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FRENCH R7T7 NUCLEAR WASTE GLASS

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OF FRENCH R7T7 NUCLEAR WASTE GLASS**

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## INTRODUCTION

The need to solidify dispersible high-level nuclear wastes (liquids, sludge or ashes) in a stable form is acknowledged world-wide, and a large number of matrices have been proposed: borosilicate glass, phosphate glass, vitroceraamics, synrocs, super calcine, tailored ceramics, etc. Borosilicate glasses have been widely selected to solidify fission product solutions generated by fuel reprocessing, as they represent one of the best tradeoffs between fabrication engineering constraints and the final material quality.

The choice of a glass composition to vitrify fission product solutions is a tradeoff between technological feasibility requirements and the search for a material ensuring the highest possible degree of containment. From a technological standpoint the principal limitations concern the glass melting temperature, its casting viscosity, volatility and corrosiveness for process equipment. The material itself must be capable of digesting in its vitreous structure virtually all of the thirty-odd chemical elements found in fission product solutions, exhibit low crystallization and high resistance to aqueous corrosion. The glass composition must therefore be adapted to each type of fission product solution.

The "R7T7" glass composition indicated in Table 1 has been selected in France to vitrify fission product solutions generated by reprocessing PWR fuels from French nuclear power stations. It is the result of several years of research to optimize its properties. This paper describes the physicochemical properties of this glass and discusses its long-term stability in a geological disposal context.

Table 1 - R7T7 glass composition (wt%)

SiO <sub>2</sub>	45.48	MoO <sub>3</sub>	1.70
Al <sub>2</sub> O <sub>3</sub>	4.91	MnO <sub>2</sub>	0.72
B <sub>2</sub> O <sub>3</sub>	14.02	CoO	0.12
Na <sub>2</sub> O	9.86	AgO <sub>2</sub>	0.03
CaO	4.04	CdO	0.03
ZnO	2.50	SnO <sub>2</sub>	0.02
LiO <sub>2</sub>	1.98	Sb <sub>2</sub> O <sub>3</sub>	0.01
Fe <sub>2</sub> O <sub>3</sub>	2.91	TeO <sub>2</sub>	0.23
P <sub>2</sub> O <sub>5</sub>	0.28	BaO	0.60
ZrO <sub>2</sub>	2.65	La <sub>2</sub> O <sub>3</sub>	0.90
NiO	0.74	Ge <sub>2</sub> O <sub>3</sub>	0.93
Cr <sub>2</sub> O <sub>3</sub>	0.51	Pr <sub>2</sub> O <sub>3</sub>	0.44
SrO	1.42	Nd <sub>2</sub> O <sub>3</sub>	1.59
Y <sub>2</sub> O <sub>3</sub>	0.20	ThO <sub>2</sub>	0.33

## PHYSICOCHEMICAL PROPERTIES

The selection of a nuclear glass involves a major characterization program to provide a comprehensive description of its properties. This work is done on nonradioactive laboratory glass specimens, radioactive glass specimens prepared in laboratory hot cells, and nonradioactive industrial glass samples fabricated in the full-scale continuous vitrification prototype facility. Additional measurements are planned on actual radioactive glass samples fabricated in the R7 facility at La Hague<sup>[1]</sup>. These characterization studies are supplemented by "sensitivity" studies to investigate the effects of minor composition variations on the glass properties.

### Homogeneity

The vitrified material is a fine black glass that is largely homogeneous at electron microprobe scale. Some rare heterogeneities include alkali metal or alkaline earth molybdates ("yellow phase"); some RuO<sub>2</sub> crystals and 10-20 μm inclusions consisting mainly of rhodium and palladium indicating that platinoids are not readily incorporated in the glass matrix; some bubbles, unmelted inclusions and chromites consisting of Fe, Ni and Cr, due mainly to corrosion of the Inconel 601 melting pot. These homogeneities account for less than 1 vol% of the glass, confirming the excellent glass homogeneity.

