Title

Student name, major; Mentor name, department

Background

A jet turbine blade is one of the most engineered materials on an airplane. During operation, turbine blades are exposed to extremely oxidizing conditions and high temperatures. The superalloy base layer of the blade melts between 1220˚C and 1330˚C, but the operating temperature of the blade can exceed 1600˚C [4]. To prevent melting of the base metal, a ceramic thermal barrier coating (TBC) is used to introduce a temperature drop of 400˚C to 500˚C across the 125-250 μm layer [5,6]. The current state of the art TBC is a zirconia material stabilized by 7 weight percent yttria (YSZ) [7]. The YSZ phase that is used in these turbines has a tetragonal structure typically referred to as tetragonal prime or supersaturated tetragonal; it is a thermodynamically metastable phase kinetically stabilized at the conditions seen in turbines [6]. Unfortunately, as turbines are in operation, air-borne calcium-magnesium alumino-silicate (CMAS) composites, a major component of sand and soil world-wide, enter into the turbine environment. The CMAS melts at the temperatures in question, sticks to the blade surface and thermochemically attacks the TBC. Eventually, this causes destabilization of the preferred tetragonal prime phase, which leads to a decrease in the temperature drop across the TBC and eventual compromise and necessary replacement of the turbine [1]. Up to this point, studies have been focused on the thermochemical mechanism of destabilization and its mitigation via alternative processing and formulations of YSZ [6]. However, all of these studies employ model CMAS with wide variations in composition [2]. This study is a systematic cross-comparison of the primary "model" CMAS compositions with each other and with "real" CMAS compositions, such as the chemical composition of Saudi Arabian desert sand and bituminous ash, and their role in influencing phase changes of the YSZ during heat treatment. Preliminary results after a 30 minute heat treatment at 1500˚C show phase changes in the YSZ as well as definitive clustering of the nineteen CMAS compositions under study. In the initial YSZ sample, both the tetragonal prime and monoclinic phases were present. After heat treatment, the monoclinic phase had disappeared, changing into tetragonal prime in contrast to literature reports.

Research Question

How does varying the composition of CMAS affect its interaction with a YSZ TBC at operating temperatures over time?

Project Objectives

1. To collaborate with the Art Department and build a furnace that can be used for high-temperature heat treatments
2. To distinguish YSZ phase evolution during high temperature treatments
3. To demonstrate the efficacy of visualization and clustering analysis tools to render complex data readily-interpretable
4. To perform a controlled-variable study on the effect of CMAS composition on tetragonal prime YSZ under elevated temperature conditions
5. To provide experimental framework and preliminary data for further studies into CMAS-resistant TBCs

Project Significance

Intense degradation of thermal barrier coatings (TBCs) by calcium-magnesium alumino-silicate (CMAS) occurs at high temperatures, such as those in a jet turbine. If a jet turbine blade fails in operation, the jet will crash. Research to improve TBCs has been a focal point in the field, but the effect of CMAS composition variation, which can influence reaction pathways and products, has not been thoroughly studied. Ultimately, this study will contribute to the body of knowledge on this important topic and influence further studies on CMAS-resistant TBC's.

Methodology

Sample Preparation

Nineteen sample CMAS compositions have been determined from the literature [2], simplified into five-component systems (the CMAS components and iron oxide), prepared from pure powders, and deposited on 19 pressed YSZ pellets in accordance with the literature [3].

Furnace Fabrication

A small kiln furnace will be constructed in collaboration with Professor Andrew Coombs of the Ceramics Program in the Art Department, who teaches a kiln design class. The furnace will be gas heated, chosen because it mimics the chemical environment of a gas-heated airfoil turbine. The heat treatments will be performed at 1500˚C, a temperature slightly higher than the 1100-1400˚C at which most studies are done but in the operation range of jet turbines [4,7,8]. This furnace will be designed, built, tested, and calibrated for its maximum temperature, temperature ramp rate and heating uniformity. This process will teach the basic concepts of furnace design.

