Sintering Behavior, Structural, and Catalytic Properties of Ytterbium Oxide (Yb2O3)

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SINTERING BEHAVIOR, STRUCTURAL, AND CATALYTIC PROPERTIES OF YTTERBIUM OXIDE (YB$_2$O$_3$)

by

ALINA AFTAB

A thesis submitted in partial fulfillment of the requirements for the Honors in the Major Program in Aerospace Engineering in the College of Engineering and Computer Science and in the Burnett Honors College at the University of Central Florida Orlando, Florida

Fall Term 2019

Thesis Chairs: Nina Orlovskaya, Ph.D. and Richard Blair, Ph.D.
ABSTRACT

Ytterbia (Yb₂O₃) is an oxide ceramic, whose magnetic properties and crystal structure were studied to some extent in the past. However, the information on Yb₂O₃’s catalytic properties is lacking. Therefore, in this work, the sintering behavior and catalytic properties of Yb₂O₃ were examined. Yb₂O₃ ceramic samples were made using pressureless sintering of the commercially available Yb₂O₃ with 99.99% purity powder. The powder was first uniaxially pressed at 20 MPa in a steel die followed by pressureless sintering at different temperatures of 900 ºC to 1600 ºC for 4 hours. The densities of these samples were measured, and the sample sintered at 1600 ºC had the largest density of 62.88% and 900 ºC samples had the lowest of 45%. To improve the density of samples, three samples were uniaxially pressed same as the previous samples; however, they were also cold isostatically pressed at 260 MPa. These samples yielded the highest density of 90% after sintering at 1700 ºC for 4 hours. Raman spectra of sintered Yb₂O₃ were collected, which corresponded very well to those published in the literature. The microstructure of sintered Yb₂O₃ was studied by scanning electron microscopy. X-ray diffraction (XRD) showed that Yb₂O₃ crystallize in an Ia₃ cubic structure. X-ray fluorescence (XRF) was performed and it confirmed high counts of Yb and high purity of Yb₂O₃ powder. The catalytic experiments were performed on highly porous Yb₂O₃ samples sintered at 900 ºC. In the first experiment, Syn gas (H₂ + CO) was passed over Yb₂O₃ at 30 psig at 400 ºC with a rate of 3 cc/m. In the second experiment, the temperature was lowered to 250 ºC with all other conditions of the experiment maintained the same. As a result of the first reaction, the products included methane, ethane, and ethylene. The products produced in the second trial were ethane, ethylene, propene, butane, butene, cyclobutene, and methyl alcohol.
ACKNOWLEDGMENT

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I would also like to acknowledge Katerina Chagoya and Alan Felix with their help with the catalytic experiment, XRD, and XRF, Ruslan Kuliiev for his help with Raman spectroscopy, and Jesse Thompson for his help with SEM.

The intent of this work was to produce Yb2O3 samples with different porosity to study their vibrational, thermal, and mechanical behavior supported by the National Science Foundation project # 1337758.
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1 INTRODUCTION AND BACKGROUND

1.1 Motivation for research

Ytterbia (Yb₂O₃) has been of significant interest as a fiber amplifier and in fiber optic technologies [1]. Another area where Ytterbia has been of use is the lasing application [2]. Moreover, Ytterbia is also considered an excellent sintering aid in ceramics such as Si₃N₄/BN Fibrous Monolith Ceramics to improve the mechanical properties of these ceramics [3]. One other potential application of Ytterbia is as the electrolyte in the Solid Oxide Fuel Cells (SOFC). SOFC offers an efficient and environment-friendly method for the conversion of chemical to electrical energy. SOFC consists of electrolytes and electrodes and the process involves providing fuel to the anode to induce an oxidation reaction which results in electrons being released to the external circuit. The cathode is then provided an oxidant to induce a reduction reaction. This process allows the electrons from the external circuit to flow from anode to cathode producing electricity [4]. Despite this interest in using Ytterbia in the applications mentioned above, there is not sufficient research on the sintering behavior or the mechanical properties of the ceramic. In order to fully explore the applications of Ytterbia, developing a complete understanding of the ceramic’s mechanical, structural, and catalytic properties is crucial. For this reason, the objectives of the proposed research include (1) developing a sintering regime that would result in the 95% or higher dense samples, (2) investigating the physical properties of the ceramic, and (3) determining the catalytic nature of the ceramic.
1.2 Background on Ytterbia (Yb₂O₃)

Ytterbia is a compound made of ytterbium and oxygen. Ytterbium is part of the Lanthanides group which are the transition metals found in the f-block of period six in the periodic table [5]. Ytterbia belongs to the cubic lanthanide sesquioxides group which consists of two cation sites C₂ and C₃i that are inequivalent [2]. The ceramic has a polymorphous crystallographic structure with three phases below 2000 °C that include cubic (bcc), monoclinic, and hexagonal phase. In the cubic phase, the space group is \textit{Ia\bar{3}} (no. 206) with 16 formula per unit cell. In monoclinic, the space group is \textit{C2/m} (no. 12) with 6 formula unit in every cell. Finally, in the hexagonal phase, the space group is \textit{P3m} 1 (no. 164) with 1 formula in each unit cell [6]. The crystal structure of Yb₂O₃ can be seen in Figure 1. Figure 2 and 3 shows the XRD and SEM of Yb₂O₃ pellets, respectively, and Table 1 lists some of the Yb₂O₃ properties.

