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HIGH STRAIN PIEZO-POLYMER

TO ALL WHOM IT MAY CONCERN:

BE IT KNOWN THAT (1) THOMAS S. RAMOTOWSKI, employee of the United States Government, (2) GEORGE J. KAVARNOS and (3) QIMING ZHANG, citizens of the United States of America, and residents of (1) Tiverton, County of Newport, State of Rhode Island, (2) New London, County of New London, State of Connecticut, and (3) State College, County of Centre, State of Pennsylvania, have invented certain new and useful improvements entitled as set forth above of which the following is a specification.

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Michael F. Oglo
APPLICANT'S ATTORNEY

21 November 2003
DATE OF SIGNATURE

1 Attorney Docket No. 83303

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3 HIGH STRAIN PIEZO-POLYMER
4

5 The present application is based on a Provisional
6 Application, No. 60/428,167, which was filed on November 21,
7 2002, and which is entitled HIGH STRAIN PIEZO-POLYMER by Thomas
8 Ramotowski, George Kavarnos, and Qiming Zhang.
9

10 STATEMENT OF GOVERNMENT INTEREST

11 The invention described herein may be manufactured and used
12 by the Government of the United States of America for
13 Governmental purposes without the payment of any royalty thereon
14 or therefor.
15

16 CROSS REFERENCE TO OTHER RELATED APPLICATIONS

17 None.
18

19 BACKGROUND OF INVENTION

20 (1) Field of the Invention

21 This invention relates to a new class of terpolymers for
22 use as high strain electrostrictive polymer films. More
23 particularly, the invention relates to a class of
24 electrostrictive terpolymers comprising vinylidene fluoride

(VDF), trifluoroethylene (TrFE) and at least one monomer having at least one bulky halogen atom side group. The monomer is preferably a chloro-monomer such as chlorofluoroethylene (CFE) or chlorotrifluoroethylene (CTFE). The chlorofluoroethylene (CFE) is preferably 1-chloro-2-fluoroethylene or 1-chloro-1-fluoroethylene.

(2) Description of the Prior Art

Many research activities in the past decade have focused on vinylidene fluoride-trifluoroethylene (VDF-TrFE) copolymers with the goal of reducing the energy barrier for ferroelectric-paraelectric phase transition and generating large and fast electric-induced mechanical responses at ambient temperatures. The close connection between the crystalline structure and electric properties led to many attempts to alter copolymer morphology by mechanical deformation, electron-radiation, crystallization, etc.

One of the main methods of processing or converting polymers into electrostrictive polymers has been by electron irradiation. Electron irradiation is the exposure to high-energy electrons. Electron irradiation of polymer films serves to break up the large crystalline regions of the polymer films into polar micro-regions resulting in a high-strain electrostrictive material.

1 Electrostriction is the high strains displayed by certain
2 materials when stressed by electric fields. The magnitude of
3 the electrostrictive strain can be described by the following
4 equation:

$$5 \quad S = QP^2,$$

6 where Q is the electrostrictive coefficient and P is the
7 polarization of the material.

8 Ferroelectric polymers such as poly(vinylidene fluoride-
9 trifluoroethylene) [P(VDF-TrFE)] films, previously annealed, can
10 be converted into electrostrictive polymers by exposure to high
11 energy electron bombardment. Electron bombardment of high
12 crystalline P(VDF-TrFE) films break up the long-range
13 ferroelectric region into polar micro-domains thereby broadening
14 the ferroelectric-to-paraelectric transition and moving the
15 transition to a lower temperature where high strains can be
16 observed when the films are driven by large electric fields.
17 These strains in the polymer films caused by electron
18 bombardment and the ensuing effects on the polymer structure can
19 be characterized by differential scanning calorimetry, X-ray
20 diffraction and infrared spectroscopy.

21 Ferroelectric polymers can contain various *trans* and *gauche*
22 configurations, including form I(β), II(α), and III(γ). In form
23 I, the chains exhibit an all-*trans* configuration. In form II,
24 the packed chains exhibit the *tgtg'* (t = *trans*; g, g' = *gauche*)

1 conformation, resulting in a nonpolar crystallite. In form III,
2 the chains exhibit *tttg'ttg'* conformation, resulting in a
3 monoclinic lattice and a polar cell.

4 Electron irradiation, i.e., electron bombardment, of these
5 ferroelectric polymers converts the polar all-trans form I(β),
6 long-range ferroelectric regions of annealed P(VDF-TrFE) films
7 into nanoregions consisting of coexisting I(β), II(α), and
8 III(γ) crystallites, preferably having Curie (polar-nonpolar
9 crystalline phase) transition at ambient temperatures. The
10 polarization of these regions give rise to a macroscopic
11 polarization and increase in the dielectric constant. The
12 macroscopic polarization provides an increase in dielectric
13 constant, large strains, much improved coupling constants and
14 large (d_{33}) signal piezoelectric constant.

