Oxidation Informed Design of Entropy Stabilized Ultra-High Temperature Ceramics

1. Introduction

Ultra-high temperature ceramics (UHTCs), most notably refractory metal carbides, nitrides and borides, hold the key to advancement in hypersonic flight technology, which enables the safe and efficient execution of both manned and unmanned flight missions. UHTCs exhibit melting temperatures exceeding 3000°C, making them appropriate candidates to withstand the extreme temperatures experienced by some areas of the craft during hypersonic flight. However, their propensity to react rapidly with oxygen limits their sustained application.^{1, 2} Researchers have attempted to address this limitation through the modification of already well-understood systems. The addition of SiC, for example, to boride UHTCs, such as ZrB₂, improves corrosion resistance above 1600°C^{2–}⁴; however, this resistance is due to the formation of a liquid borosilicate glass layer, which shears away at the speeds encountered in hypersonic flight.

The thermodynamic stability of a material is determined by the minimization of its free energy, defined as

$$G = H - TS, \tag{1}$$

where *H* is enthalpy, *T* is temperature and *S* is entropy. Traditional materials engineering relies on the enthalpic contribution to achieve stability for a desired application. An alternative and promising approach, commonly applied to metal alloys⁵ and most recently oxides⁶, which explores the effect of entropy in phase stabilization, is still in its infancy. Based on the efforts of the metals community⁷, *we hypothesize that through entropy stabilization, we can engineer a UHTC that can withstand prolonged exposure to extreme temperatures in oxidizing environments.* Through a complementary computational and experimental framework, I will investigate possible equilibrium phases of entropy stabilized UHTCs (ES-UHTC) materials based on composition and understand their subsequent oxidation properties.

2. Plan of Work





Figure 1. Calculated Ellingham diagram showing the formation of the most thermodynamically stable oxides from the Group IV, V and VI refractory metals.

The refractory metals from Group IV, V and VI comprise the elemental palette of interest for the candidate ES-UHTC compositions. Oxide formation strongly depends on the relative thermodynamic stabilities of the constituent elements. Preliminary thermodynamic models were calculated using FactSage⁸, a free energy minimization software, coupled with the relevant oxide data. These calculations have been summarized in an Ellingham diagram, shown in Figure 1. Lines lower on the curve indicate a more thermodynamically stable oxidation reaction.



Figure 2. Diagram depicting the hypothesized effect of entropy stabilization on the thermodynamics of oxidation. At a given temperature, the oxide's free energy of formation from the ES-UHTC, shown by point a, is less negative than that from conventional UHTC, shown by point b, indicating the ES-UHTC is less likely to oxidize.

While these calculations currently do not account for entropy stabilization, they provide a basis for the prediction of the oxide composition and potential morphology. I will address the effect of entropy stabilization by first calculating the configurational entropy for an entropy stabilized carbide, nitride and boride of interest. I will apply these results to correct the entropy of formation for each oxidation reaction, and modify the Ellingham diagrams accordingly. The slopes represent the negative of the change in entropy, thus an increased configurational entropy in the starting material is expected to result in a steeper positive slope for the reaction, as shown in Figure 2, meaning the oxide formed from the ES-UHTC would have reduced thermodynamic stability relative to the starting material.

2.2 Experimental Work

One of the biggest challenges of studying UHTCs is engineering an experimental set-up for achieving temperatures greater than 2000°C in a laboratory setting, without contamination or damage to the equipment. I employ direct resistive heating, wherein ~100A of current is passed through a specimen with a a thin cross section. UHTC specimens are heated in this way to ultra-high temperatures (>1600°C) through Joule heating. The temperature is monitored using an emissivity correcting infrared pyrometer. The output from the pyrometer is used to control the current passed through the

specimen, thereby maintaining the specimen at a temperature set by the program. This set-up is based on that described by Karlsdottir and Halloran⁹, and Shugart and Opila¹⁰. While the hardware for the direct resistive heating system currently exists in the Opila Lab at University of Virginia, it has only been used

for temperatures at or below 1800°C for one material system. In the last year, I have modified and optimized it to conduct oxidation experiments at temperatures above 1800°C. This included redesigning the frame for mounting the specimen chamber and pyrometer to eliminate variability in the experimental set-up, and testing a new design for the sample geometry to minimize the torque on the specimen during loading and heating, which could result in premature cracking and fracture of the specimen. I plan to upgrade the transformer and infrared pyrometer to increase the upper limit of the temperature range that can be run on this set-up.

