

Uranium encapsulation into glass

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JOINT ICTP-IAEA INTERNATIONAL SCHOOL ON
NUCLEAR WASTE VITRIFICATION

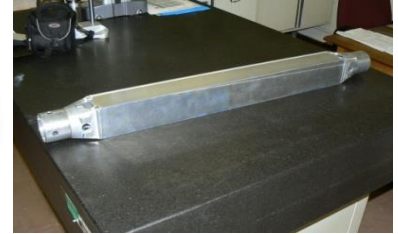
ICPT, TRIESTE

23 SEPTEMBER 2019 TO 27 SEPTEMBER 2019

1. Enriched Uranium problem

MTR Reactor

- Old reactor spent fuel (MTR fuel)
- $U(m) \geq 45\%$



Enriched Uranium

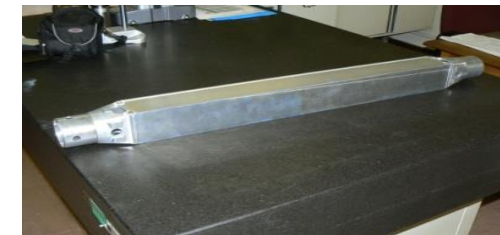
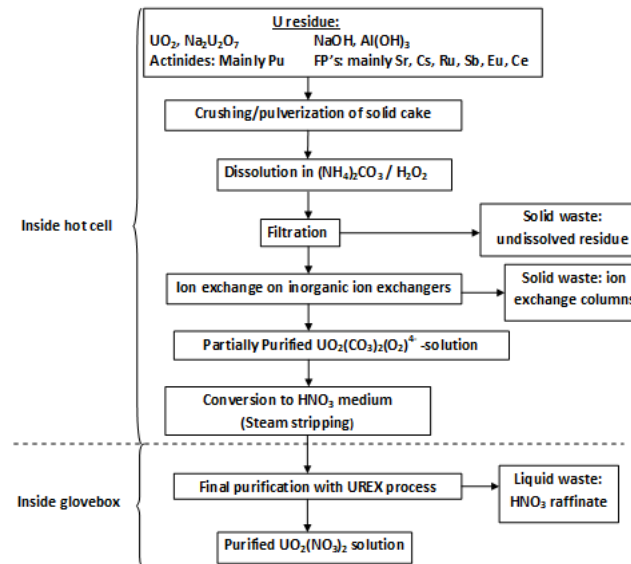
Medical isotope production

- Residue from dissolved target plates
- $U(m) \geq 45\%$



2. Alkaline reprocessing of MTR fuel (Research Reactor)

Country	Reactor (Age)	production per week (6dCi)	Processing Facility	Processing facility capacity (6dCi)	target type	processing
Canada	NRU (57)	4680	MDS Nordion	7200	HEU	acid (HNO_3)
Netherlands	HFR (53)	4680	Covidien	3500	HEU	alkaline (NaOH)
Belgium	BR-2 (53)	7800	IRE	2500	HEU	alkaline (NaOH)
South Africa	Safari-1 (49)	3000	NTP	3000	LEU	alkaline (NaOH)
Australia	OPAL (8)	1000	ANSTO Health	1000	LEU	alkaline (NaOH)
Argentina	RA-3 (47)	400	CNEA	900	LEU	alkaline (NaOH)
France	OSIRIS (48)	1200	IRE		HEU	
Czech Republic	LVR-15 (57)	2800	IRE		HEU	
Poland	MARIA (40)	1920	Covidien		HEU	
Russian Federation	RIAR:three (40)	900	IPPE	unknown	HEU	acid (HNO_3)



Alkaline re-processing chemistry of MTR spent fuel and Mo-99 residue from target plates identical

Schematic representation of the alkaline route to recover uranium bases on residue from Mo-99 target plates

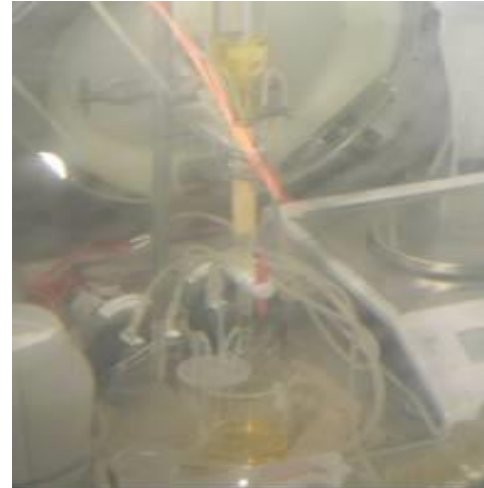
2. Alkaline reprocessing of MTR fuel (Research Reactor)



Dissolving step



Residue from
dissolving (NaOH)
process



Isolating uranium from
solid residue



High enriched Uranium
waste to be conditioned

3. Uranium waste to be conditioned for disposal

Waste characterization

Nuclide	Bq/g residue
Co-60	1.99E+04
Nb-95	2.95E+04
Zr-95	1.90E+04
Ru-106	4.24E+05
Sb-125	1.45E+06
Cs-137	3.22E+06
Ce-144	8.90E+05
Pr-144	7.86E+05
Eu-154	7.09E+04
Eu-155	2.46E+06
Fe	99.7 % m/m
Al	83 % m/m
U	98 %

