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(54) Powder coated porous substrate and a method for powder coating a porous substrate.

(57) The invention relates to a method for powder coating a substrate with a powder coating composition comprising the following steps

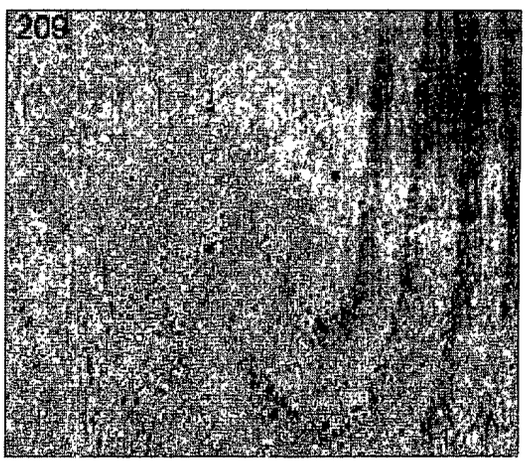
a) preheating the substrate by means of a heating source until a desired maximum temperature (T_{max}) which is at least 5 °C above the melting temperature of the powder coating composition,
b) reducing or switching off the power of the heating source before, simultaneously with or after applying the powder coating composition onto the preheated substrate, and
c) curing the powder coating composition,

with the proviso that after step a) and before step c) the temperature of the substrate should not increase.

The invention further relates to a wholly or partly powder coated porous substrate wherein no visible blisters are present.

Photograph III

photograph K



EP 1 258 296 A1

Description

[0001] The invention relates to a wholly or partly powder coated substrate. The invention further relates to a method for powder coating a substrate and to the use of the method for powder coating a substrate, especially a porous substrate.

[0002] A powder coated substrate is known from the article:

"Pulverbeschichtung von Holzwerkstoffen- ein innovatives Lackierkonzept" presented at the "Technologie-Tag der Deutsche Forschungsgesellschaft für Oberflächenbehandlung e.V. am 8. Dezember 1999 in Paderborn". In this article it is described that it is possible under certain conditions to powder coat a substrate made out of beech wood. It is stated that this kind of wood has no large pores and is lean in compounds that are trapped inside the wood structure ("Holzinhaltstoffe"). However it is also stated that other kinds of solid wood present various problems resulting in the impossibility to powder coat these kinds of wood without blisters. The article further gives only a very general description of a method for powder coating beech wood. It is only stated that the beech wood is powder coated at a temperature equal to or lower than 100°C. In that case no blisters are formed.

[0003] The purpose of the invention is to overcome the above indicated disadvantages and to provide a powder coated porous substrate and a method for powder coating a substrate with a powder coating composition, and especially a method for powder coating a porous substrate.

[0004] Here and hereinafter with "porous substrate" is meant a substrate that when being powder coated by the standard electrostatic spraying powder coating method visible blisters occur. In this standard electrostatic spraying powder coating method a standard DSM UV powder coating composition is applied in a layer with a thickness of 90-110 µm, onto the substrate with a Tribo gun (Nordson Tribo gun, type Tribomatic II Handgun). The standard DSM UV powder coating composition consists of the following components (by weight): 830 parts Uracross® P3125 polyester resin (DSM Resins), 170 parts Uracross® P3307 (DSM Resins), 10 parts Irgacure® 2959 (Ciba Specialty Chemicals Inc.) and 6.7 parts Byk® 361 (Byk Cera). Thereafter the powder coating composition is melted in an infrared oven (Triab IR machine medium wave IR lamps, temperature IR controlled at the coating surface) during 2 minutes at a temperature of 100°C. Six seconds after leaving the infrared oven the molten powder coating composition is cured by means of UV-radiation (Triab UV machine, UV lamp Fusion H-bulb (120 W/cm) UV doses 1000 mJ/cm^2)

[0005] The purpose of the invention is reached by providing a method for powder coating a substrate with a powder coating composition comprising the following steps:

a) preheating the substrate by means of a heating source until a desired maximum temperature (T_{max}) which is at least 5°C above the melting temperature of the powder coating composition,
b) reducing or switching off the power of the heating source before, simultaneously with or after applying the powder coating composition onto the preheated substrate, and
c) curing the powder coating composition,

with the proviso that after step a) and before step c) the temperature of the substrate should not increase.

[0006] The above described method is used to powder coat a substrate. It has appeared to be especially advantageous for porous substrates or heat-sensitive (either porous or non-porous) substrates.

