binder strip produced tends to be brittle and difficulty is encountered in handling the dried strip.

SUMMARY OF THE INVENTION

We have now discovered that by incorporating hygroscopic substances in the slurry the process described above can be operated satisfactorily at greater line speeds using low viscosity slurries since the additive acts as a plasticiser resulting in more flexible metal powder/binder strip. Furthermore it is found that the properties of the final sintered strip show advantages over strip produced from slurries in which the additive is not present.

According to the present invention, there is provided a process for the production of strip from powdered metal which comprises depositing on a support surface a coating of a slurry comprising a suspension of powdered metal in a binder composition containing a hygroscopic substance, drying the coating on the support surface, rolling the dried coating to effect compaction and sintering the compacted coating at a temperature below the melting point of the metal.

DETAILED DESCRIPTION OF THE INVENTION

As mentioned above, the process is generally carried out by application of the slurry of powdered metal and binder to a moving belt or drum, for example by roller coating. After drying, the metal powder/binder strip is removed from the belt or drum, compacted by rolling and then subjected to a first sintering step. The hygroscopic substance is conveniently added to the binder composition prior to dispersing the powdered metal therein. For example, where an aqueous solution or dispersion of a cellulose derivative is used as the binder composition, the hygroscopic substance may be added to the water before or after the addition of the cellulose derivative. The powdered metal is then stirred into the resultant binder composition. The hygroscopic substance should, of course, be compatible with the binder.

Preferably the hygroscopic substance is a water-soluble polyhydroxy compound such as glycerol, low molecular weight polyalkylene glycols, e.g. trimethylene glycol, polyhydric alcohols, e.g. sorbitol, mannitol, and sugars, e.g. invert sugar, Inorganic hygroscopic material may also be used, e.g. calcium chloride. The hygroscopic substance is preferably present in an amount of up to 1 percent by weight of the suspension. It is found that if substantially larger proportions of hygroscopic substance are employed, too much water tends to be retained in the cellulose derivative during the slurry drying and this causes difficulties in the subsequent processing steps.

After preparing the suspension of the powdered metal in the binder composition containing the hygroscopic substance, a self-supporting strip is produced in the manner described in our above co-pending application and the subsequent steps of the process are carried out in the manner described in that application.

In order to demonstrate the effectiveness of the process of the present invention, the following comparative experiments were carried out. An aqueous methyl cellulose solution was formed by dissolving 0.6 parts by weight of methyl cellulose in 30 parts by weight of water. 0.1 parts by weight of the sodium salt of an aliphatic dicarboxylic acid and 0.1 parts by weight of a polyoxylated polyalkylene glycol sold under the Trade Mark "Supronic" were incorporated in the methyl cellulose solution, the former additive being a corrosion inhibitor and the latter a surface-active agent. A series of binder compositions were prepared by dissolving various quantities of glycerol in samples of the methyl cellulose solution. A variety of iron powders were then stirred into the binder compositions in amounts such that the resultant slurries contained approximately 70 percent by weight of iron and correspondingly 30 percent by weight of binder composition. A series of control slurries were also prepared containing no glycerol.

A number of test strips were then made by the following steps:

1. Casting the slurries onto a substrate and drying to form self-supporting strips.
2. Compacting the resultant strips by rolling in a 12-inch diameter rolling mill at a load of 10 tons/inch width to form "-green" strips.
3. Sintering the "green" strips for 30 seconds at a temperature of 1,150° C. in a hydrogen atmosphere.
4. Re-rolling the sintered strips so as to produce a 3 percent extension in length.
5. Finally sintering the strips again for 30 seconds at a temperature of 1,150° C. in atmosphere of hydrogen.