15 Where the electrostrictive strains of materials are high
16 enough, materials having this property offer great promise in
17 applications such as sensors, underwater sonar transduction,
18 polymeric actuators, artificial muscles, and robotics. In these
19 and other applications, the high strain electrostrictive
20 materials provide higher/greater sensitivity, more powerful
21 signals and more efficient energy conversion.

22 However, electron irradiation, i.e., electron bombardment,
23 is cumbersome and expensive. Electron irradiation is also a
24 slow process because a large dose of radiation is needed to

1 achieve electrostrictive properties. Other disadvantages
2 associated with using electron irradiation which cause it to be
3 a slow process is that: (1) only a limited thickness of films
4 can be irradiated at a time; (2) the electron beam used is
5 narrow, while film size varies and can be much wider than the
6 electron beam; and (3) a vast fluctuation in conditions may
7 exist throughout the electron irradiation process.

8 The prior art discloses various polymers such as Nakamura
9 et al., U.S. Patent No. 4,543,293, which is said to disclose a
10 piezoelectric polymer comprising vinylidene fluoride,
11 trifluoroethylene and vinyl fluoride.

12 Also known in the prior art is Pantelis, U.S. Patent No.
13 4,557,880, which is said to disclose a piezoelectric film made
14 from vinylidene fluoride and tetrafluoroethylene and/or
15 trifluoroethylene.

16 Also known in the prior art is Sako et al., U.S. Patent No.
17 4,577,005, which is said to disclose a polymeric dielectric
18 material comprising a terpolymer which comprises vinylidene
19 fluoride, trifluoroethylene and hexafluoropropylene that is heat
20 treated.

21 Also known in the prior art is Preis, U.S. Patent No.
22 4,778,867, which is said to disclose a ferroelectric random

23

1 copolymer consisting essentially of vinylidene fluoride and
2 trifluoroethylene that is heat treated.

3 Also known in the prior art is Inukai et al., U.S. Patent
4 No. 5,087,679, which is said to disclose a polymeric dielectric
5 which comprises vinylidene fluoride, trifluoroethylene and
6 chlorotrifluoroethylene.

7 Also known in the prior art is Chung et al., U.S. Patent
8 No. 6,355,749, which is said to disclose a ferroelectric
9 terpolymer comprising vinylidene fluoride, trifluoroethylene and
10 chlorotrifluoroethylene or hexafluoropropene.

11 Also known in the prior art is Gervasi et al., U.S. Pub.
12 No. 2002/0132074, which is said to disclose a fluoroelastomer
13 terpolymer comprising vinylidene fluoride, hexafluoropropylene
14 and tetrafluoroethylene or chlorotrifluoroethylene.

15 Other ferroelectric and electrostrictive polymers and
16 methods of altering polymer morphology may be known. However,
17 these polymers and methods, along with those above, have various
18 shortcomings. These shortcomings are addressed by the present
19 invention. As such, the present invention provides
20 electrostrictive polymers using an alternative method of
21 altering copolymer morphology, without the need for electron
22 irradiation.

23

1 SUMMARY OF THE INVENTION

2 A primary objective of the present invention to produce an
3 electrostrictive terpolymer such as vinylidene fluoride-
4 trifluoroethylene-chlorofluoroethylene (VDF-TrFE-CFE), without
5 utilizing electron irradiation, which may be used as an active
6 material capable of generating sound in acoustic, underwater.
7 transducers and mechanical motion in actuator devices.

8 It is also a primary objective of the present invention to
9 produce an electrostrictive terpolymer such as VDF-TrFE-CFE
10 which may be used as a replacement for electron irradiated high-
11 strain P(VDF-TrFE) films.

12 It is also a primary objective of the present invention to
13 produce an electrostrictive terpolymer such as VDF-TrFE-CFE
14 which has *gauche*-type conformational defects along the polymer
15 chain which results in a broad distribution of polarizations
16 that favor higher electrostrictive strains than current
17 electrostrictive polymers.

18 It is also an objective of the present invention to produce
19 an electrostrictive terpolymer which exhibits larger mechanical
20 strains than known in the art.

21 It is also an objective of the present invention to process
22 an electrostrictive terpolymer such as VDF-TrFE-CFE which is
23 less expensive and less cumbersome than current electrostrictive
24

1 materials and methods of making those electrostrictive
2 materials.

