

REPORT DOCUMENTATION PAGE			Form Approved OMB NO. 0704-0188		
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1. REPORT DATE (DD-MM-YYYY) 23-12-2015		2. REPORT TYPE Final Report		3. DATES COVERED (From - To) 18-Aug-2014 - 17-May-2015	
4. TITLE AND SUBTITLE Final Report: Unusual Phase Transformations in Ceramics and Related Materials Under the Influence of an Electric Field				5a. CONTRACT NUMBER W911NF-14-1-0569	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER 611102	
6. AUTHORS Rishi Raj				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAMES AND ADDRESSES University of Colorado - Boulder 3100 Marine Street, Room 481 572 UCB Boulder, CO 80303 -1058				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES) U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211				10. SPONSOR/MONITOR'S ACRONYM(S) ARO	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S) 65840-MS-II.3	
12. DISTRIBUTION AVAILABILITY STATEMENT Approved for Public Release; Distribution Unlimited					
13. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.					
14. ABSTRACT We have conducted in-situ X-ray diffraction experiments at the Advanced Photon Source at the Argonne National Laboratory to explore the occurrence of unusual phase transformation or changes in crystal structure that are induced by electrical fields. Two specific results have been obtained. In one case the (1 1 0) and the (1 1 1) peaks in titanium oxide strengthen, while the (2 1 1) peak weakens, under the electric field at elevated temperature. The changes in the peak intensities are immediate, but they fluctuate with time even though the electric field remains constant. These fluctuations are ascribed to a difference in the interaction of charged defects, which are induced by					
15. SUBJECT TERMS electric field, far from equilibrium, phase transformation					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UU	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON Rishi Raj
a. REPORT UU	b. ABSTRACT UU	c. THIS PAGE UU			19b. TELEPHONE NUMBER 303-492-1029

Report Title

Final Report: Unusual Phase Transformations in Ceramics and Related Materials Under the Influence of an Electric Field

ABSTRACT

We have conducted in-situ X-ray diffraction experiments at the Advanced Photon Source at the Argonne National Laboratory to explore the occurrence of unusual phase transformation or changes in crystal structure that are induced by electrical fields. Two specific results have been obtained. In one case the (1 1 0) and the (1 1 1) peaks in titanium oxide strengthen, while the (2 1 1) peak weakens, under the electric field at elevated temperature. The changes in the peak intensities are immediate, but they fluctuate with time even though the electric field remains constant. These fluctuations are ascribed to a difference in the interaction of charged defects, which are induced by the electric field, with different planes in rutile depending on the intrinsic charged state of the planes. In a second system the reaction between alumina and titania is shown to be remarkably enhanced by the electric field. Furthermore, aluminum-titanate is shown to form below the temperature given by the phase diagram, in support of the concept that electrical fields can produce phases that are far from equilibrium.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
12/23/2015	1 S.K. Jha, J.M. Lebrun, R. Raj. Phase transformation in the alumina–titania system during flash sintering experiments, Journal of the European Ceramic Society, (02 2016): 0. doi: 10.1016/j.jeurceramsoc.2015.10.006
12/23/2015	2 S.K. Jha, J.M. Lebrun, K.C. Seymour, W.M. Kriven, R. Raj. Electric field induced texture in titania during experiments related to flash sintering, Journal of the European Ceramic Society, (01 2016): 0. doi: 10.1016/j.jeurceramsoc.2015.09.002
TOTAL:	2

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
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TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Book

TOTAL:

Received

Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Elected as a Distinguished Life Member of the American Ceramic Society, 2015.

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Shikhar Jha	0.50	
FTE Equivalent:	0.50	
Total Number:	1	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
Kiran Naik	0.50
FTE Equivalent:	0.50
Total Number:	1

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	National Academy Member
Rishi Raj	0.05	
FTE Equivalent:	0.05	
Total Number:	1	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 0.00

Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Shikhar Jha

Total Number:

1

Names of other research staff

NAME

PERCENT SUPPORTED

FTE Equivalent:

Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

The objective of this seed grant was to investigate whether unusual phase transformations can occur in ceramics under electric fields at elevated temperature. These experiments have come to be known under the general name of "flash sintering". However, the purpose here was to see if phase changes can occur under similar circumstances in pre-sintered specimens. We have been able to demonstrate that indeed unusual phase changes take place that are far from equilibrium under flash conditions. In titanium oxide, the crystal structure as measured by the X-ray diffraction peaks during in-situ experiments at the APS synchrotron we see changes that appear and then vanish when the electric field is turned on and off. In the case of the alumina-titanium dioxide system, aluminum titanate is seen to form at temperatures below the equilibrium temperature given by the phase diagram. The reaction between alumina and titania to form the titanate spinel is accelerated several fold under the electric fields. These results demonstrate the promise of using electric fields to form materials that cannot be formed by conventional methods and phase equilibria.

Technology Transfer

Visited ARL in December 2014 to present a seminar and develop interactions. Host, Dr. Raymond Brennan.