

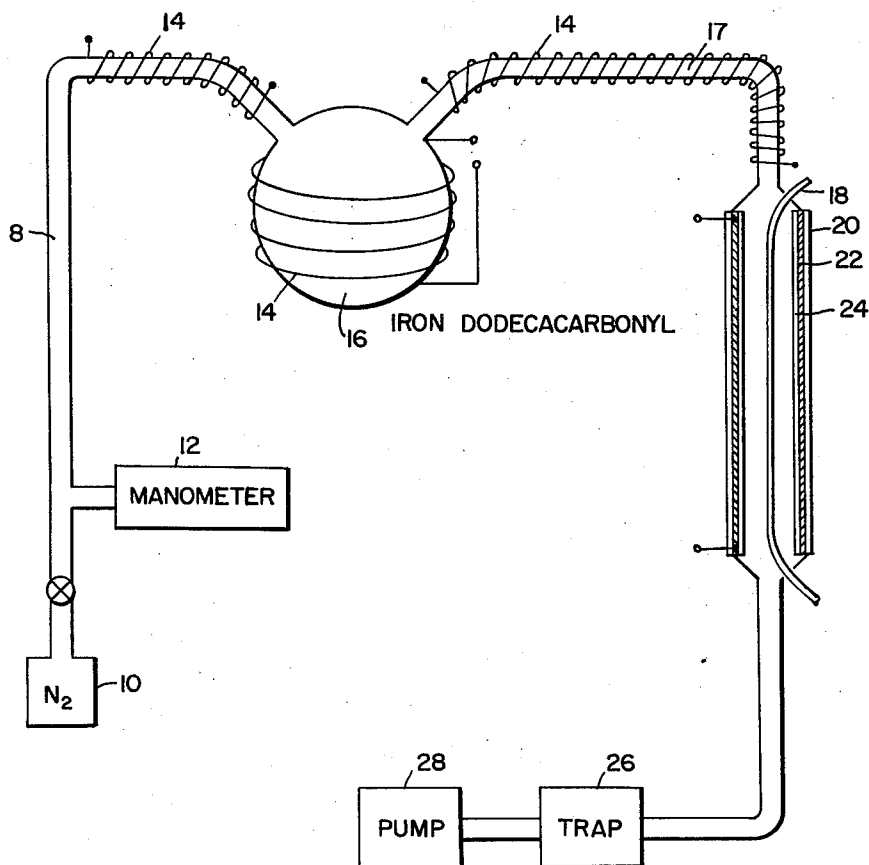
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PAPER METALLIZING PROCESS UTILIZING IRON DODECACARBONYL

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PAPER METALLIZING PROCESS UTILIZING
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1 Claim. (Cl. 117—107.2)

The present invention is concerned with metal coating processes and more particularly with vapor coating processes for depositing iron on paper cardboard, etc.

One object of the present invention is to provide vapor-coating processes for depositing iron on paper which can be carried out at temperatures below the charring point of said paper and which deposit the iron in a form having improved resistance to oxidation.

Other objects of the present invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the process involving the several steps and relation and order of one or more of such steps with respect to each of the others which are exemplified in the following detailed disclosure, and the scope of the application of which will be indicated in the claim.

For a fuller understanding of the nature and objects of the present invention, reference should be had to the following detailed description, taken in connection with the accompanying drawing wherein:

FIGURE 1 illustrates an apparatus for carrying out the steps of the present invention.

In general, the processes of this invention comprise vaporizing iron dodecacarbonyl, $\text{Fe}_3(\text{CO})_{12}$, at a temperature below the decomposition temperature of said iron dodecacarbonyl and bringing the vapors in contact with the paper, which is to be coated, while said paper is held at a temperature which is at or above the decomposition temperature of said iron dodecacarbonyl. At the decomposition temperature, the iron dodecacarbonyl breaks down to metallic iron and carbon monoxide.

In an especially useful embodiment, the process is carried out in a closed system comprising a zone for vaporizing the iron dodecacarbonyl and a zone for coating the paper. In a preferred embodiment, the vaporization of the iron dodecacarbonyl is carried out at reduced pressures. At pressures such, for example, as 10 to 50 mm., of mercury the iron dodecacarbonyl can be readily vaporized at 80 to 90° C. In a further preferred embodiment, the process is carried out in the presence of an inert gas such, for example, as nitrogen. Such inert gas serves to prevent the iron dodecacarbonyl vapors from reacting with oxygen.

