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REPORT DOCUMENTATION PAGE

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	5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME AND ADDRESS Clemson University College of Eng/Science Clemson, SC29634-1911	8. PERFORMING ORGANIZATION REPORT NUMBER
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14. ABSTRACT The main objective of the Clemson / DARPA project is to search for and achieve higher performance low temperature (100 K to 250 K) thermoelectric materials than exist to date. Dr. Tritt's group at Clemson University will perform synthesis and characterization of a number of low dimensional systems which show potential for use in thermoelectric applications. Initially Tritt's group has been and will continue to pursue studies of the low-dimensional pentatelluride materials: HfTe5 and ZrTe5. Tritt's group will perform doping studies of the thermoelectric properties of these materials as well, which will include compounds such as Hf1-XZrXTe5. Subsequent doping studies on this family of compounds will include other substitutions, Hf1-XXTe5-YZY, where M = Zr, Ta, Nb, Ti etc. and Z = S or Se. Tritt presented the preliminary results of the (Hf1-XZrXTe5) study at the XVI International Conference on Thermoelectrics in Dresden. Other promising low dimensional materials will also begin to be investigated.

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6. AUTHOR(S) Dr. Terry M. Tritt Department of Physics & Astronomy, Clemson University, Clemson, SC 29634-1911				
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12 a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited.			12 b. DISTRIBUTION CODE	
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G - Grant	TA - Task
PE - Program Element	WU - Work Unit Accession No.

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Block 10. Sponsoring/Monitoring Agency Report Number. (if known)

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REPORT DOCUMENTATION PAGE (SF298)
(Continuation Sheet)

Final Progress Report

5/15/97 thru 5/15/01 (DARPA / ARO)

DAAG55-97-1-0267

(Terry M. Tritt, PI, Clemson University)

Personnel and Their Responsibilities Being Supported by Project

Dr. B. M. Zawilski (Postdoc until February 28, 2001):

Responsible for design, fabrication and calibration of new parallel thermal conductance (PTC) thermal conductivity measurement system. Performed initial measurements on a number of standards and pentatelluride materials.

Dr. D. Ketchum (Postdoc until December 2000):

Responsible for synthesis and structure characterization of pentatelluride series. Will perform necessary doping and solid state chemistry to advance project. Will perform initial measurements on low temperature structure.

Dr. Terry M. Tritt (Professor and PI): (3/4 summer month)

Responsible for oversight and direction of project, including determination of doping studies and new materials to investigate, oversight and management of personnel, oversight and responsibility of budget and purchases.
Laboratory manager and writing reports and manuscripts.

Dr. Joe Kolis (Co-PI, Chemistry): (3/4 summer month)

Responsible for oversight of project concerning chemistry, synthesis and characterization.
Oversight and supervision of Chemistry Postdoc

Dr. Matt Marone (Postdoc-one year): Responsible for design, fabrication and calibration of new thermal conductivity system. Performed initial measurements on materials.

Dr. C. R. Feger (Postdoc): Responsible for synthesis and structure characterization of pentatelluride series. Performed necessary doping and solid state chemistry to advance project. Will perform initial measurements on low temperature structure.

Personnel and Their Responsibilities Being Supported by Project

Mr. R. T. Littleton (Graduate Student): (Full time support)

Responsible for measurements and characterization of electrical properties of all pentatelluride materials.

Design, fabrication and calibration of high temperature probe ($80 \text{ K} < T < 700 \text{ K}$)

(received Masters degree in Physics 8/98)

(received Ph.D student in MS&E at Clemson, May 2001)

Mr. Nathan Lowhorn (Graduate Student): (No support at this time)

Responsible for taking over the measurements and characterization of electrical properties of all pentatelluride materials.

(received Masters degree in Physics 8/00)

(currently Ph.D student in Physics at Clemson, degree expected Aug/Dec 2003)

Mr. Scotty Nicholson (Machine Shop Technician): (1/4 time) Responsible for machining and fabrication on thermal conductivity and Hall systems as well as other miscellaneous items.

Mr. Michael Long (Machine Shop Technician): (1/4 time) before Nicholson;

Responsible for machining and fabrication on thermal conductivity and Hall systems as well as other miscellaneous items.

Undergraduate Students:

Mr. John Cook, graduated 2001. (3 summer months, P/T during semesters 2000)

Responsible for general lab duties, sample prep, etc.

