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Search for Novel Superconductors, Thermoelectrics, and Super-Thermal-Conductors with Enhanced Performance

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14. ABSTRACT
To meet the needs of the Department of Defense for national defense and security, and at the same time to promote the health and wealth of our national economy, we have proposed, during this grant period, to search for novel materials in three areas: A) superconductors with higher Tc, higher Jc, and reduced anisotropy; B) thermoelectric materials with high figures of merit and power factors; and C) super-thermal-conductors or solids with giant thermal conductivity. During this grant period, we have: 1) achieved by thermal annealing and confirmed by microstructure study, for the first time, superconductivity up to 25 K induced through interphases between two non-superconducting allotropes of CaFe2As2, demonstrating directly a new path to high Tc via interfacial effect as predicted almost five decades ago; 2) brought down by high pressures the Tc-limits imposed by the so-called universal inverse quadratic Tc-carrier rule and brought the Tc to new heights without any sign of saturation up to ~60 GPa for the layered high Tc cuprate superconductors, e.g. Bi2Sr2Can-1CunO2n+4 and HgBa2Can-1CunO2n+2+x, demonstrating that the current record high Tcs of stable cuprate superconductors can be further enhanced and that a new avenue to higher Tc is possible; 3) induced, by pressure, superconductivity in a Mn-compound, MnSe, up to 9 K at 35 GPa, demonstrating that Mn-containing compounds are not absolutely detrimental to superconductivity and opening up a new testing ground for possible high Tc via optimization of multiple interactions in the multiferroic materials, which often contain Mn-ions; 4) discovered and synthesized > 10 new superconducting compounds with interesting electronic structures although with low Tcs < 8 K, some of which were through novel synthesis routes that may be deployed to high Tc superconductor preparations; 5) retained at ambient the high-pressure-induced metastable superconducting phases in Sb-, Bi-, and Fe-based compounds by low-temperature pressure quenching with profound implications for applications of the recently reported roomtemperature superconductivity in hydrides under ultrahigh pressure by removing the ultrahigh pressure constraint; 6) stabilized the skyrmion phase in Cu2OSeO3, a possible candidate for high density memory devices, at room temperature by enlarging the temperature window; 7) grown the first large BAs super thermal conductor single crystals as predicted for meaningful physical characterization with thermal conductivity approaching that of diamond and with immense implications for nano-electronic devices; 8) discovered several thermoelectric compounds, both p-type and n-type, with high figures of merit and power factors; and 9) refuted experimentally numerous high-profile claims of high Tc worldwide, such as those of 260 K in P-doped graphite and graphene (Florida), 52-61 K in Pd-H (Australia), > 60 K in CuCl (Brazil), and 230 K in Au-Ag (India), thus saving effort and time for researchers in the field.

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Accomplishments

- Systematic investigation of $CaFe_2As_2$ (Ca122 system)
    - Discovered the highest T_c for undoped $CaFe_2As_2$ (Ca122) by low temperature annealing at 350 °C for different times (t) over a narrow t-region. The T_c is almost constant at 25 K, which is very different from that reported previously, and insensitive to the annealing time. The superconducting volume fraction χ/χ (t) scales with the interface density D, both peaking at t ~ 16 hr. Experimental XRD results can be simulated by random P1/P2 layer stacking of no more than three layers, each using the intergrowth model of layer materials, where P1 and P2 are different phases of Ca122 and are not superconducting by themselves. The calculated interface density scaled well with the superconducting volume fraction of the sample. These results represent the best experimental evidence to date for interface-enhanced superconductivity, opening up a new avenue for higher T_c.
    - P1 was achieved by rapid cooling from 850 °C to 300 K and P2 by prolonged annealing of P1 at 350 °C. They are reversibly interchangeable by controlled annealing for different periods of time at 350 °C. Both P1 and P2 phases possess tetragonal symmetry but with a slight difference ~ 1% in their c-axes, *i.e.*, c = 11.623 A and 11.738 A for P1 and P2, respectively. Despite these similarities, they exhibit drastically different behavior on cooling: P1 undergoes a tetragonal-collapsed tetragonal phase transition at ~ 100 K, whereas P2 undergoes a tetragonal-orthorhombic phase transition at ~ 150 K. This allows us to investigate the interface effect on the induction and enhancement of superconductivity proposed by us previously in Ca122 and related compounds.
    - Detected nano-scale microstructures in superconducting samples of Ca122 by low-temperature XRD from 300 K down to 10 K and, in collaboration with Y. M. Zhu's group at BNL, by TEM investigation at both 300 K and 90 K, providing further support for our conjecture of interfacial induction of high temperature superconductivity in undoped non-superconducting Ca122 single crystals.
    - Investigated surface terminations of 122-type alkaline earth metal iron pnictides $AEFe_2As_2$ (AE = Ca, Ba) with STM/STS. Layer-resolved spectroscopy on these terminating surfaces reveals a well-defined superconducting gap on the As terminations. In contrast, the gap features become weaker and absent on *AE* and Fe terminations, respectively. Studies of the STM tip pressure effect on single-crystal $CaFe_2As_2$ were also carried out (in collaboration with S. H. Pan at TCSUH).
    - Induced interfacial superconductivity in the non-superconducting parent compounds $AEFe_2As_2$ where AE = Ca, Sr, or Ba by simple subsequent annealing of the as-grown parent compounds with As, P, or Sb. Our results indicate that the superconductivity originates from electron transfer at the interface of the hybrid van der Waals heterostructures, consistent with the two-dimensional (2D) superconducting transition observed.

