

# **FINAL REPORT**

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## **Response of Simple, Model Systems to Extreme Conditions**

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**Final Report (DE-FG02-97ER45656)**

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Extreme Conditions**

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**University of Michigan**

**ABSTRACT**

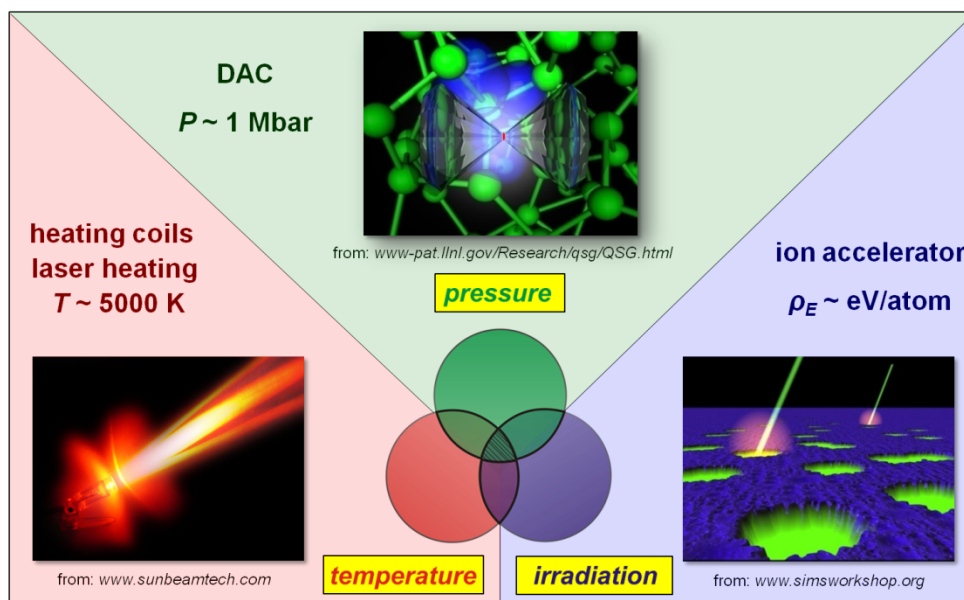
This grant was a continuation of our research program on the response of materials to extreme conditions (DE-FG02-97ER45656). However, the new focus of the proposed research was on the application of high-pressure/high-temperature techniques, together with intense energetic ion beams, to the study of the behavior of simple oxide systems (*e.g.*,  $\text{Ln}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{GeO}_2$ ,  $\text{SnO}_2$ ,  $\text{PbO}_2$ ,  $\text{Ta}_2\text{O}_5$ ) under extreme conditions. These simple stoichiometries provide unique model systems for the analysis of structural responses to pressure up to and above 1 Mbar, temperatures of up to several thousands of kelvin, and the extreme energy density generated by energetic heavy ions (tens of eV/atom). The investigations included systematic studies of radiation- and pressure-induced amorphization of high  $P$ - $T$  polymorphs. By studying the response of simple stoichiometries that have multiple structural “outcomes”, we have established the basic knowledge required for the prediction of the response of more complex structures to extreme conditions.

We especially focused on the amorphous state and characterized the different non-crystalline structure-types that result from the interplay of radiation and pressure. For such experiments, we made use of recent technological developments, such as the diamond-anvil cell and *in situ* investigation using synchrotron x-ray sources. We have been particularly interested in using extreme pressures to alter the electronic structure of a solid prior to irradiation. We expected that the effects of modified band structure would be evident in the track structure and morphology, information which is much needed to describe theoretically the fundamental physics of track-formation. Finally, we investigated the behavior of different simple-oxide, composite nanomaterials (*e.g.*, uncoated nanoparticles *vs.* core/shell systems) under coupled, extreme conditions. This provided insight into surface and boundary effects on phase stability under extreme conditions.

During the past two years, most of the funds have been used to support the research of Alex Cusick, a Ph.D. candidate in the Materials Science & Engineering Department at the University of Michigan. Alex’s research focused on irradiation studies of Sn-, Pb- and Ta-oxides. Alex’s Ph.D. defense was on June 19, 2015. At least three papers will result from his dissertation research.

