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COPYING PAPER AND METHOD OF MAKING

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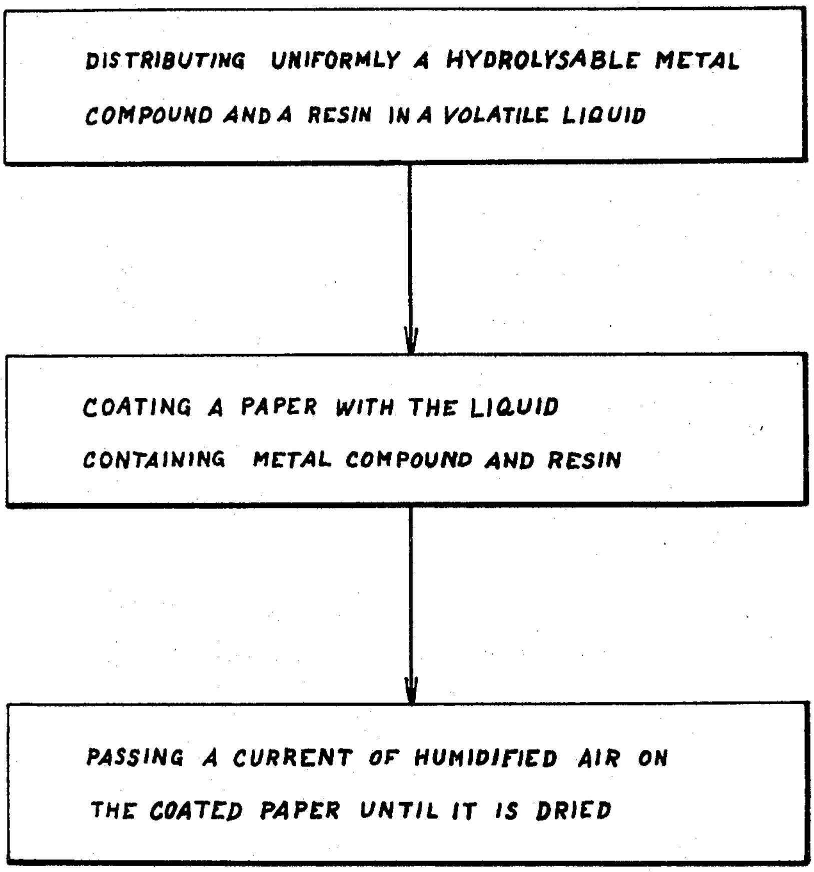

INVENTOR

Marc Henri Marie Joseph ERNST

BY Krafft & Wells

ATTORNEYS

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COPYING PAPER AND METHOD OF MAKING

Marc Henri Marie Joseph Ernst, Croix, France, assignor to Société dite: Les Petits-Fils de Leonard Danel, Loos, France, a body corporate of France

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ABSTRACT OF THE DISCLOSURE

This invention relates to manufacturing a coated paper by distributing uniformly in a volatile liquid a metal compound and a synthetic resin, said metal compounds being hydrolysable with an evolution of gas in situ, by depositing on a paper some of the liquid containing said resin and metal compound, then by hydrolysing the metal compound contained therein, and finally by drying completely the coated paper.

The present invention relates to a coated paper and to a process for its manufacture.

Coated papers already exist which have at least one side covered with at least one thin film having a microporous structure, consisting essentially of a synthetic resin.

Certain of these existing coated papers have a coating film consisting essentially of a plastic, and in particular thermoplastic, resin or including in addition a plasticiser, which may be sensitive to heat. A film of this kind, normally opaque and milky-white, becomes locally transparent under the action of pressure and/or moderate warming. Hence, a dark-coloured backing paper covered with a coating film of this kind makes it possible to write on it without ink, with the aid of a writing instrument such as a stylus, possibly warmed, by which pressure and/or warmth is applied to the free surface of the coating film. A special process for making coated paper of this kind was described, in particular, in my application Ser. No. 22,158, filed on Aug. 29, 1962, and now abandoned.

