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3,669,707

FIXING PROCESS

Filed Oct. 17, 1969

FIG. 1

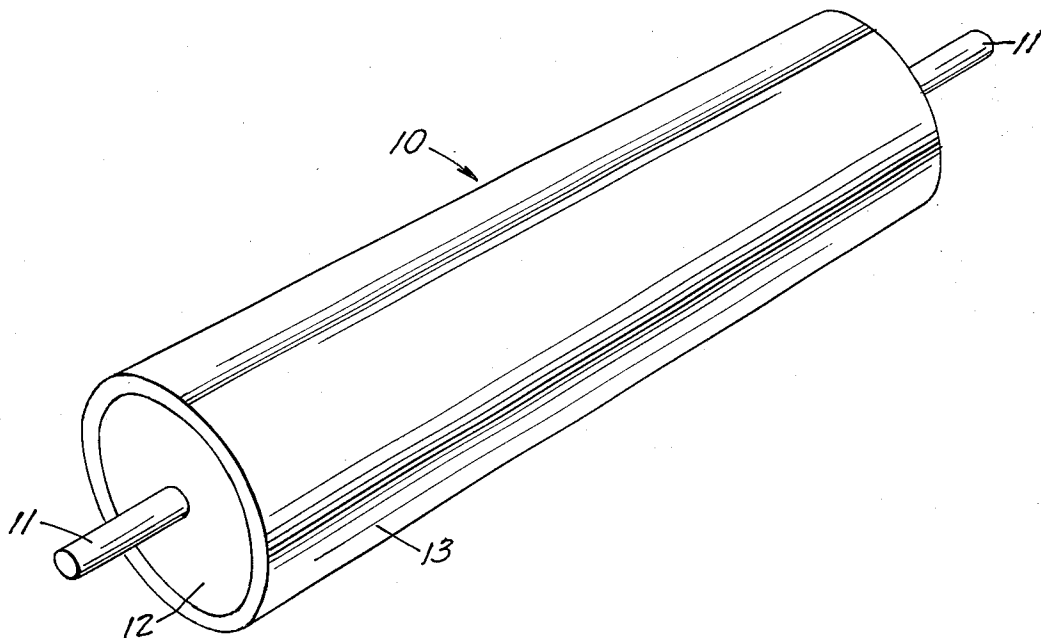
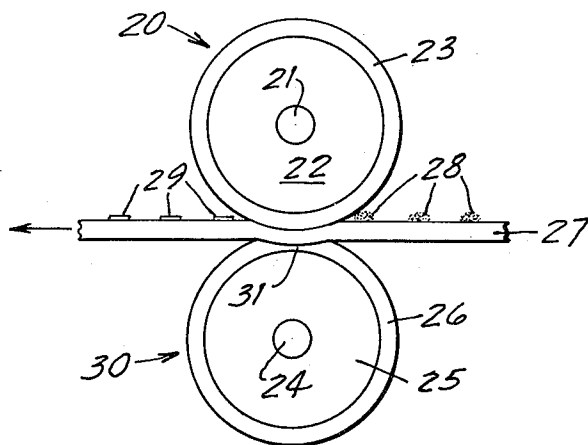


FIG. 2



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FIXING PROCESS

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6 Claims

ABSTRACT OF THE DISCLOSURE

Particulate thermoplastic toner is fixed on a receptor surface by directly contacting the toner with a silicone elastomer surface while the toner is in a fused state responsive to the adhesive nature of the silicone elastomer and to the adhesive nature of the receptor to provide for the substantially complete retention of the toner on the receptor surface in a fixed condition. Preferably, the silicone elastomer is free of high surface energy fillers and, most preferably, is both free of high surface energy fillers and contains low surface energy fillers, such as fluorinated organic polymer materials having a surface energy not greater than about 50 dynes/cm., blended therein.

This invention relates to the field of duplication; more particularly, it relates to the fixing of particulate thermoplastic toner by direct contact with a silicone elastomer surface while the toner is in a particulate non-solid or fused state.