Tests were then carried out on the resultant strips to determine their mechanical properties. The results of these tests are given as follows:

Powder	Glycerol as weight percent of slurry	Strip gauge (ins.)	Strip mechanical properties	
			UTS tons/ sq. in.	Percent elongation (¾" g.l.)
Makin (300 P1 grade)...........	0	0.0045	12	4
	0.01	0.0066	18	12
	0.1	0.0059	18	15
	1.0	0.0051	17	13
Hoganas (MH300 grade)........	0	0.0043	10	6
	0.1	0.0044	16	11
Sintrex (electrolytic annealed).	0	0.0034	15	12
	0.1	0.0047	19	22

The Hoganas, Makin and Sintrex iron powders used all had a particle size of −300 mesh. Hoganas iron powder is made by the direct reduction of high grade magnetic ore. The powder particles are irregular in shape and compact well to give good "green" strength. The powder contains certain impurities especially silica, discrete particles of oxides and surface oxides and these impurities do tend to have a deleterious effect on the mechanical properties of strip made from Hoganas powder. Makin iron powder is made from molten high grade pig iron or scrap by water atomisation, followed by crushing and heat treatment. This gives an iron powder which is feathery and ragged in shape. The powder tends to be a little more pure than Hoganas powder. Sintrex powder is produced by the electrolytic deposition of iron. The powder is dendritic in shape and of high purity which gives excellent "green" strength and sintered properties. It is, however, rather more expensive than Hoganas or Makin powders.

All tests were carried out on ¾inch gauge length samples of strip, but it is to be expected that slightly better results would be obtained with larger test specimens.

As will be seen from the above results, the effect of the presence of the glycerol is to produce a significant improvement in the ultimate tensile strength of the finished strip and more important this is accompanied by a substantial improvement in the percentage elongation of the final strip, this demonstrating greatly improved ductility.

Glycerol and the other organic hygroscopic substances mentioned above are preferably used in the practice of the present invention because their use results in the development of improved mechanical properties in the final strip as demonstrated b the above tests. Apart from the plasticising effect of the hygroscopic substances which results in better compaction during the first compaction step, these improved mechanical properties are believed to be due to reduction during the sintering step of the oxide film present on the surface of the metal particles and it is thought that the glycerol or other hygroscopic organic substance plays some part in this reduction process.

Although the inclusion of glycerol or other hygroscopic substance in the slurry is of particular advantage when using low viscosity slurries, it is, of course, also advantageous to include such additives when using higher viscosity slurries because of their beneficial effect on the mechanical properties of the final strip.

It has been found that when drying solutions of cellulose ethers 2 to 6 percent of water (by weight of cellulose ether) must be retained in order that a flexible film is formed. If the water content is reduced below the critical moisture content of approximately 2 percent inferior film properties are obtained. Hygroscopic substances having an affinity for water inhibit the removal of the last few per cent of water, resulting in more flexible metal powder/binder strip.

An important feature of solutions of certain cellulose ethers (e.g. methyl cellulose, hydroxyethyl methyl cellulose, hydroxypropyl methyl cellulose) is their thermogelling property. When, for example, a slurry containing a solution of methyl cellulose is heated to above approximately 50° C., thermogelling occurs which prevents settling out of the metal powders. Thus slurry drying above the gel temperature results in more flexible dried metal powder/binder strip because of the homogenous distribution of the metal powder.

We claim:

1. A process for the continuous production of a strip from powdered metal, which comprises:
   a. depositing on a moving support surface, a coating of a slurry comprising a suspension of powdered metal in a binder composition containing a film-forming cellulose derivative as the binder, and a hygroscopic substance compatible with said binder, said hygroscopic substance selected from the group consisting of low molecular weight polyalkylene glycols, polyhydric alcohols, sugars, and calcium chloride,
   b. drying the coating on the support surface,
   c. removing the dried coating from the support surface,
   d. rolling the dried coating to effect compaction, and
   e. sintering the compacted coating at a temperature below the melting point of the metal.
2. The process of claim 1 in which the cellulose derivative is a cellulose ether containing methyl groups.
3. The process of claim 1 in which the polyhydric alcohol is glycerol.
4. The process of claim 1 in which the hygroscopic substance is present in the binder composition in an amount of up to 1 percent by weight of the slurry.
5. The process of claim 1 in which the slurry includes a nonionic surface-active agent.
6. A process according to claim 5 in which the non-ionic surface-active agent is a polyoxyalkylene glycol.
7. The process of claim 1, wherein the slurry further contains a salt of an aliphatic dicarboxylic acid, said salt serving as a corrosion inhibitor.

* * * * *