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Kevin P. Shambrook, Ph.D.

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13. ABSTRACT (Maximum 200 words)

Ultraconductor™ polymer films have exceptional conductivity and current densities, however, at present these features are strongly anisotropic. Under this Phase I contract, a key component of a new fabrication method was successfully tested for feasibility. The technique enables the controlled patterning of conducting paths in the film's X – Y axis. The method leads to a scalable system for fabricating very high conductivity Ultraconductor materials for applications ranging from electronic interconnects to tape and windings.

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F33615-98-C-1338
Exhibit A
Page 1 of 2

BMDO PHASE I FINAL REPORT
by Kevin P. Shambrook Ph.D., Principle Investigator
Contract: F 33615-98-C-1338
Topic: BMDO98 - 016

FINAL REPORT

Patterning of Highly Conducting Microstructures in Ultraconductor™ Polymer Films.

CONTENTS

A. SUMMARY

This report describes results obtained under the Phase I SBIR contract, **Patterning of Highly Conducting Microstructures in Ultraconductor™ Polymer Films.** The contract's goal was to test a method for manipulating the inherent conducting structures of Ultraconductor polymer materials, as a means for making patterned conducting paths in the X- Y planes of Ultraconductor films.

The contract goal was successfully achieved. The conducting structures were moved and aligned as had been proposed, resulting in the formation of various configurations of X -Y patterns or "stripes". A model was also developed to explain the experimental results, with important consequences for the next phase.

In particular, the experiments clearly confirmed that electrophoresis techniques are feasible methods for Ultraconductor fabrication.

Ultraconductors are patented polymers with exceptional features, including very high electrical conductivity (more than 100,000 times higher than metals), and high current carrying capacity ($> 5 \times 10^8$ Amperes/cm^2). These features are the basis of the materials' significant commercial value for a range of electric products.

The Ultraconductor's measured properties occur in microstructures which form in the polymer after processing. In films, a proportion of these structures extend through both film surfaces, can be electrically contacted, and are called "channels".

When formed in films, the conducting channels are also highly anisotropic in the Z axis (normal to the plane of the film). This format has applications value (for example as an anisotropic interposer for the fine pitch mounting of flip-chips, presently under development at ROOTS).

However, the polymers' full applications potential can be realized when the channels are formed in the X - Y plane, or otherwise manipulated to create long conducting filaments and wire. This contract investigated and characterized such a manipulation technique.

Under the contract, a full series of tests were performed which showed clearly that the conducting channel structures can be moved and patterned in the plane of the film by electric fields.

The movement of the structures is described in this report, with images of the formation of patterns (stripes) as seen under phase contrast microscope. A theory is also presented to explain the results. This work is seen as a significant validation,

and a benchmark step toward the controlled fabrication of long conducting paths.

In this Phase, conductivity was not measured along the formed stripes. No conductivity was expected in this Phase, which was designed to characterize the motion and alignment of the structures when subjected to planar electrodes. Planar electrodes successfully mobilize and aggregate the conducting structures, but in a parallel mode. The subsequent step (proposed for the Phase II project) will be aligning the structures end to end so that the high conductivity will be continuous in the X - Y channel structures. (This type of axial conducting structure has been previously formed in the Z axis, using point electrodes.)

The Phase II manipulation of these structures under complex or additional electrode configurations will result in longer conducting lengths of high commercial value.

B. TECHNOLOGY
B1. INTRODUCTION

The contract investigated the effect of electric fields on the movement and alignment of the conducting structures (called channels) in Ultraconductor films.

B1. a) Channels

Ultraconductor channels can be observed by several means, including Atomic Force Microscope (see Appendix) and simple electric contact. They are typically 1 - 2 microns in diameter, with aspect ratios on the order of 10:1. Each channel can carry currents of over 50 amperes.

Importantly for the purposes of this contract, the conducting channels are also observable by phase contrast microscope.

In films, the conducting channels are naturally anisotropic, forming as axial structures normal to the plane of the film.

It had been previously demonstrated that externally applied field gradients will induce migration and aggregation of the conducting channels in the viscous polymer films. This translational motion is understood to result from a field-induced dipole moment in the conducting structures, and consequently, their polarization in relation to the field's gradient.

Based on experiments, it was hypothesized that "stripes" (ie, linear configurations of the conducting structures, aligned in the X - Y plane of the film) could be formed with a proper electrode configuration. This Phase I contract was to investigate that controlled patterning.

B1. b) Experimental Approach
The contract approach involved application of thin metallic electrodes to a substrate,

2

leaving a gap between them, followed by the application of the Ultraconductor polymer. This introduced Ultraconductor film into the gap, which was the locus of the applied fields.

The planar electrodes were made of aluminum foil (20 microns thick, nominal) applied to glass slides. Passive (not processed to a conducting state) dielectric polymer films were first deposited on the slides and then processed by the Ultraconductor methodology. The processing results in the formation of the electrically conducting channels (after which the polymer is considered 'active').

A large voltage gradient parallel to the plane of the substrate was then produced in the gap between electrodes.

Because the conducting channels in Ultraconductor films are aligned normal to the plane of the substrate, the applied gradient between electrodes was normal to the long axis of the conducting channels in the film.

B2. EXPERIMENTS
B2. a) The apparatus

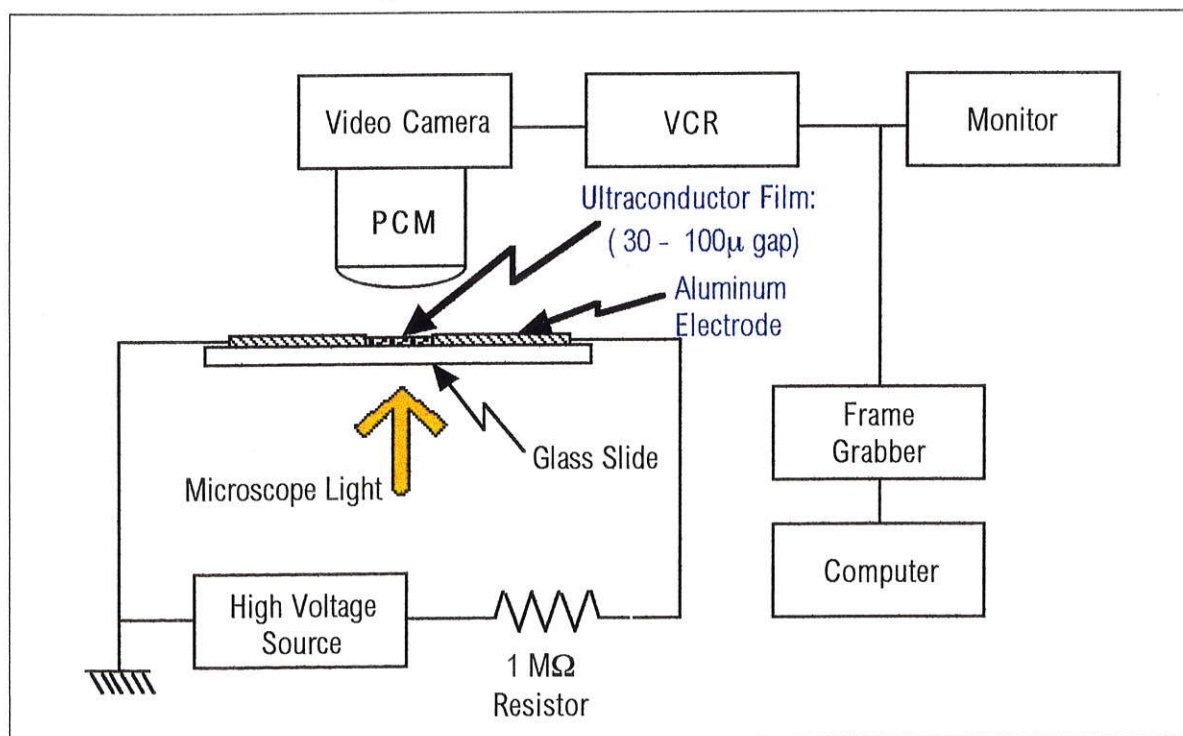Figure 1 shows a block diagram schematic of the experimental set-up.

FIGURE 1. SCHEMATIC OF EXPERIMENT
(PCM = PHASE CONTRAST MICROSCOPE

3

The phase contrast microscope (described in more detail in Appendix E) essentially processes light in such a way as to image the density of the sample. The sample, shown in plan in Fig 2, is a thin film of Ultraconductor (1 - 3 microns thick) between two electrodes. Typically 4 of these samples were made on each microscope slide, and each sample was subsequently contacted and viewed one at a time.
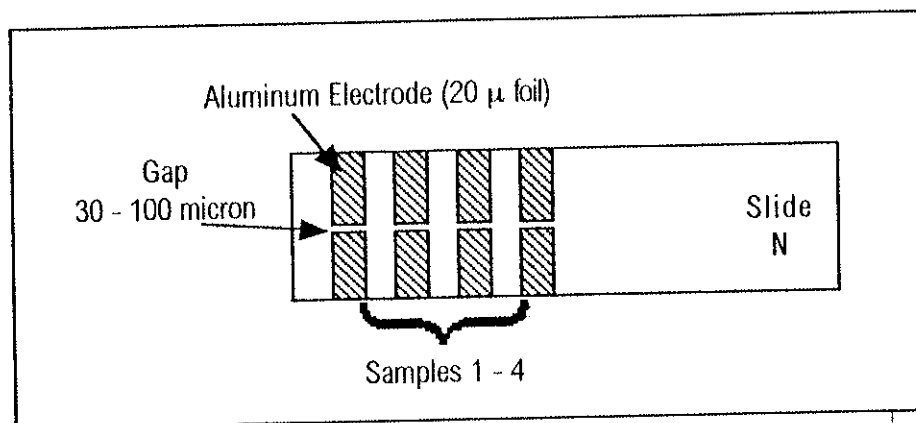

FIGURE 2.   PLAN VIEW OF A SLIDE

The phase contrast microscope image was captured by a high resolution video camera, and recorded on the VCR.   Later the VCR images were reviewed and the ones of interest entered into the computer.

Many experiments were required to optimize the preparation of electrodes and slides, to choose a suitable microscope and magnification, and to build the fixtures necessary to hold the slides and apply voltage.

In addition to these steps, a two axis micropositioner was attached to the microscope stage, to hold a specially designed probe. The probe (Figure 3), used to sample several stripes for conductivity along the stripe axis, was constructed from two parallel tungsten wires sharpened to a small radius by etching, with a 30 micron gap.
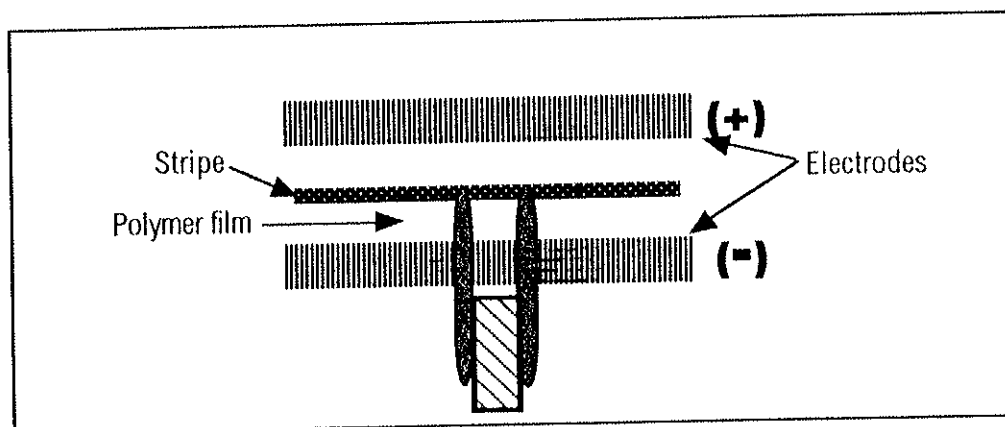

FIGURE 3. TUNGSTEN PROBES (separation ~ 30μ)

B2. b) The process

The experimental process for each sample was to apply a voltage to the electrodes and record changes in the polymer sample, as imaged by the phase contrast microscope.

The voltage was then increased in steps, leaving the sample at each voltage for 30 - 60 minutes. When there was a noticeable change at any point, the VCR was left on continuously monitoring that point. Since it was occasionally not possible to image the entire length of the electrode edge (due to magnification) only before and after images were available on certain runs.

B2. c) Controls
Several experimental controls were used to isolate the phenomenon being tested.

B2. c.1) Control samples
To ensure that patterning was solely an effect of the manipulation of the conducting structures, the above procedure was applied to sets of samples before they had been processed to create conducting structures. None of these 'passive' controls were conductive, and when tested in the apparatus, none showed any evidence of formation of stripes. When the same samples of polymer were later processed to a conducting (active) state, the samples had positive patterning results.

