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FABRICATION OF SOL-GEL-DERIVED THORIA-URANIA BY COLD PRESSING AND SINTERING

J. M. Robbins
J. G. Stradley

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J M Robbins and J. G. Stradley

Paper presented at the Sixty-Ninth Annual Meeting,
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ABSTRACT

The use of the cold-press-and-sinter technique for producing sol-gel thoria-urania fuel pellets was investigated. We studied sol-gel powder calcination temperature, moisture content, uniaxial and isostatic forming pressures, and sintering atmospheres. The sol-gel powder was characterized by DTA, TGA, and BET surface area techniques. The sintered fuel pellets were evaluated in terms of shrinkage, density, and oxygen-to-uranium ratio. Shrinkages ranged from 16 to 20%, bulk densities ranged from 78 to 95% of theoretical, and the oxygen-to-uranium ratio was changed from 2.67 to 2.00 by hydrogen reduction.

INTRODUCTION

Sol-gel-derived thoria-urania is of interest because of the amenability of the sol-gel process[1,2] to the recycle of fuels and to the production of very homogeneous mixtures of fissile and fertile materials. Sol-gel fuel materials have been prepared in two forms, fragments and microspheres. The suitability of the sol-gel material for the remote preparation of thorium-oxide-base nuclear fuels has been shown[3] by using

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[1]J. A. Lane, et al., "Thorium Utilization Systems," Vol. 6, pp. 333–343 in Proc. Intern. Conf. Peaceful Uses At. Energy: 3rd, Geneva, 1964, United Nations, New York, 1965.

[2]O. C. Dean et al., "The Sol-Gel Process for Preparation of Thoria-Base Fuels," pp. 519–542 in Proceedings of the Thorium Fuel Cycle Symposium, Gatlinburg, Tennessee, December 5–7, 1962, TID-7650, Bk. 2 (July 1963).

[3]W. S. Ernst, Jr., "Recent Vibratory-Compaction Studies on Thoria-Urania," pp. 585–605 in Proceedings of the Thorium Fuel Cycle Symposium Gatlinburg, Tennessee, December 5–7, 1962, TID-7650, Bk. 2 (July 1963).

fragments with vibratory compaction. Sol-gel microspheres, because of their sphericity and uniform size, are well suited for coating with pyrolytic carbon in fluidized beds. Irradiation test results[4] indicate that carbon-coated oxide microspheres will be satisfactory fuels for high-temperature gas-cooled reactors. Since the sol-gel process appears so promising for nuclear fuel applications, this study was conducted to determine the applicability of cold-press-and-sinter fabrication techniques for producing fuel pellets of sol-gel oxide materials. When we started, no information was found in the literature pertaining to cold pressing and sintering of sol-gel materials. Since then, some information[5,6] has been published on the fabrication and densification of sol-gel powders; however, neither investigation covered the same conditions considered in this study.

EXPERIMENTAL PROCEDURE

The material used in this study was a thoria-urania gel prepared by the Chemical Technology Division. Although the general sol-gel process has been described elsewhere, we will briefly describe the method used in preparing the material for this study. Thorium nitrate was denitrated (or converted to thorium oxide) with steam at approximately 500°C, and the resultant powder was made into an 8 M thoria sol. Next, UO_3 powder was blended in to produce a sol containing about 6.5% normal uranium by weight. Finally, the sol was dried to a gel at approximately 80°C. The dried sol-gel material was in the form of sharp-edged, flat-faced chunks ranging from approximately 0.1 to 1 in. on a side and having a glassy appearance and orange-to-red color.

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[4] J. H. Coobs and R. G. Wymer, "Preparation, Coating, Evaluation, and Irradiation Testing of Sol-Gel Oxide Microspheres," Proc. Brit. Ceram. Soc. 7(3), 61–79 (1967).

[5] H. H. Crain and C. R. Hutchison, Fabrication of Fuel Pellets From Sol-Gel Powders, WAPD-TM-581 (November 1966).

