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Discovery of New Superconductors

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Final Report

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In this project we used a novel strategy for an enlightened search for new superconducting materials. We used a broad range of synthesis methods together with highly sensitive and selective screening techniques. Materials synthesis was accomplished by natural, extraterrestrial, high pressure/temperature, phase spread alloys and conventional methods. The screening was mostly done using Magnetic Field Microwave Spectroscopy (MFMMS). Use of this technique allowed us to quickly discard large amounts of non-superconducting materials and thus focusing the effort on the interesting materials. This provided a proven, rational methodology for the search for new superconductors.

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FINAL REPORT

Introduction

In this project we used a novel strategy for an enlightened search for new superconducting materials. We used a broad range of synthesis methods together with highly sensitive and selective screening techniques. Materials synthesis was accomplished by natural, extraterrestrial, high pressure/temperature, phase spread alloys and conventional methods. The main reason to search in extraterrestrial and/or natural systems is because these are produced under extreme conditions (temperature, pressure, cooling rate, radiation), which are not available in the lab. The screening was mostly done using Magnetic Field Microwave Spectroscopy (MFMMS). Use of this technique allowed us to quickly discard large amounts of non-superconducting materials and thus focusing the effort on the interesting materials. This provided a proven, rational methodology for the search for new superconductors. Once an interesting signal was discovered, materials were subjected to a barrage of tests including energy dispersive X-ray spectroscopy (EDX), X-ray diffractometry (XRD), wet chemical analysis, magnetotransport, magnetic measurements and numerical analyses based on comparisons to databases of known superconductors.

Over the course of the award, we have discovered novel superconductivity, unusual properties in superconducting materials and superconductivity in extraterrestrial materials, among other findings. We have published many papers, given invited and contributed talks at APS march meeting and other conferences and three PhD students have successfully defended their thesis based on research supported by this grant. The research performed at UCSD and in collaboration with others, included a broad search in natural systems, samples synthesized at UCSD and samples synthesized by collaborators (even pursuing fleeting claims of superconductive materials). Our goal was to not leave any stone unturned.

Methodology

We have developed the instrumentation and proven that the methodology works in all know classes of superconducting compounds including: elements, A15s, MgB_2, cuprates, pnictides, iron arsenides iron chalcogenides. We have tested this methodology on artificially synthesized and naturally occurring minerals, micrometeorites and meteorites, nanostructures and thin films. The system we developed is the most sensitive and selective method for detection of superconductivity which can detect 10^{-12} cc of a superconductor embedded in an otherwise non-superconducting matrix. We have started research in the application of this methodology to other phenomena such as magnetism. Combined with a variety of combinatorial and divide and conquer methods this provides a unique rational search for new superconductors. We have proven that many new superconductors were discovered listed on a web site described below.

Accomplishments

Systems investigated

During the course of the research related to this grant, we have investigated many different material systems. This includes both natural as well as artificially synthesized systems. The purpose of this research was to investigate as many systems as possible and as well as to check claims that appear in the literature. We believe that our methodology has become the standard used to validate claims and theoretical suggestions.

The systems we have investigated include meteorites, naturally occurring minerals, lunar and Martian rocks and artificially synthesized thin films and bulk materials. Once an interesting signal was uncovered, we proceeded to identify the unknown compound using a full battery of tests. Below we will describe a few typical highlights from our research. Our extensive searches are described in the web site we created and described below.

As an example we have broadened our extensive searches in micrometeorites to include the Tlacotepec, Mundrabilla and GRA 95205 meteorites. A “divide and conquer” methodology allowed identification of the exact chemical composition of the material giving this signal. In our latest study of meteorites, we were able to isolate the meteorite grains containing the strongest superconducting signal. Then, by measuring with EDX and comparing to the Superconducting Materials Database (SuperCon), we were able to discover superconductive alloys of lead, indium and tin inside Mundrabilla and GRA 95205. This discovery of superconductive extraterrestrial material could have important implications for planetary science and astronomy. This manuscript is in press in the Proceedings of the National Academy of Sciences of the United States of America (PNAS). In previous studies on meteorites published during the course of this grant, we discovered an unusual electromagnetic signal in the Tlacotepec meteorite at 117 K and used MFMMS to analyze micrometeorites after segregating them based on their physical morphology.