3 In accordance with the present invention there is provided
4 a new class of terpolymers for use as high strain
5 electrostrictive polymer films. More particularly, the
6 invention relates to a class of terpolymers comprising at least
7 three monomers wherein their reaction produces terpolymers
8 having high electrostrictive properties. Specifically, the
9 electrostrictive terpolymers comprise vinylidene fluoride (VDF),
10 trifluoroethylene (TrFE) and at least one monomer having at
11 least one halogen atom side group. The monomer is preferably an
12 ethylene-based monomer and preferably selected to favor *gauche*-
13 type linkage along the polymer backbone. The halogen atom side
14 group is preferably bulky or large enough to move or cause
15 adjacent polymer chains to be farther apart from or away from
16 each other than in the absence of such halogen atom side group,
17 but not so large that it would inhibit polymer crystallites from
18 forming. The monomer is preferably a chloro-monomer such as
19 chlorofluoroethylene (CFE). The chlorofluoroethylene (CFE) is
20 preferably 1-chloro-2-fluoroethylene or 1-chloro-1-
21 fluoroethylene. The monomer may also be chlorotrifluoroethylene
22 (CTFE), but CTFE favors *trans*-type linkage instead of *gauche*-
23 type linkage. As such, electrostrictive terpolymers comprising
24

1 CFE have higher electrostrictive strains than terpolymers
2 comprising CTFE.

3 Other details of the high strain polymer of the present
4 invention, as well as other objects and advantages attendant
5 thereto, are set forth in the following detailed description and
6 accompanying drawings.

7

8 BRIEF DESCRIPTION OF THE DRAWINGS

9 Referring now to the drawings:

10 FIG. 1 is a graph illustrating the energy of *gauche* forms
11 and all-*trans* forms of a chlorinated chain polymer compared to a
12 non-chlorinated chain polymer;

13 FIG. 2 are graphs comparing the dihedral distributions
14 following a molecular dynamic simulation of 100 monomer chains
15 of $\text{-ClFC-CH}_2\text{-}$ (left) and $\text{-ClFC-CF}_2\text{-}$ (right);

16 FIG. 3 is a graph illustrating the dielectric constant
17 versus temperature for VDF-TrFE-CFE terpolymers of the present
18 invention at various frequencies;

19 FIG. 4 is a graph comparing the polarization versus the
20 applied electric field for VDF-TrFE-CFE terpolymers of the
21 present invention;

22 FIG. 5 is a graph illustrating the strain (in %) versus the
23 magnitude of an electric field (in MV/m) of two VDF-TrFE-CFE
24 terpolymers of the present invention; and

1 FIG. 6 is a graph illustrating the modulus and mechanical
2 loss tangent versus temperature for various frequencies for VDF-
3 TrFE-CFE terpolymers of the present invention.

5 DESCRIPTION OF THE PREFERRED EMBODIMENTS

6 Materials having high electrostrictive strains
7 are beneficial for use in applications such as sensors,
8 underwater sonar transduction, polymeric actuators, artificial
9 muscles, and robotics for providing higher/greater sensitivity,
10 more powerful signals and more efficient energy conversion. As
11 such, new electrostrictive materials and methods of synthesizing
12 these electrostrictive materials are being developed to replace
13 electron irradiated high strain polymer vinylidene fluoride-
14 trifluoroethylene [P(VDF-TrFE)] films while possessing all of
15 the electrostrictive properties of the electron irradiated high
16 strain P(VDF-TrFE) films.

17 Accordingly, the present invention is a new class
18 of terpolymers for use as high strain electrostrictive polymer
19 films. More particularly, the invention is a class of
20 electrostrictive terpolymers comprising vinylidene fluoride
21 (VDF), trifluoroethylene (TrFE) and at least one monomer having
22 at least one halogen atom side group. The monomer is preferably
23 an ethylene-based monomer and preferably selected to favor
24 *gauche*-type linkage along the polymer backbone.

1 The halogen atom side group is preferably bulky or large
2 enough to move or cause adjacent polymer chains to be farther
3 apart from or away from each other than in the absence of such
4 halogen atom side group, but not so large that it would inhibit
5 polymer crystallites from forming. In a preferred embodiment,
6 the halogen atom side group in the monomer is preferably
7 chlorine. The introduction of chlorine in the polymer chain
8 affects crystal packing during annealing by acting as a defect
9 that disrupts the polar all-trans long-range regions in the
10 polymer into nanoregions. Specifically, the introduction of
11 chlorine atoms into the polymer chains creates conformational
12 defects that provide the mechanism to break up the all-trans
13 long-range crystalline regions and disrupt the long-range
14 ferroelectric order, thereby converting these regions into
15 nanoregions.

16 The chlorine forces the crystalline dimensions to expand
17 and distort to accommodate the chlorine atoms. The disrupted
18 polar regions can be regarded as distorted defect structures
19 which give rise to random polar fields and electrostrictive
20 properties such as high strains. This effect is attributed to
21 the large van der Waals radius of the chlorine atom.
22 Specifically, the van der Waals radius of chlorine is 1.8 Å.