B2. c.2) Control voltages
The above procedure was applied to active samples, but with ac voltage (60 Hz) instead of dc. No stripes were formed.

In order to exclude the possibility of dielectric breakdown under voltage, polymer samples in the test apparatus were taken through voltages well above those used for the experiments, until breakdown occurred.

The effects of breakdown were obviously distinct from the striped patterns (eg, breakdown occurred between electrodes, not parallel to the electrodes in the gap; breakdown left no linear structures, etc.). Also, breakdown is an "avalanche" process which occurs in extremely short (microsecond) time scales. On the contrary, the stripe formation and migration clearly evolved and showed reversible responses to changes (both increases and decreases) in voltage.

C.) EXPERIMENTAL RESULTS

A large number of samples were subjected to the procedure. Nearly all of the active samples clearly showed the formation of structures - dark and light stripes[1]. Several samples (presumed active) showed no effects.

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[1] Dark and light features under the phase contrast microscope correspond to areas of higher material density. Higher density is a specific and unique characteristic of the conducting structures in Ultraconductor polymer films, which are otherwise amorphous and relatively homogeneous.

Typically, discernible effects were first visible in the phase contrast microscope when voltages exceeded 400v. (Based on the test results, at lower voltages the patterns were likely very close to the electrode and consequently masked from the microscope by the height of the electrode and normal light diffraction.)

Above that voltage, stripes could be observed. The stripe patterns could be generally divided into two categories: curvilinear and straight, though fine variations also occurred (described below). The curvilinear, or rounded, stripes formed adjacent to point-like asperities which extended away from the edge of the electrode into the gap. The straight or line stripes occurred along straight sections of the electrode.

Overall, the patterns were characteristically observed to conform to the shape of the local electrode edge; or more specifically, to the shape of the electric field produced locally by the electrode in the gap.

Fine variations in dimension, density, continuity and shape were typical of most samples, visible at different points along a single stripe. These variations usually corresponded to the fine shape of the electrode edge (and consequently, the strength of the local field gradient). Some variation was also likely due to differences in the local radius of the electrode edge, but this could not be confirmed with the phase contrast microscope.

The stripe patterns also showed characteristic formation stages as a function of the applied voltage. Nucleation was always at or adjacent to the positive electrode, with the pattern moving away from the positive electrode under increasing voltages.

Dark stripes were often flanked by white stripes. Since both equally indicate a relative change of density as seen by the phase contrast microscope (in fact the black and white were easily reversed by a change of focus) they can be considered as a unit.

C. 1) Experiment images

Representative images from the two types of stripe are presented here. (It is important to note that the following sequential illustrations are not time elapsed photos, but show the shapes as they appeared at the designated voltage. The times in the image captions are the time spent at the voltage, not the time of formation of the stripe. Stripes were typically persistent at the applied voltage, and changed when another voltage value was applied.)

The Figure M1, M2 series shows clearly how a curvilinear pattern or stripe has been formed relative to an electrode asperity. More important, the sequence shows the positioning and development of the stripe as a function of voltage, first nucleating on the positive electrode and gradually moving away from the electrode (in this case a corner). Figures M3, M4, M5, illustrate the second type, showing relatively straight lines, where a long stripe has formed at a straight section of the positive electrode and migrated toward the negative electrode under increasing electric fields.
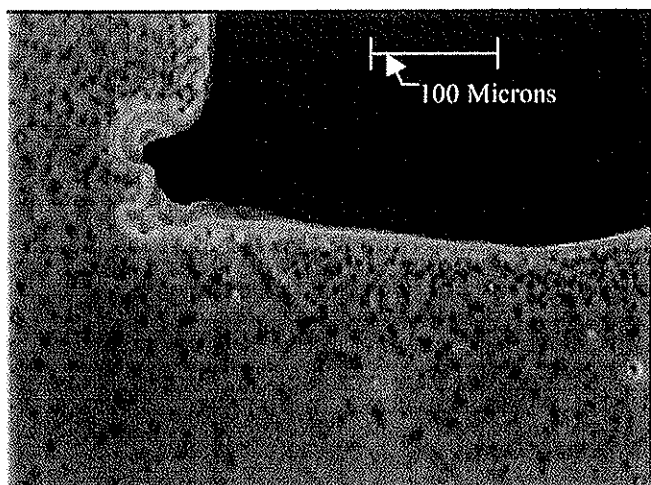
Figure M1a. Slide 22; Sample 2; V = 500, 2 min
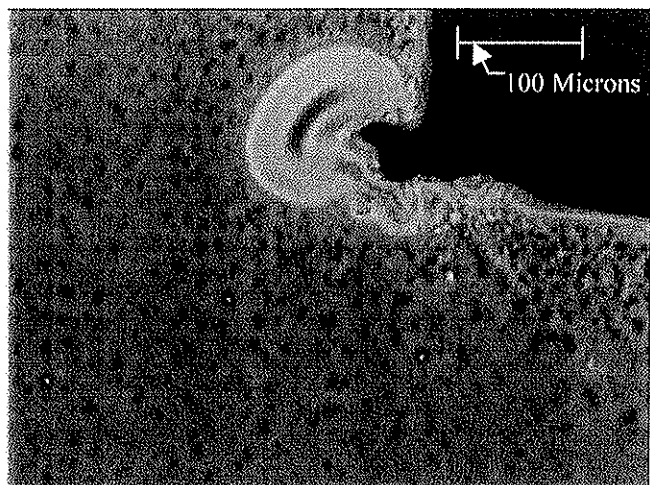

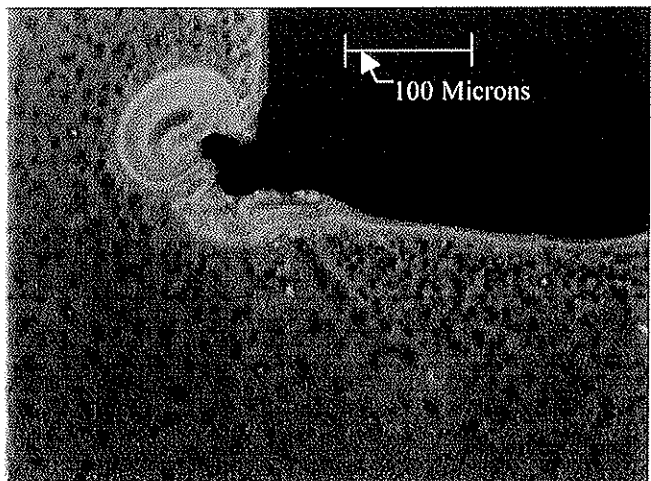Figure M1d. Slide 22; Sample 2; V = 1000, 2 min

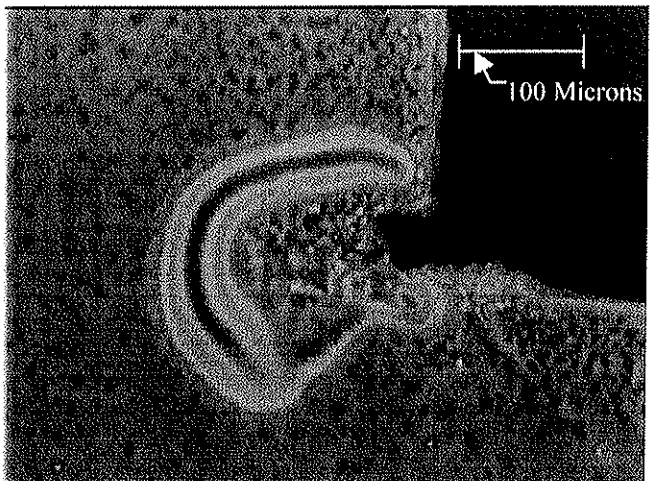Figure M1b. Slide 22; Sample 2; V = 700, 12 min

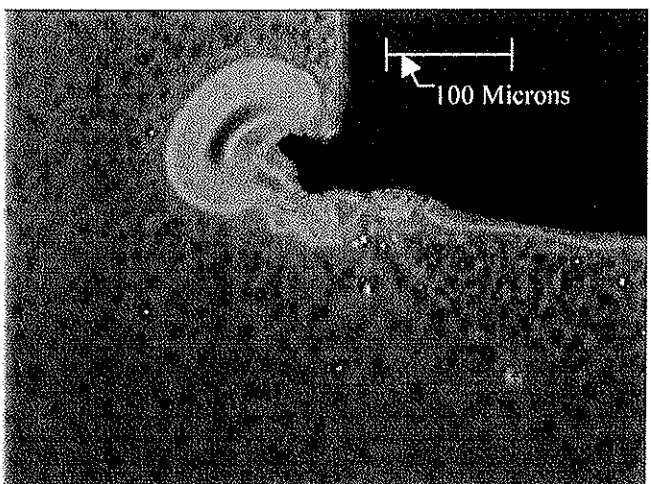Figure M1e. Slide 22; Sample 2; V = 1200, 4 min

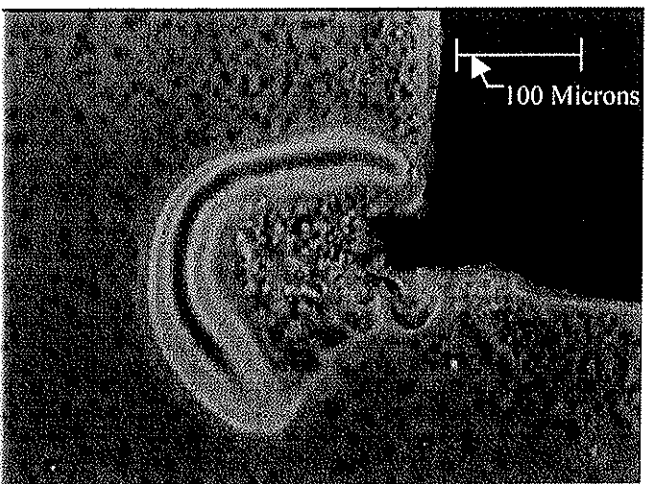Figure M1c. Slide 22; Sample 2; V = 900, 15 min

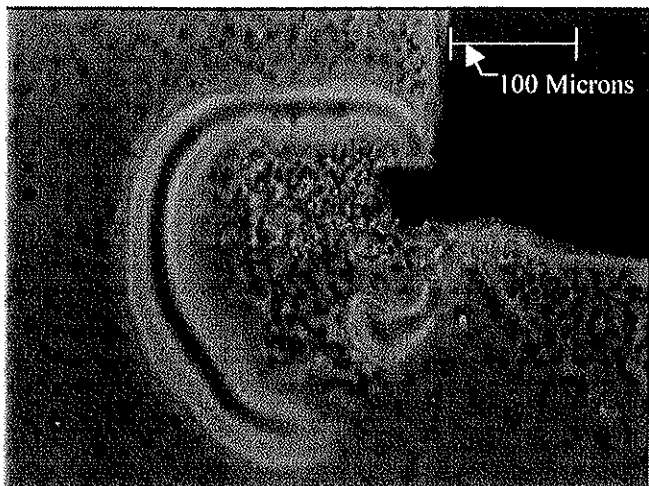Figure M1f. Slide 22; Sample 2; V = 1400, 1 min

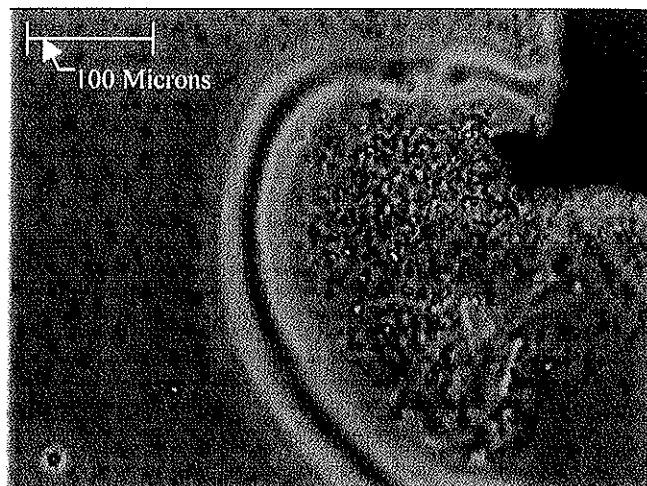Figure M2a. Slide 22; Sample 2; V = 1400, 2 min 27 sec

