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PROCESS FOR ORIENTING FERROMAGNETIC FLAKES IN PAINT FILMS

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FIG. 1.

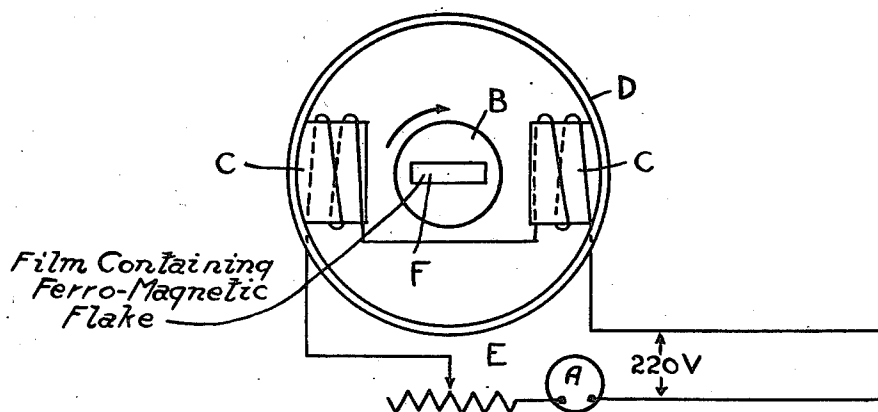


FIG. 2.

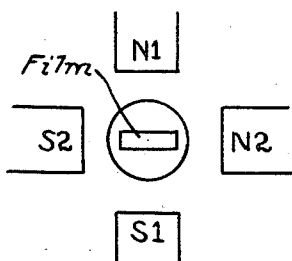


FIG. 3.

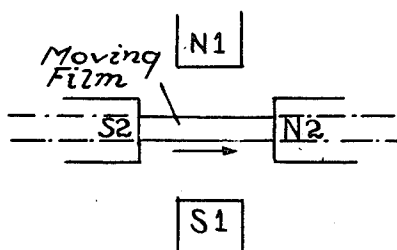


FIG. 4.

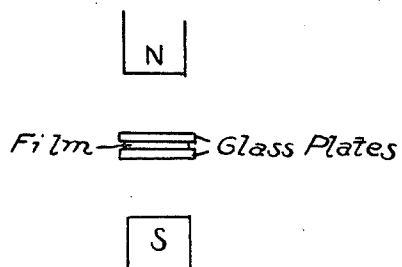
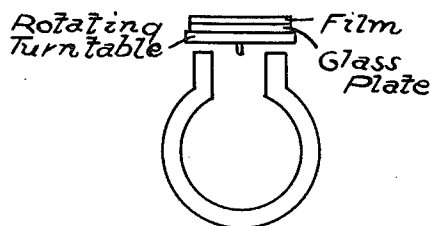


FIG. 5.



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## UNITED STATES PATENT OFFICE

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## PROCESS FOR ORIENTING FERROMAGNETIC FLAKES IN PAINT FILMS

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7 Claims. (Cl. 117-64)

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This invention relates to films containing metallic flake pigments. More particularly, it relates to a process for orienting ferromagnetic flakes in films containing the same.

Metal flake paints give coatings of high brightness only when the metal flakes are oriented, by which is meant that a large proportion of the flakes all lie in the same direction with respect to the plane of the film rather than in all directions at random. In general, brightness of the films is proportional to the degree of orientation (also called "leafing") of the metallic flakes. Brightness is a very desirable quality in a metallic paint film since it is often essential to produce artistic effects. In addition, bright coatings are useful because of certain physical properties such as that of reducing heat reflectance from hot surfaces, e. g., radiators. Heretofore, bright metal paint films have been made by subjecting the film to mechanical operations such as brushing or knifing. Such mechanical treatments are sometimes undesirable or impractical since they affect not only the film itself, but also its support, e. g., fabrics, paper, etc. and in general, they do not give complete orientation of the flakes. Moreover, the standard methods such as brushing, spraying, etc., are incapable of giving orientation of the flakes in a plane perpendicular to the plane of the film, a result which is obtainable according to one embodiment of this invention.

An object of this invention is to provide a new process for orienting metallic flakes in paint films. A further object is to provide an orienting or leafing process involving no mechanical treatment of the paint film. Another object is to provide metallic paint films having a high degree of orientation and brightness. Other objects will appear hereinafter.