[6] A. U. Daniels and M. E. Wadsworth, Densification and Sintering of Thoria and Thoria Gels, Department of Metallurgy, University of Utah, Salt Lake City, Utah, Technical Report 28 (Dec. 15, 1965).

First we crushed the chunks in a laboratory impact pulverizer until the material passed a 200-mesh (74 μ) screen. Next, a quantity of blended and screened powder was divided into four equal lots. One lot was put aside in a glass bottle, while the other three were placed in quartz combustion boats and calcined by rapidly heating in an air-atmosphere muffle furnace to the desired temperature (200, 400, and 750°C) and holding for 1 hr. The powders were then allowed to cool with the furnace.

Since sintered density normally increases with green or as-formed density, we wanted to determine the effect of pressing aids or lubricants on the green density. Water is an excellent lubricant for the forming of some ceramic powders,[7] increasing their green density. Yet, unlike various organic lubricants, it does not leave a carbonaceous residue to be removed. Therefore, each lot of powder was divided into two groups. In one group, called the control group, the moisture content was established at about 1% by the normal laboratory humidity. An additional 5% water was added to the second group of powders, called the wet group, by placing them in an atmosphere saturated with water vapor at 60°C for 40 hr.

Right-cylindrical pellets approximately 0.5 × 0.5 in. were cold formed in a steel die lubricated with a solution of stearic acid in acetone. Pellets were formed for each powder condition at three uniaxial pressure levels, 2000, 3500, and 5000 psi. Then, at least two pellets from each uniaxial pressure level were isostatically pressed at 20,000 40,000, and 60,000 psi, making a total of 18 different powder and forming combinations. After being formed, all the pellets were dried for at least 24 hr in a vacuum desiccator at 60°C. Immediately after drying, the pellets were placed on a layer of spherical thoria grog on alumina setter plates, loaded into an electric resistance furnace that had been preheated to 60°C, and sintered in air, helium, or hydrogen.

Differential thermal analyses (DTA) were performed on all the powders. Loose powder placed in platinum cups was heated in air at 10°C/min to 1100°C in a DTA unit. Surface areas of all the powders were measured by the BET method.

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[7]L. H. Van Vlack, Introduction to Physical Ceramics, Addison-Wesley Publishing Co., Inc., Reading, Mass., 1964, pp. 14-1, 14-2.

All the bulk densities, shrinkages, and weight changes reported in this study were measured by standard micrometer and analytical balance techniques.

CHARACTERIZATION OF THE SOL-GEL MATERIAL

The sol-gel material used in this study had an oxygen-to-uranium ratio of about 3.0 and contained 2.20% nitrates and 0.04% C. Spectrographic analysis of the material after grinding and screening showed that these operations introduced about 50 ppm Fe and 5 ppm Cr. If these were controlled to minimal amounts in the starting material, this additional contamination would probably be acceptable for most fuel applications. The powders we processed were screened only through a 200-mesh screen before use. A screen analysis on a typical sample of the powder is given in Table 1.

Table 1. Screen Analysis of Crushed Sol-Gel $(Th,U)O_2$

Screen Size (U.S. Standard Mesh)	Retained on Screen, %	
	Fraction	Cumulative
230	17.3	
270	14.8	32.1
325	17.0	49.1
pan	50.9	100.0

Since factors like surface area and volatiles such as nitrates and carbon affect the properties of some formed and sintered ceramic materials, we needed to determine the effects of calcination on these variables for this sol-gel material. The changes caused by calcining are presented in Table 2. As would be expected, the weight loss increased, while the nitrate content and surface area decreased with increasing calcination temperature.