23 In a preferred embodiment, a chloro-monomer which can
24 convert VDF-TrFE polymer films into high-strain electrostrictive

1 films is chlorofluoroethylene (CFE), preferably 1-chloro-2-
2 fluoroethylene or 1-chloro-1-fluoroethylene. The chloro-monomer
3 may also be chlorotrifluoroethylene (CTFE), but CTFE favors
4 *trans*-type linkage. As such, electrostrictive terpolymers
5 comprising CFE have higher electrostrictive strains than
6 terpolymers comprising CTFE. FIG. 2 illustrates the dihedral
7 distribution of monomer chains of chlorofluoroethylene
8 (-ClFC-CH₂-) and chlorotrifluoroethylene (-ClFC-CF₂-). As shown,
9 chlorofluoroethylene (-ClFC-CH₂-) has higher frequencies at
10 *gauche*-type linkages, whereas chlorotrifluoroethylene
11 (-ClFC-CF₂-) has higher frequencies at *trans*-type linkages.

12 A chloro-monomer added to the VDF-TrFE copolymer provides
13 higher electrostrictive strains than non-chloro-monomers such as
14 hexafluoropropylene (HFP), which contains a trifluoromethyl side
15 group. The trifluoromethyl group is too large and too bulky and
16 gets annealed out of the crystallites. Because of the large
17 size of the trifluoromethyl group, HFP does not favor *gauche*-
18 type linkage along the polymer chain. As such, HFP produces
19 lower electrostrictive strains than the chloro-monomer in the
20 present invention when added to P(VDF-TrFE).

21 Even if the amount of HFP was varied, these results do not
22 change. If a small amount of HFP is used to form the
23 terpolymer, some of the trifluoromethyl groups may temporarily
24 be trapped within the crystallites, but over time, those groups

1 will be annealed out. As such, its performance decreases over
2 time. If a large amount of HFP is used to form the terpolymer,
3 the increased amount of HFP added to the VDF-TrFE copolymer
4 greatly reduces crystallinity, which leads to low polarization
5 and low strains. Accordingly, adding HFP to the VDF-TrFE
6 copolymer does not provide optimum electrostrictive properties.

7 As provided by the terpolymer of the present invention and
8 as shown in FIG. 1, adding a chloro-monomer which favors *gauche*-
9 type linkage to the VDF-TrFE copolymer synthesizes a terpolymer
10 having higher energy, i.e., higher electrostrictive properties
11 such as higher electrostrictive strains, than a terpolymer
12 synthesized by adding a non-chloro-monomer, such as HFP, as the
13 monomer. This result is due to the chlorine group on the
14 chloro-monomer being not too large to inhibit polymer
15 crystallites from forming, but large enough to push or move the
16 polymer chains farther apart from or away from each other than
17 in the absence of such chlorine group, thereby distorting the
18 polymer crystal lattice. The chloro-monomer CFE of the
19 preferred embodiment of the present invention favors *gauche*-type
20 linkage along the polymer chain which produces higher
21 electrostrictive strains.

22 While CTFE is a chloro-monomer, it favors *trans*-type
23 linkage along the polymer chain and does not result in the
24 highest electrostrictive strains possible, as shown in FIG. 2.

1 Alternatively, CFE is a chloro-monomer wherein its chlorine
2 group is large enough to push or move the polymer chains farther
3 apart from or away from each other than in the absence of such
4 chlorine group, thereby distorting the polymer crystal lattice,
5 but also favors the performance-enhancing *gauche*-type polymer
6 chain configurations, as shown in FIG. 2. Therefore, the
7 terpolymer VDF-TrFE-CFE of the present invention has side groups
8 (fluorine and chlorine) which are large enough to cause a
9 crystal lattice disruption, but small enough not to seriously
10 degrade crystallinity, thereby resulting in performance-
11 enhancing *gauche*-type polymer chain configurations having higher
12 polarization and higher electrostrictive strains.

13 The properties of the VDF-TrFE-CFE terpolymers were
14 determined by molecular dynamics simulations and
15 experimentation. The results of these simulations and
16 experimentation are shown in FIGS. 3-6. As such, the
17 terpolymers of the present invention exhibit a high dielectric
18 constant at ambient temperatures as shown in FIG.3. The
19 terpolymers of the present invention also exhibit large
20 electrical responses in ambient temperatures under electric
21 fields. As such, FIG. 4 illustrates the polarization versus the
22 applied electric field of the VDF-TrFE-CFE terpolymers. FIG. 5
23 illustrates the strains (in %) versus the electrical field (in
24 MV/m) for two examples of the terpolymers of the present

1 invention, as evidenced during simulations and experimentation.
2 FIG. 6 illustrates the modulus (MPa) and mechanical loss tangent
3 versus temperature for the VDF-TrFE-CFE terpolymers of the
4 present invention.

