

**POLYMER ROOM TEMPERATURE SUPERCONDUCTORS
POWDER, THICK FILM AND SHORT WIRE APPLICATIONS**

USAF SBIR PHASE II - Contract No. FO 8630-96-C-0028

FINAL REPORT

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POLYMER ROOM TEMPERATURE SUPERCONDUCTORS

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1. SUMMARY

The preceding Phase I contract demonstrated a fundamental property of Ultraconductor™ thin (~ 10 - 40 micron) films - namely, the ability of such films to carry currents well in excess of metals. The goal of the Phase II contract was to develop the film to a thickness which would make it suitable for the first commercial applications, such as batteries and supercapacitors.

While these superior films were not demonstrated before the conclusion of the Phase II contract, all fundamental steps for their production were achieved.

The major breakthrough during the contract was demonstrating a proprietary process for producing 'enriched' Ultraconductor polymer, containing up to 100 times the density of conducting structures of non-enriched Ultraconductor material. Further, it was established that thicker films, and short lengths of wire, could be fabricated with the enriched polymer. This outcome has far greater significance - technologically and commercially - than originally anticipated for the project. Subsequent development steps are charted in the Commercialization section.

The development steps accomplished under the contract are described in the technical section of this report. During Part One, the company adapted two new laboratories for applications work. Initial contract time was spent training new technical staff in the technology; equipping the lab with special instruments, including a sensitive Magnetic Balance; and adapting known methods to create Ultraconductor films from off-the-shelf polymers manufactured in the U.S.. Measurements of the conduction and thickness of the samples by various techniques were performed.

To accomplish the contract objectives, several different techniques were used to increase sample thickness. These techniques, such as the concept of 'enrichment' (increasing the density of conducting structures in the bulk polymer), had been proposed earlier. Initially, thicker films were made without enrichment, to measure and test the limits of the unenriched polymer. Results confirmed the conclusion that enriching the polymer was a fundamental step for creating thicker films, and wire. Apparatus was built to distill out the enriched material, and the Magnetic Balance was used to make measurements; however, measurements which quantify the highest level of enrichment reached have not been completed.

This report on the results of the Phase II contract also reviews the scientific and commercial background of Ultraconductor materials and development, to enable the reader to understand the outcomes of the contract in relation to past and ongoing advances of the technology. A landmark U.S. patent on Ultraconductor materials and technology has been recently allowed.

The ability to manipulate the conductive elements shown by this work also opens up the possibility of creating an Ultraconductor "powder" technology.

2. BACKGROUND

Ultraconductors are specially processed flexible polymers which exhibit near-zero electrical resistance over the temperature range of zero to 460 Kelvin. Scientifically, the phenomena combines two branches of research - condensed matter physics, and polymer material sciences.

Standard Ultraconductor films can carry in excess of 10^6 amps/cm² without heating. This technology is the result of more than 12 years of internationally published peer reviewed research and independent replication; and more than 4 years of confidential process and applications development.

The Ultraconductor class now includes 7 tested polymers, from 4 chemically distinct polymer groups. A first, landmark, patent has been allowed in the U.S., and is awaiting allowance internationally.

Technology platforms achieved to date include consistent film production quality and control; stabilization of superconductivity; and identification of a range of additional candidate polymers. Advanced objectives completed include significant enhancements of the materials; proprietary instrumentation, processing technologies, and fabrication methods and devices; and applications research.

In addition to the thick film objectives of this contract, multiple programs in the above areas are continuing in parallel (see schematic illustrations next two pages). The technology's development benefits from access to the body of 70 years international work in polymers - an advantage not known for any other superconducting materials.

Progress in commercial applications development is being supported, in part, under this USAF SBIR Phase II Contract; and a 1997 BMDO SBIR Phase I Contract, for Ultraconductor Thermoelectric modules, many times more efficient than any available.

It is presently estimated that commercial film and short wire application prototypes will be available within the next 24 months, subject to completion of the company's present capital funding.

First applications of the technology will likely utilize films; however, Ultraconductor wire, now in early stage development, is regarded as the single largest product for the company, comprising a projected 80% of future revenues. In motors, generators, in high power transmission lines and a host of other uses, Ultraconductor wire will provide significant savings in energy consumption and tremendous advances in efficiencies.

The commercial and strategic value of Ultraconductor materials is not dependent on absolute superconductivity - which is likely - in the majority of applications, but on delivering orders of magnitude better conductivity than metals, over an extensive temperature range, with the obvious benefits of plastic materials.

Projected commercial markets by the year 2020 of \$117 billion annually for products incorporating Ultraconductor materials are based on feasible applications of fabricated film and wire, and internationally recognized studies (eg: ISIS) on projected markets for cryogenic superconductors.

Applications research also anticipates that the introduction of flexible polymer films and wire, with conductivity orders of magnitude superior to metals, holds the potential to stimulate significant redesign of existing electrical and magnetic products, and potentially revolutionize broad global industries.