Approximate solubility limits

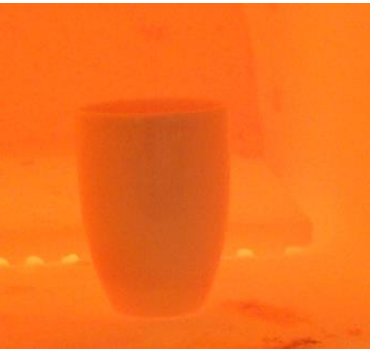
Element	Solubility limit, wt. %
Al, Si, P, Pb	25
Li, B, Na, Mg, K, Ca, Fe, Zn, Rb , Sr, Cs, Ba, Fr, Ra, U	15 - 25
Ti, Cu, F, La, Ce, Pr, Nd, Gd , Th, Bi, Zr	5 - 15
Mn, Cr, Co, Ni, Mo	3 - 5
C, S, Cl, As, Se, Tc, Sn, Sb, Te	1 - 3
H, He, N, Ne, Ar , Br, Kr, Ru, Rh, Pd, Ag, I, Xe , Pt, Au, Hg, Rn	< 0.1

Literature search

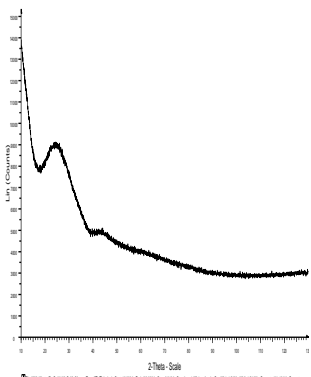
Name	Composition										Pb	Others
	SiO ₂	Na ₂ O	MgO	CaO	K ₂ O	FeO	Al ₂ O ₃	SiO ₂	Na ₂ O	MgO		
BS1	5.35	3.76	8.23	-	-	0.68	0.48	21.87	0.53	43.5	0.23	-
BS10	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS11	5.4	3.12	8.34	-	-	0.73	0.48	21.87	0.53	43.5	0.23	9.79
BS12	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS13	5.4	3.12	8.34	-	-	0.73	0.48	21.87	0.53	43.5	0.23	-
BS14	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS15	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS16	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS17	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS18	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS19	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS20	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
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BS149	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS150	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS151	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS152	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS153	5.4	3.12	-	-	-	-	-	15.5	-	-	-	-
BS154	5.4	3.12	-	-	-	-	-	15				

4. Initial Uranium vitrification studies

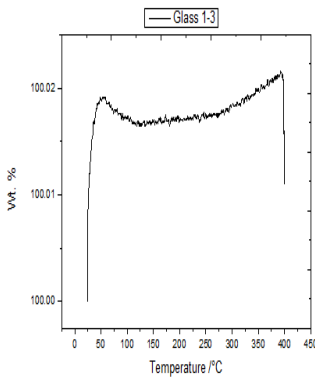
Element/ Nuclide	Surrogate compound	Mass of surrogate (g)
7% Fe	Fe ₂ O ₃	0.100
1.5% Cr	Cr ₂ O ₃	0.022
1% Al	Al(OH) ₃	0.029
U(VI)	Na ₂ U ₂ O ₇	0.736
U(IV)	UO ₂	0.111
⁶⁰ Co	CoO	6.30x10 ⁻⁸
⁹⁵ Nb	Nb ₂ O ₅	1.26x10 ⁻⁹
⁹⁵ Zr	ZrO ₂	2.45x10 ⁻³
¹⁰⁶ Ru	RuO ₂	9.14x10 ⁻⁴
¹²⁵ Sb	Sb ₂ O ₃	3.41x10 ⁻⁶
¹³⁷ Cs	CsOH.H ₂ O	1.03x10 ⁻⁵
¹⁴⁴ Ce/ ²³⁸ Pu	CeO ₂	1.59x10 ⁻³
¹⁴⁴ Pr	Pr ₆ O ₁₁	6.15x10 ⁻⁴
¹⁵⁴ Eu/ ¹⁵⁵ Eu	Eu ₂ O ₃	2.13x10 ⁻⁵
⁹⁰ Sr	SrO	5.73x10 ⁻⁴
Se	SeO ₂	3.72x10 ⁻⁵
Y	Y ₂ O ₃	3.35x10 ⁻⁴
Mo	MoO ₃	6.34x10 ⁻⁴
Rh	Rh ₂ O ₃	2.45x10 ⁻⁴
Pd	PdO	1.24x10 ⁻⁴
Ag	Ag ₂ O	2.38x10 ⁻⁶
Cd	CdO	5.32x10 ⁻⁶
Sn	SnO ₂	1.34x10 ⁻⁵
Te	TeO ₂	1.81x10 ⁻⁴
La	La ₂ O ₃	6.42x10 ⁻⁴
Nd	Nd ₂ O ₃	2.13x10 ⁻³
Sm	Sm ₂ O ₃	3.72x10 ⁻⁴
Gd	Gd ₂ O ₃	4.99x10 ⁻⁶
Total surrogate mixture mass		1.010