Figure M2d. Slide 22; Sample 2; V = 1500, 5 min
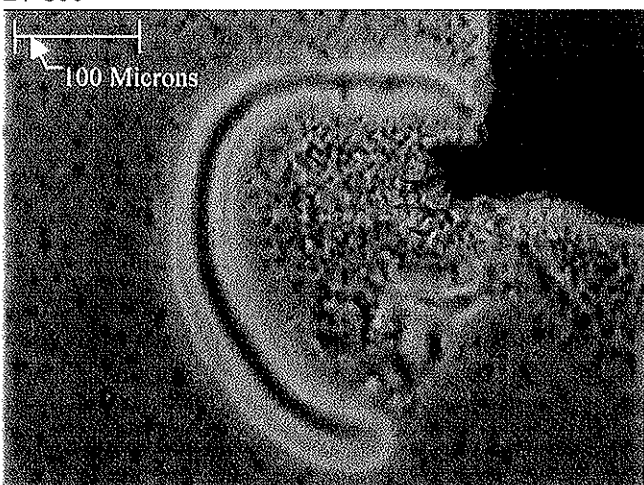
Figure M2b. Slide 22; Sample 2; V = 1500

Figure M2e. Slide 22; Sample 2; V = 1500, 5 min 20 sec

Figure M2c. Slide 22; Sample 2; V = 1500 26 sec

Figure M2f. Slide 22; Sample 2; Voltage off
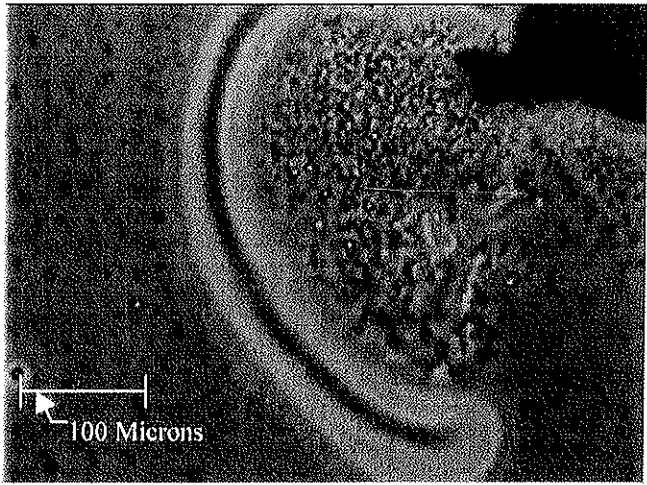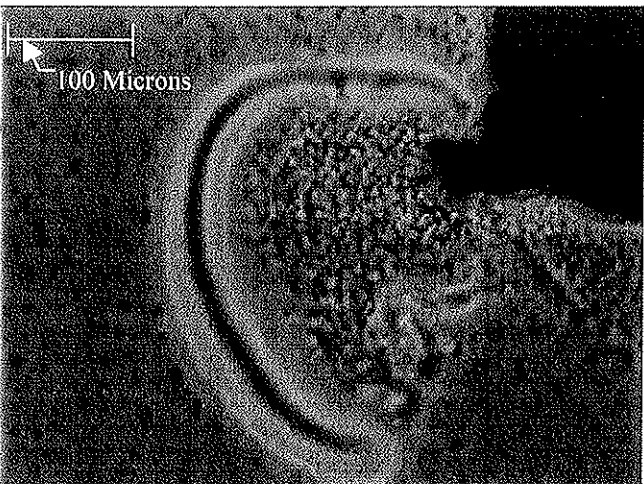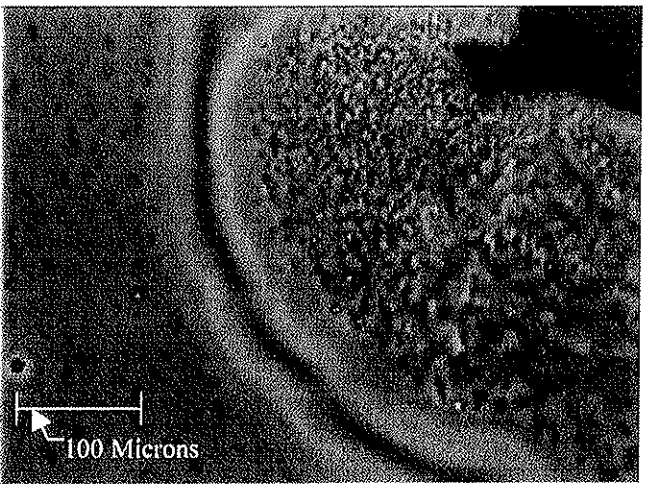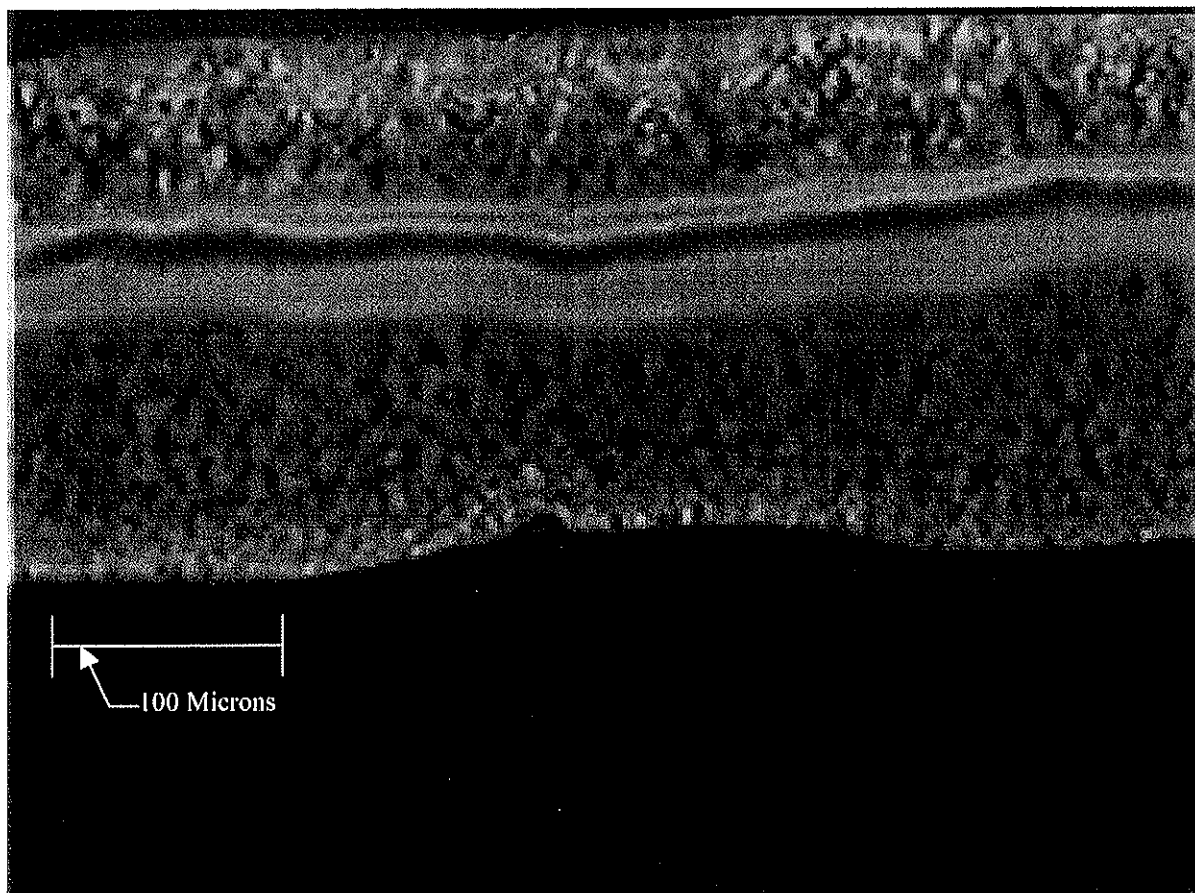
Figure M3a. Slide 22; Sample 1; V = 700, Duration 15 min.
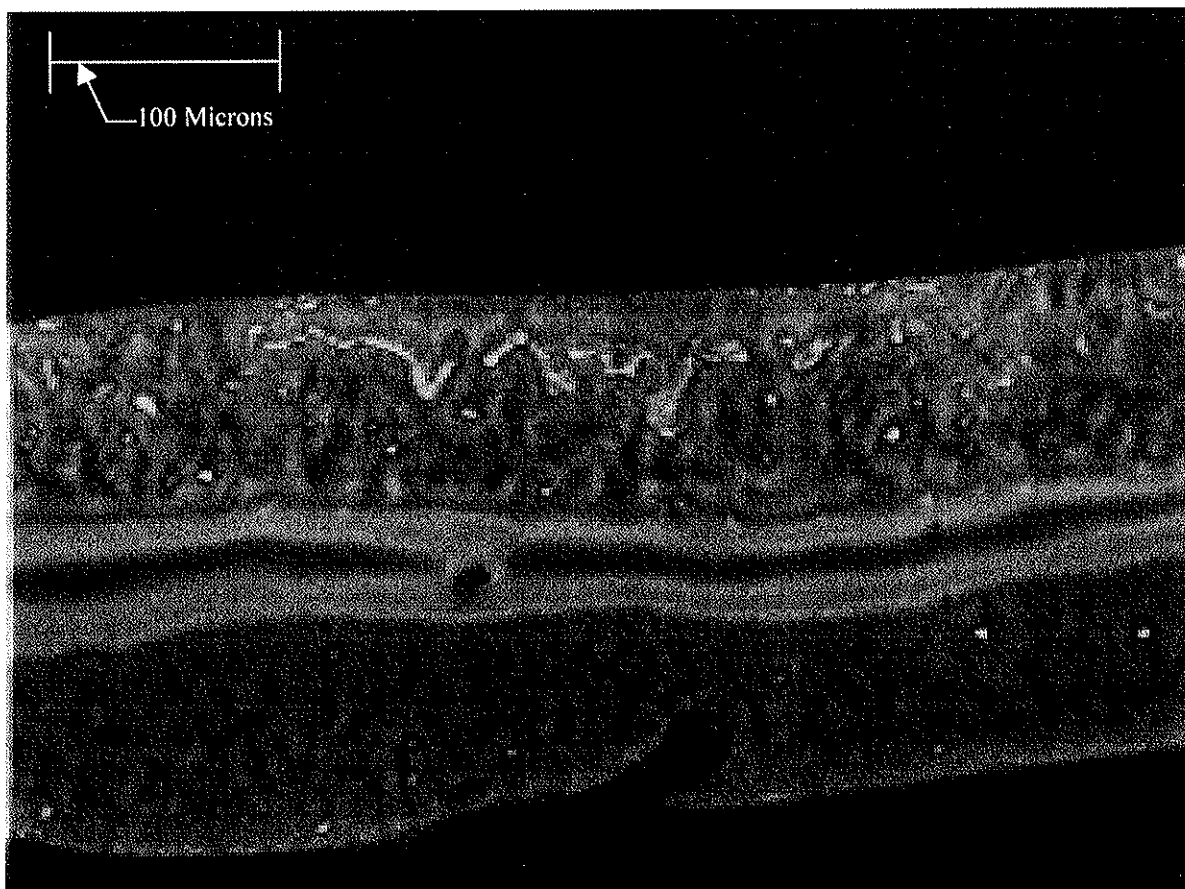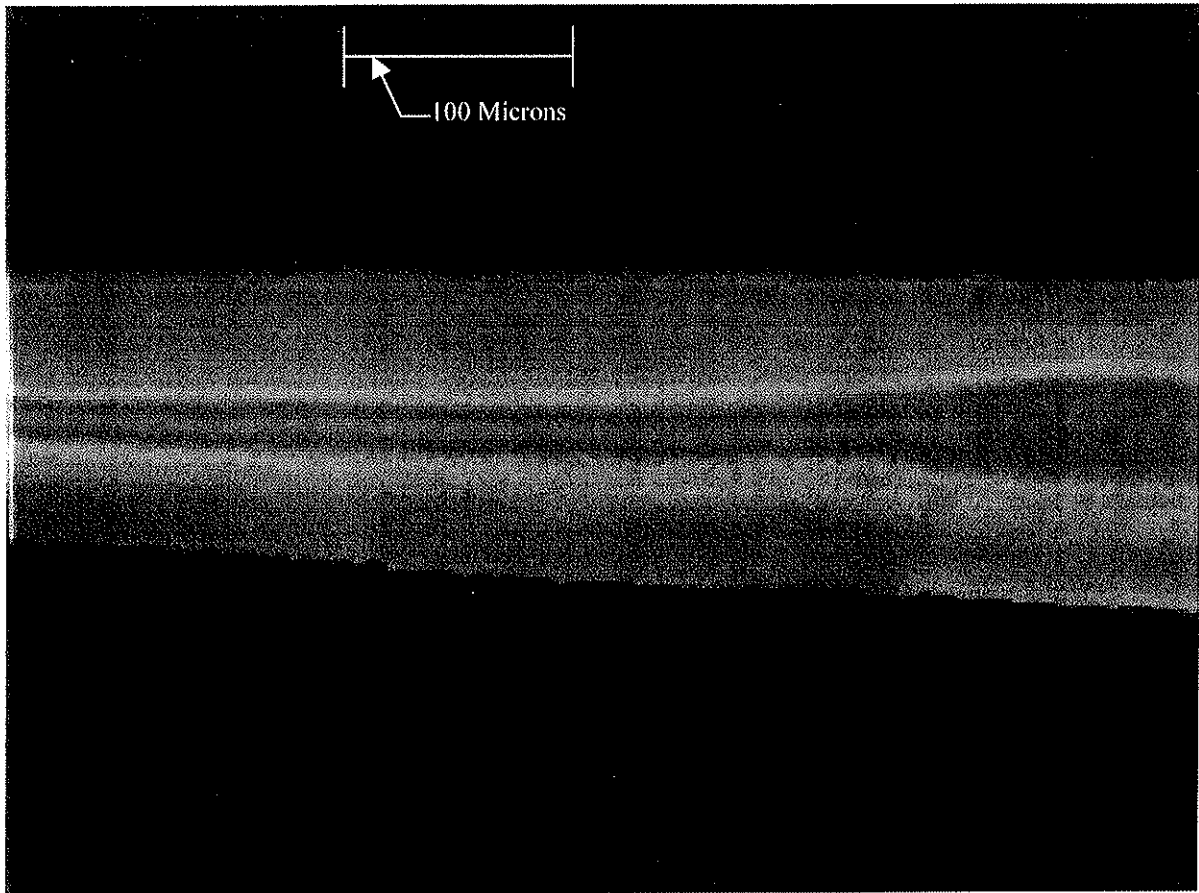