I will use this set-up to conduct oxidation experiments on samples of candidate ES-UHTCs fabricated by my collaborators at the University of California San Diego up to ultra-high temperatures. I plan to characterize the oxides that form with Xray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS), using equipment available at University of Virginia's Nanoscale Materials Characterization Facility. I also plan to characterize





the changes in composition of the base material using electron probe microscopy (EPMA) and transmission electron microscopy (TEM). I will use X-ray photoelectron spectroscopy (XPS) to interrogate the chemical structure in the oxide scale and underlying material. This may provide insight into how the different species present in the oxide are bonded and help identify new complex oxides. Oxidation kinetics will also be determined by measuring the changes in oxide

thickness, conducted using SEM, in the oxidized specimens, as a function of time. The oxidation kinetics of each individual component will be used as a baseline for comparison with the entropy stabilized ceramic. Specimen composition will be varied systematically to evaluate the effect each component has on the oxidation kinetics.

I will also conduct further experiments to better understand the oxidation mechanisms in these multi-component UHTCs, and the mechanisms and consequences of the subsurface depletion of these specimens. Preliminary results from an experiment conducted on a candidate system are shown in Figures 4 and 5. A spark plasma sintered (HfZrTiTaNb)B2 ceramic was fabricated and machined into bridge-shaped specimens shown in Figure 4(b). A bridge was exposed to an oxidizing atmosphere at ~1500°C with low PO_2 (1%) for 5 minutes in the resistive heating system (Figure 3). Figure 5 shows a secondary electron image and elemental maps of the cross section of the oxidized region of the bridge, created by fracturing the bridge in half. A porous oxide rich in Hf and Ta is seen. The region below this oxide is depleted in Hf, Zr, Ti and to a lesser extent Ta. This is an unexpected result, as the oxidation of HfB2 and ZrB2 tend to occur via oxygen diffusion inward, not metal diffusion outward.11 This depletion could also have implications for the entropy stabilization of the underlying material.



Figure 5. (top right) SEM image of the fracture cross-section and (bottom) EDS elemental maps of oxidized (HfZrTiTaNb)B2 sample

3. Expected Outcomes

At the conclusion of this study, I expect to understand the effect of entropy stabilization on the oxidation resistance of UHTCs. Additionally, I will provide recommendations for future avenues of UHTC development, and explore the viability of ES-UHTCs as a candidate for thermal protection systems in hypersonic vehicles.

4. Funding

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Investigating the effects of "sonic nets" on songbird social networks, dominance hierarchies, and dispersal

Introduction

After the "Miracle on the Hudson" in 2009, it should come as no surprise that birds can cause major damage to aircraft, and this damage can quickly get expensive. In fact, birds annually cost the aerospace industry \$973 million (Swaddle et al. 2016). In an effort to exclude birds from airfield sites in order to prevent aircraft-bird collisions, Dr.'s John Swaddle and Mark Hinders developed "sonic nets," a parametric array of sound that specifically targets airfields, but does not spread noise to surrounding areas. Sonic nets are played at ecologically relevant frequencies (pitch) that overlap with that of pest bird communication, preventing birds from hearing and responding to alarm calls, which ultimately results in long-term exclusion of birds from the area. Sonic nets have shown to successfully reduce the abundance of birds in affected areas both in a captive aviary (46% reduction) and at an airfield (82% reduction), with the potential to reduce costs associated with bird strikes by up to 96.4% (Mahjoub et al. 2015; Swaddle et al. 2016). Though the sonic nets are effective at displacing the birds we do not know whether the birds are negatively affected by the technology. This lack of knowledge is a barrier to full-scale implementation. Here, I propose to study whether the social structure of flocks of birds are fundamentally altered by a sonic net. Flock social structure affects crucial activities, such as foraging and breeding, and hence is a suitable assay of impacts on the birds.