Heat Treatment
One 30 minute heat treatment at 1500˚C with a 15˚/min cooling rate has already been accomplished in a furnace no longer available for use. After building the new furnace, the samples will be heat treated three additional times in total time increments of 3 hours, 6 hours, and 12 hours at 1500˚C. These times have been chosen to mimic short-time scale reactions occurring in turbines during operation. A sample stage will be constructed out of alumina to hold the samples in place in the furnace. Heat profiles over time during the anneals will be taken using the method determined during the fabrication stage.

Sample Characterization and Comparison
Initial X-ray diffraction (XRD) and Raman spectroscopy spectra of the pure oxides, the YSZ and the untreated CMAS-mixtures have been taken as basis spectra to facilitate future data interpretation. After each heat treatment, the samples will be investigated by XRD and Raman. In XRD, X-rays are directed toward a flat sample and diffracted from the crystallographic planes at angles characteristic of the structure (or phase) of the material under study. Raman spectroscopy is an inelastic scattering of monochromatic light, which provides information regarding the bonding environment of different chemical species. The rules determining Raman activity of a material are not as straightforward as XRD, but the positions of Raman peaks are attributable to particular structures and binding environments. XRD and Raman are used here to identify and track phase evolution in the samples. Phases will be identified by comparisons to literature references and established databases. Data will be displayed and compared within and across data sets using CombiView. CombiView is a data visualization and minimization program designed to handle large amounts of spectral data and use clustering analysis to sort the spectra into groups. A representative spectrum from each group to be compared to the literature will then be chosen, instead of analyzing each spectrum individually [9]. CombiView will be used to identify trends within the complex data sets and compare the identifying characteristics of each group to the composition of the applied CMAS. Comparisons will also be made between this project and a semi-analogous project being undertaken by a graduate student in the lab. Methods of analyzing complex spectra and presenting the analysis results will be taught.

Project Timeline

Task Description	Project Months (9 total)								
	Aug	Sep	Oct	Nov	Dec	Jan	Feb	March	April
Continue literature review and background research	X	X	X	X	X	X	X	X	X
Build furnace	X	X							
Heat treatments		X	X	X	X	X			
XRD and Raman data gathering			X	X	X	X	X		
Data analysis for phase changes, trends					X	X	X	X	
Prepare for paper, presentations, report, etc.							X	X	X

Anticipated Results and Dissemination
Expected experimental results include charting phase changes over the course of the reaction, in the YSZ as well as the final CMAS products, to determine if the reaction proceeds differently as a function of CMAS composition. It is anticipated that the results of this study will guide which model CMAS-systems will be used in future studies for accurate CMAS-TBC interactions. A manuscript with the student researcher as the primary author will be prepared with guidance of the research mentor and submitted for publication to journals such as Material Sciences and Engineering A**.** Presentations of the results will be given at Discover USC as well as at the American Institute of Chemical Engineers National Student Conference.

Personal Statement
This is currently my second semester working on this project in [mentor's] laboratory. I look forward to completing this project and contributing unique information to the general body of knowledge about YSZ thermal barrier coatings in an area that has not been well explored. This project is nested in his idea for a larger project over the course of the next several years in identifying novel CMAS resistant TBC's through combinatorial methods, and I am excited to be a part of that. I am also thrilled to be collaborating with the art department and building a kiln; I had no idea that partnerships such as these were possible in the course of academic research. My intention to pursue my PhD has been positively influenced by my experience in the lab. Conducting research is important in teaching me more about experimental design, analytical techniques, data processing and visualization, problem solving, and lateral thinking. A grant from the Magellan Scholar Program will allow me to purchase materials to build the furnace in a timely manner and be compensated for the time I spend on this project.