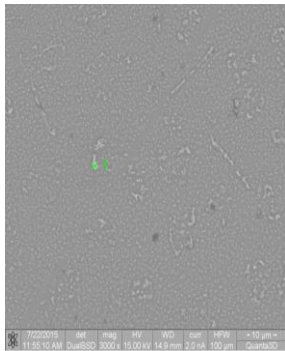
BUTTON MANUFACTURING



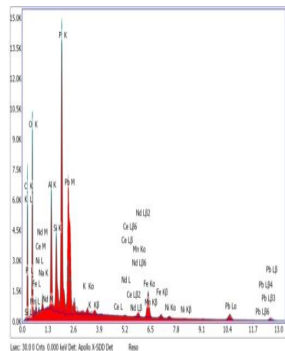
XRD



TGA



SEM



EDX

Element	Upper limit on leach rate (g/m ² /d)
B	0.06
Na	0.13
Al	0.01
Si	0.03
Ca	0.01
Cr	0.00
Fe	0.00
Ni	0.00
Zn	0.00
Se	0.00
Sr	0.00
Zr	0.00
Ru	0.00
Ag	0.00
Sb	0.08
Cs	0.03
Ba	0.00
Ce	0.00
Nd	0.00
U	0.00

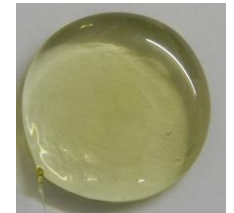
PCT-leaching

GLASS FRIT (containing 85% Uranium)

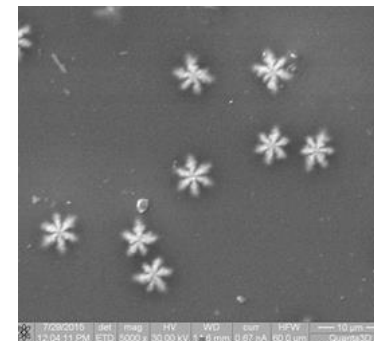
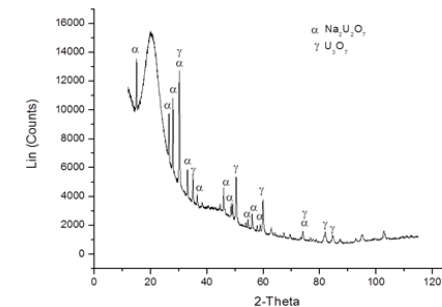
CHARATERIZATION

4. Initial Uranium vitrification studies

Properties	WS3-G-03	WS3-G-04	WS3-G-05	WS3-G-06	WS3-G-07
Processing Conditions	Melt	Melt	Melt	Melt	Melt
Process Conditions	1200°C air	1200°C air	1200°C air	1300°C air	1300°C air
Target Phases	Sodium aluminoborosilicate-3	Sodium aluminoborosilicate-4	Sodium aluminoborosilicate-5	Phosphate P_2O_5 : PbO (Average 1:1)	Phosphate P_2O_5 : PbO (Average 1:1)
Waste Form Properties					
Waste Form Density (g/mL)	2.07 g/cm ³	3.25 g/cm ³	3.36 g/cm ³	3.6 g/cm ³	3.6 g/cm ³
Waste Loading (wt.% as oxide)	20	20	40	10	20
Compressive Strength	29 Mpa	28.5 Mpa	29 Mpa	25 Mpa	25 Mpa
Thermal Stability	<0.5%	<0.5%	<0.5%	<0.5%	<0.5%
Chemical Durability	PASS	PASS	PASS	PASS	PASS
PCT-B test at 90°C for 7 days	PASS	PASS	PASS	PASS	PASS



sodium diuranate



5. Upscaled vitrification using RF technology in modified glovebox



Special glovebox for high mass of Uranium to be vitrified

5. Upscaled conditioning using RF technology

USA Type Borosilicate glass matrix (24 wt% loading)

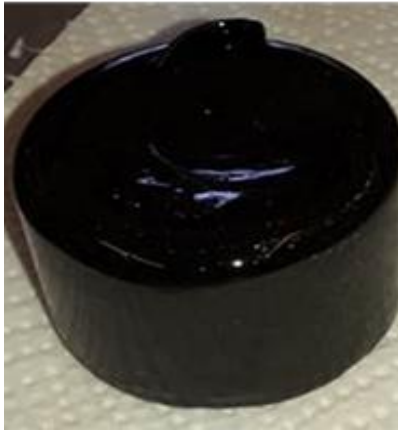
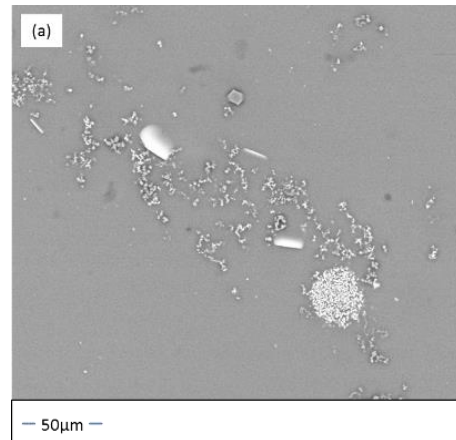
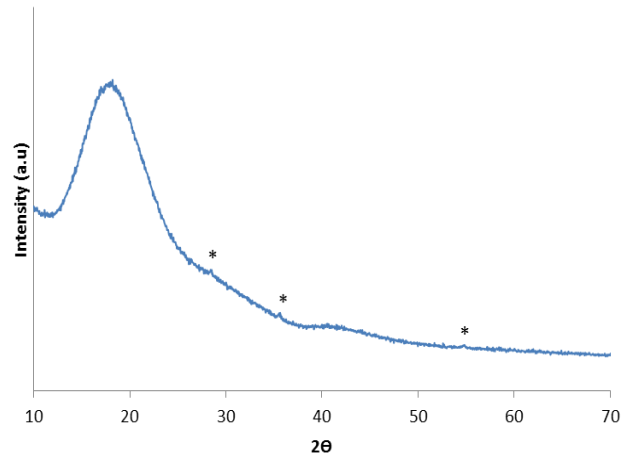