![Crystal structure of Yb₂O₃](image)

Figure 1: Crystal structure of Yb₂O₃ [7]
Figure 2: XRD pattern of Yb$_2$O$_3$ pellet [8]

Figure 3: Scanning electron microscope images of the Yb$_2$O$_3$ pellet [8]
Table 1: Crystal structure, lattice parameter, theoretical density, sintering information, average grain size and % densification for the sintered REO ceramics [8], [1]

<table>
<thead>
<tr>
<th>Compound</th>
<th>Yb$_2$O$_3$</th>
<th>Molecular weight</th>
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<td>Crystal System</td>
<td>Cubic</td>
<td>Melting point (°C)</td>
<td>2355</td>
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<tr>
<td>Lattice parameter</td>
<td>10.4361(1) Å</td>
<td>Boiling point (°C)</td>
<td>4070</td>
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<tr>
<td>Theoretical Density (g/cm$^3$)</td>
<td>9.2104</td>
<td>Exact Mass (g/mol)</td>
<td>189.934</td>
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<tr>
<td>Sintering T(°C)</td>
<td>1560</td>
<td>Average grain size (μm)</td>
<td>2 ± 2</td>
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<tr>
<td>Sintering duration (hr)</td>
<td>4</td>
<td>% Density</td>
<td>99.1</td>
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</table>
2 LITERATURE REVIEW

2.1 Hydrophobic Nature of Yb₂O₃

One study has shown that the lanthanide oxide series is intrinsically hydrophobic [8]. The researchers found that the electronic structure of lanthanide oxides resembles that of hydrophobic hydration structure and suggested that as these oxides have a full octet of electrons in their outer shell, they are less likely to bond with surrounding water molecules. The study revealed that the lanthanide oxides’ water angle range, seen in Figure 4, was between 110° and 115°, which also indicated that they are hydrophobic. Moreover, the polar component was found to not play a big part in the surface free energy. Lastly, the researchers showed that the lanthanide oxides promote dropwise condensation as they are sufficiently hydrophobic and have drop sizes that are smaller than typical dropwise promoters which indicates that they will exhibit higher heat-transfer coefficients [8].

![Figure 4: Measured values of advancing water contact angle (left axis) and the polar component of the surface free energy (right axis) of lanthanide oxides [8].](image-url)
2.2 Phase Transition in Yb$_2$O$_3$

2.2.1 Phase Transition under High Pressure at Room Temperature

One of the earlier experiments performed to study the phase transitions was done by using Mossbauer spectroscopy [6]. In this study, the researchers found concrete evidence of a phase change from a cubic, which is called C type, to a monoclinic phase, which is called B type. This phenomenon was already shown in a previous study. However, the transition pressure in this study was 13 GPa, and in previous studies, it was approximately 2.5-3.5 GPa. This low pressure was observed in static high pressure and high-temperature experiments. The experiment in this study was performed at room temperature. The study also suggested that the transition from type C to type B phase occurs by a transition to type A, which is a hexagonal phase, first and small changes in the experiment can lead to a type B phase or type A phase in the material. The reason for this could be that the transition from either phase requires very little energy, and type B is a deformation of type A phase. Figure 5 displays the change in phase type after pressure of 20 GPa was applied. The graph ‘a’ shows the Mossbauer spectra of Yb$_2$O$_3$ before the pressure of 20 GPa was applied and graph ‘b’ shows the spectra afterward. The changes in Synchrotron radiation energy-dispersive x-ray-diffraction (EDXRD) diagrams as pressure is increased can be seen in Figure 6. The phase change observed in this study was found to be irreversible after unloading [6].
Figure 5: Yb Mossbauer spectra at 4.2 K for Yb₂O₃ [6].

Figure 6: X-ray diagrams showing pressure dependence of Yb₂O₃ at room temperature [6].
2.2.2 Phase Transition under High Pressure and High Temperature

Phase transition being an important aspect for the properties of materials, a study has been performed to investigate the anharmonic and phase transition in Yb$_2$O$_3$ by Pandey et al. [9]. This study differs from that by Meyer et. al. [6] in the way that it focuses on the phase transitions occurring due to high pressure as well as temperature. The researchers found that the diffracting peaks, shown in Figure 7, were the same as what JCPDS (Joint Committee on Powder Diffraction Standards), now known as ICDD (International Centre for Diffraction Data), has established for the cubic phase of Yb$_2$O$_3$. Moreover, the study showed that the peaks in the frequency at room temperature were shifting to a higher frequency as the pressure was increased. New modes were observed at the starting of the transition to the hexagonal phase, which was at 20.6 GPa, and the peaks did not shift significantly with the increase in the pressure after the material had transitioned to hexagonal phase. The hexagonal phase did not revert to the cubic phase with the release in pressure and that indicated that the phase transition is irreversible. The research also confirmed previously reported finding that in Yb$_2$O$_3$, the Raman lines under high pressure broadens, which can be seen in Figure 8. The frequency shift with respect to temperature at constant pressure showed complex behavior as the frequency shift was low at and below 200 K but showed a significant shift at higher temperatures and also showed hardening with increasing temperatures. Using temperature and pressure data, the researchers determined that the most dominating mode in Yb$_2$O$_3$ is anharmonic and is responsible for the mode hardening [9].
Figure 7: X-ray diffraction for Yb$_2$O$_3$ powder [9]

Figure 8: Raman spectra for Yb$_2$O$_3$ at room temperature as the pressure is increased [9]
2.2.3 Phase Change until Melting Point in Yb$_2$O$_3$

A study conducted researched the thermal expansion and phase change of Yb$_2$O$_3$ up to the melting point [10]. The experiment was carried out by heating the sample by a laser which was levitated in oxygen and argon. The researchers found that the melting temperature of Yb$_2$O$_3$ was 2450 °C which was in agreement with the data available in the IUPAC reports. However, the boundaries for the melting point obtained by measuring the cooling traces were lower at 2413 °C. The researchers also found that Yb$_2$O$_3$ remained in the C-type bixbyite structure until the melting point. Previous studies had concluded there to be a transition to the hexagonal phase before reaching the melting point, but in this study, no evidence was found to support that claim. In the experiment, the researchers observed an increase in the unit cell parameter with x Yb (24d) as the temperature was raised from 25 °C to 1000 °C, which can be seen in Figure 9. The linear thermal expansion of a crystal was reported to increase from 8.27 (10^{-6}) K$^{-1}$ at 25-600 °C to 10.31 (10^{-6}) K$^{-1}$ at 600-972 °C. The dependence of Yb’s atomic displacements on temperature can also be seen in Figure 9. This dependence was found to correspond well with previously reported data [10].
2.3 Heat capacity of Yb$_2$O$_3$

