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Progress report on the ISTC project #3813: Phase relation in corium systems (PRECOS)

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20th CEG-SAM meeting
Moscow, Russia
October 11, 2011

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PRECOS project general information

Project duration	**36 months + 6 additional months**
Financial party	**Europe**
Funding	**995,610 USD**
Project status	**In Progress, Prolonged without additional funding**

Project objectives

Experimental determination of:

- liquidus – solidus temperatures
- coordinates of reference points (eutectics, etc.)
- solubility limits of solid solutions
- compositions of liquids coexisting in the miscibility gap

PRECOS test matrix

Task	Composition	Atm	Experimental data	Priority level	Number of tests scheduled /carried out
1	U-Zr-Fe-O	Argon	Selected points (liquidus, solidus, tie-lines in the miscibility gap)	1	6/8[1]
2	ZrO_2 - FeO_y	Air and p_{O2} control	liquidus, solidus, solubility limits	2	3/3[2]
	UO_2 - SiO_2	Neutral	liquidus, solidus, solubility limits, eutectic point	1	7/(5[3]+40[4])
	CaO - UO_2			1	7/7[3]
3	UO_2 – FeO – SiO_2		liquidus, solidus, solubility limits, tie-lines in the miscibility gap, ternary eutectic point	1	10/(4[3]+17[4])
	UO_2 – FeO – CaO		liquidus, solidus, solubility limits, ternary eutectic point	1	10/(4[3]+2[4])
	ZrO_2 - FeO - SiO_2		ternary eutectic point	2	2/0
	ZrO_2 - FeO - CaO		ternary eutectic point	2	2/0
4	Multicomponent prototypic corium	Argon or Air	System (atmosphere) proposed by: - French partners (1 system) - German partners (1 system) - Russian partners (1 system)	2	3/1

Notes: 1-LPH (Zr-O), 2- HTM, 3- VPA IMCC, 4- VPA in Galakhov microfurnace

- According to the 3rd Project meeting decisions these systems will be excluded from the test matrix for a more detailed study of systems having higher priority

Scope of work in quarters #12-13

System	Test	Objectives	Status
UO_2-FeO-SiO_2	PRS17,19 GPRS66-72,79-81	T_{liq}, T_{sol}, solubility limits, tie-lines in the miscibility gap, ternary eutectic composition and temperature	Tests done Post test analysis in progress
UO_2-FeO-CaO	PRS18,20 GPRS75	T_{liq}, T_{sol}, solubility limits, ternary eutectic composition and temperature	
Multicomponent prototypic corium (French system)	PRS21	T_{liq}, T_{sol}	

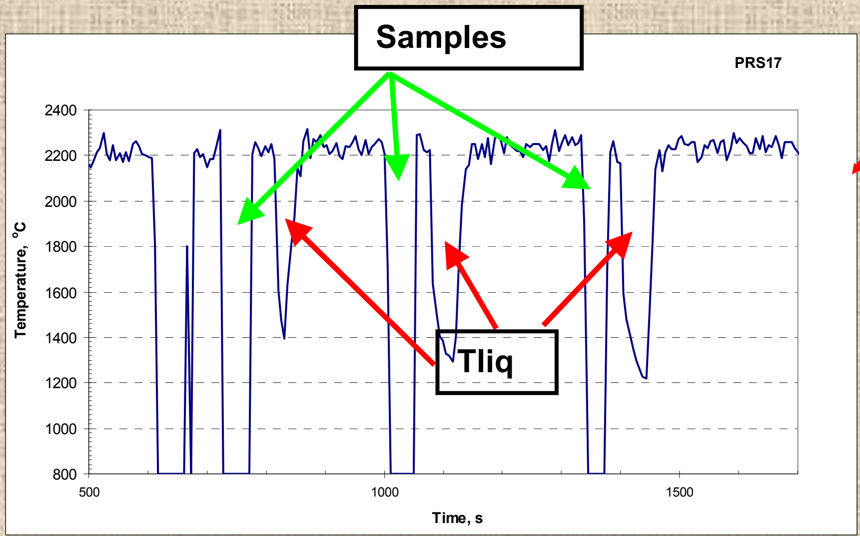
UO_2–FeO–SiO_2 system: PRS17 test results

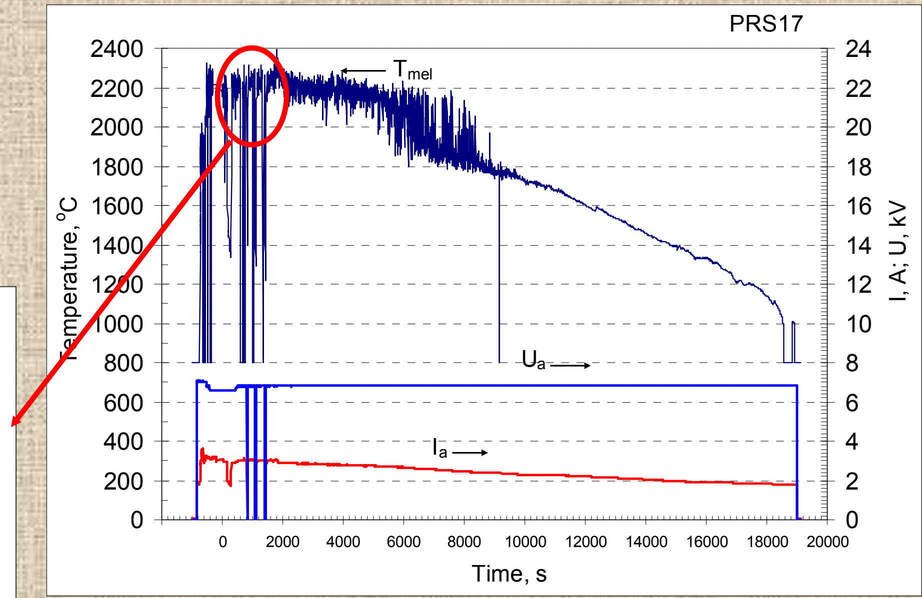
➢Experimental objectives

- T_{liq} determination by VPA IMCC
- Determination of the ternary eutectic composition

➢Charge composition

- Mol.% 10 UO_2 + 55 FeO +35 SiO_2



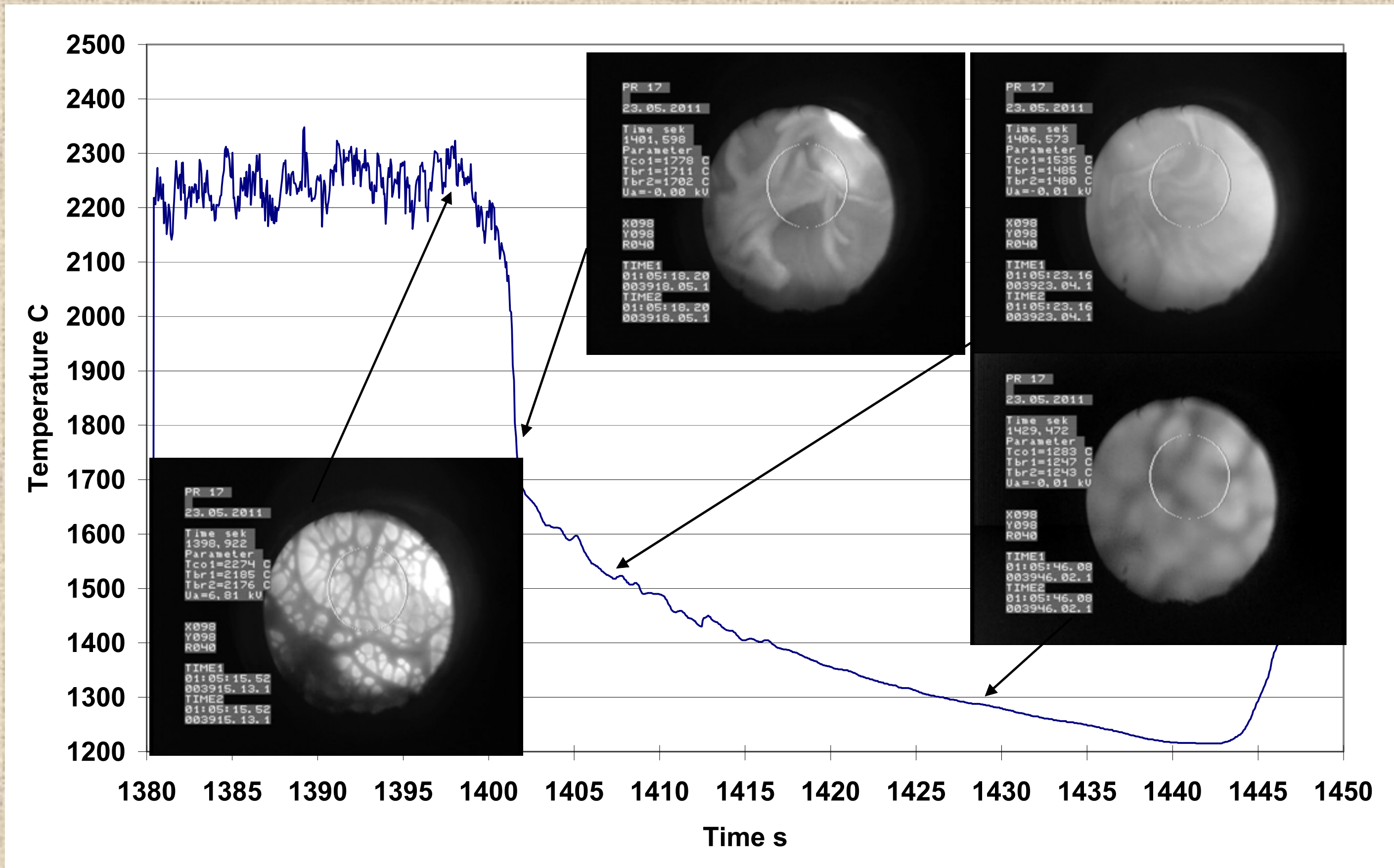


From 1894 s, the pool was pulled out from inductor at 9 mm/h for 4.8 hours. This has ensured close to equilibrium crystallization and the eutectic liquid displacement into the ingot upper part

✓T_{liq} was measured 3 times by VPA IMCC and accompanied by melt sampling

UO_2–FeO–SiO_2 system: PRS17 test results (2)

➢**VPA IMCC: Example of thermogram 3 showing melt surface**



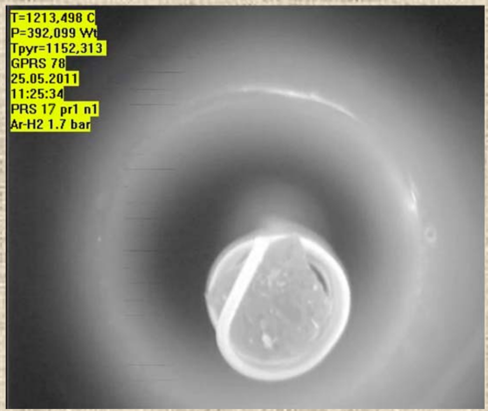
✓ **First solid phase was not detected at the melt surface. Consequently T_{liq} was not determined**

✓**Melt samples were used for VPA GM**

UO_2 - FeO - SiO_2 system: PRS17 test results (3)

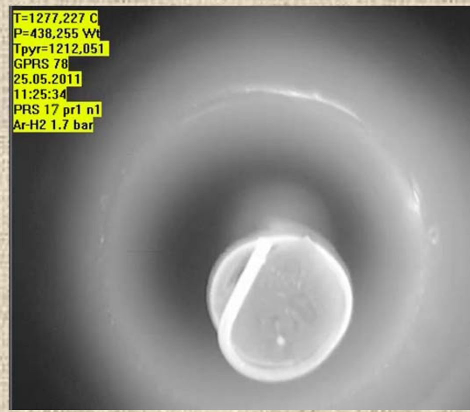
➢T_{sol} and T_{lig} determination in the Galakhov microfurnace

Sample #1

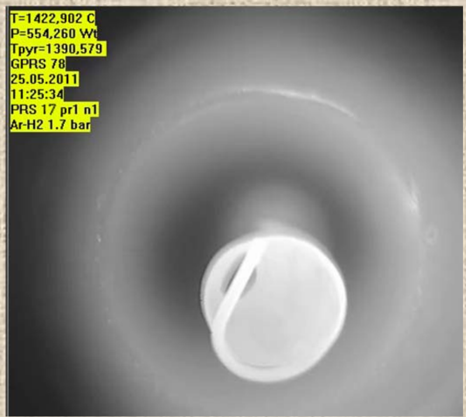


1152°C

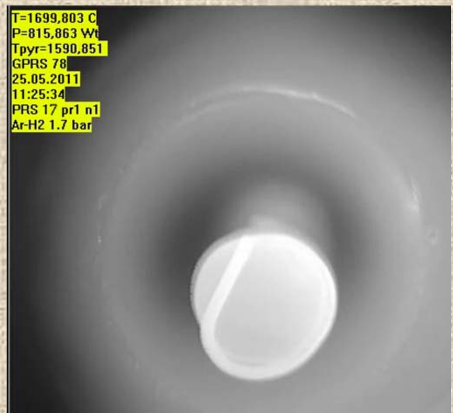
1155°C (deformation start)



1212°C



1390°C



1590°C (complete spreading)

- ✓Visual polythermal analysis in the Galakhov microfurnace T_{sol}= 1155°C; T_{liq}=1590°C
- ✓Close temperatures for other samples

UO_2–FeO-SiO_2 system: PRS17 test results (4)

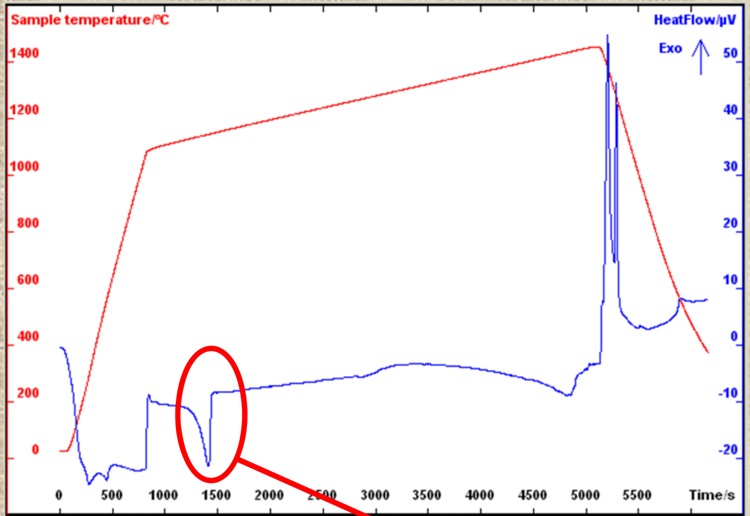
Liquidus temperatures and compositions of melt samples measured by ChA

Sample	Composition, mass/mol.%			T_{liq}/T_{sol}, ˚C
	UO_2	FeO	SiO_2	
1	27.36 8.58	47.23 55.64	25.41 35.79	1590±30/1155±15
2	26.56 8.27	48.27 56.50	25.17 35.23	
3	26.29 8.17	48.39 56.49	25.31 35.34	
Charge composition	28.8 10.0	47.8 55.0	25.4 35.0	

- ✓ T_{liq} was measured 3 times by VPA IMCC but video record of measurements was not interpreted
- ✓ T_{liq} and T_{sol} were measured by VPA in the Galakhov microfurnace. Similar temperatures were registered in all samples

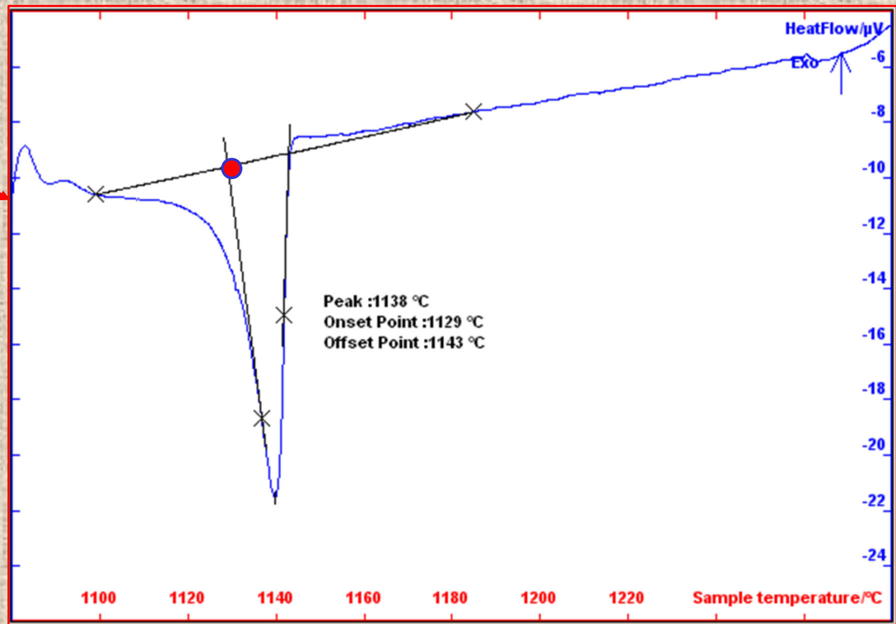
UO_2–FeO-SiO_2 system: PRS17 test results (5)

