GENERAL ENGINEERING LABORATORY

COATING BY THE FLUIDIZED BED TECHNIQUE

By

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COATING BY THE FLUIDIZED BED TECHNIQUE

ABSTRACT

A review of the patent literature on coatings via powdered solids is presented. In addition, the results of an experimental program, not yet complete, to apply polymer, glass, ceramic, and metal coatings to a variety of substrates, via a fluidized bed

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~~CONCLUSIONS~~

technique, where the article to be coated is not heated prior to exposure to the fluidized bed, are presented.

The state of the art including the work in General Electric is discussed and a plan for action suggested.

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COATING BY THE FLUIDIZED BED TECHNIQUE

Introduction

Recently, a number of departments within the General Electric Company have become interested in a fairly well publicized technique by which thick, dense, plastic coatings can be applied in one pass to a variety of shapes, including wire, by fluidizing a bed of fine polymer particles, heating the article to be coated, dipping it into the fluid bed to sinter the powder on, and then further heating to completely fuse the plastic material. Two American companies*, and one German concern ‡ claim rights to such a technique, although no American patents have been issued to either of them. However, an interesting American patent has been issued to a private investigator(20 and a German patent (5) has been issued to Gemmer upon which the above companies appear to base their claims.

The purpose of this report is to review the known prior art, present and discuss the preliminary experimental findings and development in the General Engineering Laboratory, and suggest a plan for action. This appears desireable in order to clarify somewhat the patent situation with regard to the necessity for licensing which is currently being investigated by some operating departments and suggest a centralized experimental program of benefit to those departments which have expressed interest in coating via fluidized solids.

State of the Art

In conducting a search of the patent literature, it appears that the idea of applying coatings with powders dates back to 1895 when Long (10) patented the idea of applying inorganic glazes to ceramic materials by blowing powdered glazing compounds into the kiln where the ceramic was being baked. Excellent coating was claimed even for complex shapes. Otto (13) in 1898 patented a method, together with apparatus, of enameling which constituted the application of enameling material by exposing a heated article to an atmosphere of finely divided enamel particles. Contact time was limited and only a limited number of particles were permitted to settle on the article to be coated. In 1915, Geer (4) patented a proofing process where the article to be coated was cold and the proofing compound in powder form was sprayed hot above the article. Deposition was again by gravity. Ivery (6) was granted a patent in 1917 on virtually the same process as that of Long with only minor modifications.

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* Polymer Processes Inc., Reading Pa. - American Agile Corp., Cleveland Ohio

‡ Knapsack-Griesheim Aktiengesellschaft

In 1920, Kebler (7) obtained a patent for the application of clear glass coatings to resistors in which the resistors were heated and rotated as powdered glass was sifted down on them. Davis, (1) in 1925, patented a method of sanding the interior of brick molds. Sand was sprayed against the interior surface of the hot mold and the surface was thereby coated. A process for the application of heat-plasticizable resins to various surfaces was patented by Merritt (11) in 1935. His method was one in which the article was cold and the resin, although sprayed cold, passed through a heated zone before reaching the surface to be coated. A method of impregnating paper with resinous material was patented by Lipsius (9) in 1944 where a thin coating of powder was sintered on the surface, then only the powder was heated as it was pressed into the paper. In 1954, a patent was granted to Simpson (16) for the application of powdered resin to sheet materials. Again the powder contacted the substrate by gravity.

In 1955, a German and a U.S. patent concerned with the application of powdered resin to form coatings, were issued. The German patent (5) claims:

"1. Method of preparation of protective coatings of thermoplastic plastics by dipping the object heated above the melting point of the plastic in powder form, characterized in that the plastic powder is loosened into a liquid-like floating state by a stream of gas rising through the powder.

2. Method as in Claim I characterized in that inert gas, e.g. nitrogen, is used to stir up the powder.

3. Device for using with the method described in claims I and 2 characterized by the use of a porous support for the plastic powder, for example a ceramic plate."

It is based on this patent that the Polymer Processes Co., Inc., has applied for a U.S. patent and is negotiating for licensing agreements. American Agile Corp., claims to have rights to this patent also.

It must be apparent that (1) gaseous fluidization in itself is a Chemical Engineering unit operation which can not be a claim in the United States, petroleum companies have been making extensive use of this technique for many years, (2) heating the object above the melting point of the plastic is subject to prior art as shown in the previous review, (3) the use of nitrogen or any other gas to fluidize a bed of solid particles is not novel, extensive references in the technical literature are available*, and (4) the use of a porous support for the plastic powder is not novel*. The literature

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* See references (8) and (12)

recommends the use of sintered glass, metal, and ceramic plates and discs as supports for powders to be fluidized (8), (12). Therefore, any claims now obtained in the field will have to depend for validity on whatever invention is involved in a combination of these known facts.