2.3.1 Optical Spectra and Heat Capacities Measurement of Yb$_2$O$_3$

A study focused on the interpretation of already existing data on Raman and optical spectra with heat capacity measurements of Yb$_2$O$_3$ [2]. The study discovered that Yb$_2$O$_3$ had a cubic C (bixbyite) modification and sixteen formula units are present in each elementary cell with twenty-four cations in C$_2$ site and 8 in C$_3$. In C$_3$ sites, the local axes are situated in the [111], [1$ar{1}$1], [1$ar{1}$1], and [111] directions and the C$_2$ axes are parallel to {100}, {010}, or {001}. The study presented the Raman-active as well as the infrared-active phonon and electronic transitions for Yb$_2$O$_3$ and found that the Raman spectra shifted from 3 cm$^{-1}$ to 8 cm$^{-1}$ as the temperature increased from 10K.
to 300K. The values that were found for the transitions were quite similar to those previously reported. The crystalline electric field splitting of Yb$^{3+}$ (4f$^{13}$) ions can be seen in Table 2 [2].

Table 2: Stark levels for ground state manifolds: C2 and C31 sites [2].

| Oxide   | $|SL|J$ | $Z_n$ | $E$ (cm$^{-1}$), Sp. | $E$ (cm$^{-1}$), Sch. | $E$ (cm$^{-1}$) calc. | $E$ (cm$^{-1}$), Sp | $E$ (cm$^{-1}$), S |
|---------|-------|-------|----------------------|----------------------|-----------------------|-------------------|--------------------|
| Yb$_2$O$_3$ | $^2F_{1/2}$ | 1     | 0                    | 0                    | 0                     | 0                 | 0                  |
|         |       | 2     | 388                  | 388                  | 410                   | 334               | 334                |
|         |       | 3     | 595                  | 595                  | 586                   | 740               | 740                |
|         |       | 4     | 1021                 | 1021                 | 1014                  | 980               | 980                |

2.3.2 Effect of Lattice Properties on Heat Capacity

One study investigated the contribution of electronic and lattice properties to the heat capabilities of Yb$_2$O$_3$ between the range of 10 to 350 K by extracting the contribution of the lattice property from the already measured heat capacity of Yb$_2$O$_3$ at a temperature lower than 100 K [11]. The calculated heat capacity for Yb$_2$O$_3$ can be seen in Figure 10 and the electronic contributions can be seen in Figure 11. The magnetic contribution at the temperature higher than 10 K was considered to be Schottky anomalies which are due to the electronic excitation to discrete energy levels. This phenomenon is caused as the free-ion ground term are split by the crystalline electric field. Using the lower heat capacity measurements, the researchers found that the peak of the Schottky anomaly was not reached due to which they concluded that the energy assignments could only be tentative. The practical entropy of Yb$_2$O$_3$ was calculated by extrapolation at
temperatures lower than 10 K, and Yb$_2$O$_3$ was assigned a value of 31.8 cal. (g.f.w. K.)$^{-1}$ at 298.15 K. While the enthalpy increment was established to be 4685 cal. (g.f.w.)$^{-1}$ [11].

Figure 10: Heat capacities of Yb$_2$O$_3$ [11]

Figure 11: Electronic heat capacities of Yb$_2$O$_3$ [11]
2.4 Magnetic Structure of Yb$_2$O$_3$

The magnetic structure of Yb$_2$O$_3$ has been investigated using neutron diffraction method on single crystals and powder of the material [12]. The researchers found that in Yb$_2$O$_3$, the moments were opposed in the rare-earth sides related by the body-center translation. The researchers also reported that the experiments performed by Mandel showed that for C$_{3i}$ sites, an anisotropic g tensor was present, and the major axis was present along with the local symmetry axis. In their research, researchers found that the largest g value was perpendicular to the twofold axis and along the [110] direction. They used a model, displayed in Figure 12, in which the moments were present along the local symmetry at the C$_{3i}$ sites, with sites related by the body-center translation having opposite moments. This model did not fit well with the experimental data. The other model suggested to them was similar to theirs but considered all the moments to be perpendicular to the local symmetry axis. This model fitted well with the data as compared to the previous model. The researchers could not explain the dipole-dipole interactions by calculating the lattice sum of dipole-dipole interactions. They also found that the Neel point of 2.4 °K agreed with the susceptibility data. The researchers could not come to a concrete model to describe the magnetic structure for Yb$_2$O$_3$, and dipole-dipole interactions were found insufficient to describe the magnetic structure. [12].
2.5 Electrical Properties and Permittivity of Yb$_2$O$_3$

Another study investigated the electrical conductivity and permittivity of Yb$_2$O$_3$ films [13]. The four methods used to synthesis the films are as follows: (1) using electron beam for the evaporation of Yb$_2$O$_3$ (2) using tungsten heater for the evaporation of Yb$_2$O$_3$ (3) reactive evaporation of Yb$_2$O$_3$ in air, and (4) oxidation of Yb$_2$O$_3$ films after evaporation. The researchers found that the electrical properties of the films depended on the method of deposition used. The films in which evaporation was due to tungsten heaters and films in which oxidation was carried out were amorphous and films in which deposition was due to electron beam or prepared by reactive evaporation were polycrystalline. The permittivity in the films with oxidation was higher
than in other films. The lowest resistivity was present in the films with evaporation from tungsten heater and the highest was in the ones with reactive evaporation [13].

