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# **Electronic Transitions in f-electron Metals at High Pressures: New Experimental and Theoretical Capabilities critical to Stockpile and Basic Science**

(LDRD Project Tracking Code: **04-ERD-020**)

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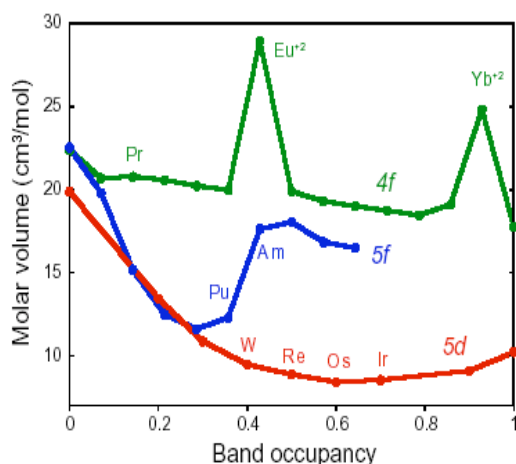
## **Abstract**

This study was to investigate unusual phase transitions driven by electron correlation effects that occur in many f-band transition metals and are often accompanied by large volume changes:  $\sim 20\%$  at the  $\delta$ - $\alpha$  transition in Pu and 5–15% for analogous transitions in Ce, Pr, and Gd. The exact nature of these transitions has not been well understood, including the short-range correlation effects themselves, their relation to long-range crystalline order, the possible existence of remnants of the transitions in the liquid, the role of magnetic moments and order, the critical behavior, and dynamics of the transitions, among other issues. Many of these questions represent forefront physics challenges central to Stockpile materials and are also important in understanding the high-pressure behavior of other *f*- and *d*-band transition metal compounds including 3d-magnetic transition monoxide (TMO, TM=Mn, Fe, Co, Ni).

The overarching goal of this study was, therefore, to understand the relationships between crystal structure and electronic structure of transition metals at high pressures, by using the nation's brightest third-generation synchrotron x-ray at the Advanced Photon Source (APS). Significant progresses have been made, including new discoveries of the Mott transition in MnO at 105 GPa and Kondo-like 4f-electron dehybridization and new developments of high-pressure resonance inelastic x-ray spectroscopy and x-ray emission spectroscopy. These scientific discoveries and technology developments provide new insights and enabling tools to understand scientific challenges in stockpile materials. The project has broader impacts in training two SEGRF graduate students and developing an university collaboration (funded through SSAAP).

## Scientific Background

The  $f$ -electron metals, actinides and lanthanides alike, exhibit a profound change in character, both for individual metals as a function of compression, or across the series as a whole at ambient pressure. It is believed that this behavior is driven by strong  $5f$ -electron correlation [1]. As we proceed through the series of the actinides, for example, two distinct parts of the series are discovered as illustrated in Fig. 1. The early part, from Th-Pu, shows a parabolic behavior of the equilibrium molar volume reminiscent of the  $d$ -transition metals, whereas the later part shows a more constant behavior of the specific volume as a function of atomic number. The similarity between the light actinides and the  $d$ -transition metals in this regard is governed by the fact that the light actinides and the  $d$ -transition metals both have delocalized electrons,  $f$  and  $d$  respectively. For the heavier actinides from americium and on, however, the  $5f$  electrons are localized similar to the lanthanide series and their properties become quite different from that of the earlier actinides. The itinerant  $5f$  electrons also show a distinguished difference compared to the  $4d$  and  $5d$  electrons; that is, their bandwidths are much narrower and in turn this leads to distortions in crystal structures of the light actinides and to exotic structures not found elsewhere in the periodic table [2]. At high compression the heavier actinides are also expected to attain distorted crystal structures because of pressure-induced delocalization of  $5f$  electrons.