## INTRODUCTION

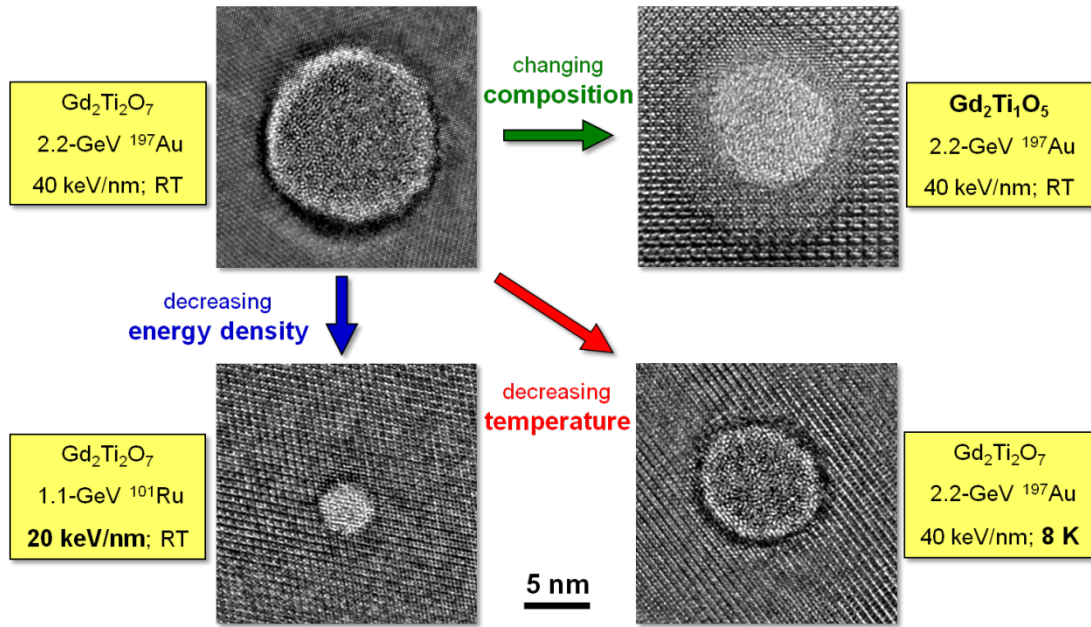
We have investigated the response of simple model systems, mainly oxides, to combinations of the extreme conditions of temperature, pressure, and high-radiation fields (Figure 1). The recent report (2007) of a Basic Research Needs workshop, *Technology and Applied R&D Needs for Materials Under Extreme Environments*, summarized by R.J. Hemley and colleagues in *Physics Today* (2009) [1], provided a strong rationale for the need to investigate materials under extreme environments. Of particular interest is the discovery of new materials in order to develop the full potential of advanced energy systems, not only for nuclear applications, but also for solar, fossil and hydrogen energy sources. Thus, the response of materials to the coupled effects of the extreme conditions of irradiation, pressure, and temperature is of technological and fundamental importance.



**Figure 1:** The purpose of this research is to investigate the response of materials to the extreme conditions of high energy deposition by ion irradiation at high pressures and temperatures.

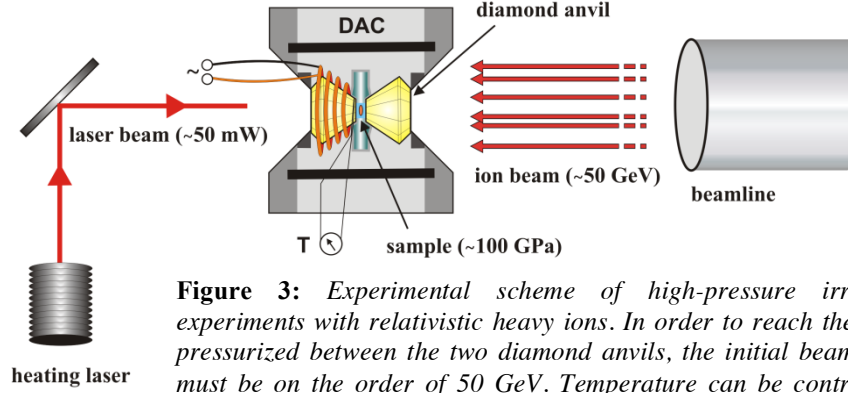
From a fundamental science perspective, irradiation, pressure, and temperature provide a unique means of creating non-equilibrium states of matter, providing new possibilities for tailoring novel materials with new properties to performance requirements in extreme environments. Important progress has been achieved utilizing temperature and pressure to modify and “manipulate” the properties of materials [2,3]. The combination of high-pressure devices with powerful laser systems has laid the foundation of major breakthroughs in extreme-condition science [1,2]. Novel structure-types have been synthesized with unusual physical and chemical properties. There are many parallels among the response of materials to temperature, pressure and irradiation [4]. Ion-beam irradiations are a standard means of nano-scale engineering of the properties of materials [5,6]. In particular, energetic ions exhibit exceptional properties because they deposit their kinetic energy within an exceedingly short interaction time ( $< \text{fs}$ ) into nanometer-sized sample volumes [7]. This high density of energy transferred to