These objects are accomplished by a method which comprises subjecting a wet paint film containing ferromagnetic flake to the action of a magnetic field, the film being positioned in the plane of the field and the directional angle between the film and the field being made to vary from the parallel to the perpendicular at short intervals of time, until the film has dried sufficiently to permit removal from the field without causing deorientation of the flakes.

In the drawing,

Fig. 1 is a diagrammatic view shown in plan of a form of apparatus suitable for carrying out the invention.

Figs. 2 and 3 are diagrammatic views in plan illustrating modified arrangements of magnets

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and film which can be used in the practice of the invention, and

Figs. 4 and 5 are diagrammatic views in elevation showing other modifications of the invention.

In the embodiment shown in Fig. 1 the change in the directional angle between the film F containing the flake ferromagnetic particles and the magnetic field is produced by rotating the film in the plane of the field. The apparatus consists of two electromagnets C mounted on opposite sides of the interior of an iron ring D. The magnets are actuated by 220 volt direct current. A rheostat E is inserted in the line to control the current which is read on ammeter A. A turntable B is mounted between the two magnets in such a way that a film placed thereon will be approximately in the center of the field. The turn-table is ordinarily run at a speed of about 20 to 60 R. P. M. by mechanism not shown which conveniently consists of a motor and suitable gears.

In another embodiment of the invention, the directional changes between the film and the magnetic field are produced by rotating the field instead of the film. An apparatus suitable for use in this modification consists, as shown in Fig. 2, of two sets of magnets, N-1, S-1 and N-2, S-2, normal to each other, which are turned on and off alternately for brief periods of time, for example, one-tenth to one-half second. The film in this embodiment is placed in a stationary position on a support in the plane of the magnetic field, whose direction is changed by an angle of 90° every time one set of magnets is turned on and the other is turned off. Alternatively, the magnets of one of the sets are hollow and the film is passed continuously at a predetermined rate through the center of these hollow magnets, thus providing a continuous operation, as shown in Fig. 3.

The preferred mode of operation, according to the first embodiment described above, is to prepare a metallic flake paint in the usual way, make a film therefrom by any method such as by knife coating, spinning, brushing, or spraying and then transfer the wet paint immediately to the turn-table. The turn-table is then rotated at about 30 R. P. M., and the current is passed through the coils so as to produce a field strength sufficient to effect orientation but insufficient to cause the paint to migrate toward the pole pieces of the magnet. The operation is continued until the paint has become dry enough to permit removal from the field without causing deorien-

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tation of the flakes. The paint may be further air-dried or baked in order to put it into condition for practical use.

The invention is illustrated by the following examples in which parts are by weight.

#### Example I

A paint is prepared from a composition comprising 80 parts of a 15% solution of ethyl cellulose in an 80/20 mixture of xylene and denatured alcohol, 90 parts of "Permalloy" flake (a magnetic alloy of iron and nickel), 18 parts of di-(beta-butoxyethyl)sebacate, 195 parts of xylene and 40 parts of denatured alcohol. The paint is formulated by mixing all the "Permalloy" flake with sufficient ethyl cellulose solution, di-(beta-butoxyethyl)sebacate and xylene to make a viscous paste. The paste is stirred for 5 minutes to produce a uniform dispersion, and the rest of the ingredients are added in small portions with agitation. The resulting paint has a pigment/binder ratio of 75/25 and an ethyl cellulose/di-(beta-butoxyethyl)sebacate ratio of 40/60. The paint has a viscosity suitable for film preparation by spinning. After aging overnight to remove air bubbles introduced during the mixing operation, a film is prepared by pouring the paint composition onto a 5" cardboard disc rotating at 150 R. P. M. The cardboard disc supporting the wet film is placed immediately on the turn-table B of the figure, which is rotated at about 40 R. P. M., and the full 220 volt current is applied, producing a magnetic field of about 100 gauss field strength. A pronounced brightening of the film is noticeable immediately upon application of the magnetic field. After one hour's rotation in the plane of the field, the film is moderately hard, flexible and very much brighter than an identical film prepared similarly in the absence of magnetic field.

A steel flake paint formulated in the same vehicle as that of the above example is oriented in the same manner to give a film of excellent brightness. In the case of steel flake, the field strength necessary to produce orientation is lower than with "Permalloy" flake.

It is not necessary for the successful operation of the process that the entire pigment consist of ferromagnetic material. As shown by the following example, it is possible to use a flake pigment comprising a large proportion of nonmagnetic material having coated thereon sufficient ferromagnetic material to make the entire pigment responsive to the orienting action of a magnetic field.