In addition to noise generated by purposefully placed sonic nets, the aerospace industry introduces a vast amount of noise into wildlife communities. In birds, excess environmental noise is associated with physiological consequences such as decreased immune function, increased cortisol, and reduced reproduction (Chloupek et al. 2009; Halfwerk et al. 2011; Kight and Swaddle 2011). In addition, noise can cause behavioral ramifications, as it masks avian communication including alarm calls, inhibits the ability of a bird to detect an approaching predator, and weakens pair bonds between males and females (Swaddle and Page 2007; Potvin 2016). A common behavioral response to noise is to simply disperse to a quieter area, and thus noise is linked to changes in community composition and species diversity in noise-polluted areas (Francis et al. 2009). While most studies focus on areas directly impacted by the noise, it is vital to understand how these noise sites are affecting non-noise areas and learn where birds disperse to in response to this disturbance.

My project seeks to assess the ecological impact of sonic nets and similar noise pollution, by understanding the ecological consequences of displacing birds away from sound. Specifically, I aim to understand where birds disperse to in response to sound and how this changes the flock social structure and dominance hierarchies within flocks. In particular, I will apply social network analysis to quantify changes in flock structure – which allows me to assess how intensely individuals are associating with and interacting with each other (Krause et al. 2007). The structure of social networks in flocks can change patterns of behavioral strategies and mating (Krause et al. 2007; Sih et al. 2009; Oh and Badyaev 2010; Formica et al. 2012). In addition, the dominance interactions within these social networks can also have implications for individual fitness, access to resources, and protection from predators (Ellis 1995).

I will perform two studies: a captive study with a model songbird, the zebra finch (*Taeniopygia guttata*); and a field study in Brisbane, Australia, with the red-backed fairy wren (*Malurus melanocephalus*) – an indicator species of ecosystem health. Social networks will be determined both at the site of induced noise disturbance via sonic nets and at surrounding quiet areas, and I will compare pre-noise and post-noise networks using the parameters of network

structure (a network-level measure of # connections/# potential connections) and degree centrality (an individual-level measurement that determines the number of connections each individual has within the network).

Objectives

I hypothesize that the sonic net will result in widespread dispersal of the flocks at that location, and that this dispersal will increase the denseness of surrounding adjacent flocks. In terms of social networks, I predict that the network structure will increase at the surrounding quiet areas while they will decrease at the sonic net site. In this research I aim to determine

- 1) If flocks retain social structure when dispersing from noise or if they distribute throughout adjacent flocks.
- 2) If the dispersal caused by sonic nets alters the dominance hierarchies within flock structure.

Methods

Captive Study:

I will observe flocks of zebra finches in a three-roomed aviary at William & Mary. Room 1 will experience the sonic net, Room 2 will serve as a connecting room, and Room 3 will be a quieter area. One flock of 10 birds (5 males, 5 females) will be tested at a time, for a total of 15 flocks over the experiment. Male (5) and female (5) zebra finches will be given individual ID's by with a unique colored band. Perches will be placed



Figure 1: Layout of Captive Study

at varying heights within each room, as dominant birds compete for the higher perches. They will also be fitted with a unique Passive Integrative Transponder (PIT) tag onto their band, with the PIT tag receivers on all of the perches throughout the three rooms. This will allow for automated data collection on flock structure and dominance hierarchies that can run throughout the course of the study. Pilot studies already indicate that a flock of finches will fully explore this aviary and will be displaced away from the sonic net.