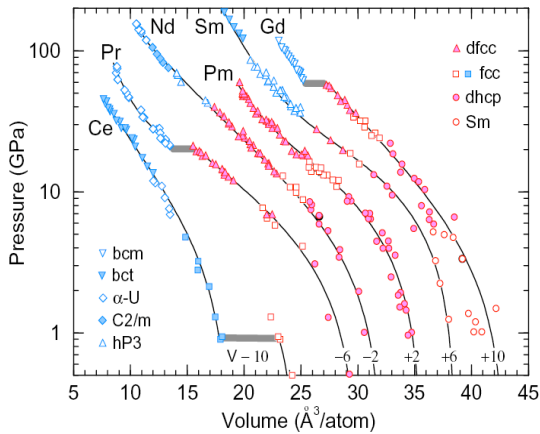
**Fig. 1.** Specific volumes of  $f$ - and  $d$ -electron transition metals plotted at their band occupancy. The actinides show a dramatic change in character between the  $d$ -band-like early actinides and the rare-earth-like late actinides. Clearly, divalent  $Eu$  and  $Yb$  are the exceptions to other trivalent rare-earth metals.

Plutonium, at the central position of the two trends, exhibits a dramatic effect with a subtle change in lattice induced by pressure, temperature and impurities. For example, Pu at ambient conditions crystallizes into low symmetry monoclinic  $\alpha$ -Pu with 16 atoms per unit cell suggesting itinerant electrons participating in bonding, whereas at slightly elevated temperatures it undergoes a series of phase transitions to high symmetry  $fcc$   $\delta$ -Pu with localized  $5f$  electrons. Because of this fundamental difference in  $5f$ -electron correlation/bonding, these two phases of  $\alpha$ - and  $\delta$ -Pu have very different thermodynamic, mechanical and chemical properties including a huge difference in density near 20 %. Such a large volume collapse in the  $\delta$ - $\alpha$  transition, of course, has a critical implication for accurate determination of the pressure-temperature trajectory of imploding Pu in nuclear stockpiles. The electron correlation also results in a very complicated phase diagram with seven polymorphs at the ambient pressure alone and several more at elevated pressures and in a highly unusual minimum in the melt curve. Because of the electronic nature of the large volume change between  $\alpha$  and  $\delta$  phases, it is conceivable that a remnant of the transition may persist into the liquid as an equation of state anomaly.

Analogous to the  $\delta$ - $\alpha$  phase transition of Pu, rare-earth metals also exhibit unusual electronic phase transitions at high pressures. Figure 2 summarizes the pressure-induced structural phase transitions in the early trivalent rare-earth metals, from high-symmetry closed-packed structures (colored in red) at low pressures to low-symmetry complex structures (in blue) at high pressures. Similarly, these transitions are considered to occur as a result of the pressure-induced  $4f$ -valence electron delocalization, but its exact

nature is not well understood. For example, some of these transitions are accompanied with large volume collapse (for example, 15 % in Ce at 1 GPa, 10 % in Pr at 20 GPa and 5 % in Gd at 60 GPa), yet the others like Nd and Sm transform to itinerant phases without any apparent discontinuity in volume. No collapse has been found in the late series of rare-earth metals. It is not known if there exist similar volume collapse transitions in Nd and Sm at low temperatures or at higher pressures than we have studied to date. Neither is known for the late trivalent rare earths. No theory has yet been able to convincingly explain the existence (or absence) of such a volume collapse. In fact, little is known about the exact nature of short-range electron correlation itself, its relation to long-range crystalline order, the possible existence of remnants of the electronic transitions in liquids, the role of magnetic moments and order, the critical behavior, and dynamics of the transitions.

The dramatic changes in behavior just described reflect equally significant changes in the underlying electronic structure of these correlated  $f$ -electron metals, so that direct examination of the electronic structure by various spectroscopic methods can provide critical insight into the nature of the transitions. Theoretical expectations are that the  $f$ -electron density of states should be “Hubbard split” by  $\sim 6$  and  $\sim 4$  eV for the rare-earths and actinides respectively, whereas in the itinerant phases this splitting is absent and the  $f$ -density of states should be a contiguous structure overlapping the Fermi level. It is an issue of current contention as to whether the itinerant phases do or do not also have some remnant Hubbard splitting in addition to the state density at the Fermi level. A variety of spectroscopic methods such as resonant inelastic x-ray scattering, x-ray absorption spectroscopy, x-ray emission spectroscopy, and x-ray magnetic circular dichroism can be used to probe the electronic structure, requiring in some cases a Be gasket if the incident photons might otherwise be absorbed in the diamond. Combining the data of these experiments one can characterize both the existence and the origin of magnetic moments, which could provide an explanation relating the large-volume transitions to screening out of the  $f$ -electron magnetic moments in the itinerant phases.