- Completed the design and construction of the state-of-art integrated MBE system with *in situ* STM, ARPES, and magnetic and transport measurement capabilities to study interface effects on superconductivity, thermoelectricity, and magnetism.
- Successfully developed a magnetically transparent ultrahigh pressure diamond anvil cell that can be used in Quantum Design's MPMS 3 to detect signals from sample masses down to nanograms in superconductors or magnets against a large magnetic background, which originates from the cell. This new cell will allow us to explore the magnetic properties of new and existing systems under ultrahigh pressures and applied fields, which is of significance to defense research.
- A new diamond anvil cell high-pressure measurement system was activated, including a diamond anvil cell that can reach pressures up to 100 GPa and a microscope that provides magnification up to 600x and 61.5 mm working distance. The optical equipment needed for diamond anvil cell work was also completely upgraded and rebuilt. The spectrograph can collect not only high-resolution Raman but also fluorescence spectra based on a micro-Raman design.
- Systematically grew large single crystals of the layered compounds β-$PdBi_2$, including both the hole-doped $PdBi_{2-x}Pb_x$ and the electron-doped Na_xPdBi_2, and studied their magnetic and transport properties. The complete phase diagram was established from hole doping to electron doping, and a high-pressure study of the undoped $PdBi_2$ was also carried out. It is worth mentioning that others later reported the same β-$PdBi_2$ materials with quite unusual half-quantum flux and topological properties.
- Continued to study the origin of the unusual non-bulk superconductivity with a T_c up to 49 K in the rare-earth-doped $CaFe_2As_2$ system. The chemical stoichiometry, magnetization, resistivity, and specific heat of the as-synthesized and 500 °C annealed (Ca,R)122 single-crystalline samples have been measured and analyzed, providing further evidence for the interface-enhanced T_c mechanism in this system.
- Attempted to grow undoped Mg_2Sn crystals by first using different metal flux for the new thermoelectric compound system $Mg_2(Sn,Ge)$, which possesses both high figure of merit *ZT* and high power factor *PF*. A new compound with refined composition $Mg_{2.9}Sn_8$ and cubic unit cell structure was also discovered.
- Discovered a new Bi-based Zintl phase $(Ca,Yb,Eu)Mg_2Bi_2$ of high thermoelectric performance with the record figure of merit *ZT* as high as 1.3 at 873 K. This *ZT* value is the highest ever reported in $CaAl_2Si_2$-based structures, especially when compared to the best antimony (Sb)-based $YbZn_{0.4}Cd_{1.6}Sb_2$ compound (in collaboration with Z. F. Ren at TCSUH).
- Carried out detailed experimental studies on one particular material, BAs, that is predicted to have an exceptionally high room temperature thermal conductivity κ above 2000 $Wm^{-1}K^{-1}$. A relatively large $\kappa \sim 196\ W\ m^{-1}\ K^{-1}$ was obtained, but this is still one order of magnitude lower than that predicted and is attributed to the domain boundaries and As deficiency in the crystals. Further optimization of the material is underway to achieve its theoretical value by perfecting the crystals (in collaboration with Z. F. Ren at TCSUH).
- <u>Investigation of topological materials: $Bi_2Se_{2.1}Te_{0.9}$, Sb_2Te_2Se, Sb_2Se_2Te, Bi_2Te_3</u>
    - Studied quantum oscillations of metallic, hole-type $Bi_2Se_{2.1}Te_{0.9}$ in magnetic fields up to 35 Tesla (at NHMFL) and found that Shubnikov-de Haas (SdH) oscillations from topological surface and bulk states could be separated and observed at low- and high-

field ranges, respectively. This unique property could arise from the cyclotron masses of bulk and surface carriers being vastly different.

- Studied the SdH oscillations in metallic $Bi_2Se_{2.1}Te_{0.9}$ in magnetic fields up to 35 Tesla. Angle-dependent measurements and calculations of the Berry phase showed two frequencies that describe oscillations from surface and bulk carriers, corresponding to the topological surface state at low field and the bulk state at high field, respectively.
- Magnetotransport measurements of the metallic, p-type Sb_2Te_2Se, a topological insulator, clearly show SdH oscillations in fields above B=15T. The value of the Berry phase β determined from a Landau level fan diagram is very close to 0.5, further suggesting that the oscillations result from topological surface states. Magnetoresistance (MR) of Sb_2Te_2Se is large and reaches a value of 1100% at B=31T without any sign of saturation. The large MR in Sb_2Se_2Te is not due to the presence of a linear Dirac dispersion as observed in many other topological systems, and it likely has a classical origin due to the mobility fluctuation and breaking of crystal structure symmetry. The large MR of Sb_2Se_2Te makes the compound suitable for utilization in electronic instruments such as computer hard discs, high field magnetic sensors, and memory devices.
- Investigated the weak antilocalization (WAL) effects in topological insulator Bi_2Te_3 single crystals at high and low bulk charge-carrier concentrations. At low charge-carrier density, the WAL curves scale with the normal component of the magnetic field, demonstrating the dominance of topological surface states in magnetoconductivity. At high charge-carrier density, the WAL curves scale with neither the applied field nor its normal component, implying a mixture of bulk and surface conduction. WAL due to topological surface states shows no dependence on the bulk charge carriers' nature (electrons or holes). The observations of an extremely large nonsaturating magnetoresistance and ultrahigh mobility in the samples with lower carrier density further support the presence of surface states. Such properties also have great technological value and can be exploited in magnetoelectric sensors and memory devices.
- Investigated the WAL effect in the p-type $Bi_2Se_{2.1}Te_{0.9}$ topological system. The WAL curves measured at different tilt angles merge together, showing that surface states dominate the magnetoconductance in the $Bi_2Se_{2.1}Te_{0.9}$. The sample also exhibits a large positive magnetoresistance that reaches 1900% under a magnetic field of 35 T at T = 0.33 K with no sign of saturation. The large magnetoresistance of topological insulators can be utilized in future technology such as sensors and memory devices.
- Studied the magnetotransport properties of a Sb_2Se_2Te single crystal where a maximum value of 1100% at B = 31 T was observed with no sign of saturation. MR shows SdH oscillations above B = 15 T. The frequency spectrum of SdH oscillations consists of three distinct peaks at $\alpha = 32$ T, $\beta = 80$ T, and $\gamma = 117$ T, indicating the presence of three Fermi surface pockets, whereas β is the prominent peak in the frequency spectrum. Further studies show that β-pocket has trivial topology.
- Investigated quantum oscillations and the associated 2D Fermi surface in a p-type metallic Bi_2Te_3 single crystal under magnetic fields up to 7 T. The presence of the 2D Fermi surface was confirmed by measuring the maxima/minima positions of oscillations. The Berry phase, $\beta = 0.4 \pm 0.05$ obtained from the Landau level fan plot, is

very close to the theoretical value of 0.5 for the Dirac particles, confirming the presence of topological surface states.