[0007] The type of heating source is not critical. Commercial equipment as for example a thermal oven, thermal convection oven, IR-radiation or NIR-radiation can be used. However it has been found that the method according to the invention can be advantageously performed by applying infrared (IR) radiation or near infrared (NIR) radiation as the heating source. IR-, and NIR- radiation are especially suitable to pre-heat, in a well-controlled manner, specific areas. An additional advantage of the use of (N)IR-radiation is that, when large substrates are involved, the resulting energy costs will be lower than when other means are used.

[0008] The preheating of the substrate is effected until the desired maximum temperature (T_{max}) is reached. The desired maximum temperature can for example depend on the nature of the substrate and/or the properties of the powder coating composition. When a substrate is heat-sensitive the desired maximum temperature will generally be lower than when the substrate is not heat-sensitive. A heat-sensitive substrate is a substrate that under the influence of increased temperature during a prolonged period of time shows changes in dimension, structure and/or colour. It can even occur that upon the action of increased temperature the substrate degrades in more or less extent. An indication whether a substrate is heat-sensitive can for example be obtained by exposing the substrate during for example 10 minutes to a temperature of for example 180°C.

[0009] The method according to the invention has appeared to be especially advantageous for these heat-sensitive substrates because by applying this method no disadvantageous effects of the heating have been observed. The properties of the powder coating composition determine amongst other things, the flow of the molten powder coating

composition. A certain level of flow should be reached to obtain a (powder) coated substrate with acceptable appearance and properties. The level of flow necessary to reach this goal is different for most powder coating compositions, therefore no specific indication can be given in this respect, however it can be easily determined by the skilled man. The desired maximum temperature can easily, without any undue burden, be determined by the man skilled in the art.

[0010] The desired maximum temperature should at least be 5°C above the melting temperature of the powder coating composition. The reason for this is to be sure that the whole powder coating composition is molten. Preferably the desired maximum temperature is at least 25°C higher than the melting temperature of the powder coating composition. The melting temperature can be determined by standard DSC-techniques, for example a DSC 2920 from TA Instruments. The DSC-measurement is performed with modulated DSC. The heating and cooling rate is 1°C/min, the modulation amplitude is 0.159 °C/60 seconds ("heat-only mode"). In case of a crystalline component the end of the melting peak is used to determine the melting temperature (T_{melt}). In case of an amorphous component the glass transition temperature (T_g) is used to determine the melting temperature.

[0011] The duration of the preheating step (a) is not particularly critical and can be chosen between wide ranges. The duration and heat power will in most cases depend on the nature of the substrate by the fact that for some substrates a longer time is needed to reach the desired maximum temperature than for others. Amongst other things this is determined by the heat capacity of the substrate. The duration can for example be chosen between 0,1 and 1800 seconds, preferably between 1 and 600 seconds, more preferred between 2 and 300 seconds.

[0012] The substrates that especially benefit from the invention are the porous substrates. The substrates that benefit most from the invention are the porous, heat-sensitive substrates. Examples of porous substrates (heat-sensitive or not) are solid wood, veneer, chip wood, wood composite material, cork, concrete, stone, brick, magnesium alloy, galvanized iron and some types of plastic, for example sheet moulding compound (SMC).

[0013] Examples of heat sensitive substrates (porous or not) are wood, for example hardwood, hard board, wood composites, for example particle board, high, medium or low density fiber board, plywood and other substrates that contain a significant amount of wood.

[0014] Examples of porous, heat sensitive substrates include all kinds of solid wood, veneer, chip wood, wood composite material, cork, paper, cardboard or plastic, for example SMC.

[0015] However the invention is also suitable for traditional heat resistant (either porous or not) substrates, for example metal, (galvanized) steel, cast iron, other alloys, glass, ceramic and bricks.

[0016] In case that the porous substrate is wood it has appeared to be advantageous to dry the wooden substrate before step a) in the method for powder coating according to the invention, to reach a humidity of the substrate between 8-22% (borders inclusive). In case this additional drying step is performed it has appeared to give a lower risk for the formation of blisters. The humidity can be determined with the aid of a hygrometer.

[0017] With powder coating composition is generally meant the final composition that is suitable for application onto a substrate. The powder coating composition generally comprises one or more resins with functional groups and/or one or more curing agents. The powder coating composition can in addition to this also comprise one or more of the following: photo-initiator, pigment, and other generally used coating additives. With (powder) coating is generally meant the resulting product from the curing of the (powder) coating composition.

