



## MIXED ALUMINA/CERIA COMPOSITIONS AS AN ENHANCED CERAMIC PROCESSING SURROGATE FOR RPS FUEL PELLETS

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Due to various safety and other considerations, the U.S. currently employs plutonium-238 in the form of <sup>238</sup>PuO<sub>2</sub> ceramic pellets as the fuel source in its current RPS (Radioisotope Power System) design. It is anticipated that a future European RPS design will also utilize a ceramic fuel form, such as <sup>241</sup>AmO<sub>2-x</sub>. Understanding the processing characteristics of the selected ceramic fuel form is one technical subject common to both efforts. While it would be ideal to perform RPS fuel processing experiments with the actual heat source radioisotope, due to radiological, cost, materials availability, and other considerations the employment of a non-hazardous and non-radioactive surrogate material could greatly ease this field of study. Identifying a cold surrogate material, that as closely as possible mimics the processing characteristics of the selected radioisotope, will enhance future development studies in support of both European and U.S. RPS activities. This paper centers on the recent application of mixed alumina/ceria compositions as an enhanced ceramic processing surrogate in support of both respective RPS programs.

### I. INTRODUCTION

Since the development of radioisotope thermoelectric generators (RTGs) at Mound Laboratories (Miamisburg, Ohio) over fifty years ago, several different fuel forms have been employed for the application of the radioisotope fuel plutonium-238.<sup>1</sup> Since plutonium-238 in its elemental state has several disadvantages, the fuel currently employed for deep space missions by the U.S is <sup>238</sup>PuO<sub>2</sub> which is an oxide ceramic.

The current U.S. MMRTG contains 32 <sup>238</sup>PuO<sub>2</sub> ceramic fuel pellets which provide all of the thermal energy that is converted into electrical power for the rover Curiosity on Mars. The process steps used in the fabrication of <sup>238</sup>PuO<sub>2</sub> fuel pellets utilize a number of classical ceramic fabrication techniques including ball milling, sieving, compaction, hot pressing, and sintering. The various ceramic processing parameters are carefully controlled which results in <sup>238</sup>PuO<sub>2</sub> fuel pellets with the required physical and mechanical properties that meet/exceeds all of the required safety and operational parameters. Since <sup>238</sup>PuO<sub>2</sub> is highly radioactive, great

care needs to be taken in the performance of all of the ceramic processing operations to ensure personnel safety.

The University of Leicester is being funded by the European Space Agency (ESA) to facilitate development of a European RTG.<sup>2,3</sup> It is expected that the European RTG will employ an <sup>241</sup>Am oxide as the heat source material also in the form of a ceramic pellet or disc. For both the U.S. and ESA RTG programs, the application of a non-radioactive surrogate material for performing various ceramic developmental processing endeavors would be very beneficial. This would have several advantages including reduced personnel radiological exposure with a corresponding significant reduction in associated costs.

### II. FUNDAMENTAL DESIRABLE CHARACTERISTICS OF A FUEL SURROGATE

It would be beneficial that any potential surrogate's physical and chemical properties be as similar as possible to the actual characteristics of the selected radioisotope containing ceramic fuel. This would help to ensure that the ceramic processing operations employed in the fabrication of fuel pellets would have comparable stress profiles within any surrogate pellets. In general, for this present discussion, there are three fundamental fuel characteristics of particular interest which should be considered in the selection of a surrogate material;

1. Fuel self-heating
2. Coefficient of Thermal Expansion
3. Fuel oxidation/reduction

#### II.A. Fuel Self-heating

Plutonium-238 has a half-life of ~87.7 years and produces ~0.5 W<sub>th</sub>/g when first processed. So each ~150 gram <sup>238</sup>PuO<sub>2</sub> pellet produces ~62.5 W<sub>th</sub>. Americium-241 has a half-life of ~432.2 years and produces ~0.1 W<sub>th</sub>/g. Previous work has studied the effect of pellet self-heating during ceramic processing employing finite element analysis.<sup>4</sup> This assisted in estimating a pellet's temperature gradients and stress fields during hot press processing of <sup>238</sup>PuO<sub>2</sub> fuel pellets. Since one of the main surrogate selection criteria is that the selected material should not be radioactive, simulating fuel self-heating was not included in this study.