➢**DTA** **Sample cut out from the eutectic core of the ingot**



Experimental conditions:

argon atmosphere;
sample mass ≈95 mg;
thermocouple of S type (Pt/Pt - 10%Rh);
sample heating rate: 5 °/min in the range between 1100 - 1400 °C,
crucible material – Pt



T_{eut}=1129 °C

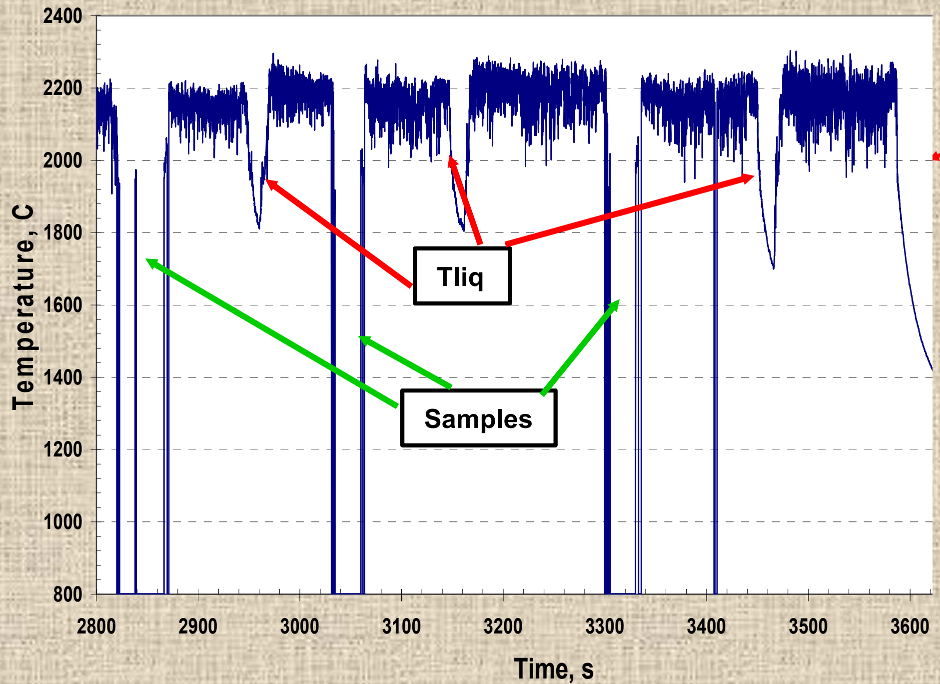
UO_2 – FeO – SiO_2 system: PRS 19 test results (6)

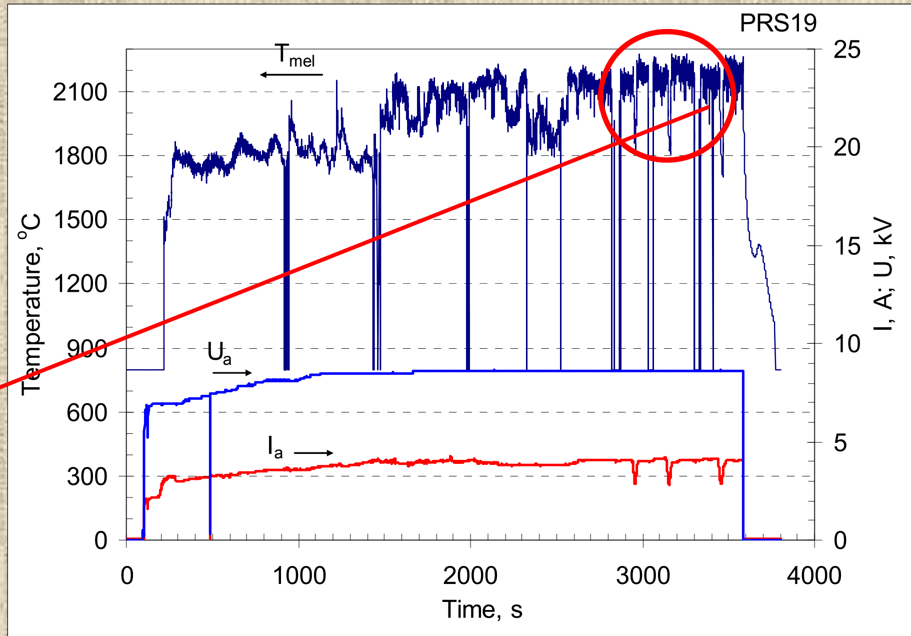
➢Experimental objectives

✓ T_{liq} determination by VPA IMCC

➢Charge composition

mol.% 50 UO_2 + 33.2 FeO +16.8 SiO_2

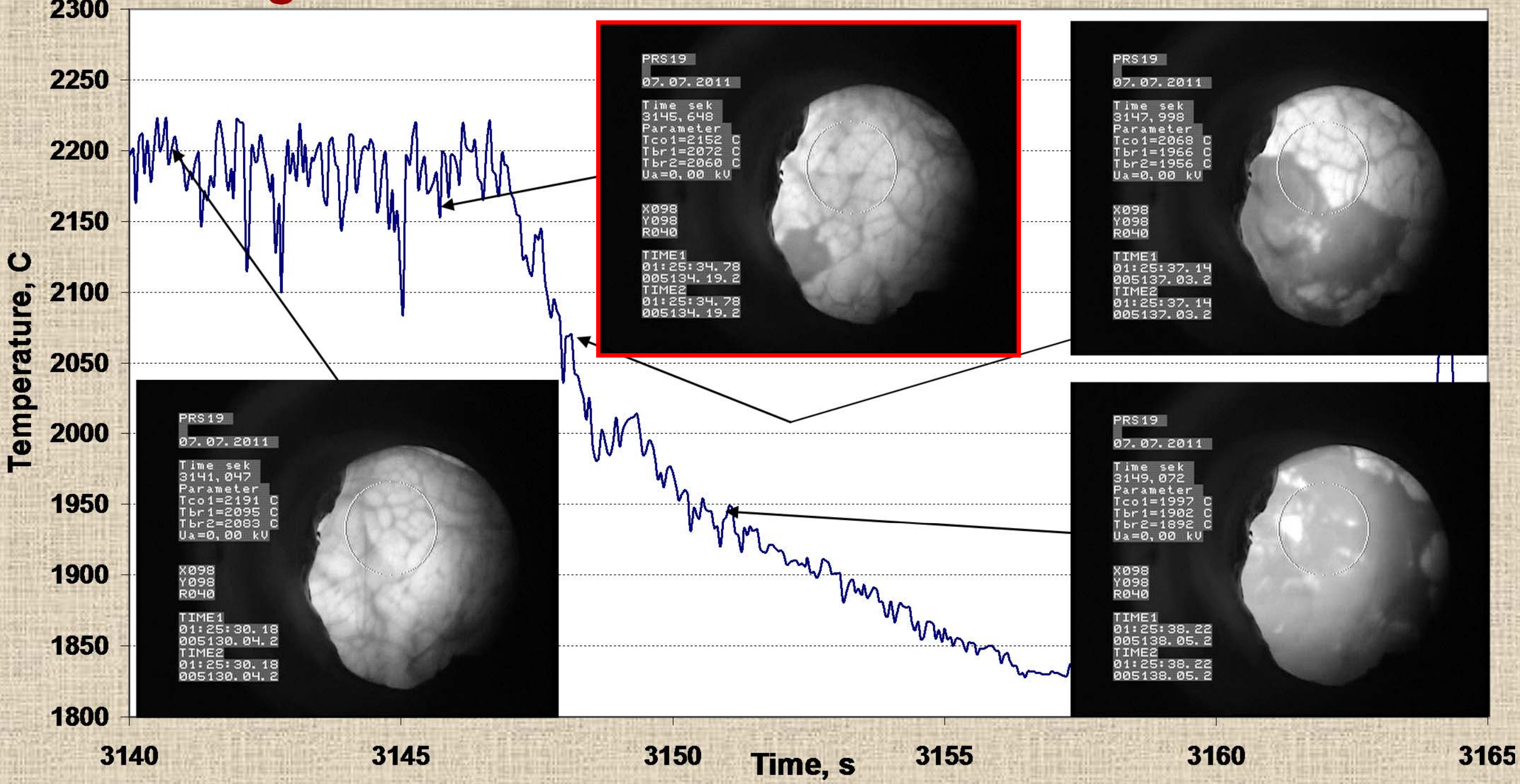




✓T_{liq} was measure 3 times by VPA IMCC with melt sampling

UO_2–FeO–SiO_2 system: PRS 19 test results (7)

- VPA IMCC: Example of thermogram 2 from the test showing melt surface

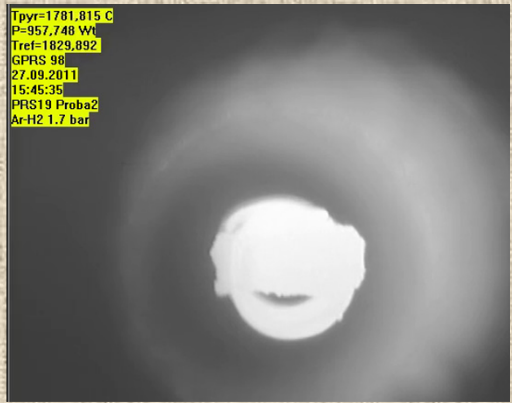


✓ Results of T_{liq} measurements: 2180, 2150, 2160°C

UO_2 – FeO – SiO_2 system: PRS19 test results (8)

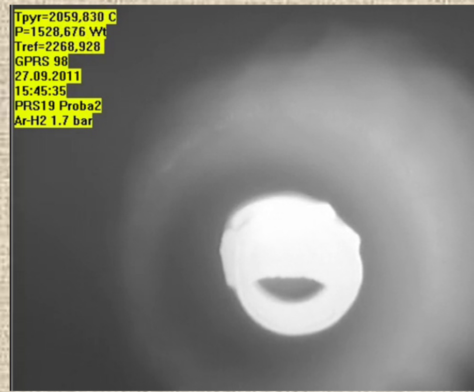
➢T_{liq} and T_{sol} determination in the Galakhov microfurnace

Sample # 2

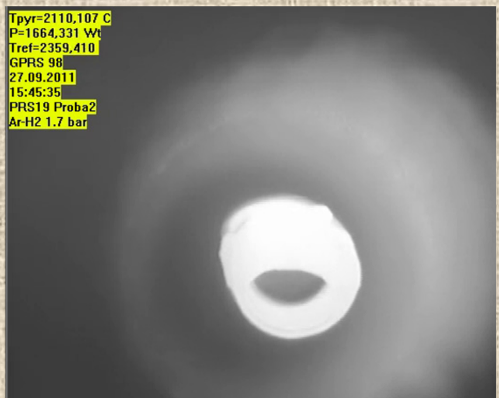


1781°C

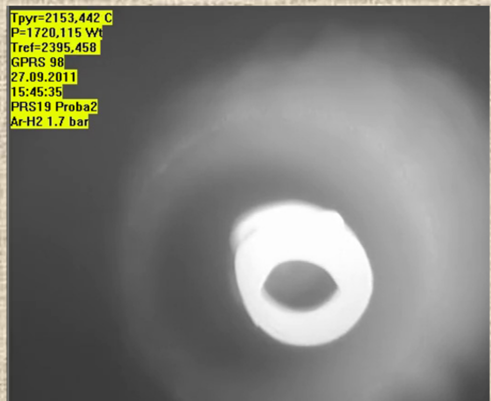
1870°C (deformation start)



2059°C



2110°C



2153°C (complete spreading)

- ✓Visual polythermal analysis in the Galakhov microfurnace T_{sol}= 1870°C; T_{liq}=2150°C
- ✓T_{liq}, determined by the VPA IMCC (2150±30°C) coincides with T_{liq} measured in the Galakhov microfurnace

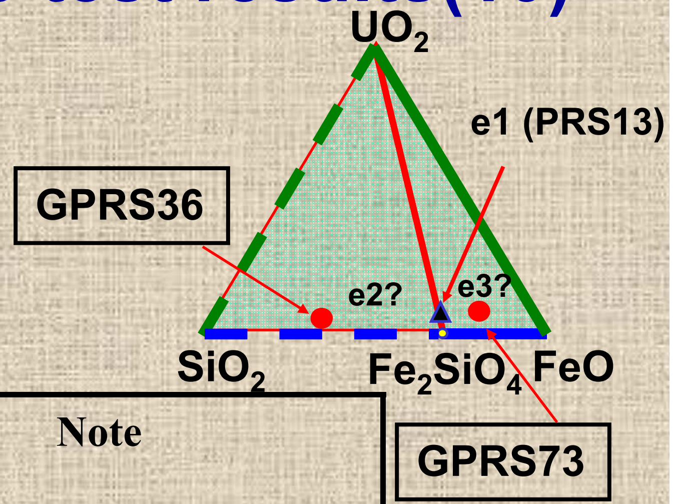
UO_2 – FeO – SiO_2 system: PRS 19 test results (9)

Liquidus temperatures and compositions of melt samples measured by ChA

Sample	Composition, mass/mol.%			T_{liq}, °C
	UO_2	FeO	SiO_2	
1	70.44 37.54	21.30 42.66	8.27 19.80	2180±35
2	70.32 37.53	22.24 44.61	7.45 17.86	2150±35
3	72.69 40.24	20.13 41.90	7.18 17.86	2160±35
Charge composition	*79.91* *50.0*	*14.12* *33.2*	*5.97* *16.8*	-

- ✓ **Change in sample composition versus the charge is explained by the crystallization of refractory phase on the cooled crucible surfaces**
- ✓ **T_{liq} for the second sample was measured by the VPA in the Galakhov microfurnace, and it coincided with the VPA IMCC data**

UO_2–FeO–SiO_2 system:GPRS36,73 test results(10)



➢Experimental objectives

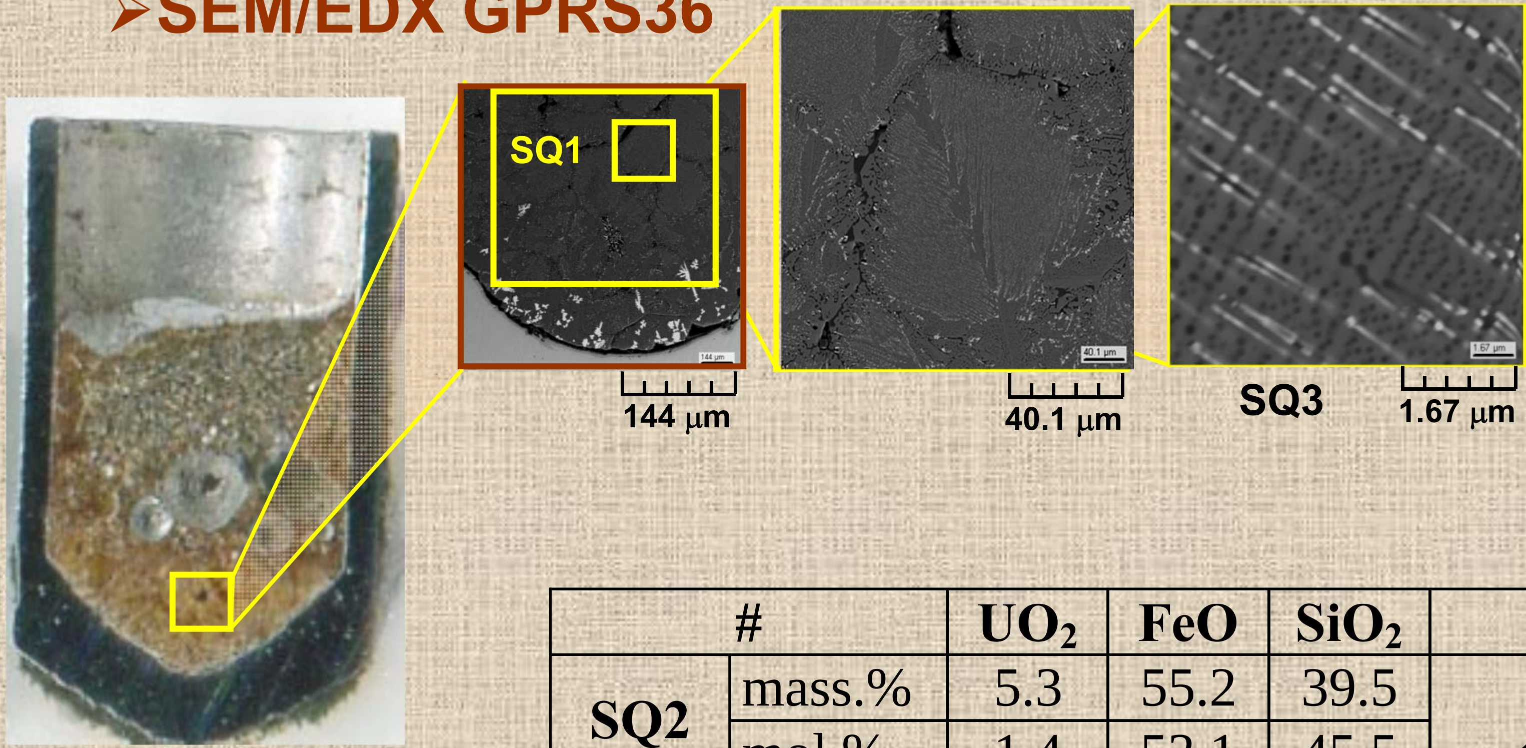
Determination of the e2 and e3 ternary eutectic composition

