Ultra-Low-Radioactive titanium alloy as a promising construction material for low background cryostats

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SINP MSU

LIDINE 2025   Oct 21 – 24, 2025

Current status of the problem

Construction metal materials characteristics

- Mechanical properties
- Radiopurity figures
- Availability in bulk quantities
- Cosmogenic activation properties
- Price

Metal materials in use:

- Copper (electroformed and preselected)
- Stainless steel (preselected)
- Titanium (preselested)
- ?? Other metals and alloys
  (Al, Ni, Zr .....)

What to do if we need 20-50-100 t of metal construction material？

1 Selecting the material from the finished rolled metal warehouse
Minimal impact on the manufacturing process
SS, Grade-1 Ti, bulk Cu

2 Development of a special process or/and equipment for the production of ultra-low-background material
Electroformed Cu (ULR Ti)

3 Minor （？） modification of industrial processes and continuous monitoring of raw materials and prevention of recontaminations
ULR Ti

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   Electroformed Cu (ULR Ti)

   much more expensive material

3. Minor （？）modification of industrial processes and continuous monitoring of raw materials and prevention of recontaminations
   ULR Ti

   a bit more expensive material

The original goal of the project was as follows:

- to investigate the possibility of creating a production cycle of parts from ultrapure titanium alloys with the following radioactivity levels:

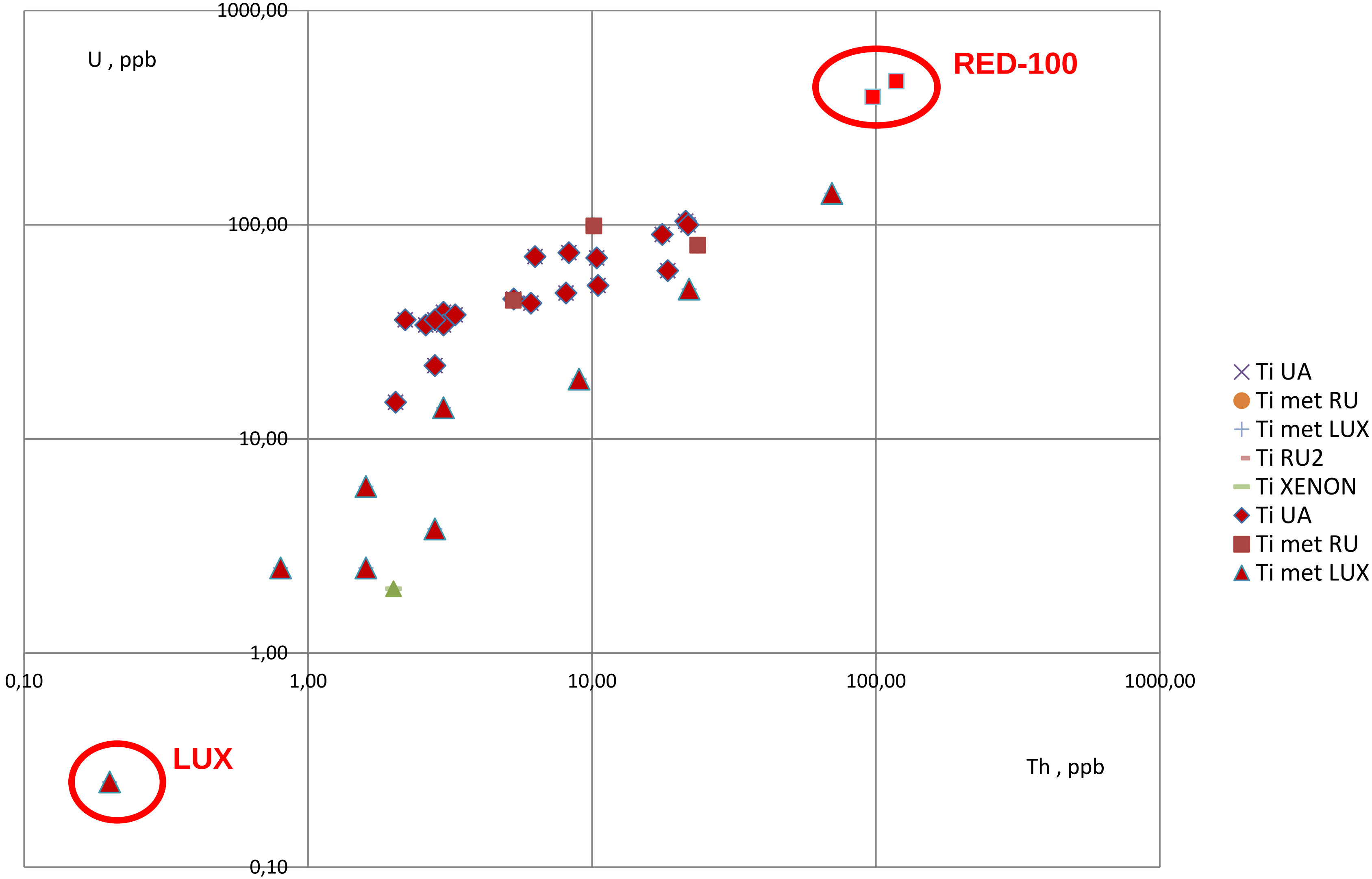
^{238}U & ^{232}Th < 1 mBq/kg (equivalent to < 0,1 ppb U & < 0,25 ppb Th)