Figure M3b. Slide 22; Sample 1; V = 800; Duration 9 min

100 Microns

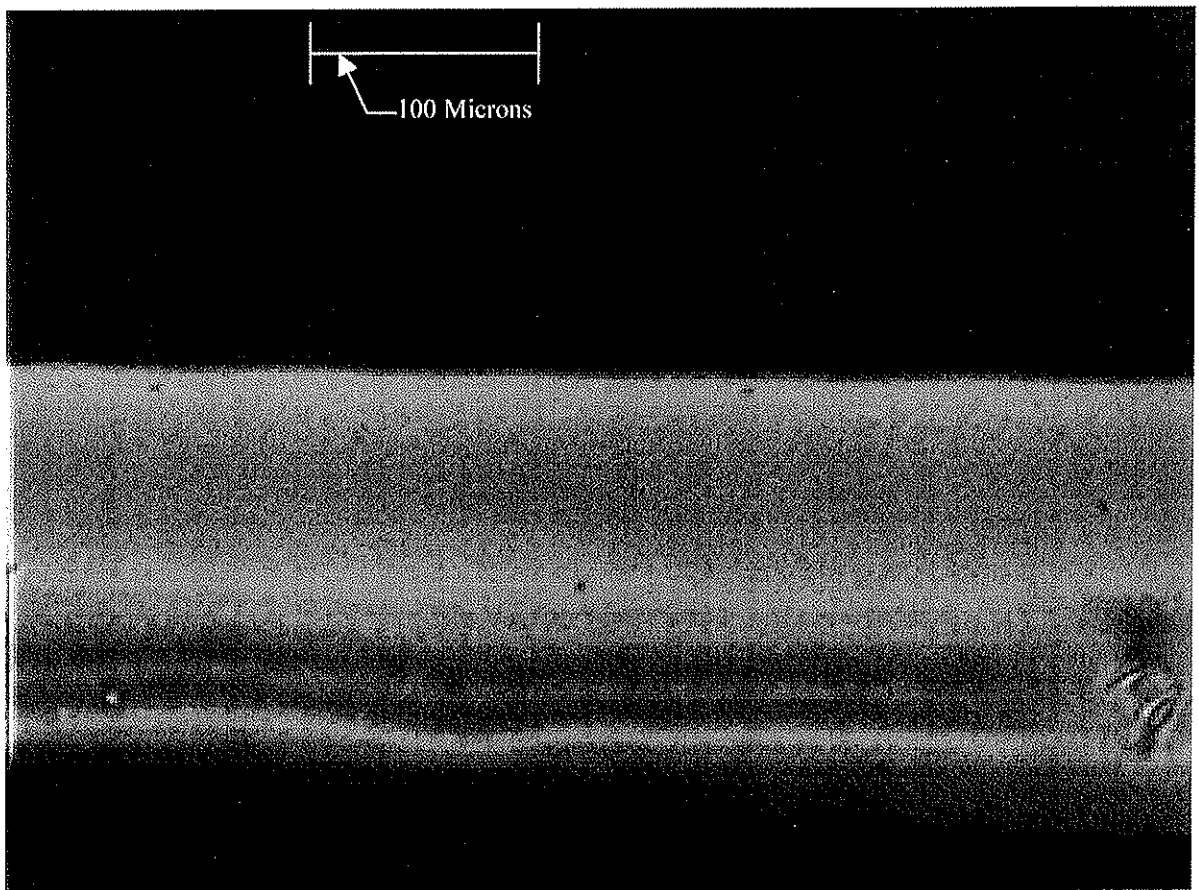Figure M4a. Slide 14; Sample 1; V = 500, 10 min.

100 Microns

Figure M4b. Slide 14; Sample 1; V = 720, 5 min.

In both types of stripe, it was evident that the structure reached an equilibrium position in the gap (relative to the positive and negative electrodes) which was determined by the value of the voltage applied.

C. 3) Experiment summary

The experiments can be summarized by the following. The application of local electric field gradients were observed to produce new structures of relatively large scale in active Ultraconductor polymers. These new structures were completely absent in passive polymer films used as controls. The new structures were observed under phase contrast microscope, indicating they are comprised of significantly higher material density than the background polymer media. That characteristic is signature of the conducting channel structures, which can be imaged by phase contrast microscope.

Consequently, the patterned stripes are understood to be conducting channels as aligned in relation to the applied field. (This phenomenon had also been previously demonstrated under different configurations of electrodes.) The formation, shape and alignment of the stripes were directly affected by the value of the local field.

D. MICROSTRUCTURE MODEL

A model of a possible mechanism to explain the experimental results is described here. As a qualitative model it is fairly simple, but not so simple to analyze quantitatively (see Appendix A).

D. 1 Review of channel formation model

Before applying this mechanism to the interpretation of the results, it will be useful first to review the model of Ultraconductor channel composition and formation.

The formation of conducting channels in Ultraconductor polymers results from an electronically driven, self-assembly of charges and polymer molecular chains. This self-assembly results from interactions due to diffusion in the polymer material (which is essentially a slow moving polar liquid). (See Appendix D for a detailed presentation.)

The sequence is initiated by mild UV irradiation of the polymer, which creates ions and free charges (electrons). These charges become solvated by polymer dipoles (polar groups), which results in the formation of stable polarons (dipoles surrounding an electron). Over time, due to their motion in the viscous polymer, the polarons contact each other and combine to form mesoscopic components called *superpolarons.*

The resulting superpolarons are prolate cylinders of approximately 200 - 500 angstroms diameter, formed of polymer molecular chains surrounding an extremely condensed, quasi-one dimensional electron 'string' (Figure 4). Over time in the viscous polymer material, the superpolarons themselves begin to aggregate, creating larger structures (Figure 5). A proportion of these structures gain sufficient dimensions to reach from film substrate to surface, and are called channels.

When formed in present Ultraconductor films, these channels show consistent characteristics: they are typically anisotropic, exceptionally conductive, with aspect ratios on the order of 10:1, and are extremely durable over ranges of temperature and high currents.
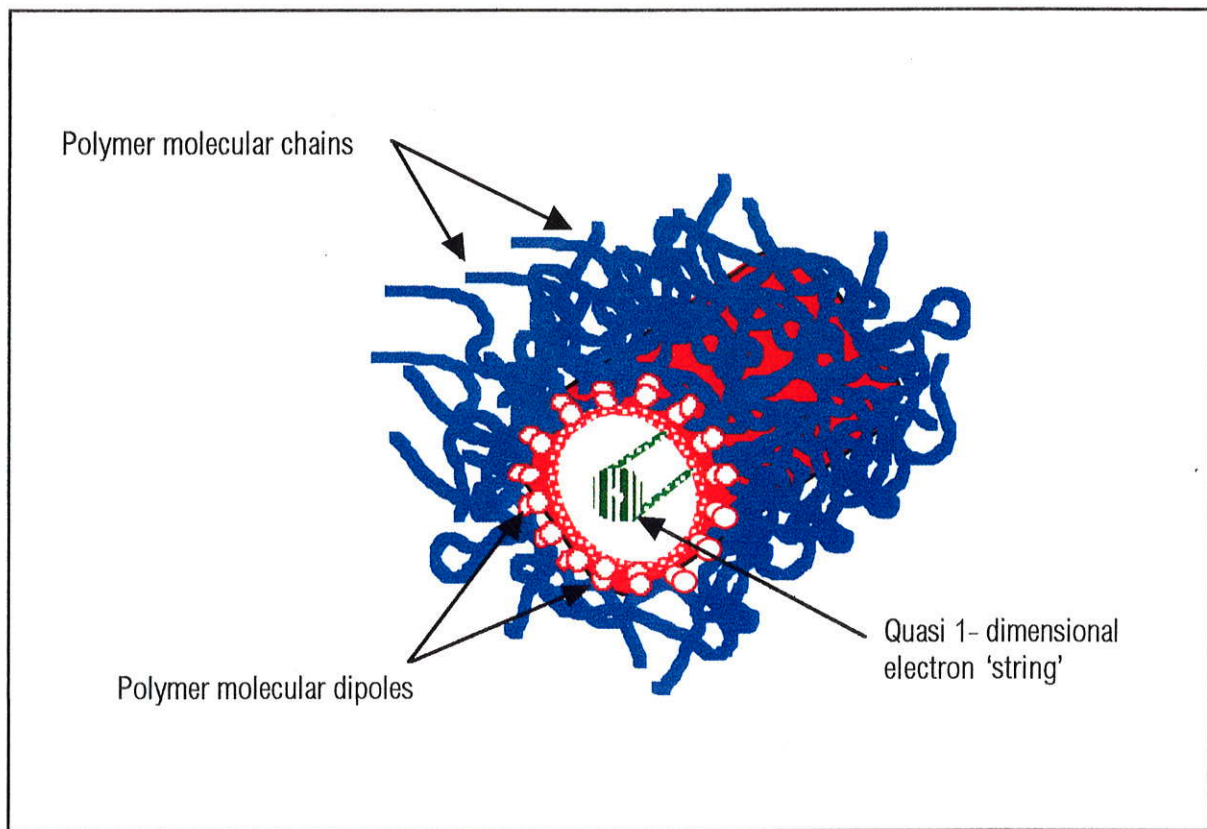
Figure 4: Cross Section of Superpolaron Structure

The lowest energy configuration for the multi-polaron units (superpolarons) is a radial distribution of polymer dipoles and a prolate structure. The illustration does not show the complete polymer chain shell which surrounds the structure, or the ions on the chains. This shell is highly condensed as a result of the high charge density of the axial electron string. Because the superpolaron's polymer 'shell' includes compensating ions, the structure has no net charge.
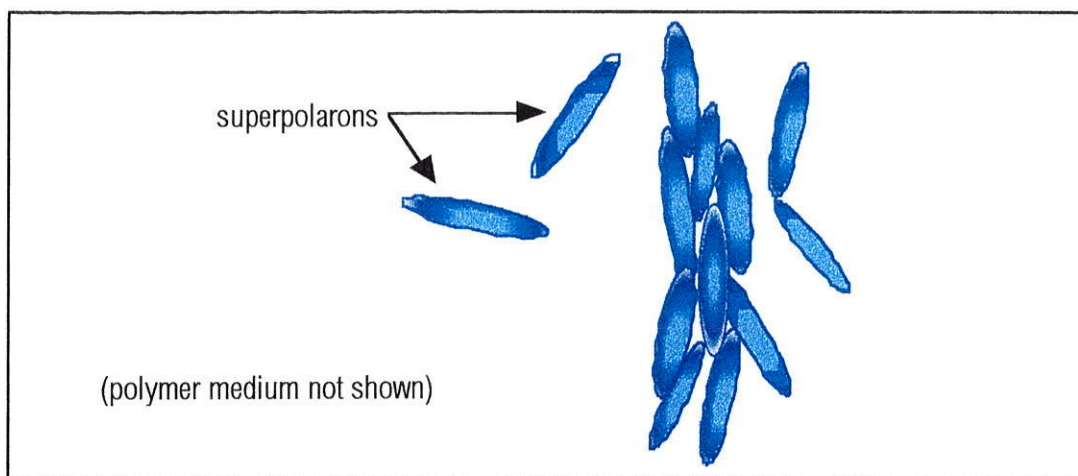
Figure 5: Initial Aggregation of Superpolarons

Motion in the viscous polymer brings superpolarons within lengths sufficient for spin exchange interaction to bind them together into aggregates. In films, the aggregates spontaneously form lengthening axial structures, channels, which can extend substrate to surface and be contacted electrically.

