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## REFLECTIVE ELECTROACTIVE PARTICLES

FIELD OF THE INVENTION

This invention relates to electroactive particles capable to reflect UV light, infrared  
5 radiation, microwave, and/or radio frequency and, more specifically, to reflective electroactive  
particles for preparing adaptive camouflage structures that may be dynamically adjusted to  
disguise the appearance of the object so that the object appears like its immediately surrounding  
environment to detectors utilizing electromagnetic radiations.

BACKGROUND OF THE INVENTION

The survivability of an object, such as military personnel, vehicles, equipments, and  
structures, in a battlefield environment is directly linked to the ability of countermeasures to  
defeat all identified or unidentified threats to the object. A key countermeasure against the  
threats to the object is to camouflage it so that its presence is difficult for an enemy to detect.  
15 Traditionally, indigenous paint camouflages have been employed to reduce visible detection of  
objects by detectors utilizing visible light. Modern warfare environments employ many other  
proven detection methods such as radar and infrared emission detectors that are not defeated by  
the current paint camouflage countermeasures. Furthermore, the time required to change the  
indigenous paint camouflage frequently for various battlefield environments prevents or delays  
20 the deployment of military assets as required by a given combat situation.

To reduce an enemy's ability to detect a military object, it is desirable that the camouflage  
(1) can be incorporated into a military object without impeding the military object to perform its  
mission or intended use; (2) can reduce the detection of the military object by a wide range of  
detectors; (3) can dynamically adapt to widely varying battlefield environments (such as night,  
25 day, or rain), movements, and multiple geographic locations and environments within a given  
battle theater; (4) can be rapidly reconfigured or changed to various patterns to suit widely  
varying geographic environments; and (5) can be deployed at acceptable cost. Improved  
camouflage can provide some of the above desirable features and significant tactical advantages  
in use.

SUMMARY OF THE INVENTION

Adaptive camouflage structures are disclosed herein that may be dynamically adjusted to  
disguise the appearance of an object, such as military personnel, vehicles, equipments, and  
structures, so that the object appears like its immediately surrounding environment to detectors

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utilizing visible light, UV light, infrared radiation, microwave, and/or radio frequency. The adaptive camouflage structures of this invention may provide a wide viewing angle and may easily be incorporated as an integral part of the object. One feature of the adaptive camouflage structures of this invention is that it may incorporate multiple incident radiation schemes into one device, thereby reducing cost, weight and installation complexity. Another feature of the adaptive camouflage structures of this invention is that the camouflage patterns created on the reflective layers of the camouflage structures are stable and may be maintained without power. A further feature of the adaptive camouflage structures of this invention is that the camouflage patterns may be retained for a long period of time until they are changed or reconfigured by a new electric field pattern.

In a first aspect, this invention features an electroactive microcapsule comprising a shell and a mixture inside the shell, wherein the mixture comprising an oil; a plurality of metal particles; and a plurality of electrically non-conducting particles and wherein the plurality of metal particles and the plurality of electrically non-conducting particles have opposite charges.

In a second aspect, this invention features an electroactive particle comprising an electrically non-conducting particle and a coating of a reflective material, wherein the coating covers a portion of the electrically non-conducting particle and wherein the reflective material is selected from the group consisting of infrared reflective materials, radar reflective materials, microwave reflective materials, UV reflective materials, and combinations thereof.

In a third aspect, this invention features an electroactive microcapsule comprising a shell and a mixture inside the shell, wherein the mixture comprising an oil and a plurality of flakes comprising of a polymer and wherein a layer of metal is coated on one of the flat surface of each flake.

In some embodiments of interest, about 25% to 75% of each of the electroactive particles in the first reflective layer reflects visible light and the remaining 75% to 25% of the particle does not reflect visible light. In other embodiments of interest, about 25% to 75% of each of the electroactive particles in the second reflective layer reflects infrared radiation, radio frequency, microwave, UV light, or a combination thereof and the remaining 75% to 25% of the particle does not reflect infrared radiation, radio frequency, microwave, UV light, or a combination thereof. In further embodiments of interest, about 25% to 75% of each of the electroactive particles in the third reflective layer reflects infrared radiation, radio frequency, microwave, UV light, or a combination thereof and the remaining 75% to 25% of the particle does not reflect infrared radiation, radio frequency, microwave, UV light, or a combination thereof.

Other features and advantages of the invention will be apparent from the following description of the particular embodiments thereof, and from the claims.

#### BRIEF DESCRIPTION OF THE DRAWINGS

5       The foregoing and other features and advantages of the invention are apparent from the following more particular description of specific embodiments of the invention, as illustrated in the accompanying drawings in which like reference numbers refer to the same parts throughout the different views. The drawings are not necessarily to scale, emphasis instead being placed upon illustrating the principles of the invention.

10       FIG. 1 is a schematic sectional view of an embodiment of an adaptive camouflage structure comprising two reflective layers and two adjustable voltage pattern interlayers, where the top reflective layer reflects visible light and the bottom reflective layer reflects infrared radiation.

15       FIG. 2 is a schematic sectional view of an embodiment of an adaptive camouflage structure comprising three reflective layers and three adjustable voltage pattern interlayers, where the top reflective layer reflects visible light, the middle reflective layer reflects infrared radiation, and the bottom reflective layer reflects radio frequency.

20       FIG. 3 is a schematic sectional view of an embodiment of a spinning disk for making electroactive bichromal beads from two different hardenable liquid materials in which the cross section is taken through the axis of the disk.

      FIG. 4 is a schematic side view of an embodiment of a double barreled nozzle for making electroactive bichromal beads from two different hardenable liquid materials having opposite charges.

25       FIG. 5 is a schematic perspective view of an embodiment of a spinning disk for making electroactive bichromal beads from two different hardenable liquid materials having opposite charges.

      FIG. 6 is a schematic side view of an embodiment of an assembly of three spinning disks for making electroactive polychromal segmented beads from different hardenable liquid materials.

30       FIG. 7 is a schematic sectional view of an embodiment of a spinning disk and a coating device for making electroactive beads in which the cross section is taken through the axis of the disk.

      FIG. 8 is a schematic side view of an embodiment of a double barreled nozzle and a coating device for making electroactive beads.

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FIG. 9 is a schematic sectional view of an embodiment of an adaptive camouflage structure comprising three reflective layers and one adjustable voltage pattern interlayer, where the top reflective layer reflects visible light, the middle reflective layer reflects infrared radiation, and the bottom reflective layer reflects radio frequency.

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#### DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

Improved adaptive camouflage structures described herein may have 2 or more layers, each of which comprises a plurality of electroactive particles that can be selectively oriented to provide a desired image. The images provided by different layers can be appropriated for different regions of the electromagnetic spectrum, such as visible light, infrared radiation, ultraviolet light, microwave, and/or radio frequency. In some embodiments, the images in the different layers are individually selectable through the application of appropriate electric fields by adjustable voltage pattern interlayers. The electroactive particles may be embedded in a suitable binder that is transparent to at least a region of the electromagnetic spectrum reflected by the electroactive particles and allows for the rotation of the electroactive particles within the layer. When oriented, the electroactive particles in each layer together present a selected image to an observer or a detector.

FIG. 1 depicts an embodiment of a two-layered adaptive camouflage structure of this invention for disguising an object 50, such as military personnel, vehicles, equipments, and structures. The adaptive camouflage structure of FIG. 1 comprises a first reflective layer 3 having a first host material and a second reflective layer 13 having a second host material, wherein the first and second reflective layers further comprise respectively a plurality of electroactive particles 60 and 61 rotatably embedded or dispersed in the respective host material. The electroactive particles 60 and 61 may be, exactly or generally, in the form of sphere, bead, oval, cylinder, disk, flake, or a combination thereof. The electroactive particles in the first reflective layer may reflect, for example, visible light 1 from the surrounding environment of the object and the electroactive particles in the second reflective layer may reflect infrared radiation 11 from the surrounding environment of the object. The first host material is transparent to both visible light and infrared radiation and the second host material is transparent to infrared radiation. A material is transparent to a particular region of the electromagnetic spectrum if it allows at least a portion of the region of the electromagnetic spectrum to pass through the material. In some embodiments, the electroactive particles 60 and 61 are, exactly or generally, in the form of sphere, bead, oval, cylinder, or a combination thereof, and the first host material and the second host material are each independently a polymeric material selected from the

group consisting of polycarbonates, polyesters, polyamides (e.g., nylons), polysiloxanes (e.g., silicones and cross-linked silicones), polyethylene, and polypropylene. In some embodiments, the electroactive particles 60 and 61 comprise flakes dispersed in a host fluid contained in cavities in the respective host material. Suitable host fluid for dispersing electroactive flakes  
5 may have a volume resistivity equal or greater than  $10^9$  ohm-cm, as measured by ASTM D 257-93. Some non-limiting examples of suitable host fluid include tetrafluorodibromoethane, tetrachloroethylene, trifluorochloroethylene, silicone oils, fluorinated oils, fluorosilicone oils, nematic liquid crystal fluids, hydrocarbons (e.g., paraffin liquids, toluene, xylene, octane, decane, tetradecane, decaline, and kerosene), oils (e.g., linseed oil, soya oil, tung oil, and olive oil), and  
10 combinations thereof. The first host material may or may not be the same as the second host material. The electroactive flakes and the host fluid are disclosed in U.S. Patent No. 6,665,042, which is incorporated herein by reference. The reflected visible light 2 may constitute a selected visible image suitable for disguising the object 50 to reduce its risk of being detected by a visible light detector, such as human eyes, telescopes, and electronic cameras. Similarly, the reflected  
15 infrared radiation 12 may constitute a selected infrared image suitable for disguising the object 50 to reduce its risk of being detected by an infrared imaging system, such as FLIRs (forward looking infrared) operating in the wavelengths from 1 to 20 microns.