Mr. Jason Jeffries, graduated:2000 (2 summer months, P/T during semesters 2000)

Responsible for general lab duties, sample prep,

Design, fabrication and calibration of high temperature probe ($80 < T < 700\text{K}$)

Mr. Raymond Tedstrom, graduated 2002: (2 summer months, P/T during semesters 2000)

Responsible for general lab duties, sample prep,

Design, fabrication and calibration of high temperature probe ($80 < T < 700\text{K}$)

Overall Program Objective

The main objective of the Clemson / DARPA-ARO project is to search for and achieve higher performance low temperature (100 K to 250 K) thermoelectric materials than exist to date. Clemson University will perform synthesis and characterization of a number of low dimensional systems, which show potential for use in thermoelectric applications. Low dimensional systems can lead to enhancement of the electronic properties over that of bulk materials. This research will be carried out within the Department of Physics and Department of Chemistry at Clemson University. Initially Clemson University will pursue studies of the low-dimensional pentatelluride materials: HfTe_5 and ZrTe_5 . These materials show promise for potential low temperature thermoelectric applications. Low temperature thermoelectric materials in the 100 - 200 K range can open potential applications for certain IR detectors, especially the mid to far infrared detectors. If HgCdTe detectors could be thermoelectrically cooled and packaged then much faster response times and higher sensitivity could be achieved over current thermal imaging systems such as bolometers. Thermoelectric cooling to 100K would make the prospect of packaged superconducting electronics more achievable. Also, enhanced computing speed through "cold computing" can be achieved by cooling computer chips and electronics to temperatures in the 100 K to 250 K range.

A secondary objective was to develop state-of-the-art facilities for the characterization of the electrical and thermal properties of thermoelectric materials over a broad range of temperature and for various sample sizes. This will allow Clemson not only to pursue high quality measurements on its own samples but to serve as a measurement facility for the greater thermoelectrics community which were involved in the Darpa Thermoelectrics program.

Previous Plans for the final 8 months of project (completion date May 2001)

Materials and Doping Strategies:

Single crystals of the pentatelluride materials will continue to be grown via solid state synthesis in the Chemistry Department and characterized in the Physics Department for their potential for these low temperature (100 K - 250 K) applications in thermoelectric refrigeration. Clemson University will perform doping studies of the thermoelectric properties of these materials as well, which will include compounds such as $\text{Hf}_{1-X}\text{Zr}_X\text{Te}_{5-X}\text{Se}_X$ and $\text{Hf}_{1-X}\text{Zr}_X\text{Te}_{5-X}\text{Sb}_X$. More general doping studies on this family of compounds will include other substitutions, $\text{Hf}_{1-X}\text{M}_X\text{Te}_{5-Y}\text{Z}_Y$, where $\text{M} = \text{Zr, Ta, Nb, Ti}$ etc. and $\text{Z} = \text{S, Se and Sb}$. In most cases small amounts of impurities including magnetic elements such as Fe, Co or Ni will be added. The addition of Bi into the structures will also be attempted as well as other broad range of doping strategies. In addition, we will pursue rare earth doping of these materials with the addition of Ce, La, Ho Eu etc. These studies will lead information important to optimize the electronic part of the figure of merit, ZT, for these materials. These materials will be characterized over a range of temperature from 350K to 20K in most cases.

In addition to the overall doping studies to investigate the optimal doping strategy we will also investigate synthesis routes to lead to much larger crystal sizes, with the overall goal crystals of approximately 1-2mm on a side and greater than 4mm in length. Since the larger crystal growth approach has proved fruitless so far, we have developed a new concept and approach to determine the absolute thermal conductivity which we call the parallel thermal conductance (PTC) technique. Initial measurements of the thermal conductivity using our standard steady state techniques will begin and approaches such as alloy or magnetic scattering for the phonons will start to be employed towards minimizing the lattice thermal conductivity.

We will continue to investigate the thermal conductivity of these materials.

We have also identified a NaCoO system (a ceramic oxide) which we have been as a potential high temperature power generation applications.

Experimental and Capability Development:

Experimental capabilities continue to be developed and enhanced. The acquisition of the Quantum Design Physical Property Measurement System under DOD-DURIP (May 1998) has been a great addition to the lab. It allows magneto-transport and heat capacity measurements to be performed. We are planning to bring on-line a new electrical transport measurement system capable of measuring the Seebeck and electrical resistivity of two samples simultaneously as a function of temperature from $6\text{ K} < T < 320\text{ K}$. When this new system is operational our existing system will be reconfigured in the same way (however it is a 10 K limited system). We have also built a high temperature probe to cover a broader range in our characterization of materials. We redesigned our thermal conductivity system (PTC) to be able to increase our measurement capability. We will put together a Hall measurement system to investigate carrier concentration of these materials.