### Physical and Mechanical Properties

Table 2 summarizes the principal physical and mechanical properties measured on simulated R7T7 glass.

Table 2 - Physical and mechanical properties of R7T7 glass

PROPERTY	VALUE	MEASUREMENT METHOD
Vitreous transition temperature	T <sub>g</sub> = 502°C	Differential thermal analysis
Viscosity at 1100°C	ν = 8 Pa·s (80 Poises)	Falling sphere viscometer
Density	ρ = 2.75 g·m <sup>-3</sup>	Water pycnometer
Linear expansion coefficient between 25 and 300°C	(α <sub>m</sub> ) <sub>25</sub> <sup>300</sup> = 8.3 × 10 <sup>-6</sup> °C <sup>-1</sup>	Adamel dilatometer
Young's modulus	E = 8.4 × 10 <sup>10</sup> N·m <sup>-2</sup>	Bending resonance method
Fracture toughness	K <sub>IC</sub> = 0.95 MPa·m <sup>1/2</sup>	Double torsion and short-rod test <sup>[2]</sup>
Thermal conductivity at room temperature	λ = 1.09 W·m <sup>-1</sup> °C <sup>-1</sup>	Hot wire method

The physical properties of this nuclear glass are thus similar to those of many industrial glasses, with mechanical properties exceeding those of pyrex or window glass.

## Chemical Durability

The inherent resistance of a glass to aqueous corrosion can be characterized by its initial dissolution rate in pure water. This parameter may be assessed either by dynamic experiments in which the glass specimen is in contact with continuously renewed water, or by static experiments in which the initial slope of the release rate curve for the major glass elements is measured. The initial corrosion rate,  $r_0$ , for R7T7 glass is typically  $2 \text{ g}\cdot\text{m}^{-2}\cdot\text{d}^{-1}$  at  $100^\circ\text{C}$ .

The evolution of this initial dissolution rate with the temperature follows an Arrhenius curve between  $50$  and  $250^\circ\text{C}$ , with a  $60 \text{ kJ}\cdot\text{mol}^{-1}$  activation energy<sup>[3]</sup>. The initial dissolution rate also varies with the solution pH, increasing by about an order of magnitude when the pH rises from  $7$  to  $10$ <sup>[4]</sup>.

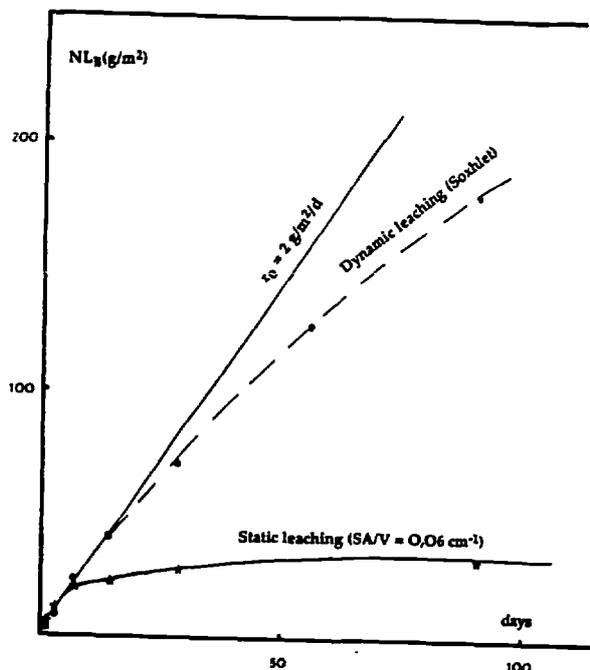


Figure 1 - Normalized mass loss versus time

Table 3 shows the normalized mass loss for R7T7 glass measured during a static test at  $90^\circ\text{C}$  with a glass surface area to solution volume (SA/V) ratio of  $50 \text{ m}^{-1}$ , compared with the results obtained under similar conditions for a “window glass” and a Pyrex glass formulation.