**Fig. 2.** Unusual electronic phase transitions of the early rare-earth metals from high-symmetry closed-packed structures of correlated phases (in red symbols) at low pressures to low-symmetry complex structures of itinerant phases (in blue) at high pressures. Note that some but not all phase transitions are accompanied with large volume collapse.

## Research activities

The objective of this study has been to understand the interplay between long-range crystal orders and short-range electron correlations in  $f$ - and  $d$ -transition metals and their compounds at high pressures and temperatures. To accomplish this objective, we have determined several key properties governing the volume collapse transitions that include magnetic moment, electronic density of state, and crystal structures, by using third-generation synchrotron x-ray at the APS and recent emerging diamond anvil cell technologies. Key progresses made in this project are summarized as the following:

1. Volume collapse transitions were determined for rare-earth metals Pr, Nd, Eu and Gd and transition metal compounds, EuN and MnO, all to 100 GPa and above, by using in-situ angle-resolved x-ray diffraction. These data are generally consistent with the previously suggested systematic phase transitions in rare-earth metals: that is, hcp  $\rightarrow$  Sm-type  $\rightarrow$  dhcp  $\rightarrow$  dist-fcc  $\rightarrow$  bcm. However, these new data also suggest that there are subtle differences in the crystal structures of localized Sm-type phase and itinerant bcm phase, in particular.
2. For the first time, we were able to obtain low energy f-electronic states of Gd in diamond anvil cells to 110 GPa by using resonance inelastic x-ray scattering (RIXS). These data clearly show the evidence for the mixed valence electronic state in Gd,  $\alpha|f^7\rangle + \beta|f^8\rangle$ , of which weight rapidly increases with increasing pressures. These results have been published by Maddox et al., in *Phys. Rev. Lett.* 96, 215701 (2006) and also in his **Ph.D. thesis** (2006, UCD).
3. Magnetic moments were determined by using high-resolution X-ray emission spectroscopy (XES) to
  - discover a complete loss of magnetic moment across the volume collapse transition in MnO at 110 GPa. These results have recently been published by Yoo et al., in *Phys. Rev. Lett.* 94, 115502 (2005).
  - Examine local f-moment changes in Gd and Eu across their volume collapse transitions. Surprisingly, these data show that f-moment in Gd does not go away even at 110 GPa. This unexpected result may suggest a possibility of different mechanism governing the large volume collapse in heavy rare-earth metals.
4. In parallel, in-depth theoretical understandings have been made for the volume collapse transitions in light rare-earth metals (Ce, Pr, and Nd), by using combined local density and dynamic mean field theory. The main results were recently published by McMahan et al., in *Phys. Rev. B.* (2005).

## Major accomplishments

This project results in major accomplishments in both science and technology areas, much of which appear in the peer-reviewed papers and a Ph. D. thesis by Maddox (2006, UCD), as listed in **Appendix**. as briefly described below. In this section, we describe the research results supporting the Mott transition in MnO and the Kondo-like electron delocalization in Gd- two major schools of thought used to explain highly unusual electron correlation-driven phase transitions occurring at high pressures.

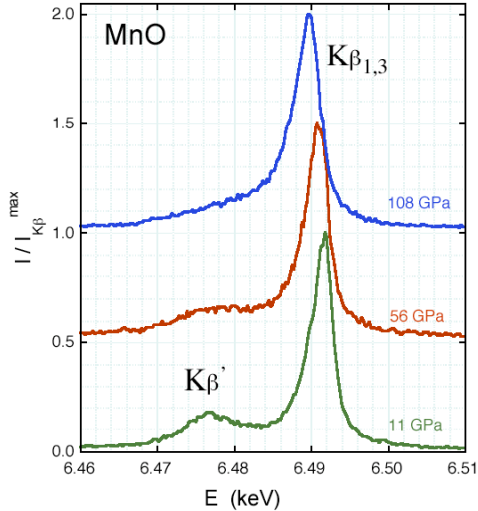
### A. The Mott insulator-metal transition of MnO at 110 GPa:

Mott's seminal work on how insulating character may arise out of the electron-electron repulsion used a 3d transition metal monoxide as an example and suggested its pressure-induced metallization, the Mott transition [3]. Yet for 55 years the Mott transition in these archetypal Mott insulators MnO, FeO, CoO, and NiO has eluded detection at room temperature due to the high pressures required. A recent resistance measurement [4] using "designer" diamond anvils, however, has seen this insulator-metal transition in MnO near 100 GPa, a pressure regime approaching conditions of the Earth's outer core. In this study [5], we obtained x-ray diffraction and x-ray emission spectroscopy data which combined with this resistance measurement provides a coherent picture of the Mott transition in MnO at a pressure of  $105 \pm 5$  GPa as the concurrence of three signatures: (1) a significant loss of magnetic moment, (2) a first-order isostructural phase transition with a  $\sim 6.6$  % volume collapse, and (3) the onset of metallic conductivity. These results exhibit profound similarities to transitions in the lanthanides and actinides as

has been predicted [6]. and should further our general understanding of electron-correlation driven phase transitions.

Figure 3 illustrates the pressure-induced changes in  $K\beta$  emission spectra of MnO, which can be classified into three groups depending on pressure: the first group below 30 GPa with the most dominate  $K\beta'$  intensity (bottom), the second between 40 and 98 GPa where the  $K\beta'$  intensity is substantially reduced yet apparent (middle), and the third above 108 GPa where the  $K\beta'$  lines nearly disappears (top). It is well known in many magnetic  $3d$  transition metal oxides that the intensity the  $K\beta'$  intensity is sensitive to local magnetic moment. Therefore, the complete loss of the  $K\beta'$  intensity above 108 GPa indicates a complete loss of magnetic moment in MnO. A small ( $\sim 1$  eV) shift of the  $K\beta_{1,3}$  band is also evident as the pressure increases from one to the other group. It is important to note that the pressure-induced spectral changes are subtle but occur abruptly at around 30 GPa and 108 GPa.

In our x-ray diffraction studies, we have further found the structural phase transitions from *cubic*- $B_1$  (*NaCl*-like) to *rhombohedral*-distorted- $B_1$  at 30 GPa, to a mixture of distorted- $B_1$  and *hexagonal*- $B_8$  (*NiAs*) at 90 GPa, and finally to isostructural  $B_8$  at 110 GPa. The isostructural phase transition at 110 GPa is based on the discontinuous changes in both specific volumes and  $c/a$  ratio. The remnant of distorted- $B_1$  phase, on the other hand, was observed well above 90 GPa to about 120-130 GPa. In contrast, the previous diffraction study suggested an “intermediate” phase in the pressure region between 90 and 120 GPa, without the characterization of crystal structure [7]. These pressure-induced structural/spectral changes of MnO observed in the present study, therefore, provide a coherent picture, suggesting the phase transitions from paramagnetic  $B_1$  to antiferromagnetic dist- $B_1$  at 30 GPa, to paramagnetic  $B_8$  at 90 GPa, and to diamagnetic  $B_8$  at 110 GPa. The 110 GPa transition is accompanied with a complete loss of magnetic moment,  $\sim 7\%$  drop in specific volume and a visual change to metallic luster.



**Figure 3.** X-ray emission spectra of MnO in the  $K\beta$  region obtained from paramagnetic  $B_1$  phase at 11 GPa, antiferromagnetic distorted- $B_1$  phase at 56 GPa, and diamagnetic  $B_8$  phase at 108 GPa. The  $K\beta$  emission represents the core-hole decay from  $1s \rightarrow 3p$  in  $Mn^{+2}$ , which splits into two branches,  $K\beta_{1,3}$  ( $^7P$ ) and  $K\beta'$  ( $^5P$ ), as a result of  $3p$ - $3d$  electron exchange intereaction. For details, see Ref [5].

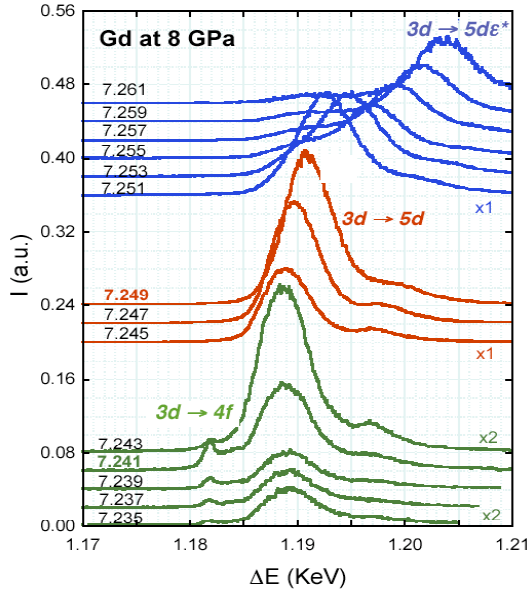
### B. The $f$ -band electronic structure of Gd at high pressures:

The goal of this study is to develop an experimental technique to probe the  $f$ -band electronic structure in  $4f$  rare-earth metals at high pressures. The most relevant information to electron correlation effects is contained in the M band (corresponding to the  $3d \rightarrow 4f$  transition in rare-earths) at approximately 1 KeV. However, high-pressure investigation of such a low energy transition is not feasible by using a conventional x-ray absorption technique, because of strong x-ray absorption by diamond and/or metal

gasket. In fact, such low energy x-ray does not even penetrate through the bulk of *f*-electron metal samples and, thus, the studies have been remained largely on surface at ambient conditions.

In this study, utilizing an intense third-generation synchrotron x-ray and resonance inelastic x-ray scattering (RIXS), we have been able to resolve the low-lying x-ray band of Gd, for the first time, at high pressures (see Fig. 4). We used a high-energy ( $\sim 7$  KeV) incident x-ray beam near the  $L_{III}$  edge ( $2p \rightarrow 5d$  or quadrupole allowed  $2p \rightarrow 4f$  transitions) through an x-ray transparent Be gasket and measured inelastically scattered x-ray photons at the resonance with the  $3d \rightarrow 4f$  transition. Figure 4 shows the  $3d \rightarrow 4f$  resonance at 7.241 keV nicely resolved from the  $3d \rightarrow 5d$  transition at 7.247 keV. Note that the dipole forbidden band can be observed in this way. More importantly, these bands are relatively sharp, because their band widths are dependent on life-time of the final state  $3d$  core-hole, which is substantially longer, than that of the  $2p$ . The spectral details, on the other hand, provide the fundamental information of crystal field splitting, spin-orbit coupling, and electron-electron exchange interaction and, thus, can be compared with theoretical results of electronic structure calculations. The importance of such data is paramount to evaluate different mechanisms, Mott transition vs. Kondo volume collapse, presently proposed to explain these unusual electronic transitions in *f*-metals [1].

Our RIXS and XES results clearly indicated Kondo-like aspects in the delocalization of  $4f$  electrons in Gd metal to 113 GPa [8]. The RIXS data obtained to 110 GPa have shown a prolonged and continuous process throughout the entire pressure range, so that the volume collapse transition at 59 GPa is only part of the delocalization phenomenon. Moreover, the  $L_{\gamma_1}$  XES spectra indicate no apparent change in the bare  $4f$  moment across the collapse, suggesting that Kondo screening is responsible for the expected Pauli-like behavior in magnetic susceptibility.



**Figure 4.** Resonance Inelastic X-ray Scattering (RIXS) of Gd at 8 GPa, plotted as a function of energy transfer  $\Delta E$  between the incident x-ray energy  $\Omega$  (marked on each spectra) and the fluorescence x-ray fluorescence  $\omega$ . At each incident x-ray energy in the vicinity of  $L_{III}$  edge (corresponding to quadrupole allowed  $2p \rightarrow 4f$  transition at 7.241 keV and dipole allowed  $2p \rightarrow 5d$  at 7.247 keV) of Ga, the inelastically scattered x-ray photons were scanned in the vicinity of the  $L\alpha$  emission line (corresponding to  $2p \rightarrow 3d$  core-hole decay at around 6.05 KeV). For detail, see Ref [8].

## Impacts

The most important impact of the proposed study has been on science and technology, resulting in significant advances of our current understandings in the forefront scientific area of condensed matter physics; namely, the electron correlation effect in transition metals and transition metal compounds at high pressures. The study results in fundamental data to challenge and validate theoretical understanding of the electronic structure and interatomic forces in *f*-electron metals as well as in new developments of



cutting-edge experimental and theoretical tools needed for Laboratory programs. This study has resulted in many high-profile scientific publications and attracted considerable academic interests (see Appendix). In fact, this LDRD study has produced two SEGRF graduate students: Brian Maddox, now working at the NIF as a PD, and Amy Lazicki in her final 4<sup>th</sup> year, both from UCD. This LDRD study has also been of strong interest to DOE/NNSA-DP, leveraging highly productive collaborations with leading scientist in the Stewardship Science Academic Alliances Program (at UCD and CDAC, Carnegie-DOE Alliance Center).