My above mentioned application also disclosed other coated papers of this kind, with coating films consisting essentially of a resin having little or no plasticity, so that it offers fairly high resistance to mechanical and thermal action. According to the degree of affinity of the synthetic resin for printing ink, it can absorb this quickly, thoroughly and very deeply, because of its microporosity. A paper faced with a coating film of this kind is therefore particularly suitable for printing, for which purpose it constitutes an excellent substitute for ordinary "art papers," which are coated with mineral substances and hence heavy and expensive.

In all the applications mentioned above, the weight per unit area (and hence the cost) of the coated paper largely depends on the thickness needed for the coating film in order to produce opacity deep enough to provide an effective mask for the backing paper, which may be dark in colour. Now the opacity of the coating films already mentioned is due almost entirely to multiple reflection of light rays from the walls of the minute air-filled spaces enclosed in the microporous films. In the case of a plastic film, it is the action of locally crushing these tiny internal spaces which, by locally compacting the film, at the same time renders it practically transparent. The texture of the existing coating films already referred to is not the most favourable to the production of deep opacity with the least possible thickness; the fact is that, because of the process by which the coating is made, the air-filled spaces it contains, or rather the solid particles bounding these spaces, are substantially spherical in shape, so that, for the volume they occupy, they present the smallest possible reflecting surface. Another factor is that these spaces or particles are of very widely varying size, which detracts from the opacity of the film as a whole.

A first object of my invention is to improve the coated papers already mentioned by reducing the thickness that must be given to their coating films to produce a given degree of opacity.

Another object of my invention is to provide a coated paper with a coating film, the internal spaces in which are mainly formed from substantially uniform microscopic gas bubbles anchored by adsorption to microscopic solid particles, said particles consisting of at least one insoluble mineral compound, more particularly metallic, such as an oxide or hydrate of a metal, and being distributed evenly and very densely throughout the film of synthetic resin.

Thickness for thickness, the film on my coated paper is distinctly more opaque than the film on coated papers known hitherto, bascially for the following reasons. The gas bubbles, being anchored to solid particles by adsorption, are not spherical in shape, so that for a given volume they present a reflecting surface greater than the minimum offered by a sphere. Moreover, the opacity obtained is enhanced by the fact that all the inner spaces in the coating film are substantially of equal size. Finally, the solid particles of mineral compounds further opacify the film on my coated paper, and all the more so because, particularly in the case of particles of metal compounds, they are better reflectors. Because of its extremely high opacity, the film on my coated paper can be reduced to very small thickness, of the order of 12 to 15 microns, and may weigh as little as 5 g./sg. m. My coated paper, which lends itself to all the existing applications already mentioned, thus has an extremely low weight per unit area and is consequently very cheap. This is due not only to the fact that only small quantities of common, very cheap substances (resins and metallic oxides) are required per unit area of the coating, but also to the possibility of employing for its backing (owing to the lightness of the coating) a paper that is thin, light and possibly of mediocre quality, which is therefore very cheap. In the case in which the film on my paper coated is so made as to be plastic (and thermoplastic in particular), its sensitivity to pressure and/or warmth applied by a writing instrument such as a stylus is distinctly greater than that of the thicker films on coated papers of similar type produced hitherto.

A further object of my invention is to provide a process for making my coated paper, this process consisting in distributing uniformly in a volatile liquid a metal compound and a synthetic resin, said metal compound being hydrolysable with an evolution of gas in situ, coating a paper by depositing thereon some of the liquid containing said resin and metal compound, then hydrolysing the metal compound contained in the liquid deposited on the paper, and finally drying completely the coated paper.

The hydrolysis of the metal compound contained in the liquid coating deposited on the backing paper thus produces simultaneously the evolution of gas in situ and the precipitation of very fine particles of an insoluble metal compound. By reason of the substantially uniform dispersion of the hydrolysable compound in the liquid phase of the coating, the formation of a bubble of gas and the precipitation of a particle of insoluble metal compound take place in conditions of proximity not only in time, but also in space, so that the gas bubble remains adsorbed, in other words superficially anchored to the solid particle formed simultaneously in close proximity. This phenomenon gives rise to the special texture of the film on my coated paper, with which the advantages already mentioned are bound up.