A person skill in the art may modify the first reflective layer 3 so that it may reflect another radiation other than visible light, such as UV light, infrared radiation, microwave, or  
20 radio frequency. Similarly, a person skill in the art may modify the second reflective layer 13 so that it may reflect another radiation other than infrared radiation, such as visible light, UV light, microwave, or radio frequency. In some embodiment of interests, the second reflective layer 13 contains electroactive particles that can reflect UV light, infrared radiation, microwave, radio frequency, or a combination thereof. Consequently, such reflective layer may be selectively  
25 used as an infrared reflective layer, a radar reflective layer, a UV reflective layer, or a microwave reflective layer.

Optionally, an adhesive layer may be applied on the outer surface of the second reflective layer 13 for adhesively mounting the two-layered adaptive camouflage structure on the surface of an object. Any suitable adhesive materials known in the art may be utilized for this purpose.  
30 Non-limiting examples of suitable adhesive materials include phenol-formaldehyde resins, polychloroprene, urea-formaldehyde adhesive, nitrile rubber-phenolic adhesives, epoxy resin adhesives, nitrile rubber-epoxy film adhesives, nylon-epoxy film adhesives, isocyanate based adhesives, hot melt adhesives, cyanoacrylate adhesives, anaerobic adhesives, silicone adhesives, high temperature resistant adhesives, hypalon toughened acrylate adhesives, bismaleimide-based

adhesives, and acrylated-based or methacrylated-based adhesives. The number average molecular weight of the polymeric adhesive materials may vary from 1000 to 10,000,000 daltons. Alternatively, the two-layered adaptive camouflage structure may be mounted on the surface of the object by any conventional mechanical fasteners, such as screws, nails, bolts and nuts, hook-and-loop type fasteners, and hook-and-hook type fasteners.

Both the visible and infrared images may be controlled by applying independently a suitable voltage pattern to each of the visible and infrared reflective layers 3 and 13 by adjustable voltage pattern interlayers 40. Each of the adjustable voltage pattern interlayers 40 may be placed over the surface 30 of the object 50 and/or at the interfaces between two adjacent layers.

The adjustable voltage pattern interlayers may form from conductive materials that are effectively transparent to visible light. Non-limiting examples of transparent conductive materials include transparent conducting oxides, such as indium tin oxide, zinc oxide, tin oxide, indium oxide, cadmium oxide, gallium oxide, copper oxide, and combinations thereof; thin metallic coating, such as gold, silver, and copper, having a thickness in the range of 0.1 micron to 0.5 nanometer; nano-particles of conducting materials such as carbon nanotubes and gold-coated silver nano-particles; transparent conductive films or stripes formed from transparent conductive ITO inks using ITO micro-particles, translucent conductive pastes using acicular (needle-like) ITO particles; transparent conductive ITO pastes using organic indium and tin compounds; transparent conductive Au-Ag inks using gold-coated silver nano-particles; and transparent conductive carbon inks using carbon micro-particles. All the transparent conductive inks and pastes are available from Sumitomo Metal Mining, Tokyo, Japan.

FIG. 2 depicts an embodiment of a three-layered adaptive camouflage structure for disguising an object 50. The adaptive camouflage structure of FIG. 2 comprises a first reflective layer 3 having a first host material, a second reflective layer 13 having a second host material, and a third reflective layer 23 having a third host material, wherein the first, second, and third reflective layers further comprise respectively a plurality of electroactive particles 60, 61, and 62 rotatably embedded or dispersed in the respective host materials. Optionally, an adhesive layer may be applied on the outer surface of the third reflective layer for adhesively mounting the three-layered adaptive camouflage structure on the surface of an object. Any suitable adhesive materials may be utilized for this purpose. Alternatively, the three-layered adaptive camouflage structure may be mounted on the surface of the object by any conventional mechanical fasteners, such as screws, nails, bolts and nuts, hook-and-loop type fasteners, and hook-and-hook type fasteners.

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In some embodiments, the electroactive particles in the first reflective layer may reflect visible light 1 from the surrounding environment of the object, the electroactive particles in the second reflective layer may reflect infrared radiation 11 from the surrounding environment of the object, and the electroactive particles in the third reflective layer may reflect radio frequency 21 from the surrounding environment of the object. In further embodiments, the first host material is transparent to visible light, infrared radiation, and radio frequency; the second host material is transparent to infrared radiation and radio frequency; and the third host material is transparent to radio frequency. In other embodiments, the electroactive particles 60, 61, and 62 are, exactly or generally, in the form of sphere, bead, oval, cylinder, or a combination thereof, and the first host material, the second host material, and the third host material are each independently a polymeric material selected from the group consisting of polycarbonates, polyesters, polyamides (e.g., nylons), polysiloxanes (e.g., silicones and cross-linked silicones), polyethylene, and polypropylene. In other embodiments, the electroactive particles are in the form of flakes dispersed in a host fluid contained in cavities in the respective host material. The reflected visible light 2 may constitute a selected visible image suitable for disguising the object 50 to reduce its risk of being detected by a visible detector. Similarly, the reflected infrared radiation 12 may constitute a selected infrared image suitable for disguising the object 50 to reduce its risk of being detected by an infrared imaging system. Similarly, the reflected radio frequency 22 may constitute a selected radar image suitable for disguising the object 50 to reduce its risk of being detected by a radar detector. The visible, infrared, and radar images can be controlled by applying independently a suitable voltage pattern to each of the visible, infrared, radar reflective layers 3, 13, and 23 by adjustable voltage pattern interlayers 40. Each of the adjustable voltage pattern interlayers 40 may be placed over the surface 30 of the object 50 and/or at the interfaces between two adjacent layers. A person skill in the art may modify the first reflective layer 3 so that it may reflect another radiation other than visible light, such as UV light, infrared radiation, microwave, radio frequency, or a combination thereof. Similarly, a person skill in the art may modify the second reflective layer 13 so that it may reflect another radiation other than infrared radiation, such as visible light, UV light, microwave, or radio frequency. Similarly, a person skill in the art may modify the third reflective layer 23 so that it may reflect another radiation other than radio frequency, such as visible light, UV light, microwave, or infrared radiation.

The visible light reflective layer 3 may comprise a host material that is sufficient transparent to visible light to allow at least a portion of the visible spectrum to pass through. Optionally, the host material for the visible light reflective layer 3 may be sufficiently transparent to at least another region of the electromagnetic spectrum, such as infrared radiation,

radio frequency, microwave, and UV light. Non-limiting examples of suitable host material for the visible light reflective layer include polycarbonates, polyesters, polyamides (e.g., nylons), polysiloxanes (e.g., silicones and cross-linked silicones), polyethylene, and polypropylene. Embedded within the host material are a plurality of electroactive particles that reflect visible  
5 light. Some non-limiting examples of electroactive particles that reflect visible light include bichromal beads or cylinders, polychromal segmented beads or cylinders, polymer liquid crystal and polymer birefringent flakes, and microencapsulated ink beads.

In some embodiments of interest, the visible light reflective layer may comprise a host material and a plurality of the bichromal beads or cylinders and/or the polychromal segmented  
10 beads or cylinders that are rotatably embedded in the host material. The bichromal beads and the polychromal segmented beads are generally spherical or elliptical particles having a size in the range of about 10  $\mu\text{m}$  to about 1 mm. In other embodiments of interest, the electroactive particles in the visible light reflective layer of the adaptive camouflage structure comprise generally spherical bichromal beads. Each of the bichromal beads has two generally equal  
15 hemispheres of contrasting colors (e.g., black and white, red and white, etc.) to achieve an optical anisotropy. The two colored hemispheres can have two different Zeta potentials due to the different materials used to achieve the optical anisotropy. The difference in Zeta potential causes each of the bichromal beads to have opposite charges so that each exhibits an electrical anisotropy corresponding to the optical anisotropy. When a voltage is applied to a bichromal  
20 bead in the visible light reflective layer, the bichromal bead rotates to present one colored hemisphere to the viewer, depending on the polarity of the applied voltage. When a voltage pattern is applied to all the bichromal beads by an adjustable voltage pattern interlayer 40 adjacent to the visible light reflective layer, the bichromal beads, each independently, rotate to present one color so as to form a selected visible image collectively. The visible image may stay  
25 in place without power for a long time until a new voltage pattern is applied.

