United States Patent Office 2,861,935
Patented Nov. 25, 1958



2,861,935

# ELECTROPHORETIC METHOD OF APPLYING A LUBRICANT COATING

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No Drawing. Application May 20, 1954
Serial No. 431,272

4 Claims. (Cl. 204—181)

Our invention relates to lubricated or low friction metal structures and methods for producing the same.

Certain inorganic compounds, i. e., the sulphides, disulphides, selenides and tellurides of such metals as molybdenum, tungsten, titanium and uranium are characterized by a laminated or plate-like crystal structure in which the metallic atoms are attached to each other in a single common plane while the non-metallic atoms are attached to the metallic atoms to form layers on both sides of this plane. The non-metallic atoms in each of the several crystals thus formed have very little attraction for each other so that these crystals will slip readily with response to each other under the action of low shearing forces. Moreover, the non-metallic atoms have an affinity for adjacent metal surfaces so that the crystals attached to such surfaces will exhibit very strong resistance to the action of forces normal to the direction of shear. Consequently, these compounds have excellent lubricating properties and also have excellent anti-seizing properties. The term "laminated metallic lubricating agents" has been selected to denote compounds of this class.

In our copending application S. N. 404,796, filed January 18, 1954, now Patent No. 2,830,017, dated April 8, 1958, we disclosed a process for depositing a solid lubricant coating on the surface of a base member. In this method a mixture of reducible metallic compounds and laminated metallic lubricating agents are electrophoretically codeposited on the surface of a base metallic member and the reducible compound is subsequently reduced to metal to form a metal matrix which is bonded to the base member and which in turn entraps and bonds the lubricating agent or agents to the structure to form a lubricant coating. The relative proportions of the reducible compounds and lubricating agents are such that the lubricant adheres uniformly to the structure until the structure is subjected to a metal-working operation. During this operation the coating, while supplying the desired amount of lubricating, is progressively worn away or stripped from the structure. The term "sacrificial layer" has been selected to denote this type of coating.

In our copending application S. N. 402,402, filed January 5, 1954, now Patent No. 2,828,254, dated March 25, 1958, we disclosed a process for depositing a low friction coating on the surface of a base member. This process is similar to that of S. N. 404,796 in that a mixture of reducible metal compounds and friction modifying agents having low co-efficients of friction are electrophoretically codeposited on the surface of a base metal member and the reducible compound is subsequently reduced to metal to bond the friction modifying agents to the base member. (These low friction modifying agents include as a subclass all laminated metallic lubricating agents and further include substances, for example, graphite, which have low friction properties and which are not included in this subclass.) In this process, however, the low friction coating thus formed is permanent and is not appreciably worn away or stripped from the structure during a metal working operation.



In order to form a sacrificial layer the percentage content by weight of reducible metal compound must fall within the range 70%–80%; in order to form a permanent coating this percentage content must fall within the range 80%–95%.

In our work on both of these processes, we have discovered that when electrophoretically deposited coatings composed of reducible metallic oxides and laminated metallic lubricating agents are formed on a metal base member, and when the subsequent reduction operation is carried out in a hydrogen atmosphere at high temperatures, a portion of the laminated metallic lubricating agent used is also reduced to metal and the lubricating or low friction properties of the resultant coating, while still evident, are reduced in effectiveness.

Accordingly, it is an object of the present invention to provide improved processes and coatings of the character indicated in which the lubricating properties are not impaired under the above conditions.

It is a further object to provide improved processes of the character indicated wherein the laminated metallic lubricating agent is rendered chemically inactive before the reducing operation is initiated.

Still a further object is to provide improved processes of the character indicated wherein particles of the laminated metallic lubricating agent are first coated with metal before the electrophoretic deposition operation is initiated.

In our invention the laminated metallic lubricating agent in comminuted form is first treated in such manner that each particle is completely coated with a thin layer of metal. These coated particles are then mixed with particles of a reducible metallic compound and this mixture is electrophoretically codeposited on the surface of a base metal member. The coated member is then heated in a reducing atmosphere until the metallic compound is reduced to metal and forms a metal matrix which is bonded to the base member and which in turn entraps and bonds the metal coated lubricating agent particles to the member to form a lubricant coating. The metal coating on the coated particles is so thin that it can be immediately worn away in use or can be readily removed by chemical action so that lubricating or low friction properties come into immediate effectiveness. In this manner the lubricating agent particles are rendered chemically inactive during the reduction operation and there is no impairment of the lubricating or low friction properties of the resultant coating.