2.6 Ion Spectra of Yb₂O₃

The single-ion spectra of Yb₂O₃ has been investigated [14]. The temperature range over which the experiment was performed was 200-300 K. The researchers determined that two sets of three electronic transitions must occur from site C₂, electric dipole, and site C₃i, magnetic dipole, which should be centered around 10,000 cm⁻¹ and have a spread of 700 cm⁻¹. Moreover, since the C₂ sites are more prevalent than the C₃i sites, transitions at these sites should dominate the spectra. The three bands chosen for electronic origins were C, E, and G, which can be seen in Figure 13. The researchers observed that the first purely electronic transitioning occurred at two different sites at 80 K. However, when the researchers doped Yb₂O₃ in Y₂O₃, then the material did not show a site preference [14].
Figure 13: Single ion spectra of Yb$_2$O$_3$ at 298 K and 80 K measured at approximately 10 Å [14]

2.7 Dielectric Properties of Yb$_2$O$_3$

An experiment studied the dielectric properties of vacuum deposited Yb$_2$O$_3$ films with the thickness of 400-2000 Å [15]. Dielectric properties show the dependence of capacitance and loss factor on frequency and temperature. The results showed an increase in capacitance with a decrease in the frequency, especially at high temperature. Moreover, the capacitance increased as the temperature was increased. The loss factor was also observed to increase with increasing temperature. The frequency at this time was seen to shift to a higher frequency region. The researchers also concluded the conduction to be ionic in nature in Yb$_2$O$_3$ [15].
2.8 Yb$_2$O$_3$ nanoparticles

2.8.1 Emission Behavior of Nanoparticles

One of the studies looked at the emission behavior of Yb$_2$O$_3$ nanoparticles [16]. Transmission electron microscopy (TEM) performed during the experiment showed that the nanoparticles clustered together due to the high-temperature calcination. Moreover, the researchers noted that the particles have a normal distribution with the diameter being 18 nm. The XRD confirmed previous studies’ conclusion that Yb$_2$O$_3$ has a cubic phase and both TEM and Debye–Scherrer equation confirmed the average crystalline size to be 17nm. The study also showed that the nanoparticles show a two-photon cooperation upconversion luminescence at 486 nm, which can be seen in Figure 14. Furthermore, the study found that Yb$_2$O$_3$ nanoparticles emit thermal radiations under a 980 nm laser excitation as the surface melts due to laser heating. Finally, the researchers concluded that the absorption cross-section of Yb$_2$O$_3$ depends on internal temperature and a higher temperature leads to a bigger absorption cross-section and in turn, a stronger luminous absorption [16].
Figure 14: The integrated intensities of Yb3+ cooperative upconversion luminescence (a), luminescence mechanism (b), and nonlinear thermal emission (c) of Yb2O3 nanoparticles as a function of the incident laser power density [16].

2.8.2 Synthesis of Nanowires, Nanorods, and Nano-Square

This research focused on the synthesis of nanowires, nanorods, and nano-square plates with Yb2O3 using the facial hydrothermal method to study the morphology and microstructure [17]. The study of the three morphologies did not reveal any change in the post thermal calcination at 550 °C and the XRD peak confirmed the phase to be C-type structure as well as with high single crystallinity. The microstructure of nanowires, nanorods, and nano-squares can be seen in Figure 15. Moreover, the three different morphologies had different phases but changed to the cubic phase with thermal annealing. The researchers also measured the interplanar distance of 0.297 nm in nanowires, 0.298 nm in nanorods, and 0.294 nm in nano-squares plates in (222) plane and NIR
absorption of 975 nm. Lastly, the XPS and UV-visible absorption revealed the presence of Yb(II) in the Yb$_2$O$_3$ nanostructures [17].

Figure 15: SEM image of nanowires, nanorods and nano-square plates [17].

2.8.3 Synthesis of Submicron and Nanomaterials

A study focused on the synthesis of submicron and nanomaterials of Yb$_2$O$_3$ prepared through the oxidation of ytterbium acetylacetonate-based precursors [18]. The researchers also performed heat treatment on Yb$_2$O$_3$ in the air atmosphere in the temperature range of 550 °C to 850 °C. From the results, the researchers concluded that the formation of morphologies of calcinated material occurred due to the presence of volatile components in the electrospun materials and these volatile materials were also the cause of small-sized crystals. With the initial heat treatment, the researchers observed the formation of small grain, high porosity, and high
specific surface area materials, as can be seen in Figure 16, as the volatile gases evaporated. With further heat treatment, the materials went through grain growth and agglomeration, leading to denser material and a decrease in the surface area. Moreover, the researchers found that the formation of these materials was due to the interconnected submicron polycrystals [18].

![Figure 16: images (a) and (b) show the SEM images before heat treatment and (c) and (d) show the SEM images after heat treatment [18].](image)

### 2.9 Yb2O3 Based Optical High-Temperature Sensor

One of the studies analyzed Yb2O3 based optical high-temperature sensor and the linearity, repeatability, and resolution were investigated [19]. Concerning the temperature of Yb2O3 and temperature of the emitting surface, the researchers found the temperature difference to be within 5% error limit which they concluded was due to the background contribution. Repeatability for the sensors was defined as the variation of the short-circuited current (Isc) at a particular temperature and the thin pellets used in the experiment were found to show small variation Isc than
thicker pellets. The researchers also observed that the order of the short-circuit fell as the distance was increased between photodetector and Yb$_2$O$_3$, as can be seen in Figure 17. X-ray diffraction was used to study the stability and repeatability of the sensor. The change in the crystal structure of oxide materials with high-temperature thermal cycling is a concern and a study was performed to observe the behavior of Yb$_2$O$_3$ as a function of thermal cycling. Yb$_2$O$_3$ was found to reduce to an unidentifiable non-stoichiometric phase under repeated cycling [19].