We have investigated a large number of different artificially synthesized systems. These studies were motivated by a variety of reasons, such as claims by other groups or because of structural similarities with existing superconductors. The large number of systems that have been investigated are summarized in Table 1 below. In order to maximize success, in some cases synthesis was performed, not only at UCSD, but also at collaborators laboratories with unique facilities (high pressure/temperature, single crystal growth).

These studies have been underway since last year and are in various stages of research and/or publication.

For instance in a recent project, we investigated lithiated iron selenide. This is an unusual Fe containing compound, which may potentially have a very high temperature transition temperature. In addition there were various reports of coexistent magnetism and superconductivity. In our report published in Physical Review B, we showed that MFMMS analysis revealed multiple superconducting transitions, in addition to the coexistent ferromagnetism. Further experiments on the intercalation of electron donating molecules in these materials show an increase in the spin coupling between the layers, resulting in an antiferromagnetic order at 50 K. These results are being prepared for a second publication.

Service

We have and continue to perform measurements as a service for a number of groups in the US and elsewhere which claimed unusual signals in a variety of compounds including graphene, CuCl, the oxychloride family and lead-doped palladium bismuth.

We have also created a new web-based database, the Superconducting Research Database, that researchers can post compounds they have investigated for superconductivity. This searchable database allows for reporting of various characteristics of material systems including: chemical formula, crystal structure, and any transitions found along with their corresponding temperatures. Researchers are also encouraged to post null results, which often do not meet the threshold for full publications, as well as comments and citations that could be useful to understand a posted system. The ultimate goal is to provide new avenues of communication and collaboration between researchers to further the goal of discovering new phases of superconductivity. At the end of this document, we have provided a list of systems in the database as well as a brief tutorial on its use.

Publication List:

1. *Magnetic field modulated microwave spectroscopy across phase transitions and the search for new superconductors.* J. G Ramírez, Ali C. Basaran, J. de la Venta, Juan Pereiro and Ivan K. Schuller, Rep. Prog. Phys. **77**, 093902 (2014).
2. *Dynamic conductivity scaling in photoexcited V_2O_3 thin films,* Elsa Abreu, Siming Wang, Juan Gabriel Ramírez, Mengkun Liu, Jingdi Zhang, Kun Geng, Ivan K. Schuller, and Richard D. Averitt, Phys. Rev. B **92**, 085130 (2015)
3. *Search for Superconductivity in micrometeorites*, S. Guenon, J.G. Ramirez, Ali C. Basaran, J. Wampler, M. Thiemens, S. Taylor, and Ivan K. Schuller, Scientific Reports, **4**, 7333 (2015).

4. *Effect of Increasing Disorder on Superconductivity of Mo/Nb Superlattices*, Juan Pereiro, Thomas Saerbeck and Ivan K. Schuller, Supercond. Sci. Technol. **28**, 085001 (2015).
5. *Enhancements of pinning by superconducting nanoarrays*, E. Navarro, C. Monton, J. Pereiro, Ali C. Basaran and Ivan K. Schuller, Phys. Rev. B., **92**, 144512 (2015).
6. *Search for New Superconductors: An Electro-Magnetic Phase Transition in an Iron Meteorite Inclusion at 117K*, S. Guenon, J.G. Ramirez, Ali C. Basaran, J. Wampler, M. Thiemens, and Ivan K. Schuller, J. Supercond. Nov. Magn., **29** (2016).
7. *Structure, magnetization, specific heat, and microwave properties of* $K_xFe_2\text{-}ySe_2$, D. Yazici, Ali C. Basaran, J.G. Ramirez, Ivan K. Schuller, M.B. Maple, Supercond. Sci. Technol. **29**, 085015 (2016).
8. *Coexistence of multiphase superconductivity and ferromagnetism in lithiated iron selenide hydroxide* $[(Li_{1-x}Fe_x)OH]FeSe$, Christian Urban, Ilya Valmianski, Ursula Pachmayr, Ali C. Basaran, Dirk Johrendt, Ivan K. Schuller, Phys. Rev. B **97**, 024516 (2018).
9. *Effects of Oxygen Annealing on Single Crystal Iron Telluride*, Nathaniel Smith, David Gelting, Ali C. Basaran, Marvin Schofield, Ivan K. Schuller, Marija Gajdardziska-Josifovska, Prasenjit Guptasarma, Physica C **567** (2018).
10. *Search Faster, Not Smarter: Using Microwaves to Search for New Superconductors*, J. Wampler, A. Hojem, Ivan K. Schuller, Proc. Int. Symp. "Superconductivity & Pressure: A Fruitful Relationship on the Road to Room Temperature Superconductivity." CERA, Ed., Madrid, Spain (2019)
11. *Superconductivity Found in Meteorites*, J. Wampler, M. Thiemens, S. Cheng, Y. Zhu, Ivan K. Schuller, Proc. Nat. Acad. Sci. (In Press)