Table 3-15 Glass properties for WS1-G-01	
Physical property	Value
Diameter (mm)	41
Height (mm)	30
Mass (g)	157.70
Volume (cm ³)	49.1
Density (g/cm ³)	3.21*
Compressive strength (MPa)	34

Element	Upper limit on leach rate (g/m ² /d)
B	0.06
Na	0.13
Al	0.01
Si	0.03
Ca	0.01
Cr	0.00
Fe	0.00
Ni	0.00
Zn	0.00
Se	0.00
Sr	0.00
Zr	0.00
Ru	0.00
Ag	0.00
Sb	0.08
Cs	0.03
Ba	0.00
Ce	0.00
Nd	0.00
U	0.00



WS1-G-01 indicating a predominantly amorphous structure with very weak peaks (*) assigned to RuO_2 (JCPDS 03-065-2824).

5. Upscaled conditioning using RF technology

USA Type Borosilicate glass matrix (24 wt% loading)

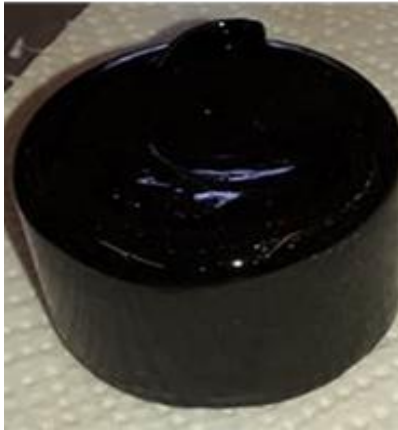
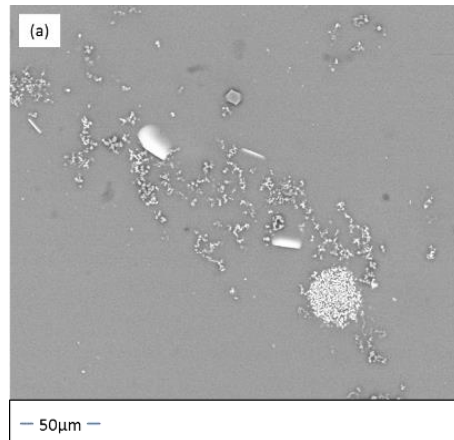
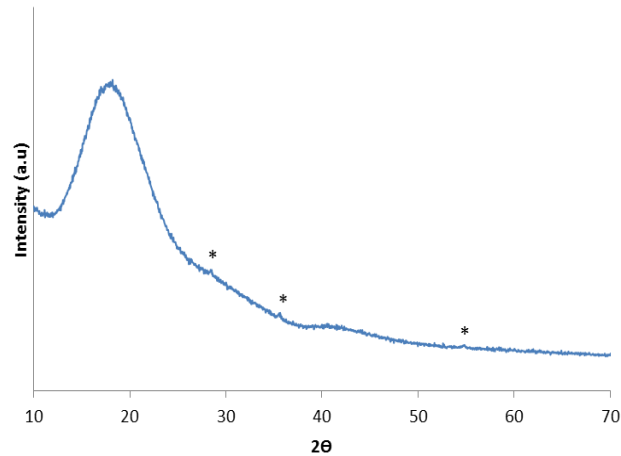


Table 3-15 Glass properties for WS1-G-01	
Physical property	Value
Diameter (mm)	41
Height (mm)	30
Mass (g)	157.70
Volume (cm ³)	49.1
Density (g/cm ³)	3.21*
Compressive strength (MPa)	34

Element	Upper limit on leach rate (g/m ² /d)
B	0.06
Na	0.13
Al	0.01
Si	0.03
Ca	0.01
Cr	0.00
Fe	0.00
Ni	0.00
Zn	0.00
Se	0.00
Sr	0.00
Zr	0.00
Ru	0.00
Ag	0.00
Sb	0.08
Cs	0.03
Ba	0.00
Ce	0.00
Nd	0.00
U	0.00



WS1-G-01 indicating a predominantly amorphous structure with very weak peaks (*) assigned to RuO_2 (JCPDS 03-065-2824).