Figure 17: The variation of $I_{sc}$ with temperature for powder at distances of (a) 24 cm; (b) 32 cm; (c) 42 cm between the detector and the emitter. Powder wt. = 0.0125 g, detector area = 5 mm$^2$ [19].
3 METHODOLOGY

3.1 Preparations of Samples

Three types of samples were created to research the properties of Yb$_2$O$_3$:

1. Non-Annealed and non-Cold Isostatically Pressed
2. Annealed and non-Cold Isostatically Pressed
3. Non-Annealed and Cold Isostatically Pressed

3.1.1 Non-Annealed and non-Cold Isostatically Pressed

The powder used in this study was provided by All-Chemie LTD and had a purity of 99.99%. XRF was performed on the powder to confirm composition and the powder was found to be a match to Yb$_2$O$_3$. 2.5 g of Yb$_2$O$_3$ powder was measured out using a standard balance and then poured into a cylindrical die to be uniaxially pressed through the Carver Hydraulic press shown in Figure 18 at a stress of 20 MPa. A total of 27 samples were created and a group of three samples was sintered at temperature range of 900 °C - 1600 °C in Thermolyne 46100 High Temperature Muffle Furnace, shown in Figure 19, for four hours. For sintering, the temperature was increased at an increment of 5 °C per minute until the set temperature was reached, at which point the dwell time started. Once the dwell time was over, the temperature ramped down to 550 °C at 5 °C per minute and stayed at that temperature for 0.1 hour. The decrement of temperature then started again at the same rate until 500 °C was reached where the temperature dwelled for 0.1 hour again and then ramped down to 20 °C. This dwelling at 550 °C and 500 °C was due to the previous
programming of the furnace and did not have any impact on the sintering or the properties of Yb₂O₃ samples. Care was taken to ensure samples were immediately sintered and were not left in the open air for an extended period of time.

Figure 18: Carver Hydraulic press used for uniaxially pressing Yb₂O₃ powder.

Figure 19: Thermolyne 46100 High Temperature Muffle Furnace used for sintering the Yb₂O₃ samples
3.1.2 Annealed and non-Cold Isostatically Pressed

In order to understand the effect of annealing on the sintering behavior of Yb\textsubscript{2}O\textsubscript{3}, two samples were created using annealed powder. 2.5 g of Yb\textsubscript{2}O\textsubscript{3} powder was annealed in the Barnstead Thermolyne 47900 Furnace, shown in Figure 20, for 26 hours at 105 °C. This annealed powder was then uniaxially pressed, same as the non-annealed samples. Both of these samples with heat-treated powder were sintered at a temperature of 1600°C. However, one sample was sintered for four hours, while the other was sintered for 24 hours. This difference in dwell time was to observe the effect of only annealing on the density of samples and then the effect of annealing and higher dwell time on the density of the samples.

![Barnstead Thermolyne 47900 Furnace used for annealing Yb\textsubscript{2}O\textsubscript{3} powder](image)

Figure 20: Barnstead Thermolyne 47900 Furnace used for annealing Yb\textsubscript{2}O\textsubscript{3} powder
3.1.3 Non-Annealed and Cold Isostatically Pressed

Six samples in total were created using this method. These samples were uniaxially pressed in a similar manner as the non-annealed samples; however, they were also cold isostatically pressed at 260 MPA in AIP Cold Isostatic Press CP360, shown in Figure 21, after the initial pressing. Afterward, these samples were sintered at 1700 °C for four hours. Again, great care was taken to not leave the samples in the open air before sintering.

Figure 21: AIP Cold Isostatic Press CP360 used for CIP of Yb₂O₃ samples
Figure 22 shows one sample of each of the different sintering temperatures and preparation methods. The gray color on four samples is due to the gold coating which was done for conducting SEM on these samples. As can be seen from the figures, as the sintering temperature increased, the volume of samples decreased.

Figure 22: 900 °C – 1600 °C samples in the first two columns followed by 1700 °C no- CIP, 1700 °C sintered late, 1700 °C CIP (2.5g), 1700 °C CIP (6g), 1600 °C 4-hr dwell time annealed sample, and 1600 °C 26-hr dwell time annealed sample.
3.2 Measuring Density and Porosity

Two different methods of density measurements were used in this study. The first was geometrical density which was used for porous samples. The dimensions of the pellets were measured using a digital caliper. Ten measurements of diameters and lengths were taken, and their average was used to find the volume which was then divided by the mass measured. The density obtained was then used to calculate the percent density and percent porosity of the samples using the given equations:

\[
\%\text{Density} = \frac{\text{Measured density}}{\text{Theoretical density}} \times 100
\]

\[
\%\text{Porosity} = (1 - \%\text{Density}) \times 100
\]

The second method of measurement of density utilized Archimedes principle and Acetone was used as the immersion liquid [20]. A beaker filled with Acetone was placed on a stand on top of an electronic balance such that the stand did not touch the balance plate. A measuring plate was suspended in the liquid using a wireframe attached to the balance and the samples were placed on this plate for mass measurement. The set up for the density measurement can be seen below in Figure 23.
The following equation was used to obtain the density where $M$ is mass of \( \text{Yb}_2\text{O}_3 \) sample in air, \( M_A \) is the mass of \( \text{Yb}_2\text{O}_3 \) sample in Acetone, \( \rho \) is the density of \( \text{Yb}_2\text{O}_3 \) sample, and \( \rho_A \) is the density of Acetone.

\[
\rho = \frac{M \ast \rho_A}{M - M_A}
\]
3.3 Catalytic Experiment

To test the catalytic nature of Yb$_2$O$_3$, a plug flow reaction was designed, and samples sintered at 900 °C were used due to their high porosity. The tube for the reaction was a smooth-bore seamless stainless-steel tube with 0.402 in. inner diameter, 0.5 in. outer diameter and a length of 12 in. The experiment consisted of two parts. The first part was purging the system with Argon and the second was the reaction by flowing Syn gas (hydrogen and carbon monoxide) through the tube. For the experiment, the tube was placed in a furnace; the temperature was set at a specific point, and the gasses being passed through the samples were analyzed by the Gas Chromatography/Mass Spectroscopy (GC/MS). The Agilent J&W HP-PLOT U column was used for the GC/MS. Purging was performed at 400 °C for one hour with a volumetric flow rate of 49.99 cc/m. The pressure was maintained at 10 psig throughout the process. During purging, GC/MS was turned off so that no sample of Argon was collected. Since the first purging was performed a day before the actual reaction, a second purging was performed for 30 minutes right before the catalytic reaction with the same parameters as before.