➢Charge composition

Test	Content, mol.%			Tempera-ture, °C	Exposure time, min	Note
	UO_2	SiO_2	FeO			
GPRS36	1.7	65.5	32.8	1100	60	Annealing
				1300	20	Melting
				1300-900	240	Cooling at 100°C/h
				900-		Cooling together with the furnace with heating switched off
GPRS73	2.5	20.0	77.5	1100	60	Annealing
				1300	5	Melting
				1300-900	240	Cooling at 100°C/h
				900-		Cooling together with the furnace with heating switched off

UO_2-FeO-SiO_2 system:GPRS36 test results(11)

➢SEM/EDX GPRS36

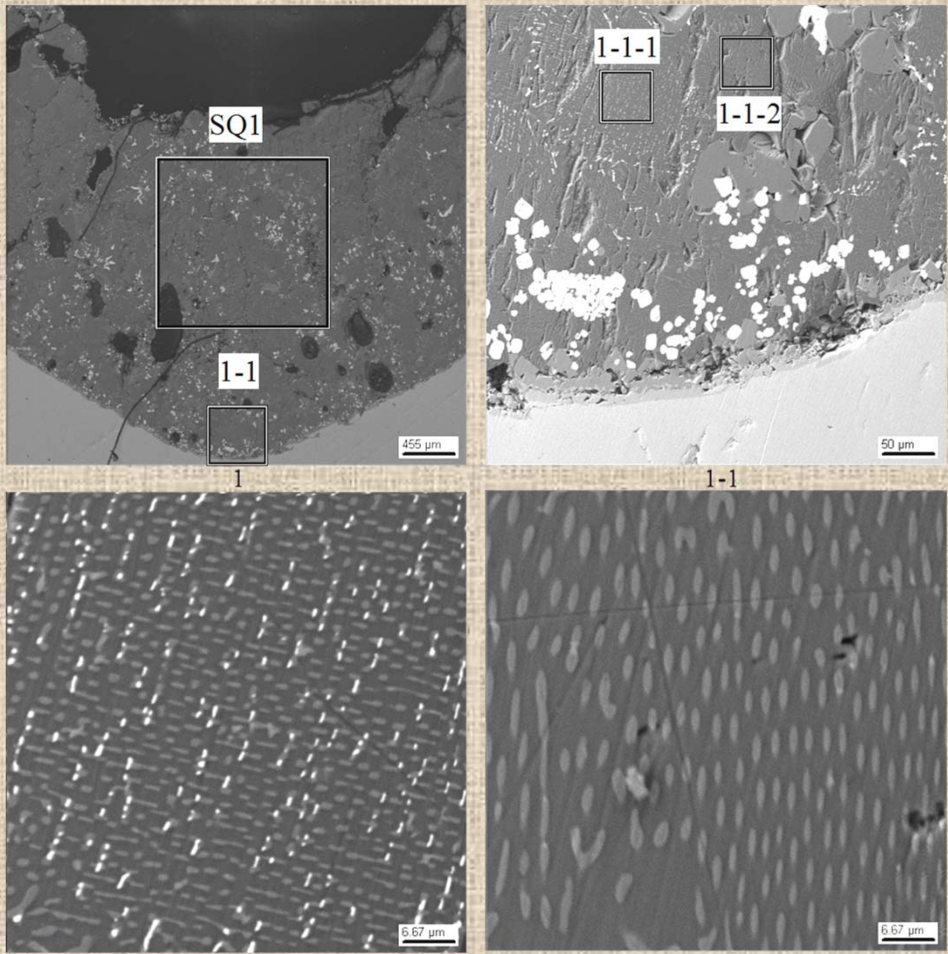


#		UO_2	FeO	SiO_2	
SQ2	mass.%	5.3	55.2	39.5	triple eutectics
	mol.%	1.4	53.1	45.5	
SQ3	mass.%	5.9	53.6	40.5	
	mol.%	1.5	51.8	46.7	

✓Microstructure in the lower part of the crucible shows the eutectic crystallization. In terms of composition, this eutectics lies within a specific triangle UO_2-Fe_2SiO_4-SiO_2

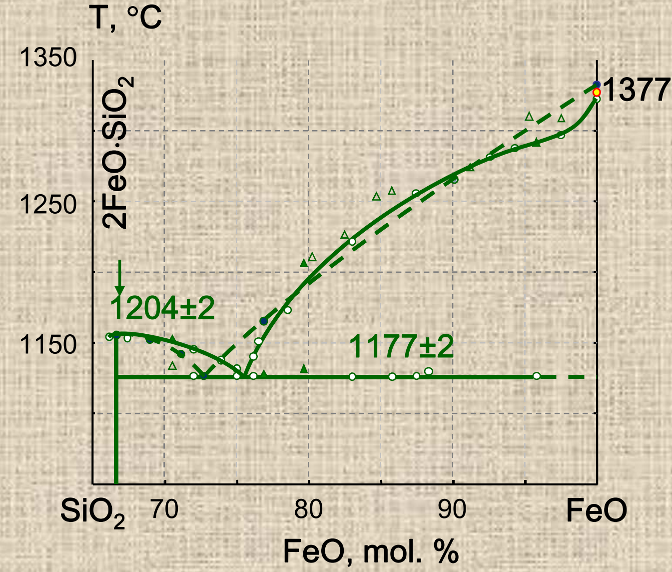
UO_2-FeO-SiO_2 system:GPRS73 test results(12)

➢SEM/EDX GPRS73



1-1-1 (SQ2) 1-1-2 (SQ3)

#		UO_2	FeO	SiO_2	Phase
SQ1	mass%	6.0	77.7	16.3	average
	mol.%	1.6	78.7	19.7	
SQ2	mass %	4.9	76.0	19.1	ternary eutectics
	mol.%	1.5	75.7	22.8	
SQ3	mass.%	-	78.4	21.6	binary eutectics
	mol %	-	75.2	24.8	



Allen W. C, R. B. Snow, Journ. Amer. Ceram. Soc, 38, № 8, 264,1955.

✓**The binary eutectics corresponds to the results obtained in previous investigations**

UO_2-FeO-SiO_2 system: GPRS #59-64 test results(13)

- **Experimental objectives**

  Study of tie lines and miscibility gap boundaries

- **Annealing, melting and quenching in the Galakhov microfurnace**

Test	Content, mol.%			Tempera-ture, °C	Exposure time, min	Note
	UO_2	SiO_2	FeO			
GPRS59	5.0	77.0	18.0	1100	60	Annealing
				1750	10	Melting and quenching
GPRS60	5.0	77.0	18.0	1100	60	Annealing
				1850	10	Melting and quenching
GPRS61	10.0	78.0	12.0	1100	60	Annealing
				1750	10	Melting and quenching
GPRS62	10.0	78.0	12.0	1100	60	Annealing
				1850	10	Melting and quenching
GPRS63	15.0	79.0	6.0	1100	60	Annealing
				1750	10	Melting and quenching
GPRS64	15.0	79.0	6.0	1100	60	Annealing
				1850	10	Melting and quenching

✓ UO_2 of >99.0 % purity, SiO_2 of 99.99% purity, FeO of >99.0 % purity, charge mass – 150 mg, molybdenum crucibles ∅ 6 mm

UO_2-FeO-SiO_2 system: GPRS #67-72 test results(14)

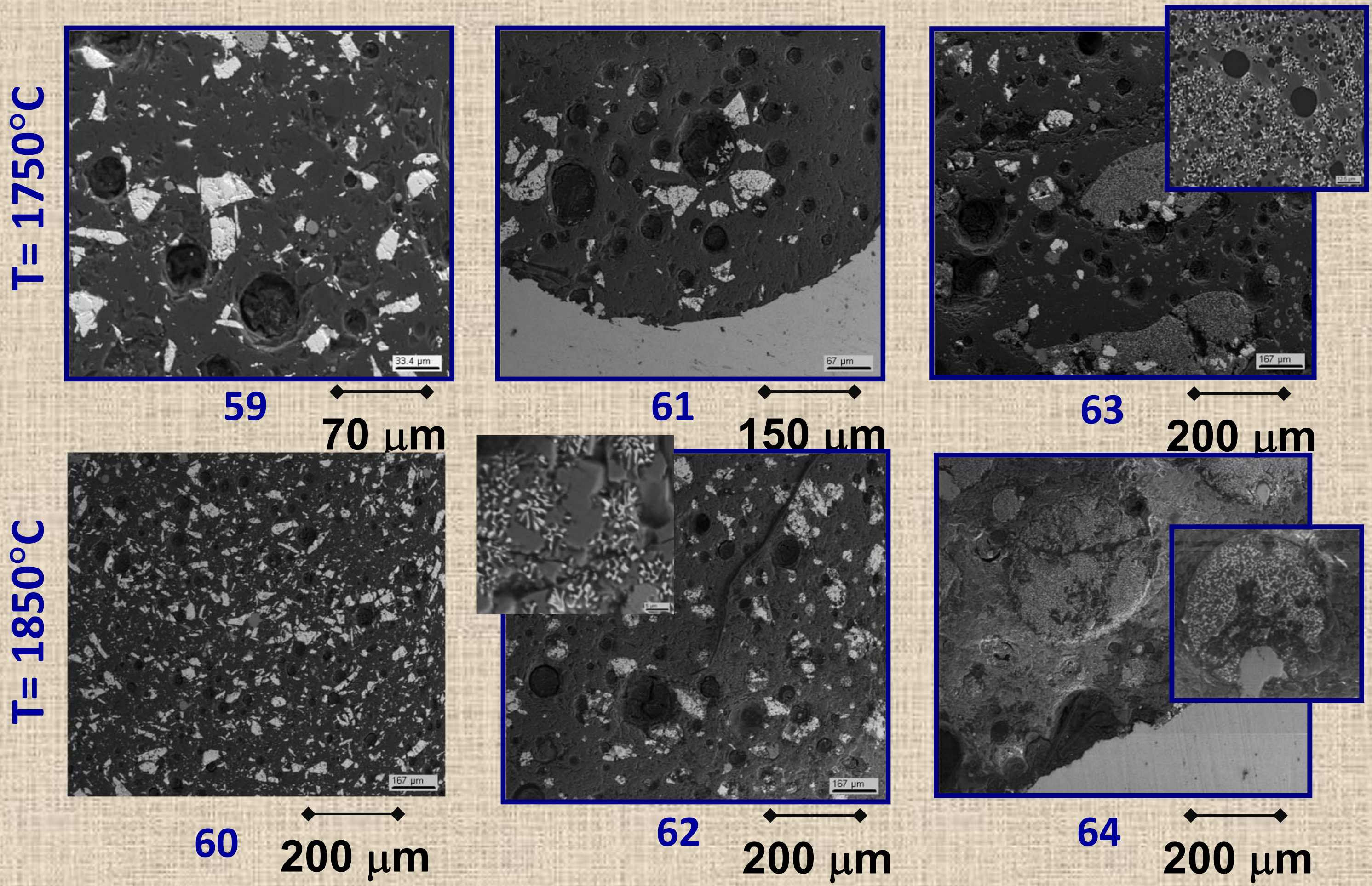
Test	Content, mol.%			Temperature, °C	Exposure time, min	Note
	UO_2	SiO_2	FeO			
GPRS67	18.0	77.0	5.0	1100	60	Annealing
				1950	10	Melting and quenching
GPRS68	18.0	77.0	5.0	1100	60	Annealing
				2050	10	Melting and quenching
GPRS69	12.0	78.0	10.0	1100	60	Annealing
				1900	10	Melting and quenching
GPRS70	12.0	78.0	10.0	1100	60	Annealing
				1950	10	Melting and quenching
GPRS71	6.0	79.0	15.0	1100	60	Annealing
				1700	10	Melting and quenching
GPRS72	6.0	79.0	15.0	1100	60	Annealing
				1950	10	Melting and quenching

UO_2-FeO-SiO_2 system: GPRS #79-81 test results(15)

Test	Content, mol.%			Temperature, °C	Exposure time, min	Note
	UO_2	SiO_2	FeO			
GPRS79	18.0	77.0	5.0	1100	60	Annealing
				2150	10	Melting and slow cooling down
				2000	10	Annealing and quenching
GPRS80	12.0	78.0	10.0	1100	60	Annealing
				2000	10	Melting and slow cooling down
				1800	10	Annealing and quenching
GPRS81	6.0	79.0	15.0	1100	60	Annealing
				2000	10	Melting and slow cooling down
				1725	10	Annealing and quenching

UO_2-FeO-SiO_2 system: GPRS #59-64 test results(16)

➢SEM

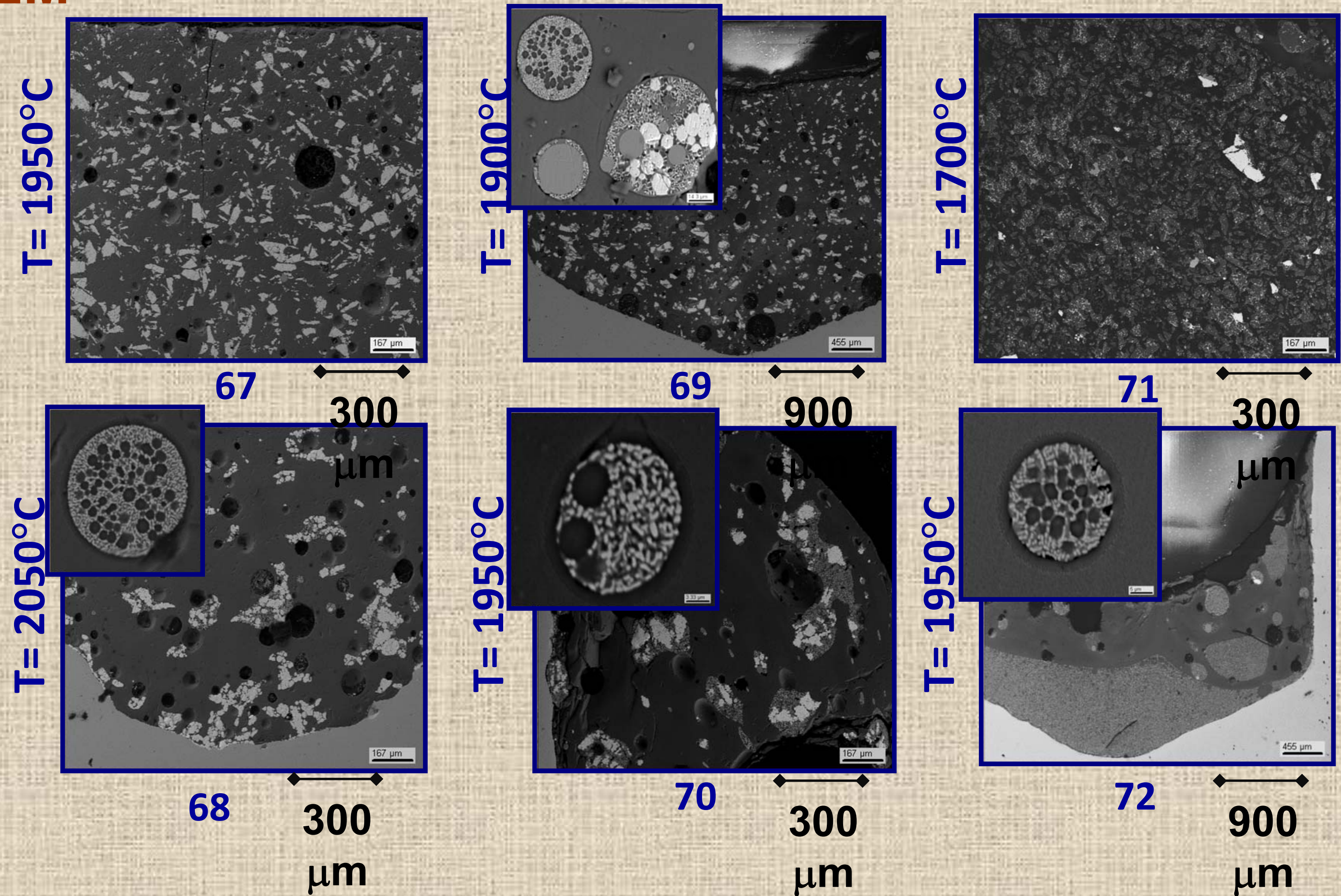


✓In GPRS 59,60,61 microstructures typical of MG are absent

✓In GPRS 62,63,64 micro segregation was found

UO_2-FeO-SiO_2 system: GPRS #67-72 test results(17)

➢SEM

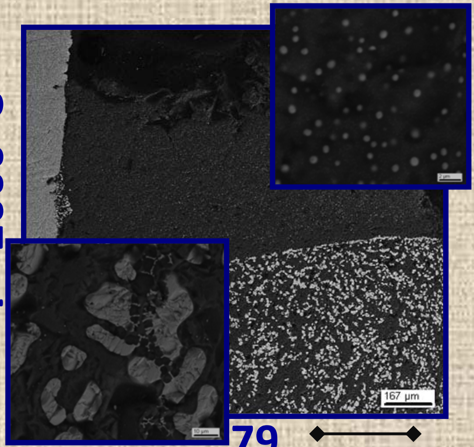


- ✓In GPRS 67,71 microstructures typical of MG are absent
- ✓In GPRS 68,69,70 micro segregation was found
- ✓In GPRS 72 macrostratification was found

UO_2-FeO-SiO_2 system: GPRS #79-81 test results(18)