In 1993, MPI (the contracting company for this Phase II) formed a subsidiary company, Room Temperature Superconductors Inc, (ROOTS) to commercialize the Ultraconductor materials and applications. ROOTS' Scientific Advisory Council presently includes Dr. James Smith, Chief Scientist of the Superconductivity Technology Center at Los Alamos National Laboratory; Dr. Matt Aldissi, an internationally recognized conducting polymer specialist with over 20 patents and 80 published papers; and Stanford Prof. Emeritus Dr. William Little, who published a forecast of room temperature polymer superconductivity in 1964.

ROOTS' scientific team - the discoverers of the material - are expert in its production, characterization, instrumentation, research and development. ROOTS has three laboratories engaged in research and technology development. In addition, the company has, and continues to expand, collaborative relationships with U.S. National Laboratories, Universities, and armed forces Laboratories.

The company has not sought, and continues to avoid early publicity.

3. OVERVIEW OF TECHNOLOGY

3.a Scientific Background

Many features of Ultraconductors are known to exist only in superconductive materials. For example:

- measurements of conductivity at ambient temperatures provide values of 10^{11} S/cm to 10^{24} S/cm and above (compared to copper, which is 10^5 S/cm)¹ ;
- between superconducting tin electrodes, at cryogenic temperatures, no measurable resistance is observed² ;
- at a critical current, resistance appears instantaneously³ (this critical current event is known only for superconducting materials);

¹ Grigorov, L.N., et al., *Polymer Science* , 1993, vol 35, no. 11, p1625

² Arkhangorodskii, V.M., et al., *JETP Lett.*, 1990, vol. 51, no 1, p 67

³ Demicheva, O.V., et al., *JETP Lett.*, 1990, vol 51, no. 4, p. 258

- thermal versus electrical conductivity is orders of magnitude in violation of the Wiedmann-Franz law (this violation is known only for superconducting current carriers, which are poor thermal conductors).

The formative conditions and processes for inducing this conductivity are well known, controlled, have been enhanced and are consistently reproduced.

3.b Self-organization of superpolarons

The nature of this conductivity in Ultraconductor polymers is distinctive.

Like metals, the Ultraconductor polymer *material* is electrically neutral. Conducting electrons are not added charges (dopants), but electrons which have been separated by ionization of the polymer. In a sequential process, these electrons are solvated by polymer molecular dipoles, which subsequently merge. The electrons at that time exist in a quasi-one dimensional charge density state which potentiates pairing, and superconductivity.

This electronic self-organization results in the formation of stable structures in the polymer, called superpolarons - essentially a complex of polymer molecules surrounding a high density electronic charge string. These superpolarons (diameter of ~ 100 angstroms) aggregate within the polymer, to create macrostructures called 'channels', which contain thousands of superpolarons. These channels, of approximately 1 - 2 micron diameters, can be oriented and moved in the viscous polymer, and frozen in place by crosslinking, or other methods.

Typically, in thin films, a proportion of these channels spontaneously orient substrate to surface, separated on average by 10 microns of dielectric polymer⁴. These channels are the regions of conductivity, and are observable by several methods, including simple electric contact, phase-contrast microscopy, and AFM .

The enhancement of these channels, or bundled superpolaron structures, was a key component of the Phase II work.

⁴ The number of conducting regions per cm² continues to increase with process enhancements.

Illustration, next page:

ATOMIC FORCE MICROSCOPE SCANS OF ELECTRIC FIELD

These AFM images, produced by Parke Scientific, are of an Ultraconductor polymer film following processing to induce conductivity.

The first image (left) is a measurement of topology of a 100 micron square area of film. The probe is in resonant mode above the surface, and two frequency shift measurements are made at each scan point. The brightest regions are raised, about .5 microns (10% of the film thickness).