Following a brief acclimation period, I will collect baseline social network and dominance data during the proceeding week to determine flock structure before exposure to sonic nets, as well as noting the location of the birds (in case there is a side-preference in the aviary). Social networks will be calculated from observations of individual bird interactions: who are they interacting with and how often? Observations will last for two hours a day throughout the week. Two birds will be observed at a time so that all individuals in the flock will be assessed within one week. As each bird is wearing a PIT tag I will also analyze proximity information for all individuals over a longer time period to assess who is interacting with whom. In addition, I will record the outcomes of dominance interactions over perches and food – precious commodities for small birds – in order to construct a dominance hierarchy.

After one week, the sonic net will be introduced to Room 1 (alternated with Room 3 every two days to remove side biases). The sonic net is a 2-10 kHz "pink" noise played through

directional speakers at approximately 80dBA. I will repeat data collection at the onset of noise to reassess flock social structure, dominance hierarchies, as well as dispersal and room choice.

Field Study

The aviary study will give me detailed information about individual and flock responses to the sonic net, but is limited in that captive birds do not face the same ecological pressures as freeliving birds. Therefore I will conduct a field study on a continent where we have received substantial commercial interest in the application of sonic nets – Australia. In Summer 2017, I will travel to a site near Brisbane, Australia for a nine-week field season. The study site has wellestablished and studied flocks of red-backed fairy wrens, most of which have unique colored leg bands. For the first week of the field season, I will collect social network data through visual observation of the unique colored bands to establish baseline network locations and structures throughout the field site. Given the larger study area compared to the captive study, I will adjust my observation method to select groups of birds (normally 4-6 birds per group) and determine their group association via coordinated movement. These groups will be followed for 20-minute sessions and rotated throughout the field site, noting any interactions the individuals within these groups may have with other nearby birds. Following this baseline data collection, I will introduce the sonic nets at the epicenter of one of these sub-networks, replicating the sound conditions of the captive study, and using an outdoor speaker system powered from a car battery. I will spend the following week reanalyzing sub-networks at noise polluted and surrounding unpolluted areas. This design will include three replicates, with noise introduced to three different sub-networks over the course of the field season.

Data Analysis

Data will be analyzed using social network R packages, such as 'igraph', and I will measure degree centrality as a node-based measure and network density for a network-level measure. Nodes will be individual birds and edges will consist of interactions between individuals. The network will be weighted to indicate frequency of interactions by edge thickness. This will give me an idea of what changes for individuals within a network, and what changes for the overall network structures. Networks can then be compared using a Mantel test.

Conclusion

Changes in social network structure could affect population-level processes, such as gene flow, disease transmission, reproductive fitness of communities, and behavioral adaptations. Therefore, it is imperative to understand how the application of sonic nets (and noise pollution more generally) can influence these social groups of animals. A reduction of degree centrality and network structure at a disturbed site could result in reduced mating success and reduced information flow. Likewise, an increase of these parameters at surrounding sites could escalate the risk for disease transmission, or homogenize any local behavioral adaptations and therefore reduce potential for cultural evolution.

The completion of this research can develop further feasibility for the implementation of the sonic nets at airports. Though we have had promising success at displacing birds from an airfield we do not know whether this displacement causes harm to the birds – this creates an implantation barrier. In addition, our data will allow scientists to understand how anthropogenic noise is affecting avian populations at and around noise sites, and with the aerospace industry serving as a prominent source of this environmental noise disturbance, it could also influence future technological development to minimize excess noise from aircraft.