The hydrolysable metal compound contained in the liquid coating deposited on the paper backing is preferably chosen from among the carbides, nitrides and hydrides of metals and the organo-metallic compounds.

In a particular embodiment of the process for making my coated paper, the hydrolysis of the metal compound is produced by the moisture with which the air used for drying is charged.

In another embodiment of the process, the hydrolysis of the metal compound is produced by the water formed in situ by chemical reaction between two substances, such as a hydroperoxide and a reducing agent added to the liquid coating, this reaction being initiated prior to the complete drying of the coated paper by the application of calorific energy, direct or resulting from the conversion of energy of another kind (electrical, electromagnetic, radiant, supersonic energy, etc.). This last embodiment enables the total amount of water formed in situ to be readily pre-determined with precision.

The liquid phase of the coating deposited on the backing paper preferably consists of at least one volatile organic liquid, free from water and chemically inert to the paper and to the other components of the coating.

The drawing sets forth a flow sheet of the process.

Various ways of carrying out the process for making my coated papers are described hereunder by way of example:

Example 1

A quantity of 85 g. of vinyl chloride and co-polymerised maleic ester and 19 g. of plasticiser are dissolved in 1 kg. of methylene chloride, 0.5 g. of finely powdered calcium hydride being added to the solution. This suspension is then fined and homogenised by grinding in a ball mill for about two hours. It is then used for coating 17 sq. m. of an ordinary dark paper, which is subsequently dried in a tunnel traversed by a current of air having a temperature of about 50° C. and a relative humidity in the neighbourhood of 90%. Drying may be completed by then passing the coated paper over suitably heated cylinders.

The moisture brought in by the drying air will have hydrolysed the calcium hydride contained in the liquid coating, the reaction being:

$$CaH_2+H_2O\rightarrow 2H_2+CaO \qquad (1)$$

Each of the molecules of calcium hydride dispersed in the liquid coating has thus been dissociated, with the formation of two molecules of hydrogen gas and one molecule of CaO. These three molecules being formed in conditions of proximity in space and time, at least one of the two molecules of gas remains anchored by adsorption to the molecule of solid CaO. Provided the particles of calcium hydride dispersed in the liquid coating were of microscopic size, the same will apply to the CaO particles formed and to the gas bubbles anchored to them by adsorption, so that the dry coating will be microporous in structure, the minute internal spaces being formed by little bubbles of gas of microscopic proportions. The way in which these spaces have been formed explains why they are closely similar in size and why (on account of their being adsorbed by solid particles) their shapes depart considerably from the spherical.

The coating film deposited on the backing paper by the method just described is extremely thin (11 to 15 microns) and is very light, weighing approximately 5–6 g./sq. m. It is nevertheless opaque enough to mask the dark backing paper completely, making it appear perfectly white. This coating film is sensitive to such pressure and heat as may be applied to it by a marking stylus, for example, which renders it locally transparent, so that the dark backing shows through. Such paper is therefore very suitable for marking with a writing instrument uncharged with ink, such as the stylus of a recording instrument. It is also very suitable for the copying of documents without the aid of ordinary carbon paper, the writing pressure then being applied, for example, by the characters on a typewriter.

In the method described above, the calcium hydride may be replaced by some other metal compound such as can be hydrolysed with the evolution of gas in situ and by some other metal compound insoluble in the liquid phase of the coating, such as an oxide or hydrate of a metal. Thus, the calcium hydride may be replaced by, for example, the hydride of another metal or by carbides or nitrides of metals which meet the above requirements.