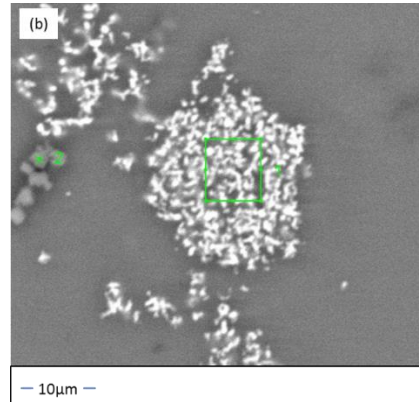
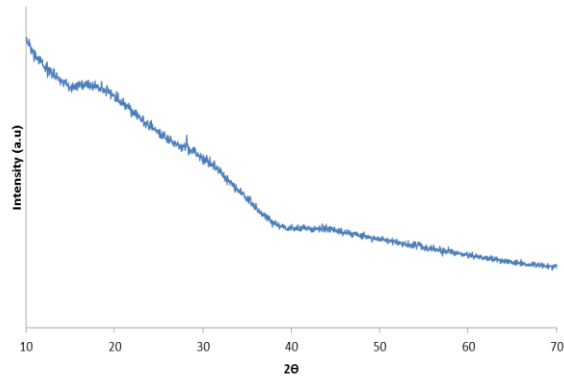
5. Upscaled conditioning using RF technology

Loffler type glass matrix (24 wt% loading)



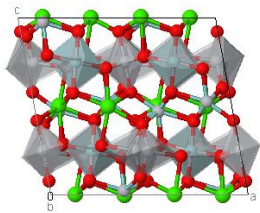
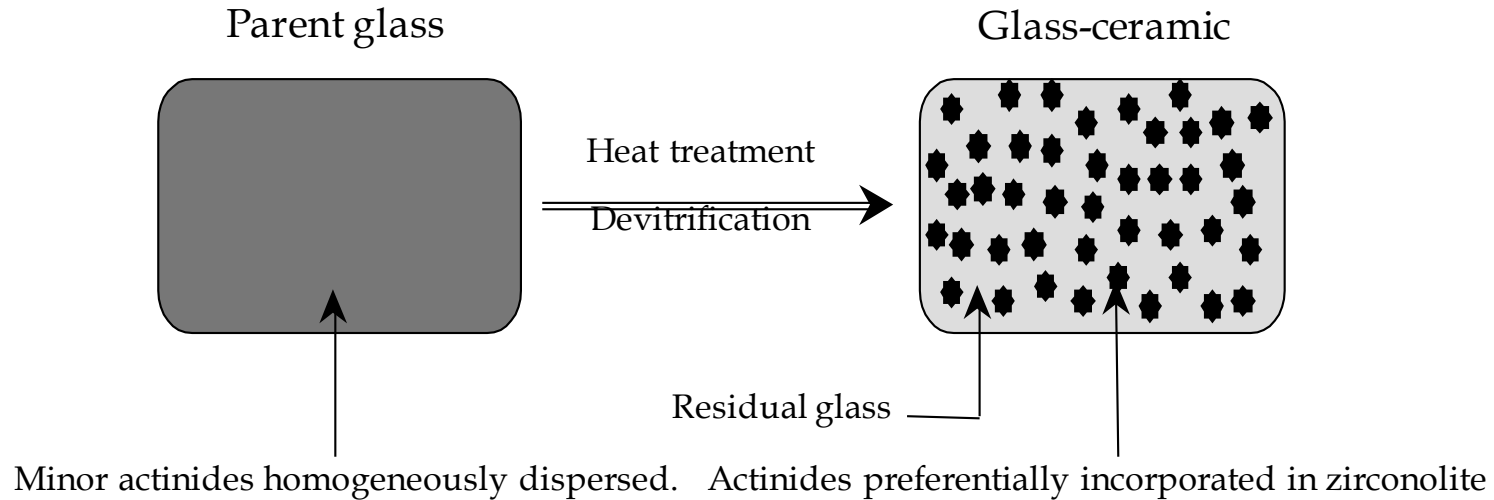
Table 3-25 Physical properties for Glass WS1-G-03	
Physical property	Value
Diameter (mm)	41
Height (mm)	34
Mass (g)	152.0
Volume (cm ³)	50
Density (g/cm ³)	3.04*
Compressive strength (MPa)	34

Element	Upper limit on leach rate (g/m ² /d)
B	0.10
Na	0.07
Al	0.01
Si	0.04
Ca	0.00
Cr	0.00
Fe	0.00
Ni	0.00
Zn	0.00
Se	0.00
Sr	0.00
Zr	0.00
Ru	0.00
Ag	0.00
Sb	0.16
Cs	0.02
Ba	0.00
Ce	0.00
Nd	0.00
U	0.00



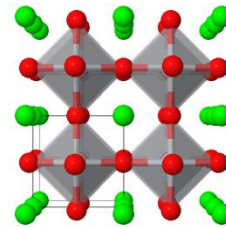
WS1-G-03 indicating a predominantly amorphous structure with very weak peaks (*) assigned to RuO_2 (JCPDS 03-065-2824).