## II.B. Coefficient of Thermal Expansion

For most bulk ceramic materials resistance to thermal shock/cracking due to a change in temperature is related to its coefficient of thermal expansion (CTE). For bulk ceramic components processed from powder formulations at high temperatures, it is really the coefficient of thermal contraction (CTC) which is of particular importance when a component is cooled from a high processing temperature. In most ceramic bulk materials, whose crystalline phases are stable, CTE and CTC are usually equivalent. In general, materials with high CTEs tend to be more susceptible to crack formation compared to materials with a low CTE. The CTE of  $\text{PuO}_2$  has been reported to be  $\sim 13 \times 10^{-6}$  cm/cm/K from room temperature to 1700K ( $\sim 1427^\circ\text{C}$ ).<sup>5</sup> Since high temperature ceramic materials exhibit a wide range of coefficient of thermal expansions, this assists in identifying potential surrogate materials which could meet this fundamental fuel characteristic.

## II.C. Fuel Reduction/Oxidation

At various stages in the fabrication of discs or pellets for RTGs, the fuel may be processed under reducing and/or oxidizing environments. This occurs in the processing of U.S. fuel pellets, and will likely also be the case in the ceramic processing of  $^{241}\text{Am}$  oxide discs/pellets for a European RPS. Both  $\text{PuO}_2$ ,  $\text{AmO}_2$  and  $\text{AmO}_{2-x}$  can be readily reduced to a sub-stoichiometric state and may even re-oxidize as a function of later processing conditions. For example, during initial processing of  $^{238}\text{PuO}_2$  fuel pellets, the powder is first placed within graphite dies and then hot pressed at an elevated temperature. It is reported that this reduces the fuel within a pellet to  $\text{PuO}_{1.88}$ .<sup>6</sup> At other stages in the pellet fabrication process it is possible that the stoichiometry of the oxide will again increase to two. For  $\text{PuO}_2$ , it is reported that as it is reduced the sub-oxide's linear expansion ( $\Delta L/L_0$ ) increases as presented in Table 1.<sup>5</sup>

**TABLE I.** Linear expansion of  $\text{PuO}_2$  and two sub-oxides.

Oxide Stoichiometry	Linear Expansion ( $\Delta L/L_0$ ) RT to 1273K
$\text{PuO}_2$	1.038
$\text{PuO}_{1.95}$	1.137
$\text{PuO}_{1.81}$	1.274

## III. CERIA AND/OR ALUMINA AS POTENTIAL FUEL SURROGATES

In both Europe and in the U.S., there have been a number of ceramic based processing studies investigating potential surrogate materials for RPS fuel. At NETS

meetings just since 2014, there have been over 20 presentations/papers related to the potential application of  $\text{CeO}_2$  as a processing surrogate for both  $^{241}\text{AmO}_{2-x}$  and  $^{238}\text{PuO}_2$ .

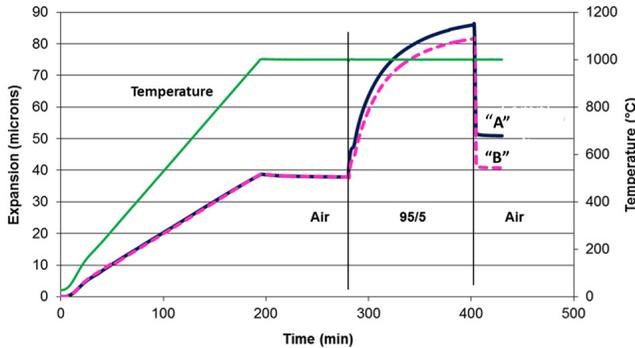
## III.A. Oxidation/Reduction Experiments on Sintered Ceria Specimens