5 The terpolymer VDF-TrFE-CFE is preferably synthesized from
6 the polymerization of vinylidene fluoride (VDF),
7 trifluroethylene (TrFE) and chlorofluoroethylene (CFE),
8 preferably either 1-chloro-2-fluoroethylene or 1-chloro-1-
9 fluoroethylene. In a preferred embodiment of the terpolymer
10 VDF-TrFE-CFE, the amount of vinylidene fluoride (VDF) used
11 preferably ranges from about 65 mole % to about 71 mole %, more
12 preferably from about 66 mole % to about 70 mole %, and most
13 preferably from about 67 mole % to about 69 mole %. The amount
14 of trifluroethylene (TrFE) used preferably ranges from about 26
15 mole % to about 33 mole %, more preferably from about 27 mole %
16 to about 30 mole %, and most preferably from about 28 mole % to
17 about 29 mole %. The amount of chlorofluoroethylene (CFE) used
18 preferably ranges from about 1 mole % to about 6 mole %, more
19 preferably from about 2 mole % to about 5 mole %, and most
20 preferably from about 3 mole % to about 4 mole %. For example,
21 a VDF-TrFE-CFE terpolymer of the present invention may comprise
22 68 mole % VDF, 28 mole % TrFE and 4 mole % CFE.

23 The terpolymer is then subjected to either solvent casting
24 or extrusion and annealed, i.e., heated and then cooled. After

1 either solvent casting or extrusion and annealing, thin films of
2 VDF-TrFE-CFE are electrostrictive, i.e., the films exhibit large
3 mechanical strains when placed in an oscillating electric field.

4 Since crystallization into large regions is prevented, as
5 described above, the terpolymer VDF-TrFE-CFE anneals as a
6 disordered material with random defect fields underlying its
7 electrostriction. As such, the electrostrictive terpolymer VDF-
8 TrFE-CFE possesses *gauche*-type conformational defects along the
9 polymer chain that result in a broad distribution of
10 polarizations that favor higher electrostrictive strains than
11 other known electrostrictive polymers.

12 The electrostrictive terpolymer VDF-TrFE-CFE of the present
13 invention can be used as an electrostrictive material in its
14 annealed state without being subjected to electron irradiation.
15 Since the chloro-monomer added to the P(VDF-TrFE) produces
16 electrostrictive properties, i.e., electrostrictive strains,
17 greater than those strains produced by electron irradiation of
18 P(VDF-TrFE), the VDF-TrFE-CFE terpolymer of the present
19 invention solves the problems associated with processing
20 polymers into electrostrictive materials by electron
21 irradiation.

22 The electrostrictive terpolymer VDF-TrFE-CFE of the present
23 invention is also conformable, robust, and chemically durable
24 which makes it good for use in hostile environments. The

1 electrostrictive terpolymer VDF-TrFE-CFE may preferably be used
2 as an active material capable of generating sound in acoustic,
3 underwater transducers and mechanical motion in actuator
4 devices. Specifically, the electrostrictive terpolymer VDF-
5 TrFE-CFE is applicable in sensors, sonars in submarines, in
6 actuators and in smart skins of vehicles or materials which are
7 used to sense vibration and control noise, such as in stealth
8 jets and submarines. In these and other applications, the
9 electrostrictive terpolymer provides higher/greater sensitivity,
10 more powerful signals and more efficient energy conversion.

11 The processing of the electrostrictive terpolymer VDF-TrFE-
12 CFE of the present invention is less expensive and less
13 cumbersome than processing electrostrictive polymers by electron
14 irradiation or other known technologies. In addition, the
15 processing of the electrostrictive polymer VDF-TrFE-CFE of the
16 present invention produces electrostrictive polymers which
17 exhibit larger mechanical strains than other known
18 electrostrictive materials.

19 The exemplary embodiments herein disclosed are not intended
20 to be exhaustive or to unnecessarily limit the scope of the
21 invention. The exemplary embodiments were chosen and described
22 in order to explain the principles of the present invention so
23 that others skilled in the art may practice the invention. As
24 will be apparent to one skilled in the art, various

1 modifications can be made within the scope of the aforesaid
2 description. Such modifications being within the ability of one
3 skilled in the art form a part of the present invention and are
4 embraced by the appended claims.

1 Attorney Docket No. 83303

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3 HIGH STRAIN PIEZO-POLYMER

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5 ABSTRACT OF DISCLOSURE

6 The present invention is a new class of terpolymers for use
7 as high strain electrostrictive polymer films. More
8 particularly, the invention is a class of electrostrictive
9 terpolymers comprising vinylidene fluoride (VDF),
10 trifluoroethylene (TrFE) and at least one monomer having at
11 least one halogen atom side group. The monomer is preferably an
12 ethylene-based monomer and preferably selected to favor *gauche*-
13 type linkage along the polymer backbone. The halogen atom side
14 group is preferably large enough to move or cause adjacent
15 polymer chains to be farther apart from or away from each other
16 than in the absence of such side group, but not so large that it
17 would inhibit polymer crystallites from forming. The monomer is
18 preferably a chloro-monomer such as chlorofluoroethylene (CFE).
19 The chlorofluoroethylene (CFE) is preferably 1-chloro-2-
20 fluoroethylene or 1-chloro-1-fluoroethylene. The
21 chlorofluoroethylene (CFE) favors *gauche*-type linkage which
22 favors high electrostrictive strains.