Table 3 - Normalized mass loss ( $\text{g}\cdot\text{m}^{-2}$ ) for R7T7 glass, soda-lime “window glass” and pyrex glass (static test at  $90^\circ\text{C}$ ;  $\text{SA}/\text{V} = 50 \text{ m}^{-1}$ )

Time (days)	Soda-lime glass			R7T7 glass			Pyrex glass		
	$\Delta m$	Si	Na	$\Delta m$	Si	Na	$\Delta m$	Si	Na
1	0.75	1.05	0.93	0.46	0.79	0.71	0.10	0.02	0.05
3	4.08	4.11	5.04	1.29	1.43	1.37	0.20	0.14	0.85
7	5.55	5.60	11.95	0.85	1.77	1.76	0.35	0.13	0.90
14	6.01	5.85	11.58	1.60	2.07	2.11	0.41	0.35	1.27
28	5.58	7.47	8.02	1.37	2.06	2.30	0.50	0.61	1.17
56	5.82	7.12	9.09	1.80	2.87	4.17	0.64	0.51	1.15

The normalized mass loss may be calculated either from the specimen mass loss:

$$NL(m) = \frac{\Delta m}{S}$$

where  $\Delta m$  is the specimen mass loss in grams  
and  $S$  is the specimen surface area in  $m^2$

or from the element analysis in the leaching solution:

$$NL(i) = \frac{C_i \times V_i}{x_i} \cdot \frac{1}{S}$$

where  $NL(i)$  is the normalized mass loss ( $g \cdot m^{-2}$ ) for element  $i$ ;  $C_i$  the concentration ( $g \cdot l^{-1}$ ) of element  $i$  in solution;  $V_i$  the solution volume (l);  $x_i$  the concentration of element  $i$  in the glass; and  $S$  the specimen surface area ( $m^2$ ).

Table 3 shows that the inherent durability of R7T7 nuclear glass is mid-way between that of ordinary window glass and that of a pyrex glass. However, the initial dissolution rate of a glass is not the principal factor affecting the long-term alteration kinetics.

## LONG-TERM STABILITY

As shown in the preceding section, the structure and properties of borosilicate glasses produced by vitrification of fission product solutions are not fundamentally different from those of conventional glass formulations. The main difference is the large number of component elements, some of which are radioactive. The problem is to determine whether this radioactivity will not induce long-term modifications of the glass structure or properties that could affect the repository conditions, and to what extent laboratory-scale aqueous corrosion tests can be extrapolated to periods covering thousands of years.

### Thermal Stability

Being radioactive, nuclear glasses release heat. The thermal power of R7T7 glass is about  $7 W \cdot kg^{-1}$  at the moment of fabrication, dropping to  $1 W \cdot kg^{-1}$  after 50 years and  $0.05 W \cdot kg^{-1}$  after 300 years. Interim storage facilities in ventilated shafts have been designed to accommodate the glass packages for several decades after fabrication in order to minimize the temperature rise.

Significant crystallization could prove detrimental to the glass integrity and containment properties by generating soluble phases, by increasing the solubility of the residual glass matrix or by inducing porosity in an initially homogeneous matrix. Considerable research has been carried out for this reason, and the composition of nuclear waste glasses has been adjusted to ensure a very low final crystallized fraction. An incipient crystallization temperature below which no crystal growth is observed (typically  $600-620^\circ C$  for R7T7 glass) is used to set the maximum storage temperature with a suitable safety margin. A maximum crystallization temperature (typically  $780^\circ C$  for R7T7 glass) has also been determined. Extended testing at this temperature provides a means for determining the maximum crystallized fraction liable to form in long-term repository conditions: this fraction increases with the amount of insoluble platinoids and chromites in the glass, but never exceeds a few volume percent for R7T7 glass and has no significant effect on glass properties<sup>(1)</sup>.

## Radiation Stability

Although  $\alpha$ -emitters are present in only small amounts in fission product glasses (0.3 wt% excluding uranium) they constitute by far the greatest potential damage source. Because of the high energy released over a short path during  $\alpha$  decay and the long half-lives of these nuclides the cumulative number of atomic displacements due to  $\alpha$ -emitters is two orders of magnitude higher over 1000 years than for  $\beta$  disintegration and spontaneous fission, as shown in Table 4. Particular attention has therefore been given to investigating the effect of  $\alpha$  decay in nuclear glasses.