$\text{CeO}_2$  has been of particular interest since it has a number of chemical and physical characteristics which makes it an attractive cold (non-radioactive) processing surrogate. One general chemical characteristic of both  $\text{AmO}_2$  and  $\text{PuO}_2$  is that they can readily release oxygen atoms under various reducing conditions which results in sub-stoichiometric oxides.  $\text{CeO}_2$  exhibits a cubic crystal structure which is maintained as it is reduced down to  $\text{Ce}_4\text{O}_7$ .<sup>7,8</sup> However, if the material is reduced even further to  $\text{Ce}_2\text{O}_3$  it will undergo a crystalline structural transformation since  $\text{Ce}_2\text{O}_3$  is hexagonal.<sup>9</sup> The reduction of  $\text{CeO}_2$  to  $\text{Ce}_2\text{O}_3$  with hydrogen has been shown to consist of a number of intermediate compositions which exhibit a range of colors.<sup>10</sup> This particular study lists over thirty intermediate compositions between  $\text{CeO}_2$  and  $\text{Ce}_2\text{O}_3$  whose color palette ranges from pale yellow, grey-blue, dark blue, blue-black, black, to olive green.

Recent work presented in Figure 1 presents expansion vs. time plots obtained on two pre-sintered  $\text{CeO}_2$  specimens "A" and "B" which were heated at  $5^\circ\text{C}/\text{min}$  from room temperature to  $1000^\circ\text{C}$  and then isothermally soaked for  $\sim 4$  hours.<sup>11</sup> Both  $\text{CeO}_2$  test specimens were initially pre-sintered to  $\sim 77\%$  theoretical density assuming a  $100\%$  theoretical density for  $\text{CeO}_2$  of  $7.1\text{g}/\text{cm}^3$ . The dilatometer system employed (Linseis Messgeraete GmbH L75 Duel Push Rod Vertical Research Dilatometer System) is capable of measuring very small changes in expansion as the y-axis scale in Figure 1 is in microns ( $\mu\text{m}$ ).

During the two experimental runs the furnace gas atmosphere during the initial ramp up and during the first segment of the isothermal soak was air. After  $\sim 90$  minutes the furnace gas atmosphere was changed from air (an oxidizing atmosphere) to  $95\%$  argon/ $5\%$  hydrogen (a reducing atmosphere). At that point the plot demonstrates a rapid increase in the linear expansion of the two specimens. The increase in measured expansion began to slow as a function of time and started to level off after  $\sim$ two hours at a magnitude  $\sim 2x$  the initial expansion due to thermal expansion as the furnace temperature initially increased from room temperature to  $1000^\circ\text{C}$ . At that point the flow of the  $95\%$  argon/ $5\%$  hydrogen cover gas was switched off and was replaced again with flowing air. The expansion vs. time plot shows an instantaneous and very rapid decrease in the expansion of the two specimens. After a very short time the expansion of "B" returns to basically its original expansion of  $\sim 40 \mu\text{m}$ . However, specimen "A" only contracts to  $\sim 50 \mu\text{m}$  and not

to its original expansion of  $\sim 40 \mu\text{m}$  likely due to crack formation. A  $5^\circ\text{C}/\text{min}$  ramp was used during the cooling of the furnace and the test specimens back down to room temperature which is not shown in Figure 1.

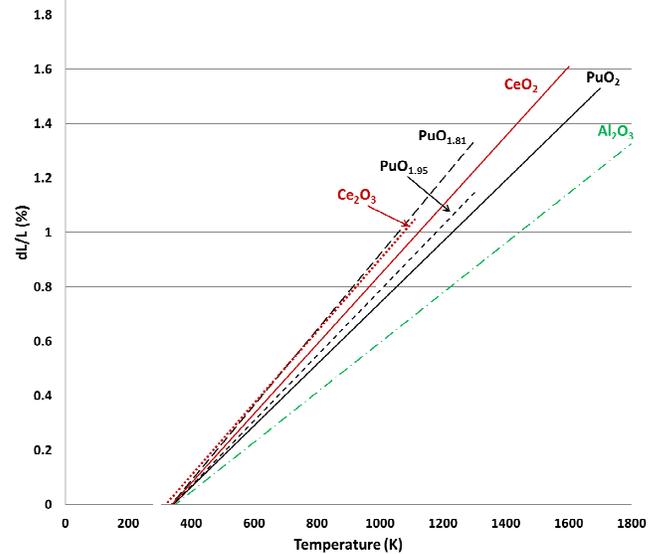


**FIG. 1.** Expansion vs. Time graphs obtained on two pre-sintered  $\text{CeO}_2$  specimens heated to  $1000^\circ\text{C}$  while the furnace gas atmosphere was changed from Air, to 95%Argon/5%Hydrogen, and back to Air.

