

has been used with a 300 kN machine in order to obtain macroscopic yielding, but there is no reason why similar designs should not be used with machines of more moderate capacity.

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### Quartz matrix isolation of radioactive wastes

One of the factors to be considered in the increased utilization of nuclear fission for energy production is the safe transportation and storage of the radioactive wastes derived from chemical reprocessing of spent nuclear fuels. Several components of the wastes will be highly radioactive and thus constitute a threat to man for  $10^4$  to  $10^6$  years. The presently conceived solution is to melt the wastes into glass forming compositions, solidify this melt in storage containers and either store these containers in shielded vaults or bury them in geologically stable strata such as bedded salt formations [1].

The solidified glass should be dense, hard, able to withstand constant temperatures of up to  $600^\circ\text{C}$  from radioactive decay and highly insoluble. Present processes use low solubility phosphate or borosilicate glass compositions. However, most phosphate based glasses devitrify above  $550^\circ\text{C}$  and borosilicate compositions often undergo liquid-liquid phase separation during melting. Both phenomena result in significantly reduced leaching resistance.

It is well known that ceramic articles fashioned

up to  $10^4$  years ago have survived burial with little apparent degradation. Certain ceramics would also be ideal matrices for isolating radioactive wastes. However, the temperatures necessary to form mixtures of these wastes and most ceramics into dense solids are hundreds of degrees above the highest values now employed in waste solidification processes ( $1000$  to  $1200^\circ\text{C}$ ). It is suggested that with the ceramic processing technique, hot-pressing, a dense solid may be formed out of the radioactive waste and a low leachability crystalline or glassy matrix at or below  $1000^\circ\text{C}$ . In order to demonstrate this idea common sand (quartz) was chosen as one matrix material. Detailed results using ten other less effective matrix materials will be described elsewhere [2].

The simulated radioactive waste chosen for this study was PW-4m, a typical light water reactor fuel reprocessing waste whose major components are  $\text{Na}_2\text{MoO}_4$  and nitrate solutions of Sr, Ba, Cs, rare earths and Zr as fission products and Fe, Cr, Ni from corrosion of fuel and reprocessing tanks. The liquid PW-4m was dried at  $110^\circ\text{C}$  and calcined at  $500^\circ\text{C}$  for 6 h and then ground to pass a  $-200$  mesh sieve. The matrix material was quartz (99.9%  $\text{SiO}_2$ ,

Pennsylvania Glass Sand Corp.) supplied at -200 mesh particle size.

The hot-pressing apparatus consists of a  $3\frac{1}{4}$  in. (84 mm) graphite cylinder with a concentric  $\frac{13}{16}$  in. (21 mm) cylindrical hole. The sample is compressed between  $\frac{13}{16}$  in. (21 mm) pistons on a standard press. The graphite reactor is inductively heated; temperature is monitored with a thermocouple recessed into the reactor. The hot-pressing is performed in air so that the graphite reactor must be replaced periodically.

In a typical experiment equal weights of simulated waste and matrix material are mixed, pre-formed into a  $\frac{3}{8}$  in. (19 mm) pellet, and placed in the graphite reactor. The sample is surrounded by graphite powder to prevent sticking to the reactor and to facilitate removal. The required temperature is achieved in 10 to 20 min. After hot pressing for a selected time the power is shut off and the reactor cools in 1 to 2 h.

Good results were obtained when the sample was heated at 1000°C under 2500 psi (176 kg cm<sup>-2</sup>) for 10 min. The product was a well consolidated disc, harder than stainless steel, which proved to be denser than expected. An approximate theoretical density for the product is an average of the density of PW-4m hot-pressed under the same conditions (4.01 g cm<sup>-3</sup>) and of quartz (2.65 g cm<sup>-3</sup>). The measured density (3.42 g cm<sup>-3</sup>) was slightly higher than this theoretical value (3.34 g cm<sup>-3</sup>). X-ray diffraction analysis indicated that although the major crystalline phases present in the product were quartz and the components of PW-4m, a small amount of one or more other unknown phases(s) was also present. These density and X-ray results demonstrate that partial reaction had occurred between the PW-4m and quartz during hot-pressing. In fact, this reaction is necessary in order to obtain a hard dense product. When PW-4m and quartz are hot-pressed separately, the pellets produced are easily crumpled to a powder.

Other important properties of these hot-pressed discs include thermal stability and low leachability. The discs showed no deterioration in properties after heating at 600°C for 2 weeks. Also, no deterioration of the discs or measurable weight loss was recorded after distilled water treatment at 100°C for 2 h and at 45°C for 2 weeks. Of course, long term leachability tests will have to be performed in order to fully evaluate the solubility characteristics of the products.

The equipment necessary for this process is not a great deal more complex than that of the melt processes. The devitrification and phase separation problems with the melt process are eliminated. The matrix material, quartz, is one of least expensive and most available substances. Hot-pressing, although costly, is becoming a routine processing technique in the ceramic industry, especially in the production of ultra-hard materials, and, if its use leads to increased stability of stored radioactive wastes its cost may be justified.

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## *Preparation of iron sesquioxide glasses by ultra-fast quenching from the melt ("splat-cooling")*

The technique of ultra-fast quenching materials from the melt ("splat-cooling"), developed in 1960 by Duwez [1] has been extensively applied to metals and alloys [2-4]. The very high cooling rates available with this technique (as high as

10<sup>7</sup> to 10<sup>8</sup>°C sec<sup>-1</sup>) have led to the formation of new metastable crystalline phases [4] as well as amorphous or microcrystalline structures [5-7]. It is interesting to note that the method has been very rarely applied to ultra-fast quenching of oxides or mixtures of oxides, e.g. [8, 9].

This letter describes splat-cooling of binary mixtures of oxides based mainly on Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and Ga<sub>2</sub>O<sub>3</sub> which has led to the formation