[0018] It is possible in the method according to the invention to maintain the substrate at the maximum temperature for a certain length of time before the power of the heating source is reduced or switched off. The length of time can be chosen in a wide range, for example between 0,1 and 300 seconds. Preferably this length of time is between 1 and 120 seconds, more preferred between 2 and 20 seconds.

[0019] After that the substrate has been preheated until the desired maximum temperature (T_{max}) in step (a), the power of the heating source is in step b) either reduced or switched off. This can be effected before, simultaneously with or after that the powder coating composition has been applied onto the preheated substrate.

[0020] After the reducing or switching off of the power of the heating source in step b), the powder coating composition is cured in step c). The curing of the powder coating composition can be effected by radiation curing, for example electron beam radiation (EB) or ultraviolet radiation (UV). It is preferred to effect the curing by the use of UV radiation. Standard UV light sources are suitable for curing the coating, for example medium pressure mercury-, iron doped mercury-, and/or gallium doped mercury-vapor lamps. Hardening of the coating is normally very quick. It can take between 1 millisecond and 25 seconds. Preferably it takes less than 10 seconds, more preferably less than 5 seconds.

[0021] The temperature at which the curing takes place is not especially critical and can vary within wide ranges. Curing can for example take place at elevated temperatures, but it is also possible to cure at lower temperatures, for example room temperature or below. However when the temperature during the curing step becomes too low the properties of the final coating are not satisfactory. The lowest possible temperature that still gives acceptable coating properties depends on the powder coating composition. This can be easily determined by the man skilled in the art. The preferred temperature range for curing is 15-200 °C, more preferred 20-175°C and most preferred between 25-150°C.

[0022] The thickness of the powder coating layer can vary between wide ranges. Generally it is between 5 and 500

μm, preferably the thickness is between 10 and 200μm, more preferred between 50 and 100 μm.

[0023] The powder coating composition can be applied onto the substrate in a repeated cycle of step a), b) and step c). By repeating step a), b) and step c) several layers of the powder coating composition are applied onto the substrate. This can be desirable when the powder coating layer is very thin and where full coverage is desired or in case layers with several colours or compositions should be applied.

[0024] The method for powder coating a substrate according to the invention has always the proviso that after step a) and before step c) the temperature of the substrate should not increase. In case these requirements are not met the appearance and properties of the final coating are not satisfactory in that blisters are visible.

[0025] Because of the fact that the substrate has a certain thickness, the temperature of the substrate will, at a fixed point in time, not be the same when measured along the thickness of the substrate. The temperature of the substrate will show a kind of temperature profile along the thickness. With the above-mentioned proviso it is meant that although the temperature at a certain position along the thickness can increase, the total temperature of the substrate should not increase. Thus a local increase in temperature should be compensated for by a decrease at another location. The total temperature of the substrate should according to the proviso not increase, but stay the same or decrease.

[0026] The temperature of the coating composition can be determined by use of a high performance infrared thermometer (Raynger MX4 from the firm Raytek). In certain cases, which can be easily determined by the man skilled in the art, it is advantageous in the method for powder coating according to the invention to allow the applied powder coating composition on the substrate sufficient time to flow after the powder coating composition is applied in step b) and before the curing of step c) is effected. The length of time that is used depends on the nature and properties of both the substrate and the powder coating composition. The length of time can vary between wide ranges but is mostly relatively short, for example between 0,1 and 900 seconds, preferably between 0,5 and 240 seconds, more preferred between 1 and 120 seconds.

[0027] The method for powder coating a substrate according to the invention can sometimes benefit from the addition, after step b), of an additional step (b2) that consists of the mildly heating the powder coating composition during the application of the powder coating composition, to compensate for the cooling action of the powder-gun, such as to reach a slowly decreasing temperature of the substrate top-layer with the applied powder coating composition, with the proviso that after step a) and before step c) the temperature of the substrate should not increase.

[0028] The additional step 2b can be very valuable when powder coating a porous, heat-sensitive substrate. As heat-sensitive substrates cannot be pre-heated to very high temperatures, there is most of the time only a limited margin between the desired maximum temperature and the melting temperature of the powder coating composition. The result of this limited margin is that the temperature of the top-layer of the substrate together with the composition can become too low because of the cooling action due to the application of the cold powder with a powder gun and the air flow of the powder gun. In such a situation it can be beneficial to apply additional (mild) heating during the spraying of the powder coating composition onto the substrate. It is also possible to consider preheated air upon spraying.