➢ **SEM**

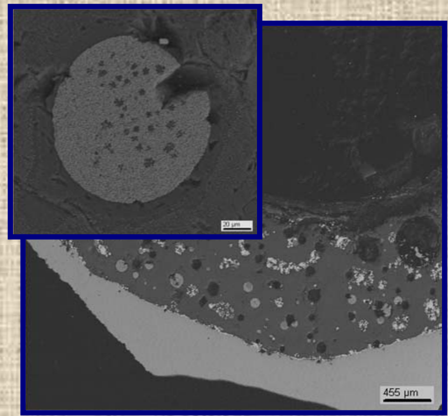
T= 2000°C



79

300 μm

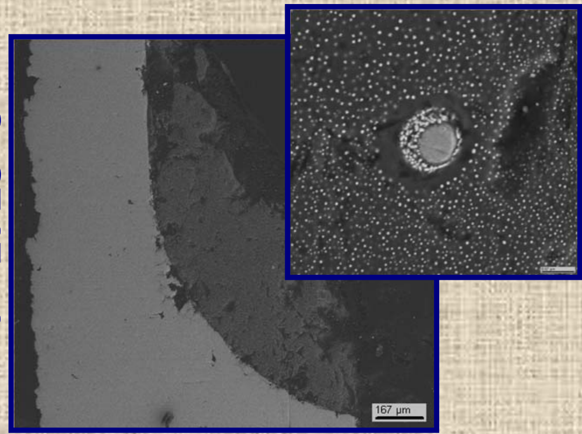
T= 1800°C



80

900 μm

T= 1725°C



81

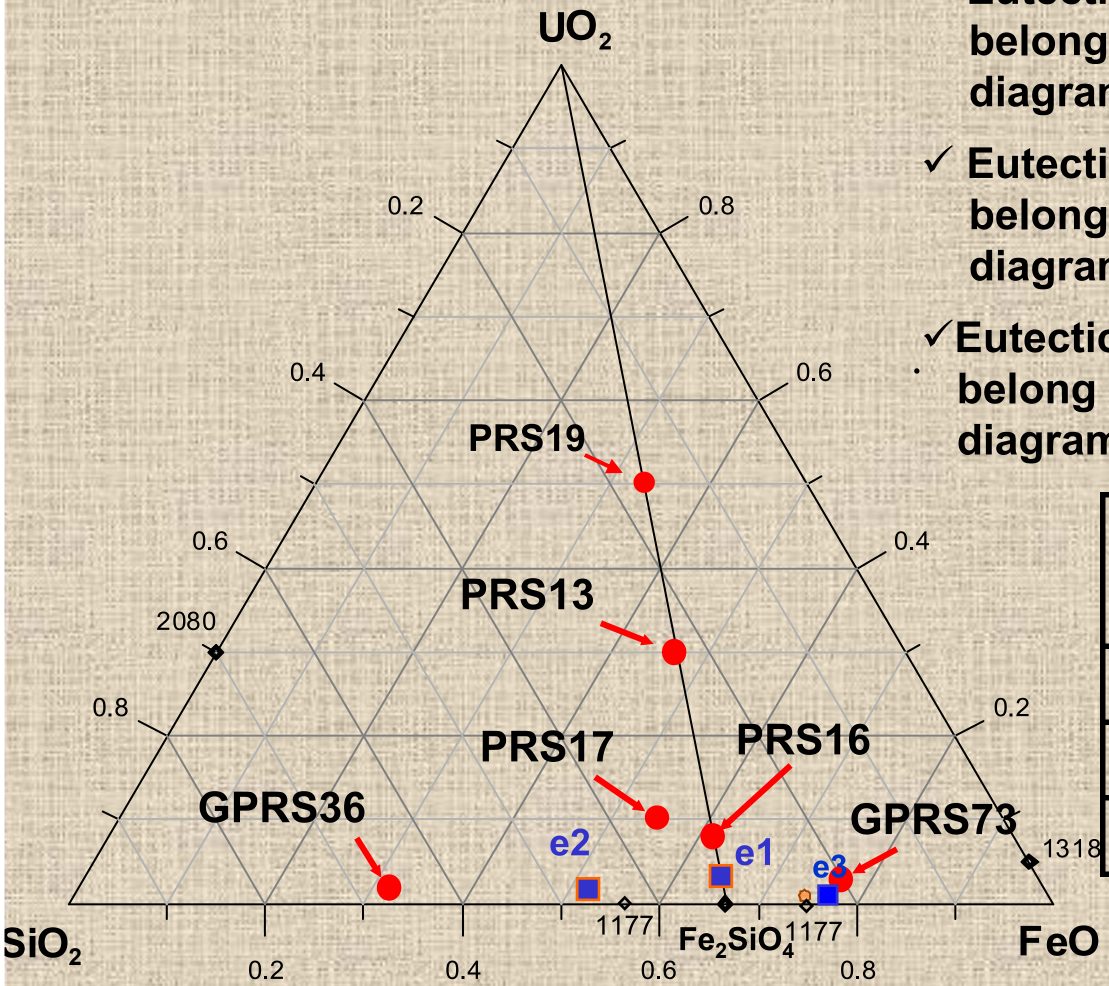
300 μm

✓**In GPRS 81 microstructures typical of MG are absent**

✓**In GPRS 80 micro segregation was found**

✓**In GPRS 79 macrostratification was found**

UO_2-FeO-SiO_2 system: test results (19)



- ✓ Eutectic point e1 has been determined to belong to UO_2-Fe_2SiO_4 section of the diagram
- ✓ Eutectic point e2 has been determined to belong to UO_2-Fe_2SiO_4-SiO_2 region of the diagram
- ✓ Eutectic point e3 has been determined to belong to UO_2-Fe_2SiO_4-SiO_2 region of the diagram

	UO_2	FeO	SiO_2
	mol.%		
e1	2.8 ± 0.3	64.9 ± 3.1	32.3 ± 3.0
e2	1.4 ±0.1	52.5 ±0.6	46.1 ±0.6
e3	1.5 ±0.2	75.2 ±0.7	22.8 ±0.8

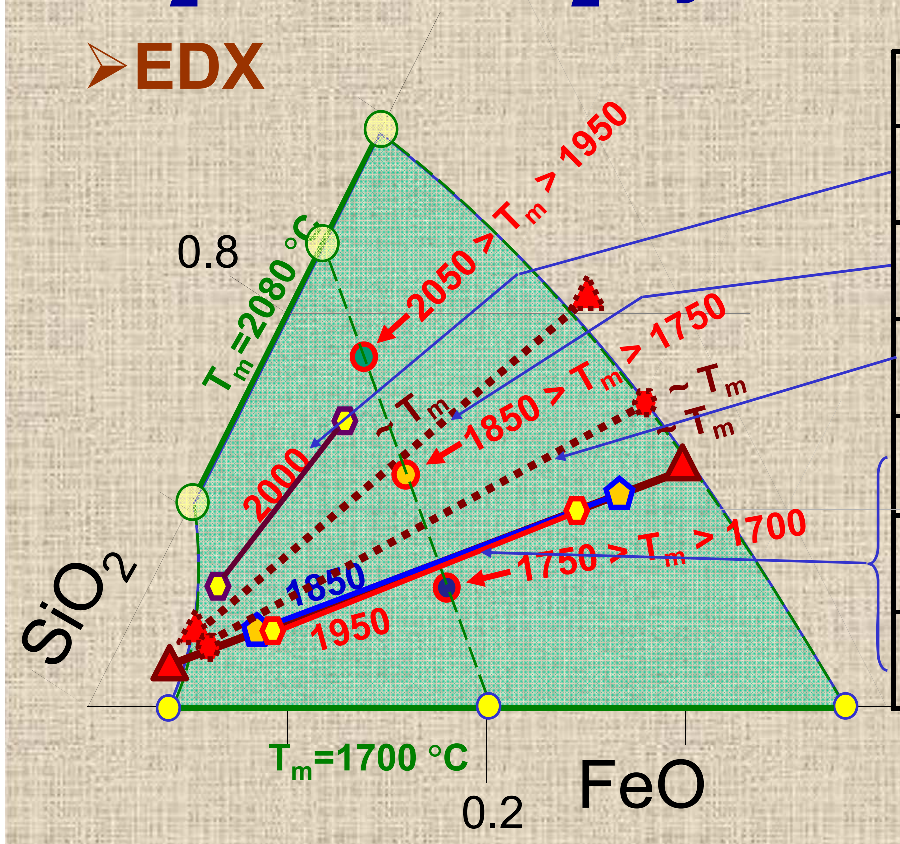
T_{e1}=1149 ± 15˚C (VPA GM)

T_{e2}=1138 ± 5˚C (DTA)

T_{e3}=?

UO_2-FeO-SiO_2 system: GPRS test results (20)

➢EDX



T, °C	Type	UO_2	FeO	SiO_2
2000	Heavy liquid	14.7±1.3	5.5±0.6	79.8±1.9
	Light liquid	6.6±0.7	2.4±0.6	91.1±1.0
~1800	Heavy liquid	20.1±8.6	15.0±1.8	64.9±6.8
	Light liquid	3.3±0.5	3.3±0.5	93.5±1.0
~1750	Heavy liquid	15.7±1.5	20.5±5.6	64.2±7.4
	Light liquid	2.6±0.6	4.5±0.9	92.8±1.2
1950	Heavy liquid	10.0±0.8	19.0±1.0	71.0±1.6
	Light liquid	3.5±0.1	7.6±1.0	88.9±1.1
1850	Heavy liquid	11.8±1.2	20.9±1.9	67.3±1.7
	Light liquid	4.0±0.3	6.6±1.3	89.4±1.6
~1725	Heavy liquid	12.2±1.5	23.4±3.6	64.4±4.4
	Light liquid	1.7±0.1	2.8±0.3	95.5±0.4

- ✓ Monotectic points of “heavy” and “light” liquids have been determined in the miscibility gap
- ✓ Three tie-lines have been determined in the miscibility gap

UO_2-FeO-SiO_2 system: test results (21)

- ✓T_{liq} and T_{sol} of PRS 17 composition has been measured by VPA in Galakhov microfurnace
- ✓T_{liq} of PRS 19 composition has been measured by VPA IMCC
- ✓T_{liq} and T_{sol} of PRS 19 composition has been measured by VPA in Galakhov microfurnace
- ✓15 GPRS experiments in the miscibility gap have been made. The SEM/EDX analysis of synthesized macro and micro structures has been completed

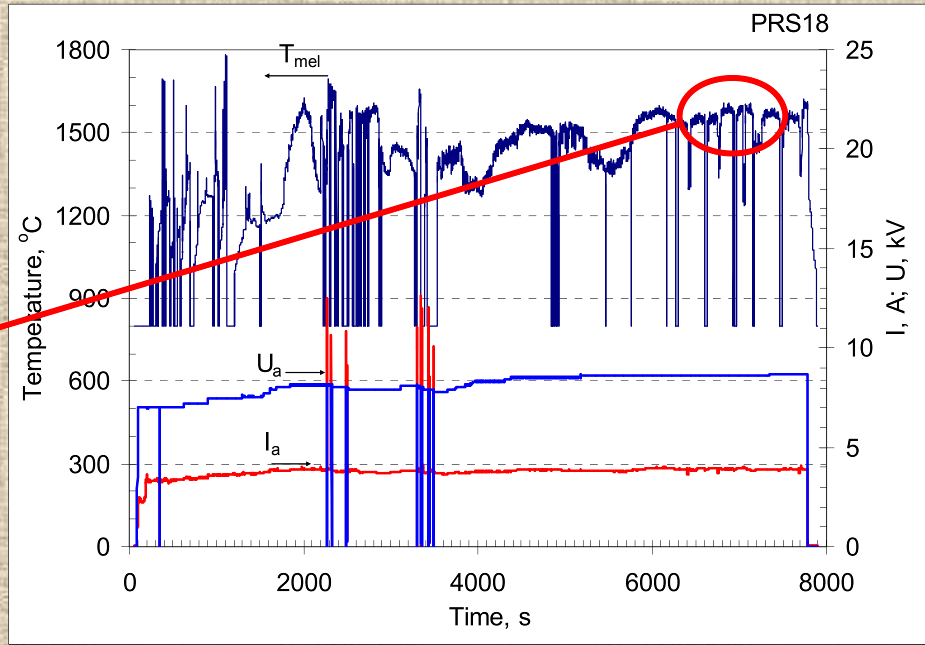
UO_2 – FeO – CaO system: PRS18 test results

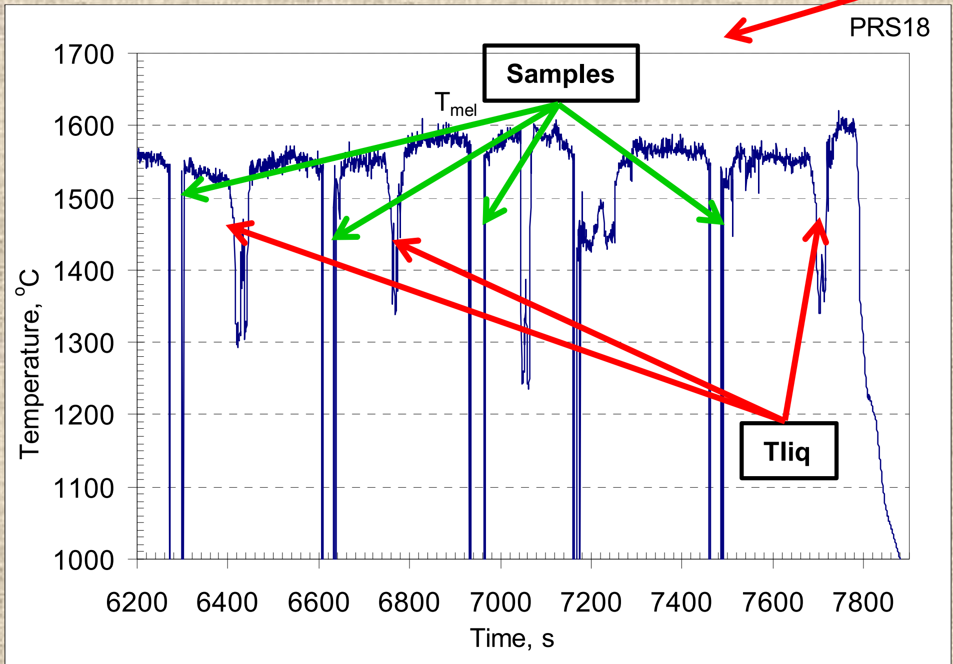
➢Experimental objectives

T_{liq} determination by VPA IMCC

➢Charge composition

mol.% 30 UO_2 + 40 FeO + 30 CaO

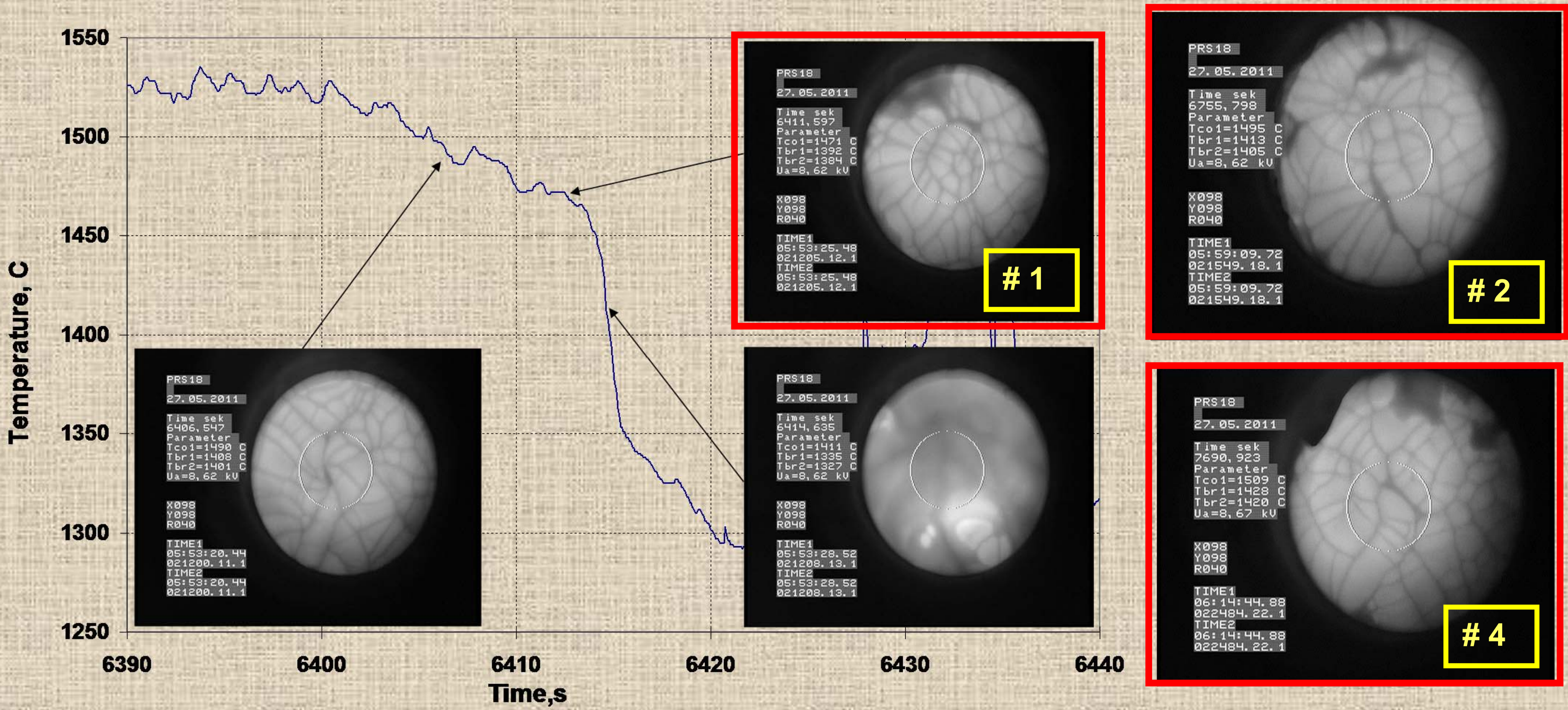




✓T_{liq} **was measured 3 times by VPA IMCC with melt sampling**

UO_2 – FeO – CaO system: PRS18 test results (2)