- to provide DarkSide experiment with ultra-pure construction material to produce the titanium apparatus (cryostat/vessel) with total mass more than 20 t

It could be done only if the industrial production of ULR titanium alloys will be carried out with the maximum possible use of industrial equipment and technology. Traditional pre-selection of materials in the product warehouse doesn't work in the case of ~20t material needs

Russian titanium industry was considered as a good candidate for the cooperation.

First stage results (2012)
Ti metal samples from different sources (U/Th difference ~4 orders !)



Why it is so widely distributed ??
What are the sources of U/Th ??

REMEMBER THE GOAL: ^{238}U < 0,10 ppb ~ 1 mBq/kg
^{232}Th < 0,25 ppb ~ 1 mBq/kg

"ULR Ti meeting" in Moscow (SINP MSU) in August 2015

ПИСЬМО О НАМЕРЕНИЯХ

Изучение физических принципов очистки титана и разработка производственных процессов для получения сверх чистого титана для нового поколения низкофоновых экспериментов.

Введение

Низкофоновые детекторы большого объема с порогом регистрации событий в области низких энергий являются одними из наиболее многообещающих инструментов исследования, способные привести к открытиям в области современной астрофизики и физики частиц.

За последнее десятилетие в этой области науки произошли события фундаментального значения. Впервые удалось шагнуть за пределы Стандартной Модели (фундаментальной теории физики элементарных частиц). Благодаря научным данным, собранным такими экспериментами по изучению солнечных нейтрино как SNO и Borexino, удалось заключить, что нейтрино обладает массой и участвует в процессе осцилляций, который заключается в изменении флейвора нейтрино. Кроме того, в процессе проведе-

LETTER OF INTENT

Study of physical principles of titanium purification and development of production processes of ultra-pure titanium for the new generation of low background detectors

Introduction

Low-background, low-energy, large volume detectors are among the most crucial tools for the next generation of discoveries in astroparticle physics.

In the last decade, this field produced a very important breakthrough by providing the first piece of physics beyond the standard model: solar-neutrino detectors such as SNO and Borexino helped establish that neutrinos are massive and undergo oscillations resulting in flavor change. In addition these two experiments, and Borexino in particular, contributed new technology able to abate radioactive backgrounds in low energy experiments by many orders of magnitude.

LNGS, включая возможность использования уникального оборудования для анализа образцов, планируется широко применять при реализации данного проекта. Разработка метода массового производства сверхчистого титана будет являться важным инновационным российским вкладом в создание первоклассных экспериментов нового поколения в области физики частиц.

Директор НИИЯФ МГУ
профессор Панасюк М.И.

Директор ИФТТ РАН
член-корреспондент РАН Кведер В.В.

Директор НИИКМТП
МГТУ им. Н.Э. Баумана
профессор Шиганов И.Н.

Директор INFN LNGS
Профессор Стефано Рагацци

The availability of a mass production method for ultra-pure titanium will represent an original and innovative contribution of Russia to first-class physics experiments.

SINP MSU director
Professor Panasiuk M.I.

ISSP RAS director
Professor Kveder V.V.

NII KM TP BMSTU director
Professor Shiganov I.