The process of this invention is particularly useful in fixing of resinous powder images produced by electrophotography onto receptor sheets such as sheets of paper. This powder image can be created through a variety of commercially known methods and its creation is not a concern of this invention. In general, the powders or toners for which this invention is directed are heat softenable, such as is provided by toners which contain thermoplastic resins.

Previous fixing techniques for heat-softenable toner powders include heating by radiant means such as by coiled wires and heat lamps, exemplary procedures being described in U.S. Pat. Nos. 3,432,639; 3,448,970; 3,449,546 and 3,452,181. These methods have proven inefficient because of the necessity of heating the sheet, usually paper, to a temperature near charring for prolonged periods which in some cases causes the paper to burn. This has led to the development of a fixing technique utilizing a heated surface which directly contacts the heat softenable resinous toner. Generally this is accomplished by means of a pair of nip rolls, one a fuser roll having a peripheral surface which has a low affinity for melted or softened toner, referred to in the art as adhesive properties, and a pressure or backup roll usually having a resilient cover. The fuser roll and/or the pressure roll may be heated internally to provide heat to soften the thermoplastic toner. A sheet, usually paper, bearing a thermoplastic powder image is passed through the nip or contact area of the two above-mentioned rolls to fix the powder image. Problems are encountered with the surface materials employed, the foremost being that as the toner is softened to become sufficiently sticky to adhere to the sheet some of the particles may stick to the surface of the fuser roll. This causes a splitting of the image and results in a partial or ghost image on the next sheet, producing what is commonly referred to in the duplicating art as an offset image.

The offset image problem has restricted the composition of the toner contacting surface to certain materials having very high surface release or adhesive characteristics. This has led to complex fixing systems such as those

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using nip rolls which may be coated with a tetrafluoroethylene resin such as Teflon and a system for dispensing a silicone oil onto the heated roll. In this cumbersome arrangement, disclosed in U.S. Pat. Nos. 3,291,466; 3,331,592; 3,449,548; and 3,452,181, problems are encountered in the liquid dispensing systems when the liquid supply is exhausted or is dispensed unevenly over the surface of the heated roll, necessitating machine down time to fill the dispenser or to replace the applicator before efficient operation can again be realized. Since the toner contacting surface of this invention operates without the need for such liquids, the attending problems are eliminated.

It is therefore an object of this invention to provide a process and article for rapidly fixing toner images without causing image splitting or offset images.

This and other objects of the invention are attained in one embodiment by means of a process for fixing thermoplastic toner comprising contacting a receptor surface bearing thermoplastic toner with a silicone elastomer surface for a time and at a temperature sufficient to permit said contacted toner to exist in a state wherein the cohesive integrity exceeds the force of adhesion exerted thereon by said silicone elastomer, and wherein the force of adhesion between said thermoplastic toner and said receptor surface exceeds the force of adhesion between said thermoplastic toner and said silicone elastomer, and separating said receptor surface and said silicone elastomer surface while said thermoplastic toner is in said state whereby said thermoplastic toner is substantially completely retained in fixed position upon said receptor surface.

FIG. 1 illustrates one embodiment of a silicone elastomer surface attached to a roll.

FIG. 2 is a sectional diagrammatic illustration of nip rolls suitable in the practice of this invention.

With reference to FIG. 1, roll 10 is shown with journals 11 for mounting and a silicone elastomer blanket 13 disposed upon the cylindrical enlargement 12 which may contain means for providing heat up to 400° F. to the blanket 13.

FIG. 2 shows a pair of nip rolls 20 and 30 in pressing relationship creating a nip 31 through which is passing a powdered image bearing sheet 27. To prevent toner offset onto roll 20 its peripheral surface is provided with a blanket 23 of silicone elastomer.

Rolls 20 and 30 are conventionally cooperatively rotated to draw the sheet 27 therebetween. When this combination is used as a toner fixing or fusing device, either roll 20, roll 30, or both are heated or heat may be provided by an external device prior to the arrival of the sheet 27 into the nip 31. As the sheet 27, bearing a powdered image 28 is drawn into the heated nip 31, the powdered image 28 is fixed to provide a permanent image 29. Roll 20 conventionally is called a fuser roll in a fixing device wherein the roll must make direct contact with the toner image, and, therefore, must be capable of fixing the toner without retaining fused toner which could cause an offset image on the following sheet.