the electrons along the ion path leads to local states of intense electronic excitation, a confined plasma-like state that screens the Coulomb field and decreases rapidly away from the ion trajectory. Through electron-phonon coupling, the excited electrons transfer their energy to target atoms by exceeding the average binding energy (tens of eV/atoms), which leads to local melting with an accompanying shock wave to the surrounding material. These collective processes, which occur over pico- to nano-second time frames, drive the local atomic structure far from equilibrium, resulting in rapid phase transition pathways through equilibrium and non-equilibrium states that trigger complex structural modifications within a cylindrical zone of diameter  $\sim 10$  nm, the so-called ion track [7]. *It is within this zone that one finds the most extreme conditions, and new advances in transmission electron microscopy allowed us to investigate in detail the internal structure of such tracks under a wide range of experimental conditions (Figure 2).*



**Figure 2:** HRTEM images of ion tracks under changing conditions at ambient pressure [7]. (top left) Track in  $\text{Gd}_2\text{Ti}_2\text{O}_7$  irradiated with 2.2 GeV Au ions (40 keV/nm) at room temperature (RT). (top right) Track in  $\text{Gd}_2\text{TiO}_5$  irradiated with 2.2 GeV Au ions, illustrating the effect of change in composition. (bottom left) Track in  $\text{Gd}_2\text{Ti}_2\text{O}_7$  irradiated with 1.1 GeV Ru ions (20 keV/nm) at RT, illustrating the effect of a decrease in deposited energy density. (bottom right) Track in  $\text{Gd}_2\text{Ti}_2\text{O}_7$  irradiated with 2.2 GeV Au ions (40 keV/nm) at 8 K, illustrating the effect of decreased temperature.

Combining such energy depositions simultaneously with extremes in pressure and temperature adds complexity and provides unique access to an extended pressure-temperature space, which is difficult to access by any other means. The coupling of pressure-temperature with ion beams has been recently achieved [8-12] by injecting relativistic heavy projectiles through the diamond anvil of a diamond-anvil cell into the pressurized sample (Figure 3). *The study of the interaction of nano-scale structures and surfaces with different types of irradiation over a range of temperatures and pressures opened up an entirely new research field that has mainly developed at the University of Michigan through a web of international collaborations.*



**Figure 3:** Experimental scheme of high-pressure irradiation experiments with relativistic heavy ions. In order to reach the sample pressurized between the two diamond anvils, the initial beam energy must be on the order of 50 GeV. Temperature can be controlled by either heating wires [8] or an intense heating laser beam.

The integration of the concept of non-equilibrium thermodynamics, introduced by irradiation, pressure, and temperature provided a new means of investigating fundamental phenomena and developing new materials with improved properties. *During the past three years, we have investigated model systems of materials that have simple stoichiometries, but for which there are many structural possibilities. In particular, we investigated the separate and coupled effects of high pressure, elevated temperature, and high radiation fields on phase transformations in these materials* in order to address the following fundamental questions:

- *How do solids respond to the simultaneous exposure of several extremes, such as pressure, temperature, and rapid, high-energy deposition?*
- *What is the relation of pressure-induced and radiation-induced amorphization?*
- *Are novel structure types, such as ultra-dense glasses, accessible by the combined application of extreme conditions?*
- *Can nanoscale manipulations by swift heavy ions “tune” the thermodynamic properties of unstable high-pressure phases?*
- *How does radiation damage and track formation evolve in different high-pressure polymorphs with the same composition?*
- *Are the known pressure-temperature phase boundaries relevant for radiation-damaged materials?*
- *What is the behavior of different simple-oxide, composite nanomaterials under coupled extreme conditions?*