Theses

1. S. Wang, *Inhomogeneous Phase Transition of Vanadium Oxide on Mesoscopic Scale*, (2014)
2. I. Valmianski, *Vanadium oxide phase transitions: fast, small, and strained*, (2017)
3. J. Wampler, *The Search for Novel Superconductivity in Inhomogeneous Materials using Magnetic Field Modulated Microwave Spectroscopy* (2019)

Personnel

This project was an ideal interdisciplinary project where materials scientists, physicists, chemists, and astronomers. Because of this a large number of researchers at different levels were involved in this. This includes: 2 undergraduates, 4 graduate students, and 5 postdoctoral fellows. 2 postdoctoral fellows are still at UCSD and have moved into other related research areas. The undergraduates are currently pursuing doctoral work in other universities and the graduate students and postdocs have moved into academic institutions or the private industry.

Invited Talks at Conferences:

1. *35 Years of Metallic Superlattices: Hybrids,* Keynote Speaker, Advanced Materials

and Nanotechnology AMN7, February 9, 2015.

2. *Physics at the Nanoscale*, Energy Symposium, XXIV International Materials Research Congress, Cancun, Mexico, August 18, 2015.
3. *Magnetic Field Modulated Microwave Spectroscopy (MFMMS) across Phase Transitions and the Search for New Superconductors*, 11th Conference on Materials and Mechanisms of Superconductivity, Geneva, Switzerland, August 23-28, 2015.
4. *An elightened method to search for new superconductors,* M2S-2018, 12th International Conference on Materials and Mechanisms of Superconductivity & High Temperature Superconductors, August 19-24, Beijing, China
5. *An enlightened method to search for new superconductors,* International Symposium "Superconductivity & Pressure: A Fruitful Relationship on the Road to Room Temperature Superconductivity." May 21-22, Madrid, Spain (2019)

Invited Talks/ Research Organizations:

1. *35 Years of Metallic Superlattices: Oxide/Magnetic*, Ivan K. Schuller, Center for Nanoscience and Nanotechnology CEDENNA, January 5, 2015.
2. *35 Years of Metallic Superlattices: Benefits and Threats to Industry*, Ivan K. Schuller, Pontificia Universidad Catolica: Center for Innovation, January 6, 2015.
3. *35 Years of Metallic Superlattices: Hybrids*, Universidad de Chile, Faculty of Science, January 7, 2015.
4. *Hybrids*, RWTH, Julich, Germany, January 15, 2015.
5. *35 Years of Metallic Heterostructures,* Peter Grunberg Institute (PGI), Julich, January 16, 2015.
6. *35 years of Metallic Heterostructures,* Auckland University, February 18, 2015.
7. *Why Physics: Beyond Atom Bombs and Big Bangs*, New York Univ, New York, NY, May 7 2015
8. *35 Years of Metallic Superlattices*, Univ Minnesota, Twin Cities, MN, June 14, 2015.
9. *35 Years of Metallic Superlattices*, Chalmers Univ of Technology, Gothenburg, Sweden, September 16, 2015.
10. *35 Years of Metallic Superlattices*, Tsing Hua University, Taipei, Taiwan, October 12, 2015.
11. *Joint Colloquium among Five Institutions of NTU and Academia Sinica*, Taipei University, Taipei, Taiwan, October 13, 2015.
12. *35 Years of Metallic Superlattices*, Universidad del Pais Vasco, Bilbao, Barcelona, Spain, November 6, 2015.
13. *35 Years of Metallic Superlattices*, Universidad Autonoma de Madrid, IEEE Spanish Chapter, Barcelona, Spain, November 9, 2015.