In the second part, which was the reaction, the temperature was raised to 500 °C and hydrogen and carbon monoxide was passed through the tube at 3 cc/m. The back-pressure regulator was set to 30 psig and the reaction ran for six hours with a sample from the system taken and injected into GC/MS very hour. The schematic of the catalytic experiment can be seen in Figure 24.
After the reaction, the Yb₂O₃ samples were heated up to 800 °C for two hours with a rate of 100 °C per hour to remove the coat of carbonization. Afterward, a second run of the experiment was performed. The purging parameters were the same as the first experiment except argon flow rate was 54 cc/m and the process lasted for two hours. The second catalytic reaction was completed at the same flow rate and pressure as before; however, the temperature was lowered to 250 °C and the reaction went on for 12 hours. Samples were taken every 35 minutes by the GC/MS. Two main difference between the first and second experiment was the orientation of the furnace and the placement of thermocouple. In the first experiment, the furnace was placed in a vertical position with the thermocouple at the middle of the tube; however, the vertical position was changed in the

Figure 24: Set up of the catalytic experiment
second experiment to a horizontal position to allow a more even flow of heat with three thermocouples placed at the start, middle, and end of the tube. The furnace read the average of these temperatures.

3.4 Raman Spectroscopy

Raman spectroscopy was performed on a sample of each temperature using the Renishaw InVia Raman microscope, shown in Figure 25. A 530 nm laser and 50x magnifying lens was used in this study. Before testing, a silicon sample was used to calibrate the microscope through InVia’s Wire software and a health check was performed. The spectrum showed a Raman shift at 520.455 cm\(^{-1}\) which is the same as the accepted Raman shift for silicon [21]. After calibration, the sample was focused under 50x magnifying lens. To acquire the spectrum of Yb\(_2\)O\(_3\), the grating scan type was set at extended. The lower range was set at 100 cm\(^{-1}\) and the high range was set at 8000 cm\(^{-1}\) which is the maximum range that can be used for Raman spectroscopy. The exposure time for this experiment was set at 10 seconds and the confocality was chosen to be standard. Pictures were taken after the sample was focused under the lens.

![Figure 25: Renishaw InVia Raman microscope](image-url)
3.5 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) was performed using a microscope by Tescan Digital Microscope Imaging. The sample sintered at 1600 °C for four hours was used for SEM. This sample was placed on a carbon tape attached to a sample plate and then the plate was carefully secured on the SEM stage. TEM grade with gold deposited on it was used as a calibration standard in this experiment. A speck on the standard was brought into focus such that the resolution was same as the resolution to be used for imaging Yb₂O₃. The images were acquired at a magnification of 1kx, 5kx, 10kx, and 30kx and the working distance was set at 7.054 mm.

3.6 X-Ray Diffraction

PANalytical Empyrean XRD was used in this experiment. Yb₂O₃ in powder form was put on the wafer plate and methane was used to adhere the powder to the plate. The amount of methane was small enough to not affect the experiment. The start angle for XRD was 5° and the end angle was 80°. The step size used during XRD was 0.0334 and time per step was 11 seconds. The total time for the experiment was three minutes and eight seconds.
4 RESULTS AND DISCUSSION

4.1 Density and Porosity Measurements

After sintering, the density was calculated for each sample of different sintering temperatures. Table 3 shows the density with a confidence interval of 95%, dimensions, and the average weight of the samples of different sintering temperatures as well as preparation methods.

Table 3: Density, height, weight, percent density and porosity of samples with different sintering temperature and preparation methods