FIG. 1

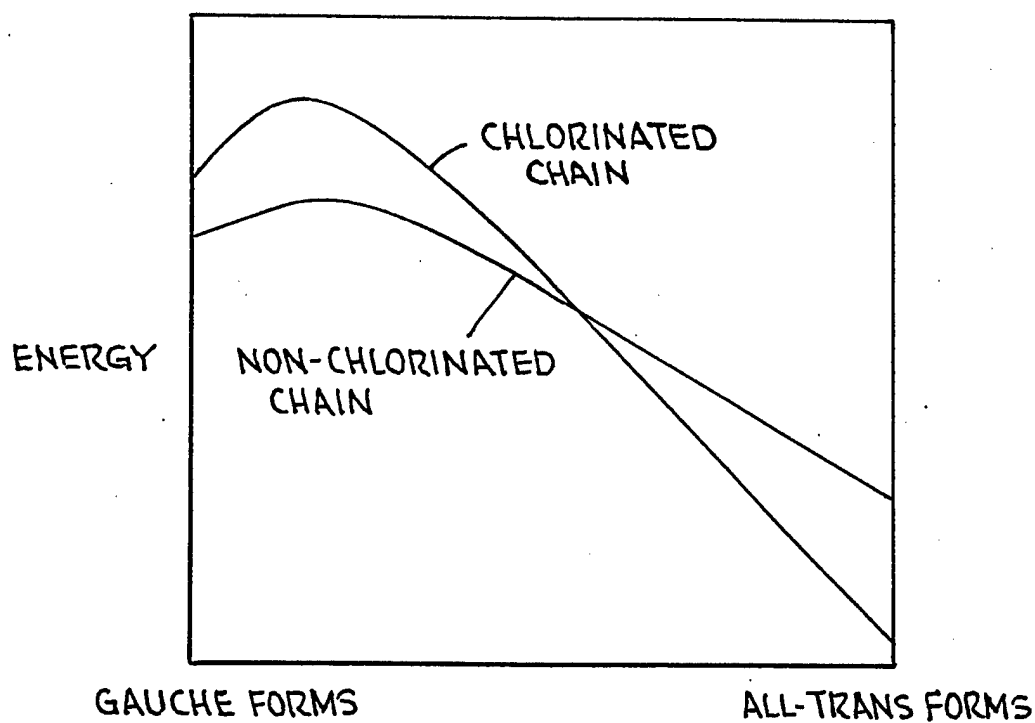


FIG. 2

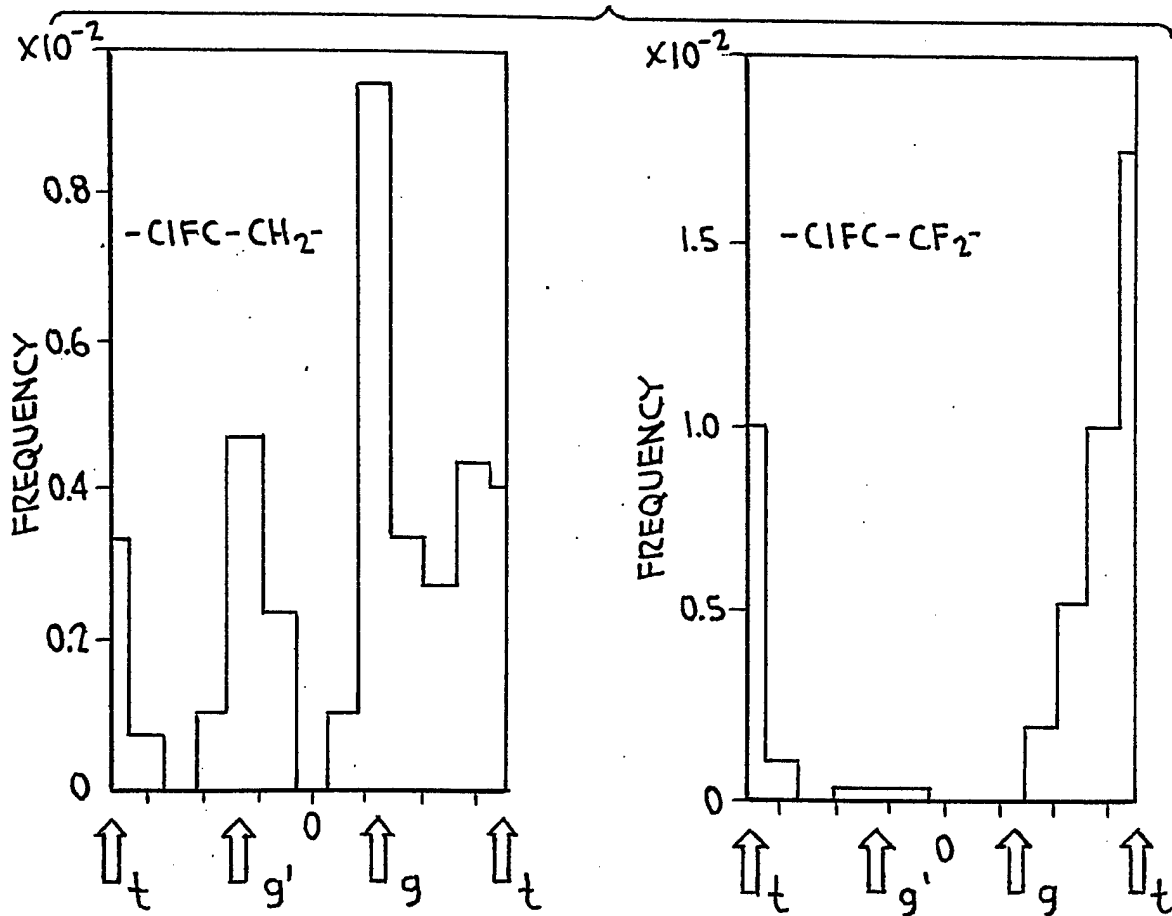