D. 2 Experimental effect on channel microstructures

The conducting microstructures in Ultraconductor films are normally distributed throughout the volume. When the polymer media is left viscous (ie, not crosslinked) the microstructures show a susceptibility to applied fields, which can cause them to move.

This phenomenon is directly analogous to electrophoresis, in which particle motions can result from their polarization due to the application of field gradients. The present contract investigated and further demonstrated this effect on Ultraconductor conducting structures (channels).

The observed phoretic effects on the Ultraconductor channels were distinctive. Figure 6 shows that in the experimental scheme used for this contract, two opposing forces may be acting on the conducting microstructures.

Figure 6a illustrates the presumed mechanism for this effect. Near the positive electrode, the electric field induces a dipole in the conductor, and the dipole is attracted to the region of highest field strength, which is the edge of the electrode. Note that the local field strength is highly dependent on the radius of curvature of the electrode in the cross-section shown, and also in the plan view if the electrode is not perfectly straight (rare since the edge was cut, not electrodeposited).

FIGURE 6. FORCE MODEL

It is proposed that this dipole force attracts the elements to the electrode, where some of the elements may acquire a positive charge - probably by losing electrons to the electrode.

This leads to 6b, showing the Coulomb repulsion between the charged particle and the electrode. The element then migrates under the net effect of these two forces, and they tend to balance at a distance from the electrode. At that point a raft of these elements will tend to form a line, and be spread homogeneously because, being charged they have mutual repulsion.

When the voltage is removed, the stripe starts to dissipate in minutes, which is another indicator that the particles in the stripe are charged. Normal diffusion under thermal agitation would be hours or days.

E. INTERPRETATION

The experiment clearly shows the formation of certain patterns as a result of the application of electric field gradients to polymer films containing Ultraconductor conducting structures.

The experiment confirmed that the conducting structures were visible in a phase contrast microscope, a feature which directly corresponds to the higher density one would expect from the tight electrical coupling within the structure, and as indicated by the known morphology of the channel (for example, as revealed by Atomic Force Microscope scans).

The experiment also confirmed by direct imaging that these structures were mobilized by electric fields; this was previously known rather indirectly by concentration studies as described in the U.S. Patent.

New information was also generated by the project. It was observed that the conducting structures were likely being repulsed from the field source, indicating with high probability that the structures were being charged as a result of their migration toward the positive electrode edge. This information will be of enormous value in designing equipment to concentrate the structures in a region, and also for the case where a conducting chain of structures is to be formed between two electrodes. In general it indicates that an optimal voltage exists for achieving attachment of the structures to an electrode.

The model proposed indicates the engineering parameters for developing long conducting paths in the next phase. The Phase II objective, using the concentrated stripe, will be to reorient the elements along the stripe to form a conducting filament. This will involve a local field, and possibly a method of discharging the stripe.

F. BENEFITS OF ELECTROPHORESIS / OUTLINE OF PHASE 2

In general terms, phoresis may be defined as the translational or rotational motion of particles which have been polarized in a solution or suspension by an applied field. Electric, magnetic and dielectric modes of phoretic motion have been described in the literature, each mode distinguished primarily by a) the nature of the applied field; and b) the relative natures of the particles and suspensions.

For example, magnetophoresis results from polarization of ferromagnetic particles in a magnetic field gradient, while dielectrophoresis results from the polarization of dielectric particles in a dielectric suspension by an electric field.

Electrophoresis is a now a well understood precision technology used for particle sizing, separation of biological cells and other uses. This contract has shown that the Ultraconductor conducting structures move under electric fields, which is the fundamental effect of electrophoresis.

In addition to their analytic value, phoretic techniques have been shown to enable the fine control and construction of colloidal or particle based materials, such as new composites. Phoretic methods can also induce phase changes in certain

suspensions, with practical applications.

In the latter category, certain types of suspensions of magnetic or electrically charged particles show dramatic responses to certain field strengths. These "smart fluid" systems (respectively called magnetorheological [MR] and electrorheological [ER] fluids) exhibit a complex evolution when fields are applied. Specifically (and of interest here) these suspensions evolve from homogeneously dispersed particles to highly oriented chains under applied fields, as the colloidal particles acquire an induced dipole moment.

While Ultraconductor materials are not precisely equivalent to ER or MR systems, the inherent conducting structures do demonstrate phoretic responses to both types of applied fields, as well as particle chaining and aggregation effects.

Consequently, Phase II will extend upon the present contract results by incorporating data from such phoretic systems, with the ultimate goal of creating a chain of structures forming a long conductor. The Phase II goal will be the development of a commercially scalable controlled fabrication system, employing these techniques.

Phase II objectives may be summarized as:
- Utilize the electrophoresis techniques to measure particle density, particle sizing and aggregation phenomena;
- Utilize these results to optimize applied fields for maximum aggregation and chaining of structures;
- Using shaped electrodes, develop the technique to create conducting paths, and extend these paths to useful lengths;
- Characterize the conductors developed.

G. COMMERCIALIZATION PATH

Commercial development of the Ultraconductor technology is proceeding with proprietary fabrication and applications work at ROOTS three laboratories. The company was awarded its first, landmark U.S. patent on Ultraconductors in July, which is pending worldwide. Additional patents have also been filed.

The company's commercialization strategy is based principally on joint development and licensing contracts with corporate strategic partners. This strategy is consistent with existing practices for companies which have developed superconducting materials. ROOTS has initiated substantive discussions with several Fortune 500 companies for specifically targeted applications development.

Of particular significance here, ROOTS has participated with one corporation in testing present Ultraconductor films for use as a highly anisotropic interposer for very fine pitch flip chip mounting. Initial testing of samples at the corporation's

laboratories support this application.

The present contract goal leads to development of fabrication technology for making extremely low resistance / high current density X - Y conducting paths in Ultraconductor films. This method is also believed to be a scalable approach for fabrication of unlimited length tape and wire. These fabricated forms are of specific utility for a very wide range of commercial applications.

Initially, applications development utilizing the fabrication technique will be focused on small scale products, as appropriate to the present state of film production capability. Targets include electronic applications (eg, interconnects), small sensors, EMF shielding, and compact antennas.

Subsequent targets (on development of continuous loop structures) include extremely compact magnetic energy storage and magnetic levitation components.

Ultimately, as the fabrication method is extended to production of filamentary tape, suitable for increasingly larger scale windings. These windings are the basis for a diverse range of electromagnetic (eg, motor, generator) devices and products.

The Phase II proposal will address in more specific detail the commercial path planned for the patterning development.

H. CONCLUSION

Work performed under the Phase 1 contract was very successful in accomplishing the goals originally proposed - the creation of patterns in the film from conducting structures; and the clear demonstration of the future utility of electrophoresis for the development of useful conductors and as a set of tools for the scientific scale up their production.

In particular, the electrophoretic method investigated in this Phase I was demonstrated as a feasible approach for a principle fabrication technique for Ultraconductor materials. The technique is inherently suited for production scale fabrication, due to the flexibility of the method, and its potential for incorporation into a through-processing system.

Further, phoretic techniques in general have been widely developed and investigated, providing useful resources applicable to the Ultraconductor process development. This factor alone significantly reduces the novelty - and consequently, risks - of the next Phase.

The commercial utility and value of the Ultraconductor materials, when fabricated as proposed under the contract (and in Phase II), are believed to be extremely high.

17

APPENDIX A. MATHEMATICAL MODEL

The qualitative model described in Section D of this Report is repeated here for completeness:

MICROSTRUCTURE MODEL

A model of a possible mechanism to explain the experimental results is described here.   Figure 6 shows that two opposing forces may be acting on the conducting microstructures.

6a shows that near the positive electrode the electric field will induce a dipole in the conductor, and the dipole is attracted to the region of highest field strength, which is the edge of the electrode. Note that the local field strength is highly dependent on the radius of curvature of the electrode in the cross-section shown, and also in the plan view if the electrode is not perfectly straight (rare since the edge was cut, not electrodeposited).
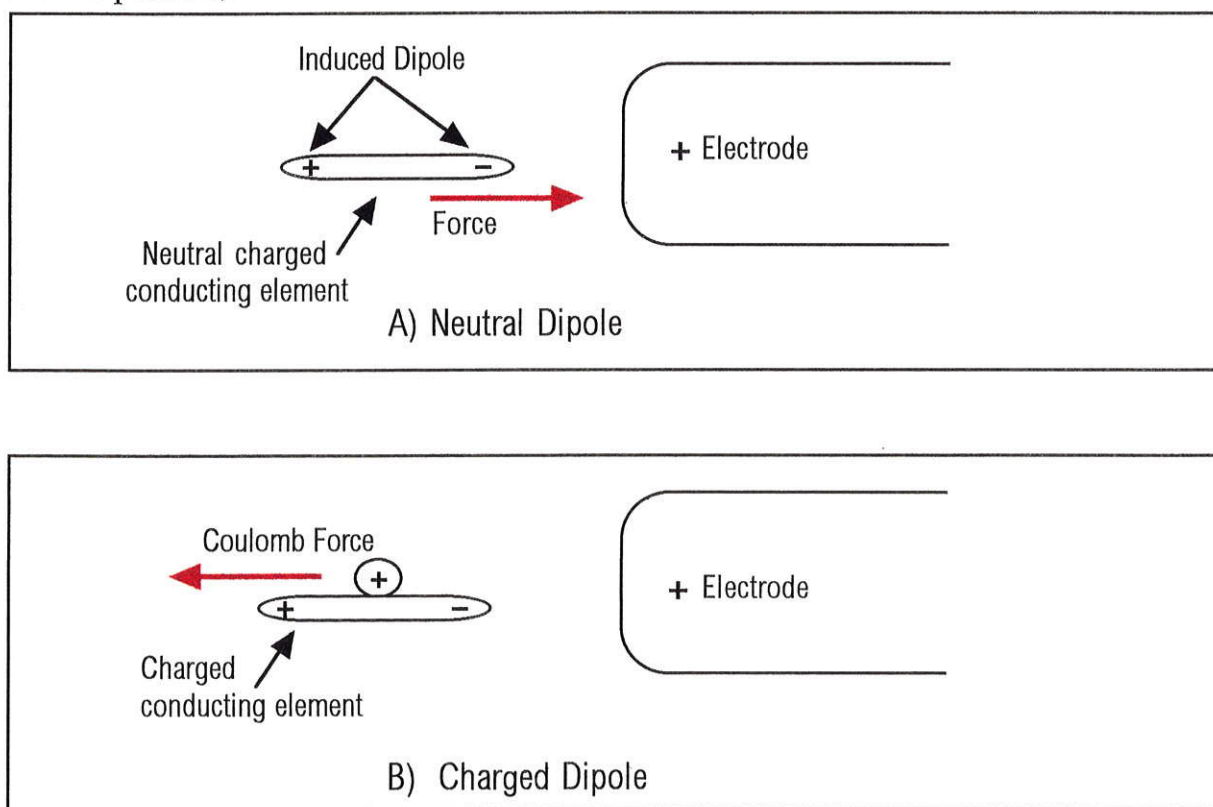

FIGURE 6. FORCE MODEL

It is proposed that this dipole force attracts the elements to the electrode, where some of the elements may acquire a positive charge - probably by losing electrons to the electrode.

This leads to 6b, showing the Coulomb repulsion between the charged particle and

the electrode. The element then migrates under the net effect of these two forces, and they tend to balance at a distance from the electrode. At that point a raft of these elements will tend to form a line, and be spread homogeneously because, being charged they have mutual repulsion.

When the voltage is removed, the stripe starts to dissipate in minutes, which is another indicator that the particles in the stripe are charged. Normal diffusion under thermal agitation would be hours or days.