Table 4 - Radiation dose of R7T7 glass as a function of time

Radiation	Cumulated disintegration per gram of glass	Atomic displacements per gram of glass	Deposited energy in Gray	
			After 10 <sup>3</sup> Y	After 10 <sup>7</sup> Y
$\alpha$	$\sim 1.7 \cdot 10^{18}$	$\alpha : 2 \text{ to } 3 \cdot 10^{20}$ $\text{RN} : 2 \text{ to } 3 \cdot 10^{21}$	$1.4 \cdot 10^9$	$5.6 \cdot 10^{10}$
$\beta$	$\sim 4.3 \cdot 10^{19}$	$\sim 4 \cdot 10^{19}$	$2.4 \cdot 10^9$	$2.7 \cdot 10^9$
$\gamma$	$\sim 2 \cdot 10^{19}$	$\ll 2 \cdot 10^{19}$	$\ll 2 \cdot 10^9$	$\ll 2 \cdot 10^9$

Glass samples doped with small quantities of actinides with high specific activities (<sup>241</sup>Am, <sup>238</sup>Pu, <sup>244</sup>Cm or <sup>242</sup>Cm) sustain in only a few years the same number of  $\alpha$  disintegrations per gram as the actual vitrified waste glass in several thousand years. Actinide-doped glasses have been fabricated in many countries<sup>[5]</sup> (notably the USA, France, Germany and the United Kingdom) and have logged cumulative doses exceeding  $5 \times 10^{18}$   $\alpha$  disintegrations per gram, simulating 100 000 years of irradiation of an actual nuclear glass. The principle results<sup>[6]</sup> are summarized below.

- **Density changes.** The glass density diminishes slightly, indicating minor swelling under irradiation. However, the volume change stabilizes at about 0.5% for an integrated dose of some  $2 \times 10^{18}$   $\alpha \cdot \text{g}^{-1}$  and remains constant thereafter. This suggests that a state of equilibrium is reached above this dose between the number of defects created in the glass and the effects of spontaneous annealing.
- **Leaching.** No significant variation in the glass dissolution rate was observed at integrated doses of up to  $5 \times 10^{18}$   $\alpha \cdot \text{g}^{-1}$ .
- **Mechanical properties.** The glass microhardness diminishes and its fracture toughness increases when the dose exceeds  $10^{18}$   $\alpha \cdot \text{g}^{-1}$ . As for swelling, this variation stabilizes above  $2 \times 10^{18}$   $\alpha \cdot \text{g}^{-1}$ . This effect favors glass stability by increasing its crack propagation resistance and deformability.

Thus, unlike most ceramic materials (Synroc, zirconolites, etc.), nuclear glass shows no adverse effects of irradiation damage.

## Aqueous Corrosion

### Basic Mechanisms

Aqueous corrosion will be the principal long-term glass alteration factor. Numerous studies have been conducted to identify the basic mechanisms of this corrosion in order to develop long-term behavior models. These mechanisms can be summarized as follows.

- **Hydration and interdiffusion.** After an initial glass hydration step, a thin reaction zone is created within which a  $H^+ \rightleftharpoons$  alkali metal interdiffusion profile is formed. This is the principal phenomenon involved in acid media, resulting in selective dissolution of the glass component elements.
- **Silica network dissolution.** In most cases, however, the release of alkali metals from the glass quickly results in an alkaline pH in which the dissolution kinetics of the silicated network are faster than the interdiffusion kinetics. This forms a hydrated glass front that penetrates into the pristine glass at a rate  $r$ . Aagaard and Helgeson<sup>[7]</sup> showed that for most silicated minerals this rate is controlled by the silicic acid activity in solution according to a first-order relation. Hence the relation<sup>[8]</sup>:

$$r = r_0 \left( 1 - \frac{a_{Si}}{a^*_{Si}} \right) \quad \text{where:}$$

$r$  is the rate of progression of the corrosion front,  
 $r_0$  the initial dissolution rate,  
 $a_{Si}$  the  $H_4SiO_4$  activity in solution  
 and  $a^*_{Si}$  the  $H_4SiO_4$  activity at saturation