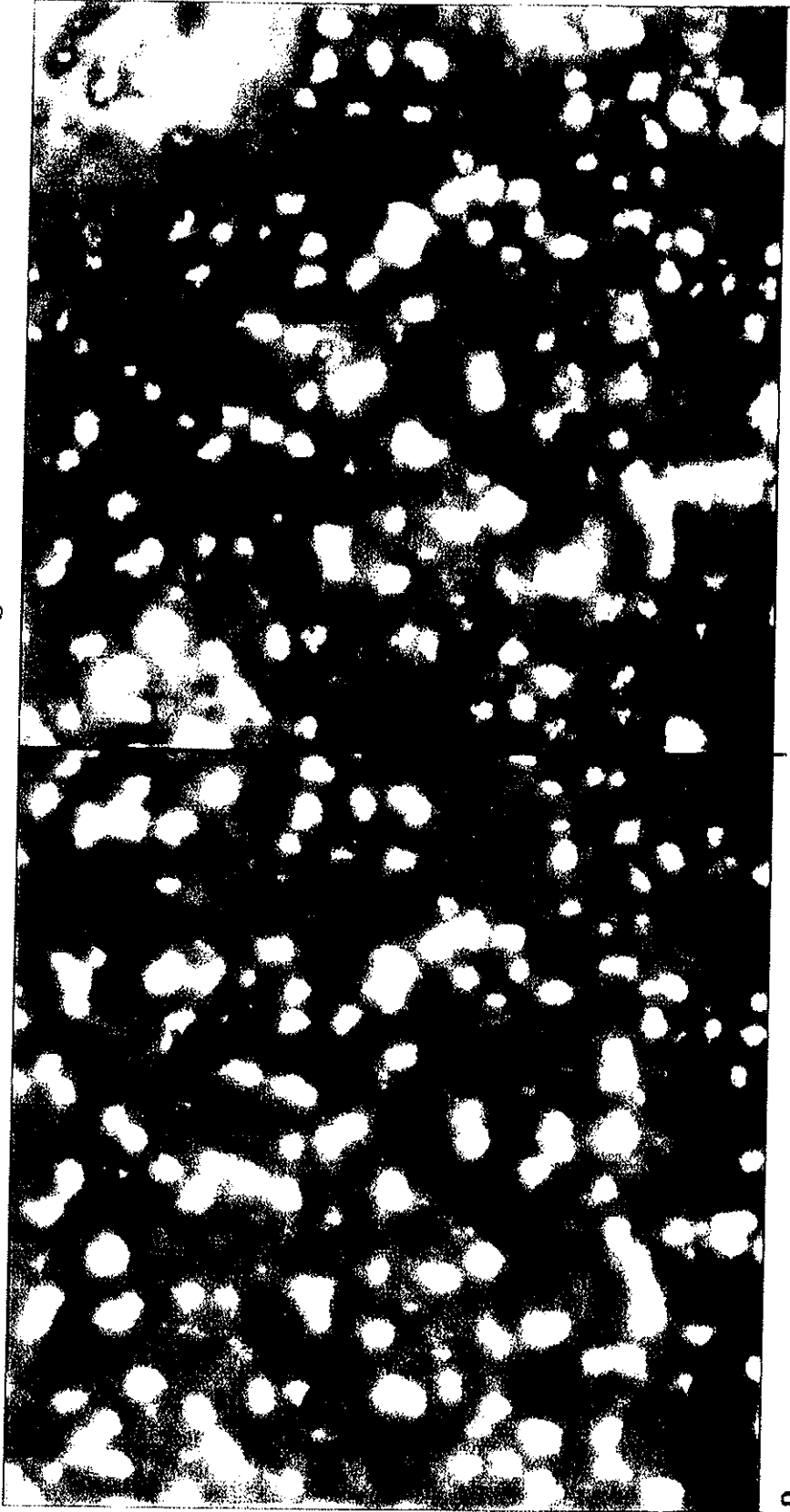
The second image (right) shows the electrostatic field measured at the probe tip, based on an alternating voltage applied to the substrate, and indicates a significant field change at the same points imaged in the topographic scan, presumed to be the conducting channels.

Though not conclusive, (these measurements were not simultaneously corroborated by other testing), it is thought that the lower brightness regions show channels, or aggregates of superpolarons, which are partially grown (eg; not entirely from surface to substrate), and hence are invisible to an electric probe.

The observation of (conducting) points in these images coincides with previous estimates of their distribution, made by sampling with electric probe. The topological features also correspond to the present model of channel structure and formation.

As a way of measuring the size and density of conducting points, AFM can be a valuable tool for monitoring the effect of process changes, making visible channel growth patterns, and enabling advanced experiments on the effects of local electric or magnetic fields.

RSTMAFM Images



0 100 μm 0 100 μm
Data type Z range Height 2.00 μm Data type Z range Frequency 20.0 Hz

23vfm.000
23 Volts

#2

3.c Advanced Development Approaches to Thick Films

It was known that as the thickness of Ultraconductor films was increased, the number of conducting channels which could be measured diminished proportionally. This was determined to reflect the distribution of lengths of the conducting channels themselves. Consequently, the primary task for creating thick films was considered to be the creation of longer channels in the films.

There is no theoretic limit to the length of the conducting channels. From prior experimental evidence, it was shown that the length of the conducting channels (and consequently thicker conducting films) depended on the relative abundance and availability of the superpolarons which create them.

Several experimental approaches, which focused on increasing these superpolaron concentrations, had shown significant promise.

This Phase II contract investigated, and subsequently developed, these process and fabrication techniques, based on the experimental demonstrations.