#### Example II

A mixture of 50 parts of magnetite (magnetic iron oxide) and 40 parts of a hydrocarbon plasticizer marketed by the Wilmington Chemical Company under the trade name "Naftolen R-100" and consisting of aromatic and cycloaliphatic hydrocarbons is milled on a 3 roll mill in sufficient passes to produce a well dispersed paint. Six parts of the resulting paint is added with stirring to a paste of 30 parts of fine aluminum powder (flake) in 35 parts of carbon tetrachloride. After the composition is well mixed, 27 parts of a 15% solution of polystyrene in xylene, 4 parts of the same hydrocarbon plasticizer, and 25 parts of carbon tetrachloride are added, and the mixture is stirred until homogeneous. The resulting paint, in which the magnetite/aluminum powder ratio is 10/90, the pigment/binder ratio is 75/25 and the polystyrene/plasticizer

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ratio is 40/60, has a viscosity suitable for knife coating. A 20 mil coat of the paint is knifed on Cellophane attached by rubber cement to a 5" Bakelite disc. The disc is placed on the turntable, which is rotated at 30 R. P. M., and the magnetic field is applied at full strength of about 100 gauss. The film is rotated in the magnetic field for one hour, after which it is almost dry. The film is allowed to air-dry overnight, after which the Cellophane is stripped from the Bakelite, soaked in water for one hour, and finally stripped from the pigmented film. The resulting detached film is pliable, stronger and brighter than a control film prepared outside the magnetic field.

It is not essential that the nonmagnetic pigment be coated with the magnetic pigment. As shown in the following example, subjecting a film containing an intimate mixture of both pigments to the action of a magnetic field causes satisfactory leafing of the paint because of the mechanical arranging action of the magnetic particles on the nonmagnetic particles.

#### Example III

A mixture of 11 parts of "Permalloy" flake, 33 parts of fine aluminum powder and 20 parts of xylene is ground in a mortar for 10 minutes. Thirty parts of xylene and 60 parts of a 25% solution of polystyrene in xylene are added, and the paint is stirred until homogeneous. The resulting paint, which has a pigment/binder ratio of 75/25 and a "Permalloy"/aluminum powder ratio of 25/75, has suitable viscosity for film preparation by spinning. The paint is aged overnight to remove air bubbles introduced during mixing, after which a film is prepared by pouring a portion of the paint on a 5" paper disc rotating at 250 R. P. M. The paper disc is transferred immediately to the turn-table, after which a magnetic field of about 100 gauss is applied. After 60 minutes' rotation at 30 R. P. M., a second coat is applied and the procedure repeated. The film is air-dried 6 hours and baked at 65° C. for two days. The resulting film is brighter than a control film prepared outside the magnetic field.

Orientation of the flakes in a direction perpendicular, rather than parallel, to the plane of the film may be accomplished by a variation of the already described procedure, as shown in the following example.

#### Example IV

A paint is prepared by stirring two parts of "Permalloy" flake in 40 parts of a 15% solution of polyisobutylene (Vistanex B-60) in xylene. A film is spread on a glass plate and allowed to dry on the rotating turn-table in a magnetic field, as in Example I, for one hour, producing a very bright film. The film is then covered with another glass plate, the "sandwich" heated with a heat lamp until the film is soft, then placed immediately between the poles of a magnet perpendicular to the film, as shown in Fig. 4, which is an elevation looking from the side. When the magnetic field is applied, the bright film turns immediately to a dead, nonreflecting black, the flakes being now oriented in a direction perpendicular to the plane of the film.

If the above process is carried out without the glass cover or with an open, wet film, the paint is pulled toward the magnet, producing a very rough, grey film. It is, however, possible to use a wet, uncovered film, by rotating it on a rigid

non-ferromagnetic support (for example, glass) above the poles of a magnet, as shown in Fig. 5, which is an elevation looking from the side. In this case, a dull black film is obtained, as in Example IV.

In the process of this invention, any ferromagnetic flake pigment can be used, such as iron, iron alloys, steel, alloy steels, magnetic iron oxide, as well as pigments comprising ferromagnetic pigment in intimate contact with nonferromagnetic flake pigment such as aluminum copper, bronze, silver, gold, etc. In the latter case, there should be enough ferromagnetic pigment present to render the pigment mixture responsive to the action of a magnet of practical field strength. For practical purposes, this means the total pigment should contain at least about 5% by weight of ferromagnetic pigment.