The rapid change in expansion at the soak temperature when the furnace atmosphere was changed from air to 95% argon/5% hydrogen was likely due to the rapid reduction of the  $\text{CeO}_2$  to  $\text{CeO}_{2-x}$ . After the furnace atmosphere was switched from 95% argon/5% hydrogen back to air, the specimen contracted sharply since it very rapidly re-oxidized from  $\text{CeO}_{2-x}$  back to  $\text{CeO}_2$ . The oxygen reduction of the specimen was anticipated but the exact value for "x" is unknown. This is mainly due to the inability of "quenching" and maintaining the sub-stoichiometric form of  $\text{CeO}_{2-x}$  produced during the experiments when re-exposed to air.

### III.B. Comparison of the Linear Expansion Characteristics as a Function of Temperature of $\text{CeO}_2$ , $\text{PuO}_2$ and some of their Sub-oxides

The fabrication of  $^{238}\text{PuO}_2$  pellets (and very likely  $^{241}\text{Am}$  oxide pellets/discs) can result in the reduction/oxidation of the oxide fuel during various stages in the process. One physical property which can be directly impacted by change in stoichiometry is the expansion (or contraction) characteristics of  $\text{PuO}_{2-x}$ . Fortunately, other researchers have reported linear expansion values of  $\text{PuO}_{2-x}$  as a function of temperature.<sup>5</sup> Figure 2 shows a comparison of linear expansion vs. temperature for  $\text{PuO}_2$  and for the two sub-oxides of  $\text{PuO}_2$  listed in Table 1. Also, presented in the figure is the linear expansion vs. temperature for  $\text{CeO}_2$  and one of its sub-oxides, and for alumina which will be discussed later in this paper. The data demonstrates that the magnitude of the linear expansion increases as either  $\text{PuO}_2$  or  $\text{CeO}_2$  is reduced due to a pellet processing operation.



**FIG. 2.** Linear expansion vs. temperature for  $\text{PuO}_2$ ,  $\text{CeO}_2$ , and several of their respective sub-oxides.

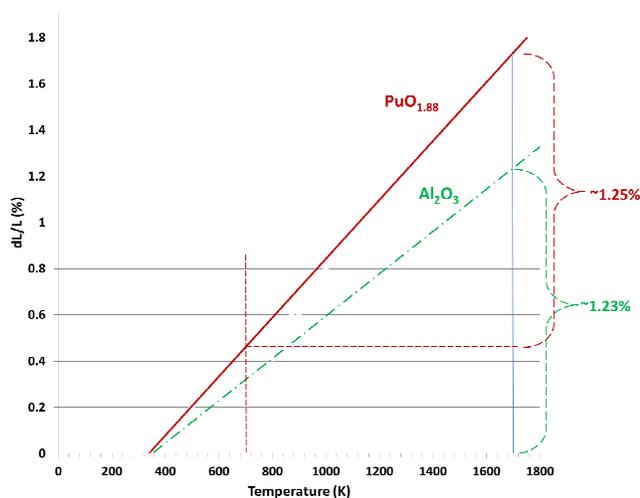
Comparing the linear expansion vs. temperature data in Figure 2 for  $\text{PuO}_2$  and  $\text{CeO}_2$  suggests that they are reasonably comparable with only a  $\sim 10$  to  $15\%$  difference between room temperature and  $\sim 1600\text{K}$ . As discussed previously, the hot pressing of  $^{238}\text{PuO}_2$  pellets in graphitic dies results in the reduction of the pellets to reportedly  $^{238}\text{PuO}_{1.88}$ .<sup>6</sup> It is also shown in Figure 2 that reduction of  $\text{PuO}_2$  to  $\text{PuO}_{1.95}$  or  $\text{PuO}_{1.81}$  actually results in an increase in linear expansion as a function of temperature. The authors have not found in the literature linear expansion vs. temperature data for  $\text{PuO}_{1.88}$ . However, a reasonable estimate would be between the data presented in Figure 2 for  $\text{PuO}_{1.95}$  and  $\text{PuO}_{1.81}$ .