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Diameter (mm)</th>
<th>Height (mm)</th>
<th>Weight (g)</th>
<th>Density (g/cm³)</th>
<th>%Density</th>
<th>%Porosity</th>
</tr>
</thead>
<tbody>
<tr>
<td>900</td>
<td>9.74</td>
<td>7.84</td>
<td>2.43</td>
<td>4.14 ± 0.093</td>
<td>44.92</td>
<td>55.08</td>
</tr>
<tr>
<td>1000</td>
<td>9.67</td>
<td>7.59</td>
<td>2.43</td>
<td>4.35 ± 0.192</td>
<td>47.23</td>
<td>52.77</td>
</tr>
<tr>
<td>1100</td>
<td>9.60</td>
<td>7.25</td>
<td>2.40</td>
<td>4.58 ± 0.074</td>
<td>49.65</td>
<td>50.35</td>
</tr>
<tr>
<td>1200</td>
<td>9.53</td>
<td>7.32</td>
<td>2.40</td>
<td>4.60 ± 0.134</td>
<td>49.93</td>
<td>50.07</td>
</tr>
<tr>
<td>1300</td>
<td>9.42</td>
<td>7.20</td>
<td>2.42</td>
<td>4.82 ± 0.096</td>
<td>52.26</td>
<td>47.74</td>
</tr>
<tr>
<td>1400</td>
<td>9.29</td>
<td>7.01</td>
<td>2.41</td>
<td>5.06 ± 0.098</td>
<td>54.85</td>
<td>45.15</td>
</tr>
<tr>
<td>1500</td>
<td>9.10</td>
<td>6.91</td>
<td>2.41</td>
<td>5.37 ± 0.138</td>
<td>58.28</td>
<td>41.72</td>
</tr>
<tr>
<td>1600</td>
<td>8.91</td>
<td>6.72</td>
<td>2.43</td>
<td>5.80 ± 0.178</td>
<td>62.88</td>
<td>37.12</td>
</tr>
<tr>
<td>1600 (Annealed, dwell: 4 hr.)</td>
<td>9.14</td>
<td>6.62</td>
<td>2.41</td>
<td>5.55</td>
<td>60.23</td>
<td>39.77</td>
</tr>
<tr>
<td>1600 (Annealed, dwell: 24 hr.)</td>
<td>9.32</td>
<td>6.80</td>
<td>2.43</td>
<td>5.24</td>
<td>56.78</td>
<td>43.22</td>
</tr>
<tr>
<td>1700</td>
<td>8.64</td>
<td>6.44</td>
<td>2.42</td>
<td>6.40</td>
<td>69.46</td>
<td>30.54</td>
</tr>
<tr>
<td>1700 (Delayed sintering)</td>
<td>8.65</td>
<td>6.31</td>
<td>2.41</td>
<td>6.50 ± 0.252</td>
<td>70.50</td>
<td>29.5</td>
</tr>
<tr>
<td>CIP 1700</td>
<td>8.056</td>
<td>5.92</td>
<td>2.42</td>
<td>8.44 ± 0.151</td>
<td>91.51</td>
<td>8.49</td>
</tr>
</tbody>
</table>
Plotting the percent density and percent porosity as a function of sintering time, as shown in Figure 26, shows that the average density increased with the increase in the temperature while the porosity of the samples decreased. The average density was lower than what was expected at the higher temperatures which indicated that the method for preparation of samples needed to be changed to improve the density of the samples.

![Temperature vs Density and Porosity](image)

**Figure 26: Effect of sintering temperature on density and porosity of Yb$_2$O$_3$ ceramics.**

To determine whether Yb$_2$O$_3$ was hydrophilic enough that the moisture content in the powder affected the density, the Yb$_2$O$_3$ powder was annealed. First, the sample from the annealed powder was sintered for the normal four hours. When the density of the sample did not show improvement, the dwelling time was increased to observe whether the amount of time affected the density. The density of the sample sintered for 24 hours was the same as the sample sintered for
four hours at the same temperature. In the article on the hydrophobicity of Yb₂O₃, mentioned earlier in the paper (page 5), the researchers had first pressed the powder at 270 MPa and then at 350 MPa to obtain a density of 99.1% with the sintering temperature of 1560 °C and dwell time of four hours [8]. In order to create that high pressure for compacting the Yb₂O₃ powder, CIP was performed. The original stress at which CIP was to be performed was 300 MPa; however, due to the limitations of the compressor used for CIP, the maximum stress achieved was 260 MPa. CIP vastly improved the percent density even at 260 MPa and the average wet density calculated for the sample was approximately 90% which leads to the assumption that had the pressure went to 300 MPa or higher, the density would have improved further. Table 4 lists the density of CIP samples calculated through geometrical density as well as wet density and Figure 27 displays a comparison of the densities resulting from each sintering temperatures and preparation methods.

Table 4: Geometric density, wet density, and percent density of CIP samples sintered at 1700 °C

<table>
<thead>
<tr>
<th>Geometric density (g/cm³)</th>
<th>Wet density (g/cm³)</th>
<th>Wet %density</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.96</td>
<td>8.39</td>
<td>90.97</td>
</tr>
<tr>
<td>8.07</td>
<td>8.47</td>
<td>91.85</td>
</tr>
<tr>
<td>8.04</td>
<td>8.45</td>
<td>91.70</td>
</tr>
</tbody>
</table>
Three samples were sintered approximately five days after being uniaxially pressed, unlike the rest of the samples that were sintered immediately. These samples were covered by Yb$_2$O$_3$ powder to prevent exposure to air. The samples were sintered at 1700 °C for four hours and density was calculated. The average density of the three samples was 6.5 g/cm$^3$ which is approximately the same as the density of 1700 °C non-CIP sample sintered immediately after being pressed. This evidence suggests that not a significant amount of moisture from the atmosphere gets absorbed by the powder in a short amount of time.

Figure 27: Comparison of different sintering temperatures and sample preparation methods
4.2 Catalytic Experiment Result:

The analysis of the four samples collected by GC/MS from the first reaction showed the production of methane, ethane, and ethylene. The first sample, taken an hour after the start of the reaction, showed the presence of only ethylene; however, both methane and ethane were found in the second and third samples along with ethylene. The GC/MS chromatogram in Figure 28 shows the products formed with respect to retention time.

![GC/MS chromatogram](image)

Figure 28: GC/MS data for catalytic experiment at 400 °C
The graph in Figure 29 displays the amount of each alkane and alkene made with respect to the total amount of alkane/alkene produced in the third sample.

![Alkane Fraction](image)

Figure 29: Alkane fraction of Methane, Ethane, and Ethylene on the catalytic experiment

Figure 30 shows the Flory-Schulz plot for the experiment. In the plot, ethane and ethylene were combined as they both have two carbons.
It can be seen from the graph that the reaction follows a Flory-Schulz plot which is a probability distribution. The results show that methane production was favored in the reaction at this particular temperature and pressure. Although only ethylene was observed in the sample taken after one hour, it is possible that methane and ethane were produced for a short amount of time but were then used to form ethylene. The reaction formation of methane can be shown by:

\[
\text{C}_2\text{H}_4 + \text{CH}_4 \xrightarrow{-\text{H}_2\text{O}} \text{C}_2\text{H}_6
\]
The samples, after the experiment, showed black patches on the surface, as can be seen in Figure 31. These black patches were considered to be coke. To reduce the surface where carbonization had occurred, the samples were heated to 800 °C.