14. *35 Years of Metallic Superlattices*, University of Barcelona, Barcelona, Spain, November 11, 2015.
15. *35 Years of Metallic Superlattices*, Pontificia Universidad Catolica, Santiago, Chile, November 26, 2015.
16. *Why Physics: Beyond Atom Bombs and Big Bangs*, Univ of Texas, San Antonio, TX, April 29, 2016.

Contributed Talks at Conferences:

1. *Ferromagnetism in partially oxidized CuCl*, T. Saerbeck, J. Pereiro, J. Wampler, J. Stanley, J. Wingert, O.G. Shpyrko, and Ivan K. Schuller, International Conference on Magnetism, Barcelona, Spain, July 5-10, 2015.
2. *Search for New Superconductors in Extraterrestrial Materials Using Magnetic Field Modulated Microwave Spectroscopy*, Stefan Guenon, G. Ramirez, A.C. Basaran, J. Wampler, S. Taylor, M. Thiemens, and Ivan K. Schuller, 4th International Symposium on Energy Challenges & Mechanics – working on small scales, Aberdeen, Scotland, UK, August 11-13, 2015.
3. *Superconducting Properties of Ferromagnetic Lithiated Iron Selenide Hydroxide*, C. Urban, A. Basaran, U. Pachmayr, D. Johrendt, Ivan K. Schuller, MMM/Intermag 2016, San Diego, CA, January 11-15, 2016.
4. *Search for Superconductivity in Extraterrestrial Materials: An Electro-Magnetic Phase Transition with Spin-Glass Characteristics*, J.G. Ramirez, Ali C. Basaran, J. Wampler, S. Taylor, M. Thiemens, Ivan K. Schuller, APS March Meeting, Baltimore, MD, March 14-18, 2016.
5. *Characterizing phase transitions in known materials with Magnetic Field Modulated Microwave Spectroscopy (MFMMS)*, J. Wampler, J.G. Ramirez, A. Basaran, I.K. Schuller, APS March Meeting, Baltimore, MD, March 14-18, 2016.
6. *Ultrasensitive detection of superconducting transitions within doped Sr2IrO4*, J. Wampler, J. Trastoy, G. Cao, I.K. Schuller, APS March Meeting, New Orleans, LA, March 13-17, 2017.
7. *Transport and magnetization effects in strain coupled VO2/FeRh heterostructures*, S. Bennett, C. Urban, J. Trastoy, I. Valmianski, I.K. Schuller, APS March Meeting, New Orleans, LA, March 13-17, 2017.
8. *Pressure tuning of coercivity states in Ni-V2O3 magnetic heterostructures*, C. Urban, I.K. Schuller, APS March Meeting, New Orleans, LA, March 13-17, 2017.
9. *Natural Superconductivity Observed in Meteorites Above 5K*, J. Wampler, M. Thiemens, I.K. Schuller, APS March Meeting, Los Angeles, CA, March 5-9, 2018.
10. *Modulating Metal-Insulator Transitions in VO_X by Tuning Oxygen Stoichiometry*, M. Lee, Y. Kalcheim, J. del Valle, I.K. Schuller, American Physical Society March Meeting, Boston, MA, March 4-8, 2019.
11. *New magnetic phases and the absence of superconductivity in doped Hematophanite, $Pb_4Fe_3O_8Cl$*, A. Hojem, J. Wampler, I.K. Schuller, APS March Meeting, Boston, MA, March 4-8, 2019.