In the case of aluminium carbide, for instance, the hydrolysis reaction employed would be as follows:

$$Al_4C_3+12H_2O\rightarrow 3CH_4+4Al(OH)_3 \qquad (2)$$

In this case, the gas bubbles formed initially and adsorbed by the simultaneously formed particles of aluminium hydroxide consist of methane. Inasmuch as the density of the resultant internal spaces, which corresponds substantially to that of the aluminium carbide originally dispersed in the liquid coating, is high enough for these spaces to be in communication with one another and hence with the exposed surface of the coating, the methane initially filling them will finally be replaced by air. Magnesium and lithium hydrides, calcium and lithium carbides, calcium and magnesium nitrides and so forth may be used, for example, for manufacturing my coated paper, the gas bubbles formed in situ by their hydrolysis then consisting respectively of hydrogen, methane, ethane, acetylene, nitrogen or ammonia, which is finally replaced in the internal spaces by atmospheric air.

The synthetic resin which constitutes the base of the coating of my paper may be chosen from among the cellulose esters or ethers, polyvinylidene, polyesters, natural or artificial rubber, various co-polymers, etc.

In the example described above, the synthetic resin is dissolved in a volatile organic liquid (methylene chloride) and the hydrolysable metal compound is placed in suspension in the same liquid. It is equally possible, however, to dissolve the hydrolysable metal compound in a different volatile organic fluid from that in which the synthetic resin is dissolved, inasmuch as the two solutions can then be mixed together to form the liquid coating. Again, the synthetic resin may itself be in suspension in the organic liquid or liquids constituting the liquid phase of the coating, in which the hydrolysable metal compound is dispersed or dissolved. The liquid phase of the coating must always consist of one or more volatile organic liquids, free from water so as to avoid soaking the backing paper, in addition to being chemically inert to this backing and to the other components of the coating (synthetic resin, hydrolysable metal compound and various additives, such as, for example, where at least one of the solid components of the liquid coating is in suspension, an anti-sedimentation agent, such as amine salts, xanthogens, etc.).

Example 2

A solution (known as a Grignard solution) of magnesium methyl iodide is prepared by mixing, for example, 200 g. of ethyl alcohol, 71 g. of methyl iodide and 12.2 g. of magnesium. Another solution is made of 100 g. of polyvinyl chloride in 1 kg. of methylene chloride and this solution is mixed with 8 g. of the magnesium methyl iodide solution and 25 g. of dibutyl phthalate. After filtration, the mixture is used for uniformly coating 20 sq. m. of dark backing paper. This paper is then dried as described in Example 1. The moisture brought in by the drying air causes hydrolysis of the magnesium methyl iodide, the reaction being:

$$2CH_3\text{—}MgI+2H_2O\rightarrow 2CH_4+Mg(OH)_2+MgI_2 \qquad (3)$$

The process by which the inner spaces are formed is the same as that described in Example 1, the coated paper thus obtained also having very similar properties. The magnesium methyl iodide used in this example may be replaced by some other suitable hydrolysable organometallic compound.

The sensitivity to pressure of the coated papers made in accordance with the foregoing two examples is bound up with the fact that their coating has a microporous and plastic structure, the proportion of metal compound dispersed in the coating being sufficiently low not to reduce greatly the plasticity of the synthetic resin used; in point of fact, the amount of metal compound dispersed in the coating in these examples is less than 6% by weight. The heat-sensitivity of my coated papers can be increased if desired, primarily with a view to marking them with a warmed point or by thermal or supersonic radiation (partially intercepted by a screen, for example), if, for the coating film, one uses a highly thermoplastic resin or adds a plasticiser, possibly a heat-sensitive one, to a resin of medium plasticity. For such purposes, the amount of mineral compound dispersed in the coating film should still preferably be less than 6% by weight.