ANALYSIS

a) Dipole force Fd

The dipole is induced by a field E at the element (quasi one dimensional conducting structure). It is assumed that the element is rotated by the induced dipole torque to align with the E field. (See Fig A1).
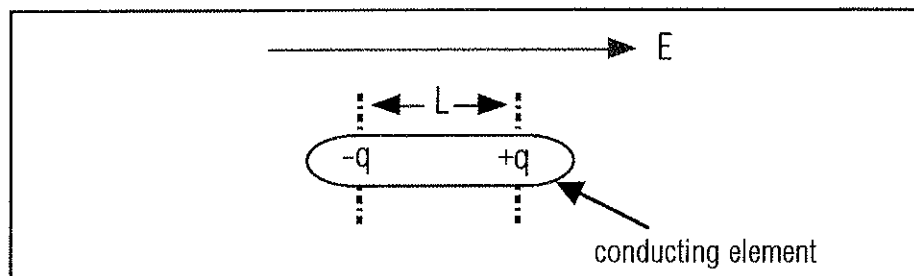

FIGURE A1: Induced Dipole

For equilibrium the field at "-q" must be zero or the charge would redistribute. The analysis is simplified by considering the virtual dipole rather than the actual distribution of charge which creates the dipole.

The field due to "+q" = q/l^2 (divided by the factor k = 4 (Pi)e_0e which will be assumed henceforth)

Hence $E + q/l^2 = 0$ $\hspace{4cm}$ (A1)

$$El^3 = ql$$

The force on a dipole ql is
$\hspace{1cm}$ Fd = ql(dE/dx). $\hspace{3cm}$ (A2)
Then,
$\hspace{1cm}$ Fd = El^3 (dE/dx) $\hspace{3cm}$ (A3)

b) Coulomb force (Fc)
$\hspace{1cm}$ Let the net charge on the element be Q, then,

19

$Fc = EQ$

At a given voltage, after acquiring the net charge the element will move away from the electrode until Fd = Fc

$$El^3 (dE/dx) = EQ$$
$$Q/l^3 = dE/dx$$

If, for each element, we assume Q and l are constant, the element moves until the equation is satisfied:

Case 1: $Q/l^3 < dE/dx$ at the electrode - the element will remain at the electrode.

Case 2: $Q/l^3 > dE/dx$ at the electrode - the charge moves away from the electrode.

Fig A2 is a diagram of the voltage V, the field E, and the dipole driving function dE/dx. It is clear that if these were the only forces on the element then dE/dx becomes weaker, and then reverses to enhance the Coulomb force - hence the element would migrate to the negative electrode.

FIGURE A2: VOLTAGE (V) AND FIELD (E) BETWEEN ELECTRODES WITH RADIUS R (NOT TO SCALE)
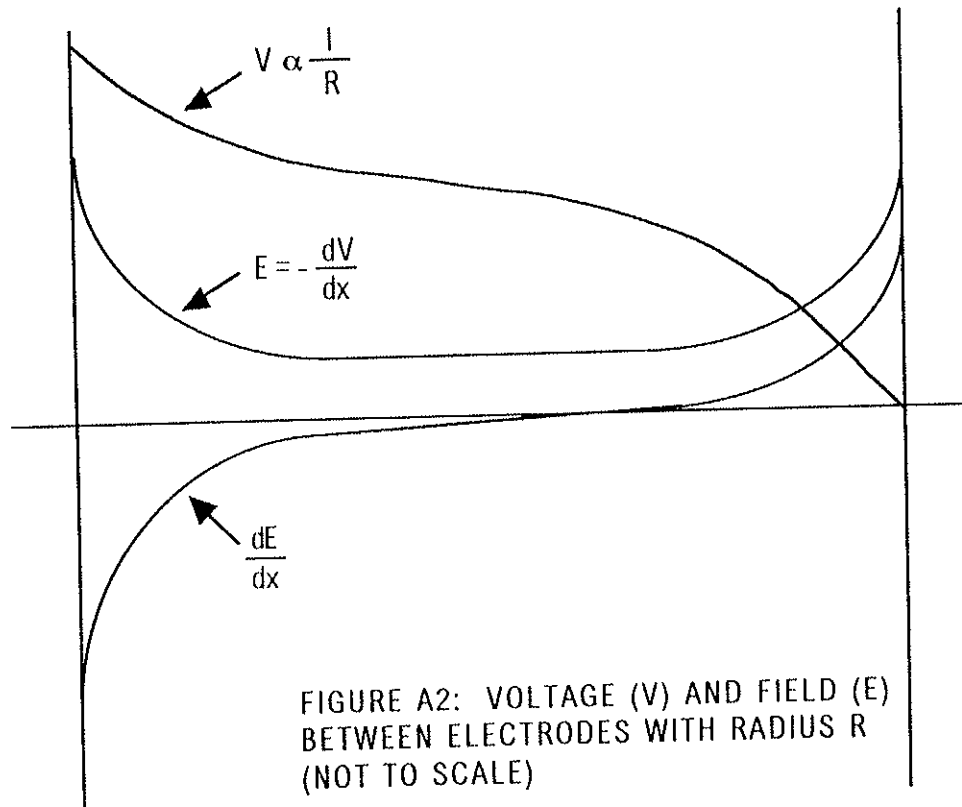
This analysis shows that the Coulomb and dipole forces on an individual element will not provide the stable configurations we see in the experiment.
A modification of the theory may explain stability of the patterns. The force away from the positive electrode is provided by the Coulomb repulsion of molecules which have a positive charge from contact with the electrode. These molecules migrate to the negative electrode and the viscous force they apply to the raft of conducting elements, balances the dipole attraction in a dynamic balance.

Another approach to modeling the results is to recognize that the assumption that Q and I are constant may not be correct.

Q may be distributed to other conducting elements as they join; or in effect, for a given Q, the length I associated with Q increases. This would increase the dipole force without changing the Coulomb force.

The length I could be reduced for elements near the electrode, since the induced dipole creates an internal stress, and the charge Q will add to that stress. Breakup of long elements near the electrode is therefore to be expected.

It is important to note that the experimental results show utility, independent of the actual model at this time. It is presumed that the model will be further refined as we progress in the planned development.

APPENDIX B.   ATOMIC FORCE MICROSCOPE SCANS OF ELECTRIC FIELD

Illustration, next page:

The following illustration, provided by Parke Scientific,
is an Atomic Force Microscope view of a single
Ultraconductor channel structure, imaged at the surface of
the film.

Topology of the channel end is typical, presenting as a
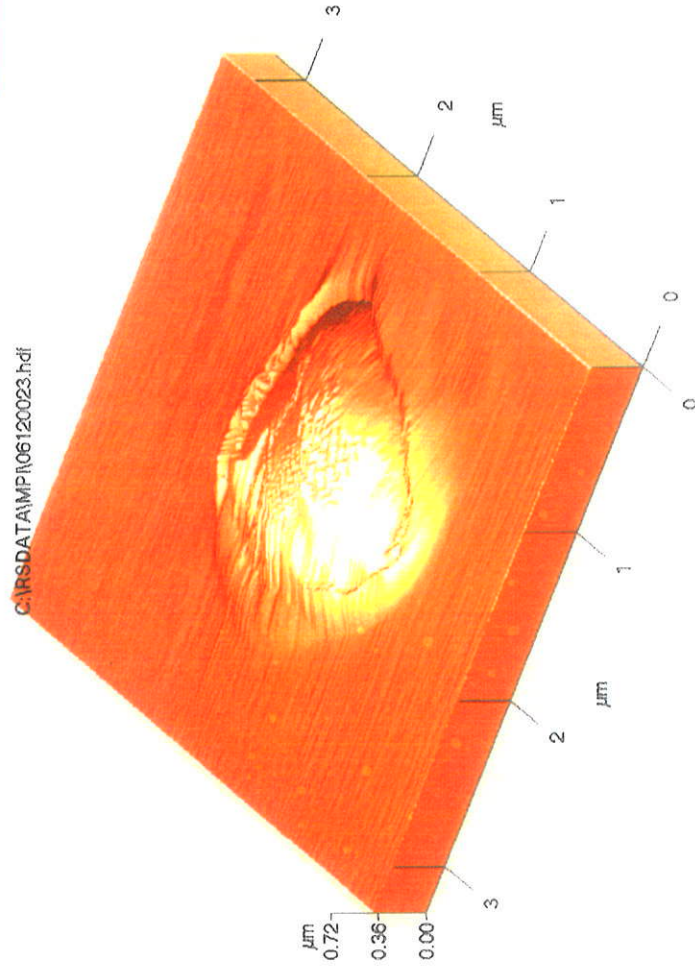'bump' of approximately 0.5 microns in height, and 2
microns diameter.

The offset illustration (right) is an Electric Force Scan (scan
of electric field) of the same feature, indicating a
pronounced gradient over the channel end.

EFM Analysis

TOPOGRAPHY

C:\RSDATA\IMP\06120022.hdf

EFM

C:\RSDATA\IMP\06120023.hdf

23
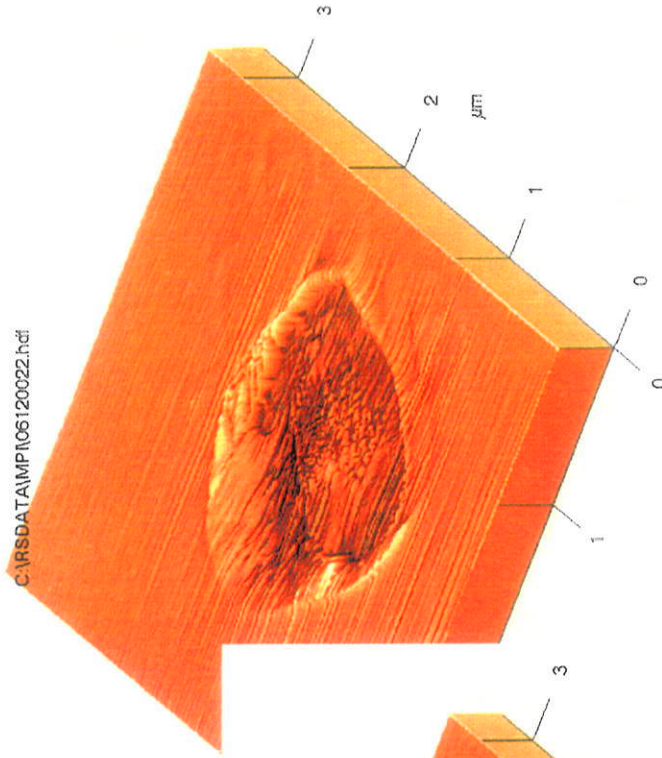
C. Description of Phase Contrast Microscope

A phase contrast microscope (PCM) was used, which can distinguish the higher density conducting channels from the surrounding dielectric medium.

The PCM is able to convert differences in phase or path length of light, to differences in brightness, which can be seen, even when all light paths are transparent. The phase change or optical path difference d introduced by an object is defined by the relationship $d = (n_o - n_m)t$, where n_o is the refractive index of the object, n_m that of the surrounding medium, and t the object thickness. Although the PCM converts variations in d into variations in intensity, the relationship between these quantities is not a simple one. In general, as d increases, the image becomes darker; but beyond a certain value of d, it becomes lighter again, and finally, for very large values of d the contrast actually becomes reversed, so that the image becomes brighter than the background.

The system chosen is a Meiji, model ML5000, including a Zernicke phase plate and objectives, with a high resolution color CCD camera. This was connected to both a VCR and a computer, using a frame grabber, for recording and analysis.

The Meiji was the least expensive industrial quality microscope with phase contrast. Phase contrast was determined by experiment to be essential as a means of clearly viewing the variations of polymer density. The higher quality images and versatility of the more expensive brands was not essential to foreseeable experiments.

It was found that structure could be seen within the polymer without phase contrast, by closing down the diaphragm to its smallest aperture. This provides an effect analogous to a pinhole camera, providing a large depth of focus. This approach appeared to have two effects - first, it integrated all light in its path, keeping it all in focus; whereas the lens with a larger diaphragm will have a short focal length, and the image is obscured by the layer of polymer surrounding the layer in focus. Second, by cutting down the intensity of the light, areas of slightly greater absorption appeared dark, because they were close to the threshold of visibility; whereas they would at higher intensity appear only a small percentage different.

The phase contrast, in comparison, could highlight the differences in density at a particular depth in the film, at the point where it was focused. Hence one can examine different points in the film by changing the focus.

The images were generally viewed with a X10 objective for a total magnification of x100.

2. Background

Commercial development of the Ultraconductor technology was begun in 1993, following 12 years of research begun by a scientific team at the Polymer Institute, Russian Academy of Sciences, led by Dr. Leonid Grigorov, Ph.D, Dc.S. (Dr. Grigorov, a Physicist, has published more than 100 papers, holds 20 patents, and is an active member of ROOTS.)

Dr. Grigorov's research into the phenomenon began in 1981 with an investigation of anomalous conductivity in a common insulating polymer, polypropylene. By 1983, he had identified a process which could replicate the unexpected conductivity in thin films of the polymer.

The first published paper describing the discovery was translated into English in 1991, under the auspices of the American Institute of Physics. Through 1993 (scientific publication was suspended until 1998 by ROOTS) a range of tests, including measurements of conductivity and other features of the polymers were made and published. Samples were independently reproduced and tests were also published by other laboratories, including the prestigious Joffe Institute in St. Petersburg.