Roll 30, the backup or pressure roll, may have a silicone blanket 26 disposed upon the core 25 in certain situations; however, in operational situations where this roll does not contact the toner it is not required.

The fixing roll 20 is operated at a surface speed of from 2 to 30 inches per second and at a temperature sufficient to cause the toner to exist in a fused, non-transfer state. Generally, the temperature ranges between 220° F. and 400° F. depending upon surface speed and physical characteristics of the toner. The time-temperature relationship of the toner in the heated nip 31 is controlling in bringing the toner to the desired state as hereinafter explained and

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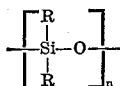
thereafter fixing it upon the receptor 27 with no retention upon the silicone elastomer surface 23. Toner residence time in the heated nip can be controlled in a variety of ways, e.g., by adjusting roll speed or nip width, the latter by varying nip pressure or deformability of nip materials. These factors as well as the temperature are varied to provide the proper time-temperature relationship to achieve the desired state of the fused toner for non-transfer of the toner, in whole or in part, during the fixing step.

The fusion of the thermoplastic toner is accomplished according to this invention by utilization of forces and properties inherent in the material involved in the process, i.e., silicone elastomer, thermoplastic toner, and various receptor surfaces upon which the toner is fixed. When the thermoplastic toner is contacted with the heated silicone surface certain conditions must prevail to achieve the desired results of fixing the toner to the receptor surface. The thermoplastic toner must achieve a gross physical state (commonly referred to as a rubbery or compliant state) wherein the toner has a cohesive integrity greater than the force of adhesion exerted on it by the silicone elastomer surface and the force of adhesion between the silicone elastomer surface and the toner must be less than the force of adhesion between the receptor and the fused toner. The term "gross" as used herein refers to the entire mass of thermoplastic toner, as opposed to individual particles, for example.

The silicone elastomers required in the practice of this invention are formed from the cure or further polymerization of silicone gums. Silicone elastomers have an adhesive quality which can be quantitatively described in terms of release value. Release values are determined on an "Instron," Model TM operating at a crosshead speed of 12 inches per minute and chart speed of 2 inches per minute. One-inch Johnson and Johnson "Red Cross" brand waterproof adhesive tape is used, selecting only a roll having a retention force of about 450 grams (425-475) as measured at 80° F. on a 24 gauge, No. 4 finish stainless steel test panel. In determining either the retention force of the tape to be used or the release value of a sample, a ten-inch strip of tape is applied to a 6-inch by 1½ inch panel by passing a 4½ pound rubber-faced tape roller twice over the tape, using only the weight of the roller. The sample is immediately placed in the Instron and the force in grams necessary to strip the tape at an angle of 180° is determined. The amount of force required to strip the tape is referred to as the "release value," and the larger the release value, the more adhesion there has been between the adhesive tape and the surface.

A small release value indicates an effective release coating and a large release value indicates an ineffective release coating. Standard tests for release value are described in TAPPI (Technical Association for the Pulp and Paper Industry), vol. 43, No. 8, p. 164A (August 1960) and TAPPI Routine Control Method "RC-283 Quality of Release Coatings," issued 1960. Many silicone elastomer surfaces have been found to have a release value of only 1 gm./in., and none greater than 30 gm./in. Such materials have been found satisfactory in the process of this invention. Materials which have a release value greater than 100 gm./in. will not provide an adequate toner contacting surface because when contacted and fused, part of the toner will transfer from the receptor sheet and thus split the image being fixed. The next sheet to be fused would be exposed to that toner retained on the elastomer surface and an offset image would result.