My coated paper can also have a microporous film which is practically insensitive to moderate mechanical and thermal actions. Such a coated paper is particularly suitable for printing, because of its ability to absorb ink (primarily printing ink) thoroughly, quickly and very deeply, by virtue of the microporous structure of its coating. It can therefore be used in printing in place of what are known as "art" papers, over which it has the essential advantage of a far lower weight per unit area and greater cheapness. These merits arise from the fact that the coating film on my paper has superior opacity, thickness for thickness, compared to the coating films, generally based on mineral compounds, used hitherto in the making of usual art papers. Quality for quality, therefore, my "art paper" can have a far thinner coating film than that of the usual art papers—only 12 to 15 microns, for example—and hence can be very light, weighing at most 5 g./sq. m. Owing to its lightweight nature, such a film can be carried by a backing which itself is very thin, very light and possibly of mediocre quality, such as a paper weighing about 20 g./sq. m. which is far thinner and lighter (and hence far less expensive) than the papers used for backing the usual art papers. The coating film on my art papers is distinguishable from that on my marking papers only by the proportion of mineral compound incorporated in them, this proportion being distinctly higher in the case of the former and lying between 20% and 40% by weight, for example, so as to make the coating film more resistant to pressing, scratching and heating, for example. With that same purpose in mind, it is advisable to use a resin having little or no plasticity for its manufacture, such as synthetic resins that are highly polymerised or poly-condensed. Finally, the nature of the resin chosen should be suited to that of the ink to be used, especially in the case of printing ink. For an ink that is water-bound or has an affinity for water, one should choose a resin having a similar affinity, such as methyl cellulose, whereas, for a greasy or oily ink, a resin having an affinity for grease or oil should be preferred, such as polyvinyl chloride or chloro-acetate, polystyrene, etc.

It is also within the scope of my present invention to improve the coated paper for manifolding, which is described in my Patent 3,294,571, filed on May 13, 1963, for "Sheets of Coated Paper." With these sheets of paper, coated front and back, marks made on the front of the first coated sheet can be transferred to the front of at least one other coated sheet placed below the first, by virtue of one of their faces being coated with at least one thin, friable coating adhering poorly to the paper, their other face having a thin coating adhering well to the paper, these two coatings being such that they adhere locally to each other wherever the two superimposed sheets are subjected to the marking pressure, so that the corresponding parts of the poorly adhering coating of one of these sheets is pulled away by the well adhering coating of the other sheet when the sheets are subsequently separated.

Although the manufacturing process proposed in my present application is applicable to all embodiments of the coated papers described in my above mentioned patent, all that will be given hereunder is an example of the making of sheets having the front covered with a coating that adheres well to the paper and the back covered with a thin coating of colouring matter, of a different colour from the coating on the front or, should this latter be transparent, from the surface of the backing paper to which it is applied, while this coating of colouring matter is itself covered with a poorly adhering coating, the two differently adhering coatings having the same microporous structure but the former being resistant while the latter is friable. The use of paper coated both sides in this way for manifolding is illustrated in FIGURE 2 of my above mentioned patent, and therefore need not be explained herein detail.

Example 3

The back of a 20 sq. m. run of paper is coated with an aqueous suspension of carbon black, with or without the addition of a wetting agent, so as to give, after drying, a non-cohesive coating of carbon weighing from 1 to 1.5 g./sq. m. Next, 90 g. of polyvinyl chloro-acetate is dissolved in 1 kg. of dry methylene chloride. To this solution is added 3 g. of cumene hydroperoxide, 1.2 g. of propyl amine and 0.5 g. of calcium hydride, which could be replaced by 0.75 g. of calcium carbide or 8 g. of the Grignard reagent solution mentioned in Example 2. With the resulting mixture, that side of the paper that is already covered with carbon is coated uniformly. This liquid coating is then allowed to dry partially, the run of paper then being passed across an infra-red radiator placed transversely to it. The heat received by the still liquid coating brings about the reduction of the cumene hydroperoxide by the propyl amine, the water thus formed in situ at once hydrolysing the calcium hydride (or the analogous metal compound) according to reaction (1) above. This, of course, enables the amount of water liberated in situ in the liquid coating to be adjusted very accurately to the amount of metal compound therein, which requires hydrolysing. The heat energy put into the liquid coating deposited on the backing paper, necessary to initiate the reduction of the hydroperoxide, may equally well be introduced directly, by contact, or by the conversion of energy of another kind, such as electrical energy (glow discharges), electromagnetic energy (high-frequency electric field), supersonic energy, etc. The formation of water in situ can naturally be obtained not only by a reducing reaction, but by any other suitable chemical reaction between two judiciously chosen additives in the liquid coating. The hydrolysis reaction is accompanied by opacification of the coating, which, once dry, is friable. The still exposed front of the run of paper is next coated with one of the solutions mentioned in Examples 1 and 2, and then dried as described in Example 1. The coating thus deposited on the front of the paper is not friable like that deposited on the back. If the front and back of two sheets cut from the run of coated paper made as described above be superimposed one on the other, it will be found that the two microporous films brought into contact with each other will stick together under the action of pressure, for instance, and that when the two sheets are pulled apart the adhering portions of the friable coating on the back of one sheet will be pulled away by the resistant, i.e. non-friable coating on the front of the other sheet, along with the corresponding portions of the intermediate layer of carbon on the back of the first sheet. The adhesion between the two coatings can be further enhanced by increasing the proportion of plasticiser contained in the non-friable coating adhering well to the front, or by adding an adhesive substance to it.