- Investigated pressure effects on the multiferroic state of $GdMn_2O_5$ and discovered that external pressure decouples the Mn-spin and Gd-moment systems, resulting in a second ferroelectric phase with higher transition temperature in this compound.
- Ultrafast optical spectroscopy was used to study carrier dynamics in slightly underdoped $(BaK)Fe_2As_2$ crystals without a magnetic transition. We found that the relaxation component of superconducting quasiparticles persisted from the superconducting state up to ~70 K in the normal state. Our findings suggest that the pseudo-gap-like feature in the normal state is possibly the precursor of superconductivity (in collaboration with K. H. Lin and M. K. Wu at Institute of Physics, Academia Sinica, Taiwan).
- Systematically studied the three representative families of iron chalcogenide superconductors: $FeTe_{1-x}Se_x$; $K_xFe_{2-y}Se_2$; and monolayer FeSe film grown on $SrTiO_3$ using angle-resolved photoemission spectroscopy. In addition to all three systems having universal and strong orbital-dependent correlation effects, all three families also have a superconducting state in close proximity to an orbital-selective Mott phase (in collaboration with Z. X. Shen at Stanford University).
- Successfully grew single-layer FeSe/STO thin films. STM measurements observed the superconducting gap above 50 K, making us the third group outside China to successfully grow the high temperature superconducting monolayer FeSe/STO films.
- Successfully obtained epitaxial $CaFe_2As_2$ films grown on $SrTiO_3$ substrate by choosing a Ca:Fe flux ratio of 1:1. Island growth mode rather than layer-by-layer growth mode, as well as $\sqrt{2} \times \sqrt{2}$ surface reconstruction, were both observed.
- Efforts are being made to raise the T_c in stable cuprates to above its current record of 164 K, which was established by us in 1993 in Hg1223, by inducing a possible electronic transition in the CuO_2-layers in the layered cuprates under ultra-high pressures.
- Developed a new synthetic strategy to grow large-sized black phosphorus through ternary clathrate $Sn_{24}P_{22-x}I_8$, under lower synthetic temperature and pressure, which will help future development of thin film growth on substrates.
- Observed a weak 3.8 K superconducting transition in alkaline-intercalated black phosphorus with a magnetic field effect similar to Sn. Careful investigations, including black phosphorus crystal growth mechanism, have demonstrated that the reported superconductivity in intercalated black phosphorus (BP) crystals with a T_c of 3.8 K [R. Zhang et al., *Nature Comm.* 8, 15036 (2017)] is not an intrinsic property of the BP crystals examined but is associated with the minute Sn-residue in the sample from crystal processing.
- Carried out detailed studies of CuCl samples under electrical and magnetic fields. Experimental observations showed a large resistive drop induced by an electrical field and two magnetic transitions in CuCl, without any trace of superconductivity.
- Systematic doping studies were conducted for Zr_5Ge_3, where Ru was doped onto different crystallographic sites: $Zr_{5-x}Ru_xGe_3$, $Zr_5Ge_{3-x}Ru_x$, and interstitial-filled $Zr_5Ge_3Ru_x$. Superconductivity was successfully induced in compounds where the Ge sites were partially doped by Ru, but not when Ru was doped into the Zr-site down to 1.8 K. Both magnetic and transport studies observed bulk superconductivity, with a T_c around 5.7 K for the $Zr_5Ge_{2.5}Ru_{0.5}$ sample. The high upper critical field, enhanced electron correlation, and extremely small electron-phonon coupling suggest the observed superconductivity is possibly unconventional.

- Carried out systematic doping studies of Mn_5Si_3-type Zr_5Ge_3 with other 5-3 type structures such as Cr_5B_3 and W_5Si_3 type compounds and discovered superconductivity up to 3.8 K in the $Zr_{5-x}Ta_xGe_3$ before structural instabilities occurred. The sample is a type-II superconductor with an upper critical field ~0.6 T; therefore, its superconductivity cannot be due to elemental Ta as tantalum is a type-I superconductor with a T_c ~4.5 K and H_c ~800 Oe. It should also be noted that superconductivity was not observed in the Y- and Cr-doped samples.
- Systematic transition metal doping studies were conducted for Zr_5Ge_3 and found that superconductivity was only induced for Ru- and Pt-doping at the Ge-site, *i.e.*, $Zr_5Ge_{3-x}Ru_x$ and $Zr_5Ge_{3-x}Pt_x$, but not in other sites, nor with different transition metal doping. The superconducting phase diagram was also further established.
- Carried out systematic doping studies of YFe_2Ge_2, which is isoelectronic and isostructural with the Fe-pnictide superconductor KFe_2As_2, with different species such as Ca, Zr, Cr, Ta, Ru, and Si. While measurements down to 2 K have not detected any signature of superconductivity to date, interesting magnetoresistance results were observed. NMR studies on the $YFe_2Ge_xSi_{2-x}$ compounds with different doping levels have shown strong ferromagnetic spin fluctuations within the Fe(Ge,Si) layers, whereas between-layer coupling is antiferromagnetic (in collaboration with D. Arcon at the University of Ljubljana).
- Discovered high thermoelectric performance with the record figure of merit (*ZT)* as high as 1.3 at 873 K in the Bi-based Zintl phases $(Ca,Yb,Eu)Mg_2Bi_2$. This *ZT* value is the highest ever reported in $CaAl_2Si_2$ based structures, especially compared to the best antimony (Sb)-based $YbZn_{0.4}Cd_{1.6}Sb_2$ compound (in collaboration with Z. F. Ren at UH).
- Achieved a record output power density of ~22 $W \cdot cm^{-2}$ in the p-type half-Heusler $Nb_{0.95}Ti_{0.05}FeSb$ based on a single-leg device operating at between 293 K and 868 K for possible large-scale power generation. The technique deployed is transferable to other thermoelectric materials (in collaboration with Z. F. Ren at UH).
- Grew large Cu_2OCl_2 crystals with multiferroic characteristics.
- Discovered a highly active catalyst derived from a 3D foam of $Fe(PO_3)_2/Ni_2P$ for extremely efficient water oxidation and found that this catalyst yields current densities of 10 mA/cm^2 at an overpotential of 177 mV, 500 mA/cm^2 at only 265 mV, and 1,705 mA/cm^2 at 300 mV, with high durability in an alkaline electrolyte of 1 M KOH even after 10,000 cycles, representing activity enhancement by a factor of 49 in boosting water oxidation at 300 mV relative to the state-of-the-art IrO_2 catalyst (in collaboration with Z. F. Ren at UH).
- Investigated the fluorination of electrically insulating hexagonal boron nitride (h-BN) and the subsequent modification of its electronic band structure to a wide bandgap semiconductor *via* the introduction of defect levels and found that the electrophilic nature of fluorine causes changes in the charge distribution around neighboring nitrogen atoms in h-BN, leading to room-temperature weak ferromagnetism, in agreement with theoretical calculations considering various possible configurations of fluorinated h-BN structures and their energy states. This chemical functionalization approach expands its functionality to electronic and magnetic devices (in collaboration with P. M. Ajayan at Rice University).
- Carried out systematic structural, optical, magnetic, magnetocaloric, and high-pressure studies of $HoCrO_3$ powder. The large magnetocaloric entropy change observed in $HoCrO_3$ and its tunability by Tm and Gd doping makes $HoCrO_3$ a promising system for next-generation low-temperature magnetic refrigeration (in collaboration with M. Jain at the University of Connecticut).