The bichromal beads may be prepared by conventional techniques known in the art. For example, a bichromal bead may be prepared by sputtering a light reflective material, such as titanium oxide, on one hemisphere of a bead comprising a black polyethylene. Alternatively, a bichromal bead may be prepared by coating on one hemisphere of a white glass bead of about 10  
30  $\mu\text{m}$  to 1 mm in diameter, with an inorganic coloring material such as indium by evaporation. Alternatively, a bichromal bead may be prepared by coating on one hemisphere of a glass bead heavily loaded with a white pigment (e.g., titanium oxide) in a vacuum evaporation chamber with a layer of nonconductive black material (e.g., graphite, titanium carbonitride, and a combination of magnesium fluoride and aluminum). Bichromal beads may also be prepared by

(a) forming a bichromal fiber by coextruding a semi-circular layer of a polyethylene pigmented white with a semi-circular black layer of polyethylene containing magnetite, (b) chopping the resultant bichromal fiber into fine bichromal particles ranging from 10 microns to about 10 millimeters, (c) mixing the bichromal particles with clay or anti-agglomeration materials, and (d) heating the mixture with a liquid at about 120 °C to spherodize the bichromal particles, followed by cooling to allow for solidification into bichromal beads.

Many other methods, such as those methods known in the art, also may be utilized to prepare the bichromal beads or the polychromal segmented beads. Non-limiting examples of suitable apparatuses to implement suitable methods include the devices depicted in FIGS 3-6.

Referring to FIG. 3, a first colored hardenable liquid material 66 and a second colored hardenable liquid material 67 are applied by delivery means (not shown) to opposite sides 64, 65 of a spinning disk 62, which rotates uniformly about shaft 63. The colored hardenable liquid materials 66, 67 may comprise a molten liquid, such as molten carnauba wax and molten polyethylene, and a color pigment. Centrifugal force causes the first and second colored hardenable liquid materials 66, 67 to flow toward the periphery of disk 62, where they combine at the edge to form bichromal streams 68 that become unstable and eventually break up into bichromal beads 69. When liquids 66, 67 flow with equal rates to the edge of disk 62, the technique produces bichromal beads with equal hemispheres of color. The bichromal beads may solidify by cooling at room temperature or the cooling may be accelerated by having the bichromal beads passing through a cooling zone containing cold liquid nitrogen or CO<sub>2</sub> vapors (not shown). The bichromal beads 69 prepared by this method generally are from about 5 to about 200 microns in diameter, depending on the spinning speed of the spinning disk 62 and the viscoelastic properties of the molten liquid.

Referring to FIGS. 4 and 5, alternative methods are shown for producing bichromal beads 99 and 101 having an implanted dipole moment by means of combining two differently colored hardenable liquid material 93 and 94 in a double barreled nozzle 91 or on a spinning disk 100. The liquid materials 93 and 94 are charged by means of positive electrode 95 and negative electrode 96 and combine by means of electrostatic attraction at the exit of the double barrel nozzle or at the rim of the spinning disk to form respectively bichromal beads 99 and 101, all of which have an implanted dipole moment.

In other embodiments of interest, the electroactive particles in the visible light reflective layer of the adaptive camouflage structure comprise polychromal segmented beads. Referring to FIG. 6, polychromal segmented beads may be prepared by using an assembly of three spinning disks 71. Different colored hardenable liquid materials may be introduced by delivery means

(not shown) to each side of each of the three spinning disks 72, 73, 74 rotating uniformly about a common shaft 75. The colored hardenable liquid materials combine at the edge of the disks to form polychromal segmented streams that become unstable and eventually break up into polychromal segmented beads. Optionally, the colored hardenable liquid materials, each  
5 independently, may be charged by means of a positive electrode and/or a negative electrode.

The preparations and the addressing or orienting of the bichromal beads or cylinders and the polychromal segmented beads or cylinders are disclosed in U.S. Patent Nos. 6,690,350, 6,542,283, 6,524,500, 6,497,942, 6,496,298, 6,492,967, 6,441,946, 6,428,868, 6,421,035, 6,396,621, 6,362,915, 6,335,818, 6,262,707, 6,120,588, 6,055,091, 5,982,346, 5,922,268,  
10 5,919,409, 5,917,646, 5,904,790, 5,894,367, 5,891,479, 5,815,306, 5,808,593, 5,777,782, 5,767,826, 5,760,761, 5,751,268, 5,717,515, 5,717,514, 5,717,283, 5,708,525, 5,344,594, 5,262,098, 5,075,186, 4,143,103, and 4,126,854, all of which are incorporated herein by reference.

In other embodiments of interest, the electroactive particles in visible light reflective  
15 layer of the adaptive camouflage structure comprise microencapsulated ink beads. Each microencapsulated ink bead is a generally spherical microcapsule containing positively charged particles and negatively charged particles suspended in a clear liquid, such as oils, where the positively charged particles and the negatively charged particles can reflect and/or absorb visible light. In some embodiments, both the positively charged particles and the negatively charged  
20 particles are colored. In other embodiments, each microencapsulated ink bead contains positively charged white particles and negatively charged black particles, all of which are suspended in an oil within the bead. Therefore, when a negative electric field is applied to such a microencapsulated ink bead, the negatively charged black particles move to the bottom of the microencapsulated ink bead where they are hidden, whereas the positively charged white  
25 particles move to the top of the microencapsulated ink bead where they can reflect visible white light to an observer. This process can be reversed by applying a positive electric field which causes the negatively charged black particles to move to the top of the microencapsulated ink bead where they absorb visible light. Microencapsulated ink beads may be prepared by many conventional encapsulation techniques, such as those disclosed in U.S. Patent No. 6,067,185,  
30 which is incorporated herein by reference.

Numerous suitable procedures for microencapsulation are detailed in both Microencapsulation, Processes and Applications, (I. E. Vandegaer, ed.), Plenum Press, New York, N.Y. (1974) and Gutcho, Microcapsules and Microencapsulation Techniques, Nuyes Data Corp., Park Ridge, N.J. (1976), both of which are incorporated herein by reference. The

encapsulation processes fall into several general categories: interfacial polymerization, in situ polymerization, physical processes, such as coextrusion and other phase separation processes, in-liquid curing, and simple/complex coacervation, all of which can be applied to form electroactive beads for reflecting visible light or other electromagnetic radiations. In one embodiment of preparing microencapsulated ink beads, an oil phase comprising a mixture of positively charged particles and negatively charged particles, an oil, and sebacoyl chloride (from Aldrich) is dispersed in water with stirring at a high speed, such as 200-500 rpm, and at room temperature to form a dispersion of oil droplets containing the particles and a dicarboxylic acid dichloride. Then microencapsulated ink beads are formed by adding to the dispersion an aqueous solution of a diamine, which reacts with the dicarboxylic acid dichloride at the interface between the droplets and the surrounding aqueous medium to form a polyamide shell around each droplet. Non-limiting example of suitable dicarboxylic acid dichloride include malonyl chloride, succinyl chloride, glutaryl chloride, adipyl chloride, pimeloyl chloride, suberoyl chloride, azelaoyl chloride, sebacoyl chloride, and terephthaloyl chloride. Non-limiting example of suitable diamine include 1,10-diaminodecane, 1,8-diaminooctane, 1,7-diaminoheptane, 1,6-diaminohexane, 1,5-diaminopentane, 1,4-diaminobutane, 1,3-diaminopropane, 1,2-diaminoethane, and 1,4-benzenediamine. Similarly, a polyurea shell may be formed by replacing the dicarboxylic acid dichloride with an oil soluble diisocyanate such as methylene diphenyl diisocyanate, 1,6-hexamethylene diisocyanate, 1,5-naphthalene diisocyanate, and 2,4-toluene diisocyanate. Similarly, a polyester shell may be formed by replacing the diamine with a water soluble diol such as ethylene glycol, 1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 1,8-octanediol, 1,10-decanediol, and hydroquinone. Similarly, a polyurethane shell may be formed by reacting a water soluble diol with an oil soluble diisocyanate. More detailed description of the preparations and applications of microencapsulated ink beads are disclosed in U.S. Patent Nos. 6,753,999, 6,738,050, 6,727,881, 6,724,519, 6,721,083, 6,710,540, 6,704,133, 6,652,075, 6,422,687, 6,120,588, 6,118,426, 6,067,185, and 6,017,584, all of which are incorporated herein by reference.

In other embodiments of interest, the electroactive particles of the visible light reflective layer of the adaptive camouflage structure comprise liquid crystal polymer electroactive flakes or birefringent polymer electroactive flakes. The electroactive flakes may be prepared from liquid crystal polymer materials, such as nematic liquid crystal polymer materials and cholesteric liquid crystal polymer materials. The electroactive flakes may also be prepared from birefringent polymers that do not possess liquid crystalline properties. The electroactive flakes may be prepared by casting on a silicon substrate at an elevated temperature a solvent-free film

comprising a liquid crystal polymer or a birefringent polymer. Next, the cast film is fractured into electroactive flakes by pouring liquid nitrogen over the silicon substrate. The electroactive flakes range in size from 0.1 to 500 microns. The size of the electroactive flakes may be further reduced by conventional milling methods such as ball mills, sand mills, and hammer mills.