The method is applicable to compositions comprising any desired film vehicle, such as drying oils, varnishes, the various alkyd resins, polyvinyl chloride interpolymers, polyisobutylene, polyethylene, nitrocellulose, amide-formaldehyde resins, polyvinyl alcohol, etc. The vehicle may contain other ingredients such as plasticizers, fillers, dispersing agents, nonmetallic pigments, dyes, etc. For practical purposes, it is desirable that the paint have a viscosity of at least 0.3 poise at 25° C.

The operating conditions, i. e., the strength of the magnetic field and the speed of rotation of the film or of the field, may be suitably varied, depending, among other things, on the amount and kind of ferromagnetic pigment in the total pigment and on the viscosity of the vehicle. Some effect is obtained with a field strength as low as 5 gauss. In general, a field strength of 100 to 500 gauss is sufficient to orient the flakes even in vehicles of rather high viscosity. The field strength should not exceed that at which the entire paint composition begins to be attracted toward the magnet, although the latter effect, if it takes place, may be offset by applying the magnetic field intermittently. In general, it may be said that, the less magnetic pigment there is in the total pigment, or the more viscous the vehicle is, the higher the field strength should be.

While directional changes of the film with respect to the field, or vice-versa, are essential to obtain orientation parallel to the plane of the film, the speed of this motion is not very critical. When the film is rotated, any speed within about 10 and 250 R. P. M. is suitable, the range between 20 and 60 R. P. M. being, in general, optimum. When the field is rotated by means of two sets of magnets normal to each other and operating alternately, intervals of between  $\frac{1}{15}$  and 1 second are suitable, preferably between  $\frac{1}{10}$  and  $\frac{1}{4}$  second.

The invention is applicable to the treatment of film on any desired flat support such as paper, cardboard, regenerated cellulose, cloth, earthenware, wood, fabrics of any kind, sheet metal, etc. Objects of any shape such as rods, tubes, etc., may be subjected to orientation to obtain novel effects. A ferromagnetic metal support can be used when perpendicular orientation is desired.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that we do not limit ourselves to the specific embodiments thereof except as defined in the appended claims.

We claim:

1. A process for preparing a metal-pigmented film which comprises applying a wet paint film containing metal particles comprising ferromagnetic metal in flake form to a non-ferromagnetic base and subjecting said wet paint film to the action of a magnetic field with the film positioned in the plane of said field, and at short intervals of time varying the directional angle between an axis in the plane of the film and the said magnetic field through at least 90° while keeping the film in the plane of the field until the film has dried sufficiently to permit removal of the film from said field without causing deorientation of said metal flake particles.

2. A process for preparing a metal-pigmented film which comprises applying a wet paint film containing metal particles comprising ferromagnetic metal in flake form to a non-ferromagnetic base and subjecting said wet paint film to the action of a magnetic field with the film positioned in the plane of said field, and rotating said film in the plane thereof and in the plane of said field until the film has dried sufficiently to permit removal of the film from said field without causing deorientation of said metal flake particles.

3. The process set forth in claim 1, in which said particles of flake form are a mixture of ferromagnetic flake pigment and nonmagnetic flake pigment, and in which the ferromagnetic pigment is present in amount of at least 5% by weight of said mixture.

4. The process set forth in claim 2, in which said particles of flake form are a mixture of ferromagnetic flake pigment and nonmagnetic flake pigment, and in which the ferromagnetic pigment is present in amount of at least 5% by weight of said mixture.

5. The process set forth in claim 1, in which said particles of flake form comprise particles of ferromagnetic flake pigment adherent to particles of nonmagnetic flake pigment, and in which at least 5% by weight of the total weight of the ferromagnetic flake pigment and nonmagnetic flake pigment consists of ferromagnetic flake pigment.

6. The process set forth in claim 2, in which said particles of flake form comprise particles of ferromagnetic flake pigment adherent to particles of nonmagnetic flake pigment, and in which at least 5% by weight of the total weight of the ferromagnetic flake pigment and nonmagnetic flake pigment consists of ferromagnetic flake pigment.

7. In a process for preparing a metal-pigmented film containing metal particles in flake form the plane of which is oriented parallel to that of the film, the steps comprising subjecting a wet paint film which contains ferromagnetic flake pigment in amount of at least 5% of the metal pigment in said film, to the action of a magnetic field with the film positioned in the plane of said field, and at short intervals of time varying the directional angle between an axis in the plane of the film and said magnetic field through at least 90° while keeping the film in the plane of the field until the film has dried sufficiently to permit removal from said field without causing deorientation of the oriented metal flake particles.

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