- Investigated the competition between the spin and charge orders in $Nb_2O_2F_3$ and the associated unusual phase transition with extremely slow kinetics, which was recently discovered by us with an unusual oxidation state of the Nb ion between 3+ and 4+. Two unusual magnetic transitions are also detected at 90 K and 50 K. With the successful synthesis of large-size single crystals of $Nb_2O_2F_3$, we want to unravel the origin of physics responsible for the transition and the associated slow kinetics (in collaboration with A. Guloy, UH; and C. R. dela Cruz and H. Cai, ORNL).
- The phase diagrams for the multiferroic compound $Mn_{1-x}Co_xWO_4$ with respect to pressure and doping effects were both constructed. Magnetic field effects on the magnetic and multiferroic properties of Co-doped $Mn_{1-x}Co_xWO_4$ were also investigated. Complementary neutron measurements were conducted on CORELLI at Oak Ridge National Laboratory, which helped confirm the phase diagram and establish the magnetic ordering for doping and pressure effects on the magnetic and ferroelectric phases in $Mn_{1-x}Co_xWO_4$. Initial pressure measurements were conducted employing dielectric (polarization) measurements and then followed up with additional high-pressure neutron scattering measurements to establish the phase. It was found that for the doping range of 0.1 to 0.15, the paraelectric AF1 phase becomes unstable under pressure and transforms into the conical spin phase, which is also multiferroic. The ferroelectric transition temperature also increases, and there is an enhanced stability of the multiferroic state. For the lower doping range examined, only a minimal increase is observed for the ferroelectric transition. We concluded that for doping of $0.1 < x < 0.15$, pressure suppresses the AF1 and thereby allows for stabilization of the conical spin phase. (in collaboration with F. Ye at ORNL)
- Discovered bulk superconductivity at 1.4 K in the ternary $SrPt_{10}P_4$ with a complex new structure.
- Conducted joint experiments with Eremets on the unstable hydrogen-sulfur system to confirm and unravel the nature of the system's reported transition at temperatures up to 203 K under 200 GPa.
- Achieved high thermoelectric performance in n-type Mg_3Sb_2-based materials by manipulating the ionized impurity scattering, with a ZT value of ~ 1.7 at 773 K in $Mg_{3.1}Co_{0.1}Sb_{1.5}Bi_{0.49}Te_{0.01}$ (in collaboration with Z. F. Ren at UH).
- Achieved high thermoelectric performance in cubic GeTe by Bi and Mn codoping and suppression of the phase transition, with a ZT~ 1.5 at 773 K and an average ZT of ~1.1 from 300 to 773 K in cubic $Ge_{0.81}Mn_{0.15}Bi_{0.04}Te$ (in collaboration with Z. F. Ren at UH).
- Synthesized high-quality BP and isotopically enriched ^{11}BP crystals, demonstrated high thermal conductivity > 500 W m^{-1} K^{-1} at room temperature, and studied the isotopic effects on the thermal conductivity and Raman spectroscopy. The measured thermal conductivities for BP and ^{11}BP are in good agreement with our first-principles calculations above 250 K (in collaboration with D. Cahill at UIUC and D. Broido at Boston College).
- Synthesized boron arsenide single crystals with seeded techniques with a size of 400–600 μm and an improved thermal conductivity of 351 ± 21 W m^{-1} K^{-1} at room temperature, which is almost twice as conductive as the previously reported BAs crystals by our group that was reported back in 2015 (in collaboration with Z. F. Ren at UH). (2017-2018)
- Developed a new synthetic approach to grow large-size BAs crystals up to mm size using different transport agents. Single-crystal X-ray, TEM, and STEM studies show single-domain crystals with no large-scale structural defects, such as grain boundaries or twinning. TDTR shows ultrahigh thermal conductivity of 1000±90 W m^{-1} K^{-1} at 300 K, a factor of 3

higher than the conductivity of silicon carbide and surpassed only by diamond and the basal plane thermal conductivity of graphite (in collaboration with D. Cahill at UIUC).