6. Composite glasses for U and FP



Zirconolite: 30%

Act³⁺, Ln



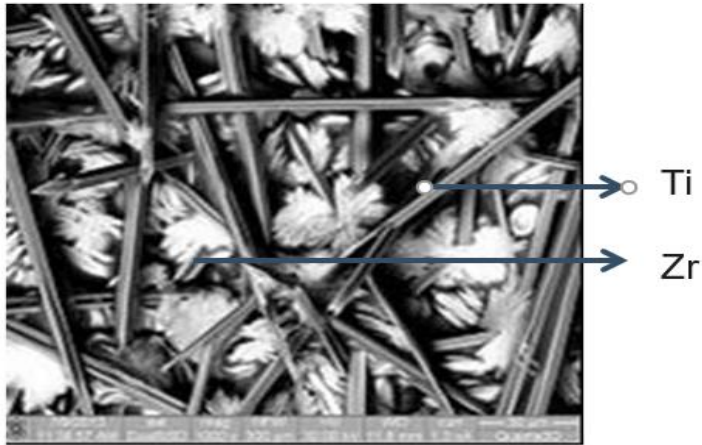
Perovskite: 20%

Na, Act⁴⁺, Ln

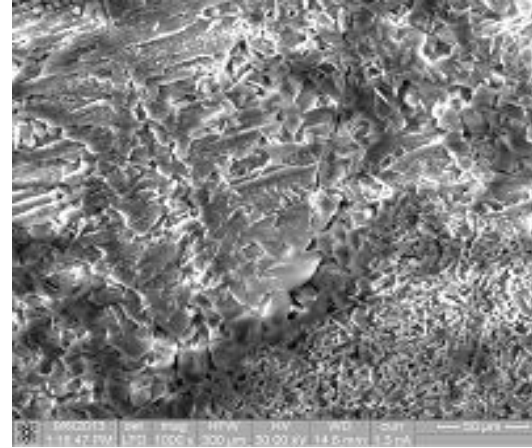
Content

1. Enriched uranium problem
2. Alkaline reprocessing of MTR fuel (Research Reactor)
3. Uranium waste to be conditioned for disposal
4. Initial Uranium vitrification studies
5. Upscaled conditioning using RF technology
6. Composite glasses for U and FP
7. Conclusion

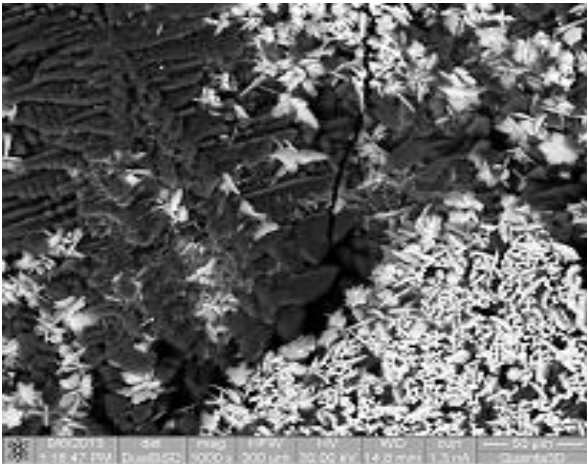
6. Composite glasses for U and FP



GCM (borosilicate glass) containing
10% Titanate : 10% Zirconia



GCM (borosilicate glass) containing
20% Titanate : 10% Zirconia



GCM (borosilicate glass) containing
10% Titanate : 20% Zirconia



GCM (lead iron phosphate glass) containing
10% Titanate : 10% Zirconia

6. Composite glasses for U and FP

Al borosilicate glass with ceramic forming components added

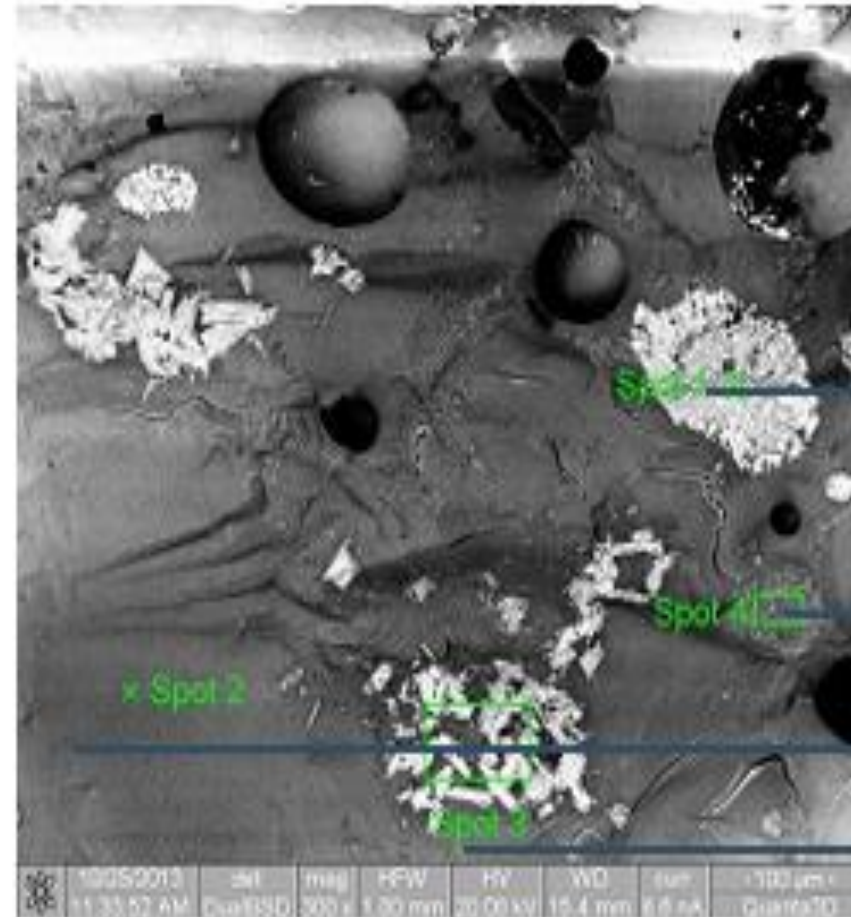
43.3 Wt% SiO_2 : 6.3 Wt% B_2O_3 :
18.8 Wt% Al_2O_3 : 2.5 Wt% CaO :
7.5 Wt% BaO : 3.4 Wt% Na_2O :