We noted earlier that calcined sol-gel materials are hygroscopic. While moisture pickup can be controlled or prevented by bottling and

Table 2. Changes in Nitrate Content, Surface Area, and Weight Loss Caused by Calcination

Calcination Temperature (°C)	Nitrate Content (%)	Surface Area (m^2/g)	Weight Loss (%)
Uncalcined	2.20	65.3	0.00
200	0.04	68.7	1.36
400	0.001	61.6	2.38
750	0.0001	36.3	4.02

careful handling of the powder, it is difficult to protect formed pellets without resorting to a glove box operation. For example, pellets formed from hygroscopic sol-gel powder developed cracks, apparently caused by the pickup of moisture, between forming and sintering. An exothermic reaction between moisture and the dry hygroscopic sol-gel powder may explain the cracking observed in formed pellets. Further, the cracking can be eliminated by allowing the powder to pick up free moisture before forming. The three calcined lots of powder in this study increased approximately 1% in weight when exposed for 48 hr to the uncontrolled humidity of the laboratory. The uncalcined powder contained about 1.1% free moisture, which, as stated in the procedure, represents the control group. All of the powders of the wet group, calcined or uncalcined, absorbed an additional 5% moisture during the 40 hr exposure to a saturated humidity at 60°C.

Differential thermal analyses were performed on the control powders to establish a desirable sintering cycle. The DTA for the uncalcined powder is shown in Fig. 1. At approximately 120°C there is a strong endotherm due to moisture removal, then a strong exotherm at about 265°C, which is believed to be a carbon-nitrate reaction. Another endotherm occurs near 430°C due to nitrate decomposition, and finally the exotherm in the 650 to 1000°C range shows the release of surface energy from the powder. The DTA for the powder calcined at 200°C was very similar to that for the uncalcined powder. However, both lots of powder calcined

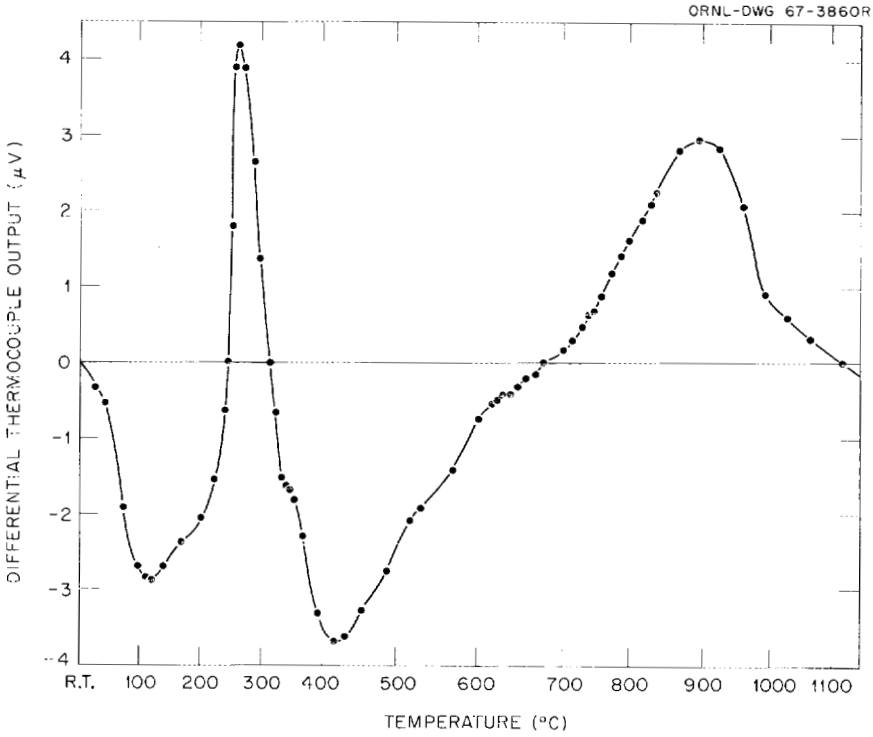

Fig. 1. Differential Thermal Analysis (DTA) Pattern of Sol-Gel-Derived Thorium-Uranium Oxide.

at 400 and 750°C had lost the 265°C exotherm and had a reduced endotherm at 430°C. Also, the surface energy release exotherm was slightly depressed for the powder calcined at 750°C, as would be expected because of its reduced surface area (Table 3). Based on the DTA results for uncalcined powder, we established an 80°C/hr firing cycle with 2-hr soak periods at 240 and 400°C and finally a 1.25-hr soak period at 1425°C.