- Synthesized both thin plates and bulk crystals of BAs with size up to 4x3 mm^2 through a modified seeded technique. In bulk crystals the highest measured local thermal conductivity exceeds 1000 $Wm^{-1}K^{-1}$ at room temperature, where an average bulk value near 800 $Wm^{-1}K^{-1}$ is obtained. These findings confirm the recently proposed paradigm of phonon band engineering as a new route to achieve ultrahigh lattice thermal conductivity (in collaboration with Z. F. Ren at UH).
- Investigated the pressure-induced multiferroicity in $PrMn_2O_5$. We observed the emergence of a new pressure-induced magnetic phase in $PrMn_2O_5$ determined by powder neutron diffraction. The new magnetic phase becomes completely suppressed at 8 GPa. The magnetic structure stabilized under pressure should induce a strong spontaneous electric polarization due to the nearly perfect collinearity of the Mn^{3+} and Mn^{4+} spins (in collaboration with P. Foury-Leylekian at Laboratoire de Physique des Solides, France).
- Investigated a high-voltage Na_4NiTeO_6 cathode material for Na-ion batteries. It delivers 111 mAh g^{-1} at 0.1 C with 87% contribution from the inductive effect at voltage beyond 3.5 V. With the addition of fluoroethylene carbonate, a stable fluorine-containing solid electrode interface is formed, which significantly improves its cyclic performance (in collaboration with S. Chen at UH and Y. H. Huang at Hubei University of Technology, China).
- Developed an experimental setup to measure the Seebeck coefficient of a variety of samples down to 2 K and under magnetic fields up to 7 T employing the commercially available Quantum Design's Physical Property Measurement System. The developed puck and software control are suitable for studying the thermoelectric power of various sizes of samples, from length ~2 to greater than 5 mm with excellent temperature control. This new set-up is also suitable for studying superconductors, semiconductors, thermoelectrics, or topological materials in a wide temperature (2-400 K) and magnetic field (0-7 T) ranges. We have used this technique to measure the thermoelectric power of various bismuth-based topological single crystals ($Pb_{0.8}Sn_{0.2}Te$, Bi_2Te_3, $Bi_2Se_{2.1}Te_{0.9}$, and Sb_2Te_3).
- Studied phase transformations in niobium oxyfluoride Nb_2O_2F using Raman scattering spectroscopy. A phase transition from the high-temperature monoclinic (*I2/a*) phase to the low-temperature triclinic (P1) phase at $T_c \approx 90$ K is observed due to charge disproportionation of $(Nb{-}Nb)^{7+}$ dimers and creation of crystallographically nonequivalent dimers with long and short Nb-Nb bonds. Nb-Nb dimers are found to possess stretching vibrational frequencies as high as 382 cm^{-1}. Strikingly, the kinetics of structural transformation are strongly dependent upon the sample cooling rate. Fast cooling results in a "freezing" of the high-temperature monoclinic phase and allows us to observe a spin-ordered state below $T_N \approx 49$ K (in collaboration with A. M. Guloy at UH).
- Investigated a new two-dimensional material, "hematene," obtained from natural iron ore hematite (α-Fe_2O_3) by liquid exfoliation. The two-dimensional morphology of hematene is confirmed by transmission electron microscopy. Magnetic measurements coupled with density functional theory calculations confirm the ferromagnetic order in hematene while its parent form exhibits antiferromagnetic order. When loaded on titania nanotube arrays, hematene exhibits enhanced visible light photocatalytic activity. Our study indicates that photogenerated electrons can be transferred from hematene to titania despite a band alignment unfavorable for charge transfer (in collaboration with P. M. Ajayan at Rice University).

- Synthesized a metastable narrow gap semiconducting germanium allotrope Ge(oP32) as polycrystalline powders and single crystals from the oxidation of a layered Zintl phase Li_7Ge_{12} in ionic liquids. This germanium allotrope, which features a complex covalent network of 4-bonded Ge, results from a well-ordered topotactic oxidative condensation of $[Ge12]^{7-}$ layers. It is a diamagnetic semiconductor (E_g = 0.33 eV) and transforms exothermically and irreversibly to α-Ge at 363 °C (in collaboration with A. M. Guloy at UH).
- Studied a new 2D material, "ilmenene," exfoliated from the naturally occurring titanate ore ilmenite ($FeTiO_3$) by employing liquid phase exfoliation in dimethylformamide (DMF) solvent by ultrasonic bath sonication. The 2D nature makes ilmenene ideal for photocatalytic water splitting in combination with titania nanotube array electrodes, capable of harvesting a broad spectrum of sunlight, which yields a quantum efficiency up to ~7% at wavelengths above the absorption edge of anatase titania. Photoelectrochemical cells consisting of ilmenene (2D $FeTiO_3$)-1D TiO_2 photoanodes were fabricated for solar hydrogen fuel generation from water with improved performance characteristics (in collaboration with P. M. Ajayan at Rice University).
- Discovered a common T_c resurgence and a large T_c-enhancement of the monolayer $Bi_2Sr_2CuO_{6+\delta}$ and bilayer $Bi_2Sr_2CaCu_2O_{8+\delta}$ to beyond the maximum T_cs predicted by the universal relation between T_c and doping (*p*) or pressure (P) at higher pressures. This T_c resurgence has been attributed to a possible pressure-induced Fermi surface topology change in the compounds leading to a charge transfer between different bands, and an increase of the density of states at the Fermi level. This discovery suggests that higher T_cs than those previously reported for the layered cuprate high-temperature superconductors can be achieved by breaking away from the universal T_c-P relation through the application of higher pressures.
- Successfully stabilized the high-pressure superconducting phase of Sb at ambient pressure upon the removal of pressure by examining the metastability of the phase and following a specific thermodynamic path. The results offer hope to retain the high T_c high pressure phases in cuprates and molecular solids of hydrides without the application of pressure.
- Observed a signature of the chiral magnetic effect in single-crystalline Sb at ambient pressure below ~ 60 K, *i.e.*, the magnetoresistance is huge and positive when H is perpendicular to current, while small and negative when the H is parallel to the current, offering a new avenue for quantum computation. A manuscript is being prepared for publication.
- Induced superconductivity in single crystals of $Ca(Fe_{1-x}Co_x)_2As_2$ and $AeFe_2As_2$ (Ae = Ca, Sr, Ba) grown by self-flux solution method, with onset-T_c at 15 K, 16 K, 22 K, and 24 K, respectively. Systematic characterization, including resistivity, Hall coefficient, magnetization, X-ray diffraction, and wavelength dispersive spectroscopy results were performed to reveal the underlying mechanism for the reported superconductivity in the Fe-based 122 system.
- Synthesized Weyl semimetal samples of $Mo_{1-x}W_xTe_2$ with x = 0.10, 0.25, 0.30, 0.50, 0.60, 0.70, and 0.90 and conducted detailed chemical analysis, transport measurements, and pressure measurements up to 18 kbar. Structural transitions were observed for $Mo_{0.25}W_{0.75}Te_2$ under 12.3 kbar, and for $Mo_{0.60}W_{0.40}Te_2$ and $Mo_{0.90}W_{0.10}Te_2$ at ambient pressure. Superconductivity was induced in $Mo_{0.60}W_{0.40}Te_2$ with an onset T_c of 1.7 K under 8.4 kbar and in $Mo_{0.90}W_{0.10}Te_2$ with an onset T_c of 1.4 K under 3.9 kbar. T_c has been observed to increase continuously with pressure. Additional measurements are in progress using diamond anvil cells to investigate the properties of this system at higher pressures.