Figure 3 shows such an estimate for the linear expansion vs. temperature of  $\text{PuO}_{1.88}$  from room temperature to  $\sim 1800\text{K}$  based on some of the information presented in Figure 2. Interestingly, it appears to be very similar to the linear expansion vs. temperature of  $\text{CeO}_2$  also from room temperature to  $\sim 1800\text{K}$ . This is likely one of the reasons why  $\text{CeO}_2$  has been of considerable interest as a possible surrogate in the radioisotope power systems community. However, it needs to be stated this apparent similarity is somewhat misleading when the actual processing aspects of a ceramic fuel pellet is taken into consideration.

In actuality,  $^{238}\text{PuO}_{1.88}$  fuel pellets do not attain an average equilibrium temperature equivalent to  $\sim 300\text{K}$  or "room temperature" after hot pressing. This is due to the self-heating of the fuel which can be estimated to result in an average equilibrium pellet temperature of  $\sim 700\text{K}$ , and not room temperature. Assuming a pellet processing temperature of  $1700\text{K}$  in combination with an average equilibrium pellet temperature of  $700\text{K}$  yields a first-order hot pressed pellet's total cooling gradient of  $1000\text{K}$ .

Figure 3 shows an estimated magnitude of the linear expansion (actually contraction) for a  $^{238}\text{PuO}_{1.88}$  pellet when cooled from 1700K to 700K of  $\sim 1.25\%$ . Whether the estimated equilibrium temperature of a hot pressed pellet of  $\sim 700\text{K}$  is highly accurate, it does not dramatically impact the current analysis. The authors do recognize that “ $^{238}\text{PuO}_2$ ” fuel pellets are not isotopically 100%  $^{238}\text{Pu}$ .

Figure 3 also shows the linear expansion (or contraction) of alumina or  $\text{Al}_2\text{O}_3$  which is a very classical common ceramic material. Alumina is not radioactive or self-heating, so if a pellet of alumina is cooled from 1700K it will eventually have an average equilibrium of 300K when at room temperature. Even though alumina has a significantly lower linear expansion/contraction compared to  $\text{PuO}_{1.88}$ , the actual magnitude of the linear contraction of  $\sim 1.23\%$  for alumina is very comparable to a  $\text{PuO}_{1.88}$  pellet which appears to have a total linear contraction of  $\sim 1.25\%$  when the self-heating of plutonium-238 is taken into account. For comparison, the data shown in Figure 2 for  $\text{CeO}_2$  suggests it has a much greater linear expansion/contraction between 1700K and 300K or room temperature of over  $\sim 1.6\%$  with its sub-oxides even being greater. This strongly suggests that alumina, from an expansion/contraction vs. temperature prospective, is likely an enhanced processing surrogate for  $^{238}\text{PuO}_2$  compared to  $\text{CeO}_2$ .



**FIG. 3.** Estimating the linear expansion/contraction vs. temperature for  $\text{PuO}_{1.88}$  (1700K to 700K) and  $\text{Al}_2\text{O}_3$  (1700K to 300K).

### III.C. Fuel Reduction/Oxidation Discussion

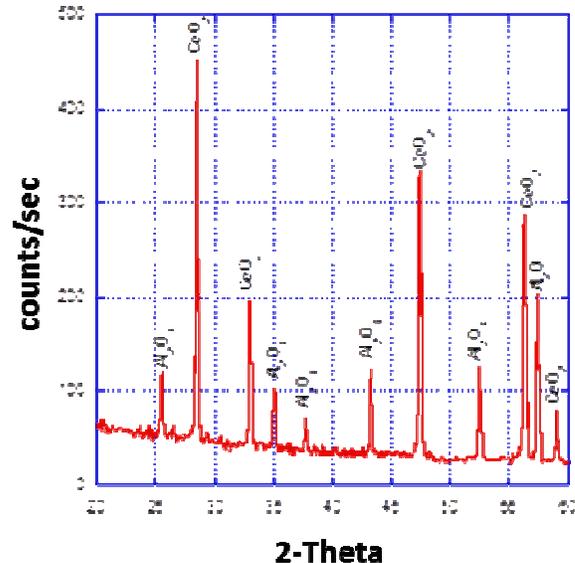
#### III.C.1. X-ray diffraction studies on sintered alumina/ceria mixed oxide specimens