5       The electroactive flakes may be dispersed in a host fluid contained in cavities throughout the host material in the visible light reflective layer of the adaptive camouflage structure. Alternatively, the electroactive flakes may be dispersed in host fluid contained within a microcapsule having a shell that is transparent to visible light. The preparation and application of the liquid crystal polymer or birefringent polymer flakes are disclosed in U.S. Patent No.  
10   6,665,042, which is incorporated herein by reference.

      In general, the visible light reflective layer reflects into a visible light detector the visible light coming from the sky and the surrounding environment, making the camouflaged object appear to look like the surrounding environment. In some embodiments of interest, the visible light reflective layer may be prepared by embedding a plurality of the above-mentioned visible  
15   light reflective electroactive particles in a host material, such as cross-linked silicones, which is then cured and soaked in an oil, such as silicone oils. The host material absorbs the oil and swells. The swelling process creates tiny oil-filled cavities holding the visible light reflective electroactive particles. The visible light reflective electroactive particles may rotate within such cavities when a voltage is applied. The angle of rotation can be controlled by the strength of the  
20   electric field. To overcome adhesive forces between the visible light reflective electroactive particles and the cavity wall, an electric field above an electrical threshold is applied before they rotate. This provides on the visible light reflective layer a stable visible image which remains until it is changed by the application of another electrical field. Electrical signals applied in this way allow various selected images to be placed on the visible light reflective layer as desired.

25       The infrared reflective layer 13 may comprise a host material that is sufficiently transparent to infrared radiation to allow at least a portion of the infrared spectrum to pass through. Optionally, the host material for the infrared reflective layer 13 is sufficiently transparent to other electromagnetic radiations, such as radio frequency, microwave, UV light, and a combination thereof, to allow at least a portion of the other electromagnetic radiations to  
30   pass through. Non-limiting examples of suitable host material for the infrared reflective layer 13 include polycarbonates, polyesters, polyamides (e.g., nylons), polysiloxanes (e.g., silicones and cross-linked silicones), polyethylene, polypropylene, and conducting polymers such as polyaniline, polypyrrole, polythiophene, poly(p-phenylene), polyacetylene, poly(p-phenylenevinylene), and combinations thereof. Embedded within the host material is a plurality

of infrared reflective electroactive particles that reflect at least a portion of the infrared spectrum. The infrared reflective electroactive particles may be, exactly or generally, in the shape of sphere, oval, cylinder, disk, flakes, microcapsules, or a combination thereof.

In some embodiments, the infrared reflective electroactive particles comprise infrared  
5 reflective electroactive beads or cylinders. The infrared reflective electroactive beads may be prepared by coating at least a portion of each of electrical anisotropic beads having a diameter between 10  $\mu\text{m}$  to 1 mm with an infrared reflective material. Similarly, the infrared reflective electroactive cylinders may be prepared by coating at least a portion of each of electrical anisotropic cylinders with an infrared reflective material. The electrical anisotropic beads or  
10 cylinders may be prepared by the same or similar methods described above for the bichromal beads or cylinders or polychromal segmented beads or cylinders.

The infrared reflectivity of a material is its ability to reflect infrared radiation. Materials having a high infrared reflectance generally reflect strongly while materials having a low infrared reflectance generally reflect very little. A thermally warm object can be made to appear  
15 cooler to an infrared detector if its surface is made reflective to background infrared radiation by using a material having a high infrared reflectance. Generally, the infrared reflectivity of a material increases with the electron mobility of the material. In general, a good electrical conductor with a high electron mobility, such as metals, is highly reflective with an infrared reflectance greater than 50%. Non-limited examples of good infrared reflective material (i.e.,  
20 material having an infrared reflectance greater than 50%) include metals and metal oxides. A poor electrical conductor, such as glass and organic materials, generally is a very poor reflector with a low infrared reflectance (i.e., infrared reflectance less than 50%). In general, the sky and surrounding environment of an object are thermally cooler than the object to be camouflaged in the infrared region. The Infrared reflective layer 13 of this invention reflects into an infrared  
25 detector the lower level of infrared radiation coming from the sky and the surrounding environment while at the same time reflecting away from an infrared detector infrared radiation of the object, masking the temperature of the object by making it appear to be thermally cooler and look like the surrounding environment.

A non-limiting specific example of preparing an infrared reflective electroactive bead or  
30 cylinder include the step of coating an infrared reflective material, such as metals and metal oxides, on at least a portion of a bead or cylinder comprising an electrically non-conducting material, such as glass and organic material (e.g., polyethylene), having an infrared reflectance less than 50%. The bead is generally spherical or elliptical. The bead or cylinder have a size in the range of about 10  $\mu\text{m}$  to about 1 mm. Non-limiting examples of suitable metal include

aluminum, scandium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, germanium, yttrium, zirconium, niobium, molybdenum, technetium, ruthenium, rhodium, palladium, silver, cadmium, indium, tin, antimony, lanthanum, gadolinium, hafnium, tantalum, tungsten, rhenium, osmium, iridium, platinum, gold, thallium and lead, and metallic alloys. Non-limiting examples of suitable metal oxide include indium oxide, tin oxide, indium tin oxide, cadmium stannate ( $\text{Cd}_2\text{SnO}_4$ ), copper-doped cadmium stannate, stannic oxide, and fluorine-doped stannic oxide. Because the non-metal and the infrared reflective material have different Zeta potentials, the resulted bead or cylinder should exhibit an electrical anisotropy and therefore, be electrical active. Alternatively, an infrared reflective electroactive bead may be prepared by coating on one hemisphere of a low infrared reflectance glass bead of about 10  $\mu\text{m}$  to 1 mm in diameter with an infrared reflective material by evaporation. The resulted bead should exhibit an electrical anisotropy and therefore, be electrical active because the glass and the infrared reflective material have different Zeta potentials. Alternatively, an infrared reflective electroactive bead or cylinder may be prepared by coating on at least a portion of a precoated metalized bead or cylinder having a high infrared reflectance with a material having a low infrared reflectance.

Coating of the beads or cylinders may be accomplished in any manner conventional for the application of the particular coating material to be employed, e.g., liquid coating, fluidized bed coating, spraying, rotary atomizing, monomer atomization/polymerization, vapor deposition, sputter deposition, etc. Optionally, the beads may be levitated by acoustical levitation or by the well-known fluidized bed technique.

In other embodiments of interest, the infrared reflective electroactive particles in the Infrared reflective layer 13 comprise infrared reflective electroactive microcapsules. Each infrared reflective electroactive microcapsule contains a shell, an oil or a liquid carrier within the shell, and dispersed in the liquid carrier a plurality of two different kinds of particles having opposite electrical charges or polarities. The first is a plurality of particles of an electrically conducting material, such as metals and metal oxides. The second is a plurality of particles of an electrically non-conducting material, such as various glasses and organic materials. The electrically conducting particles and the electrically non-conducting particles may be, exactly or generally, in the form of irregular shape, sphere, bead, oval, cylinder, disk, flake, or a combination thereof. The infrared reflective electroactive microcapsule generally is spherical or elliptical in shape. In some embodiments, the electrically conducting particles that are capable to reflect infrared radiation are positively charged, and the electrically non-conducting particles that do not reflect infrared radiation are negatively charged. When a negative electric field is

applied to such an infrared reflective electroactive microcapsule, the negatively charged non-reflective particles move to the bottom of the infrared reflective electroactive microcapsule where they are hidden, whereas the positively charged infrared reflective particles move to the top of the infrared reflective electroactive microcapsule where they can reflect infrared to an infrared detector. This process can be reversed by applying a positive electric field which causes the negatively charged particles to move to the top of the infrared reflective electroactive microcapsule where they absorb infrared radiation.

In one embodiment of preparing infrared reflective microcapsules, an oil phase comprising a mixture of electrically conducting particles, electrically non-conducting particles, an oil, and a dicarboxylic acid dichloride such as sebacoyl chloride (from Aldrich) is dispersed in water with stirring at a high speed, such as 200-500 rpm, and at room temperature to form a dispersion of oil droplets containing the particles and the dicarboxylic acid dichloride. Then microencapsulated ink beads are formed by adding to the dispersion an aqueous solution of a diamine which reacts with the dicarboxylic acid dichloride at the interface between the droplets and the surrounding aqueous medium to form a polyamide shell around each droplet. Similarly, a polyurea shell may be formed by replacing the dicarboxylic acid dichloride with a diisocyanate which reacts with the diamine. Similarly, a polyester shell may be formed by replacing the diamine with a diol which reacts with the dicarboxylic acid dichloride. Similarly, a polyurethane shell may be formed by reacting a diol with a diisocyanate. Non-limiting examples of suitable oil or liquid carrier for the oil phase include straight-chain, branched-chain, and cyclo-aliphatic hydrocarbons such as petroleum oils, naphtha, ligroin, hexane, pentane, heptane, octane, isododecane, isononane and cyclohexane; aromatic hydrocarbons such as benzene, toluene and xylene; halocarbon liquids such as 1,1,2-trichloro-1,2,2-trifluoroethane, trichloromonofluoromethane and carbon tetrachloride; and isoparaffinic hydrocarbons such as various Isopar<sup>TM</sup> carrier liquids from Exxon Company, Houston, TX, and Norpar<sup>T</sup> carrier liquids from ExxonMobile Chemical, Houston, TX. The size of the infrared reflective electroactive microcapsules may vary from 1 to 1000 microns.