Depending on the curing mechanism to be used, specific silicone gums are prepared, all having the central, repeating linear unit



when n may be as small as 2 or as large as 20,000 or more, and where all R's in the chain may be the same, but need

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not be, each individual R being monovalent alkyl or aryl group, halogenated alkyl or aryl group or cyano alkyl group, with not more than a few percent of total R being vinyl, phenyl or halogenated vinyl or phenyl, the major portion of R usually being methyl groups. Where milling is employed to incorporate the low surface energy filler into the silicone elastomer, n is a number such that the gum is of a millable molecular weight. Dimethyl polysiloxanes containing 1 to 4 mol percent of vinyl substituents on the main chain are preferred. Due to compatibility with Teflon, another preferred gum is one having methyl and perfluoroalkyl R group.

Silicone elastomers, formed by further polymerizing the gums just referred to, can be characterized generally as the very sparsely cross-linked (cured) dimethyl polysiloxane of high molecular weight, e.g., 100,000-1,000,000 average molecular weight. The sparsity of cross-linking is indicated by R/Si ratios very close to 2, generally above 1.95, or even above 1.99, and generally below 2.1 or even below 2.01, there usually being 200-500 dimethyl units between cross-link sites. In contrast, the much more densely cross-linked silicone resins which are considered commercially useful fall in the range of R/Si ratios of 1.2-1.5.

Exemplary millable silicone gums useful in the practice of this invention are dimethyl polysiloxane sold under the tradename Silastic 400, polymerized vinyl dimethyl polysiloxane, sold under the tradename Silastic 430, polymerized vinyl and phenol polysiloxane, sold under the tradename Silastic 440, polymerized trifluoropropyl and vinyl dimethyl polysiloxane, sold under the tradename Silastic LS 420 and the like. The preferred gum is polymerized vinyl dimethyl polysiloxane sold under the trade name Silastic 430, but others are equally useful.

Other silicone elastomers can be used in the practice of this invention. Exemplary of these are moisture curing silicone gums such as the acetoxy terminated silicone gums and room temperature vulcanizable silicones such as those cured using catalysts including dibutyl tin dilaurate, tin octoate and lead octoate.

The type of fillers which may be compounded with the silicone elastomers is a significant consideration in the present invention. Fuser copy life or the number of copies fixed before failure of the fuser blanket is a controlling factor in the choice of a material for a fuser blanket. Fusers which are capable of fixing many thousands of copies are desired in the field of duplication. Fusers which fix over one hundred thousand copies are preferred. Conventional compositions of silicone elastomers formulated with high surface energy fillers such as silica, titanium oxide, and iron oxide have a very short copy life. For example, in a silicone elastomer fuser blanket having 20 weight percent silica (a high surface energy filler) only 1000 copies are fixed before objectionable offset occurs and the blanket becomes useless. Silicone elastomers having substantially no reinforcing fillers (at least less than 1% by weight of the silicone elastomer) used under the same conditions will fix as many as 35,000 copies before failure occurs from mechanical breakdown of the elastomer. It has been discovered that the addition of low surface energy reinforcing fillers which resist thermal degradation at the fixing temperature (e.g. 220°-400° F.) drastically improve the copy life of silicone elastomer fusing blankets. Silicone elastomer fusing blankets having low surface energy fillers blended therein, such as Teflon, under similar operating conditions have been used to fix in excess of 100,000 copies with no offset or mechanical breakdown. A surprisingly long copy life is thereby obtained through the addition of low surface energy fillers.

In general, low surface energy materials are the organic polymers, which have surface energies of about 50 dynes/cm. or less. The preferred organic polymers are the fluorinated resins having surface energies below 30 dynes/cm. Polytetrafluoroethylene, available under the

trade marks Teflon, is considered to have a surface energy of about 18–20 dynes/cm. The conventional fillers are inorganic materials such as silica, iron oxide, titanium dioxide, etc. which have surface energies above 50 dynes/cm., and generally above 70 dynes/cm. Surface energies of various materials are reported by F. M. Fowkes, "Surfaces and Interfaces I—Chemical and Physical Characteristics," pp. 197–223 (1967). In addition to surface energy requirements, the filler must be able to withstand the fixing temperatures for prolonged periods, generally 220° F. to 400° F. for at least 50 hours and, preferably 100 hours or more. Fluorinated resins, particularly polytetrafluoroethylene, uniquely meet these requirements due to their release abilities, temperature resistance, and reinforcing properties when milled into silicone elastomers.