The non-friable adhesive coating on the front can also be produced by the use of the same solution as for the friable coating on the back, but with the addition of a plasticiser to this solution, such as, in the case of the preceding numerical example, 20 g. of plasticiser. The hydrolysing and drying operations will then be the same for the two microporous coatings on front and back.

What I claim is:

1. Sheets of paper coated with thin films of different kinds on their two faces, said coated sheets, when superposed with each face thereof having a coating film of a first kind in contact with another face thereof having a coating film of a second kind, being adapted to transfer the inscribing made on the upper sheet by a pressure-exerting member to all the sheets placed thereunder, each thin coating film of a first kind prepared by the method of claim **5**, whereby each coating film of a first kind is locally broken into sharply outlined fragments by the inscribing pressure, whereas each thin coating film of a second kind is adapted to retain the broken fragments of the frangible coating film of a first kind in contact therewith, when the superposed sheets, the inscribing having been made thereupon, are separated from one another.

2. The sheets of coated paper of claim **1**, in which each thin coating film of a second kind consists essentially of a slightly adhesive substance.

3. The sheets of coated paper of claim **1**, in which each thin coating film of a second kind consists essentially of a film-forming, synthetic resin, deposited as a microporous, pressure resistant film, whereby the microporous films of different kinds which are in contact with each other, are superficially interpenetrated by each other under the inscribing pressure, but only each frangible coating film of a first kind is broken by said inscribing pressure into sharply outlined fragments, which are retained by the unbroken, microporous film of a second kind in contact therewith, when the superposed sheets are separated from one another.

4. The sheets of coated paper of claim **1**, in which an intermediate, thin and friable layer of a coloring substance is inserted between each sheet of paper and the friable film of a first kind thereon.

5. A process for manufacturing coated paper, consisting in distributing uniformly a mixture comprising a film-forming, synthetic resin and a metal compound in a proportion not over 40% by weight, in at least one volatile liquid, free from water and chemically inert to said resin and metal compound as well as to usual papers, said metal compound being selected from among the metal compounds hydrolysable with evolution of gas in situ and precipitation of microscopic particles of insoluble metal oxides, depositing on a paper a liquid coating with said resin and metal compound being distributed uniformly throughout, then hydrolysing completely the metal compound contained in said liquid coating on the paper, whereby plenty of microscopic particles of insoluble metal oxide are precipitated with a uniform distribution throughout said liquid coating and, at each place thereof, where such a metal oxide particle is precipitated, a microscopic gas bubble is evolved and anchored on said particle by adsorption, all of said anchored, non-spherical, rather prolate bubbles having substantially a same uniform, microscopic size, and finally drying completely said liquid coating, whereby there is obtained on said paper a dry coating, in which said anchored gas bubbles form micropores in a sufficient amount to opacify said dry coating.

6. The process of claim **5**, in which at least one of the resin and metal compound is dissolved in the volatile liquid.

7. The process of claim **5**, in which at least one of the resin and metal compound is dispersed uniformly throughout the volatile liquid.