In order that the iron dodecacarbonyl may be decomposed to metallic iron, the paper, to be coated, should be heated to the decomposition temperature of said iron dodecacarbonyl. At normal atmospheric pressure iron dodecacarbonyl decomposes at 140° C. At reduced pressures, as employed in the preferred embodiment of this invention, the decomposition point may be slightly lower. It has been found that, at pressures of 10 to 50 mm., of mercury, the iron will be readily deposited on the paper when it is heated to a temperature of about 135 to 140° C.

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Referring now to FIGURE 1, there is shown a complete system for carrying out the processes within the scope of this invention. At one end of the system, an inert gas such, for example, as nitrogen is introduced into the system from a source 10 such, for example, as a gas cylinder. The nitrogen flows through a first conduit 8 into retort 16 which contains a supply of iron dodecacarbonyl. The first conduit 8, the retort 16 and the second conduit 17, which leads out of said retort 16, are provided with heating elements 14. The heating element around the first conduit serves to elevate the temperature of the inert gas it enters the retort 16. The heating element 14 around the retort serves to vaporize the iron dodecacarbonyl and the heating element 14 around the second conduit serves to prevent the vapors from condensing in the second conduit 17. The second conduit 17 leads into plating chamber 18. Said chamber 18 is provided with a heating element 20 which is used to elevate the temperature of the paper 22 to at least the decomposition temperature of said iron dodecacarbonyl. When the paper is so heated, the vapors decompose and leave a layer of iron 24 on said paper. Pressures within the system are controlled by vacuum pump 28 which is connected to the system through trap 26.

Example

Iron dodecacarbonyl was heated to a temperature of about 80 to 90° C., in a system similar to that set forth above at a pressure of about 10 to 50 mm. The vapors were carried by means of dry nitrogen gas at a flow rate of 25 cc./per minute (0.4 cu. ft./per hour) to the plating chamber. The paper was placed in the chamber and heated from the reverse side to a temperature of about 140° C. A lustrous and ductile film of metallic iron was deposited on the paper.

The iron dodecacarbonyl for use in the processes of this invention may be prepared from iron pentacarbonyl, $\text{Fe}(\text{CO})_5$, through dehydrogenation by using active oxidizing agents such, for example, as manganese dioxide. The following procedure will illustrate the preparation:

42 ml. of iron pentacarbonyl in 180 ml. of methanol were added to a nitrogen flushed 2 liter flask, equipped with a nitrogen inlet, a reflux condenser and a stirrer. A cooled solution, comprising 45 gm. of sodium hydroxide dissolved in 100 ml. of water, was added with stirring over a 20 minute period. Afterwards, 125 ml. of a saturated ammonium chloride solution were added. Then, a thick brown aqueous paste of manganese dioxide was added to the resulting mixture. This paste had been prepared by heating over a steam bath 67 gm. of potassium permanganate in a solution comprising 300 ml. of water and 100 ml. of 95% ethanol until a vigorous reaction started. Upon adding the paste, a mild exothermic reaction occurred. After the reaction had proceeded for about an hour, the excess manganese dioxide was decomposed by first adding a solution comprising 40 gm. of ferrous sulfate, $(\text{FeSO}_4 \cdot 7\text{H}_2\text{O})$, dissolved in 250 ml. of dilute sulfuric acid and then adding over about a half hour period a cooled solution comprising 150 ml. of concentrated sulfuric acid and 150 ml. of water. The mixture was further stirred for an extra half hour using ice cooling. The black crystals of iron dodecacarbonyl were filtered and washed with (a) two 150 ml. portions of hot dilute sulfuric acid, (b) two 150 ml. portions of water

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(c) two 100 ml. portions of 95% ethanol and (d) two 100 ml. portions of pentane. The washed yield was sucked dry for about 10 minutes; care was used to prevent excessive drying. Such excessive drying causes the iron dodecacarbonyl to be less resistant to oxidation.

Since certain changes may be made in the above process without departing from the scope of the invention herein involved, it is intended that all matter contained in the above description or shown in the accompanying drawing shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

A process for metallizing paper with iron, said process comprising heating iron dodecacarbonyl to a temperature of approximately 80 to 90° C. and generating an iron dodecacarbonyl vapor at a pressure of approximately 10 to 50 mm. Hg, intermingling said iron dodecacarbonyl vapor with a flow of dry inert gas to provide a gaseous mixture, directing said gaseous mixture to one face of said paper, heating the other face of said paper to a temperature of approximately 135 to 140° C. in order to cause iron dodecacarbonyl to break down to metallic

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iron and carbon monoxide, and inhibiting oxidation of said carbon monoxide by the presence of said inert gas.

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