[0029] The invention further relates to a wholly or partly powder coated porous substrate wherein in the powder coating no blisters are present with a size larger than 30 μm. It is generally accepted that blisters with a size larger than 30 μm are at least visible. It is preferred to have no blisters present with a size larger than 15 μm. It is even more preferred to have no visible (by the naked eye) blisters at all present.

[0030] The thickness of the finally obtained powder coating on the substrate is chosen on demand. Mostly depending on the final application wherein the wholly or partly powder coated substrate will be used. However it is preferred that the thickness of the powder coating is between 10 and 1000 μm, because between these ranges the best appearance of the coated substrate can be obtained.

[0031] The substrate can be coated with one or with various layers of coating compositions mostly depending on the final thickness or properties that is/are desired. In case that various layers are applied, the coating compositions used can all have the same composition and properties or they can have different compositions and properties, for example different colours or different outdoor resistance. The various coating compositions that are used to apply the various layers can even belong to different kinds of systems, for example one layer can be obtained after applying a powder coating composition and the other layer or layers can be obtained by applying another powder coating composition or even a wet coating composition. No real limitations exist in the combination of coating compositions. However the preferred substrate comprises one or more coating layer(s) wherein the only or first layer is the powder coating layer. The more preferred substrate consists of only one coating layer.

[0032] Coating compositions are applied onto a substrate for various reasons, for example to protect the substrate against weather influences (ageing, discoloration) or to give the substrate a better appearance. Depending on the reasons why the substrate is coated various powder coating compositions can be chosen. For example powder coating compositions exist that result in a clear coat or powder coating compositions that on the other hand result in a pigmented coat. Substrates that have an attractive appearance of themselves are preferably powder coated with a clear coat. Examples of this kind of substrates are all kinds of wood. Substrates whose appearance can be improved are preferably coated with a pigmented coat.

[0033] The invention further relates to the coating and the powder-coated substrate that is obtainable by the method according to the invention, which method is described above.

[0034] The invention will now be elucidated by means of the following non-restrictive examples.

EXAMPLES

[0035] In the following examples a powder coating composition with a melting temperature of approximately 100°c was used. it had the following composition:

COMPONENT	PARTS (BY WEIGHT)	REMARKS
URACROSS® P 3125	830	EX DSM COATING RESINS
URACROSS® P 3307	170	EX DSM COATING RESINS
IRGACURE® 2959	10	EX CIBA SPECIALTY CHEMICALS INC.
BYK® 361	6,7	EX BYK CERA

Deal coated with powder-UV

[0036] Deal was coated with the powder coating composition using 4 different methods:

Example I (according to the invention)

[0037] The deal was pre-heated by means of IR-radiation to 150°C in 1 minute. The powder coating composition was sprayed onto the substrate upon which the substrate was cooled down to 120°C, in such a way that no outgassing from the deal occurred. The coating was melted to a homogeneous, well-flown film and was subsequently cured by means of UV-radiation (1 J/cm2). The result is shown in photograph 1.

Comparative Example A:

[0038] The deal was sprayed at room temperature with the same powder coating composition as in Example I. Thereafter the coating was melted at 100°C by means of IR-radiation. After proper flowing the powder coating composition was cured by means of UV-radiation (1 J/cm2). The result is shown in photograph A.

Comparative Example B:

[0039] Method in which pre-heating was used to prepare the substrate. The deal was pre-heated in 10 min. to 120°C in an oven. Due to transport from oven to spray cabin and spraying with the powder coating composition, the temperature of the substrate became under the melting temperature of the powder coating composition and the top layer of the powder coating composition did not show proper melting. To obtain a homogeneous film the powder coated deal was heated up again by means of IR-radiation in 2 minutes to 120°C and then cured by means of UV-radiation (1 J/cm2). The result is shown in photograph B.

Comparative Example C:

[0040] Method in which pre-heating was used to prepare the substrate as described in Comparative Example B. The temperature of the substrate became under the melting temperature of the powder coating composition and the top layer of the powder coating composition did not show proper melting. Now the coated surface was heated-up in 2 minutes to 100°C (in stead of 120°C). The result is shown in photograph C.

[0041] From photograph I and A-C it is obvious that you only can reach a good performance without blisters if you coat the (porous, heat-sensitive) substrate deal according to the invention.

Oak coated with powder-UV

[0042] Oak was coated with the powder coating composition using 4 different methods:

Example II (according to the invention)

[0043] The oak was pre-heated by means of IR-radiation to 150°C in 1 minute. The powder coating composition was sprayed onto the substrate, subsequently it was cooled down to 120°C, in such a way that no outgassing from the oak occurred. The powder coating composition was melted to a homogeneous well-flown film and subsequentlycured by means of UV-radiation (1 J/cm2).