In some embodiments, the infrared reflective electrically conducting particle include particles of metals and metal oxide, such as aluminum, gold, silver, indium oxide, tin oxide, indium tin oxide, cadmium stannate ( $\text{Cd}_2\text{SnO}_4$ ), copper-doped cadmium stannate, stannic oxide, and fluorine-doped stannic oxide. In other embodiments of interest, the size of the metal particles is in the range of 1 nanometer to 1 micron. infrared reflective metal particles may be prepared according to methods known in art such as the metal salt reductive method, metal evaporation method, surfactant-stabilized-metal dispersion method, and the methods disclosed in

U.S. Patent Nos. 4,892,798, 5,089,362, 5,312,683, 5,322,751, 5,332,646, and 5,437,912, all of which are incorporated herein by reference. In further embodiments of interest, the infrared non-reflective particles are particles of glass or an organic material such as plastics and elastomers. The metal loading in the infrared reflective electroactive microcapsules may be in the range of 0.01 to 5.0 g/100 ml of the oil.

In other embodiments of interest, the infrared reflective electroactive particles in the Infrared reflective layer 13 comprise infrared reflective electroactive microcapsules. Each infrared reflective electroactive microcapsule contains a shell, an oil or a liquid carrier within the shell, and dispersed in the liquid carrier a plurality of infrared reflective flakes. The infrared reflective electroactive microcapsule generally is spherical or elliptical in shape. The infrared reflective flakes may be prepared by casting on a silicon substrate at an elevated temperature a solvent-free film comprising a liquid crystal polymer or a birefringent polymer. Next, the outer surface of the cast film is coated with an infrared reflective metal such as aluminum, scandium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, germanium, yttrium, zirconium, niobium, molybdenum, technetium, ruthenium, rhodium, palladium, silver, cadmium, indium, tin, antimony, lanthanum, gadolinium, hafnium, tantalum, tungsten, rhenium, osmium, iridium, platinum, gold, thallium and lead, and metallic alloys. The metal coating may be applied by any conventional coating device. Non-limiting examples of suitable coating device include chemical vapor deposit systems, such as Plasma-Enhanced CVD System, Model PD-200D, available from SAMCO, Sunnyvale, CA, PECVD system, model NPE-4000, available from Nano-master Austin, TX, Ultra-High Vacuum CVD Process System, Model UHV-CVD-5000, available from Tek-Vac Industries, Brentwood, NY; sputtering systems such as sputter coaters, models NSC 4000 and 3000, available from Nano-master, Austin, TX, and EXPLORER™ 14 Magnetron Sputtering System, DISCOVERY® 18 DC/RF Research Magnetron Sputter Deposition System, DISCOVERY® 24 Multi-Cathode DC/RF Magnetron Sputter System, and Bench Top Turbo IV High Vacuum Deposition System, all available from Denton Vacuum, Moorestown, NJ; gas evaporation reactors such as that described in U.S. Patent No. 4,871,790, incorporated herein by reference; the Klabunde-style static reactors; and rotary reactors of the Torrovap™ design (available from Torrovap Industries, Markham, Ontario, Canada). Next, the metalized coating is fractured into a plurality of electroactive infrared reflective flakes by pouring liquid nitrogen over the silicon substrate. The electroactive infrared reflective flakes may be dispersed in an oil and then turned into microcapsules according to the encapsulation techniques described above.

Optionally a surfactant may be used to facilitate the dispersing of the infrared reflective flakes, the electrically conducting particles, or the electrically non-conducting particles in an oil. Non-limiting examples of suitable surfactant for infrared reflective flakes and the electrically conducting particles include epoxide terminated polyisobutylenes such as Actipol<sup>TM</sup> E6, E16, and E23 (available from Amoco Chemical Co., Chicago, IL); commercial oil additives such as Lubrizol<sup>TM</sup> 6401 and Lubrizol<sup>TM</sup> 6418 (available from The Lubrizol Corporation, Wickliffe, OH), AMOCO<sup>TM</sup> 9250 (available from AMOCO Petroleum Additives Company, Naperville, IL), and OLOA<sup>TM</sup> 1200 (a low molecular weight polyisobutylene attached to a diamine head group by a succinimide linkage, available from Chevron Chemical Company, San Francisco, CA); and hydrocarbon compatible hyperdispersants such as Solsperse<sup>TM</sup> 17,000 (available from ICI Americas Inc., Wilmington, DE). In general, the concentration of the surfactant is in the range of 0.001 to 10.0 g/100 mL based on the total fluid.

Optionally a charge director or charge control agent may be used to control the charge of the infrared reflective flakes, the electrically conducting particles, or the electrically non-conducting particles. The charge director may impart either positive charge or negative charge to the particles or flakes. Non-limiting examples of suitable positive charge director include organic acid metal salts consisting of polyvalent metal ions and organic anions as the counterion. Non-limiting examples of suitable metal ions include Ba(II), Ca(II), Mn(II), Zn(II), Zr(IV), Cu(II), Al(III), Cr(III), Fe(II), Fe(III), Sb(III), Bi(III), Co(II), La(III), Pb(II), Mg(II), Mo(III), Ni(II), Ag(I), Sr(II), Sn(IV), V(V), Y(III), and Ti(IV). Non-limiting examples of suitable organic anions include carboxylates or sulfonates derived from aliphatic or aromatic carboxylic or sulfonic acids, preferably aliphatic fatty acids such as stearic acid, behenic acid, neodecanoic acid, diisopropylsalicylic acid, abietic acid, naphthenic acid, octanoic acid, lauric acid, tallic acid, and the like. In some embodiments, the positive charge directors are the metallic carboxylates (soaps) described in U.S. Pat. No. 3,411,936, incorporated herein by reference, which include alkaline earth- and heavy-metallic salts of fatty acids containing at least 6-7 carbons and cyclic aliphatic acids including naphthenic acid. In other embodiments, the positive charge directors are polyvalent metal soaps of zirconium and aluminum. In further embodiments, the positive charge director is the zirconium soap of octanoic acid (Zirconium HEX-CEM from Mooney Chemicals, Cleveland, Ohio). Non-limiting examples of suitable negative charge directors are polymers or copolymers having nitrogen-containing monomer, quaternary ammonium block copolymers, lecithin, basic metallic petronates such as basic barium petronate, basic calcium petronate, and basic sodium petronate, metal naphthenate compounds, and polyisobutylene succinimide available as OLOA<sup>TM</sup> 1200 from Chevron Oronite Company LLC, Houston, TX,

and the like. Non-limiting examples of the nitrogen-containing monomer are (meth)acrylates having an aliphatic amino group, vinyl monomers having nitrogen-containing heterocyclic ring, cyclic amide monomers having N-vinyl substituent, (meth)acrylamides, aromatic substituted ethylenic monomers having nitrogen-containing group, nitrogen-containing vinyl ether  
5 monomers, etc. In some embodiments, the negative charge directors are lecithin, basic metallic petronate, and polyisobutylene succinimide. The charge director levels depend upon a number of factors, including the chemical composition of the dispersion and the particle size of the electroactive infrared reflective particles or flakes. Those skilled in the art know how to adjust the level of charge director based on the listed parameters to achieve the desired results for their  
10 particular application.

In some embodiments of interest, the infrared reflective layer 13 may be prepared by embedding a plurality of the above-described infrared reflective electroactive particles in a host material, such as cross-linked silicones, which is then cured and soaked in an oil, such as silicone  
15 oils. The host material absorbs the oil and swells. The swelling process creates tiny oil-filled cavities holding the infrared reflective electroactive particles. The infrared reflective electroactive particles may rotate within such cavities when a voltage is applied. The angle of rotation can be controlled by the strength of the electric field. To overcome adhesive forces between the infrared reflective electroactive particles and the cavity wall, an electrical field above an electrical threshold is applied before they rotate. This application of the field provides  
20 a stable image that remains on the infrared reflective layer 13 until it is changed by the application of another electrical field. Electrical signals applied in this way allow selected images to be placed on the infrared reflective layer 13 as desired.

In general, a good electrical conductor, such as a metal, has a high radio frequency reflectivity. Non-limited examples of good radar reflective material include metals such as  
25 aluminum, scandium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, germanium, yttrium, zirconium, niobium, molybdenum, technetium, ruthenium, rhodium, palladium, silver, cadmium, indium, tin, antimony, lanthanum, gadolinium, hafnium, tantalum, tungsten, rhenium, osmium, iridium, platinum, gold, thallium and lead, and metallic alloys. A poor electrical conductor, such as various glasses and organic materials, has little  
30 electron mobility and thus a low radio frequency reflectivity. The radar reflective layer will reflect into a radar detector the lower level of radio frequency coming from the sky and the surrounding environment while at the same time reflecting away from the radar detector all other radio frequencies, making the camouflaged object appear to look like the surrounding environment.