The American patent by Voris (20) claims both pulverization to reduce particle size and protective coating of resins, thermoplastic and thermosetting, in a single apparatus. Pulverization is made to occur by collisions of particles as they are propelled toward each other at high velocities in high pressure gaseous or liquid streams. These fine particles are then propelled upward, due to imparted energy, to the exposed surface of an object heated above the melting point of the resin. The powder sinters on the surface and this is then heated further to produce a continuous coating. In this case what appears novel is the application of a jet pulverizer to a coating technique. Blaw-Knox has jet pulverizers for sale and the inventor states that these can be used. The portion which claims the coating of a heated article with powder appears to be established prior art.

In addition to the foregoing patents other activities within the General Electric Company can be reported. In 1954, Root (14) in the General Engineering Laboratory, submitted a patent disclosure and later issued a report (15) describing a technique by which to coat laminates with powdered resin continuously. Heated laminates were passed through a chamber in which plastic powder was being fluidized by churning paddles. The plastic powder coated the lamination and no further treatment was necessary. A patent reviewing committee rated this disclosure not patentable in view of the existing prior art.

In 1955, Gaynor (2) submitted a patent disclosure in which coating a heated article in a fluidized bed of particles and subsequent heating for the purpose of complete fusion was claimed. Coating materials cited were plastic, glass, ceramic and metal and articles to be coated were metal, glass, ceramic and plastic. In addition, cross-linking to produce thermosetting plastics by the inclusion of catalyst powder in the fluid bed was also claimed. A variety of gaseous fluidizing media are also mentioned. A subsequent disclosure (3) cites deposition in a fluidized bed on cold articles with subsequent heating to fuse the coating. The means by which deposition is made to occur are twofold. First, it has been shown that particles in the course of fluidization acquire an electrostatic charge (dielectrics carry electrostatic charges even without fluidization(17)), and that there is an electrostatic component of adhesion (19), (20). Therefore, it was conceivable that such charges would be sufficient to cause the particles to adhere to the article to be coated. (This has been shown, as is later discussed.) Second, if these naturally

acquired charges were not sufficient to result in adhesion of particles an electrical potential difference of appropriate sign between an electrode and the article to be coated was claimed as a method of enhancing deposition. Since the claim of applying powdered coatings via natural electrostatic charges and/or electrical fields in a fluidized bed has not been uncovered in either the patent or technical literature, this invention is being considered by the General Engineering Laboratory for patentability.

EXPERIMENTAL WORK

A cursory advanced development project with limited funds was established by this laboratory to investigate the process for coating in a fluidized bed. In order to obtain maximum information in a short time with limited funds, a batch coating apparatus was set-up as shown in Fig. 1.

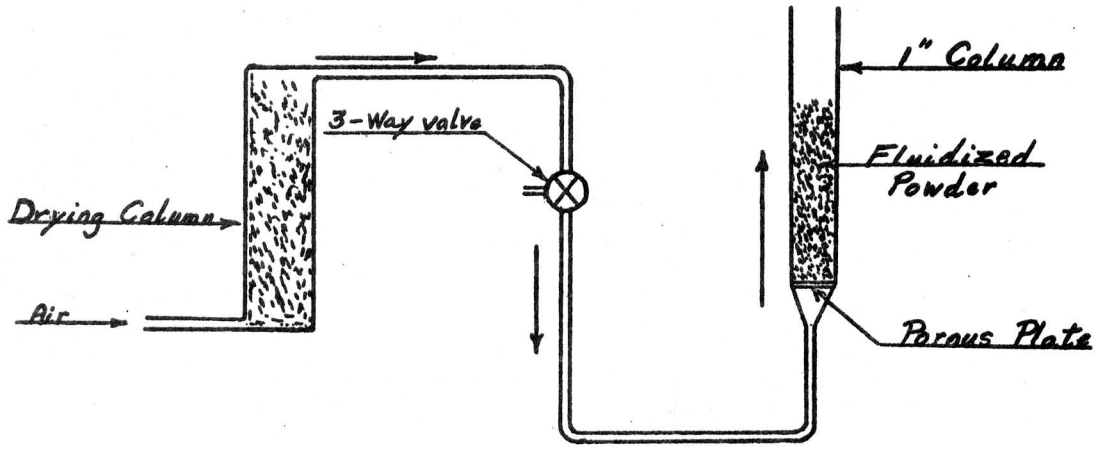

Figure 1

Polymer powders varying in particle size from 50 - 300 microns were fluidized in dry air and copper wire samples (50 mil) were dipped into the bed. In every case the powder clung to the wire without the aid of either heat or an electrical field. The wire with the powdered particles on it was then removed from the bed and fused in an oven. It was noted that even the sharp edges where the wire had been cut were coated by the powder. The polymer powders investigated were polyethylene, polystyrene, polymethylmethacrylate, Kel-F, polyvinyl chloride, polyvinylidene chloride, cellulose acetate, cellulose acetate butyrate, ethyl cellulose, polyvinyl alcohol, phenol-formaldehyde, and polyester resin. Other materials successfully applied to metal substrates are copper powder, sodium tetraborate, ceramic frits and powdered glass.