➢VPA IMCC: Example of thermogram 1 from the test showing melt surface



✓ Results of T_{liq} measurements: 1470, 1495, 1510°C

UO_2 – FeO – CaO system: PRS18 test results (3)

➢Liquidus temperatures and compositions of melt samples measured by XRF and ChA

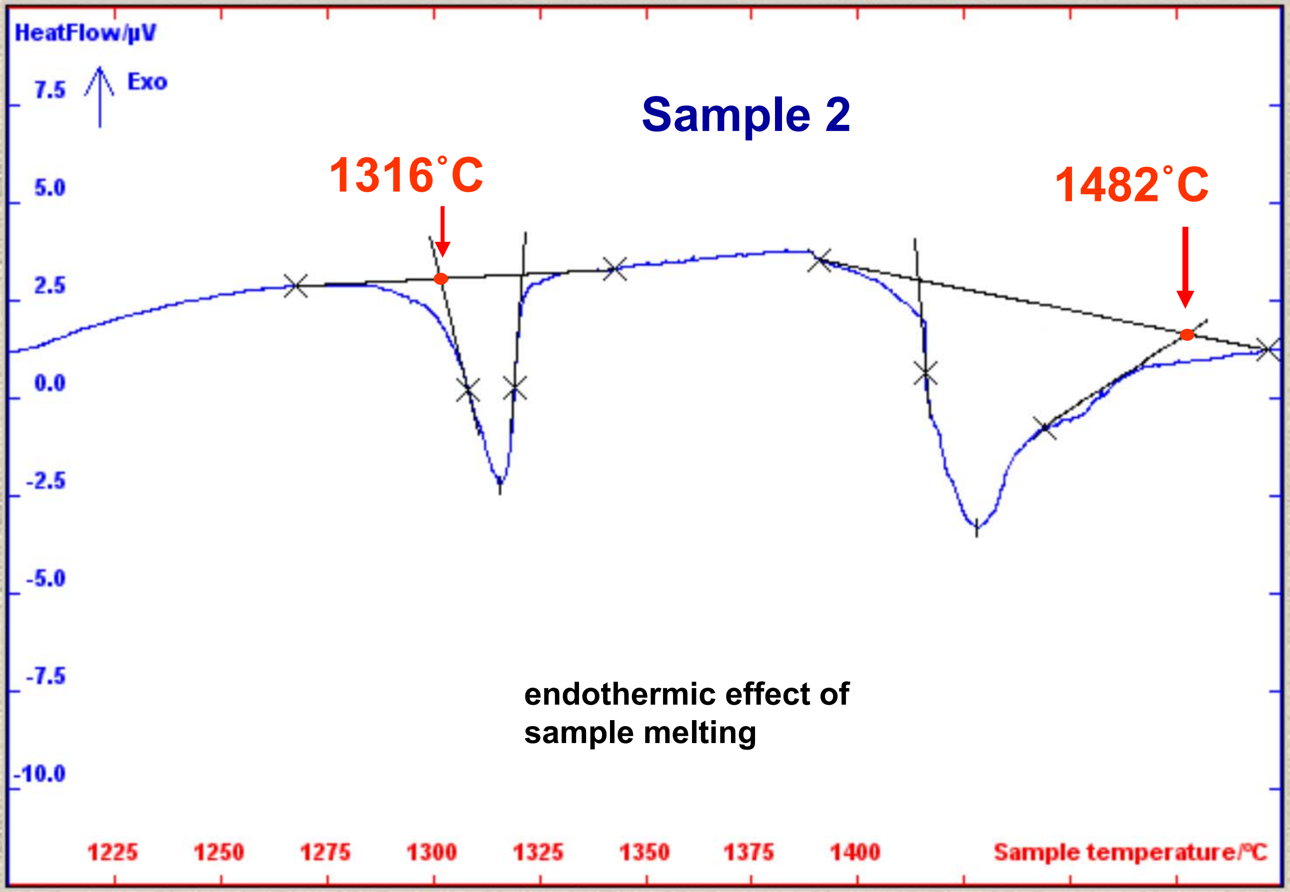
Sample	Composition, mass/mol. %						T_{liq} °C
	XRF			Cha			
	UO_2	FeO	CaO	UO_2	FeO	CaO)*	
1	30.76 10.20	59.28 73.89	9.96 15.91	30.96 10.29	59.18 73.93	9.86 15.78	1470±22
2	31.91 10.69	58.05 73.11	10.04 16.20	31.97 10.71	57.74 72.69	10.29 16.60	1495±23
4	30.80 10.20	58.75 73.13	10.45 16.67	30.75 10.17	58.55 72.79	10.70 17.04	1510±23

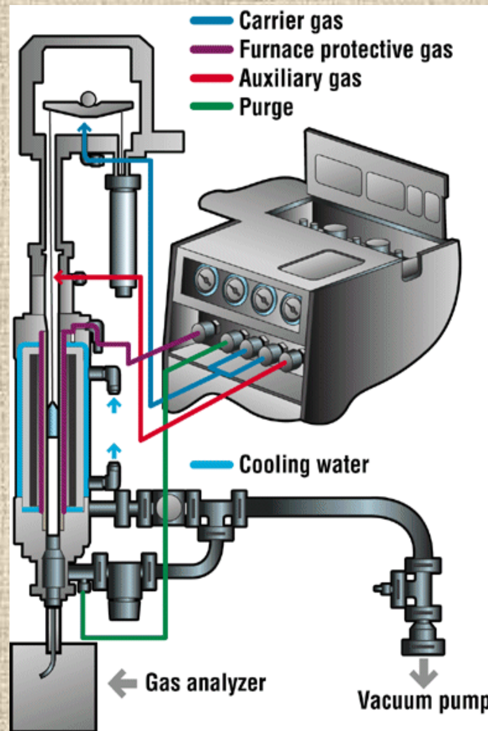
* - determined from residue

✓During the molten pool preparation, the initial crystallization phase is UO_2 that crystallizes on the pool bottom.

UO_2 – FeO – CaO system: PRS18 test results (4)

➢ **DTA measurements**





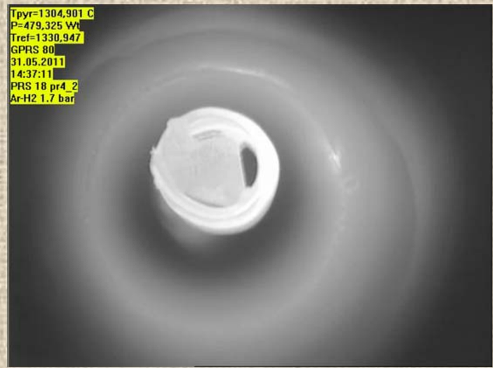
✓ **In accordance with DTA of sample #2**
T_{sol}=1316C, T_{liq}=1482C

Analysis conditions:
mass - ≈ 100 mg;
carrier gas – argon;
thermocouple type –
S (Pt /Pt - 10% Rh);
the working area heating rate – 5°C/min;
crucible material –Pt

UO_2 – FeO – CaO system: PRS18 test results (5)

➢ T_{liq} and T_{sol} determination of sample #4 in Galakhov microfurnace

1174°C



1305°C (deformation start)

1491°C

1532°C

1543°C (complete spreading)

✓ **Visual polythermal analysis in the Galakhov microfurnace T_{sol}=1305°C; T_{liq}=1543°C**

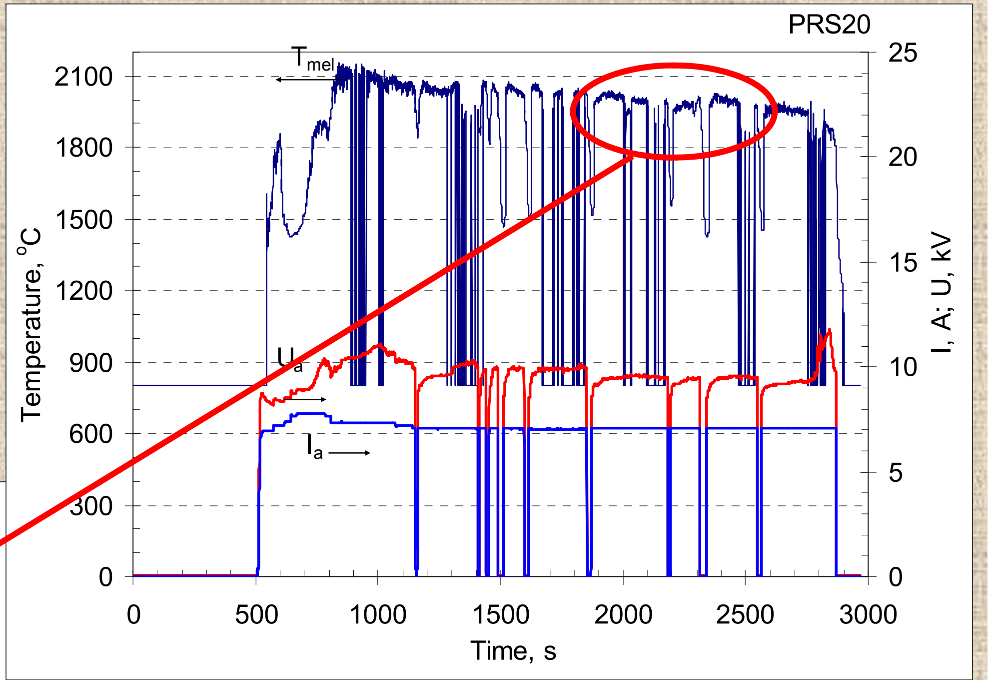
UO_2 – FeO – CaO system: PRS20 test results (6)

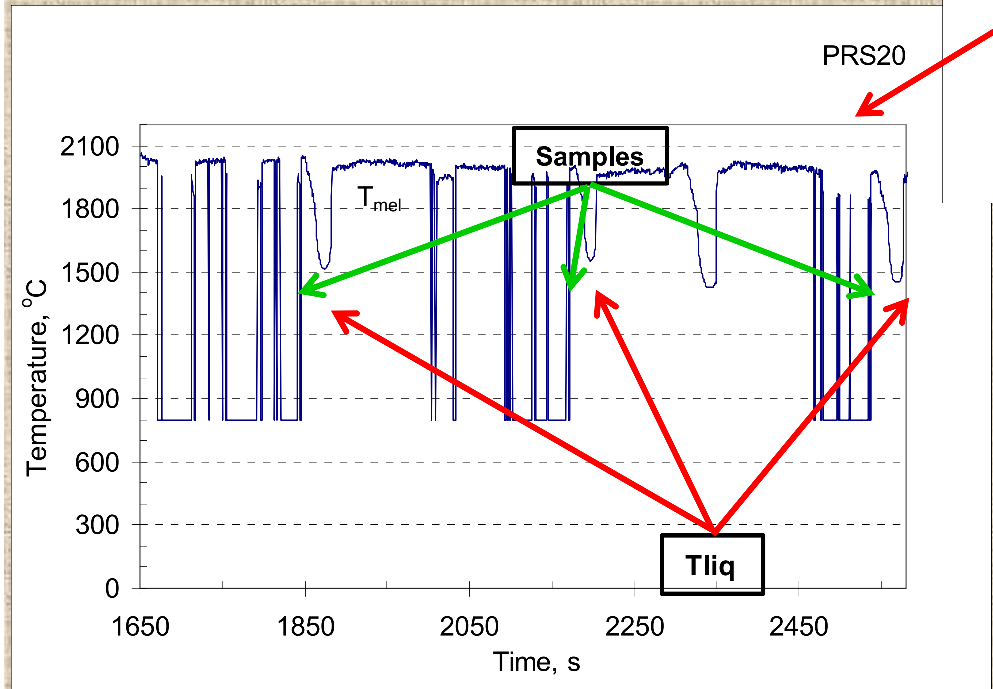
➢ **Experimental objectives**

T_{liq} determination by VPA IMCC

➢ **Charge composition**

mol.% 30 UO_2 + 40 FeO + 30 CaO



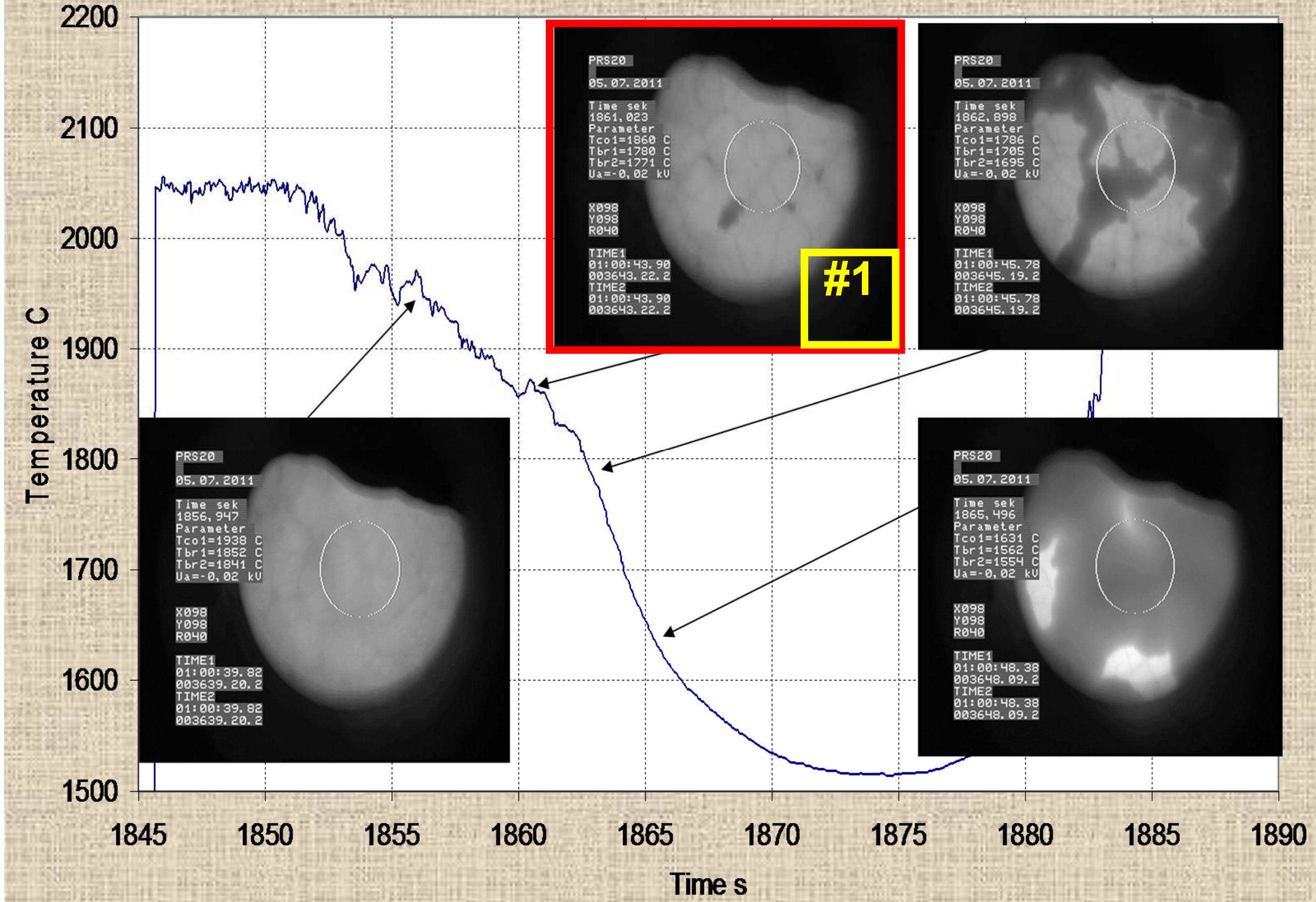


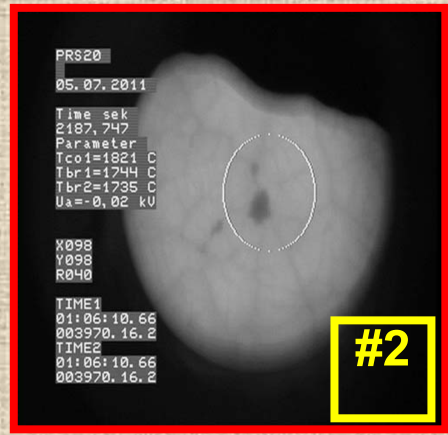
✓ **Initial crucible charge composition corresponds to PRS18**