The radar reflective layer 23 may comprise a host material that is sufficiently transparent to radio frequency radiation to allow at least a portion of the radio frequency spectrum to pass through. Optionally, the host material for the radar reflective layer may be sufficiently transparent to one or more other electromagnetic radiations, such as infrared radiation, microwave, and UV light. Non-limiting examples of suitable host material for the radar reflective layer include polycarbonates, polyesters, polyamides (e.g., nylons), polysiloxanes (e.g., silicones and cross-linked silicones), polyethylene, polypropylene, and conducting polymers such as polyaniline, polypyrrole, polythiophene, poly(p-phenylene), polyacetylene, poly(p-phenylenevinylene), and combinations thereof. Embedded within the host material are a plurality of radar reflective electroactive particles that can reflect at least a portion of the radio frequency spectrum. In some embodiments of interest, the radar reflective electroactive particles comprise radar reflective beads or cylinders. The radar reflective beads may be prepared by coating the electrical anisotropic beads having a diameter between 10  $\mu\text{m}$  to 1 mm with a radar reflective material. Similarly, the radar reflective cylinders may be prepared by coating electrical anisotropic cylinders with a radar reflective material. The electrical anisotropic beads or cylinders may be prepared by the same or similar methods mentioned above for the bichromal beads or cylinders or polychromal segmented beads or cylinders.

Since metals are both good infrared reflective materials and radar reflective materials, the above-described infrared reflective microcapsules containing metal particles or flakes may be used as radar reflective electroactive microcapsules for reflecting radio frequency. In some embodiments of interest, the radar reflective electroactive particles in the radar reflective layer 23 comprise radar reflective electroactive microcapsules.

In some embodiments of interest, the radar reflective layer 23 may be prepared by embedding a plurality of the above-described radar reflective electroactive particles in a host material, such as cross-linked silicones, which is then cured and soaked in an oil, such as silicone oils. The host material absorbs the oil and swells. The swelling process creates tiny oil-filled cavities holding the radar reflective electroactive particles. The radar reflective electroactive particles may rotate within such cavities when a voltage is applied. The angle of rotation can be controlled by the strength of the electric field. To overcome adhesive forces between the radar reflective electroactive particles and the cavity wall, an electric field above an electrical threshold is applied before they rotate. This application of a field provides a stable image that remains on the radar reflective layer 23 until it is changed by the application of another electrical field. Electrical signals applied in this way allow selected images to be placed on the radar reflective layer 23 as desired.

In some embodiments of interest, one of the layers of the adaptive camouflage structure may comprise a UV reflective layer. The UV reflective layer may comprise a host material that is sufficiently transparent to UV light to allow at least a portion of the UV spectrum to pass through. Optionally, the host material for the UV reflective layer may be sufficiently transparent to one or more other electromagnetic radiations, such as infrared radiation, microwave, and radio frequency. Non-limiting examples of suitable host material for the UV reflective layer include polycarbonates, polyesters, polyamides (e.g., nylons), polysiloxanes (e.g., silicones and cross-linked silicones), polyethylene, polypropylene, and conducting polymers such as polyaniline, polypyrrole, polythiophene, poly(p-phenylene), polyacetylene, poly(p-phenylenevinylene), and combinations thereof. Embedded within the host material are a plurality of UV reflective electroactive particles that can reflect at least a portion of the UV spectrum. In some embodiments of interest, the UV reflective electroactive particles comprise UV reflective electroactive beads or cylinders. The UV reflective electroactive beads may be prepared by coating electrical anisotropic beads having a diameter between 10  $\mu\text{m}$  to 1 mm with a UV reflective material. Similarly, the UV reflective electroactive cylinders may be prepared by coating electrical anisotropic cylinders with a UV reflective material. The electrical anisotropic beads or cylinders may be prepared by the same or similar methods mentioned above for the bichromal beads or cylinders or polychromal segmented beads or cylinders.

In general, a good electrical conductor, such as a metal, has a high UV reflectivity. Non-limited examples of good UV reflective material include metals such as aluminum, scandium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, germanium, yttrium, zirconium, niobium, molybdenum, technetium, ruthenium, rhodium, palladium, silver, cadmium, indium, tin, antimony, lanthanum, gadolinium, hafnium, tantalum, tungsten, rhenium, osmium, iridium, platinum, gold, thallium and lead, and metallic alloys. A poor electrical conductor, such as various glasses and organic materials, has little electron mobility and a low UV reflectivity. In general, a UV reflective layer may reflect into a UV detector the lower level of UV light coming from the sky and the surrounding environment while at the same time reflecting away from the UV detector all other UV light, making the camouflaged object appear to look like the surrounding environment.

Since metals are both good infrared reflective materials and UV reflective materials, the above-mentioned infrared reflective microcapsules containing metal particles or flakes may be used as UV reflective microcapsules for reflecting UV light. In some embodiments of interest, the UV reflective electroactive particles in the UV reflective layer comprise UV reflective electroactive microcapsules.

In some embodiments of interest, the UV reflective layer may be prepared by embedding a plurality of the above-described UV reflective electroactive particles in a host material, such as cross-linked silicones, which is then cured and soaked in an oil, such as silicone oils. The host material absorbs the oil and swells. The swelling process creates tiny oil-filled cavities holding the UV reflective electroactive particles. The UV reflective electroactive particles may rotate within such cavities when a voltage is applied. The angle of rotation can be controlled by the strength of the electric field. To overcome adhesive forces between the UV reflective electroactive particles and the cavity wall, an electrical field above an electrical threshold is applied before they rotate. This application of the electrical field provides a stable image that remains on the UV reflective layer until it is changed by the application of another electrical field. Electrical signals applied in this way allow selected images to be placed on the UV reflective layer as desired.

In some embodiments of interest, one of the layers of the adaptive camouflage structure of this invention may comprise a microwave reflective layer. The microwave reflective layer may comprise a host material that is sufficiently transparent to microwave to allow at least a portion of the microwave spectrum to pass through. Optionally, the host material for the microwave reflective layer may be sufficiently transparent to one or more other electromagnetic radiations, such as infrared radiation, UV, and radio frequency. Non-limiting examples of suitable host material for the microwave reflective layer include polycarbonates, polyesters, polyamides (e.g., nylons), polysiloxanes (e.g., silicones and cross-linked silicones), polyethylene, polypropylene, and conducting polymers such as polyaniline, polypyrrole, polythiophene, poly(p-phenylene), polyacetylene, poly(p-phenylenevinylene), and combinations thereof. Embedded within the host material are a plurality of microwave reflective electroactive particles that can reflect at least a portion of the microwave spectrum. In some embodiments, the microwave reflective electroactive particles comprise microwave reflective electroactive beads or cylinders. The microwave reflective electroactive beads may be prepared by coating electrical anisotropic beads having a diameter between 10  $\mu\text{m}$  to 1 mm with a microwave reflective material. Similarly, the microwave reflective electroactive cylinders may be prepared by coating electrical anisotropic cylinders with a microwave reflective material. The electrical anisotropic beads or cylinders may be prepared by the same or similar methods described above for the bichromal beads or cylinders or polychromal segmented beads or cylinders.

In general, a good electrical conductor, such as a metal, has a high microwave reflectivity. Non-limited examples of good microwave reflective material include metals such as aluminum, scandium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium,

germanium, yttrium, zirconium, niobium, molybdenum, technetium, ruthenium, rhodium, palladium, silver, cadmium, indium, tin, antimony, lanthanum, gadolinium, hafnium, tantalum, tungsten, rhenium, osmium, iridium, platinum, gold, thallium and lead, and metallic alloys. A poor electrical conductor, such as various glasses and organic materials, has little electron mobility and a low microwave reflectivity. In general, the microwave reflective layer will reflect into a microwave detector the lower level of microwave coming from the sky and the surrounding environment, making the camouflaged object appear to look like the surrounding environment.

Since metals are both good infrared reflective materials and microwave reflective materials, the above-described infrared reflective microcapsules containing metal particles or flakes may be used as microwave reflective microcapsules for reflecting microwave. In some embodiments of interest, the microwave reflective electroactive particles in the microwave reflective layer comprise microwave reflective microcapsules.

In some embodiments of interest, the microwave reflective layer may be prepared by embedding a plurality of the above-described microwave reflective electroactive particles in a host material, such as cross-linked silicones, which is then cured and soaked in an oil, such as silicone oils. The host material absorbs the oil and swells. The swelling process creates tiny oil-filled cavities holding the microwave reflective electroactive particles. The microwave reflective electroactive particles may rotate within such cavities when a voltage is applied. The angle of rotation can be controlled by the strength of the electric field. To overcome adhesive forces between the microwave reflective electroactive particles and the cavity wall, an electrical field above an electrical threshold is applied before they rotate. This application of the electric field provides a stable image that remains on the microwave reflective layer until it is changed by the application of another electrical field. Electrical signals applied in this way allow selected images to be placed on the microwave reflective layer as desired.