RESULTS AND DISCUSSION

Satisfactory green pellets were produced from both groups of powders under all the forming conditions investigated. No pressure capping, laminating, or moisture squeeze-out was seen in any of the specimens at the three uniaxial pressures investigated. As expected, pellets formed from all powders increased in apparent strength with increased uniaxial and isostatic forming pressures and with moisture content. However, the apparent strength of the pellets formed from calcined powders decreased as the calcining temperature increased. Pellets of the 750°C calcined

Table 3. Effect of Moisture Content Upon the Bulk Density of Formed and Air-Sintered Material

Forming Pressure, psi		Density[a] of 200°C Calcined Powder				Sintered Density (g/cm^3)	
		Uniaxially Formed		Isostatically Formed			
Uniaxial	Isostatic	Control	Wet	Control	Wet	Control	Wet
2000	20,000	3.62	3.64	4.74	5.21	8.56	9.42
3500	20,000	3.79	3.85	4.71	5.23	8.63	9.42
5000	20,000	3.93	4.16	4.71	5.21	8.72	9.44
2000	40,000	3.62	3.84	5.21	5.28	9.14	9.47
3500	40,000	3.79	3.90	5.23	5.30	9.20	9.49
5000	40,000	3.95	4.08	5.24	5.32	9.21	9.53
2000	60,000	3.63	3.83	5.23	5.33	9.38	9.56
3500	60,000	3.80	3.86	5.55	5.34	9.42	9.55
5000	60,000	3.95	4.33	5.55	5.34	9.46	9.55

[a]Calculated on a dry weight basis.

powder formed at the 2000 psi uniaxial level required care in handling to prevent chipping. No abnormal die wear was noticed with the sol-gel materials as others[8] have reported; however, our highest uniaxial pressure level, 5000 psi, was very low compared to the reported 100,000 psi and greater.

As mentioned earlier, increasing the green density increases the sintered density. Hence, as high a green density as practical was desired. Figure 2 compares the influence of the three variables studied that affect the green bulk density: forming pressure, calcining temperature, and moisture content. The family of curves for the control group shows that the green density increased with forming pressure and decreased with increased calcination temperature. The effect of calcination became more pronounced with increasing forming pressure for the control group. While only one of the family of curves for the wet group is shown, it illustrates the decrease in slope caused by the additional moisture. This additional moisture equalized the isostatic forming pressure, allowing us to achieve approximately the same green density at 20,000 psi with the wet group as

[8] H. H. Crain and C. R. Hutchison, Fabrication of Fuel Pellets From Sol-Gel Powders, WAPD-TM-581 (November 1966).

5, all pg 2

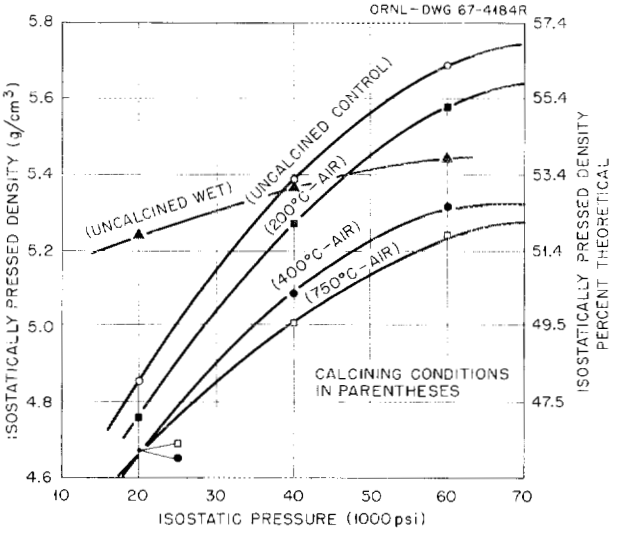

Fig. 2. Effect of Isostatic Pressure and Calcining Conditions on the Bulk Density of Isostatically Pressed Material.

at 40,000 psi in the control group. The additional moisture also decreased the maximum green density achieved; however, this was not detrimental to the final or sintered density.

