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6. AUTHORS Ivar E. Reimanis		8. PERFORMING ORGANIZATION REPORT NUMBER	
7. PERFORMING ORGANIZATION NAMES AND ADDRESSES Colorado School of Mines 1500 Illinois St. Golden, CO 80401 -1890			
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Report Title

Final Report for "The Potential for a Novel Transformation-Toughened Composite"

ABSTRACT

Research was conducted to examine the possibility to design a novel transformation-toughened composite utilizing a phase transformation in beta-eucryptite. Specimens comprising zirconium oxide as the matrix and beta-eucryptite as the particulate phase were made with varying amounts of eucryptite particle size. Indentation fracture experiments were conducted and four-point bend notch bars were fabricated. The indentation experiments showed significant crack deflection, suggesting that toughening may occur. However, quantitative results (from the four-point bend bars) have not yet been achieved.

List of papers submitted or published that acknowledge ARO support during this reporting period. List the papers, including journal references, in the following categories:

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

I. E. Reimanis, C. Seick, K. Fitzpatrick, E. R. Fuller, and S. Landin, "Spontaneous Ejecta from b-Eucryptite Composites", in press in Journal of the American Ceramic Society (2007).

Number of Papers published in peer-reviewed journals: 1.00

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

Number of Papers published in non peer-reviewed journals: 0.00

(c) Presentations

Invited: Ivar E. Reimanis, "Spontaneous Ejecta in Low Thermal Expansion Ceramic Composites" Materials Department, Indian Institute of Science, Bangalore February 1, 2007

Invited: Edwin R. Fuller, Jr., Andrew T. Durnford, Thomas Wanner, Chris Seick, Ivar E. Reimanis, "Three-Dimensional Microstructure-Property Simulations for Low Thermal Expansion Ceramic Composites" Annual TMS Meeting, Orlando, FL, February 26 2007.

Invited: Ivar E. Reimanis, "Spontaneous Ejecta in Low Thermal Expansion Ceramic Composites" ARCI, Hyderabad, India, April 5, 2007.

C. Seick, K. Fitzpatrick, I. E. Reimanis and E. R. Fuller, Jr., "Spontaneous Ejecta in a Low Thermal Expansion Ceramic Composites" MS&T2006 Symposium: Deformation Mechanisms in Complex Materials, in Cincinnati, OH October 16 2006

Edwin R. Fuller, Jr. & Andrew T. Durnford, Chris Seick & Ivar E. Reimanis, Ravi Kappiyoor & Thomas Wanner, "Microstructure-Property Simulations for Low Thermal Expansion Ceramic Composites" MS&T2006 Symposium: Deformation Mechanisms in Complex Materials, in Cincinnati, OH October 16 2006

Chris Seick, Ivar E. Reimanis, James A. Valdez, Kurt Sickafus and Ming Ting, "Radiation Damage Effects in Beta-Eucryptite (?-LiAlSiO₄)" MS&T2006 Symposium: Irradiation Effects, in Cincinnati, OH October 18 2006

C. Seick*, K. Fitzpatrick, I. E. Reimanis, T. Wanner, E. R. Fuller, Mechanical Behavior of ?-Eucryptite Composites, 31st International Cocoa Beach Conference and Exhibition on Advanced Ceramics and Composites, Daytona Beach, FL January 25, 2007.

Number of Presentations: 7.00

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

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts): 0

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

(d) Manuscripts

Number of Manuscripts:0.00

Number of Inventions:

Graduate Students

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>	National Academy Member
Ivar Reimanis	0.10	No
FTE Equivalent:	0.10	
Total Number:	1	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>
Chris Seick	1.00
Kyle Fitzpatrick	1.00
Amanda Younessian	0.50
FTE Equivalent:	2.50
Total Number:	3

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: 2.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 2.00

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:..... 1.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale): 2.00

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

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Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PhDs

NAME

Total Number:

Names of other research staff

NAME

PERCENT SUPPORTED

FTE Equivalent:

Total Number:

Sub Contractors (DD882)

Inventions (DD882)