References

1. Wellman R, Whitman G, Nicholls J. CMAS corrosion of EB PVD TBCs: Identifying the minimum level to initiate damage. International Journal of Refractory Metals and Hard Materials 2010; 28:124-132.

2. Levi C, Hutchinson J, Vidal-Sétif M, et al. Environmental Degradation of TBCs by Molten Deposits. MRS Bulletin 2012; 37:932-941.

3. Gledhill A, Reddy K, Drexler J, et al. Mitigation of damage from molten fly ash to air-plasma-sprayed thermal barrier coatings. Materials Science and Engineering: A 2011; 528:7214-7221.

4. Besmann T. Interface Science of Thermal Barrier Coatings. Journal of Material Science 2009; 44:1661-1663.

5. Krämer S, Faulhaber S, Chambers M, et al. Mechanisms of cracking and delamination within thick thermal barrier systems in aero-engines subject to calcium-magnesium-alumino-silicate (CMAS) penetration. Materials Science and Engineering: A 2008; 490:26-35.

6. Levi C. Emerging materials and processes for thermal barrier systems. Current Opinion in Solid State and Material Science 2440; 8:77-91.

7. Aygun A, Vasiliev A, Padture N, et al. Novel thermal barrier coatings that are resistant to high-temperature attack by glassy deposits. Acta Materialia 2007; 55:6734-6745.

8. Borom M, Johnson C, Peluso L. Role of environmental deposits and operating surface temperature in spallation of air plasma sprayed thermal barrier coatings. Surface and Coatings Technology 1996; 87:116-126.

9. Long C, Hattrick-Simpers J, Murakami M. Rapid structural mapping of ternary metallic alloy systems using the combinatorial approach and cluster analysis. The Review of Scientific Instruments 2007; 78:072217.

Magellan Scholar BUDGET FORM

Student's Name:

Student salary	**Hours** Estimated number of hours student will work	**Rate** Enter the hourly wage	**Subtotal**
Research hours during semesters when enrolled in classes	240	$10.00	$2,400.00
Research hours during semesters when NOT enrolled in classes	0	$10.00	$0.00
Fringe: Student salary * student fringe rate (What is fringe? See budget instructions or guidebook)			
Enrolled in classes	$2,400.00	0.55%	$13.20
NOT enrolled in classes	$0.00	8.29%	$0.00
Materials/Supplies		Enter sub-total from below:	$238.87
Travel		Enter sub-total from below:	$715.00
		TOTAL:	**$3,367.07**
		Amount requested for MGS award:	**$3,000.00**

Budget Justification/Description

Student Salary: Indicate estimated number of student research hours per week and hourly rate separated by semesters when student is enrolled in classes or not enrolled in classes (generally fall or spring vs summer semesters).

Enrolled in classes: 11 hours per week for 12 weeks per semester for 2 semesters at $10 per hour.
Not enrolled in classes: 0 hours

The pay for approximately 200 of these hours will be covered by the Magellan Grant; the remainder will be covered by the research mentor.

Materials/Supplies: Indicate items, quantity, and estimated price. *Be sure to include taxes on all purchases.*

Item	Cost per unit ($)	Quantity	Cost ($)
Super Duty Bricks	7.06	15	105.90
Thermocouple	18.57	1	18.57
Alumina Plate	64.40	1	64.40
Poster Printing	50.00	1	50.00
		Total Cost:	$238.87

Travel: Indicate location, purpose of travel, estimate itemized costs (transportation, lodging, registration, etc).

American Institute of Chemical Engineers Annual National Student Conference – Salt Lake City, Utah

Airfare (roundtrip) - $395 (from Orbitz)
Lodging - $80/night at Salt Lake City Plaza Hotel for 3 nights - $240
Food - $20/day for 4 days - $80
Local transportation – airport shuttle provided by hotel, conference within walking distance - $0

Total: $715

Approximately $500 will be covered by the Magellan Grant. The student will seek departmental funds and pay out-of-pocket for the remainder of the cost.