12. *Detection of Spin Canting Using Magnetic Field Modulated Microwave Spectroscopy*, A. Hojem, J. Wampler, I.K. Schuller, APS March Meeting, Denver, CO, March 2-6, 2020 (Cancelled)

Other Talks

1. *The Search for Novel Superconductivity in Inhomogeneous Materials using Magnetic Field Modulated Microwave Spectroscopy,* J. Wampler, Thesis Defense: University of California San Diego, San Diego, August 29, 2019

Changes in research objectives, if any: None

Change in AFOSR program manager, if any: None

Extensions granted or milestones slipped, if any: None

Include any new discoveries, inventions, or patent disclosures during this reporting period (if none, report none): None

Search projects

Material System		Motivation	Status	Synthesis
Doped $BaFe_2S_3$ (Sr, Ca)		* Superconducting under pressure. * Stabilize at ambient pressure via doping and quenching samples made under pressure.	*Synthesis high and ambient pressure * New transition discovered, but no superconductivity	*Spain-Alario-Franco *UCSD
Doped Sr_2IrO_4	Polycrystalline	* Band structure similar to cuprates * Induce superconductivity via chemical doping.	* Superconductor found in Pt-Doped samples, likely known material ($SrIr_2$). * Paper in preparation	*UCSD
	Single Crystal		* Finished	*G. Cao-University of Colorado
Lead Oxychloride Doped: • $Pb_7MoO_9Cl_2$ • $Pb_4Fe_3O_8Cl$ • Potentially others		* Distorted perovskite, similar to some cuprates. *Some Pb perovskites superconduct at low T.	* Magnetic phases discovered in gallium and cobalt doped hematophanite (presented at APS march meeting 2019)	*UCSD
Meteorites (Tlacotepec, Mundrabilla, GRA 95205)		* Iron chalcogenides superconduct * Inclusions include FeS, Pb compounds. * Unusual synthesis conditions (temperature, pressure, cooling rate) * Measured a range of meteorites (15 meteorites as well as a collection of micrometeorites)	* Unusual electromagnetic phase transition in Tlacotepec at 117K * Superconductivity discovered in Mundrabilla, GRA 95205. $T_c \sim 5\text{-}6$ K * 2 papers published * 1 more in print	* Mark Thiemens - UCSD
Minerals		* Similar motivation as meteorites, though their synthesis conditions are less extreme.	* No superconductivity discovered	* Johnpierre Paglione - UMD
Copper Chlorides		* Occasional reports of CuCl and CuCl + Si superconductivity * Magnetism in partially oxidized CuCl	* CuCl+Si does not superconduct. *Novel magnetic phase published.	*Spain-Alario-Franco * UCSD
Lithiated Iron Selenides Hydroxide		Conflicting reports of coexistence of magnetism and superconductivity.	*Paper Published *Subsequent paper on intercalated samples in preparation	Germany-D. Johrendt

Iron Flouride	Benchmark sample to show presence or non-presence of AFM transition in MFMMS	* No transition detected	*UCSD
Iron Selenide (telluride)	Oxygen doping and oxidation state in single crystals	*Paper Published	P. Guptasarma, U. Wisconsin
Palladium Bismuthate	Possible topological phases	*Measurements underway	Bing Lv- UT Dallas
Superconductor-VOx host, hybrids	Enhance Tc using structural phase transition from the host	* Synthesis underway	*UCSD
MXY (M=Fe,Pb,Y - X=O,F - Y=Cl,F), MNX (M=Zr,Hf – X=Cl,Br,I)	Compounds SC at low T and might superconductivity at high T ?	* Synthesis underway	*UCSD
Palladium Hydride, Deuteride	Claims of SC at 50-60K	* Collaboration underway	Australia-E. Gray
$K_2Cr_3As_3$	SC+magnetism	* Underway	ORNL-K. Taddei
YBCO/PLCMO Heterostructures	Field induced SC	* Superconductivity observed but suppressed for thinner YBCO layers.	Switzerland-C. Bernhard
Rare Earth Hydride	Recent experiments show	* Measured CeLiH, LaLiH, NdLiH, SmLiH and CeNaH, LaNaH, NdNaH, SmNaH * No superconductivity discovered	*Spain-Alario-Franco
FeSi Single Crystals	Observe insulator to metal transition in MFMMS	* superconducting-like peak observed	*UCSD-M. B. Maple
Website Database	Creating tools to compare and understand existing data	* Available at srd.physics.ucsd.edu	*UCSD
Computer algorithm	Correlations study underway	* Expecting info	V. Stanev-U. Maryland

Superconducting Research Database – Tutorial

The Superconducting Research Database (SRD) is an open database meant to allow researchers to share information on materials they have tested for superconducting phases. The database can store and display information on crystal structure, through photo or crystal structure file (.cif), as well any transition, either superconducting or nonsuperconducting, that a researcher has found in a particular material system.