4. TECHNOLOGY DEVELOPMENT: PART ONE

4.a Preparation and Instrumentation

Since, prior to 1993, the Ultraconductor material had been researched primarily in Moscow, the state-of-the art instruments, know-how and methods for thick film technology had to be transferred to MPI. To further accelerate commercialization, it was also a goal to use materials which are readily available and standard in the U.S.. Fulfillment of these steps coincided with the Phase II contract. Hence there were several parallel tasks in the short term of the project.

Two labs were expanded and set up for applications and development work. The smaller, which needed a stable concrete foundation, was devoted to the Magnetic Balance, a very sensitive instrument. The larger lab was dedicated for the bulk of the work, including material processing, instrument making, and measurements.

4.a.1 Polymer Preparation:

Three atactic polypropylene polymers from Scientific Polymer Products, Inc. were acquired to be used for the project. Softening points of the polypropylene varied from 20° C to 150° C. Initially the polymers were processed as-received from the supplier.

It was later determined preferable to make a solution of the polymers prior to further handling during the processing steps. Solutions could be filtered to

eliminate undesirable components⁵, and solutions were superior for storing the purified polymer, and were used to deposit polymer films on various substrates.

Hexane and heptane were initially chosen to make the polymer solutions. With further testing it was discovered that toluene was superior, due to its greater solvating characteristics, especially for the longer chain polymers.

The three standard phases of Ultraconductor polymer preparation are 1) oxidation at elevated temperatures, 2) Ultraviolet irradiation and 3) mechanical stressing. The oxidation procedure was not changed throughout. The latter two steps, which are done simultaneously, were.

Initially procedures 2 and 3 were accomplished by depositing a layer of polymer on a rotating disc located under a UV lamp with a stationary stirrer made from an angled wire inserted into the polymer. This stirrer was replaced with a stationary upper disc made from UV transmitting fused quartz, which enabled a shearing stress⁶ to be applied to the polymer during irradiation.

A polymer preparation carousel was built to enable the processing of polymer samples on 10 substrate discs simultaneously. All of the samples were rotated around the carousel so that each received an identical amount of UV radiation, while being subjected to shearing stresses between UV transmitting quartz discs.

Samples were then prepared by differing exposure times to UV radiation; film thicknesses; with and without shear stressing; and differing exposure times at oxidation temperatures. The materials produced by each of these methodologies was characterized, in order to measure the effectiveness of each of these processing steps.

As an adjunct to the primary method of film deposition, a technique was developed for laying down a uniform film of polymer of any thickness on a disc by evaporation of a known volume of a polymer solution of a known concentration. A solvent recovery system was developed which was 98% efficient for recycling solvent used in making the polymer solutions.

4.a.2 Instrumentation:

Three types of instrument were built to enable the measurement of the Ultraconductor samples and to develop methods of increasing the thickness or

⁵ UV transmission measurements were conducted for the various polymers, and it was found that the unfiltered polymer was opaque to UV at the wavelengths that were most effective, while filtered polymers transmitted well up to thicknesses of 100 microns.

⁶ The ultimate objective of thick films with high densities of conducting channels was approached by increasing the concentration of conducting elements (superpolarons) in two ways. The initial approach was mechanical stressing, and a later approach was concentration.

The mechanical stressing approach was based on an experiment that indicated a higher concentration of free electrons could be produced.

length. The first investigations were to determine whether the films' thickness (ie, the length of continuously conducting channels through the film) could be increased without simultaneous electric contact.

The first device was essentially a pair of plates with the sample between, which would elastically separate as force was decreased, thereby gradually increasing the thickness of the film.

In the second device the film was placed between the polished ends of two brass strips, which could be moved apart with sub-micron resolution by a cantilever technique.

The third and most successful instrument used a micrometer with an LED readout which controlled the motion of two brass pin electrodes into the polymer, which was placed on a flat faced electrode. One of the brass pins was set to contact the flat electrode in a dry area, free of polymer. This was the control electrode, which established the zero position of the micrometer when it was conducting.

5. TECHNOLOGY DEVELOPMENT: PART TWO

5.a Initial Approaches

Each instrument was tested with a variety of samples prepared from 3 types of polypropylene.

In general the instrument and sample were heated to ensure that the viscosity was appropriate for the sample to flow as the gap (thickness) was increased. Measurement of gap distance required great care, as the instrument was inevitably subject to some temperature gradients.

Two procedures were commonly used to extend the distance while maintaining conductivity, via a channel. In the third test device, as the pin-to-substrate distance increased, the polymer was extended and subjected to a high voltage gradient at the tip of the pin. This voltage gradient was attractive to the conducting elements, and effective in extending the conducting path.

When conductivity was lost, the voltage was increased, up to tens of volts, with a maximum of 50 volts. (The current was always limited under these conditions.) Often conductivity would be regained; at other times the distance had to be decreased to regain contact.