Comparative Example D

[0044] The oak was sprayed with the powder coating composition at room temperature. Thereaftere the powder coating composition was melted at 100°C by means of IR-radiation, followed by UV-curing (1 J/cm2). The result is shown in photograph D.

Comparative Example E

[0045] The oak was pre-heated in 10 min. to 120°C in an oven. Due to transport from oven to spray cabin and spraying with the powder coating composition the temperature of the substrate became under the melting temperature of the powder coating composition. As a result of that the powder coating composition did not show proper melting of the top layer. To obtain a homogeneous film the coated oak was heated up again by means of IR-radiation in 2 minutes to 120°C and then cured by UV-radiation (1 J/cm2). The result is shown in photograph E.

Comparative Example F:

[0046] Method in which pre-heating was used to prepare the substrate as described in Comparative Example E. Here also the temperature of the substrate dropped below the melting temperature. The coated surface was heated-up by means of IR-radiation in 2 minutes to 100°C (in stead of 120°C).
The result is shown in photograph F.

[0047] From photograph II and D-F it is obvious that you only can reach a good performance without blisters if you coat the porous substrate oak according to the method according to the invention.

Walnut coated with powder-UV

[0048] Walnut was coated with the powder coating composition using 4 different methods:

Example III (according to the invention)

[0049] Walnut was pre-heated by means of IR-radiation to 150°C in 1 minute. The powder coating composition was sprayed and the sprayed substrate was cooled down to 120°C, in such a way that no out gassing from the walnut occurred. The coating was melted to a homogeneous well-flown film and subsequently cured by UV-radiation (1 J/cm2). The result is shown in photograph III.

Comparative Example G:

[0050] The walnut was sprayed with the powder coating composition at room temperature. Subsequently the powder coating composition was melted at 100°C by means of IR-radiation, followed by UV-curing (1 J/cm2). The result is shown in photograph G.

Comparative Example H:

[0051] The walnut was pre-heated in 10 min. to 120°C. Due to transport from oven to spray cabin and spraying with the powder coating composition, the temperature of the substrate became under the melting temperature of the powder coating composition and the top layer of the powder coating composition did not show proper melting. To obtain a homogeneous film the powder coated walnut was heated up again by means of IR-radiation in 2 minutes to 120°C and then cured by means of UV-radiation (1 J/cm2).
The result is shown in photograph H.

Comparative Example K:

[0052] Method in which pre-heating is used to prepare the substrate as described in Comparative Example H and wherein also the temperature of the substrate became under the melting temperature of the powder coating composition. Subsequently the powder coated surface was heated-up by means of IR-radiation in 2 minutes to 100°C (in stead of 120°C).
The result is shown in photograph K.

[0053] From photograph III, G, H and K it is obvious that you only can reach a good performance without blisters if you coat the porous substrate walnut according to the method according to the invention.

Claims

1. Wholly or partly powder coated substrate **characterized in that** the substrate is porous and that in the powder coating no blisters are present with a size larger than 30 μm

2. Substrate according to claim 1 **characterized in that** in the powder coating no visible blisters are present

3. Substrate according to any one of claim 1-2 **characterized in that** the thickness of the powder coating is between 10 and 1000 μm

4. Substrate according to any one of claim 1-3 comprising one or more layers **characterized in that** the powder coating is the only or first layer

5. Substrate according to any one of claim 1-4 **characterized in that** the coating consists of only one layer

6. Substrate according to any one of claim 1-6 **characterized in that** the porous substrate is solid wood, veneer, chip wood, wood composite material, cork, concrete, stone, brick, magnesium alloy, galvanized iron or plastic

7. Substrate according to any one of claim 1-5 **characterized in that** the substrate is heat-sensitive

8. Substrate according to claim 7 **characterized in that** the heat-sensitive substrate is hardwood, hard board or a wood composite

9. Substrate according to any one of claim 1-8 **characterized in that** the powder coating is a clearcoat

10. Substrate according to any one of claim 1-9 **characterized in that** the powder coating is a pigmented coat

11. Method for powder coating a substrate with a powder coating composition comprising the following steps

    a) preheating the substrate by means of a heating source until a desired maximum temperature (T_{max}) which is at least 5 °C above the melting temperature of the powder coating composition,
    b) reducing or switching off the power of the heating source before, simultaneously with or after applying the powder coating composition onto the preheated substrate, and
    c) curing the powder coating composition, with the proviso that after step a) and before step c) the temperature of the substrate should not increase.