FIG. 3

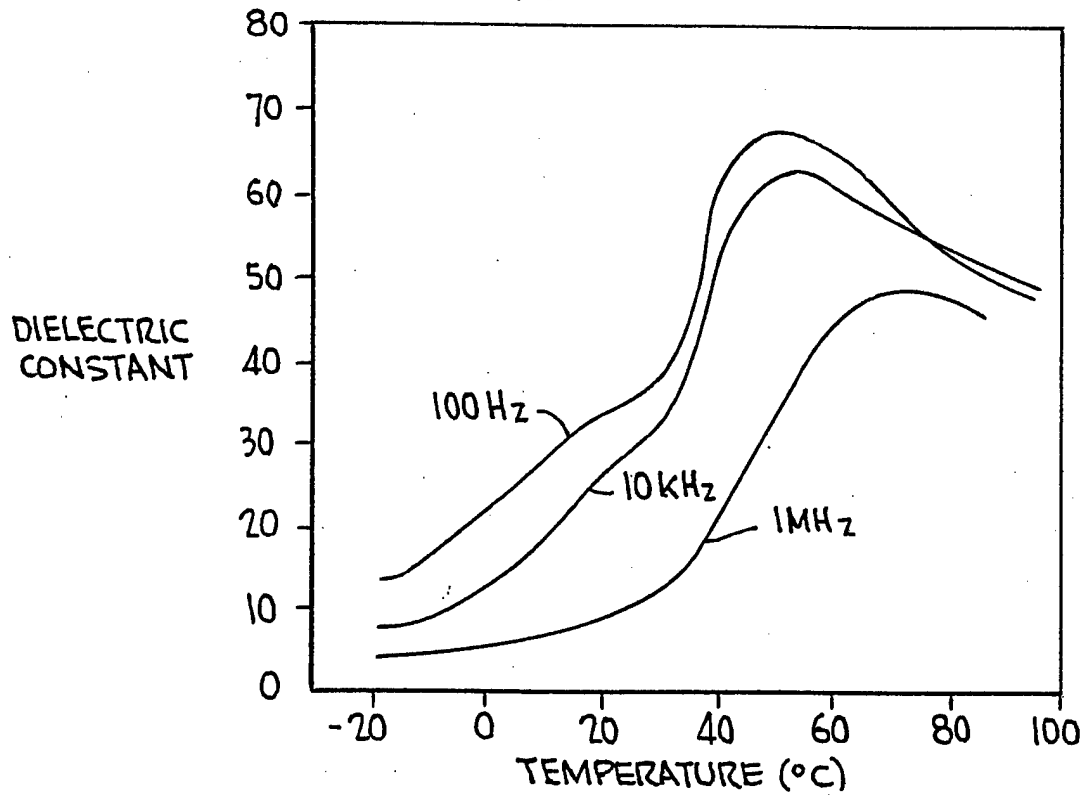


FIG. 4