RESULTS AND DISCUSSION

The following presentation is based on a limited amount of experimental work. Therefore, results are limited and in most cases qualitative. However, the discussion of a number of phenomena which

occur repeatedly appears in order.

Adhesion of Powder

The adhesion of the powder to the substrate before heating appears to be adequate. A sharp rap will cause most of it to fall off the wire, however, the coated specimen can be removed from the fluidized bed where there are medium velocity air currents, and carried a reasonable distance (approx. 5 ft.) to an oven without losing any of the powder. It is estimated that the adhesion in this case is at least as good as that found in electrophoretic coating.

Particle Size

It appears that the smaller the particle the better the coating and the stronger the adhesion. However, particles from 50 - 300 microns have been applied successfully. For good fluidization, particle size much below 50 microns and above 500 microns are not practical. Coating thickness is also affected by the size of the particles, the smaller the particle sizes the thicker the coating.

Fluidized Bed Density, Air Velocity and Humidity

It has been observed that thicker coatings are obtained in the denser portions of the fluidized bed for identical immersion times which are very short (fraction of a second). Therefore, coating thickness is subject to control in this way as well as via particle size.

The air velocity controls the bed density and up to a limiting velocity affects the electrostatic charge on the powder.

Humidity affects both the electrostatic charge and the character of the coating. In both cases, it is detrimental in that it reduces the charges and is adsorbed by the powder making the fused coating less smooth, adherent, and coherent.

Fusion Temperature and Atmosphere for Fusion

It was apparent that for some of the materials fusion was too slow and that as a result oxidation accompanied the fusion. Therefore, some temperature above the melting point is desirable which will melt the powder in a short time such that oxidation will be limited. This temperature will vary depending on the material.

The atmosphere present in the oven obviously affects both the coating and the substrate. In order to limit or preclude oxidation it is apparent that either an inert or partially reducing atmosphere is required.

Present Status

Since Polymer Processes Co., Inc., has not obtained a U.S. patent as yet, it can not be determined at the present time whether or not such a patent will cover the technique evolved at the General Engineering Laboratory. The licensing negotiations are temporarily at a standstill, with the next step up to Polymer Processes Co., Inc. The experimental program in this laboratory is proceeding apace. The work is aimed at investigating many of the more fundamental aspects of the "electrostatic" coating process such as (1) the effect of particle size, fluid bed density, fluid bed height, air velocity, etc., on powder coating thickness and the ease of coating, (2) optimum fusion temperature, (3) most desirable atmosphere for fusion, and (4) ratio of powder coating thickness to fused coating thickness later to be correlated with particle size used in coating. In addition the application of (1) a mixture of crosslinkable resin (in the thermoplastic state) and a material which catalyzes the cross-linking reaction during fusion, (2) a mixture of polymer and inert filler and/or pigment, (3) glass, ceramics, and metals, and (4) a mixture of the constituents of metallic alloy, to a variety of substrates is under investigation. The adaptation of the technique so that it may be used as the major operation in a continuous process is also being studied. An example of such a process might be a continuous process for coating wire. The study outlined above is limited by the availability of funds.

It appears that the general technique of applying coatings via a mixture of powder and gas is not only advantageous but has broad applications within the Company. However, it has not been definitely shown that the use of a fluid bed is technically and economically more advantageous for all applications than a dense mixture of powders and gas which is not a fluidized bed. This latter technique has been described and is therefore prior art. It should be noted that the investigation currently being conducted at this laboratory is so directed as to yield data which would be applicable to any coating technique employing powder and gas. It appears then that more information is requisite before any judgement is possible. Furthermore, even if it is decided that a license should be purchased from Polymer Processes, there still remains a great deal of technical data to uncover.

Suggested Future Plan

In view of the foregoing statements, the broad company interest, the fact that this technique can be used to coat everything from the very largest to the very smallest objects regardless of shape, and the fact that it is not the function of the General Engineering

Laboratory alone to financially support a very extensive program, it appears that it would be of maximum value to all interested departments to support one rather large pooled development project, at a central site, fundamental in nature and broad in scope. The ultimate aim of this project being the development of continuous processes for coating many substrates such as sheets, gears, housings, flash bulbs, laminates, coils, wire, etc., with a variety of powders. The advantages of such a program over individually supported programs are many-fold and apparent. In order to submit a proposal, however, the breadth and degree of interest must be ascertained first. Therefore, the detachable letter on the following page is included. The nature of the response will determine the scope and the cost of the proposal, which will be submitted shortly after the letters are returned.

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Date: _____

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The _____ Department is _____
(very, mildly, not)

interested in evaluating a proposal for a pooled development project to investigate the coating of _____
(wire, sheet, gears, coils, etc.) with _____ (Specify if possible), by the powder-gas coating technique.
(polymers, glass, ceramics, metals)

All comments, questions, and suggestions will be sincerely appreciated.

_____

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