The coupled effects of pressure, temperature and irradiation are a grand scientific challenge that requires the combination of novel experimental design and the use of state-of-the-art *in situ* and *ex situ* characterization techniques. For a successful technical implementation, three experimental conditions need to be fulfilled: (1) the sample must be enclosed in a high-pressure cell, (2) the ions require sufficient kinetic energy to travel completely through one of the pressure anvils to reach and traverse the sample under investigation, and (3) a heating laser (or heating wire) has to be connected with the pressure cell at the irradiation site (Figure 3). With a typical anvil thickness of about 2 mm, the minimum ion energy is on the order of 200 MeV/u (MeV per nucleon, i.e., for  $^{238}\text{U}$  ions 47.6 GeV), which is only available at a few accelerator facilities [13]. The first

ion irradiation experiments of pressurized samples showed that the combination of swift heavy ions and high pressure triggers dramatic material modifications in solids that are not observed with the application of only pressure or only ion irradiation [8]. It has been demonstrated that this approach provides a wide spectrum of geoscience applications from nanoscale simulations of fission-track formation in Earth's crust [9] to phase transitions of radiation-damaged minerals resulting from meteorite impact [14]. By manipulating the structure of pressurized solids at the nanoscale, this new technique provided interesting applications for materials research, such as the stabilization of novel materials to ambient conditions that otherwise could not be recovered [11]. Based on this success, we extended this approach and investigated the response of simple oxide systems to combinations of pressure up to and above 1 Mbar (1 Mbar = 100 GPa), temperature of up to several thousands of kelvin, and the extreme energy density generated by energetic heavy ions (tens of eV/atom). These extreme conditions were applied to single crystals, nano-materials, and amorphous structures.

The research objectives included:

- (a) ***Consequences of non-equilibrium energy depositions of ion beams into solids at extremely high pressures and temperatures:*** Relativistic ions provide an exceptional physical regime including local states of intense electronic excitation, confined plasma-like states, local melting with an accompanying shock wave [7]. It is of great importance to study the consequences of such projectiles in pressurized simple oxides. In particular, the propagation of ion-induced nanoscale shock waves in materials at Mbar-pressure is a new way of coupling static and dynamic compression methods. This will give rise to novel thermodynamic conditions in solids, which are inaccessible by other means.
- (b) ***Radiation response of unique high-pressure/ temperature structure types:*** Oxides have complex phase diagrams at high pressure and temperature and can form exotic solids with exceptional properties (e.g., dense glasses with enhanced hardness) [2]. Since most of these structure types are unstable at ambient conditions, they have never been exposed to energetic ions before. It is of fundamental scientific importance to investigate the influence of ion-induced energy deposition in such high-pressure phases.
- (c) ***Ion-beam induced stabilization of unstable high-pressure/ temperature phases:*** Radiation-induced fragmentation into nanocrystals at high pressure has been demonstrated in complex ceramics as a unique tool for *in situ* modification of the thermodynamic pathways upon pressure release [11]. Since simple oxides, such as SiO<sub>2</sub> have many technological applications and are of importance in Earth science, it is of major relevance to test whether ion-beam irradiation at high pressure leads to the stabilization of non-quenchable structure types. This may yield useful new materials with interesting applications (e.g., hard dense glasses) and have important implications for an improved understanding of geodynamic properties (e.g., post-stishovite).

- (d) ***Relation between pressure- and radiation-induced amorphization:*** Energetic ion irradiation [7] and high pressure [15] are two different means of inducing amorphization in crystalline solids. In particular, simple oxides are prone to periodic-aperiodic transformations and form novel amorphous structures. The details of pressure- and radiation-induced amorphization have never been directly compared. Systematic studies are required in order to gain a more fundamental understanding of the process of amorphization. The atomic-scale disordering mechanisms and related energetics need to be investigated over a wide range of pressure and radiation conditions.
- (e) ***Influence of pre-existing radiation damage on phase-transitions at high-pressure and high-temperature:*** Phase diagrams of simple oxides are well established and transformations to high pressure/ temperature phases are fully characterized (e.g., quartz  $\rightarrow$  coesite  $\rightarrow$  stishovite with increasing pressure). In natural environments, minerals are exposed to the radiation of radioactive-decay products resulting in defects and amorphization. Pressures and temperatures of phase transitions can be significantly changed by the microstructure that results from radiation damage [14]. Thus, we will investigate the influence of radiation damage on phase-transformations in simple oxides.