To contribute to the database a user first creates a user profile with their name, institution and contact email. Once approved the user is allowed to add entries and add information to already created entries.

To create a new entry users are prompted to enter a material system name, subtitle, crystal structure type, any transitions found along with temperatures of transitions and chemical formula when applicable. They are also encouraged to include an image or .cif files to better visualize the system they are working with.

Once an entry is created users are able to add citations for their publications where the system was investigated. Users are also encouraged to add brief comments to further expand on any information they feel important to share. As the SRD is meant to foster new avenues of collaboration all entries, comments, and publications are associated with a user that can be contacted via the user provided email address.

Superconducting Research Database – Table of materials

The following is a list of materials investigated and included into the superconducting research database by the Schuller lab:

Project	Material	Dopants
Minerals	Akinite	
	Aramayoite	
	Berthierite	
	Bismuthnite	
	Bronite	
	Colusite	
	Cubanite	
	Maucherite	
	Pyrrhotite	
	Psuedobrookite	
	Stephanite	
	Wittichenite	
	Marcasite	
Meteorites	Abee	
	ALHA77216.85	
	LAP 91900	
	PCA 82502	
	EET 87511	
	North County Oldhamite	
	PCA 91241	
	ALHA 81021	
	EET 83213	
	Santa Clara	
	Allende	
	LEW 85311	
	Murchison	
	Mundrabilla	
	GRA 95205	
Lead Oxychlorides	Hematophanite($Pb_4Fe_3O_8Cl$)	Ga, Bi, Mn, Co
	Parkinsonite ($Pb_7MoO_9Cl_2$)	Fe, La, Ta, V, Mn, V, Ga, Bi
	Asisite($Pb_7SiO_9Cl_2$):	Al, Ga, Bi
Yttrium Barium Strontium Copper Oxide	$YBaSrCu_3O_7$	
$K_2Cr_3As_3$	$K_2Cr_3As_3$	

Project	**Material**	**Dopants**
Rare earth hydrides	CeLiH	
	LaLiH	
	NdLiH	
	SmLiH	
	CeNaH	
	LaNaH	
	NdNaH	
	SmNaH	
MFe_2S_3 (M=Ba, Ca, Sr)	$BaFe_2S_3$	Ca, Sr
AMN	$BaZrN_2$	
	$SrZrN_2$	
	$SrHfN_2$	
	$CaNbN_2$	
	$SrTiN_2$	
	$CaTaN_2$	
	$BaHfN_2$	
	$NaNbN_2$	
Barium Iron Arsenide	BaFeAs	Ag, Ti, Th, Sc, Zr, Ni
Strontium Irridate	*Sr2IrO4*	Pt,Os,Rh,La,Rb
	Lithiated Iron Selenide Hydroxide: Li(OH)FeSe	
Palladium Hydride, Deuteride	PdH_x/PdD_x	
YBCO/PLCMO Heterostructures	YBCO	
	$Pr_{0.5}La_{0.2}Ca_{0.3}MnO_3$	
Miscellaneous	AlB_2	
	Granular Al	
	BaCoSO	Fe
	$BiF_{0.5}S_2(La,Ce)O_{0.5}$	
	Bismuth strontium calcium copper oxide (BSCCO)	
	Q-carbon	B
	diamond	B
	$CaMn_2Al_{10}$	
	Eu_5Si_3	
	FeF_2	
	Fe_2O_3	
	Fe_3O_4	
	FeS film and powder	
	FeSi	
	$LaFe_2Si_2$	

Project	**Material**	**Dopants**
Miscellaneous(cont.)	MgB_2	
	MnP	
	Ni film	
	Nb_3Sn	
	$Nd_{2-x}Ce_xCuO_4$	
	$PdBi_2$	Pb
	$RuSr_2GdCu_2O_8$	
	$RuSr_2Gd_{0.9}Ce_{0.1}Cu_2O_8$	
	SmB_6	
	V_3Si	