Silicone elastomer filled with high surface energy fillers initially provides a good fusing blanket. However, as the elastomer contacts the toner and the receptor sheet upon which it is borne, abrasion occurs exposing high surface energy sites within the blanket. Thereafter, fused toner has a tendency to adhere and accumulate at the exposed high surface energy sites resulting in an offset image problem. This can be temporarily remedied by cleaning the toner accumulation from the elastomer surface but cannot be completely eliminated. The exposure of high surface energy sites in silicone elastomers filled with high surface energy fillers is totally unexpected in the silicone elastomer formulation art. It has been thought that high surface energy fillers would be completely wet by the silicone elastomer and therefore not present this problem.

Silicone elastomers substantially free of high surface energy fillers provide fuser blankets without the above described offset problem. Silicone elastomers free of fillers provide a fusing blanket capable of use in a conventional copy machine. However, this blanket lacks the physical strength required for prolonged trouble free use which is a prime requisite of a fuser blanket in copying devices. The preferred blanket is provided by blending low surface energy fillers such as fluorocarbon resins, preferably Teflon, into silicone elastomers which are substantially free (less than 1% by weight) of high surface energy fillers. Silicone elastomers having low surface energy fillers blended therein provide fusing blankets capable of prolonged use without the attendant problem of exposure of high surface sites that are seen in silicone elastomers strengthened with high surface energy fillers.

Silicone elastomer roll surfaces can be fabricated by a number of techniques. The desired roll covering should be smooth and of controlled thickness. The preferred elastomer containing low surface energy fillers requires milling to blend the fillers therein and therefore requires millable silicone gums. Milled compositions are easily shaped into blankets by pressing in a suitable die.

A suitable method for making the silicone elastomer fusing blanket involves slowly adding a low surface energy filler to a high viscosity silicone gum in a high shear mixing device to achieve intimate blending of the components. The high shear mixing of the components in the case of Teflon appears to cause the elongation of particles of Teflon into threads or filaments thereby providing a fiber structure within the silicone elastomer. In order to achieve the critical reinforcing effect of the fluorocarbon resins, e.g., Teflon, the resins must be thoroughly intermixed such as by milling into a millable silicone elastomer. The reinforcing properties are not obtained, for example, by simply mixing the fluorocarbon resin with a silicone gum without milling and then curing the mixture.

In the preparation, conventional silicone gum curing agents are added to the mixture after high shear mixing of the low surface energy filler and the silicone gum. Exemplary curing agents are benzoyl peroxide, 2,4-dichlorobenzoyl peroxide, tertiary-butyl perbenzoate, dicumyl peroxide and other commercially recognized sili-

cone polymer curing agents. Curing conditions required by these agents vary somewhat depending upon the curing agent and the silicone gum. Generally, heat is required to cure the silicone gums and conventionally temperatures up to 340° F. are used. Post-curing of the silicone elastomer composition may be required if it is to be used at high temperatures. Post-curing is accomplished by heating the elastomer in a forced air oven from 2 to 4 hours or more at temperature up to 450° F.

The low surface energy fillers preferably are fluorinated or partially fluorinated organic resins. Exemplary of these are polytetrafluoroethylene sold under the trade name Teflon, the copolymer of vinylidene fluoride and hexafluoropropene sold under the trade name Fluorel, and the terpolymer of vinylidene fluoride, hexafluoropropene and tetrafluoroethylene, sold under the trade name Viton B.

The amount of low surface energy filler employed may vary over a wide range, generally from about 0.1 to about 20.0 weight percent of the total, preferably from about 0.5 to 5.0 weight percent, and most preferably about 2.0 weight percent.

Generally, as the content of the low surface energy filler is increased, the hardness of the composition increases. It is desirable that there be intimate contact between the silicone elastomer fixing surface and the substrate bearing toner powder. Thus, fixing surfaces having a Shore A durometer hardness of less than 80 are desirable. Hardnesses greater than 80 Shore A durometer of the composition of a fuser-blanket generally require very high nip pressures to get satisfactory fixing results; therefore, the content of low surface energy filler in the preferred composition is limited by this factor.