- **Surface layer formation.** As the glass silicate network dissolves, all the glass components are hydrated. Many of them, however, including iron, zirconium, the rare earths and the actinides, form hydroxides, hydrosilicates or hydrated oxides which are highly insoluble in alkaline media. These elements do not enter solution, but recombine *in situ* to form an amorphous surface "gel" layer. Glass dissolution remains nonselective (all the glass elements are hydrated at the same time) but becomes incongruent (not all the elements enter solution).

The formation of this gel affects the glass corrosion kinetics in two ways. First, it constitutes a diffusion barrier that impedes the release of silicic acid formed at the pristine glass surface; an  $H_4SiO_4$  activity gradient appears in the interstitial water of the gel, and the activity at the pristine glass/gel interface ( $a_{Si}^{interf}$ ) must be taken into account in the kinetics (cf. Figure 2):

$$r = r_0 \left( 1 - \frac{a_{Si}^{interf}}{a^*_{Si}} \right)$$

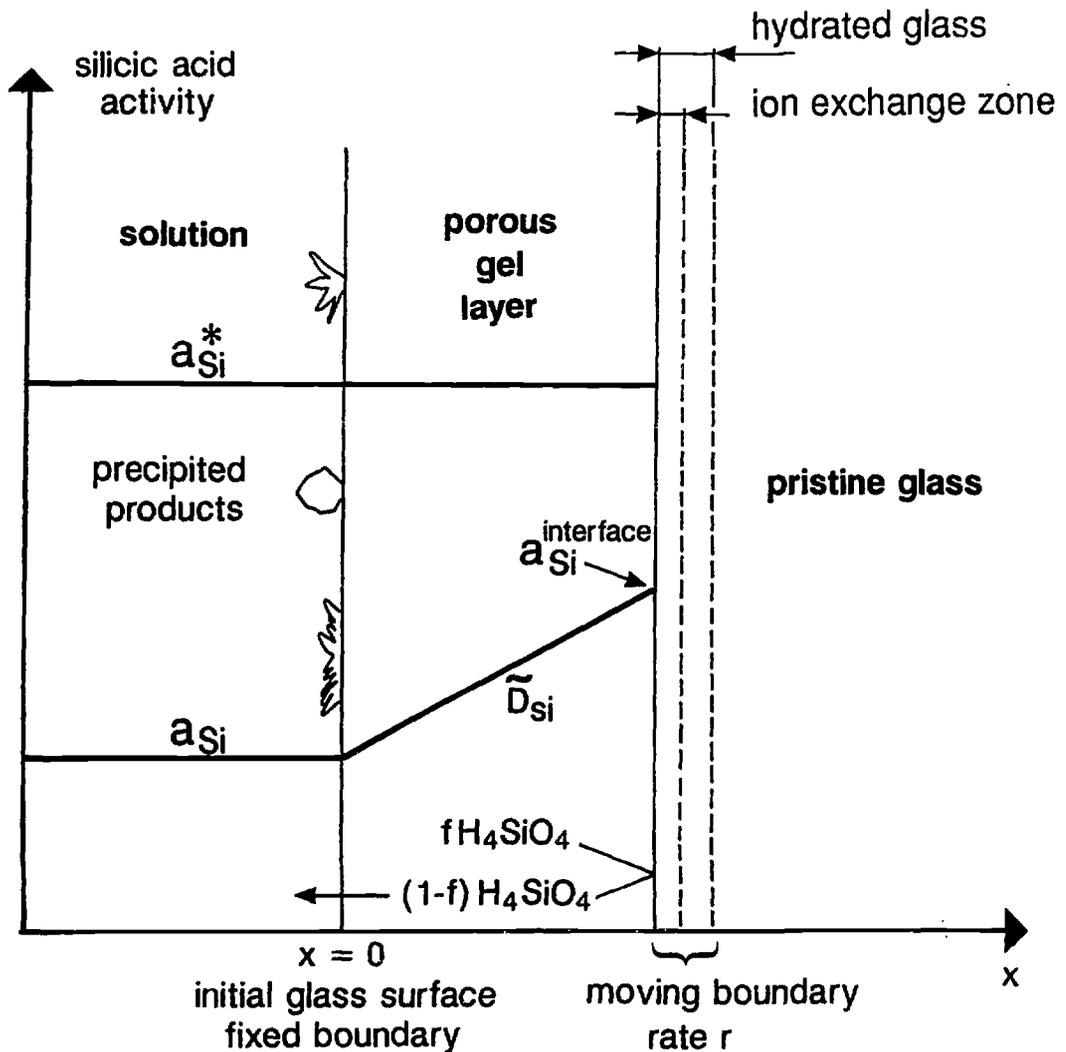


Figure 2 - Schematic representation of the principal corrosion mechanisms

Second, the gel also traps part of the hydrated silica. The silicon fraction,  $f$ , chemisorbed by the gel shows a virtually linear relation with the silicon concentration in solution<sup>[9]</sup>:

$$f\% = \alpha[Si] + \beta.$$

At 90°C, as saturation conditions are approached, about 75% of the hydrated silicon remains trapped in the gel layer.