To date 7 chemically distinct polymers have been used to create Ultraconductors, including olefin, acrylate, urethane and silicone based plastics. The total list of candidate polymers suited to the process is believed to number in the hundreds.

Candidate polymers must be polar without significant crystalline or glass phase at the time of processing. (Intrinsically conducting [conjugated] polymers cannot be used.)

The processing treatment (a description follows) results in the formation of discrete macromolecular structures. These structures (called 'channels') are persistent, and are distributed throughout the bulk polymer. In films, these channels can be observed by several methods, including phase contrast optical microscope, Atomic Force Microscope (AFM) (see illustration at the end of this paper), magnetic balance, and simple electric contact. The channel structures can be moved and manipulated in the polymer.

The characteristics of the bulk polymer are not changed by the processing, and it remains dielectric. Dopants are not used.

Ultraconductor films are prepared on metal, glass, or semiconductor substrates. The polymer is initially viscose (during processing). For practical application the channels are subsequently "locked" in the polymer, by crosslinking, or glass transition. The channel's characteristics are not affected by either mode.

3. Characterization

Characterization of the conducting channels in films was begun in 1983. To date measurements have focused on macroscopic features, specifically, measurements of the magnetic, electric, thermal, chemical, and morphologic nature of the channels.

3.a Magnetic Characterization

The processing treatment initiates characteristic changes in the magnetic state of the polymer, as measured in a sensitive faraday magnetic balance. The most consistent feature is a growing ferromagnetism simultaneous with the appearance of electrical conductivity. Transient Meissner-like diamagnetic curves have been observed, rarely, in samples when tested over periods of days, in moderate magnetic fields.

The magnetic readings for the Ultraconductor samples are established against baseline readings obtained for each sample (before processing) and film substrate. The ferromagnetic response attributable to the changed electronic state of the polymer is therefore quite direct, and is always present in all samples which are conductive.

Ferromagnetic field gradients local to the channel structures are also observed by AFM in magnetic mode.

Due to the connection between the ferromagnetic signature and electric conductivity, Ultraconductor samples are routinely tested for ferromagnetic response, as a process control. Higher values of ferromagnetism correspond to increased densities of conducting regions in the samples.

The magnetic responses typical of the processed Ultraconductor samples are entirely absent in the unprocessed base polymers, as tested and in the literature.

3. b Electric Characterization

3. b.1 Conductivity

The channels were early found to be electrically conductive, for ac and dc currents, at voltages as low as 0.1 volt. In addition, AFM electric field scans (using non-contact, tapping mode) indicate pronounced field gradients localized to the conducting channels. The AFM scans also reveal a higher density of points than can be measured by conductive probe, indicating that a proportion of the channel structures do not fully extend substrate to surface.

A significant body of experimentation has tested the value of the channel's conductivity, both under ambient conditions, and over a range of temperatures, pressures, and magnetic field strengths. Test methods include 4 point probe, and superconducting tin electrodes[3] . The measurements indicate that the channels'

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[3] Arkhangorodskii, V.M., et al., *JETP Lett.*, 1990, vol. 51, no 1, p 67

conductivity (10^{11} - 10^{24} S/cm) is dramatically higher than metals (~ 10^5 S/cm); and that the high conductivity is insensitive to temperature (from 1.8 K to 700 K) or magnetic fields (to 9 Tesla)[4].

3. b.2 Resistance

Electric resistance of the channels has also been measured under various experimental configurations. Typically, newly formed channels have measured resistivities of ~ 1 Ohm. This initial measured resistance can be lowered by several means, including a) application or release of modest local (electrode) pressure; and b) application of pulsed ac currents of increasing amperage over time. Following method b), called 'training', channel resistivity is reduced to the range of 25 milliohms. (Training also increases the current carrying capacity of individual channels; see below).

The disparity between the conductivity of the channels (estimated from a variety of measurements) and the measured resistance is understood to indicate that a significant portion, if not all, of the resistance is at the contact. This conclusion was also supported by 4 point probe measurements, made independently at the Joffe Institute.

3. b.3 Thermal

Subsequent tests also determined that the conductivity is not metallic: the thermal conductivity of the conducting channels was found to be equivalent to the surrounding dielectric polymer (indicating that the charge carriers in the conducting channels are poor thermal conductors). The disparity between electric and thermal conductivity of the channels is six to seven orders of magnitude beyond metals in this respect[5]. Such a large scale violation of the Wiedemann-Franz law is known only for Cooper pair electrons, in superconductors.

3. b.4 Current Measurements

Experiments indicated that individual channels (approximately 1 - 2 microns diameter) have a maximum current density: exceeding a threshold current results in a jump-like rise in resistance, melting of electrode and substrate, and vaporization of a small volume of polymer[6]. While high currents below the threshold do not affect the polymer, a very small step increase above the maximum current results in the characteristic microexplosion event. The threshold current value is increased significantly by the training procedure (application of incrementally stepped increases of pulsed ac currents over time).

The microexplosion event was investigated, and several consistent features for the phenomenon were measured. The event (in trained channels of approximately 2 microns diameter) typically occurs between currents of 50 to 100 amperes; occurs in extremely short (ns) time scales; and corresponds to a sharp, nearly instantaneous

[4] Grigorov, L.N., et al., *Polymer Science* , 1993, vol 35, no. 11, p1625
[5] Grigorov, L.N., et al., *Superconductivity*, 1991, (4), p 345
[6] Demicheva, O.V., et al., *JETP Lett.*, 1990, vol 51, no. 4, p. 258

rise in resistivity of the channel.

The rise in resistance in the channel was measured to occur prior to the rise in temperature which accompanies it, indicating that the event is not thermally triggered. In combination, these factors strongly suggest a 'critical current' event, analogous to those known for superconductors.

3. c Chemical and Morphologic Characterization

Conductive Ultraconductor samples (post processing) have been tested and compared against samples of the unprocessed base polymer for contaminants, chemical composition, and metal inclusions. They are found to be identical in all respects to the base material.

Examinations of morphologic features of samples, such as transparency, visco-elasticity, and so forth, also indicate that the bulk Ultraconductor polymer (excepting the channels) is unchanged from the base polymer.

In films, the channel structures are distributed randomly in the polymer, and are of varying lengths to a maximum measured of 100 microns. A proportion present themselves through top and bottom film surfaces, as indicated by direct electric contact. At the free surface, they are typically 1 - 2 microns in diameter, roughly circular, and present as 'bumps' approximately 0.6 microns high (see Appendix B).

4. Potential Explanations for the Anomalous Features

Several potential mechanisms for certain of the anomalous features have been investigated and experimentally discounted.

a. The possibility of holes, or punctures, in the Ultraconductor polymer.

While holes in very thin, spin-cast polymer films are likely for amorphous elastomers, this probability statistically falls to zero at thicknesses exceeding 2 - 3 microns. Further, the highly elastic state of the material makes it largely self-healing at scales which would escape visual (microscopic) observation.

The conclusion of holes is also inconsistent with the observed magnetic features; resistance and conductivity values; the Wiedemann-Franz measurements; and the critical current observations.

b. The possibility of metallic inclusions or asperities in the channels.

The growth of metallic inclusions, or the presence of metal asperities, in the polymers was investigated. In order to be consistent with measured features, this possibility requires the spontaneous growth of multiple, highly anisotropic metal asperities approaching 100 micron lengths, from metal and non-metallic substrates,

at the densities measured, in a variety of chemically distinct dielectric polymers. This phenomenon has not been reported in the literature.

Further, no metal asperities or inclusions have been observed or measured in the Ultraconductor polymers. In addition, metallic contamination is inconsistent with the uniform ferromagnetic changes of samples prepared on metal and non-metallic substrates; thermal measurements, such as the Wiedemann-Franz violation; measurements of conductivities; and the reduction of resistance by increased pulsed currents.

c. The possibility of polymer breakdown under voltage.

Breakdown in dielectric polymers such as polypropylene (ubiquitous as an insulator) has been extensively studied and published. Breakdown typically leaves visible damage; has no preferred direction or orientation in the polymer; and does not conduct current without measurable resistance.

In contrast, the conductivity in Ultraconductor films is highly anisotropic, and measurable at 0.1 volts, well below the breakdown field (which in polypropylene films, is 1.2×10^5 V/cm).

Further, since any breakdown of the polymer remains resistive, polymer heating, visible damage, and decomposition by heating should be observable well in advance of the current densities measured for the Ultraconductors.

Breakdown is also inconsistent with the ferromagnetic measurements; the spontaneous appearance of conducting regions in the Ultraconductor polymers prior to electric contact; the critical current event; and the Wiedemann-Franz violation.

5. Processing of Ultraconductors from Dielectric Polymers

The experimental observations of the Ultraconductor phenomenon reveal an apparent metamorphosis of dielectric insulators into ferromagnetic conductors with non-Fermi properties.

This transformation is initiated by a processing of certain polymers.

5. a Candidate Polymers

Polymers which successfully respond to the Ultraconductor process must meet certain physical criteria. Specific chemical formula is not important, provided those criteria are met.

5. a. 1 Polymers With Very Low Crystalline or Glass Phase

Microscopically, polymers are comprised of repeating molecular units which form long chains. The ordering of molecular groups on the chains affects the material qualities of the polymer. For example, the regular molecular order of isotactic polypropylene (PP) allows individual chains to interlock with each other, producing a hard, crystalline-like material.

Another type of PP - the atactic version - instead has random molecular order. Its polymer molecular chains do not significantly interlock, but wander with heat convection, resulting in a material like a slow moving liquid. The material remains amorphous, viscose, and elastic.

Isotactic PP and other polymers with significant crystalline or glassy phase have not been successfully processed to Ultraconductors. However, atactic PP and other amorphous, viscous, elastomers respond reliably, indicating that the free movement of molecular chains is essential.

5. a. 2 Polymers with Polar Groups

Another precondition is that the polymer should be polar. Many polymers have molecular groups ('side groups') which extend off the axis of the long molecular chain. Certain amorphous polar polymers have side groups with high mobility and large dipole moment, which can be easily oriented in low electric fields. These respond to the Ultraconductor processing.

5. b Process Steps

The process for creating Ultraconductors is based primarily on mild ionization methods, which introduce free electrons into the dielectric polymer. Dopants are not required.

The following are the processing steps, for the dielectric polymer atactic polypropylene (PP).

5. b. 1. Oxidation
The PP is oxidized by exposure to ambient air at slightly elevated temperature. When PP is oxidized, the oxidation produces electric dipoles (C=O groups) on the molecular chain. Their total average concentration reaches ~ 10^{22} /cm^3. (With other polymers, dipoles may already exist on the molecular chain, and oxidation is unnecessary.)

5. b. 2. Ionization
The oxidized PP (OPP) is then ionized by mild irradiation, typically UV light. Ionization occurs as electrons are knocked from the polymer molecules, creating ions on the polymer molecular chain.

6. Model of Process-induced Ultraconductor Formation

When taken together, the known preconditions and process strongly suggest that the mechanism for Ultraconductor formation involves a self-organization of charge and molecular lattice. A model for this system has been qualitatively and quantitatively described (reviewed below), and subsequently a quantum mechanical theory of the phenomenon has been published. This novel self-organization has no equivalence in solid state materials.

6. a Charge Separation

The polymers used to form Ultraconductors are dielectrics, with no conducting (free) electrons. The ionization process is used to introduce free electrons into the polymer through an unusual mechanism.

In dielectric polymers, ionized molecules normally will reverse in very short time scales, and the electrons recombine. However, in a *polar media* it is possible for dipole groups to prevent this recombination, with the result that charges (such as electrons and ions) remain stably separated.

This stabilization of separated charges due to electrolytic dissociation in polar solvents is well known. For example, when NaCl is dissolved in water, the H_2O dipoles maintain the charged elements (Na^+ and Cl^-) in solution separately.

The viscous polar polymer acts in essentially the same manner: the highly mobile molecular dipoles surround and hold the freed electrons in their electric fields. These electrons, chemists say, are "solvated". Physicists call these solvated electrons "polarons".

6. b Charge Localization and Self-organization

At this stage, polymer molecular dipoles have solvated (captured) a quantity of electrons freed by ionization (mean concentration ~ 10^{18} / cm^{-3}), and the compensating ions. The dipoles and ions remain attached to the long polymer molecular chains, and are distributed in the polymer.

Due to the high mobility of the polymer media, and thermal energy, these randomly distributed charges can move relative to each other. Over time separate polarons (electrons surrounded by polymer dipoles) are brought closely together, and collide.