All five of these research objectives were investigated and the results presented in a series of twenty-one papers. Additionally, one paper has been submitted and four papers are in preparation.

## ACCOMPLISHMENTS OF RESEARCH PROGRAM

(2011 to 2015)

The principal scientific accomplishment of the past three years has been to develop the methodology for swift heavy ion irradiation in combination with high pressure (up to 100 GPa) experiments. This has involved, not only developing the irradiation techniques, but the use of synchrotron sources (NSLS, CHESS, APS and ALS) to investigate *in situ* phase transformations at high pressures. In addition, we have perfected procedures that allow us to reclaim DAC samples for examination by high-resolution transmission electron microscopy. Very few research groups have such a range of spectroscopic, x-ray diffraction and TEM techniques available for the study of the response of materials to extreme conditions. We have also applied a combination of quantum mechanical calculations (DFT) and Monte Carlo simulations to understand the response of materials on the nanoscale (see for example, the recent paper in the *Journal of Materials Research*, 2010, that describes and simulates the internal structure of individual tracks created by swift heavy ions [7]). Most interestingly, by collaboration with Patrick Kluth at ANU in Australia, we have combined small angle x-ray scattering studies with high pressure conditions (in preparation).

Professor Ewing continues to be actively involved in issues related to the nuclear fuel cycle as a member of the *ad hoc* committee for the Materials Research Society symposium entitled, *Scientific Basis for Nuclear Waste Management*. He was a member of the International Advisory Board for the *Thirty-Fifth International Symposium on the Scientific Basis for Nuclear Waste Management*, Buenos Aires, Argentina, 2011. He is the co-chair of the symposium to be held in 2016 at the fall meeting of the Materials Research Society (40<sup>th</sup> anniversary).

Professor Ewing is a co-organizer for the symposium N “Nuclear Materials” for *E-MRS IUMRS International Conference*, June, 2011, Nice, France, and was a co-organizer of a symposium on the nuclear fuel cycle at the IUMRS meeting in Kuyushu, Japan, in 2015. Ewing is presently on the International Committee of *Radiation Effects in Insulators* for which the next meeting will be in Jaipur, India in October of 2015.

In 2012, Ewing was appointed chair of the Nuclear Waste Technical Review Board by President Obama.

Professor Maik Lang is a co-chair for the NUMAT 2016 conference in France and on the International Committee for the Swift Heavy Ion Meeting. He also was a panel member of the Basic Research Needs workshop during July, 2015.

### Honors for research team:

1. Maik Lang received the Alvin Van Valkenburg Award, which recognizes a young scientist who has made seminal contributions to high-pressure science (2010).



2. Maik Lang received the Powe Junior Faculty Enhancement Award, ORAU, Oak Ridge, TN, (2015).
3. Professor Ewing was named the recipient of the Roebling Medal of the Mineralogical Society of America, its highest honor (2015).
4. Professor Ewing was named the recipient of the IMA Medal for superlative research in mineral sciences from the International Mineralogical Association (2015).
5. Professor Ewing named the Edward H. Kraus Distinguished University Professor by the University of Michigan (2010 – 2013).

## PUBLICATIONS & INVITED PRESENTATIONS

(2011 to 2015)

### Papers in preparation:

A.B. Cusick, M. Lang, F.X. Zhang, Z.G. Li, K. Sun, C. Trautmann, and R.C. Ewing, *Decomposition and multiple phase transformations in heavy ion irradiated PbO<sub>2</sub>*, in preparation.

A.B. Cusick, M. Lang, F.X. Zhang, Z.G. Li, K. Sun, C. Trautmann, and R.C. Ewing, *Amorphization of Ta<sub>2</sub>O<sub>5</sub> under swift heavy ion irradiation*, in preparation.

A.B. Cusick, M. Lang, F.X. Zhang, J.M. Zhang, M. Rodriguez, P. Kluth, C. Trautmann, R.C. Ewing, *Swift Heavy Ion-Induced Decomposition and Phase Transformation in Nanocrystalline SnO<sub>2</sub>*, in preparation.

C.L. Tracy, M. Lang, F.X. Zhang, C. Trautmann, R.C. Ewing, *Phase transformations of Ln<sub>2</sub>O<sub>3</sub> materials irradiated with swift heavy ions*, submitted to *Physical Review B*.