## Modeling

Figure 3 shows the evolution of the silicon and boron concentrations in solution during a series of 1-year static leaching experiments at 90°C with a glass surface area to solution volume (SA/V) ratio of 50 m<sup>-1</sup>. These concentrations are compared with the values predicted by a numerical model taking the following factors into account.

1. The glass dissolution rate is controlled by the first order relation:

$$r = r_0 \left[ 1 - \frac{C_{\text{Si}}^{\text{interf}}}{C^*_{\text{Si}}} \right]$$

(Considering the low ionic strength of the solutions, the activities are assimilated with the concentrations).

2. The dissolved silicon concentration at the interface between the gel and the pristine glass,  $C_{\text{Si}}^{\text{interf}}$ , is calculated at each iteration with allowance for a constant H<sub>4</sub>SiO<sub>4</sub> diffusion coefficient in the gel ( $\tilde{D} = 10^{-2} \text{ cm}^2 \cdot \text{d}^{-1}$  at 90°C) and a linear gradient in the gel.
3. The  $C^*$  value is recalculated at each step in the dissolution process for the experimentally measured pH, with allowance for dissociation of silicic acid according to the following equations:

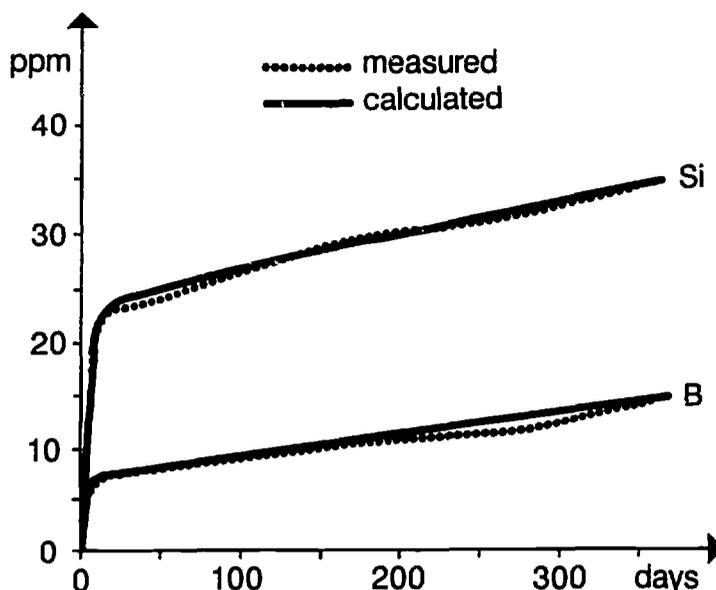


Figure 3 - Calculated and measured concentrations for silicon and boron

4. As the glass corrodes only a fraction,  $f$ , of the hydrated silicon enters solution. This fraction varies with the silicon concentration in solution according to the expression:

$$f = 0.10 + 0.115[\text{Si}] \quad (\text{where } [\text{Si}] \text{ is expressed in } \text{mg}\cdot\text{l}^{-1}).$$