12. Method according to claim 11 **characterized in that** the substrate is a porous substrate or a heat-sensitive (porous or non-porous) substrate

13. Method for powder coating a substrate according to any one of claim 11 or 12 **characterized in that** it after step b) comprises an additional step (b2):

    b2) during the application of the powder coating composition the powder coating composition is mildly heated to compensate for the cooling action of the powder-gun, such as to reach a slowly decreasing temperature of the substrate top-layer with the applied powder coating composition, with the proviso that the temperature of the top-layer together with the applied powder coating composition should not exceed T_{max} of step a)

14. Method for powder coating according to any one of claim 11-13 **characterized in that** the substrate is maintained at the maximum temperature for a certain length of time before the power of the heating source is reduced or switched off

15. Method for powder coating according to any one of claim 11-14 **characterized in that** the heating source is infrared (IR) radiation, near infrared (NIR) radiation, thermal oven or thermal convection oven

16. Method for powder coating according to any one of claim 11-15 **characterized in that** the curing is effected by UV-radiation

17. Method for powder coating according to any one of claim 11-16 **characterized in that** the maximum temperature (T_{max}) is at least 25 °C higher than the melting temperature of the powder coating composition

18. Method for powder coating according to any one of claim 11-17 **characterized in that** the preheating step (a) is effected within 1800 seconds

19. Method for powder coating according to any one of claim 11-18 **characterized in that** the powder coating composition is applied in a repeated cycle of step a), b) and step c)

20. Method for powder coating according to any one of claim 11-19 **characterized in that** after the powder coating composition is applied in step b) and before the curing of step c) is effected, the applied powder coating composition on the substrate is allowed sufficient time to flow

21. Method for powder coating according to any one of claim 11-20 **characterized in that** the porous substrate is made of wood and that it was dried before step a) to reach a humidity of the substrate between 8-22% (borders inclusive)

22. Use of a method according to any one of claim 11-21 for powder coating a substrate

23. Use of a method according to any one of claim 11-21 for powder coating a porous, heat-sensitive substrate

24. Use according to claim 23 **characterized in that** the heat-sensitive porous substrate is solid wood, veneer, chip wood, wood composite material, cork, paper, cardboard or plastic

25. Use of a method according to any one of claim 11-21 for powder coating a porous substrate **characterized in that** the porous substrate is concrete, stone, brick, magnesium alloy or galvanized iron

26. Use of a method according to any one of claim 11-21 to apply a powder coating as a primer

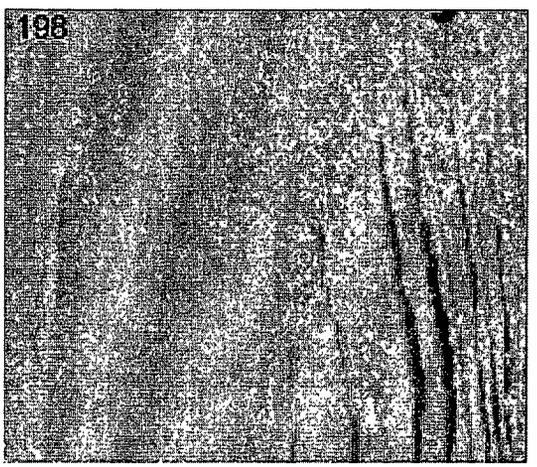
27. Coating obtainable by the method according to any one of claims 11-21

28. Powder coated substrate obtainable by the method according to any one of 11-21

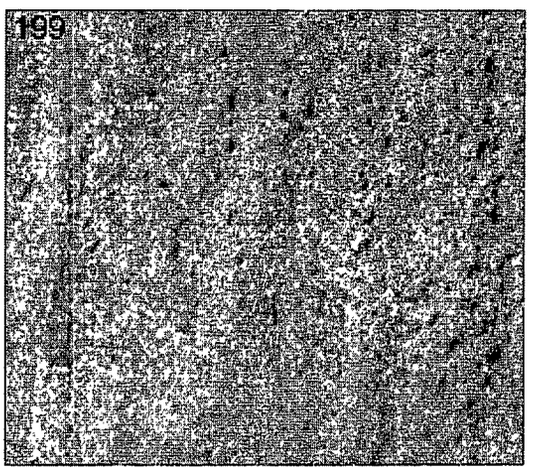
Photographs (scale 10x10 cm) of the obtained results in the Examples

photograph I, A, B, C:

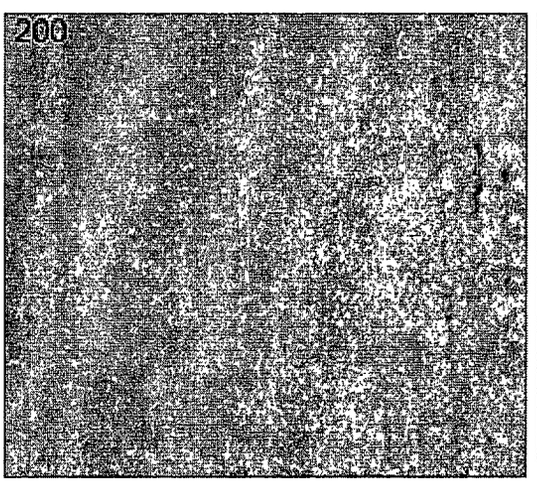
photograph I



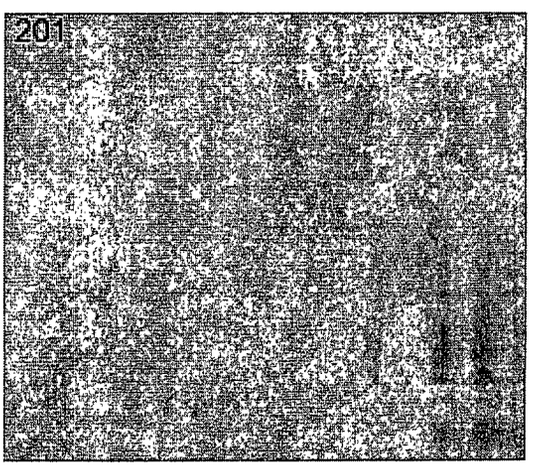
photograph A



photograph B

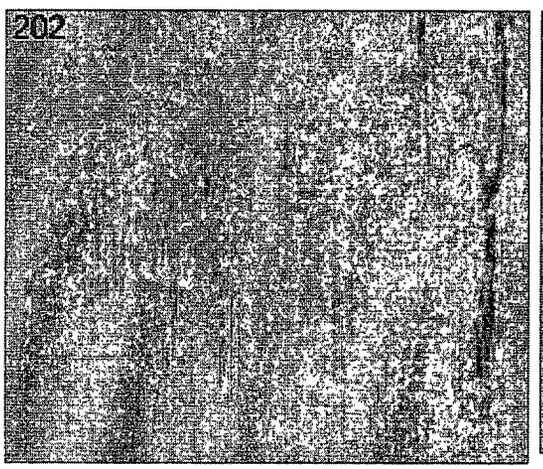


photograph C

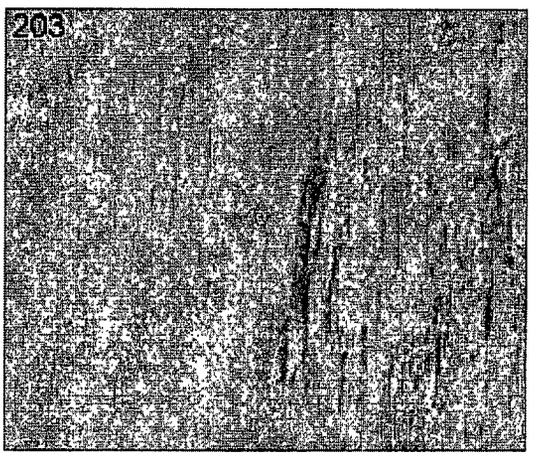


photograph II, D, E, F

photograph II



photograph D



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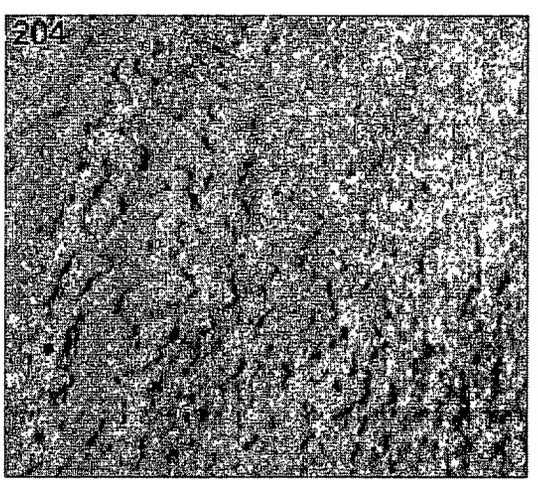