5 Implementing a Pressure-Induced Phase Transformation in Beta-Eucryptite to Impart Toughening

Patent Filed in US? (5d-1) Y

Patent Filed in Foreign Countries? (5d-2) N

Was the assignment forwarded to the contracting officer? (5e) N

Foreign Countries of application (5g-2):

5a: Ivar Reimanis

5f-1a: Colorado School of Mines

5f-c: 1500 Illinois Street

Golden CO 80401

5a: Chris Seick

5f-1a: Colorado School of Mines

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5a: Kyle Fitzpatrick

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Golden CO 80401

The Potential for a Novel Transformation Toughened Composite

PI: Ivar Reimanis, Colorado School of Mines

Statement of Problem Studied:

The objective of the study was to establish whether or not it is possible to use β -eucryptite to make a transformation toughened composite. The approach was to add particles of β -eucryptite to an inert ceramic matrix, press and sinter to high density, and conduct mechanical properties tests. The mechanical testing included Vickers indentation tests to qualitatively evaluate crack paths, and notched four point bend tests.

The concept was based on a recent observation that β -eucryptite composites exhibit a unique phenomenon in which particles are ejected from the surface when the material is indented with a sufficient load. A pressure-induced phase transformation was reported to occur in β -eucryptite [1,2], and it is hypothesized that the transformation is involved in causing particle ejecta. The present work seeks to utilize the transformation by promoting it during cracking, thereby toughening a ceramic.

Summary of the Most Important Results:

Progress was made to establish that a transformation is indeed involved when β -eucryptite composites are indented with any kind of indenter. This was written and published in one of the papers for this project.

Composites comprising β -eucryptite particles contained within a ZrO_2 matrix were made by mixing powders, and hot pressing. The β -eucryptite was provided by CoorsTek. It contained about 15 vol. % of a second phase, lithium aluminate (LiAl_5O_8), according to x-ray diffraction and SEM. Our efforts to produce pure β -eucryptite were not successful in the time period of this project. Three different particle sizes were used; these were made by grinding and sieving β -eucryptite. Densities achieved were relatively high, but specimens with the larger β -eucryptite particle sizes cracked extensively around the particles. SEM images of the indents and the associated cracks formed are shown in Figures 1 and 2 for pure ZrO_2 , and for one of the composites produced. It is apparent that significant crack deflection occurs when β -eucryptite particles are present in the ZrO_2 , and such deflection suggests toughening is operative. The measured crack length, a representation of the toughness, was not significantly different between the various specimens; applying the Anstis model for toughness, a toughness of $4.1 \text{ MPam}^{1/2}$ was determined for both specimens. However, because the cracking mechanism may be different in the two specimens (i.e., the model used to calculate toughness may be different), it is not appropriate to compare the quantitative results at this time.

The notched four-point bend bars have not been tested at the time of the writing of this report.