The initial moisture content significantly affected the density of the air-sintered sol-gel material. The uniaxial, isostatic, and air-sintered densities for the sol-gel powder calcined at 200°C are shown. Two points are outstanding about this data. One, mentioned previously, pertains to the narrow spread in the sintered density of the wet group: it runs from 9.42 to 9.56 g/cm^3, compared to 8.56 to 9.46 g/cm^3 for the dry group. Thus, the additional moisture appears to lower the pressure required to achieve the greatest sintered density. The second point pertains to the increased densification that occurred in the wet group. For example, a wet pellet (6% moisture) formed at 2000 and 20,000 psi had a green density of 5.21 g/cm^3 and a sintered density of 9.42 g/cm^3, while a control pellet (1% moisture) formed at 2000 and 40,000 psi also had a green density of 5.21 g/cm^3 but sintered to only 9.14 g/cm^3. The reason is not completely clear, but apparently the presence of additional moisture during forming promotes sintering. The effect of moisture must occur during forming since 1.0 to 6.0% moisture was removed during drying at 60°C in vacuum. The additional moisture may cause cementing between the individual crystallites to enhance densification. We believe that this cementing is possible because the sol-gel thoria-urania behaves much like some other colloidal materials (sodium silicate, etc.) in the presence of water. If excess water is added, the material gets sticky and will develop a tenacious bond to other materials. All the data in Table 3 are for the powder calcined at 200°C, but they are representative of the behavior of the other three powders.

The shrinkage of a ceramic material is an important consideration when one attempts to produce parts to a predetermined size with close tolerances. The effect of powder calcination and green density on the diametral shrinkage during sintering to 1425°C in air is shown in Fig. 3. Pellets of the uncalcined powder and from that calcined at 200°C differed very little in shrinkage. The decrease shown for the powders calcined at 400 and 750°C reflects the reduced surface areas.

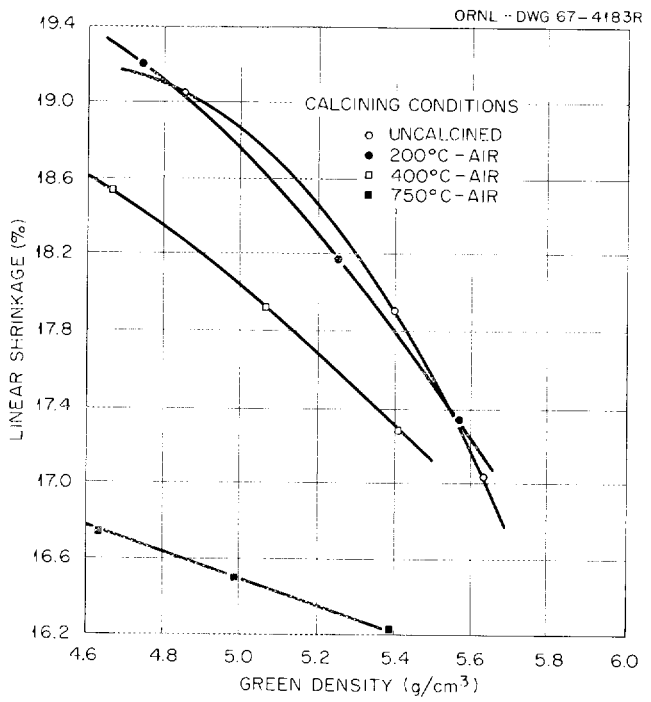

Fig. 3. Effect of Green Density and Calcining Conditions on the Diametral Shrinkage During Sintering.