R.I. Palomares, V.B. Prakapenka, F.X. Zhang, C. Trautmann, R.C. Ewing, M. Lang, *Swift heavy ion irradiation of dense GeO<sub>2</sub> glass at high pressure: Formation and stabilization of a disordered NiAs-type structure*, in preparation.

### Journal articles (most recent listed first):

M. Lang, F.X. Zhang, J.M. Zhang, C.L. Tracy, A.B. Cusick, J. VonEhr, Z.Q. Chen, C. Trautmann, R.C. Ewing, *Swift heavy ion-induced phase transformation in Gd<sub>2</sub>O<sub>3</sub>*, *Nuclear Instruments and Methods in Physics Research B* **326** (2014) 121-125.

M.D. Rodriguez, W.X. Li, F. Chen, C. Trautmann, T. Bierschenk, B. Afra, D. Schauries, R.C. Ewing, S.T. Mudie, P. Kluth, *SAXS and TEM investigation of ion tracks in neodymium-doped yttrium aluminum garnet*, *Nuclear Instruments and Methods in Physics Research B* **326** (2014) 150-153.

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W.X. Li, P. Kluth, D. Schauries, M.D. Rodriguez, M. Lang, F.X. Zhang, M. Zdorovets, C. Trautmann, and R.C. Ewing, *Effect of orientation on ion track formation in apatite and zircon*, *American Mineralogist* **99** (2014) 1127-1132.

- B. Afra, M. Lang, T. Bierschenk, M.D. Rodriguez, W.J. Weber, C. Trautmann, R.C. Ewing, *Annealing behaviour of ion tracks in olivine, apatite and britholite*, Nuclear Instruments and Methods in Physics Research B **326** (2014) 126-130.
- D. Schauries, B. Afra, T. Bierschenk, M. Lang, M.D. Rodriguez, C. Trautmann, W.X. Li, R.C. Ewing, P. Kluth, *The shape of ion tracks in natural apatite*, Nuclear Instruments and Methods in Physics Research B **326** (2014) 117-120.
- D. Schauries, M. Lang, O.H. Pakarinen, S. Botis, B. Afra, M.D. Rodriguez, F. Djurabekova, K. Nordlund, D. Severin, M. Bender, W.X. Li, C. Trautmann, R.C. Ewing, N. Kirby, and P. Kluth, *Temperature dependence of ion track formation in quartz and apatite*, Journal of Applied Crystallography **46** (2013) 1-6.
- W.X. Li, M.D. Rodriguez, P. Kluth, M. Lang, N. Medvedev, M. Sorokin, J.M. Zhang, B. Afra, M. Bender, D. Severin, C. Trautmann, and R.C. Ewing, *Effect of doping on the radiation response of conductive Nb-SrTiO<sub>3</sub>*, Nuclear Instruments and Methods in Physics Research B **302** (2013) 40-47.
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- H.Y. Gou, Z.P. Li, H. Niu, F.M. Gao, J.W. Zhang, R.C. Ewing, and J. Lian, *Unusual rigidity and ideal strength of CrB<sub>4</sub> and MnB<sub>4</sub>*, Applied Physics Letters **100** (2012) 111907.
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- W.X. Li, Y.B. Chen, J.M. Zhang, L.M. Wang, and R.C. Ewing, *Controlling the Structure and Size of Au Nanocrystals by Annealing and Ion Sputtering*, Langmuir **28** (2012) 51-55.
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J.W. Wang, Z. Rak, F.X. Zhang, R.C. Ewing, U. Becker, *Electronic structure and energetics of tetragonal SrCuO<sub>2</sub> and its high-pressure superstructure phase*, Journal of Physics-Condensed Matter **23** (2011) 465503.

B. Afra, M. Lang, M.D. Rodriguez, J.M. Zhang, R. Giulian, N. Kirby, R.C. Ewing, C. Trautmann, L. Chadderton, M. Toulemonde, and P. Kluth, *Annealing kinetics of latent particle tracks in Durango apatite*, Physical Review B **83** (2011) 064116.

W.X. Li, L.M. Wang, M. Lang, C. Trautmann, R.C. Ewing, *Thermal annealing mechanisms of latent fission tracks: Apatite vs. zircon*, Earth and Planetary Science Letters **302** (2011) 227-235.