In the previous section it was demonstrated that in terms of linear expansion/contraction as a function of temperature,  $\text{Al}_2\text{O}_3$  would be an enhanced surrogate for

the processing of plutonium-238 fuel pellets. However, another aspect desirable for a processing surrogate for  $^{238}\text{PuO}_2$  or  $^{241}\text{AmO}_{2-x}$  fuel pellets/pucks is associated with aspects of fuel reduction/oxidation. Oxidation/reduction of ceramic based fuel forms can result in the formation of strains resulting in stresses which may initiate crack formation and growth. It is known that alumina is extremely stable in both oxidizing and reducing atmospheres, while as presented in Figure 1,  $\text{CeO}_2$  does readily “donate or acquire” oxygen atoms depending on environmental conditions as do  $^{238}\text{PuO}_2$ ,  $^{241}\text{AmO}_2$  and  $^{241}\text{AmO}_{2-x}$ . If possible, it would be advantageous if a fuel surrogate exhibited expansion/contraction vs. temperature and to some degree oxidation/reduction characteristics similar to the selected radioisotope fuel form.

This led the authors to perform investigations on the processing of  $\text{Al}_2\text{O}_3/\text{CeO}_2$  mixed oxide ceramic specimens. The initial focus was to determine whether any inter-reactions (melting, intermediate compounds, phase transformations, etc.) occur during high temperature sintering that could impact application of an  $\text{Al}_2\text{O}_3/\text{CeO}_2$  mixed oxide formulation as a fuel surrogate. Previous experience has shown that  $\text{CeO}_2$  and of course  $\text{Al}_2\text{O}_3$  sintered discs can be made using standard ceramic processing operations.<sup>12</sup>

For the initial experiments mixed alumina/ceria discs (50mole%alumina/50mole%ceria) were cold pressed and air sintered at either 1400, 1500, or 1600°C for up to 60 minutes. X-ray diffraction (XRD) was performed on the mixed oxide sintered discs at both the University of Leicester and at the University of Dayton. Figure 4 shows the diffraction pattern obtained on a 50m%alumina/50m%ceria disc sintered at 1600°C.



**Fig. 4.** XRD pattern on a 50mole%alumina/50mole%ceria disc specimen that was sintered at 1600°C for 60 minutes.

The XRD studies at both universities determined that for the sintering conditions discussed, there was no apparent interaction between the two oxides. In all cases the only diffraction lines obtained corresponded to either alumina or ceria.

### III.C.2. Dilatometer experiments on alumina/ceria mixed oxide sintered specimens as a function of oxidizing and reducing test atmospheres

Since the XRD results on the 50m%alumina/50m%ceria sintered specimens showed no formation of any intermediate compounds (Figure 4), additional alumina/ceria test specimens were fabricated to investigate some of their oxidizing/reduction characteristics. A series of mixed alumina/ceria ceramic disc specimens ~2.5cm in diameter were cold pressed containing from 100%  $\text{Al}_2\text{O}_3$  to 100%  $\text{CeO}_2$  with three intermediate molar powder mixtures of either 75m% $\text{Al}_2\text{O}_3$ /25m% $\text{CeO}_2$ , 50m% $\text{Al}_2\text{O}_3$ /50m% $\text{CeO}_2$  and 25m% $\text{Al}_2\text{O}_3$ /75m% $\text{CeO}_2$ . These green discs were sintered at 1400°C, and then ~6mm in diameter test specimens were obtained using a diamond core drill. Similar to the oxidation/reduction experiments on sintered ceria specimens discussed in Section III.A., individual test discs were heated to 975°C in a dilatometer which again has the ability to measure small dimensional changes of a specimen as a function of temperature and gas atmosphere.