A technique referred to as training the polymer was developed. This is a process of subjecting the sample to voltage pulses of increasing magnitude. It was previously known that the current carrying capacity of individual channels could be increased in this manner until a level of up to 50 amps would flow through a

channel in short pulses⁷. The technique was demonstrated to result in permanently higher current carrying capacities.

Thereafter, training was done before increasing the distance to enhance the current carrying capability of the extending channel. The current would then be reduced to about 1 mA before increasing the distance, typically by one micron increments. Consequently, as the brass pins were moved away from the flat-faced electrode, conduction was maintained through training.

These techniques - though difficult to implement, as a consequence of working in regions of micron dimensions - were successfully employed to increase the film thickness (length of conducting path) by up to 10 microns, demonstrating their effectiveness. However, the limits of the approach were clear. It was reasonably resolved that the density of conducting elements were still insufficient to enable longer channel formation.

This conclusion, that a higher density of conducting elements was required, shifted the focused development and investigation to the enrichment approaches.

5.b Measurement: the MAGNETIC BALANCE

Counting the number of conducting regions, or measuring the film thickness, provides only a crude indicator of the density of conducting elements in the polymer. In order to measure changes in that density in a useful way for development, a sensitive instrument is required. The Magnetic Balance was designed and built for that purpose several years ago, and has evolved through three iterations to date.

The Magnetic Balance measures the magnetic properties of samples of polymer in a non-uniform magnetic field. The magnetic measurement is directly proportional to the number of "free" electrons in the sample, and hence the density of superpolarons.

The main coils can provide fields up to 2 Tesla, and there is also an auxiliary minor coil for fine adjustments. When adjusted the Balance is sensitive to forces as low as 10ngm, or 10^{-10} Newtons! At 10 ngm, it is sensitive to an electron concentration of 10^{15} electrons/cc, which is 0.1% of the desired concentration of 10^{18} electrons/cc and hence is excellent for monitoring the early growth of polarons.

⁷ The highest experimentally measured amperage per channel to date was ~ 100 Amps. Pulsed currents are used in order to prevent heating / melting of metal electrodes and circuit leads.

An analysis was done of the Magnetic Balance sensitivity.

Force = 10 ng = 50 "units" ("shift" or "rotation")

(Repeatability if no contamination)

Sensitivity: 10 ng = 10×10^{-6} dynes

Magnetic moment (emu) = 10^{-5} dynes / 150 gauss/cm = 6.7×10^{-8} emu

SENSITIVITY

<u>FIELD</u>	<u>Mag.moment</u>	<u>emu/unit</u>	<u>electrons</u>
kGauss	$\times 10^{-8}$ emu (50 "units")	$\times 10^{-8}$	per cc (in 1 mg)
0.1	50	1	10^{17}
1	5	10	10^{16}
10	0.5	100	10^{15}

COMPARISON OF SENSITIVITIES

Magnetic Balance: 1 kGauss -	5×10^{-8} emu
10 kGauss	0.5×10^{-8} emu
SQUID: All fields	1×10^{-8} emu
National Magnet Lab: (Cantilever force magnetometer)	10×10^{-8} emu
George Assoc. Gradient coil	5×10^{-8} emu

Hence the Balance is equal to or better than the best magnetometers available. Note that a SQUID is not suitable for measurements of Ultraconductors, since the polymers do not produce a spatially large field to link with a pickup coil.

In the final months of the project, components of the Magnetic Balance failed, requiring significant repair, and major calibration. This delayed full quantification of the measurements of the successful enrichment process, described next. (A more detailed review of calibration, alignment and measurements using the Magnetic Balance are reviewed in Appendix A.)

5.c The Enrichment Process

5.c.1 Overview

The previously described results of tested approaches to making thicker films had verified that a higher density of conducting structures were necessary. A good analogy to the effect of enriched material is the percolation probability in normal materials processing.

Prior to this contract, a series of experiments had given preliminary confirmation of two techniques for concentration - resulting in density increases of greater than a factor of ten. To "concentrate" means to separate in some fashion the superpolaron from the non-superpolaron polymer, and to either collect the superpolaron, or to remove the non-superpolaron material. By adopting this approach, the initial density of superpolarons produced in a film, which up to now had been the focus, became less critical.

In addition to the concentration, process steps for collecting the polymer were developed. The techniques investigated utilized the fact that superpolarons have an electric dipole moment while the bulk of the polymer is electrically neutral. One technique involved collecting superpolarons from many large plates (actually in cylindrical form).

In the first embodiment of the test setup for isolating superpolarons, the prepared polymer was in the form of a thin film on a metallic tube which was rotated. A thin wire was placed in close proximity to the tube surface. Prior to testing, the copper wire used to collect the superpolarons was evaluated on the magnetic balance.

In this setup, a high voltage was maintained between the wire and the tube, creating a non-uniform electric field⁸. The electric field in the vicinity of the wire surface produced a steep gradient, which attracts superpolarons in its vicinity.