**Accomplishments under this contract during the term of the contract
(5/15/97 thru 5/15/01)**

Single crystals of the “parent” pentatelluride materials HfTe_5 and ZrTe_5 have been synthesized as well as extensive doping of the other phases: $(\text{Hf}_{1-x}\text{Zr}_x\text{Te}_{5-x}\text{Y}_x)$ ($\text{Y} = \text{Se}, \text{Sb}$). The thermoelectric power and resistivity have been measured on all the samples as a function of temperature from 320 K to 10 K.

We have had some substantial successes during the project period. These centered around substitutional doping of Sb and Se on the Te site in the HfTe_5 materials, $(\text{Hf}_{1-x}\text{Zr}_x\text{Te}_{5-x}\text{Y}_x)$ ($\text{Y} = \text{Se}, \text{Sb}$). The addition of small amounts of Sb resulted in totally suppressing the resistive anomaly in the pentatelluride materials. The resistivity is somewhat lower and has a metallic temperature dependence, resistivity decreasing with decreasing temperature. The relatively high thermopower values are maintained, $S \approx 100 \mu\text{V/K}$ at $T = 300\text{K}$ and decreasing with decreasing temperature. These results on the electronic properties exhibit power factor values greater than that of Bi_2Te_3 materials, over the temperature range of interest.

We continued the doping studies but the results of period warranted an intensive effort into measuring the thermal conductivity of these small ribbon-like single crystal materials. This is where we have focused our efforts over the two years of the project and with much success. We developed a new technique which we call the parallel thermal conductance (PTC) technique. The issue is that because of the size of these materials, it is very difficult for them to support a heater and the necessary thermocouples. The PTC technique allows us to bypass this problem. Briefly, the PTC technique uses a sample holder which has very low thermal conductance and the heaters and thermocouples are attached to this holder. The thermal conductance is measured of this “system” which gives the background thermal conductance when the sample is added in parallel. The background is then subtracted leaving the thermal conductance of the sample. There is still an issue concerning the resolution of the sample dimensions, which yield an uncertainty of 40-50 %. This technique was published in the Review of Scientific Instruments in 2001. The results on the thermal conductivity of the pentatellurides has been published in Appl. Phys. Lett. We have also published several papers on some of our novel measurement techniques and apparatus which can be found in the publications section.

The addition of small amounts of Se resulted in reducing the overall resistivity and enhancing or increasing the thermopower to values over $200 \mu\text{V/K}$ at $T = 100\text{K}$. These are very large thermopower values at these temperatures in a system that is still a relatively good conductor. These effects resulted in power factor values greater than that of BiTe materials over the temperature range $100 \text{K} < T < 320\text{K}$. This was especially the case in the range of interest $T = 150\text{K}$. Initial determination of the thermal conductivity indicates that it is a factor of 2-3 times higher than desired. We are continuing the project with doping with heavy mass atoms to reduce the thermal conductivity.

Accomplishments under this contract during the term of the contract

(5/15/97 thru 5/15/01)

Cont.

A secondary objective of the project was to develop state-of the art facilities for the characterization of the electrical and thermal properties of thermoelectric materials over a broad range of temperature and for various sample sizes. This will allow Clemson not only to pursue high quality measurements on its own samples but to serve as a measurement facility for the greater thermoelectrics community which were involved in the Darpa Thermoelectrics program. We developed excellent facilities and participated in other Darpa related projects, such as the opals, the half-Huesler alloys and work on the skutterudites. Many of our novel measurement techniques and apparatus were written up as scientific publications and published in review of Scientific Instruments, Cryogenics and the MRS Proceedings.

Final Progress Report
5/15/97 thru 5/15/01 (DARPA / ARO)
DAAG55-97-1-0267
(Terry M. Tritt, PI, Clemson University)

CLEMSON GROUP PRODUCTIVITY ON
ARO/DARPA Grant # DAAG55-97-1-0267
1/1/00 THRU 12/31/00

PATENTS SUBMITTED:

- Transition Metal Pentatellurides and Transition Metal Chalcogenide Compounds for Potential Thermoelectric Materials: **Terry M. Tritt, (PI)**
Terry M. Tritt, (PI)
submitted to University committee Oct 15 1997
approved for further action-November 24, 1997
action by lawyers to file August 1998, Dec 2000. May 2002

Editorials:

- * 2000 **Semimetals and Semiconductors** (3 Volumes: Volume **69**, **70** and **71**)
Thermoelectric Materials - Current Trends and Approaches
Editor **Terry M. Tritt**, Academic Press

Volume 69: **Thermoelectric Materials – Recent Developments in Thermoelectric Materials: Bulk Materials and Overview – Part I**