Figure 5 shows linear expansion data obtained on the mixed oxide test specimens as the initial air atmosphere was changed at temperature to a reducing gas mixture of 95%Ar/5% $\text{H}_2$ , and then back again to an air atmosphere. The results indicate that the majority of the reduction of the cerium oxide within the mixed oxide sintered discs occurs on the order of several minutes, while the majority of the re-oxidation occurs even more rapidly when the specimens are re-exposed to air. The magnitude of the change in linear expansion of the various ceria/alumina mixed oxide specimens as a function of gas environment in general ranges from ~50% to over 100%. A rapid linear expansion (or corresponding volume change) of this magnitude due to reduction/re-oxidation within a  $^{238}\text{PuO}_2$  or  $^{241}\text{Am}$  oxide ceramic fuel form will potentially induce strains resulting in the formation of micro-cracks leading to a decrease in a pellet/disc's mechanical strength.

For plutonium-238 containing fuel pellets an estimate can be made for the linear expansion change due to re-oxidation to  $^{238}\text{PuO}_2$  of fuel pellets after the hot press process. It has been previously reported that the hot press process results in the reduction of the pellet to  $^{238}\text{PuO}_{1.88}$ . Employing information contained within Figures 2 and 3 it can be determined that this re-oxidation corresponds to a change in linear expansion of ~0.2%. Intriguingly, Figure 5 suggests that an alumina/ceria mixed oxide

formulation containing ~75m% $\text{Al}_2\text{O}_3$ /25m% $\text{CeO}_2$  would exhibit roughly a ~0.2% change in linear expansion due to re-oxidation. While the precise mixed oxide ratio may still need to be determined, the indication is that it would likely result in an enhanced ceramic processing surrogate.

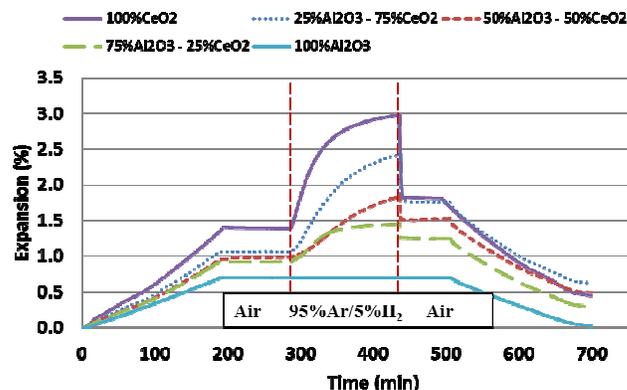


Fig. 5. Expansion vs. Time graphs obtained on a series of alumina/ceria mixed oxide sintered specimens heated to 950°C while the furnace gas atmosphere was changed from Air, to 95%Argon/5%Hydrogen, and back to Air.

## IV. CONCLUSIONS

The selection of a cold ceramic processing surrogate for  $^{238}\text{PuO}_2$  or  $^{241}\text{Am}$  oxides would be highly advantageous in helping to minimize personnel exposures and cost. Two fundamental desirable characteristics of a potential surrogate have been discussed including; a) thermal expansion/contraction and b) fuel reduction and/or oxidation, both of which can be significant factors in strain/stress/micro-cracking within a ceramic fuel pellet or disc.

Experiments focused on the processing of  $^{238}\text{PuO}_2$  fuel pellets employing various alumina/ceria mixed oxide ceramic specimens indicate that it may be possible to mimic several aspects of the fuel's thermal expansion/contraction and reduction/oxidation by employing ~75m% $\text{Al}_2\text{O}_3$ /25m% $\text{CeO}_2$  mixed oxide powder in the fabrication of surrogate ceramic "fuel" pellets. It is anticipated that a similar analysis as outlined in this paper may also prove useful in identifying an alumina/ceria mixed oxide formulation for the  $^{241}\text{Am}$  oxide based fuel being considered for a European RPS.

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