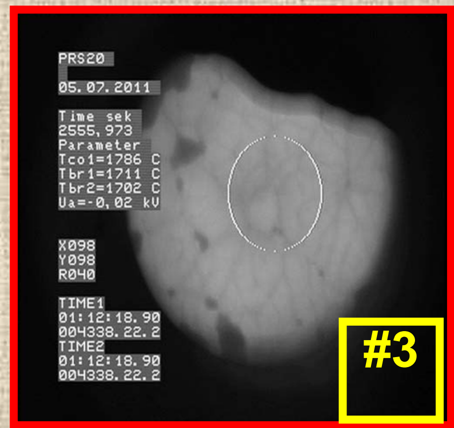
✓ **T_{liq} was measured 3 times by VPA IMCC with melt sampling**

UO_2 – FeO – CaO system: PRS20 test results (7)

➢VPA IMCC: Example of thermogram 1 from the test showing melt surface







✓ **Results of T_{liq} measurements: 1860, 1821, 1786 °C**

UO_2 – FeO – CaO system: PRS20 test results (8)

➢ Liquidus temperatures and compositions of melt samples measured by XRF and ChA

Sample	Composition, mass/mol. %						T_{liq} °C
	XRF			Cha			
	UO_2	FeO	CaO	UO_2	FeO	CaO)*	
1	61.41 27.83	25.15 42.84	13.44 29.33	61.23 27.54	24.22 40.95	14.55 31.41	1860±28
2	59.02 25.93	27.15 44.82	13.83 29.25	59.49 26.22	26.18 43.37	14.33 30.41	1821±27
3	59.76 26.50	26.49 44.15	13.75 29.36	59.05 26.16	28.91 48.14	12.05 25.70	1786±27

* - determined from residue

✓ The results of corium samples XRF and chemical analysis were found to differ significantly

✓ XRF results are believed to be more accurate, since the content of CaO was not determined by chemical analysis, but calculated from the residue

UO_2-FeO-CaO system: GPRS66, 75 test results (9)

➢ **Experimental objectives**

Validation of eutectic composition

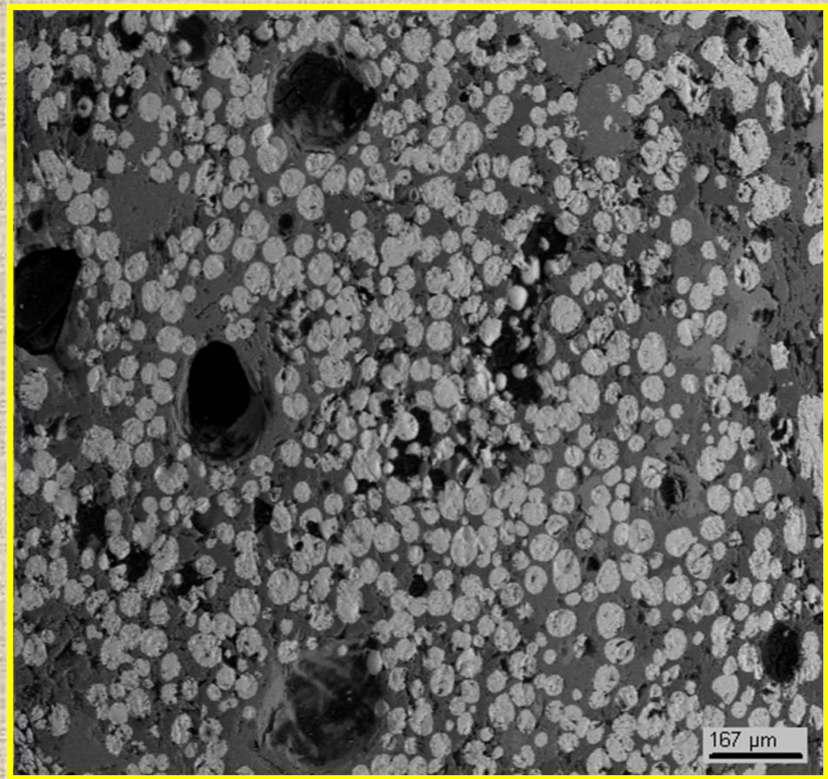
➢ **Annealing, melting and quenching in the Galakhov microfurnace (estimation of ternary eutectic position)**

Test	Content, mol.%			Temperature, °C	Exposure time, min	Note
	UO_2	FeO	CaO			
GPRS66	15.0	80.0	5.0	1000	5	Annealing
				1750	5	Melting
				1750-900	240	Cooling at 100°C/h
GPRS75	3.0	87.0	10.0	1000	60	Annealing
				1400	5	Melting
				1400-900	240	Cooling at 100°C/h

✓ **UO_2 of >99.0 % purity, SiO_2 of 99.99% purity, FeO of >99.0 % purity, charge mass – 150 mg, molybdenum crucibles ∅ 6 mm**

UO_2 – FeO – CaO system: GPRS66 test results (10)

- SEM/EDX



167 µm

✓Eutectic structures in GPRS 66 are absent.
Apparently, the initial melt composition is far from the ternary eutectics

✓SEM/EDX GPRS75 in progress

UO_2–FeO–CaO system: test results (11)

- ✓ T_{liq} of PRS 18 and 20 compositions has been determined by VPA IMCC
- ✓ T_{liq} and T_{sol} of PRS 18 compositions has been determined by VPA in Galakhov microfurnace
- ✓ An additional experiment on the validation of the ternary eutectics composition has been made
- ✓ SEM/EDX of melt samples and determination of final solubility is in progress

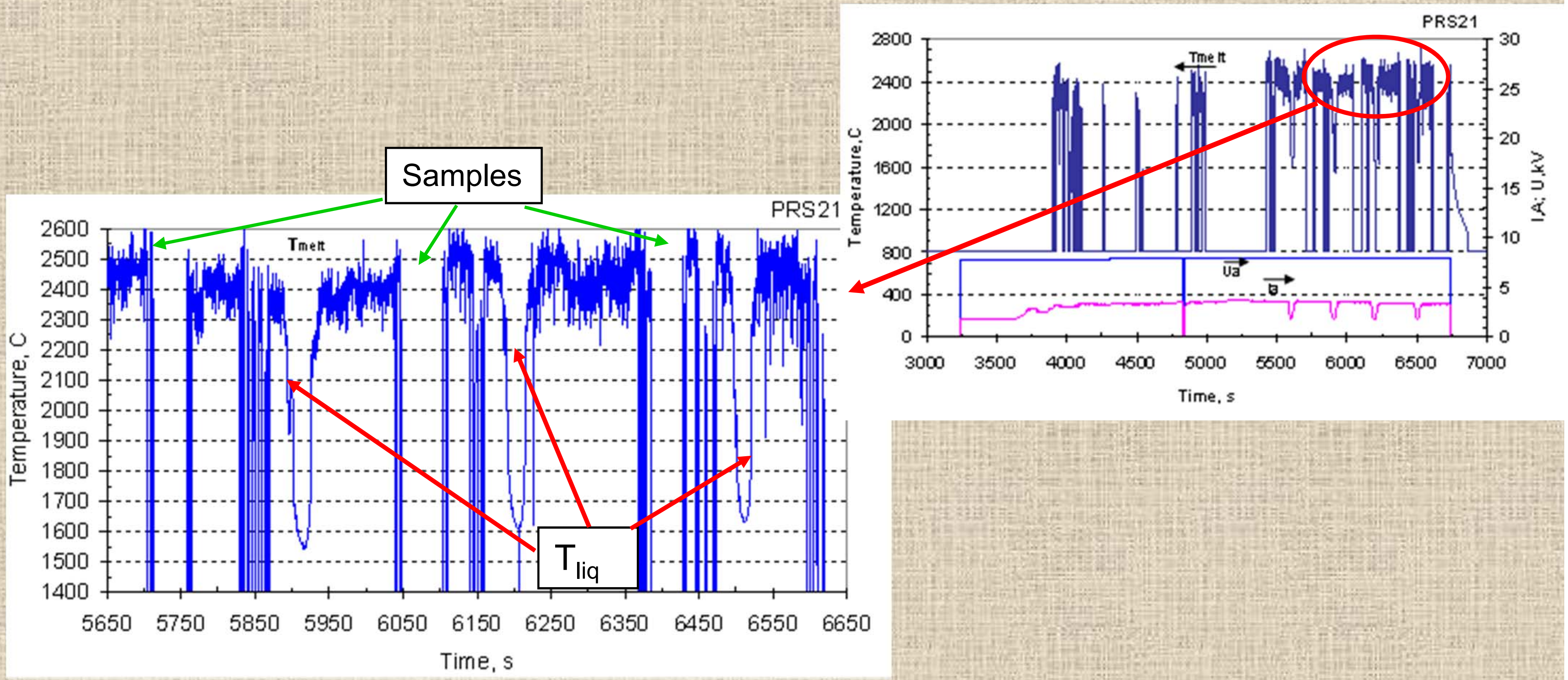
Multicomponent prototypic corium. French system

➢Experimental objectives

T_{liq} determination by VPA IMCC

➢Charge composition

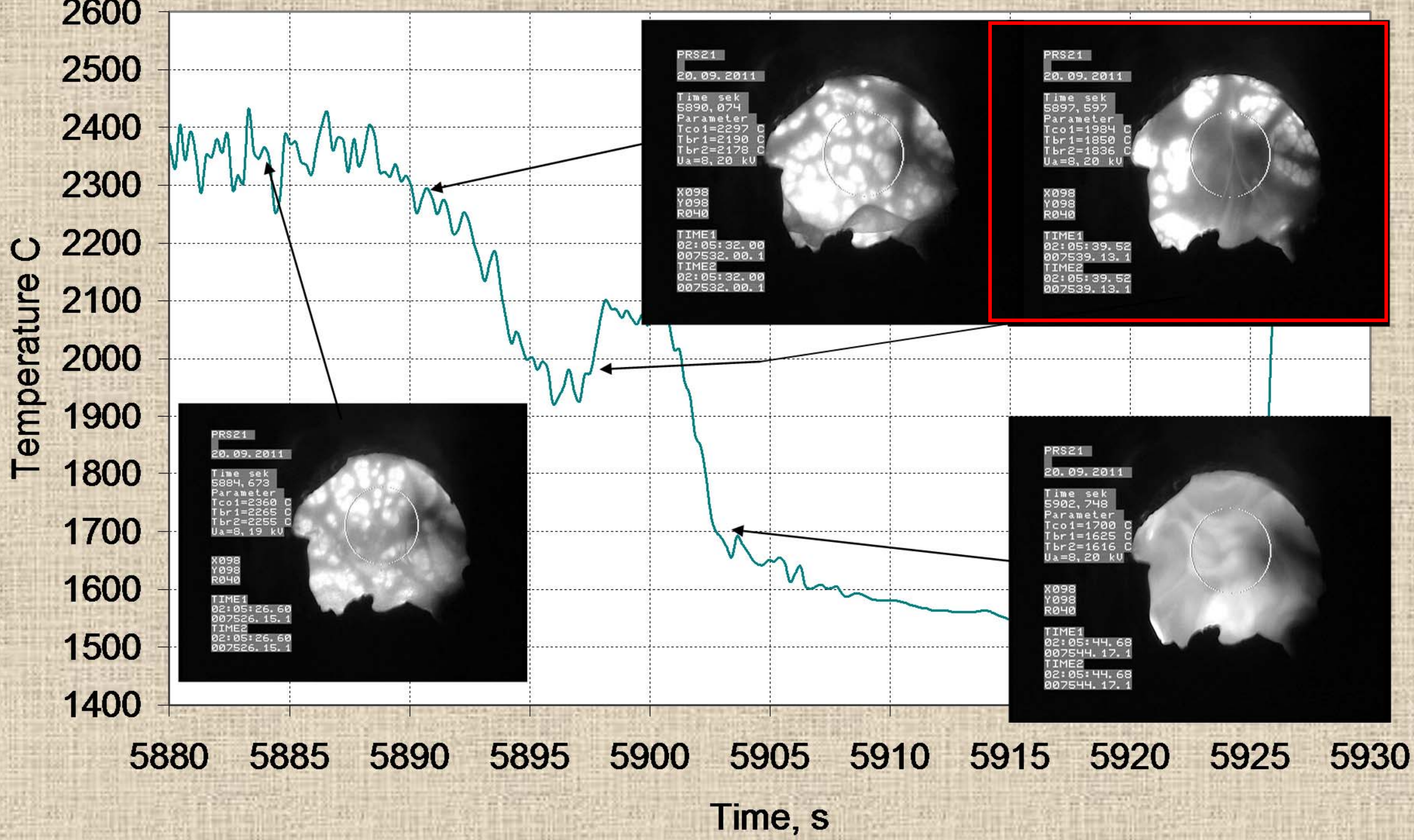
wt.% 33.5 UO_2+21.2 ZrO_2 +22.2 SiO_2+6.0 CaO+15.6 FeO+0.1 MgO+1.4 Al_2O_3



✓T_{liq} was measured 3 times by VPA IMCC with melt sampling

Multicomponent corium : PRS21 test results (2)

➢VPA IMCC: Example of thermogram 1 from the test showing melt surface



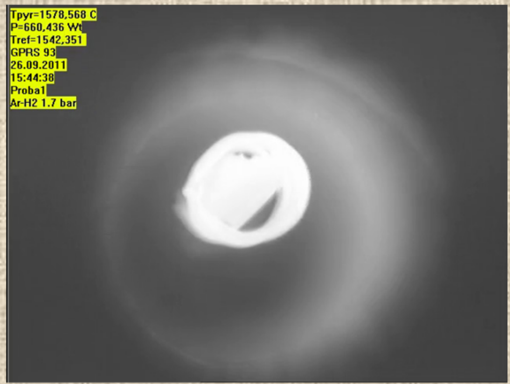
✓ Results of T_{liq} measurements: 2000±70°C

✓It was not possible to interpret the second and third measurements

Multicomponent corium : PRS21 test results (3)

➢T_{sol} and T_{lig} determination in the Galakhov microfurnace

Sample #1



1528°C (deformation start)

1652°C

1750°C

1957°C (complete spreading)

✓**Visual polythermal analysis in the Galakhov microfurnace**
T_{sol}=1528°C; T_{liq}=1957°C

Multicomponent corium : PRS21 test results (4)

- ✓ T_{liq} of PRS 21 compositions has been determined by VPA IMCC
- ✓ T_{liq} and T_{sol} of PRS 21 compositions has been determined by VPA in Galakhov microfurnace
- ✓ Cha, XRF and SEM/EDX in progress

Results obtained with laser pulse heating facility

Joint Institute for High Temperatures, Moscow ***(IVTAN)***

The main task of IVTAN laser-heating experiments in the PRECOS project is an issue of possible immiscibility of liquid in the U-Zr-O system. The required set of samples have been prepared at NPO “LUCH” however the transfer of the samples to IVTAN is delayed due to the license required for experiments with natural uranium. Therefore it has been mutually agreed that in the mean time the experimental work is focused on the following systems not containing natural uranium: Zr-ZrO_2, ZrO_2 –FeO and CaO.

The licensing procedure was completed in August 2011

ФЕДЕРАЛЬНАЯ СЛУЖБА
ПО ЭКОЛОГИЧЕСКОМУ, ТЕХНОЛОГИЧЕСКОМУ И АТОМНОМУ НАДЗОРУ

ЛИЦЕНЗИЯ

Регистрационный номер ЦО-09-501-6157 от 19 июля 2011 г.

Лицензия выдана Учреждению Российской академии наук Объединенный институт высоких температур РАН (ОИВТ РАН)

Юридический адрес лицензиата: 125412, г. Москва, ул. Ижорская, д. 13, строение 2

Лицензия дает право на использование радиоактивных веществ при проведении научно-исследовательских и опытно-конструкторских работ

Основание для выдачи лицензии: заявление Учреждения Российской академии наук Объединенный институт высоких температур РАН, решение руководителя Центрального межрегионального территориального управления по надзору за ядерной и радиационной безопасностью Федеральной службы по экологическому, технологическому и атомному надзору от 19.07.2011 г. № 6157

Срок действия лицензии до 04 июля 2016 г.