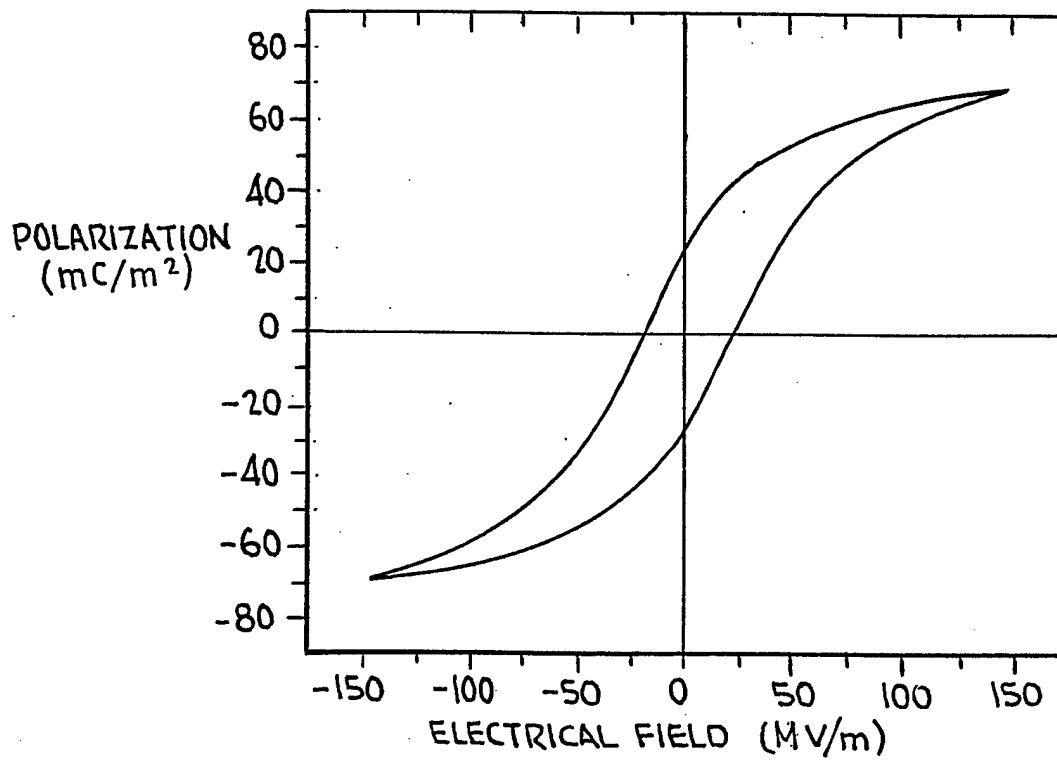


FIG. 5

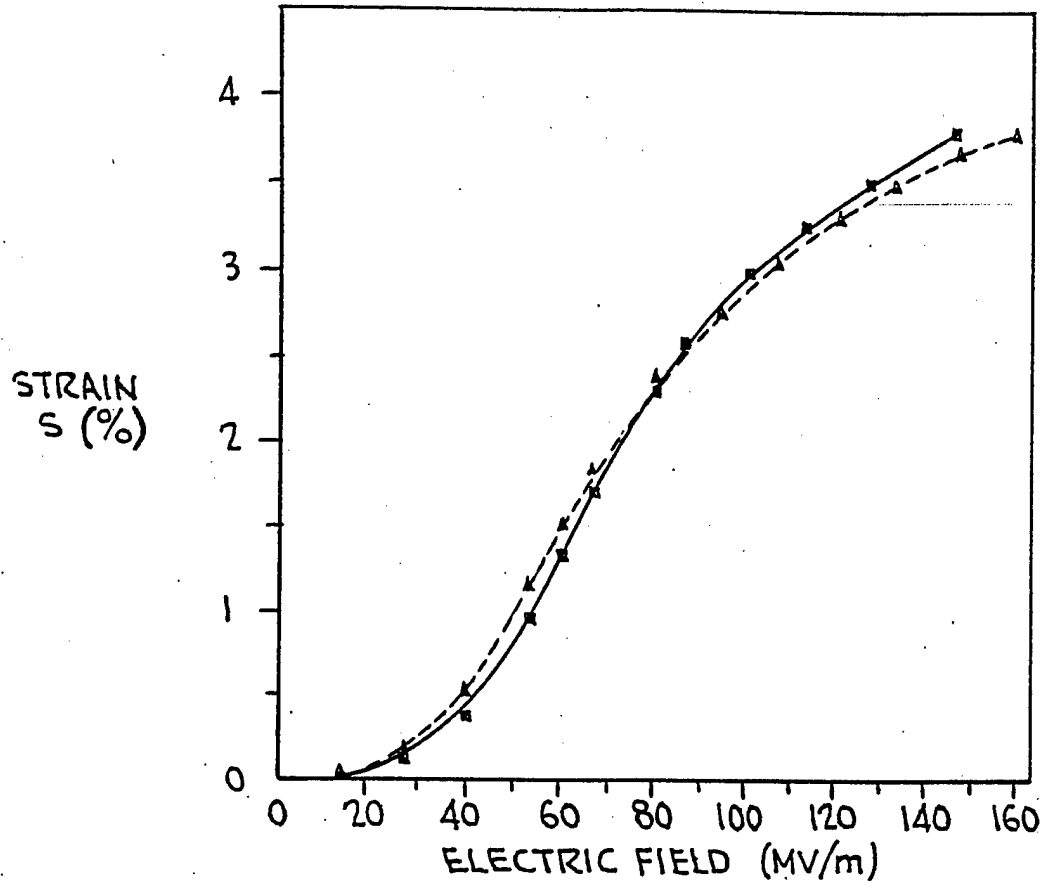


FIG. 6