Average bulk densities, which show the effects of different forming pressures, calcination states, and moisture contents for the sintered pellets, are summarized in Table 4. As expected from the discussion of green bulk densities, the sintered bulk densities decreased uniformly with the increase in calcination temperature. Also, the sintered bulk densities increased with increasing forming pressure and moisture content. In fact, calcination effects can apparently be overcome by increasing the forming pressure. Figure 4 shows that the sintered densities of pellets from the uncalcined powders and the powders calcined at 200 and 400°C are about the same for equivalent green densities. For example, if a pellet of a 400°C-calcined powder was formed at a green density of 5.7 g/cm^3, it should display a sintered density equal to that of the uncalcined powder with the same green density.

Figure 5 shows typical microstructures of the sol-gel thoria-urania after sintering in air at 1425°C. Sol-gel material has such a small grain size that only particles can be distinguished by conventional metallography. However, the microstructures in Fig. 5 do show something

Table 4. Bulk Densities of Sintered Thorium-Uranium Oxide Derived From the ORNL Sol-Gel Process

Forming Pressure, psi		Density (g/cm^3) for Different Calcination Temperatures, °C							
		Uncalcined		200		400		750	
Uniaxial	Isostatic	Control	Wet	Control	Wet	Control	Wet	Control	Wet
2000	20,000	8.73	9.29	8.56	9.42	8.26	8.75	7.89	8.10
3500	20,000	8.76	9.27	8.63	9.42	8.33	8.72	7.93	8.12
5000	20,000	8.79	9.33	8.72	9.44	8.34	8.75	7.92	8.12
2000	40,000	9.21	9.38	9.14	9.47	8.86	9.35	8.43	8.66
3500	40,000	9.25	9.49	9.20	9.49	8.87	9.38	8.46	8.66
5000	40,000	9.24	9.46	9.21	9.53	8.90	9.41	8.52	8.71
2000	60,000	9.42	9.43	9.38	9.56	9.24	9.52	8.73	8.99
3500	60,000	9.44	9.50	9.42	9.55	9.22	9.53	8.77	9.05
5000	60,000	9.46	9.56	9.46	9.55	9.33	9.58	8.83	a

[a]Pellets got wet during isostatic pressing; data not valid.

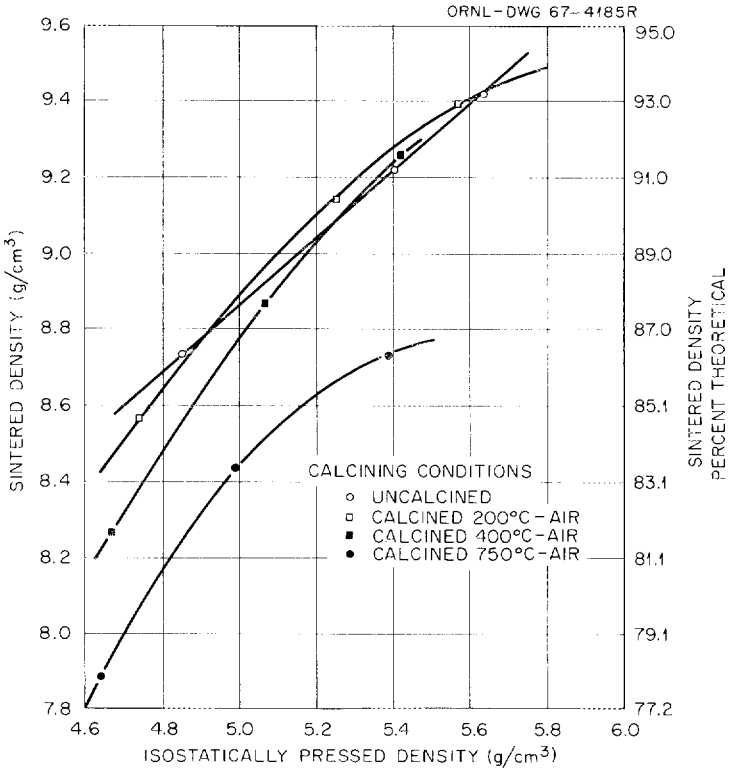

Fig. 4. Effect of Green Density and Calcining Conditions on the Sintered Density.