Basic chemicals to form

6.0 Wt% Perovskite

6.0 Wt% Hollandite

6.0 Wt% Zirconolite



Chemicals
representing:

Zirconolite

Perovskite

Glass

Hollandite

SEM image of Glass-ceramic matrix:300x Mag

6. Composite glasses for U and FP

Al borosilicate glass with real ceramic-forming components and two FP's

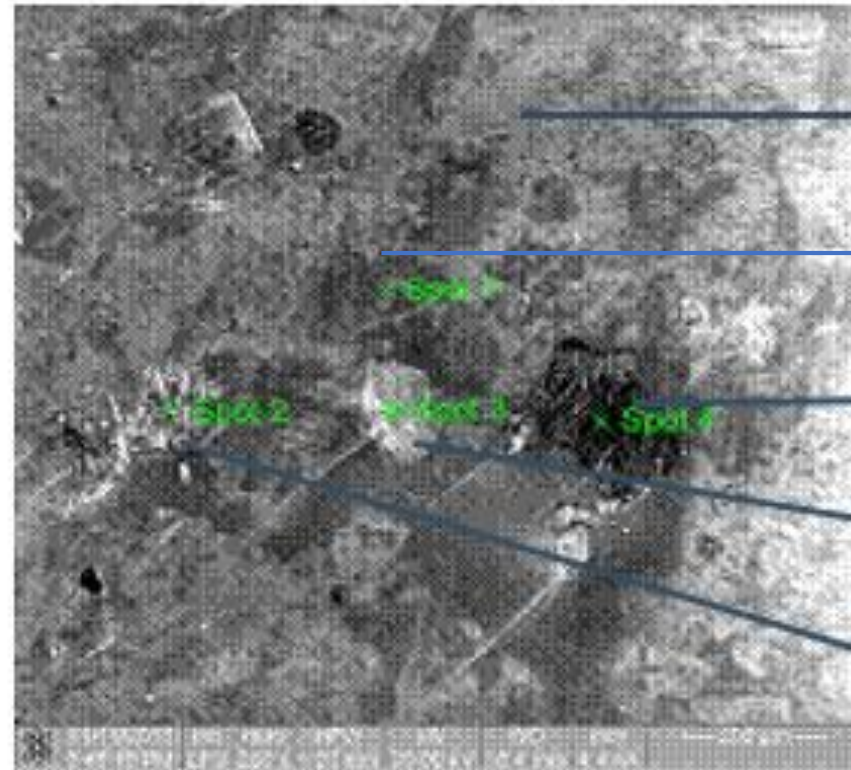
43.3 Wt% SiO_2 :	6.3 Wt% B_2O_3 :
18.8 Wt% Al_2O_3 :	2.5 Wt% CaO :
7.5 Wt% BaO :	3.4 Wt% Na_2O :
4.8 Wt% Nd_2O_3 :	4.0 Wt% CeO_2

Basic chemicals to form

5.0 Wt% Perovskite

5.0 Wt% Hollandite

5.0 Wt% Zirconolite



Chemicals representing:

Glass matrix + Nd??

Perovskite +Ce + Nd

Hollandite +Ce

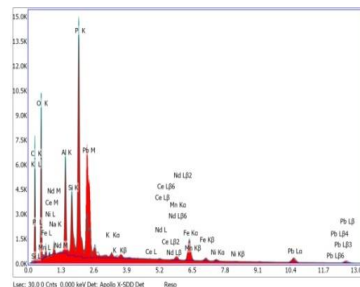
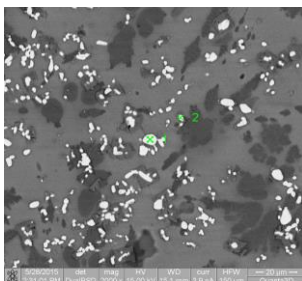
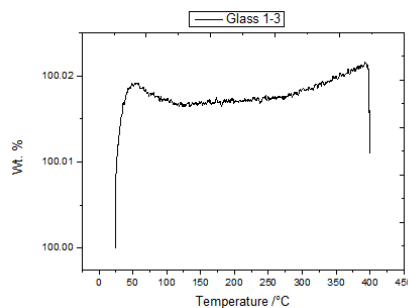
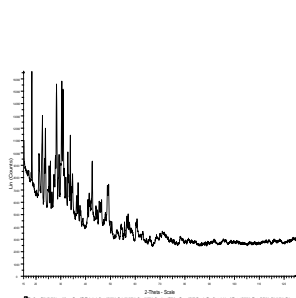
Zirconolite +Ce