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Dark Matter Content of the Local Group Dwarf Galaxies

Dark matter is the most abundant form of matter in the observable universe, comprising almost 85 percent of the total mass, and outweighing regular matter, which makes up stars, by almost six-to-one. Yet, to date, no scientist has been able to image dark matter directly. Instead, to infer its presence, we study other observable implications of dark matter. Dark matter exerts the gravitational force on regular matter, like stars, but does not interact with regular matter through the electromagnetic force. Therefore, we can only study dark matter in environments where stars, gas, and dust are gravitationally interacting – in galaxies, for instance. Dwarf galaxies are key elements in the study of dark matter, as dwarf galaxies have the highest inferred dark matter fractions (Majewski et al., 2008; McConnachie, 2012). However, dwarf galaxies also tend to contain far fewer luminous stars (~ 100 million to a few billion stars) than galaxies like the Milky Way (\sim several hundred billion stars), so it is inferred that nearly 99 percent of the total mass in dwarf galaxies is made up of dark matter. (For comparison, the typical spiral galaxy, like the Milky Way, is made up of roughly 80 to 90 percent dark matter.) Due to their relative proximity, dwarf galaxies in the Local Group have played a valuable role in the search for dark matter (e.g. Aaronson & Olszewski, 1987; Hargreaves et al., 1994, 1996; Kleyna et al., 2002).

We currently have over a dozen epochs of radial velocity measurements from the Sloan Digital Sky Survey (SDSS) Apache Point Observatory Galactic Evolution Experiment (APOGEE) for stars in two of the Local Group dwarf galaxies, Ursa Minor (UMi) and Draco. These precise radial velocity measurements are then used to derive the velocity dispersion in the galaxy, which can then be used to determine the total mass of the galaxy from the virial theorem. We have specifically selected these two dwarf galaxies because previous velocity dispersion (σ) measurements for these low luminosity dwarfs are very high, on the order of $\sigma \sim 10$ km s⁻¹ (Aaronson & Olszewski, 1987; Wilkinson et al., 2004; McConnachie, 2012). Such large σ values correspond to a total inferred galaxy mass far exceeding what can be accounted for by the luminous stellar component alone. This indicates that these galaxies have a very high dark matter fraction. These data, taken over the course of a year, gives the most precise radial velocities for individual stars in these dwarf galaxies, and the most precise measurement of their radial velocity dispersions.

It is currently thought that the dispersion in the measured radial velocity of the stars in the UMi and Draco dwarf galaxies reflects the actual motions of stars within the galaxy, but does not account for the possible additional motions attributable to stars wobbling due to motions of fainter companion stars. (Aaronson & Olszewski, 1987; Wilkinson et al., 2004) A binary system, which consists of two bodies, typically one star and a smaller companion – like a smaller star, brown dwarf, or planet – orbiting a common central point or barycenter, can inflate the measured velocity of a star beyond its orbital motion within the galaxy. A large number of stars with companions in a dwarf galaxy might artificially inflate the calculated velocity dispersion due to additional reflex motion about their barycenters, which would then artificially inflate to the total inferred mass of the galaxy derived from the virial theorem. Until now, the inflation of σ due to a high fraction of companions in the UMi and Draco dwarf galaxies has been ignored, because the few rather cursory probes for companions have suggested that binary effects seem small. However, it is fair to say this has not been rigorously tested with a comprehensive survey of numerous stars. Previous probes for dark matter in Local Group dwarf galaxies had only a few epochs of observation over a baseline of a couple of months, at most. (Greevich & Putman, 2009; Walker et al., 2007, 2009; Wilkinson et al., 2004) The APOGEE instrument has completed a comprehensive sampling of a large number of dwarf galaxy stars over many epochs (> 24 epochs for stars in UMi, > 19 epochs for Draco) with more precise radial velocity measurements than were previously possible with instruments used in previous studies – the William Herschel or Magellan Telescopes, for example. The APOGEE instrument can achieve radial velocity precisions of $< 1 \,\mathrm{km \ s^{-1}}$. whereas instruments on Herschel or Magellan can only achieve radial velocity precisions of $\sim 2 \,\mathrm{km \ s^{-1}}$. APOGEE observations also span a baseline longer than one year. APOGEE is especially equipped to undertake the test of binarity in dwarf galaxies, much more effectively than previous studies. With the apOrbit pipeline (Troup et al., 2016) APOGEE data can be used to fit orbits to binary systems. Early analysis of these APOGEE data indicates that about half of the stars in the UMi and Draco dwarf galaxies potentially have companions, with a slightly higher fraction of binary systems in Draco. Such a significant binary fraction might substantially artificially inflate σ , and therefore, artificially inflate the total inferred mass and dark matter content, if those companions are not accounted for in previous calculations.