- Studied the new superconducting phases $K_2Mo_3As_3$ and $Rb_2Mo_3As_3$ with $T_c \sim 10$ K and proposed the possible existence of Tomonaga-Luttinger liquid (TLL) behavior in this system.
- Investigated the surface terminations and reconstruction of 122-type alkaline-earth metal iron pnictides $AeFe_2As_2$ (Ae = Ca, Ba) with scanning tunneling microscopy/spectroscopy. Layer-resolved tunneling spectroscopy on these terminating surfaces reveals a well-defined superconducting energy gap on the As terminations, while the gap features become weaker on the Ae terminations and absent on the Fe terminations. (in collaboration with S. H. Pan at UH)
- Successfully expanded and enhanced the skyrmion phase region from the small range of 55 to 58.5 K to 5 to 300 K in single-crystalline Cu_2OSeO_3 by applying pressures up to 42.1 GPa through a series of phase transitions. The results are in agreement with our Ginzburg–Landau free energy analyses. The observations also indicate that the skyrmion state can be achieved at higher temperatures in various crystal symmetries, suggesting the insensitivity of skyrmions to the underlying crystal lattices and thus the possible more ubiquitous presence of skyrmions in helimagnets.
- Suppressed the rhombohedral-cubic phase-transition in GeTe by a simple Bi and Mn codoping and achieved a peak thermoelectric figure of merit (ZT) of ∼1.5 at 773 K and an average ZT of ∼1.1 from 300 to 773 K in cubic $Ge_{0.81}Mn_{0.15}Bi_{0.04}Te$. (in collaboration with Z. F. Ren at UH)
- Investigated thermoelectric properties of Zintl-phase Eu_2ZnSb_2 and achieved maximum ZT value reaching ∼1.0 at 823 K for $Eu_2Zn_{0.98}Sb_2$ by regulating the Zn deficiency. (in collaboration with Z. F. Ren at UH and Q. Zhang at Harbin Institute of Technology)
- Synthesized high-quality BAs crystals and demonstrated high thermal conductivities >1000 $W\ m^{-1}\ K^{-1}$ at room temperature, opening a new paradigm for thermal management in power electronics. (in collaboration with Z. F. Ren at UH)
- Investigated the coefficient of thermal expansion (CTE) of BAs and BP materials through X-ray and neutron diffraction in extended temperature ranges from 100 K to 1150 K. Demonstrated the room-temperature CTE values, $3.6 \pm 0.15 \times 10^{-6}$/K for BAs and $3.2 \pm 0.2 \times 10^{-6}$/K for BP, which indicate that these compounds could be better candidate materials for heat spreaders in future power electronic devices.
- Synthesized $CaMn_2O_4$ samples and explored the effects of oxygen deficiencies under different annealing conditions. A spontaneous polarization (P_S) was observed near 253 K only for the oxygen-annealed sample without applied magnetic field, while no P_S was observed for the argon-annealed sample. Further dielectric measurements revealed anomalies above 260 K with the time- and magnetic-field-dependent phenomena in both samples.
- Found that $HoFeWO_6$ orders antiferromagnetically below $T_N \sim 17$ K. Observed an anomaly in the dielectric constant and dielectric loss, as well as a polarization switchable by an electric field at the same temperature, revealing the ferroelectric nature of this transition. We further found that below the temperature $T_{C2} \sim 5$ K, there appears a polarization ($\mathbf{P}_2$) in the opposite direction of the ($\mathbf{P}_1$) at $T_{C1} \sim 17$ K. While external magnetic field suppresses the polarization in the first transition ($\mathbf{P}_1$), at a critical field $H_{C1} \sim 1$T, the direction of $\mathbf{P}_2$ is flipped.
- Investigated magnetic property changes in $RE_2Fe_4Sb_5$, which is structurally related to the Fe-based superconductors, with Co doping, and showed that the incorporation of Co into the Fe triangular sublattice leads to an itinerant magnetic system.
- Discovered a non-stoichiometric ternary Zr-Pt-Sb antimonide with a new structure type. Detailed chemical bond analysis and calculations show that partial occupancy at one

particular Zr site c creates a pseudogap and accounts for the chemical stability of this compound.