![Figure 31: Samples before the heat treatment.](image1)

The surface was not reduced. Instead, the black patches turned brown, as shown in Figure 32, which showed that the black patches were, in fact, iron and not coke and the brown color was due to oxidization of iron during heating. These samples were used again for the second catalytic experiment as the surface area excluding the coke area was considered enough to not affect the experiment.

![Figure 32: Sample after the heat treatment](image2)

For the second experiment at the lower temperature, formation of alcohol was expected; however, only one alcohol—methyl alcohol—was formed. The graph in Figure 33 displays the GC/MS chromatogram for the 13th sample and Figure 34 shows the type of products formed and their amount in the 13th sample.
Figure 33: GC/MS data for catalytic experiment at 250 °C
In this experiment, more types of alkanes and alkenes were observed. In the 13 samples obtained throughout the experiment, none showed the presence of methane which was formed in the largest quantity in the first experiment. A possible reason for the absence of methane in the data is that it was formed for a short amount of time and was used to form higher levels of hydrocarbons before the next sample was taken. Some samples also showed the presence of cyclopropane, 1-propene, 2 methyl, and various structures of butene. The Flory-Schulz plot for the second catalytic experiment can be seen in Figure 34. In the Flory-Schulz plot, only the alkanes and alkenes were included. Moreover, as before, the fraction of all the alkanes and alkenes with the same carbon numbers were combined.
A Flory-Schulz plot is supposed to show a linear relation and the amount of each higher-level hydrocarbon is supposed to be less than the previous hydrocarbon. This is not the case in this reaction as methane was not observed even in the first sample and the trendline shown is of polynomial nature with the order of two.

4.3 Raman Spectroscopy Result

For the first Raman spectroscopy, samples one and two of 1600 °C sintering temperature sample batch were used to ensure the composition of the samples. The peak was found to be at 358.766 cm\(^{-1}\) for both samples which is similar to the previously reported Raman spectrum peak.
of 360 cm\(^{-1}\) at room temperature with a laser of 532 nm [22]. The Raman spectra of \(\text{Yb}_2\text{O}_3\) can be seen in Figure 36.

4.4 Scanning Electron Microscopy Results

The images acquired from SEM showed white lines. One possible reason for this could be charging which occurs when the material being imaged is not a conductor and does not absorb the electron being released to its surface. \(\text{Yb}_2\text{O}_3\) was found to have a flak-like structure which could be a possible reason for the difficulty faced with densifying the ceramic. Moreover, the grains
appear to be agglomerating. This agglomeration could have been caused by the sintering of Yb$_2$O$_3$.

Figure 37 displays the SEM images of Yb$_2$O$_3$ at the magnification of 5k and 30k.

![SEM images of Yb$_2$O$_3$](image)

Figure 37: SEM of 1600 °C sample acquired at the magnification of 5k and 30k
4.5 X-Ray Diffraction Spectroscopy

Figure 38 shows the intensity of x-rays at angles between 15°- 80°. A comparison of the obtained data on Yb₂O₃ and the JCPDS data on Yb₂O₃ was performed. The triangles are the peaks that matched the peaks in JCPDS data. As can be seen from the graph, the XRD is a close match to the JCPDS data. The highest intensity was observed at 29.7°.

![XRD spectrum of Yb₂O₃ obtained from testing](image)

Figure 38: XRD spectrum of Yb₂O₃ obtained from testing
CONCLUSION AND FUTURE WORK

In this work the sintering regime of Yb$_2$O$_3$ was studied. The densification of Yb$_2$O$_3$ powder was done by pressureless sintering. A density of 90% was achieved with a sintering regime that included uniaxial pressing, cold isostatic pressing, and sintering temperature of 1700°C. Raman spectroscopy and X-ray diffraction performed on 1600 °C sintering temperature Yb$_2$O$_3$ sample and Yb$_2$O$_3$ powder respectively agreed well with the already existing data. In order to achieve a density of 99%, further research needs to be performed to improve the sintering regime of Yb$_2$O$_3$. Increasing the pressure during cold isostatic pressing could potentially improve the density. Performing the Raman spectroscopy on the sample from each sintering temperature is recommended, as that will allow the observation of the molecular strain at each temperature. The Scanning electron microscopy on sample sintered at 1600 °C showed agglomeration and flake-like structure. Performing SEM at each sintering temperature as well is the next step as valuable information can be obtained, such as grain growth and crystal size. The catalytic reaction at 400 °C performed on Yb$_2$O$_3$ samples sintered at 900 °C produced methane, ethane, and ethylene; while, the reaction at 250 °C yielded ethane, ethylene, propene, butane, butene, cyclobutene, and methyl alcohol. The Flory-Schulz curve of the second reaction was found to be parabolic. Further research is especially needed to study the catalytic nature of Yb$_2$O$_3$. One potential next step could be to increase the duration of reaction at each temperature and determine whether higher-level hydrocarbons are produced with more time. Also, the deformation behavior of Yb$_2$O$_3$ oxide needs to be studied as there is almost a complete gap in knowledge of Yb$_2$O$_3$’s strength, hardness, fracture toughness, and stress-strain behavior in bending and compression. While the intent was to perform compression tests of Yb$_2$O$_3$ as a function of porosity in this HIM research work, the lack of access
to the testing facility prevented the measurement of the deformation behavior of Yb$_2$O$_3$ on time. Thus, this work will be done in the future. The information gained through these additional experiments will assist in better understanding the structural, mechanical as well as catalytic properties of Yb$_2$O$_3$ and will in turn, help in developing new and exciting applications of Yb$_2$O$_3$. 
LIST OF REFERENCES