To improve on previous measurements of σ in these galaxies, our first goal is to define which of the members of each of these dwarf galaxies do, in fact, have companions. We can then exclude those binary systems from the sample that we use to calculate velocity dispersion, which would yield a more accurate total mass for the galaxy. Essentially, we want to answer the question, if we include the population of stars which have a companion in our calculation of σ , how much is the estimate for the velocity dispersion inflated and how much is the total calculated mass inflated?

Accurate calculation of the total mass and dark matter contained in dwarf galaxies is especially important to models of their formation and evolution, particularly as it relates to their ability to retain gas and form stars. Accurate mass estimates will allow astronomers to understand how dwarf galaxies contribute to the mass density of the entire universe, as well as understand how dwarf galaxies contribute to the evolution of larger Milky Way-like galaxies. We would then be better able to map the way mass is distributed through the universe.

We are particularly interested in studying the chemical content or metallicity, kinematic properties, and spatial distribution of the stars that are part of binary systems. A radial gradient in metallicity – which is the relative abundance of heavy atomic nuclei, like carbon, nitrogen, etc. compared to the abundance of hydrogen and helium – in the binary population which differs from the radial metallicity gradient observed in the population of stars without companions could indicate that the binary systems formed during a different stage of the dwarf galaxy's evolution, because metallicity is an indicator of age. We expect metal poor stars, consisting of mostly hydrogen and helium, to be a very old population of stars, since they would have formed prior to the synthesis of heavy metals from hydrogen and helium in the very early universe. Younger stars tend to have much higher metallicities. Differences in the radial metallicity gradient for the stars with companions, as compared to the metallicity gradient for the stars without companions, could indicate that binary systems in these dwarf galaxies formed during a certain burst of star formation (SF), and may enable us to point to a certain time-period when binary systems were forming in the UMi and Draco dwarf galaxies.

This study of binary systems might also be an important tool in understanding the evolution of dwarf galaxies, and how they impact the evolution of even larger galaxies. SF is an important contributor to the evolution of any galaxy. It has been posited that the UMi dwarf galaxy had a single burst of SF around 14 billion years (Gyr) ago, which lasted less than 2 Gyr (Mighell & Burke, 1999; Carrera et al., 2002), whereas the Draco dwarf had a large burst of SF which ended roughly 10 Gyr ago, followed by a low, constant rate of SF throughout its history, and a second burst of SF around 2 to 3 Gyr ago (Aparicio et al., 2001). As stated previously, early analysis of these data indicate that the Draco dwarf galaxy contains a higher fraction of binary systems than does the UMi dwarf, which could provide further evidence for the differences in the proposed SF histories of these galaxies. Understanding how the pieces that impact the formation of binary systems, like metallicity and SF history, will give important insight to the formation and evolution of dwarf galaxies.

Studying Local Group dwarf galaxies provides a unique laboratory to observe the most abundant form of matter in our universe. Our contributions will provide a more accurate measure of the total masses and dark matter content of two Local Group dwarf galaxies, and with further observations, provided by the continued operation of the APOGEE North (SDSS) and South (Las Campanas Observatory/LCO) instruments, we might also be able to better constrain the mass and dark matter content of other dwarf galaxies. These APOGEE instruments will obtain observations of the Bootes I dwarf from the Northern hemisphere, and the new APOGEE instrument at LCO will allow APOGEE to obtain observations of the Sculptor, Carina, and Sextans dwarf galaxies. Ultimately, by studying the binary population and APOGEE stellar chemical abundances of the Local Group dwarf galaxies, we expect to be able to better understand their star formation and evolution history.

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