- Continued to investigate a new 2D material "ilmenene" exfoliated from the naturally occurring titanate ore ilmenite ($FeTiO_3$). Probable charge transfer excitation from $Fe^{2+}Ti^{4+}$ to $Fe^{3+}Ti^{3+}$ results in ferromagnetic ordering along with the antiferromagnetic phase accompanied by enhanced anisotropy due to surface spins. The 2D nature and band gap states help ilmenene form a heterojunction photocatalyst with titania nanotube arrays, capable of broad-spectrum light harvesting and separating/transferring the photogenerated charges effectively for solar photoelectrochemical water splitting (in collaboration with P. M. Ajayan at Rice U).
- Successfully obtained ultrathin two-dimensional sheets of manganese (II) telluride having an average thickness of ∼2 nm and flake size of ∼100 nm by liquid phase exfoliation presumably for the first time. Vanishing of exchange interactions in two dimensions results in paramagnetic ordering in manganese (II) telluride sheets while its pristine form prefers antiferromagnetic order. The exfoliated 2D sheets were used to sensitize titania nanotubes to broaden the absorption spectrum and utilize visible light for photoelectrochemical water splitting (in collaboration with P. M. Ajayan at Rice U).
- Studied the unconventional structural transformation of fluorinated WS_2 (FWS_2) into the 1T phase. The fluorination enhances the stability of 1T FWS_2 and makes it energetically favorable at higher F concentration. Investigation of the electronic and optical nature of FWS_2 is supplemented by possible band structures and bandgap calculations. Friction force microscopy is used to determine this effect of functionalization accompanied phase transformation (in collaboration with P. M. Ajayan at Rice U).
- Investigated hexagonal boron nitride (h-BN) by carbon doping. The h-CBN is prepared by using GQDs as seed nucleations for the epitaxial growth of h-BN along the edges of GQDs without the assistance of metal catalysts. An enhanced ferromagnetism in the h-CBN emerges due to the spin polarization and charge asymmetry resulting from the high density of C-N and C-B bonds at the boundary between the GQDs and the h-BN domains. The saturation magnetic moment of h-CBN reaches 0.033 emu g^{-1} at 300 K, which is three times that of as-prepared single carbon-doped h-BN (in collaboration with P. M. Ajayan at Rice U).
- Studied the new oxohalide-based multiferroic Cu_2OCl_2 and established a novel mechanism for the origin of charge ordering in this compound through detailed magnetization, specific heat, neutron diffraction, and dielectric measurements (in collaboration with H. D. Yang at National Sun Yat-Sen U, Taiwan)
- Investigated magnetization and dielectric properties of Pb_2MnO_4 as a function of temperature, magnetic field, pressure, and electric field to address the magnetoelectric coupling and identify the underlying mechanism for this phenomenon (in collaboration with H. D. Yang at National Sun Yat-Sen U, Taiwan).
- Studied $Cu_9O_2(SeO_3)_4Cl_6$, which has robust antiferromagnetic interactions and a $T_N = 37$ K, through magnetization, specific heat, and dielectric measurements as a function of temperature, magnetic field, and electric field to investigate possible magnetoelectric coupling below T_N and antiferroelectric order below $T_E \sim 267$ K (in collaboration with H. D. Yang at National Sun Yat-Sen U, Taiwan).
- Synthesized polycrystalline samples $Co_3Sn_2S_{2+x}$ ($0 \leq x \leq 0.34$), $(Co_{1-y}Ni_y)_3Sn_2S_{2.26}$ ($0 \leq y \leq 0.05$), and $(Co_{1-z}Fe_z)_3Sn_2S_{2.26}$ ($0 \leq z \leq 0.05$). Discovered that a so-called A' phase within the A phase can be sketched in the H-T phase diagram of $Co_3Sn_2S_2$. Investigated the effects of

external high-pressure and chemical substitutions. The possible origin of the field-induced phase A' could be ascribed to the non-collinear skyrmion-like phenomenon. Our findings in kagome $Co_3Sn_2S_2$ provide a new candidate in spin-frustrated systems to explore the complexity of magnetic-field-induced magnetism (in collaboration with H. D. Yang at National Sun Yat-Sen U, Taiwan).

- Investigated the magnetic structures of the multiferroic $GdMn_2O_5$ *via* single-crystal neutron diffraction. The system undergoes a first-order incommensurate-to-commensurate magnetic transition below 33 K, accompanied by the appearance of electric polarization P. Upon cooling, P increases smoothly while the magnetic order shows an abrupt enhancement in intensity below 20 K owing to the large increase of the rare-earth Gd moment. The contrasting temperature evolution of the magnetic order and polarization indicates the polarization is mainly driven by the exchange striction between the magnetic Mn ions (in collaboration with F. Ye at ORNL).
- Investigated a highly active oxygen evolution catalyst in neutral pH, Brownmillerite Sr_2GaCoO_5, with specific activity about one order of magnitude higher than that of the widely used iridium oxide catalyst. Using Sr_2GaCoO_5 to catalyze oxygen evolution, the integrated CO_2 reduction achieved an average solar-to-CO efficiency of 13.9% with no appreciable performance degradation in 19 hours of operation. Our results not only set a record for efficiency in sunlight-driven CO_2 reduction, but open new opportunities toward the realization of practical CO_2 reduction systems (in collaboration with Y. Yao at UH and C. Ling at Toyota Research Institute of North America).
- Discovered sensitive but non-invasive methods that not only allow direct correlation of luminescence with the underlying structure, but also the distinguishing of point defects from embedded nanostructures. Identified the difference between emissive and non-emissive Cs_4PbBr_6 crystals and revealed the existence of $CsPbBr_3$ nanocrystals in emissive Cs_4PbBr_6. Investigated the hydrostatic-pressure effect on luminescence centers. Our observations excluded Br vacancies as possible luminescent centers. The resolution of this longstanding controversy paves the way for further device applications of low-dimensional perovskites (in collaboration with J. M. Bao at UH).
- Discovered superconductivity in the intercalated 2D material $SnSe_2$ and reported the highest superconducting T_c of 7.8 K thus far with organic cointercalations. We also determined its unusual superconducting phase diagram. The T_c is independent of the doping level but rather scales with the interplanar spacing during organic intercalation. The new method has been adopted to other systems with similar T_c enhancement observed.
- Discovered new phases in the Pd-Se-Te system with superconducting T_c at 2.8 K.
- Synthesized high-quality complex Fe chalcogenides $BaFe_2Se_4$ and $K_3Fe_2Se_4$ with a quasi-1D $FeSe_4$ feature, carried out detailed magnetic and transport studies, and discovered unusual canted antiferromagnetism in this system.
- Discovered a new layered $BaPt_4Se_6$ compound with mixed Pt valence and multiple anomalies at low temperatures.
- Discovered a new ternary nonstoichiometric chalcogenide $Zr_{6.5}Pt_6Se_{19}$, which has both 2D and 1D features in the layers. Detailed magnetic, transport, and band structure calculations suggest it is a very narrow semiconductor with a band gap ~ 0.1 eV.
- Discovered a new polymorphic phase of $BaCu_2As_2$ with a much larger *c* lattice through different flux growth. This new phase is an intergrowth structure of the well-known $ThCr_2Si_2$ and $CaBe_2Ge_2$ types and could be a prototype to design new superconductors among the Fe