8. The process of claim **5**, in which the hydrolysable metal compound is selected from among the group consisting of metal carbides, metal nitrides, metal hydrides and organo-metallic compounds.

9. The process of claim **5**, in which the coated paper is dried by passing a current of air thereon.

10. A coated paper prepared by the process of claim **5**.

11. The coated paper of claim **10**, whose dried coating has a weight substantially equal to 5 g. per sq. m. and a thickness of 12 to 15 microns.

12. The coated paper of claim **10**, whose dried coating contains particles of a hydrated metal oxide.

13. A coated paper for writing and printing thereon without ink, prepared by the process of claim **5**, whose dried coating is pressure-transparentizable and contains microscopic particles of an insoluble metal oxide in an amount not over 6%.

14. A coated paper for writing and printing thereon without ink, prepared by the process of claim **5**, whose dried coating is heat-transparentizable and contains microscopic particles of an insoluble metal oxide in an amount not over 6%.

15. A coated paper for writing and printing thereon without ink, prepared by the process of claim **5**, in which the synthetic resin is of a nonplastic kind and in which the insoluble metal oxide is present in an amount of 20 to 50% by weight of the coating.

16. A process for manufacturing coated paper, consisting in distributing uniformly a mixture comprising a film-forming, synthetic resin and a metal compound in a proportion not over 40% by weight, in at least one volatile liquid, free from water and chemically inert to said resin and metal compound as well as to usual papers said metal compound being selected from among the metal compounds hydrolysable with evolution of gas in situ and precipitation of microscopic particles of insoluble metal oxides, depositing on a paper a liquid coating with said resin and metal compound being distributed uniformly throughout, then passing a current of humidified air on the coated paper, whereby the metal compound contained in said liquid coating is completely hydrolysed by water deposited thereon from said humidified air, a plenty of microscopic particles of insoluble metal oxide are precipitated with a uniform distribution throughout said liquid coating and, at each place thereof, where such a metal oxide particle is precipitated, a microscopic gas bubble is evolved and anchored on said particle by adsorption, all of said anchored, non-spherical, rather prolate bubbles having substantially a same uniform, microscopic size, then said liquid coating is completely dried by said current of air, whereby there is obtained on said paper a dry coating, in which said anchored gas bubbles form micropores in a sufficient amount to opacify said dry coating.

17. The process of claim **16**, in which the current of humidified air passed on the coated paper has a temperature substantially equal to 50° C. and a relative humidity substantially equal to 90%.

18. A process for manufacturing coated paper, consisting in distributing uniformly a mixture comprising a film-forming, synthetic resin, a metal compound in a proportion not over 40% by weight, and a pair of reactive substances, in at least one volatile liquid, free from water and chemically inert to said resin, metal compound and reactive substances as well as to usual papers, said metal compound being selected from among the metal compounds hydrolysable with evolution of gas in situ and precipitation of microscopic particles of insoluble metal oxides, and said pair of reactive substances being selected from among the pairs of substances which are reacted by a heat energy supply with a formation of water, depositing on a paper a liquid coating with said resin, metal compound and reactive substances being distributed uniformly throughout, then supplying heat energy to said liquid coating, whereby said reactive substances are reacted with one another and form water for hydrolysing said metal compound, a plenty of microscopic particles of insoluble metal oxide are precipitated with a uniform distribution throughout said liquid coating and, at each place thereof, where such a metal oxide particle is precipitated, a microscopic gas bubble is evolved and anchored on said particle by adsorption, all of said anchored, non-spherical, rather prolate bubbles having substantially a same uniform, microscopic size, and finally drying completely said liquid coating, whereby there is obtained on said paper a dry coating in which said anchored gas bubbles form micropores in a sufficient amount to opacify said dry coating.

19. The process of claim **18**, in which the reactive substances are a hydroperoxide and a reducing agent.

20. The process of claim **18**, in which the heat energy supplied to the liquid deposited on the paper is converted therein from another physical form of energy.

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MURRAY KATZ, *Primary Examiner.*