A fusing blanket is prepared by placing the silicone elastomer which has the low surface energy filler and a curing agent blended therein on a polished die which may be coated with a release assisting substance. The die is fitted with shims to provide a blanket of a certain thickness. The loaded die is pressed in a platen press heated sufficiently to cause the cure of the elastomer. The resultant sheet is adhesively or mechanically attached to a fuser roll core to provide a completed fuser roll. Likewise, the composition can be pressed in a similar die which has a sheet such as a stainless steel sheet covering the bottom of the die. The sheet preferably is coated with an adhesive. The resultant composition bonded to the steel sheet can be mechanically or adhesively bonded to the roll core.

To attain fixing without offsetting in whole or in part of thermoplastic toner from the receptor surface to the silicone elastomer requires that the cohesive integrity of the toner powder be greater than the force of adhesion exerted thereon by the silicone elastomer and further that the receptor have a greater force of adhesion for the toner in the state suitable for fixing than the silicone elastomer. Exemplary receptor surfaces are paper, clay, ceramic, glass, plastic, and metals, e.g., aluminum or stainless steel etc. In general, to accomplish fixing without offsetting requires that the thermoplastic toner be in a rubbery, compliant state which may be defined in terms of viscoelastic properties as the state wherein the fused thermoplastic toner has a creep modulus (defined as

$$G_{(t)} = \frac{1}{J_{(t)}}$$

where $J_{(t)}$ is creep compliance) in the range of between about 10^8 dynes/cm.² and about 10^4 dynes/cm.². Under process operating conditions, the creep modulus of fused thermoplastic toner is dependent on two parameters—the temperature of the fused thermoplastic toner and the time in which two surfaces are in contact with each other with the fused thermoplastic toner between the contacting surfaces.

For a given temperature, the range of contact time over which the fused toner exhibits a modulus within the 10^4 to 10^8 dynes/cm.² range (generally in what is known as the rubbery region) depends on the material and on its molecular weight. For high molecular weight amorphous polymers the rubbery region is called the entanglement plateau and it may extend over several decades of reduced time. Some semi-crystalline materials, low molecular weight polymers, and other organic compounds may not exhibit a plateau and thus may be in the rubbery region for a relatively short period of reduced time. Similarly, for a given contact time, such fused toner exhibits rubbery response characteristics (generally a modulus of 10^4 to 10^8 dynes/cm.²) over a relatively narrow temperature range.

Suitable toners for the above described process are compositions containing thermoplastic materials such as those which at temperatures below 80° C. maintain a dry particulate state but yet achieve a rubbery or compliant state with a creep modulus from 10^4 to 10^8 dynes per cm.² when heated. Exemplary thermoplastics useful in compounding toner powders are epoxy resins such as that sold under the trade name Epon 1004, polystyrene resins sold under the trade name Piccolastic D 125 and D 150. An exemplary toner powder is the following wherein all percentages are by weight and the average particle size is 7 microns:

	Percent
Epon 1004	44
Magnetite	52
Carbon black	4

The powder is made by spray drying this formulation from a solvent such as chloroform. Another suitable toner powder for the above described process consists of 65% polystyrene and 35% carbon black.

Thermoplastic toner can be prepared by several techniques; for example, by spray drying an organic solution or emulsion of the developer material or by an extrusion-grinding process. Particles can be classified into the desired size range. The particle size range of the transfer medium generally ranges from 0.5 to 50 microns, preferably between about 2 and about 15 microns for most applications. However, specific applications may demand lower or higher size ranges. Generally, spherical particles are preferred. The powders preferably have a flowability angle of repose ranging from 80 to 125 degrees. Flowability is measured by feeding a thin stream of powder to the upper flat surface of a circular pedestal from a vibrating funnel, thereby creating a conical deposit of powder on the pedestal. The angle of repose is defined by the angle between the side of the cone and the pedestal at 25° C.