The wire was removed from the apparatus and analyzed in the magnetic balance to determine the density of superpolarons.

5.c.2 Superpolaron concentration

The first attempt at isolating the superpolarons from a polymer sample was made using SP # 780 which had been dissolved in toluene and then filtered. An aluminum tube was substituted for the brass tube to duplicate the previous successful experiment as closely as possible.

The aluminum tube (measuring 4.775 cm diameter, 4 cm in length and 1.65

⁸Since we do not have a high voltage generator at kilohertz frequencies, a circuit was built to generate the waveforms using two 1000v amplifiers. An alternating frequency is desirable in case there are ions present in the medium - which could bombard the wire in a dc field.

mm thickness), was weighed and then dipped into the polymer/toluene solution for 3 minutes. The toluene was then allowed to evaporate and the tube was weighed a second time. The difference in weights was assumed to be due to the polymer coating on the tube surface. Calculation of the average polymer thickness gave a result of 1.17 microns.

The tube was allowed to bake in the oven for 2 hours at a temperature of 100° C to oxidize the polymer. The inner tube surface was then irradiated for 0.5 hours with the UV lamp placed axially inside the tube, using a cooling fan to limit the tube temperature to 50 degrees during the process. The 30 minute irradiation time was chosen because the lamp-to-surface distance in this setup was one-half of that for the previous irradiation setups and the film thickness was much less.

A pure copper wire (99.9985 % pure, 0.25 mm dia.) was placed on a copper mount such that it was parallel to the lower part of the tube inner surface and within 0.5 mm of the surface. The tube was then placed in a hollowed out aluminum block and was rotated at a rate of 4 mm every 15 minutes, to expose the polymer to the electric field generated by the wire. The wire was connected to an alternating voltage of 1000v, to induce the superpolarons to migrate through the medium to the wire, to which they should adhere strongly - first, by electrostatic attraction. This creates intimate contact, and adhesion, by Van der Waals forces.

By evaluating the copper wire's magnetic moment on the Magnetic Balance, before and after the test run, results were obtained which demonstrated the increased concentration density of superpolarons in the enriched batch sample.

Though the test results were positive as expected, further runs could not be obtained in order to accurately quantify the results for this report. This data will be collated following completion of the Magnetic Balance repairs.

However, the preliminary results for the enrichment process obtained under this contract are consistent with precise experimental measurements performed in the initial research of the technique, by an independent ROOTS team.

Consequently, the results of the tests done under the contract may be summarized:

- It was verified that unlimited lengthening of the conducting regions is not possible without enrichment of the polymer material prior to fabrication;
- Enrichment was demonstrated, and techniques were tested;
- An optimum technique was verified and enhanced, which will enable commercially viable production of Ultraconductor polymers, fabricated thick and thin films, and short wire lengths;
- Scale up of these and modified techniques were modeled.

With the successful demonstration of enrichment, the development

challenge shifts to one of production volume. The design and testing of scaled apparatus for enrichment is the remaining objective necessary for the consistent production of enriched Ultraconductor material.

Consequently, the Phase II project resulted in the key goal necessary for producing thick films and wire, for commercial applications.

6. CONCLUSIONS AND COMMERCIAL IMPLICATIONS

The accomplishments of the Phase II project may be summarized as follows:

- Two laboratories were set up;
- Key additional personnel were trained in the technology;
- An instrument essential to the technology (Magnetic Balance) was installed, calibrated, and brought on line, and personnel trained in its use, calibration and maintenance;
- Multiple techniques were developed and compared for producing Ultraconductors using standard, commonly available materials;
- A variety of methods were developed and compared, to increase the number of superpolarons in the material, resulting in higher value films;
- Finally, an enrichment technique was developed which increases the density of superpolarons by an order of magnitude, creating the ideal material to fabricate thicker film and wire.

Thick film was not achieved in the time frame of the project (18 months). However, while disappointing to the project team, that objective has limited significance, relative to the achievement of the enrichment process, which has far more profound implications for the subsequent development of the technology.

The enriched Ultraconductor material enables fabrication of films and wire with unlimited thickness and length. Confirmation of the enrichment process establishes a standard production methodology, which can be licensed for industrial scale Ultraconductor manufacture.

Further, the enriched polymer provides optimal qualities necessary for additional commercial breakthroughs. One aspect likely to benefit is the development effort to create three dimensional conductivity in Ultraconductor films.

Presently, the Ultraconductor film is an anisotropic conductor, with conductivity only normal to the plane of the film. This results from a preferential spontaneous orientation of channels.

Techniques have already been developed which enable the selective

reorientation, manipulation and alignment of these structures in the film. However, as was the case with thicker film, the limit of these techniques is determined by the abundance of superpolarons, which is addressed by enrichment.

Consequently, the apparent limited success of this project will undoubtedly make multiple paths to commercial products shorter and less risky, while opening new ones - a very significant step.