The metal coating operation can be carried out by a number of well-known techniques, including, for example, ball milling, electroplating, precipitation and spraying.

Electrophoretic deposition occurs when an electrostatic field is established between two electrodes immersed within a colloidal or gross dispersion of charged particles, thus causing migration of the suspended particles toward one of the electrodes and producing a deposit of an adherent coating on that electrode. Exceptional uniformity of coating thickness and compactness (with a relatively high coating density) are obtained as compared with dipping, spraying, brush and other more conventional methods of application. Irregularly shaped objects can be coated with excellent uniformity and reproducibility of coating. A complete description of the electrophoretic process will be found in our copending application S. N. 386,882, filed October 19, 1953.

The following examples set forth certain well-defined instances of the application of this invention. They are, however, not to be considered as limitations thereof since many modifications can be made without departing from the spirit and scope of this invention.

*Example I*

A coating 1 to 10 mils thick containing 77% by weight of copper oxide and 22% by weight of molybdenum disulphide particles was electrophoretically deposited out of organic media onto a stainless steel sheet in the manner outlined in the above-mentioned copending application S. N. 404,796.

The sheet was then fired in an atmosphere of hydrogen at a sintering temperature for a maximum period of 30 seconds. Subsequent cross-sectional analysis established that the copper oxide had been reduced to copper in the form of a matrix bonded by codiffusion to the surface of the sheet. Molybdenum disulphide particles were found to be entrapped within the pores of the copper matrix. However, particles of metallic molybdenum were also found to be entrapped within the pores of the copper matrix. It was therefore concluded that some of the molybdenum disulphide particles had been reduced to metallic molybdenum during the reduction operation.

*Example II*

Comminuted molybdenum disulphide particles were each coated with a thin layer of metallic copper in accordance with a conventional coating process of the type outlined, for example, in circular No. 389 of the U. S. Bureau of Standards. These coated particles were then electrophoretically codeposited with copper(ous) oxide particles on the surface of a stainless steel plate and then heated in a hydrogen atmosphere under the same conditions as outlined in Example I. Subsequent cross-sectional analysis established that a copper matrix was formed bonded by codiffusion to the surface of the stainless steel plate. The copper coated molybdenum disulphide particles were found to be entrapped within the pores of this matrix and no metallic molybdenum was found to be present.

*Example III*

Comminuted particles of molybdenum disulphide were electrolytically coated with metallic silver. The silver coated particles were then electrophoretically codeposited with nickel oxide particles on the surface of a stainless steel plate in the general manner outlined in Example I. The coated plate was then fired in hydrogen to a temperature of 2000° F. for a period of 15 seconds. Subsequent cross-sectional analysis established the nickel oxide had been reduced to nickel in the form of a matrix bonded by codiffusion to the surface of the sheet. Silver coated molybdenum disulphide particles were found to be entrapped within the pores of the nickel matrix. No metallic molybdenum particles were found to be present.



The coated plate was substantially dipped in a bath of nitric acid to dissolve that portion of the silver coating which was exposed on the outermost surface of the plate.

As many apparent different embodiments of this invention may be made without departing from the spirit and scope thereof, and it is to be understood that we do not intend to limit ourselves to the specific embodiments shown, but are limited only to the invention as defined in the claims which follow.

We claim:

1. The method of applying a lubricant coating to the surface of a metal which comprises the steps of electrophoretically codepositing out of liquid media onto the surface of a metal a coating consisting essentially of a mixture of between 70%–95% by weight of comminuted particles of at least one reducible metallic oxide and between 30%–5% by weight of metal clad comminuted particles of at least one lubricating agent selected from the group consisting of the sulphides, disulphides, selenides and tellurides of molybdenum, tungsten, and titanium; and heating said coated metal in a reducing atmosphere until said oxide particles are reduced to metal in the form of a matrix bonded to said metal, said metal clad particles being entrapped within the pores of said matrix.

2. The method as set forth in claim 1 wherein said liquid media is an organic media.

3. The method as set forth in claim 1 wherein said reducing atmosphere is a hydrogen atmosphere.

4. The method as set forth in claim 1 including the further step of chemically removing the metal cladding from the surface exposed portions of the entrapped metal clad particles.

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# CERTIFICATE OF CORRECTION

Patent No. 2,861,935 November 25, 1958

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 1, for "substantially" read -- subsequently --.

Signed and sealed this 3rd day of March 1959.

(SEAL)
Attest:

KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON
Commissioner of Patents