Under these conditions an excellent fit with the experimental results is obtained. A first-order law may thus be applied to nuclear glasses, provided allowance is made for a number of additional phenomena related to the formation of the surface layer.

### Long-Term Evolution

Under purely static conditions or with very low renewal rates, the corrosion rate therefore drops considerably. Various experiments<sup>[10]</sup> have shown that if a residual corrosion rate exists, it is less than  $2 \times 10^{-4} \text{ g}\cdot\text{m}^{-2}\cdot\text{d}^{-1}$ , i.e. about four orders of magnitude lower than the initial corrosion rate. At this rate more than a million years would be required to alter the glass block: the corrosion kinetics under saturation conditions are therefore not a problem for the long-term chemical stability of the glass.

The major parameters will be the solution renewal rate, and any phenomena liable to decrease the  $\text{H}_4\text{SiO}_4$  activity below the saturation value,  $a^*_{\text{Si}}$ . Two essential phenomena can maintain this activity below its saturation value: precipitation of silicated minerals, and consumption of silicon released from the glass by environmental materials.

- *Role of newly-formed phases.* Many surface studies have been conducted to identify the crystallized phases liable to form during glass alteration. Practically no crystallized phases have been evidenced at 90°C, and hydrothermal alteration experiments have been performed at temperatures up to 250°C<sup>[11]</sup>. The major alteration products that form at high temperatures are clays (smectites), aluminosilicates (zeolites) and hydrated calcium silicates.

Complex geochemical codes must be used to determine whether these minerals are liable to control the long-term silicon activity in solution. The initial geochemical model of R7T7 glass now being developed<sup>[12]</sup> shows that the minerals identified at the surface of the glass tend to delay the onset of saturation, but do not control the long-term silicon activity in solution. The formation of these minerals is therefore not expected to lead to a subsequent renewal of R7T7 glass corrosion.

- *Influence of environmental materials.* A large number of R7T7 glass alteration experiments conducted with a variety of environmental materials<sup>[13]</sup> have shown that sand, granite or salt have a secondary effect on the glass alteration kinetics. Conversely, most clays act as veritable silica sinks, consuming silicon released by glass corrosion and thus delaying or preventing the rise to saturation conditions. The result is sustained corrosion of the glass at a higher rate than in the other materials.

## CONCLUSION

The French R7T7 nuclear glass composition was carefully selected to allow incorporation of some thirty different oxides found in fission product solutions. The resulting glass exhibits very low crystallization, and its physical and chemical properties are very similar to those of standard industrial glasses.

From the standpoint of self-irradiation, nuclear glasses have been shown to withstand  $\alpha$  doses corresponding to several hundred thousand years under repository conditions without any significant modification of their containment properties.

Predicting the long-term behavior of fission product glasses subjected to aqueous corrosion is no doubt the most difficult aspect of the problem. There are no simple means of simulating glass alteration in a deep underground geological formation over periods of several tens of thousands of years: predictions are necessarily based on mathematical models. A substantial research effort has been undertaken to identify all the basic corrosion mechanisms liable to control long-term alteration. These mechanisms are now relatively well understood, and provide the basis for developing the indispensable models.

It may already be affirmed that realistic storage conditions exist under which glass alteration occurs at a very slow rate, and can fulfill its role as the first containment barrier for several tens of thousands of years. In conjunction with other engineered barriers and the very long-term retention capacity of the geological repository site itself, underground disposal of vitrified nuclear waste can be considered a safe solution for future generations.

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