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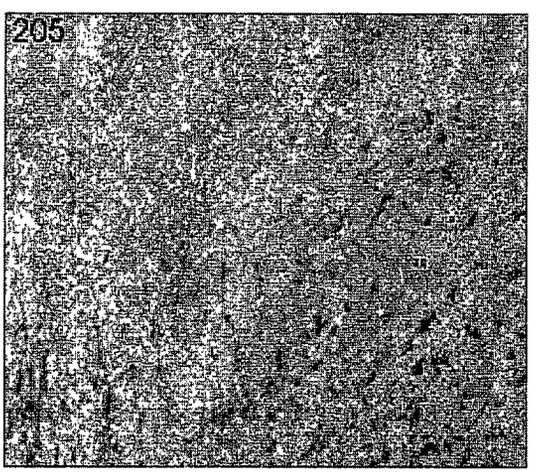
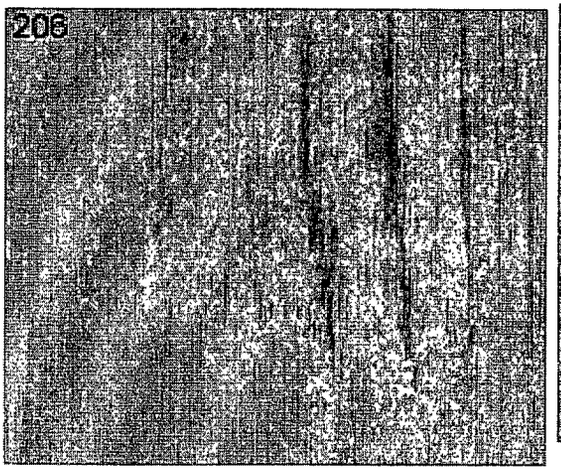
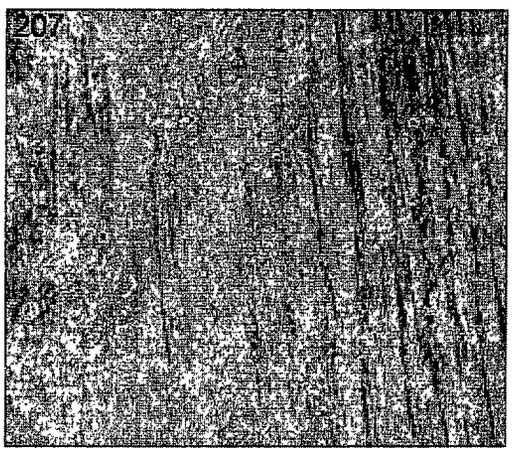


photograph III, G, H, K:

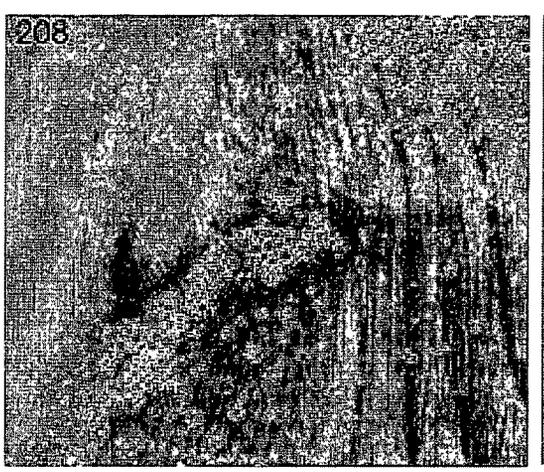
Photograph III



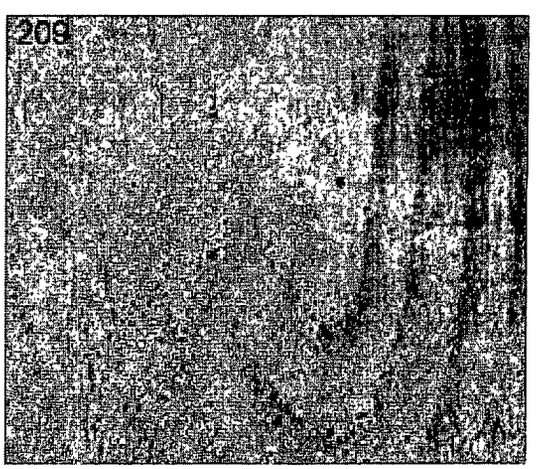
photograph G



photograph H



photograph K



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EUROPEAN SEARCH REPORT

Application Number
EP 01 20 1888

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
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