To better illustrate the invention, the following non-limiting examples are provided wherein all parts and percentages are by weight unless otherwise stated.

EXAMPLE 1

A roll was prepared by banding 980 grams of filler free silicone gum sold under the trade name Silastic 430 on a rubber mill having a 13 inch roll. Twenty grams of powdered polytetrafluoroethylene sold under the trade name Teflon powder grade 6 (surface energy 19-20 dynes/cm.) was slowly added to the banded silicone gum. The milling was continued in this matter for about 15 minutes to blend the components. Thereafter the mixture was transferred to a Banbury high shear mixer and therein worked for 15 minutes at a temperature of 250-290° F. This mixture was then allowed to cool and returned to the rubber mill. Fifteen grams of benzoyl peroxide in an equal weight of silicone gum paste was blended into the mixture on the rubber mill. After 15 minutes of blending, the mixture was placed on a 5 mil by 10 inch by 18 inch stainless steel sheet which had previously been primed with Dow Corning silicone rubber primer 2260 to promote adhesion of the silicone elastomer to the

sheet. The sheet was shimmed to give a final blanket thickness of 0.020 inch and pressed in a platen press at a pressure of 100 tons for 10 minutes at 260° F. The blanket was then post cured for four hours at 400° F. in a forced air oven and after cooling, adhesively bonded to the 6 inch diameter by 12 inch long fuser roll of an experimental copy machine. The roll had an internal heating element which maintained the blanket surface temperature at 325° F.

Powdered images were deposited on bond paper according to the electrographic process described in French Pat. No. 1,456,993 with a toner consisting of 60 percent magnetite and 40 percent epoxy resin (trade name Epon 1004) which had been spheroidized with carbon on the outside. Thereafter, each sheet was passed in turn through the nip created by the above described fuser roll which was in contact with a pressure roll at a nip load pressure of 25 to 50 p.s.i. which created a nip width of 1/2 inch with the pressure roll. The fuser roll was rotated counterclockwise at a surface speed of 16 inches per second and thus was used to successfully fix in excess of 100,000 powdered image copies.

EXAMPLES 2-4

Fusing blankets were prepared and evaluated according to the methods described in Example 1 using silicone elastomer Silastic 430 with variations only in the content of fillers. Both high surface energy and low surface energy filler containing compositions were prepared and the results of their evaluation are tabulated below with those of Example 1.

Example No.	Weight percent filler surface energy		Copies made before failure	Reason for failure
	High ^a	Low ^b		
1	0	2.0	100,000	No failure—end test.
2	0	0	36,000	Rubber failure.
3	24.7	0	1,000	Objectionable offset.
4	20.0	1.95	15,000	Do.

^a Silica.
^b Teflon.

What is claimed is:

1. A process for fixing particulate thermoplastic toner to a receptor surface at image defining areas comprising:

- (a) contacting a receptor surface bearing image defining areas of particulate thermoplastic toner with the surface of a silicone elastomer layer for a time and at a temperature sufficient to permit heating and fusion of said contacted fused toner to said receptor, said silicone elastomer containing fluorinated organic polymer filler having a surface energy not greater than about 50 dynes/cm. and being substantially free of high surface energy filler, said fused toner having adhesion for said receptor greater than its adhesion to said silicone elastomer surface and less than the cohesion of said fused toner; and
- (b) separating said receptor from said silicone elastomer surface while said thermoplastic toner is in said fused state whereby said thermoplastic toner is substantially completely retainer at said image defining areas on said receptor surface.

2. The process of claim 1 wherein said receptor surface is paper.

3. The process of claim 1 wherein said silicone elastomer low surface energy filler is polytetrafluoroethylene.

4. The process of claim 1 wherein said silicone elastomer has from 0.1 to 20.0 weight percent polytetrafluoroethylene blended therein.

5. The process of claim 1 wherein said silicone elastomer has from 0.5 to 5.0 weight percent polytetrafluoroethylene blended therein.

6. The process of claim 1 wherein said thermoplastic toner assumes a modulus within the range of about 10^4 to about 10^8 dynes/cm.² in said fused state.

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