7. COMMERCIALIZATION RESULTING FROM THIS CONTRACT

The practical and commercial value of Ultraconductor thick films is illustrated by the following comparison.

Estimates from measurements of Ultraconductor films show a significant advantage to the current carrying capacity of the state-of-the-art HTS ceramic YBCO superconducting tape recently produced at Los Alamos National Laboratory.

COMPARISON OF ULTRACONDUCTOR FILM CURRENT DENSITY

	<u>Current density (A/sq cm)</u>	<u>Engineering current density</u>
Ultraconductor	5×10^9	1.5×10^7
LANL Tape (YBCO)	1×10^6	2.0×10^4
Copper		2.0×10^2

The Phase II project has brought Ultraconductor technology several steps closer to use in commercial applications. In particular, enrichment of the polymer is key to optimal fabrication of thick films, leading to a variety of products.

At the beginning of the Phase II project, it was expected that batteries and supercapacitors might be the first products commercialized and introduced, but several factors have advanced the application to thermoelectric devices, making them the likely early product. The achievements of this Phase II - putting in place the means to make thick films in dimensions and quality required for passive thermoelectric components - are the basis for the thermoelectric SBIR contract(s) with BMDO.

Subsequently, the steps to complete the Phase II work will be to compare the two most promising enrichment techniques under development, and to scale up production of materials for the one chosen. Quantities produced are presently limited by the size of the experimental devices (which has made production of films prior to contract expiration difficult). Initially minuscule volumes of enriched

polymer will be available, and so increased output is regarded as critical to advanced testing of film and wire fabrication.

Specifically with regard to the commercial objectives of this contract, ROOTS has initiated discussions with individual corporate alliance partners for thick film applications to thermoelectric modules; connectors; advanced capacitors; and polymer film fabrication.

Consequently, the achievement of thick films from this contract directly enables the commercialization of Ultraconductor-enhanced products.

8. COMMERCIALIZATION STRATEGY

Following development and prototype introduction, ROOTS will not internally produce or sell Ultraconductor products, except for pilot or highly profitable segments. Instead, the company will enter joint development and licensing contracts to ensure the rapid commercialization of the technology.

This approach, which is consistent with existing superconductor commercialization, effectively leverages the interests and resources of international corporations which already manufacture and sell conventional versions of the products proposed, such as cable, and motors. The largest have excellent market position, experience in the field, and significant capital resources.

Licensing or joint development agreements with these corporations will enable the experience and resources of these companies to accelerate each step of the development to market process. Competitive or simultaneous agreements, if acceptable, would further drive commercialization. Importantly, this approach enables our company to focus on core technology, and further development.

Such strategic alliances will reduce or eliminate duplicative start-up costs, allow parallel design and production cycles, reduce time-to-market, increase market penetration, and increase the speed with which industrial sectors can adopt and employ the new technology. They also ensure early technical and design feedback, essential to our own development approaches and objectives, a process already begun.

9. ULTRACONDUCTOR MARKET OPPORTUNITIES

Fundamentally, Ultraconductor materials - fabricated as film and wire - will deliver exceptional advantages in weight, size and electrical efficiencies over present day conductors. These advantages have already been anticipated by global industrial sectors, based on the expected application of cryogenic superconductors.

The potential markets for products incorporating those superconductors are established at International Superconductivity Industry Summit (ISIS) conferences

Ultraconductor™ Product Opportunity Chart

Product Distribution By Market Segment and Application Category

Scale of distribution does not reflect market value or product sales in dollars.

MARKET SEGMENTS	ELECTRIC GENERATION	ELECTRIC DISTRIBUTION	ELECTRIC USE
DEFENSE <i>8 Product Groups</i> 	GENERATORS (Induction) (TE)	WIRE ENERGY STORAGE	MOTORS
AEROSPACE <i>8 Product Groups</i>		Connectors Assemblies	SMES Ultracapacitors Battery Systems TE (cold) MAGNETS
MANUFACTURING / PROCESS INDUSTRIES <i>8 Product Groups</i>			
TRANSPORTATION (Mass and Private) <i>10 Product Groups</i>		CABLE Transformer Current Limiter	
UTILITIES <i>7 Product Groups</i>			
COMMUNICATIONS <i>6 Product Groups</i>			
MEDICAL & SCIENTIFIC <i>10 Product Groups</i> <i>(TE not shown)</i>		Coax Fiber Optic Cable Connectors Interconnects	Magnets Special Motors
ELECTRONICS <i>5 Product Groups</i> <i>(TE not shown)</i>			IC'S Josephson Junctions
ELECTRO-OPTICS <i>3 Product Groups</i>		Photonic Switches Electro-Mag Shielding	Sensors Actuators

of superconductor experts and more than 70 companies. According to these studies, international (cryogenic) superconductor applications markets will grow to \$150 - \$200 billion by the year 2020.