Volume 70: **Thermoelectric Materials – Recent Developments in Thermoelectric Materials: Bulk Materials and Overview – Part II**

Volume 71: **Thermoelectric Materials – Recent Developments in Thermoelectric Materials: Thin Film and Superlattice Materials – Part III**

- * Proceedings of 2000 Materials Research Society **Volume 626**
New Materials for Small Scale Thermoelectric Refrigeration and Power Generation
Applications.
Edited by **Terry M. Tritt**, G. Nolas, M. Kanatzidis, G. Mahan and D. Mandrus

Review Articles:

- * 1999 Annual Review of Materials Science:
Skutterudites: A Phonon-Glass-Electron-Crystals Approach To Advanced Thermoelectric Materials Research. by: G. Nolas , D. Morelli and **T. M. Tritt**
Annual Review of Materials Science, **29**, 89-116, 1999

- * 2000 *Semimetals and Semiconductors* (Volume **69**)
Overview of Measurement and Characterization Techniques for Thermoelectric Materials: pp. 25-50
Terry M. Tritt and Valerie M. Browning

- * 2000 *Semimetals and Semiconductors* (Volume **70**)
Thermoelectric Properties of Quasicrystals
pp. 77 - 116
Terry M. Tritt, A. L. Pope and Joseph W. Kolis

- * 2000 *Semimetals and Semiconductors* (Volume **70**)
Thermoelectric Properties of Transition Metal Pentatellurides
pp. 179-206
Terry M. Tritt and Roy T. Littleton, IV **Conference Executive Participation:**

- * Proceedings of 2000 Materials Research Society **Volume 626**
New Materials for Small Scale Thermoelectric Refrigeration and Power Generation Applications.
Edited by **Terry M. Tritt**, G. Nolas, M. Kanatzidis, G. Mahan and D. Mandrus

Conference Organizer
Conference Budget Officer

- * 1999-2002 International Thermoelectrics Society: **T. M. Tritt**, Executive Board

INVITED PRESENTATIONS (1997 –2001 related to project or TE's):

- *Measurement and Characterization of Thermoelectric Materials.*
Terry M. Tritt,
1997 Materials Research Society, Spring March 31-April 4th (San Francisco)
Symposium Q: Thermoelectric Materials, New Directions and Approaches
p 290, 1997 Spring MRS Meeting Abstracts, April 1997
- *Thermoelectric Properties of the Pentatelluride Materials*
 $Hf_{1-x}M_xTe_5$ ($M = Zr, Ti, Ta$ and Nb).
Terry M. Tritt, DARPA Review on Thermoelectric Materials Program
August 11& 12, 1997, Pasadena CA
- *Low Temperature Thermoelectric Materials for Small Scale Refrigeration Applications.*
Terry M. Tritt, October 23, 1998
ICSICT-98 (Solid State and Integrated Circuit Technology) Beijing China
- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, Senate Liason to Armed Forces Committee
Clemson University, Clemson SC, December 16, 1997
- *Thermoelectric Materials for Automotive Applications*
Terry M. Tritt,
Automotive Resarch Opportunities at Clemson University
BMW Vice President George West
Clemson University, Clemson SC, December, 1997
- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, Davidson College Evening Seminar Series
Davidson NC, February 17, 1998
- *Thermoelectric Properties of the Pentatelluride Materials*
 $Hf_{1-x}M_xTe_5$ ($M = Zr, Ti, Ta$ and Nb).
Terry M. Tritt, Beijing (had to be withdrawn due to travel constraints)
International Conference on Integrated Electronics (ICSICT '98)
- *Low Temperature Thermoelectric Materials for Small Scale Refrigeration Applications.*
Terry M. Tritt, October 23, 1998
ICSICT-98 (Solid State and Integrated Circuit Technology) Beijing China
Had to be withdrawn due to travel constraints

- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, Ames National Lab and Iowa State University
Ames Iowa, September 11, 1998

- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, Michigan State University,
Condensed Matter Physics Seminar
East Lansing Michigan, September 14, 1998

- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, University of Michigan,
Condensed Matter Physics Seminar
Ann Arbor Michigan, September 15, 1998

- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, University of Virginia,
Condensed Matter Physics Seminar
Charlottesville VA, October 23, 1998

- *Thermoelectric Materials Run Hot and Cold*
Terry M. Tritt, University of Virginia,
Society of Physics Students Seminar
Charlottesville VA, October 23, 1998

- *Transport Properties Of Group IV Metal Tellurides: Possible Low Temperature Thermoelectrics.*
Joseph W. Kolis*, Christopher Feger.,**Terry M. Tritt**, Roy Littleton IV,
1998 Fall MRS Meeting, Symposium Z, *New Materials for Small Scale Thermoelectric Refrigeration and Power Generation Applications.*
editors: T. M. Tritt, M. Kanazidis, G. Mahan and H. B. Lyons, Jr.

- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, Oak Ridge National Laboratory,
Condensed Matter Physics Seminar
Oak Ridge , Tenn., January 15, 1999

- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, University of South Carolina,
Condensed Matter Physics Seminar
Columbia, SC, January 21, 1999
- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, University of Idaho,
Condensed Matter Physics Seminar
Moscow ID, Feb. 2, 1999
- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, University of Oregon,
Condensed Matter Physics Seminar
Eugene, OR, February 5, 1999
- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt, Allied Signal,
Condensed Matter Physics Seminar
Morristown, NJ, February 17, 1999
- *AOP Regional Science Fair Awards Ceremony: Keynote Address*
Terry M. Tritt, CoES,
Clemson University, March 24, 1999
- *Research and Investigations of New and Novel Materials for Thermoelectric Applications.*
Terry M. Tritt, CoES College Advisory Board,
Clemson University, Clemson SC, April 23, 1999
- *Research and Investigations of New and Novel Materials for Automotive Applications.*
Terry M. Tritt, BMW Corporation,
Clemson University, May 5, 1999
- *DARPA DSRC Special Panel on Potential Applications of Nanostructured Materials Thermoelectric Materials.*
Terry M. Tritt,
Washington D.C. May 19, 1999
- *Investigation of Novel and Advanced Materials*
Terry M. Tritt,
1999 Summer Science Research Institute
Clemson University, Clemson SC, July 20, 1999
- *Investigation of Novel and Advanced Materials*
Terry M. Tritt,
Clemson University Research Foundation
Clemson University, Clemson SC, September 10, 1999

- *Thermoelectric Materials for Small Scale Refrigeration and Power Generation Applications.*
Terry M. Tritt,
Condensed Matter Physics Seminar
Auburn University, Auburn, AL, October 22, 1999
- *The Next Generation Thermoelectric Materials,*
Terry M. Tritt,
(Special Invited Session on Thermoelectric Materials)
Bull. Am. Phys. Soc., **Vol 44**, , 26, SESAPS-99,
Chapel Hill, NC Nov 7-9, 1999
- *Introduction to Thermoelectric Materials*
Terry M. Tritt,
Special Dept. of Energy Workshop on
Future Directions in Thermoelectric Materials Research
Oak Ridge National Laboratory,
Oak Ridge, Tenn., January 10 & 11, 2000
- *The Next Generation Thermoelectric Materials Under Investigation at Clemson University*
Terry M. Tritt,
Special Dept. of Energy Workshop on
Future Directions in Thermoelectric Materials Research
Oak Ridge National Laboratory,
Oak Ridge, Tenn., January 10 & 11, 2000
- *Electrical and Thermal Transport Rapid Measurement Equipment Using a Cryocooler Based Cooling System.*
Terry M. Tritt and **Roy T. Littleton, IV**,
Quantum Design
San Diego, CA, January 14, 2000
- *Strategies for the Investigation of New Bulk Materials for Thermoelectric Applications*
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Terry M. Tritt
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editors: G. S. Nolas, D. Mandrus and D. Johnson
MRS Fall 2001 Symposium G: (# **G1-1**).

- *Raman scattering in Sb doped Transition Metal pentatellurides $ZrTe_{5-x}Sb_x$*
K. McGuire, Nathan D. Lowhorn, T. M. Tritt and A. M. Rao
2001 Fall MRS Meeting, Symposium G, Thermoelectric Materials,
editors: G. S. Nolas, D. Mandrus and D. Johnson
MRS Fall 2001 Symposium G: (# **G5-3**)

- *Transformation of Multiwalled Carbon Nanotube into Strings of Carbon Nanoshells*
B. Sadanadan, J. Gaillard, T. Savage, S. Bhattacharya, **T. M. Tritt**,
and A. M. Rao et. al.
2001 Fall MRS Meeting, Symposium Z, *Making Functional Materials with Nanotubes.*
editors: P. Bernier, P. A. Ajayan, Y. Iwasa and P. Nikolaev
MRS Fall 2001 Symposium Z: (# Z6-18)

- *Effects of various Grain structure and Sizes on the Thermal Conductivity of Ti-based Half-Heusler alloys*
S. Bhattacharya, **Terry M. Tritt**, Y. Xia, V. Ponnambalam, S.J. Poon
and N. Thadhani
Ga. Tech. Conference on Nanomaterials and Nanotechnology, (Sept. 2001)

Enclosure 2

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