F.X. Zhang, J.W. Wang, M. Lang, J.M. Zhang, and R.C. Ewing, *Pressure-induced structural transformations in lanthanide titanates La<sub>2</sub>TiO<sub>5</sub> and Nd<sub>2</sub>TiO<sub>5</sub>*, Journal of Solid State Chemistry **183** (2010) 2636-2643.

W.X. Li, L.M. Wang, K. Sun, M. Lang, C. Trautmann, R.C. Ewing, *Porous fission fragment tracks in fluorapatite*, Physical Review B **82** (2010) 144109.

## **Keynote or Invited Presentations:**

### **R.C. Ewing**

“Pyrochlore & Plutonium: The Elegant Response of a Simple Structure to Extreme Conditions,” University of Illinois at Chicago, March 4, 2010.

“Pyrochlore & Plutonium: The Elegant Response of a Simple Structure to Extreme Conditions,” invited seminar at University of Texas, El Paso, April 22, 2010.

“Response of Solids to Extreme Conditions: Coupling High-Pressure Cells with Large Accelerators”: Invited Seminar, Bhabha Atomic Research Centre, Mumbai, India, February 28, 2011.

“Fluorite Structure & Derivatives”: Workshop on *Thermodynamics of Actinides Materials: Theory and Experiment*, Notre Dame University, September 25, 2011.

“Response of Simple, Model Systems to Extreme Conditions”: invited lecture, Mechanical Behavior and Radiation Effects Contractors Meeting, Basic Energy Sciences, Potomac, Maryland, March 26<sup>th</sup>, 2013.

“The Effect of Nuclear Radiation on the Structure of Zircon”: invited for the symposium in honor of Heinrich Holland at the Goldschmidt Conference, Florence, Italy, August 27, 2013.

“Nuclear Materials under Extreme Condition”: keynote in the symposium on nuclear materials at the Goldschmidt Conference, Florence, Italy, August 29, 2013.

“Extreme Conditions: Combining energetic ion beams with high pressure cells”: invited lecture in session in honor of Frank Hawthorne, Roebling Medalist, 125<sup>th</sup> Annual Meeting of the Geological Society of America, Denver, CO, October, 27, 2013.

- “Response of Solids to Extreme Conditions: Coupling High-Pressure Cells with Large Ion Accelerators”: Earth & Environmental Science Seminar Series, Wayne State University, Detroit, MI, December 4, 2013.
- “The Simplicity and Complexity of the Fluorite Structure” invited lecture at the Workshop on Reducing Uncertainty in Performance Prediction (REDUPP) sponsored by EURATOM-7 and the European Community, Stockholm, Sweden, February 18, 2014.
- “Response of Actinide-Bearing Materials to Highly Ionizing Irradiation”: invited presentation on the occasion of the 50<sup>th</sup> Anniversary of the Nuclear Division of the American Chemical Society, Denver, CO, March 25<sup>th</sup>, 2015.
- “Projecting Risk into the Future:” seminar in the Nuclear Engineering Department at the University of Tennessee, Knoxville, TN, April 1, 2015.
- “The Future of Nuclear Energy:” panelist at symposium sponsored by the Yale Climate & Energy Institute, New Haven, CN, April 24, 2015.

### **Maik Lang**

- “Structural modifications in  $A_2B_2O_7$  pyrochlore induced by swift heavy ions with and without pressure”: invited planetary at the Ionenstrahlphysik workshop, Jena, Germany, April 2009.
- “Review of  $A_2B_2O_7$  pyrochlore response to irradiation and pressure”: invited planetary at the 15<sup>th</sup> International Conference on Radiation Effects in Insulators, Padua, Italy, Sept. 2009.
- “Combined high pressure and heavy-ion irradiation: a novel approach”: invited seminar talk at the National Synchrotron Light Source Friday seminar, Brookhaven, NY, USA, March 2009.
- “Response of complex oxides to extreme conditions of irradiation and pressure”: invited talk at the Materials Research Society Spring Meeting, San Francisco, CA, USA, April 2010.
- “Structural modifications in  $A_2B_2O_7$  pyrochlore induced by swift heavy ions with and without pressure”: talk at the Minerals, Metals & Materials Society TMS annual meeting, San Francisco, CA, USA, Feb. 2009.
- “Swift heavy ion induced crystalline-crystalline transformations in rare earth sesquioxides”: talk at the 17<sup>th</sup> International Conference on Radiation Effects in Insulators, Helsinki, Finland, July 2013.

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