Лицензия действует при соблюдении прилагаемых условий действия лицензии, являющихся ее неотъемлемой частью

М.П. **Руководитель органа лицензирования** **В.А. Снигирев**

Серия А В № 336837

Laboratory Room Dedicated for Handling of Natural Uranium Samples

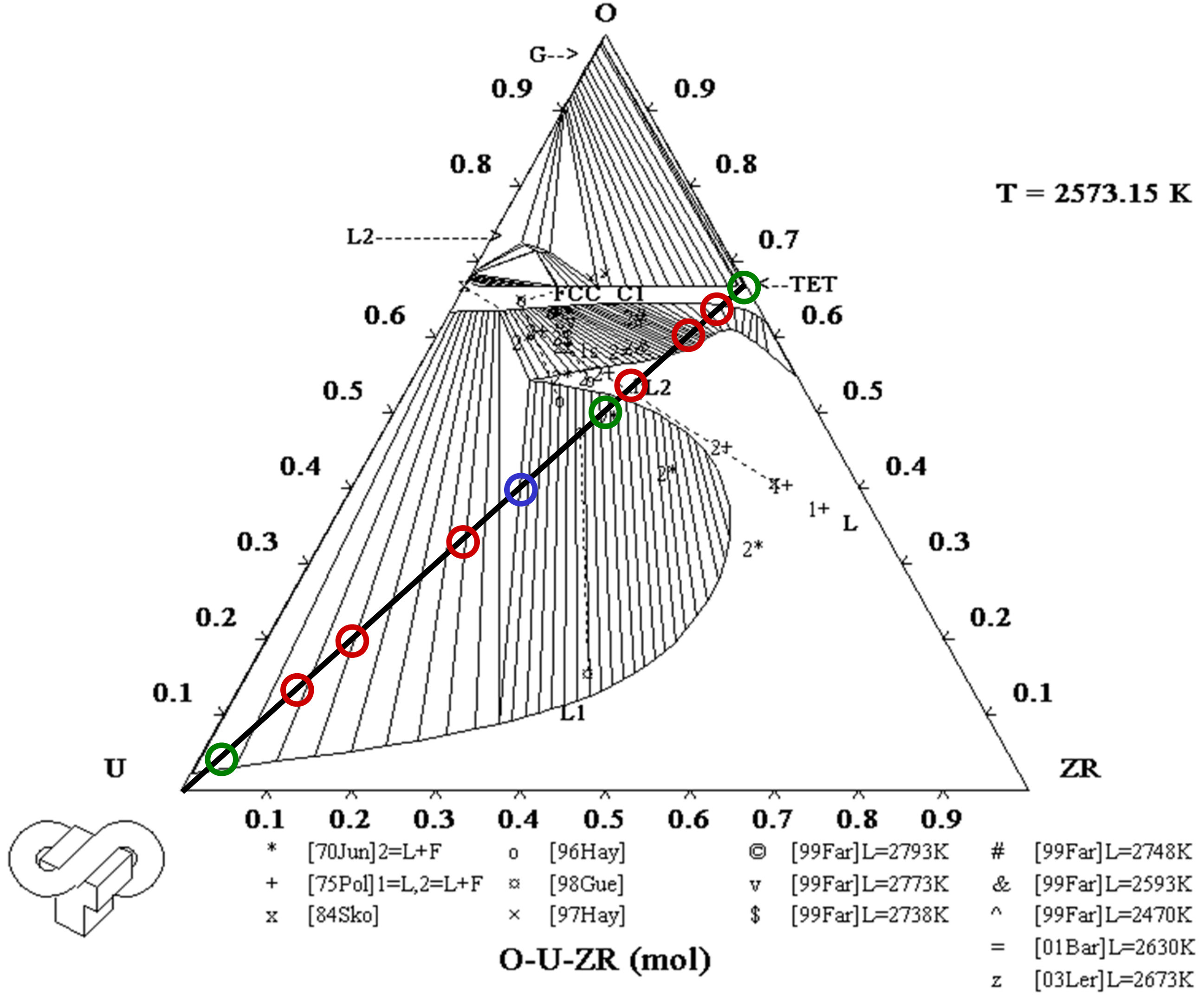
U-ZrO_2 samples for laser-heating experiments

Recently it turned out that some samples prepared at NPO Luch are completely disintegrated into powder.

Since we have much less samples at our disposal that it was planned previously one has to update the test matrix.

Disintegration of U-ZrO_2 samples sintered at NPO "Luch"

- Samples remaining intact (green circles)
- Samples partially disintegrated (blue circles)
- Samples completely disintegrated (red circles)



ZrO_2-FeO system

Main tasks:

• Testing of applicability of the mixed oxide samples prepared by blending followed just by pressing without any heat treatment

• Definition of number of the laser-melting cycles for complete homogenization of initially inhomogeneous low density samples

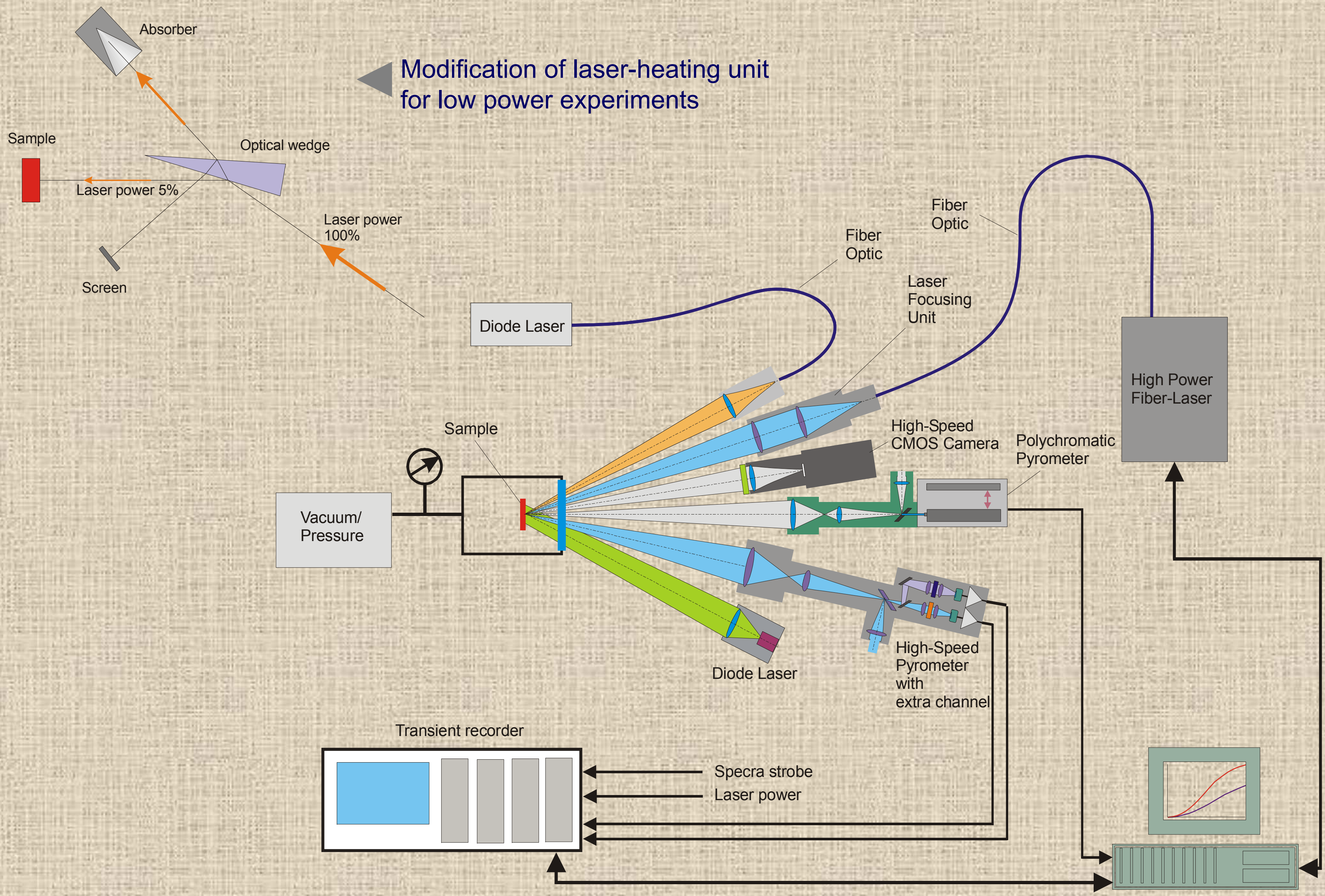
•Slight modification of the setup for experiments with low density samples

Prepared samples:

1. Cylindrical samples (dia 12x3 mm) of various compositions are prepared at IVTAN by blending followed by pressing. The samples have low density (ca. 70% of the theoretical density). The latter means that test samples have to be prepared by the self-crucible melting using several consecutive laser shots;

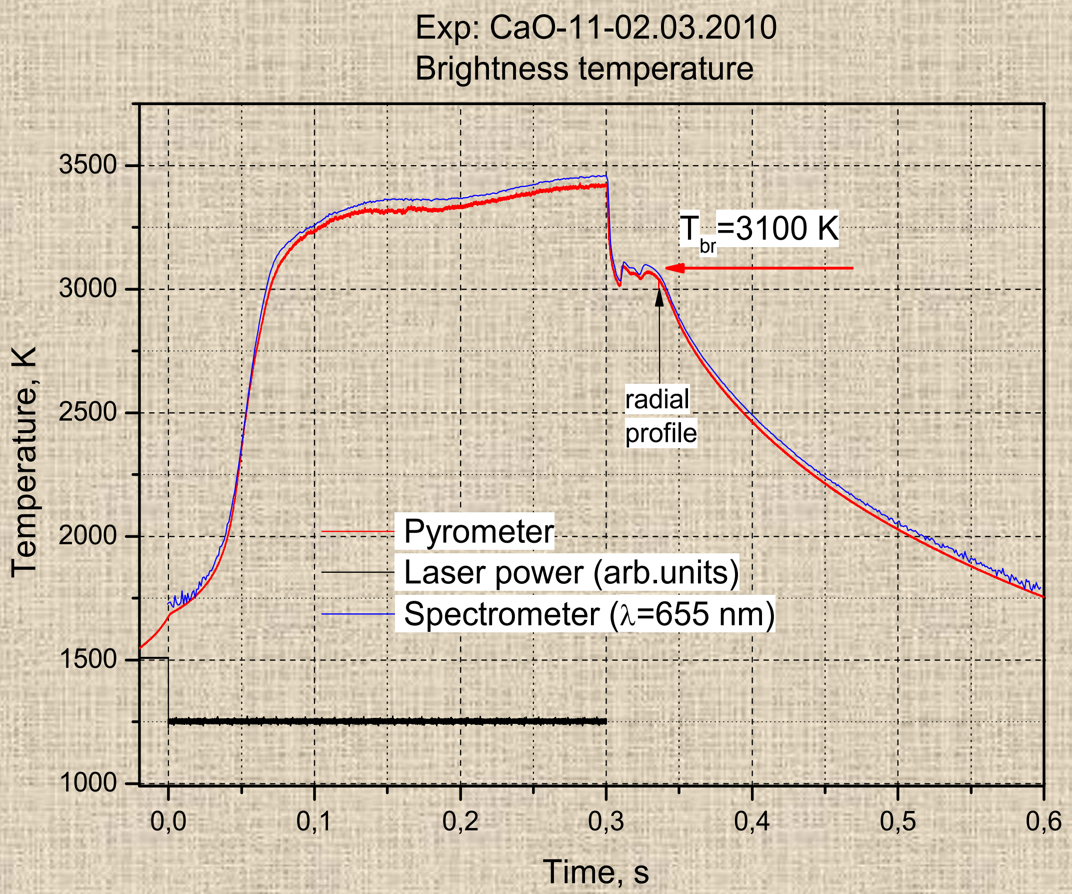
2. Samples cut from ingots formed in course of CORD-10 and CORD-15 tests. The samples are very dense and even the first laser shot can be informative. However the further control of the composition is required.

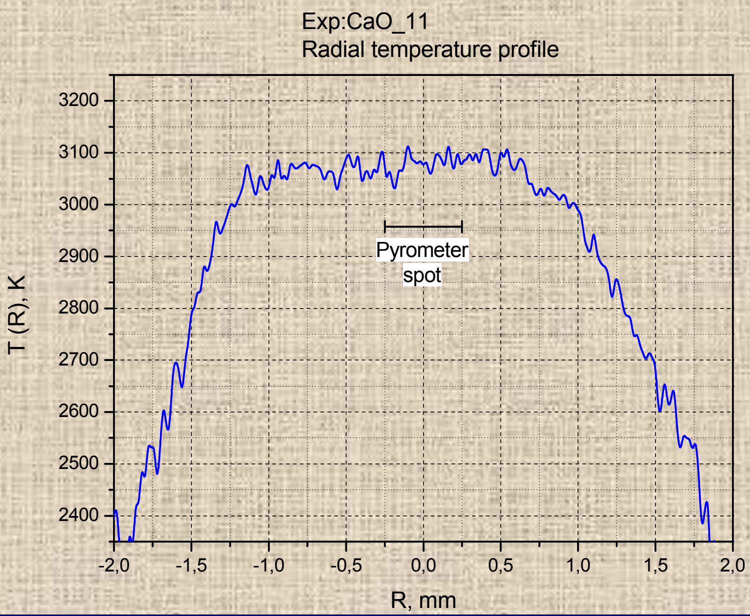
Schematic of the Set-Up



Modification of laser-heating unit
for low power experiments

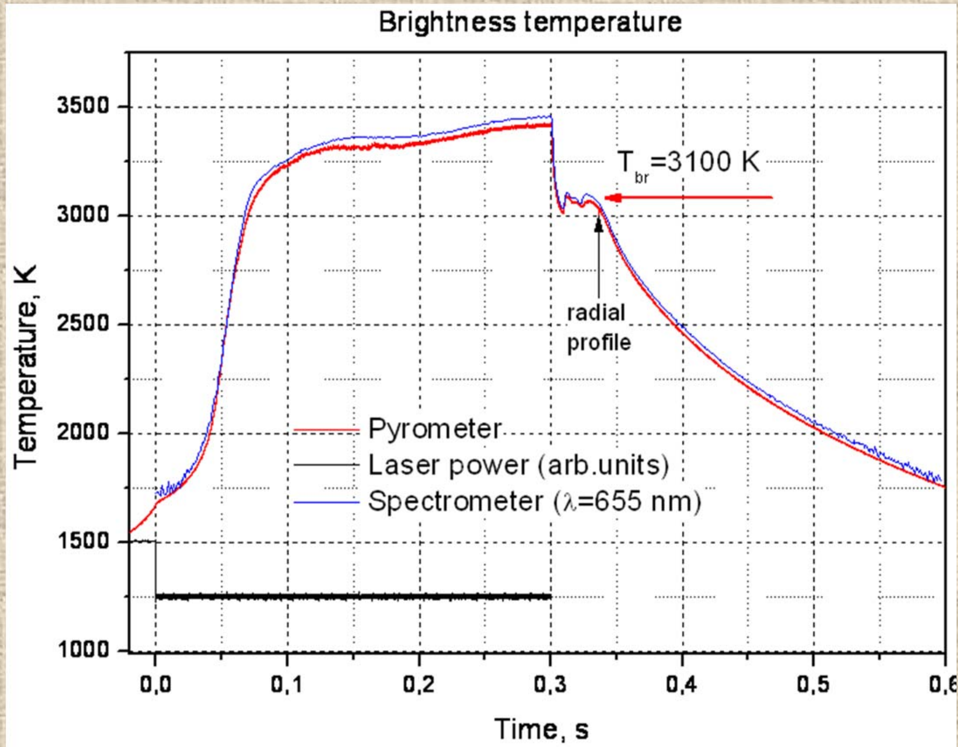
Laser-Pulse Melting of CaO



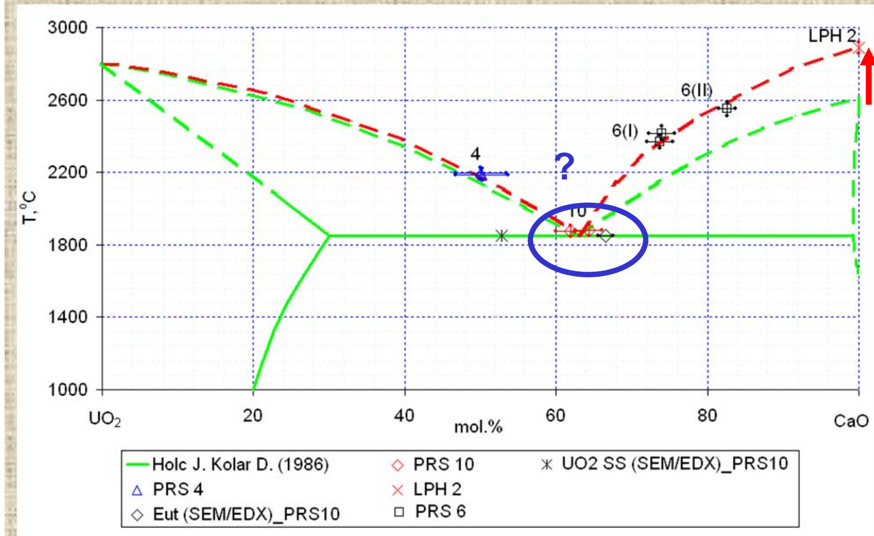


CaO Melting Point Measurements

Experimental Thermogram



Test results on UO_2 - CaO system



Peculiarities of CaO melting:
Very low absorption coefficient below 1500-2000K
The boiling temperature is less than 200 K higher than the melting temperature

- **Assuming that ε=0.85-0.9 →Tmelt= 3160 ± 30K**
- **It is planned to make direct measurements of ε at the M.P. of CaO.**