## Acknowledgement

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8. B. Maddox *et al.*, Kondo-like 4f Delocalization in Gd at High Pressures, **Phys. Rev. Lett.** 96, 215701 (2006)

## **Appendix: Summary for technical activities of this LDRD project (04-ERD-020):**

### **Ph.D. Thesis**

Brian Maddox, Pressure-Induced Electronic Phase Transitions in Transition Metal Oxides and Rare Earth Metals, UC-Davis (2006).

### **Published in peer-reviewed journals:**

- A. Lazicki, C. S. Yoo, H. Cynn, W. J. Evans, W. E. Pickett, J. N. Olamit, Kai Liu and Y. Ohishi, 'Search for superconductivity in LiBC at high pressure, **Phys. Rev. B** (2007) in print
- V. Iota, J-H. Park, C-S. Yoo, J. Lang, D. Haskel, G. Srajer. Electronic Structure and Magnetism in Highly Compressed 3d-Transition Metals, **Appl. Phys. Lett.** (2007) in print
- B.R. Maddox, A. Lazicki, C. S. Yoo, V. Iota, M. Chen, A.K. McMahan, M.Y. Hu, P. Chow, R.T. Scalettar, and W.E. Pickett, Kondo-like 4f Delocalization in Gd at High Pressures, **Phys. Rev. Lett.** 96, 215701 (2006)
- A. K. Lazicki, C. S. Yoo, W.J. Evans, W.E. Pickett, and R.T. Scalettar, Pressure-Induced Antifluorite-to-Anticotunnite Phase Transition in Lithium Oxide, **Phys. Rev. B** 73, 184120 (2006)
- D. Kasinathan, J. Kunes, A. Lazicki, H. Rosner, C.-S. Yoo, R.T. Scalettar, and W. E. Pickett, Singular Kohn Anomoly Surfaces and Superconductivity in Compressed Lithium, **Phys. Rev. Lett.** 96, 47004 (2006)
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- C.S. Yoo, B. Maddox, J-H. P. Klepeis, V. Iota, W. Evans, A. McMahan, M. Hu, P. Chow, M. Somayazulu, D. Hausermann, R. Scalettar, W. Pickett, First-Order Isostructural Mott Transition in MnO, **Phys. Rev. Lett.** 94, 115502 (2005).
- McMahan, Combined local-density and dynamic mean field theory calculations for the compressed lanthanides Ce, Pr, and Nd, *Phys. Rev. B.* (2005), in print

### **Invited talks:**

- Yoo, High-pressure materials research using DAC and Synchrotron x-ray, a plenary talk at Fifth International Conference on Synchrotron Radiation in Material Science (SRMS-5), July 30-Aug. 2, 2006, Chicago, IL60611
- Yoo, Correlation driven phase transitions in transition metals at high pressures, presented at the workshop on Inelastic X-ray Scattering, Advanced Photon Source, Argonne National Laboratory, Argonne, IL, May 3-5, 2006
- Yoo, Electronic phase transitions in transition metals at high pressures, SEMC 2005, April 17-21, 2005, Miami, Florida

Yoo, LLNL activities at the HPCAT/APS, presented at the sector 16 review, APS/ANL, Argonne, IL, March 3<sup>rd</sup>, 2005

Yoo, High pressure stockpile materials research: DAC studies with advanced x-ray and laser, presented at the DOE-BES/NNSA Joint meeting, Washington D. C., Feb. 18, 2005

Yoo, LLNL's high pressure science with advanced x-ray synchrotron source, presented at the Materials R&D for national security at DOE user facility, Los Alamos National Laboratory, Los Alamos, NM, Feb. 23-24, 2005

McMahan, Correlation effects in the compressed rare earth metals, APS march meeting, March 21-25, 2005, Los Angeles, CA

Yoo, Mott transition in compressed MnO, AIRAPT meeting, Carshlru, Germany, June 27- July 1, 2005

Yoo, Pu  $\delta\alpha$ -like electronic phase transitions in MnO and Gd, PDRP seminar, LLNL, June 9<sup>th</sup>, 2005