For example, the application of superconductor motors, generators, and transmission cable in U.S. utilities has the potential of reducing losses by 35 - 50%, translating to savings of as much as \$5 billion per year.

Growth in world energy consumption presents further new market opportunities. The level of world energy consumption is projected by the World Resources Council to increase as much as 50% over the next 25 years, led by growth in the use of electricity. In 1993, worldwide markets for underground power cable alone was estimated at \$700 million. According to the U.S. National Energy Plan, the estimated international market potential for U.S. energies technologies is more than \$200 billion through 2010. The World Bank estimates that investments of \$4 trillion will be needed during the next 30 years to meet developing countries electricity needs alone.

The actual potential markets (see chart next page) for products incorporating Ultraconductors are expected to exceed present projections for several reasons. Versus present superconductors, ambient temperature polymer Ultraconductor material enables commercially equivalent products in almost every case, but at potentially lower capital and lifetime costs, with reduced risk of failure, and without cryogenic support. Additionally, Ultraconductors create expanded market potential, since it is ideal for applications which would be impossible for refrigerated superconductors.

The Nikkei Industrial Research Institute concluded that markets would be seven times greater for ambient temperature superconductor products, than for cryogenic superconductors.

APPENDICES

Appendix A

MAGNETIC BALANCE: ALIGNMENT AND MEASUREMENTS

The following actions were undertaken.

The mechanism for locking the balance beam (BB) in place was not working, necessitating the replacement of a plastic membrane. It was observed that the locking mechanism screws were aluminum and could affect the capacitance measurement, which is the heart of the balance, so plastic end sections were machined for the screws. Readjusting these screws so that, when locked, the beam is centered is a tedious procedure.

The balance mechanism sits on a foam pad, which was replaced and re-levelled. In addition the magnet structure was detached from the balance, so that it could be leveled independently. This also allowed the centering of the quartz rod (which holds the sample cage) in its protective tube.

The fixed capacitive plates were then reset using a special gauge, and the screws locked in place. The trimming resistors and capacitors were adjusted to balance the voltage bridges which measure the capacitance. The suspension threads for the BB were set closer to increase the sensitivity. These threads are of fine glass, and later broke as a sample was being hung on the quartz rod. Replacing them was difficult. The last task in the calibration was to adjust the suspension points to perfectly center the beam, replace the screens, and then measure the sensitivity.

The balance was calibrated with a resolution of about 10 ng force. To put this in perspective, when a modest field of 1.1 Kgauss is applied (10 volts on the coils) it creates a differential of 300 gauss (from readings on the Hall sensors) across the 2 cm gap, or a field gradient of 150 gauss/cm. The size of sample measured is generally about 1 mg, and a good sample of Ultraconductor may contain 10^{17} electrons/cc, or 10^{14} electrons per sample. The magnetic moment of each electron is 1 Bohr magneton = 9.27×10^{-21} ergs/gauss (approx 10^{-20}). Hence, the force on the sample is 10^{14} electrons $\times 10^{-20}$ ergs/gauss $\times 150$ gauss/cm = 150×10^{-6} dynes = 150 nanograms.

It was found that the quartz rod, which should be diamagnetic, was contributing 1600 ng paramagnetic, due to impurities. The end of the rod was broken off and replaced with a good piece, which became diamagnetic, and then a small quantity of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ was added to bring it as close as possible to zero.

One method of calibration is to apply a known voltage to one of the plates of the capacitors in the balance. This produces a force on the beam proportional to the square of the voltage. A force of 100 ng is induced by 0.045 volts, and this corresponded to about 500 "units" of measured capacitance change = torque = force.

To make measurements of Ultraconductors the sample is adhered to, or coated on a copper wire. The measurement is the difference between the wire before and after coating. Several samples of copper wire were tested and all were paramagnetic, indicating impurities - this is undesirable since impurities being of unknown origin could be changed by handling or processing. A pure wire was bought - 99.9985%. The first sample tested showed 10^{-5} ferrous.

Since the signal of a ferromagnetic sample may be 200 million times larger than a diamagnetic or paramagnetic sample of equal weight, even a microscopic ferromagnetic impurity can, if undetected, result in a significant measurement error.

Other samples tested diamagnetic as desired. The samples of wire to be used are cut with a piece of glass to avoid impurities from steel blades. The first run of the concentration experiment was inconclusive because the sample came into contact with a ferrous holder. Holders are now non-ferrous.

Measurements on the Magnetic Balance presently take about 30 minutes per sample, assuming there is no contamination. The sample must be handled with great care. Loading the sample upsets the Balance and a few minutes are required for it to settle - normally not in the same "zero zone" as before. The balance is re-zeroed by adjusting the voltage on the plates. The reading is taken, typically at several levels of flux intensity. The sample is then carefully unloaded.