FIG. 7 depicts a specific, but not limiting, method of making the infrared reflective beads, the radar reflective beads, the UV reflective beads, or the microwave reflective beads. Two hardenable liquid materials 106 and 107 are applied by delivery means (not shown) to opposite sides 104, 105 of a spinning disk 102, which rotates uniformly about shaft 103. The hardenable liquid materials 106, 107, each independently, may comprise a molten liquid, such as molten carnauba wax and molten polyethylene. Centrifugal force causes the hardenable liquid materials 106, 107 to flow toward the periphery of disk 102, where they combine at the edge to form liquid streams 108 that become unstable and eventually break up into beads 109. The beads may solidify by cooling at room temperature or the cooling may be accelerated by having the liquid

beads passing through a cooling zone containing cold liquid nitrogen vapors (not shown). In some embodiments, one of the hardenable liquid materials may further comprise particles or flakes comprising an infrared reflective material, a radar reflective material, a UV reflective material, or a microwave reflective material. Therefore, each of the beads 109 has one hemisphere containing particles or flakes of an infrared reflective material, a radar reflective material, a UV reflective material, or a microwave reflective material, such as aluminum, and the other hemisphere having none.

In other embodiments, the hardenable liquid materials 106 and 107 are the same. The reflective beads 109 may be obtained by coating the solidified beads with a reflective material 82, such as an infrared reflective material, a radar reflective material, a UV reflective material, and a microwave reflective material, with a coating device 81 which emits tiny droplets or vapors 83 of the reflective material 82 on one hemisphere of each of the solidified beads. Non-limiting examples of the coating device include gas evaporation reactor such as that described in U.S. Patent No. 4,871,790, incorporated herein by reference; the Klabunde-style static reactor; a rotary reactor of the Torrovap<sup>TM</sup> design (available from Torrovap Industries, Markham, Ontario, Canada); chemical vapor deposit systems, such as Plasma-Enhanced CVD System, Model PD-200D, available from SAMCO, Sunnyvale, CA; PECVD system, model NPE-4000, available from Nano-master Austin, TX; Ultra-High Vacuum CVD Process System, Model UHV-CVD-5000, available from Tek-Vac Industries, Brentwood, NY, and sputtering systems such as sputter coaters, models NSC 4000 and 3000, available from Nano-master, Austin, TX; and EXPLORER<sup>TM</sup> 14 Magnetron Sputtering System, DISCOVERY<sup>®</sup> 18 DC/RF Research Magnetron Sputter Deposition System, DISCOVERY<sup>®</sup> 24 Multi-Cathode DC/RF Magnetron Sputter System, and Bench Top Turbo IV High Vacuum Deposition System, available from Denton Vacuum, Moorestown, NJ.

FIG. 8 depicts another specific, but not limiting, method of making the infrared reflective beads, the radar reflective beads, the UV reflective beads, or the microwave reflective beads. Two hardenable liquid materials 106 and 107 are applied by delivery means (not shown) to opposite side of a double barrel nozzle 91. The hardenable liquid materials 106, 107, each independently, may comprise a molten liquid, such as molten carnauba wax and molten polyethylene. The hardenable liquid materials 106 and 107 are charged by means of positive electrode 95 and negative electrode 96 and combine by means of electrostatic attraction at the exit of the double barrel nozzle 91 to form beads 120 having an implanted dipole moment. In some embodiments, one of the hardenable liquid materials may further comprise particles or flakes comprising an infrared reflective material, a radar reflective material, a UV reflective

material, or a microwave reflective material. Therefore, each of the beads 120 has one hemisphere containing particles or flakes of an infrared reflective material, a radar reflective material, a UV reflective material, or a microwave reflective material, such as aluminum, and the other hemisphere having none. In other embodiments, the hardenable liquid materials 106 and 107 are the same. The reflective beads 120 may be obtained by coating the solidified beads with a reflective material 82, such as an infrared reflective material, a radar reflective material, a UV reflective material, and a microwave reflective material, with a coating device 81 which emits tiny droplets or vapors 83 of the reflective material 82 on one hemisphere of each of the solidified beads. Non-limiting examples of the coating device include chemical vapor deposit systems and sputtering systems, such as those mentioned above.

The desired camouflage image for each reflective layer of the adaptive camouflage structure of this invention may be determined by one or a combination of methods known in the art. The methods vary from preset programmed images to actively changing images that are generated by sensors that monitor changes in the indigenous environment that result from, for example, vehicle motion or changing light conditions. Some of the conventional methods of determining the desired images are disclosed in U.S. Patent Nos. 6,682,879, 6,342,290, and 5,307,162, all of which are incorporated herein by reference.

Once a desired camouflage image for each reflective layer of the adaptive camouflage structure of this invention is selected. The desired camouflage images in the reflective layers of the adaptive camouflage structure may be generated by means of adjustable voltage pattern interlayers, each of which is adjacent to a reflective layer for controlling the camouflage image reflected by that layer. Each adjustable voltage pattern interlayer may be a conventional addressing schemes such as the canted-field electrode configuration in U.S. Patent No. 5,717,515, the multithreshold addressing system in U.S. Patent No. 5,739,801, the dual vector field addressing system in U.S. Patent No. 6,690,350, and the various addressing systems in U.S. Patent Nos. 6,753,999, 6,693,620, 6,531,997, 6,504,524, and 6,445,489, all of the above-mentioned patents are incorporated herein by reference.

In some embodiments of interest, each adjustable voltage pattern interlayer comprises an active matrix addressing scheme. The active matrix addressing scheme divides each reflective layer into an array of tiny cells defined by columns and rows. Each cell representing a pixel and, in general, contains one thin-film transistor for each cell. Each thin-film transistor controls the voltage applied to at least a reflective electroactive particle so as to control their orientation for reflecting a particular radiation, such as visible light, UV light, infrared radiation, microwave,

and/or radio frequency. In some embodiments, the active matrix addressing scheme controls the voltage in each cell by the following procedure:

(1) a selected voltage is applied to the gates of a selected row (e.g., the first row) of the active matrix while non-selected voltages are applied to the gates of all other rows;

(2) data voltages are applied at the same time to all of the column electrodes to charge each pixel in the selected row to the desired voltages;

(3) the selected voltage applied to the gates in the selected row is charged to a non-selected voltage; and

(4) steps 1-3 are repeated for each succeeding row until all of the rows have been selected and the pixels charged to the desired voltages.

In other embodiments of interest, the addressing scheme comprises two electrode assemblies located on either side of a reflective layer in which the reflective electroactive particles are embedded. Both electrode assemblies are made of an optically transparent conductor with a very high resistivity. Each of the electrode assemblies is divided into rectangular regions. The rectangular regions are separated by high-resistivity separators which may be made of glass or other substrate material. Both electrode assemblies can be connected to a power supply. Within each of the rectangular regions are located individually addressable bus bars. The electrode bus bars in one electrode are situated parallel to and directly above the electrode bus bars in the other electrode. The voltage at each individual bus bar can be set using active matrix addressing electronics. Accordingly, each of the rectangular regions can be individually addressed. The above electrode configuration is described in U.S. Patent No. 6,492,967, which is incorporated herein by reference.

In further embodiments of interest, each of the reflective layers of the adaptive camouflage structure of this invention can be addressed separately from the other reflective layers by one of the above addressing schemes. In additional embodiments of interest, a single addressing scheme may be used to control all the reflective layers of the adaptive camouflage structure if the reflective electroactive particles in different layers of the adaptive camouflage structure have different rotational thresholds. Referring to FIG. 9, if the visible reflective electroactive particles 60 in the visible reflective layer 3 will begin to rotate only upon application of a high voltage  $V_1$ , the infrared reflective electroactive particles 61 will begin to rotate in the infrared reflective layer 13 upon application of an medium voltage  $V_2$ , and the radar reflective electroactive particles 62 will begin to rotate in the radar reflective layer 3 upon application of a low voltage  $V_3$ , then an adjustable voltage pattern interlayer 40 comprising a single addressing scheme may be used to address the reflective electroactive particles in all the

three layers. For example, the application of the high voltage  $V_1$  will cause reflective electroactive particles 60, 61, and 62 in all three layers to rotate. The application of the medium voltage  $V_2$  will cause only the infrared reflective electroactive particles 61 and the radar reflective electroactive particles 62 to rotate. The application of the low voltage  $V_3$  causes only the radar reflective electroactive particles 62 to rotate. Thus the reflective electroactive particles in all three layers can be rotated as desired by successive application of high, medium, and low addressing voltages.

The embodiments described above are intended to be illustrative and not limiting. Additional embodiments are within the claims below. Although the present invention has been described with reference to preferred embodiments, workers skilled in the art will recognize that changes may be made in form and detail without departing from the spirit and scope of the invention.

CLAIMS

1. An electroactive microcapsule comprising a shell and a mixture inside the shell, wherein the mixture comprises an oil; a plurality of metal particles; and a plurality of electrically non-conducting particles, and wherein the plurality of metal particles and the plurality of electrically non-conducting particles have opposite charges.

2. The electroactive microcapsule of claim 1 wherein the oil is selected from the group consisting of straight-chain, branched-chain, and cyclo-aliphatic hydrocarbons, aromatic hydrocarbons, halocarbon liquids, and isoparaffinic hydrocarbons.

3. The electroactive microcapsule of claim 1 wherein the concentration of the plurality of metal particles is in the range of 0.01 to 5.0 g of metal particles per 100 ml of the oil.