pnictides. Transport studies reveal that the carriers are p-type and that the magnetoresistance increases up to 22% at 5 K and under a magnetic field of 7 T.

- Induced superconductivity in a new polymorphic phase of $BaCu_2As_2$ with Z=10 (β-$BaCu_2As_2$) by pressure. Three superconducting phases were observed in this Sn flux grown crystal. One occurs below 9 GPa, the second occurs at ~9 to ~20 GPa, and the third occurs above 20 GPa and continues to about 60 GPa with a maximum onset T_c of 7 K at 24 GPa. We confirmed that the third superconducting phase at pressure above 20 GPa is from β-$BaCu_2As_2$. The other two superconducting phases at P below 20 GPa, although similar to that of Sn, which was used as flux, are also most likely from β-$BaCu_2As_2$ rather than Sn residue.
- Conducted high-pressure resistance measurements on quasi-1D multiferroic $LiCu_2O_2$ crystal up to 60 GPa. The pressure dependence of electrical anisotropy was studied. Preliminary results show that the electrical anisotropy increases with increasing pressure and then decreases with further increasing pressure. The maximum anisotropy is located at ~30 GPa at room temperature and at ~36 GPa at 4 K. The crystal becomes less insulating in both directions of the ab plane as the pressure increases, and the resistance value decreases about 10E5 times from ambient to 60 GPa.
- Performed high-pressure transport measurements on novel chalcogenide $BaFe_2Se_4$. Preliminary data shows that the band gap could be tuned by application of pressure, a semiconductor-to-metal transition was observed at pressure of ~10 GPa, and trace superconductivity was detected at pressure above ~20 GPa.
- Conducted high-pressure resistivity measurements up to 63 GPa on non-superconducting single-crystalline antimony, in which several structural transitions were detected. Superconductivity associated with the m-HGL phase was observed with a T_c of 3.4 K above ~ 9 GPa and for the bcc phase a T_c of 4.0 K above ~ 30 GPa was successfully retained up to 135 K and 110 K, respectively, upon the complete and rapid removal of the pressure at 77 K.
- Discovered a unique approach in which copper (Cu) atoms from bulk Cu solid intercalate spontaneously into van der Waals (vdW) gaps of group IV and V layered TMDs at room temperature and atmospheric pressure. The Fermi level of NbS_2 shifts up because of the intercalation of Cu, resulting in the improvement of the electrical conductivity in the z-direction. Intercalation of Cu into vdW gaps of NbS_2 systematically suppresses the superconducting transition temperature and the superconducting volume fraction (in collaboration with P. M. Ajayan at Rice University).
- Discovered that high pressure can effectively suppress the complex magnetism of crystalline MnSe observed at ambient condition and successfully induce superconductivity. Superconductivity with T_c ~ 5 K was first observed in magnetic measurements at a pressure of ~12 GPa. The highest T_c is ~ 9 K at ~35 GPa. Our observations suggest the interfacial effect between the metallic and insulating boundaries may play an essential role in the pressure-induced superconductivity in MnSe (in collaboration with M. K. Wu at Institute of Physics, Academia Sinica, Taiwan).
- Resistivity measurements were performed on single crystals of Bi and FeSe under high pressure using diamond anvil cells. Discovered an approach to lock in metastable superconductivity by pressure quenching for enhanced T_c in both of these systems.
- Systematically characterized the atomic structure and electronic properties of $CaBi_2$(010) thin films grown by molecular beam epitaxy (MBE) and found that their growth follows a Stranski-Krastanov mode. A nonreconstructed I_{Bi} layer and a (1 × 2) reconstructed II_{Ca} layer were found to be the most common surfaces. Nonreconstructed III_{Bi} and V_{Ca} layers were

further exposed with reduced bismuth growth flux. All of these constituent layers exhibit unique features in the STS spectra, indicating that unique electronic properties exist in each specific constituent layer.

- Investigated the strain-modified electronic states of three-monolayer (3-ML) Bi(110) films grown on graphene/6H-SiC(0001) by using low-temperature scanning tunneling microscopy/spectroscopy (STM/S). We located inversion domain boundaries (IDBs) across which atoms are configured in the opposite order. The defect has extended spatial distribution and 1D parabolic energy dispersion. We suggest this defect as a potential candidate for nontrivial 1D edge states.

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L. Z. Deng, Ph.D., University of Houston, Physics, August 2015, Dissertation: "Possible Interface Enhanced Superconductivity in Iron Pnictides and Chalcogenides"

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N. Poudel, Ph.D., University of Houston, Physics, May 2017, Dissertation: "Tuning of the multiferroic properties of selected materials by pressure, magnetic field and chemical doping"

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L. Mastalli-Kelly, Ph.D., University of Houston, Physics, August 2019, Dissertation: "Systematic Investigation of the Pr-Ba-Cu-O Phase Diagram"

S. Y. Huyan, Ph.D., University of Houston, Physics, December 2019, Dissertation: "Investigation of the Possible Mechanism Underlying Superconductivity in the Parent Compounds of Iron-Based 122 Material Systems"

X. Liu, Ph.D., The University of Texas at Dallas, Physics, May 2020, Dissertation: "Superconductivity, Magnetism and Topological Properties of Several Quasi-1D Materials"