unusual. The bulk densities of (a) and (b) are about the same, yet the particle size and pore distribution for the two are quite different. The bulk densities of (b) and (c) are vastly different, but the structures are very much alike. Studying these and many other microstructures for pellets made in this program, we deduce that forming pressure has more of a bearing on the sintered structure than does sintering. In (b) and (c) some of the coarse particles were apparently sheared with the 60,000 psi forming load, thereby decreasing the size of the pores.

The effects of sintering atmosphere on bulk density are shown in Table 5. These pellets were all uniaxially formed at 3500 psi and isostatically pressed at 40,000 psi from control powder. Pellets sintered in air had the highest bulk density, those sintered in helium had a slightly lower density, and those sintered in hydrogen had the lowest density. While those sintered in air decreased some in density when refired in hydrogen to reduce their oxygen-to-uranium ratio, their final

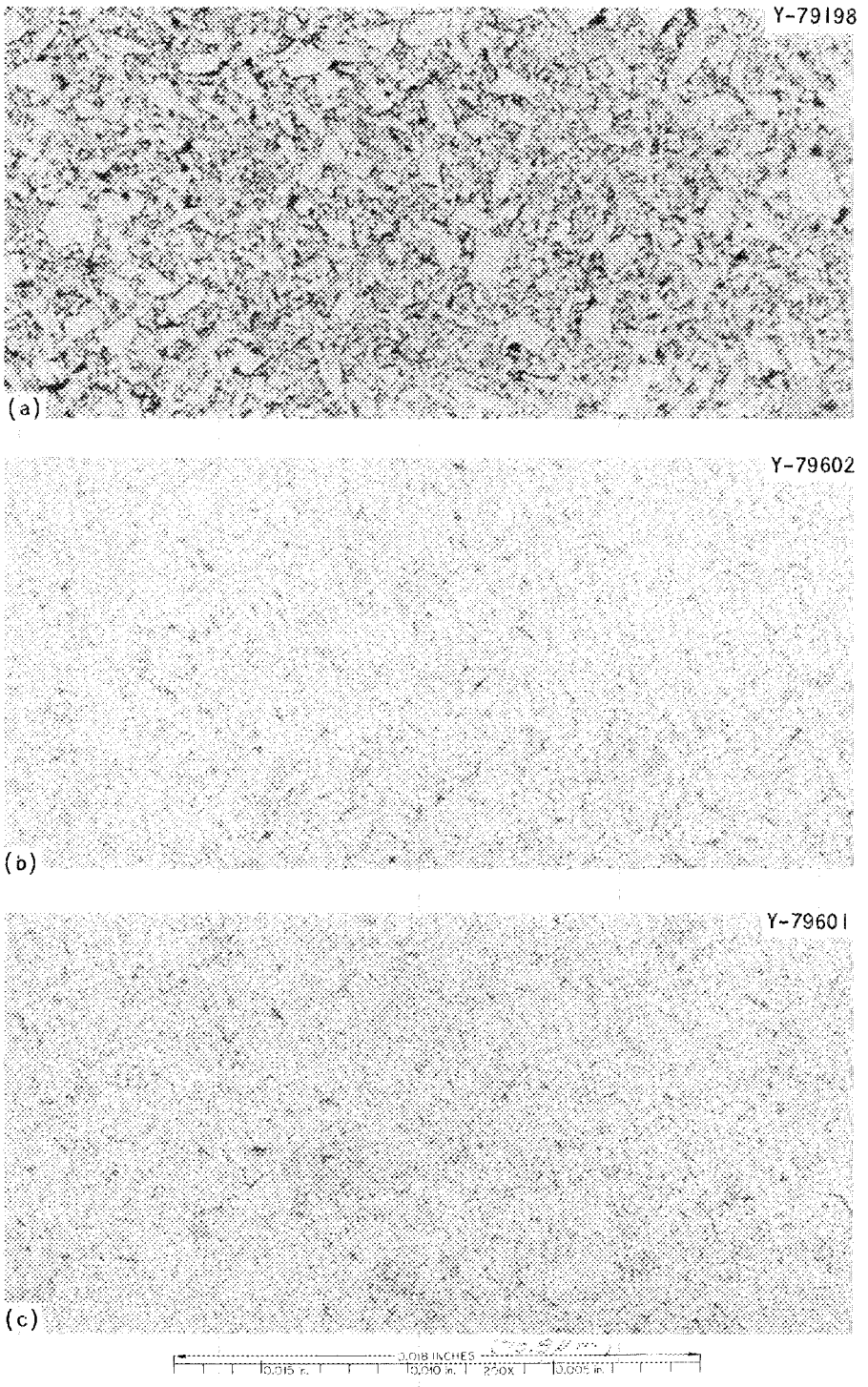

Fig. 5. Thoria-Urania Sol-Gel Sintered in Air. (a) Uncalcined control powder formed at 1200 psi uniaxial and 20,000 psi isostatic pressures; 8.76 g/cm^3. (b) 750°C calcined control powder formed at 1800 psi uniaxial and 60,000 psi isostatic pressures; 8.86 g/cm^3. (c) Uncalcined wet powder formed at 1800 psi uniaxial and 60,000 psi isostatic pressures; 9.58 g/cm^3. Etchant: 30 H_3PO_4–1 HF.

Table 5. Effect of Sintering Atmosphere on the Bulk Density of Sintered Pellets

Calcination Temperature (°C)	Bulk Density (g/cm^3) for Different Sintering Atmospheres		
	Air	Helium	Hydrogen
Uncalcined	9.25	9.06	8.91
200	9.20	9.12	8.95
400	8.87	8.78	8.58
750	8.46	8.31	8.12