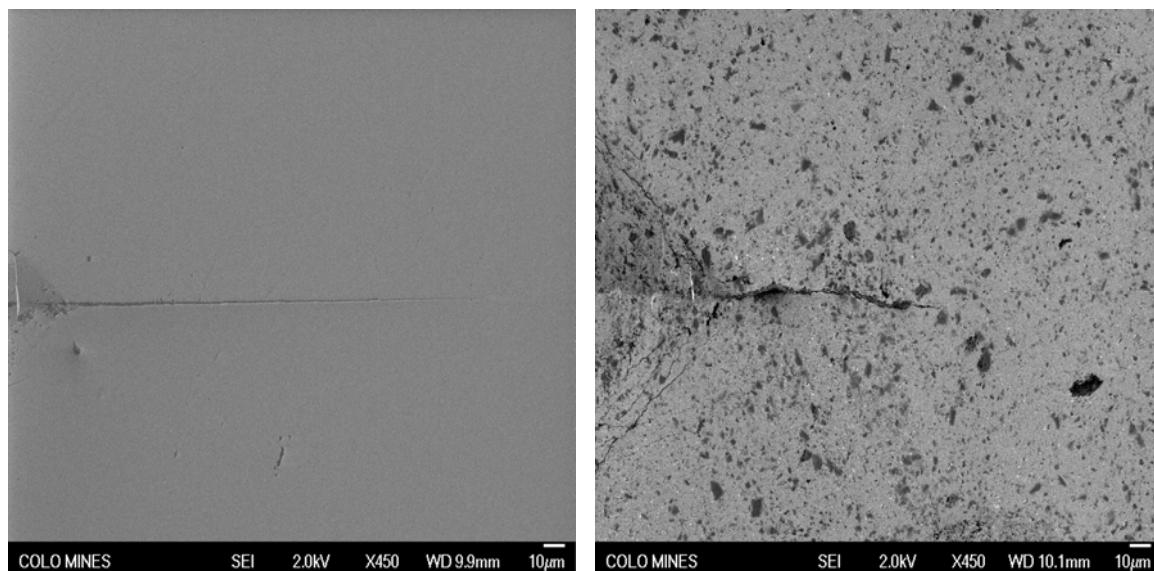


Figure 1. Pure ZrO_2 on left. Composites with 10 wt. % beta-eucryptite on right. Both figures show a crack emanating from the indent apparent in the left side of the figure. Significant crack deflection and tortuosity is apparent in the figure on the right, an indication of enhanced toughness.

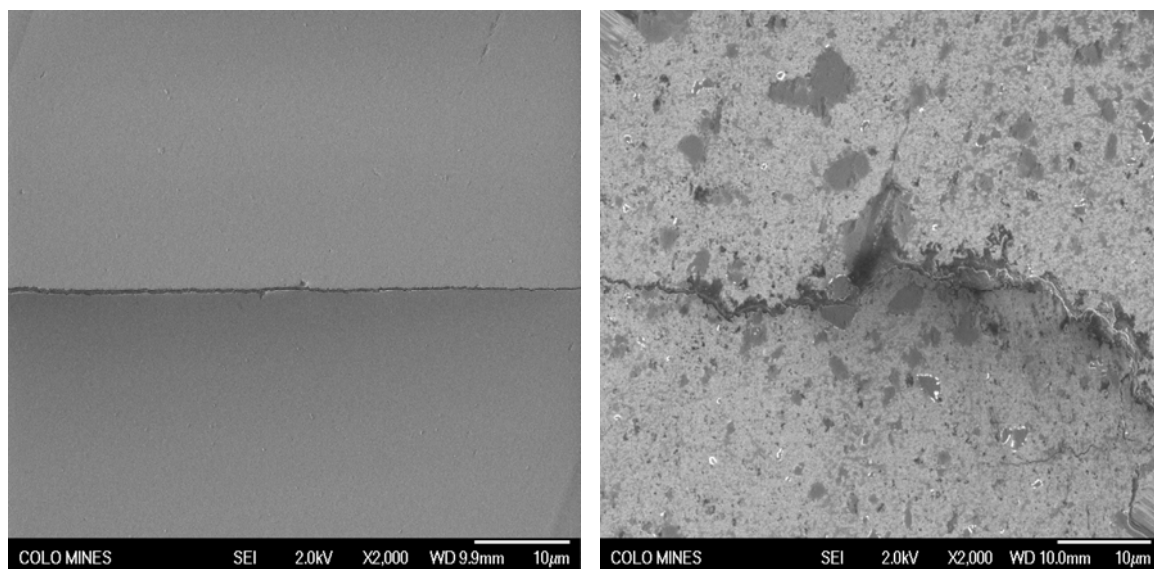


Figure 2. In the composite (right picture), the dark contrast represents the beta-eucryptite particles which are clearly interacting with the crack.

Bibliography

1. J. Zhang, A. Celestian, J. B. Parise, H. Xu and P. J. Heaney, "A New Polymorph of Eucryptite (LiAlSiO_4), ϵ -Eucryptite, and Thermal Expansion of α - and ϵ -Eucryptite at High Pressure" *Am. Mineralogist*, Vol. 87, 566-571 (2002).
2. J. Zhang, Y. Zhao, H. Xu, M. V. Zelinskas, L. Wang, Y. Wang and T. Uchida, "Pressured-Induced Amorphization and Phase Transformations in β - LiAlSiO_4 ", *Chemistry of Materials*, 17, 2817-2824 (2005).