A simple theoretic analysis shows that two polarons together form a lower, and hence preferred, energy state, than solitary polarons: consequently, polarons which collide, join. The electrons are brought together in a "sack" of surrounding dipoles. This event, repeated throughout the polymer over time, has enormous consequences on the organization of charges and the polymer matrix (See following Figure A.):

32

1. POLYMER CHAINS of polypropylene after oxidation

Dipoles of polar groups (oppositely charged ends)

Molecular chain
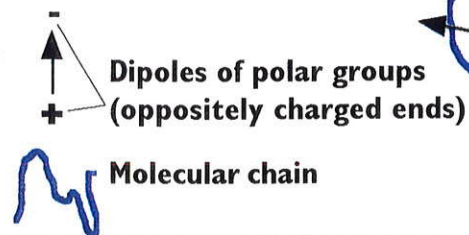
2. IONIZED STATE after UV irradiation showing polarons

ions

polarons

(Drawing not to scale)
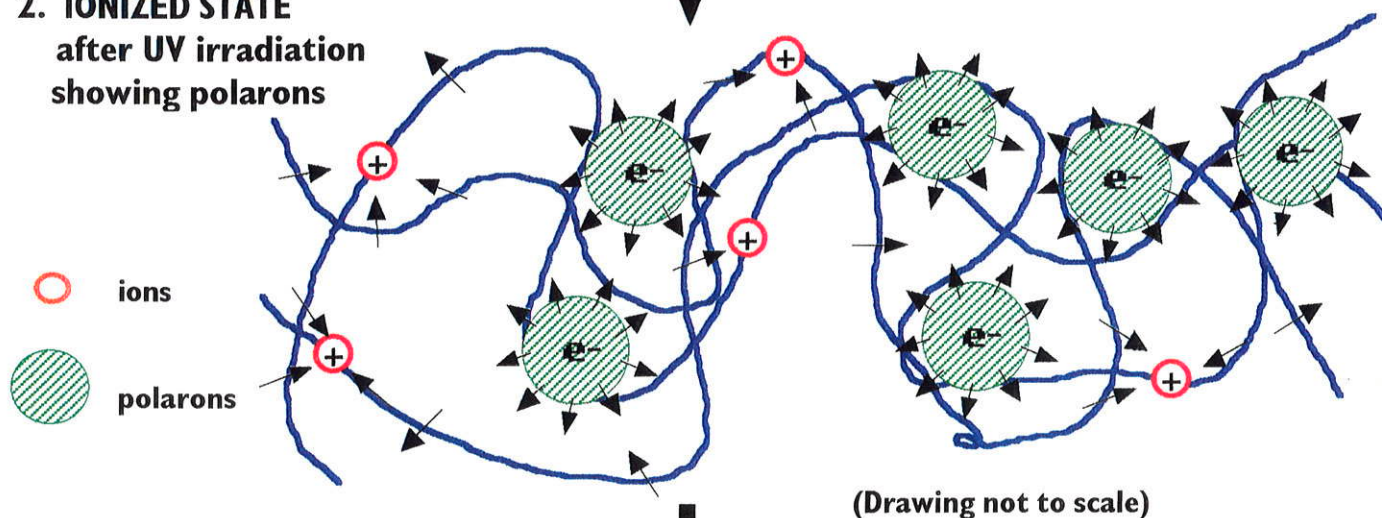
3. SUPERPOLARONIC STATE (polymer matrix not shown)

Polymer

Electron 'string"

Multi-Polaron Shell

Polymer

33

Figure A - Process Sequence
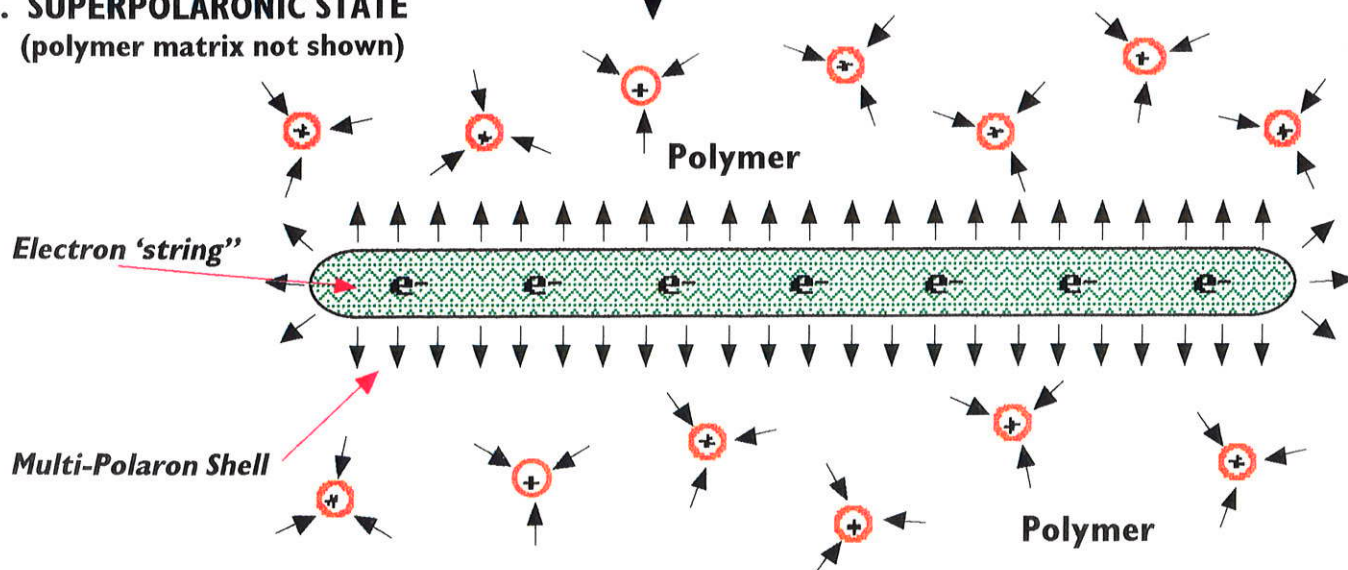
- When polarons join, axial multi-polaron units are formed, called 'superpolarons'.
- These lengthening electron strings form a growing electronic potential valley. The increasingly negative charge density strongly attracts the positively charged ions, and dipoles.
- The polymer molecular chains to which they are all attached are drawn densely together, forming a corresponding cylindrical shell around the electron string. At this time, the strongly condensed electronic system is quasi-one dimensional.
- In these conditions, the charge density in the quasi one-dimensional potential valley ($\sim 10^{21}$ electrons / cm^3) must find an energetically stable state. The lowest ground state is found to be achieved by electron pairing.[7]

6. c Channel Formation

The superpolaron 'threads' aggregate in the polymer, bound together by spin exchange coupling, to form bundles which grow to contain thousands of superpolaron structures.

It is these bundles, or channels, which are the subject of present fabrication approaches. It has been demonstrated that these superconducting structures can be manipulated and 'grown' in a manner akin to crystal growth, and consequently may be extended and combined effectively in order to make wire.

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[7] The quantum mechanical theory cannot be adequately treated here; the paper has been recently published in Philosophical Magazine B, 1998, Vol 78, No.4 353-363.

APPENDIX E. DETAILS OF PHASE I EXPERIMENTS

The typical sample was prepared as follows:

a) Amorphous polypropylene (4% to 10%) was dissolved in toluene or hexane.

b) A glass slide was dipped in the solution and allowed to evaporate at room temperature. (Heating to speed evaporation was found to create less homogeneous film, which as a result produced dark spots or dots as seen in the microscope.)

c) Step b was repeated to create a thicker and more uniform film.

d) 4 aluminum strips were laid across the slide, adhering to the polymer film.

e) Each strip was cut in the center, and the two halves drawn slightly apart, to create two electrodes with a gap of 30 to 100 microns. Hence 4 pairs of electrodes are created on each slide.

f) Further layers of polymer were added, to partially fill the gap between the electrodes. The first layer covered all pairs, the second was dipped to cover only three pairs etc. Hence one slide contains samples with from 1 to 4 additional layers.

Examination:

At this point each slide was examined under the microscope and the image recorded. Next a dc voltage of up to 600 volts was applied and the image recorded under voltage.

In general these "passive" slides did not show any change with voltage. The word, passive, is used here to mean polymer which has not been treated with UV light.

The next step was to expose the slide to UV light for 20 to 60 minutes, creating an "active" slide with conducting channels (superpolarons) in the polymer.

The active slide was then examined.

Experimental Notes:

Glass slides were also coated with submicron thicknesses of Indium tin oxide (ITO), to create thinner electrodes for comparison. However, no technique was found to make a clean cut or gap.

The dark stripes which appeared in the Ultraconductor films under dc voltage fields, were sometimes very clear; however, there were also signs of incipient stripes which were masked by the background "noise", and which were difficult to identify. The analysis and characterization are based on the clear stripes. This defines the usage of the word "stripe" for this report - there may be other dark or bright stripes in the polymer which are not created by the fields, and these are considered as part of the background noise (generally fairly stable images). The stripes are also subject to interpretation since they are essentially a phase contrast microscope image of polymer density differences.

Observations:

1 There were no "stripes" in passive polymer (that is, without UV treatment), while stripes were induced in active polymer (that is, Ultraconductor).

2 They originated at a short distance from the positive terminal.

3 A few stripes originated near the negative terminal.

4 The stripe initially formed when a critical voltage was applied, typically 300 to 400 volts.

5 At a set voltage the stripe migrated away from their origin (often toward the negative electrode), and then stabilized.

6 When the voltage was increased the stripe migrated to a new position.

7 Often there was a thin dark line followed by a white area, and a wide dark stripe appeared in this area.

8 The area defining the stripe, white and dark, seemed to eliminate the normal background dots.

9 AC voltages of the same magnitude did not create a stripe.

10 As the dc voltage was increased in passive polymer there was a range where the polymer showed electro-hydrodynamic (EHD) activity.

11 As the voltage was further increased, breakdown occurred. The EHD and breakdown occurred in similar fashion in the active polymer, often with flashes of blue light, indicating the breakdown path was in the air above the film.

12 In active polymer the breakdown had also been observed between an electrode and the stripe.

These observations start to create a picture of what is happening. Point 12 is an indication that the stripe may be conducting in part. This was tested directly by probes.

To probe the stripe, aluminum electrodes were used as probes along the gap - this was not successful since they tended to distort the formation of the stripe, and it was not possible to create a stripe connecting these stationary electrodes. Microprobes were developed using tungsten tips held in a micromanipulator mounted on the microscope. No conduction was found using this technique.

36

APPENDIX F. PHASE I CONTRACT SUMMARY: CHRONOLOGY

Contract started 5-5-98

May
This was the first month of the contract: "Patterning of highly conductive Microstructures in Ultraconductor^{TM} polymer films", a Phase I SBIR project to develop a practical fabrication method for orienting superpolaron molecular structures.

The month was spent on the following activities to initiate the contract:

1. Planning
   Technical
   Budgets and cash-flow

2. Laboratory preparation

3. Planning and assignment of personnel.

4. Review of scientific papers on Ultraconductors(TM), and the experiments relating to electric field properties.

5. Review of the theory of phase contrast microscopes.

6. Design of the experiment

7. Investigation of phase contrast microscope.

June
In June, we continued to review the use of phase contrast microscopes, and started the preparation of suitable samples.

The phase contrast microscope was needed to see structures in the polymer, since the polymer is essentially transparent. Three phase contrast microscope's have now been demonstrated; Olympus last month, Leica and Nikon this month. They all seem to be high quality microscopes. The Nikon representative was able to leave the microscope with us for a week, which was very helpful.

July
In July we examined two more microscopes, the Zeiss and the Meiji, and decided to purchase the Meiji. The system is a model ML5000, including a Zernicke phase plate and objectives, with a high resolution color CCD camera. This will be connected to both a VCR and a computer, using a frame grabber, for recording and analysis.

August
All the equipment is now in place for the investigation. The phase contrast microscope is working well, and the image is captured by a high resolution video camera, and recorded both on a VCR and in a computer, through a frame grabber. The computer link was completed at the end of the month.

Most of the effort this month went into the preparation of samples and the examination of the samples under the microscope. A significant amount of data was collected and recorded, indicating that the "active" samples respond to the electric field. However, it is too early in the investigation to ascribe a cause to the changes, or to detect a pattern.

September
In September, there was further work on computer capture of the images, more samples were made and investigated, and methods of probing the "stripes" were tried.

October
Further investigation of stripe formation with a variety of electrode shapes. Probing the stripes did not show any conduction in the direction along the stripe. Images were captured and documented for the final report.

November
November was spent reviewing and cataloging all data. In addition, a review and evaluation of electrophoretic literature was made, especially as applied to aggregation and chaining of polarized particles. The theoretic model and mathematic models for interpretation of the experimental results were prepared. Preparation and writing of the final report began.