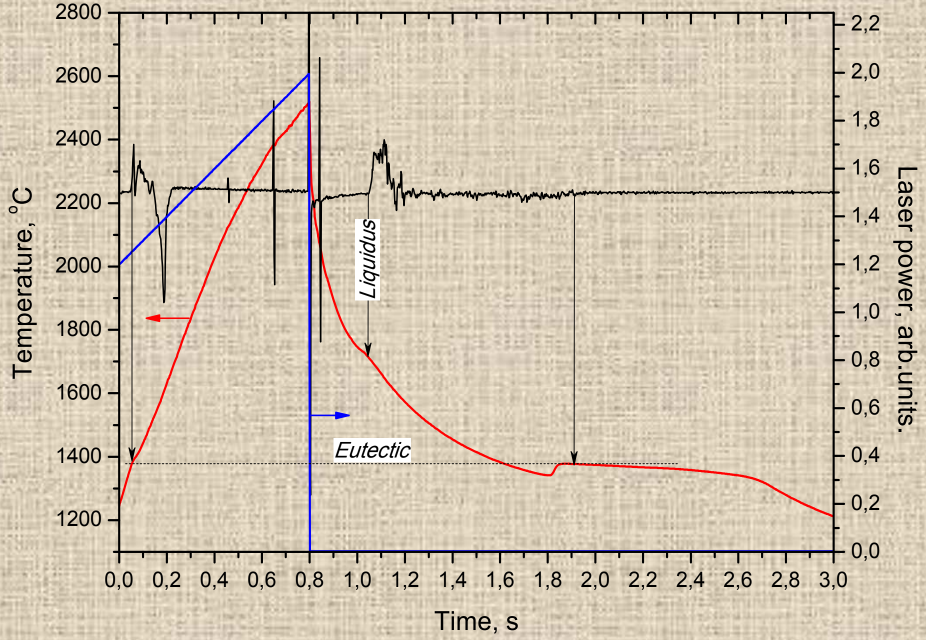
ZrO_2-FeO System

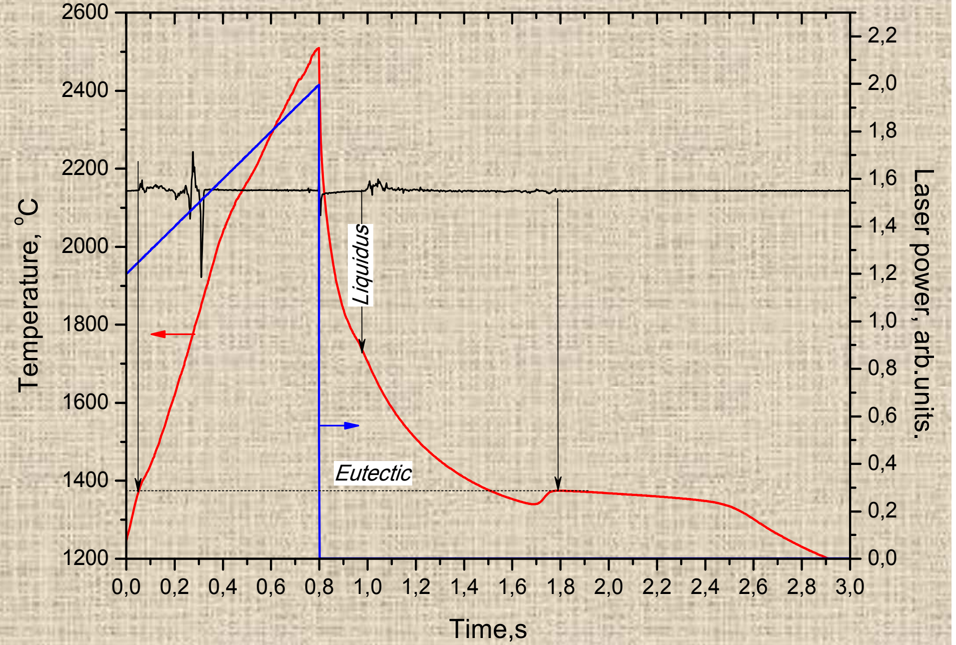
•Verification of the laser-heating technique on relatively well studied binary system

• Testing of applicability of the mixed oxide samples prepared by blending followed just by pressing without any heat treatment

• Definition of number of the laser-melting cycles for complete homogenization of initially inhomogeneous low density samples

Example of the thermogram on IVTAN samples



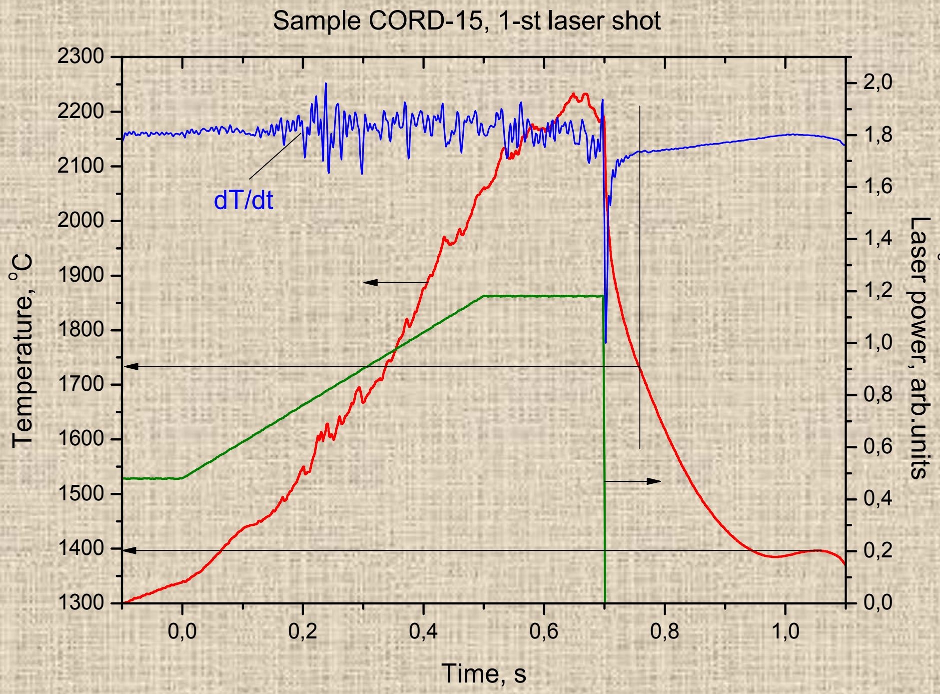


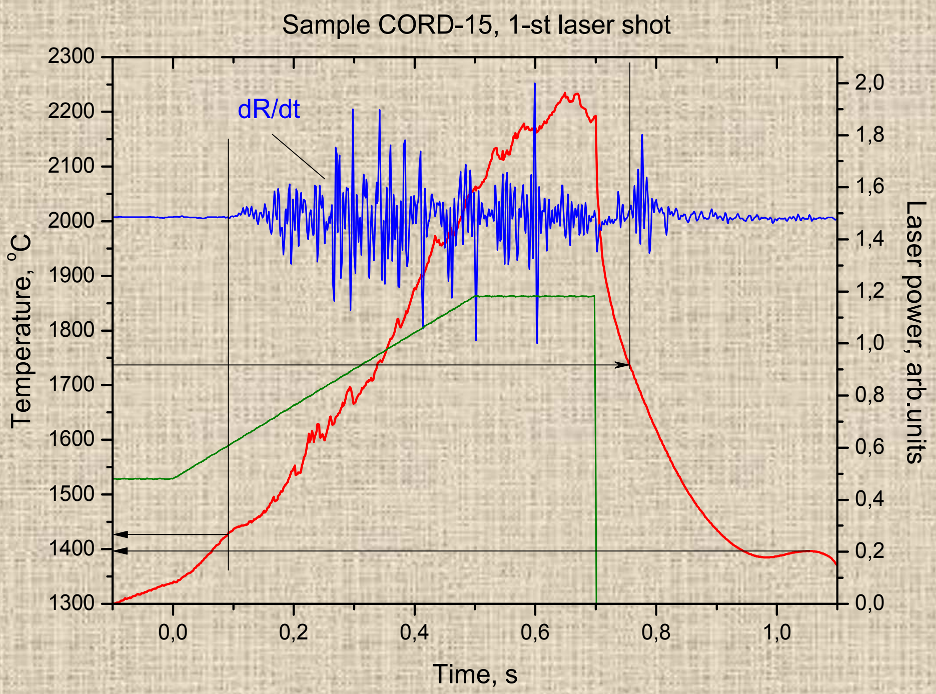
Laser-melting of CORD-15 sample

-Only the first shot is successful

-Eutectic point is clearly seen both on ascending and descending flanks of the thermogram

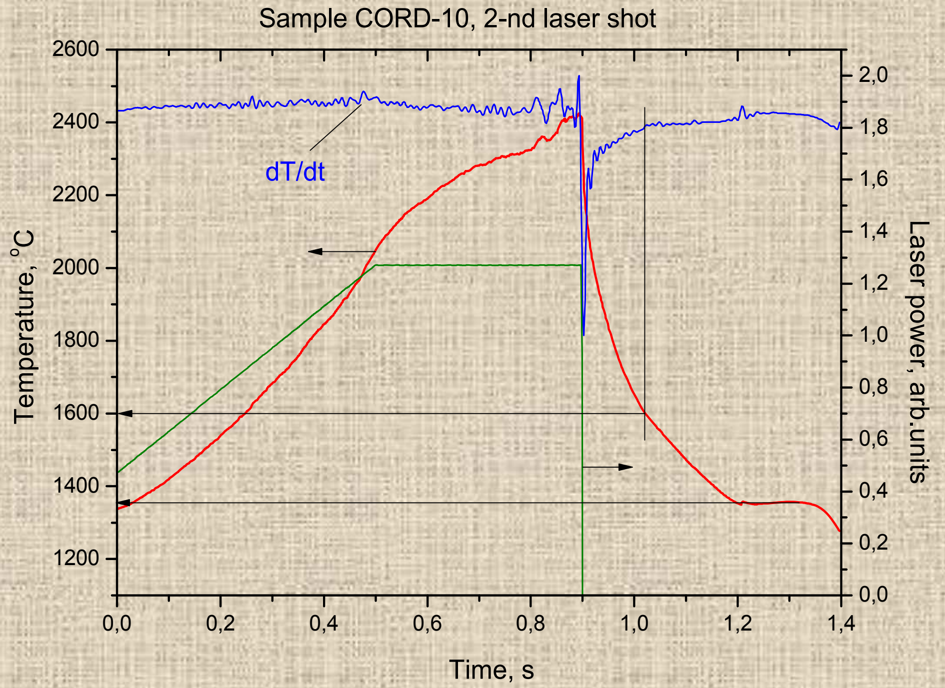
- Both variation of the reflection signal and the thermogram are used for identification of the solidus and liquidus points

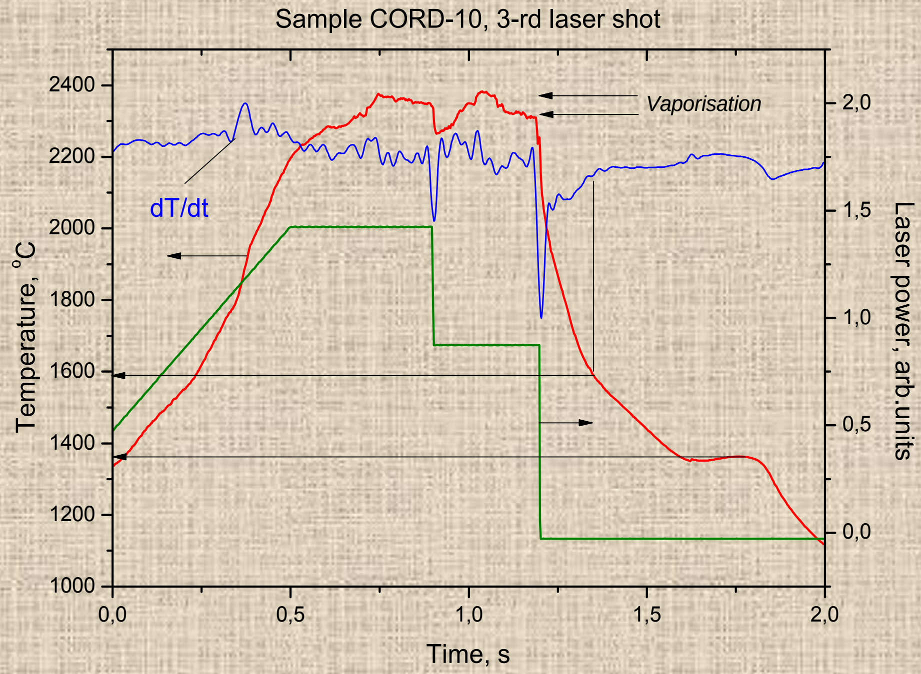




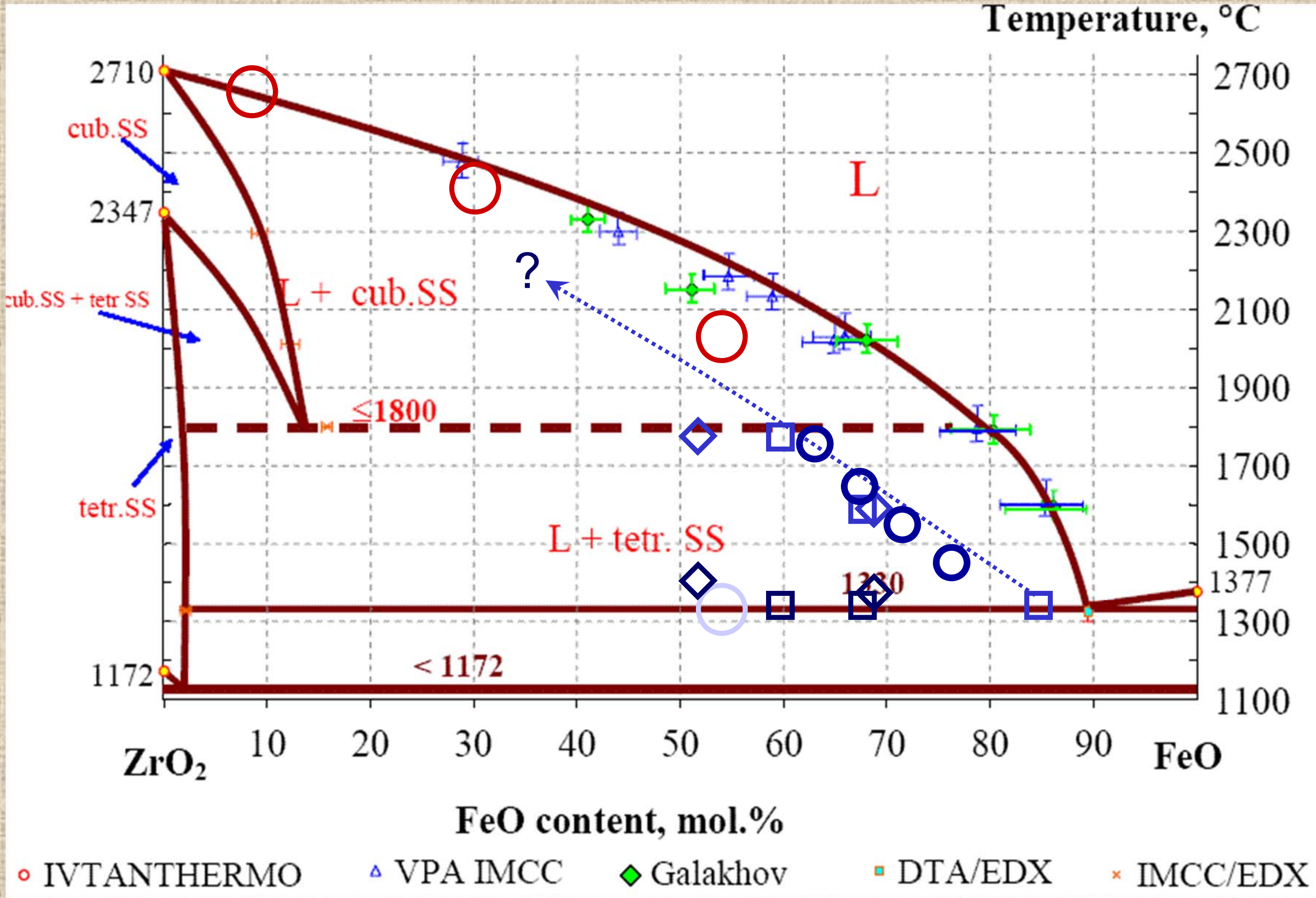
Laser-melting of CORD-10 sample

One can see reproducibility of eutectic temperature and suggested liquids temperature. Their positions are independent on laser power density and intense vaporisation (in case of shot 3)





ZrO_2-FeO System: Laser-Heating Results



○ - IVTAN (2010), laser-heating □ -March 2011 ◇ -May 2011, CORD samples

○ - Sept. 2011

Proposed tasks for the remaining extension period

1. Measurements on suboxidized Zr-Fe-O system to verify the existence and parameters of immiscibility cupola. Further experiments with ZrO_2-FeO system on IVTAN samples and CORD samples. Possible use of the IVTAN high pressure setup can be considered
2. Study of possible immiscibility in Zr-U-O system on samples prepared at NPO “Lutch”
3. CaO?

Conclusion

•The licensing problem is still topical however all the documents are accepted by “Gosatomnadzor” and the bureaucratic process there is expected to be finalized at the end of the month

•The great number of laser-melting experiments are performed on ZrO_2 –FeO system. It is observed that liqudus values for both IVTAN and CORD samples are remarkably lower than previously obtained by conventional methods (Galakhov microfurnace, *etc*).

4rd PRECOS project meeting

(June 8, 2011, St. Petersburg)

Objectives:

- **Discuss test results ternary oxidic sistems:**

  UO_2-FeO-SiO_2; UO_2-FeO-CaO

- **Discuss the compositions of multicomponent prototypic corium for fulfilling Task 4 of the experimental matrix**

- **Discuss and agree upon future works**

- **To discuss PRECOS project prolongation without additional funding**

Concluding remarks

- ✓ On agreement with collaborators it is planned to prolong the project for 6 months without additional funding
- ✓Work scope for quarters # 14 - 15
  - Complete studies in the FeO-SiO_2-UO_2, FeO-CaO-UO_2 systems
  - Continue studies of the multicomponent prototypic corium (compositions of German and Russian partners)
  - Start studies of the U-Zr-O system (by LPH in IVT RAN)
  - Prepare final reports on the SiO_2-UO_2, FeOy-ZrO_2, CaO-UO_2 systems