density was still greater than that of a pellet sintered only in hydrogen. For example, the density of 9.25 g/cm^3 for a pellet sintered in air becomes 9.20 after hydrogen reduction. There is no difference in the microstructures of pellets with equivalent densities sintered in different atmospheres.

The effect of oxygen-to-uranium ratio on the color of the sintered pellets was quite pronounced even though the starting material contained only 6% uranium. As previously mentioned, the starting sol-gel material was orange to red and had an oxygen-to-uranium ratio of approximately 3.00. After air sintering, the oxygen-to-uranium ratio was about 2.67 (U_3O_8), and the product was dark brown. Helium and hydrogen sintering produced pellets with a light green color and an oxygen-to-uranium ratio of 2.00.

CONCLUSIONS

1. Fuel pellets may be produced from sol-gel thoria-urania by the cold-press-and-sinter technique with bulk densities 95% of theoretical.

2. The use of moisture, at least in amounts up to 6%, lowers the forming pressure required to achieve a pellet with a specific sintered density.

3. The sintered bulk density is controlled by powder calcination temperature, powder moisture content, forming pressure, and sintering atmosphere. Proper combinations of these may be used to produce pellets with specific sintered bulk densities.

4. The sintered structure appears to be established by the forming load rather than sintering conditions.

5. Sintering initially in air, followed by hydrogen reduction, produces a denser pellet than sintering completely in hydrogen; sintering in helium produces only slightly lower density.

6. Specimens formed from uncalcined powder achieved the highest bulk density. Equivalent densities can be achieved from powders calcined at 200 and 400°C, but higher forming pressures are required.

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