4. The electroactive microcapsule of claim 1 wherein the metal particles are particles of a metal selected from the group consisting of aluminum, scandium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, germanium, yttrium, zirconium, niobium, molybdenum, technetium, ruthenium, rhodium, palladium, silver, cadmium, indium, tin, antimony, lanthanum, gadolinium, hafnium, tantalum, tungsten, rhenium, osmium, iridium, platinum, gold, thallium, lead, and combinations thereof.

5. The electroactive microcapsule of claim 1 wherein the metal particles are generally in the form of sphere, bead, oval, cylinder, disk, flake, or a combination thereof.

6. The electroactive microcapsule of claim 1 wherein the electrically non-conducting particles are particles of an electrically non-conducting material selected from the group consisting of glasses and organic materials.

7. The electroactive microcapsule of claim 1 wherein the electrically non-conducting particles are generally in the form of sphere, bead, oval, cylinder, disk, flake, or a combination thereof.

8. The electroactive microcapsule of claim 1 wherein the metal particles are positively charged and the electrically non-conducting particles are negatively charged.

9. The electroactive microcapsule of claim 1 wherein the metal particles are negatively charged and the electrically non-conducting particles are positively charged.

10. The electroactive microcapsule of claim 1 wherein the shell comprises a polymer  
5 derived from a diamine and a dicarboxylic acid dichloride.

11. The electroactive microcapsule of claim 10 wherein the diamine is selected from the group consisting of 1,10-diaminodecane, 1,8-diaminooctane, 1,7-diaminoheptane, 1,6-diaminohexane, 1,5-diaminopentane, 1,4-diaminobutane, 1,3-diaminopropane, 1,2-  
10 diaminoethane, and 1,4-benzenediamine.

12. The electroactive microcapsule of claim 10 wherein the dicarboxylic acid dichloride is selected from the group consisting of malonyl chloride, succinyl chloride, glutaryl chloride, adipyl chloride, pimeloyl chloride, suberoyl chloride, azelaoyl chloride, sebacoyl  
15 chloride, and terephthaloyl chloride.

13. The electroactive microcapsule of claim 1 wherein the shell comprises a polymer derived from a diisocyanate and a diamine or a diol.

14. The electroactive microcapsule of claim 13 wherein the diamine is selected from the group consisting of 1,10-diaminodecane, 1,8-diaminooctane, 1,7-diaminoheptane, 1,6-diaminohexane, 1,5-diaminopentane, 1,4-diaminobutane, 1,3-diaminopropane, 1,2-diaminoethane, and 1,4-benzenediamine.  
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15. The electroactive microcapsule of claim 13 wherein the diol is selected from the group consisting of such as ethylene glycol, 1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 1,8-octanediol, 1,10-decanediol, and hydroquinone.  
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16. The electroactive microcapsule of claim 13 wherein the diisocyanate is selected from the group consisting of methylene diphenyl diisocyanate, 1,6-hexamethylene diisocyanate, 1,5-naphthalene diisocyanate, and 2,4-toluene diisocyanate.  
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17. The electroactive microcapsule of claim 1 wherein the shell comprises a polymer derived from a diol and a dicarboxylic acid dichloride.

18. The electroactive microcapsule of claim 17 wherein the diol is selected from the group consisting of such as ethylene glycol, 1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 1,8-octanediol, 1,10-decanediol, and hydroquinone.

5

19. The electroactive microcapsule of claim 17 wherein the dicarboxylic acid dichloride is selected from the group consisting of malonyl chloride, succinyl chloride, glutaryl chloride, adipyl chloride, pimeloyl chloride, suberoyl chloride, azelaoyl chloride, sebacoyl chloride, and terephthaloyl chloride.

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20. The electroactive microcapsule of claim 1 wherein the mixture further comprises a positive charge director, a negative charge director, or a combination thereof.

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21. The electroactive microcapsule of claim 1 wherein the mixture further comprises a surfactant.

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22. The electroactive microcapsule of claim 21 wherein the surfactant is selected from the group consisting of epoxide terminated polyisobutylenes, commercial oil additives, a low molecular weight polyisobutylene attached to a diamine head group through a succinimide linkage, and hydrocarbon compatible hyperdispersants.

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23. An electroactive particle comprising an electrically non-conducting particle and a coating of a reflective material, wherein the coating covers a portion of the electrically non-conducting particle and wherein the reflective material is selected from the group consisting of infrared reflective materials, radar reflective materials, microwave reflective materials, UV reflective materials, and combinations thereof.

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24. The electroactive particle of claim 23 wherein the coating covers generally one half of the electrically non-conducting particle.

25. The electroactive particle of claim 23 wherein the electroactive particle is generally in the form of a bead or a cylinder.

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26. The electroactive particle of claim 23 wherein the electrically non-conducting particle comprises of a glass or an organic material.

27. The electroactive particle of claim 23 wherein the reflective material comprises a metal selected from the group consist of aluminum, scandium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, germanium, yttrium, zirconium, niobium, molybdenum, technetium, ruthenium, rhodium, palladium, silver, cadmium, indium, tin, antimony, lanthanum, gadolinium, hafnium, tantalum, tungsten, rhenium, osmium, iridium, platinum, gold, thallium, lead, and combinations thereof.

28. An electroactive microcapsule comprising a shell and a mixture inside the shell, wherein the mixture comprises an oil and a plurality of flakes comprising of a polymer and wherein a layer of metal is coated on one of the flat surface of each flake.

29. The electroactive microcapsule of claim 28 wherein the metal is selected from the group consist of aluminum, scandium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, gallium, germanium, yttrium, zirconium, niobium, molybdenum, technetium, ruthenium, rhodium, palladium, silver, cadmium, indium, tin, antimony, lanthanum, gadolinium, hafnium, tantalum, tungsten, rhenium, osmium, iridium, platinum, gold, thallium, lead, and combinations thereof.

30. The electroactive microcapsule of claim 28 wherein the oil is selected from the group consisting of straight-chain, branched-chain, and cyclo-aliphatic hydrocarbons, aromatic hydrocarbons, halocarbon liquids, and isoparaffinic hydrocarbons.

31. The electroactive microcapsule of claim 28 wherein the polymer comprises a liquid crystal polymer or a birefringent polymer.

32. The electroactive microcapsule of claim 28 wherein the shell comprises a polymer derived from a diamine and a dicarboxylic acid dichloride.

33. The electroactive microcapsule of claim 32 wherein the diamine is selected from the group consisting of 1,10-diaminodecane, 1,8-diaminooctane, 1,7-diaminoheptane, 1,6-

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diaminohexane, 1,5-diaminopentane, 1,4-diaminobutane, 1,3-diaminopropane, 1,2-diaminoethane, and 1,4-benzenediamine.

34. The electroactive microcapsule of claim 32 wherein the dicarboxylic acid dichloride is selected from the group consisting of malonyl chloride, succinyl chloride, glutaryl chloride, adipyl chloride, pimeloyl chloride, suberoyl chloride, azelaoyl chloride, sebacoyl chloride, and terephthaloyl chloride.

35. The electroactive microcapsule of claim 28 wherein the shell comprises a polymer derived from a diisocyanate and a diamine or a diol.

36. The electroactive microcapsule of claim 35 wherein the diamine is selected from the group consisting of 1,10-diaminodecane, 1,8-diaminooctane, 1,7-diaminoheptane, 1,6-diaminohexane, 1,5-diaminopentane, 1,4-diaminobutane, 1,3-diaminopropane, 1,2-diaminoethane, and 1,4-benzenediamine.

37. The electroactive microcapsule of claim 35 wherein the diol is selected from the group consisting of such as ethylene glycol, 1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 1,8-octanediol, 1,10-decanediol, and hydroquinone.

38. The electroactive microcapsule of claim 35 wherein the diisocyanate is selected from the group consisting of methylene diphenyl diisocyanate, 1,6-hexamethylene diisocyanate, 1,5-naphthalene diisocyanate, and 2,4-toluene diisocyanate.

39. The electroactive microcapsule of claim 28 wherein the shell comprises a polymer derived from a diol and a dicarboxylic acid dichloride.

40. The electroactive microcapsule of claim 39 wherein the diol is selected from the group consisting of such as ethylene glycol, 1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 1,8-octanediol, 1,10-decanediol, and hydroquinone.

41. The electroactive microcapsule of claim 39 wherein the dicarboxylic acid dichloride is selected from the group consisting of malonyl chloride, succinyl chloride, glutaryl

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chloride, adipyl chloride, pimeloyl chloride, suberoyl chloride, azelaoyl chloride, sebacoyl chloride, and terephthaloyl chloride.

42. The electroactive microcapsule of claim 28 wherein the mixture further comprises  
5 a positive charge director, a negative charge director, or a combination thereof.

43. The electroactive microcapsule of claim 28 wherein the mixture further comprises  
a surfactant.

10 44. The electroactive microcapsule of claim 43 wherein the surfactant is selected  
from the group consisting of epoxide terminated polyisobutylenes, commercial oil additives, a  
low molecular weight polyisobutylene attached to a diamine head group through a succinimide  
linkage, and hydrocarbon compatible hyperdispersants.