Perovskite +Ce + Nd

SEM image of GCM with surrogates:250 x Mag

6. Composite glasses for U and FP

Element/ Nuclide	Surrogate compound	Mass of surrogate (g)
7% Fe	Fe ₂ O ₃	0.100
1.5% Cr	Cr ₂ O ₃	0.022
1% Al	Al(OH) ₃	0.029
U(VI)	Na ₂ U ₂ O ₇	0.736
U(IV)	UO ₂	0.111
⁶⁰ Co	C ₆₀ Q	6.30x10 ⁻⁸
⁹⁵ Nb	Nb ₂ O ₅	1.26x10 ⁻⁹
⁹⁵ Zr	ZrO ₂	2.45x10 ⁻³
¹⁰⁶ Ru	RuO ₂	9.14x10 ⁻⁴
¹²⁵ Sb	Sb ₂ O ₃	3.41x10 ⁻⁶
¹³⁷ Cs	CsOH.H ₂ O	1.03x10 ⁻⁵
¹⁴⁴ Ce/ ²³⁸ Pu	CeO ₂	1.59x10 ⁻³
¹⁴⁴ Pr	Pr ₆ O ₁₁	6.15x10 ⁻⁴
¹⁵⁴ Eu/ ¹⁵⁵ Eu	Eu ₂ O ₃	2.13x10 ⁻⁵
⁹⁰ Sr	SrO	5.73x10 ⁻⁴
Se	SeO ₂	3.72x10 ⁻⁵
Y	Y ₂ O ₃	3.35x10 ⁻⁴
Mo	MoO ₃	6.34x10 ⁻⁴
Rh	Rh ₂ O ₃	2.45x10 ⁻⁴
Pd	PdO	1.24x10 ⁻⁴
Ag	Ag ₂ O	2.38x10 ⁻⁶
Cd	CdO	5.32x10 ⁻⁶
Sn	SnO ₂	1.34x10 ⁻⁵
Te	TeO ₂	1.81x10 ⁻⁴
La	La ₂ O ₃	6.42x10 ⁻⁴
Nd	Nd ₂ O ₃	2.13x10 ⁻³
Sm	Sm ₂ O ₃	3.72x10 ⁻⁴
Gd	Gd ₂ O ₃	4.99x10 ⁻⁶
Total surrogate mixture mass		1.010



Element	Upper limit on leach rate (g/m ² /d)
B	0.06
Na	0.13
Al	0.01
Si	0.03
Ca	0.01
Cr	0.00
Fe	0.00
Ni	0.00
Zn	0.00
Se	0.00
Sr	0.00
Zr	0.00
Ru	0.00
Ag	0.00
Sb	0.08
Cs	0.03
Ba	0.00
Ce	0.00
Nd	0.00
U	0.00

Properties	WS3-GC-02	WS3-GC-03	WS3-GC-04	WS3-GC-05
Processing Conditions	Melt	Melt	Melt	Melt
Process Conditions	1. 1300 °C, 2. 600 °C, 3. 900 °C In air	1. 1300 °C, 2. 600 °C, 3. 900 °C In air	1. 1300 °C, 2. 600 °C, 3. 900 °C In air	1. 1300 °C, 2. 600 °C, 3. 900 °C In air
Target Phases	Borosilicate glass with 30% Al and 5% perovskite 5% zirconolite	Borosilicate glass with 30% Al and 5% perovskite 5% zirconolite	Brorsilicate glass with 30% Al and 5% perovskite 5% zirconolite	Phospate glass with 30% Al and 5% perovskite 5% zirconolite
Waste Form Properties				
Waste Form Density (g/mL)	2.42 g/cm ³	2.17 g/cm ³	2.01 g/cm ³	2.01 g/cm ³
Waste Loading (wt.% as oxide)	40	38	22	20
Compressive Strength	30MPa	32MPa	35MPa	29 Mpa
Thermal Stability	<0.1%	<0.1%	<0.1%	<1%
Chemical Durability	PASS	PASS	PASS	PASS
PCT-B test at 90°C for 7 days	PASS	PASS	PASS	PASS

Added Basic chemicals to form
5.0 Wt% Perovskite
5.0 Wt% Hollandite
5.0 Wt% Zirconolite

7. Summary

Vitrification of Uranium waste

1. Alkaline reprocessing technology possible for **Al-U Type fuels**
2. Selective removal of enriched U possible (with traces of other activities)
3. High waste loadings of Uranium into glass possible (24% waste - 20 wt. % U previous literature 1.2 %)
4. **Glass matrix with 25 wt.% loading pass all WAC requirements for disposal (Compression, stability, leaching)**
5. High waste loadings of Uranium into glass-ceramics (composite glasses) possible (24% waste - 12 wt. % U - no available literature values)
6. **Glass-ceramic matrix with 12 wt.% loading pass all WAC requirements for disposal (Compression, stability, leaching)**

8. References

1. L. Stassen Necsa report AC-UMo0101-REP-10001
2. L. Stassen Necsa report NWR-UMo01-REP-14026
3. Necsa report RC-PVLG-0027 (2007).