INFN LNGS director
professor Stefano Ragazzi

"ULR titanium" driving forces :

The project was started in 2011 within the DarkSide collaboration

LLC «MODERN TECHNOLOGIES»

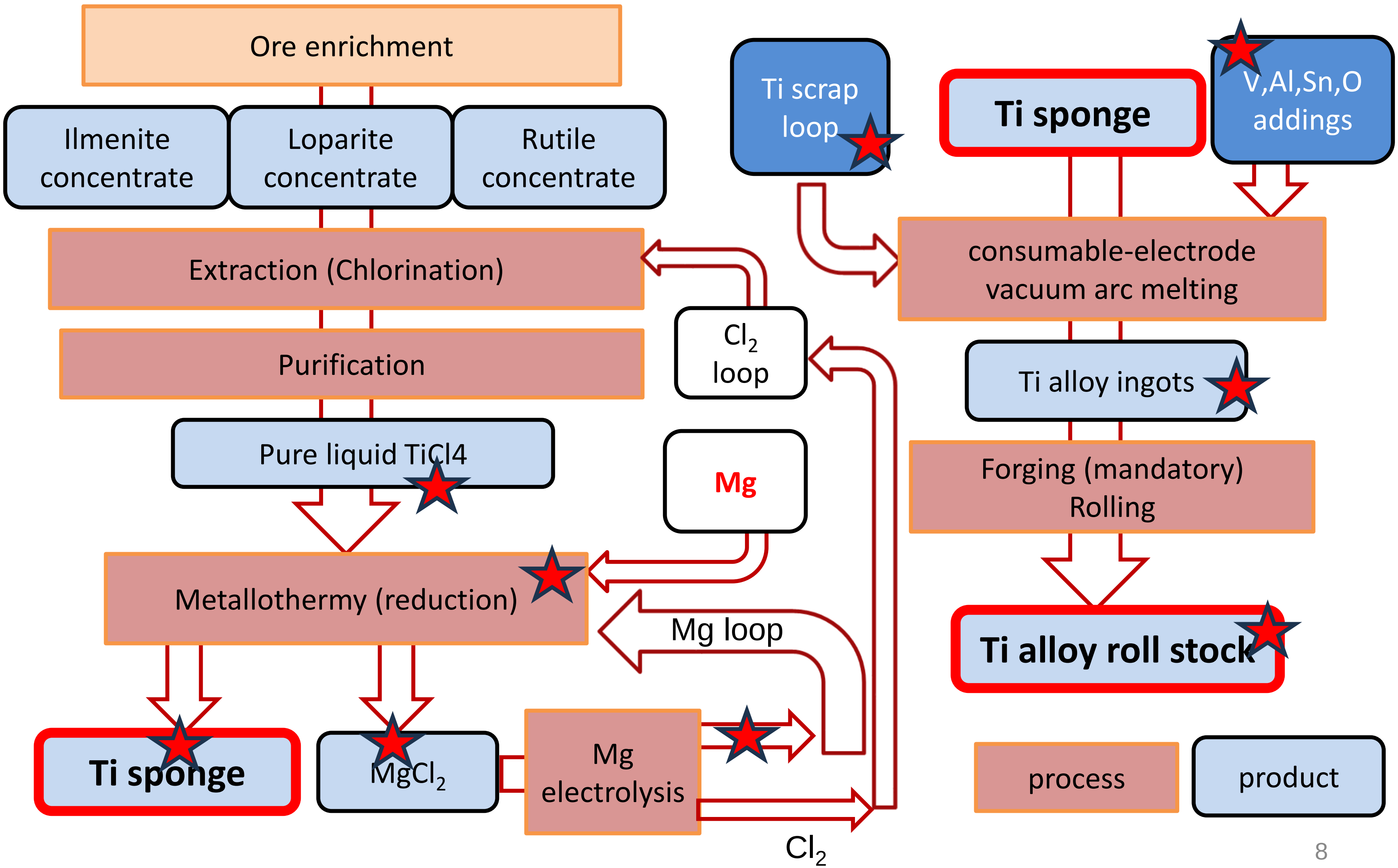
Lomonosov MSU Skobeltsyn Institute of Nuclear Physics
INFN supported the project till 2022
Tsung-Dao Lee Institute is in the game since 2025
Statement of the physical problem, R&D program development

JPSC "Solikamsk Magnesium Plant" (Solikamsk) (till 2020)
PJSC «VSMPO-AVISMA» (Verhnyja Salda)
Industrial partners from Russian titanium industry

Institute of Solid State Physics of RAS (Moscow) and
Belgorod State Research University (Belgorod)
LLC «MODERN TECHNOLOGIES» (Solikamsk)
Sapienza Universita di Roma
Theory of titanium alloys, unique metallurgical cleaning methods and installations, melting, rolling, welding, mechanical test

D. Mendeleyev University of Chemical Technology (Moscow),
INFN LNGS, INR RAS
ICP-MC & HPGe U/Th screening

Industrial titanium production cycle
"Kroll-process"



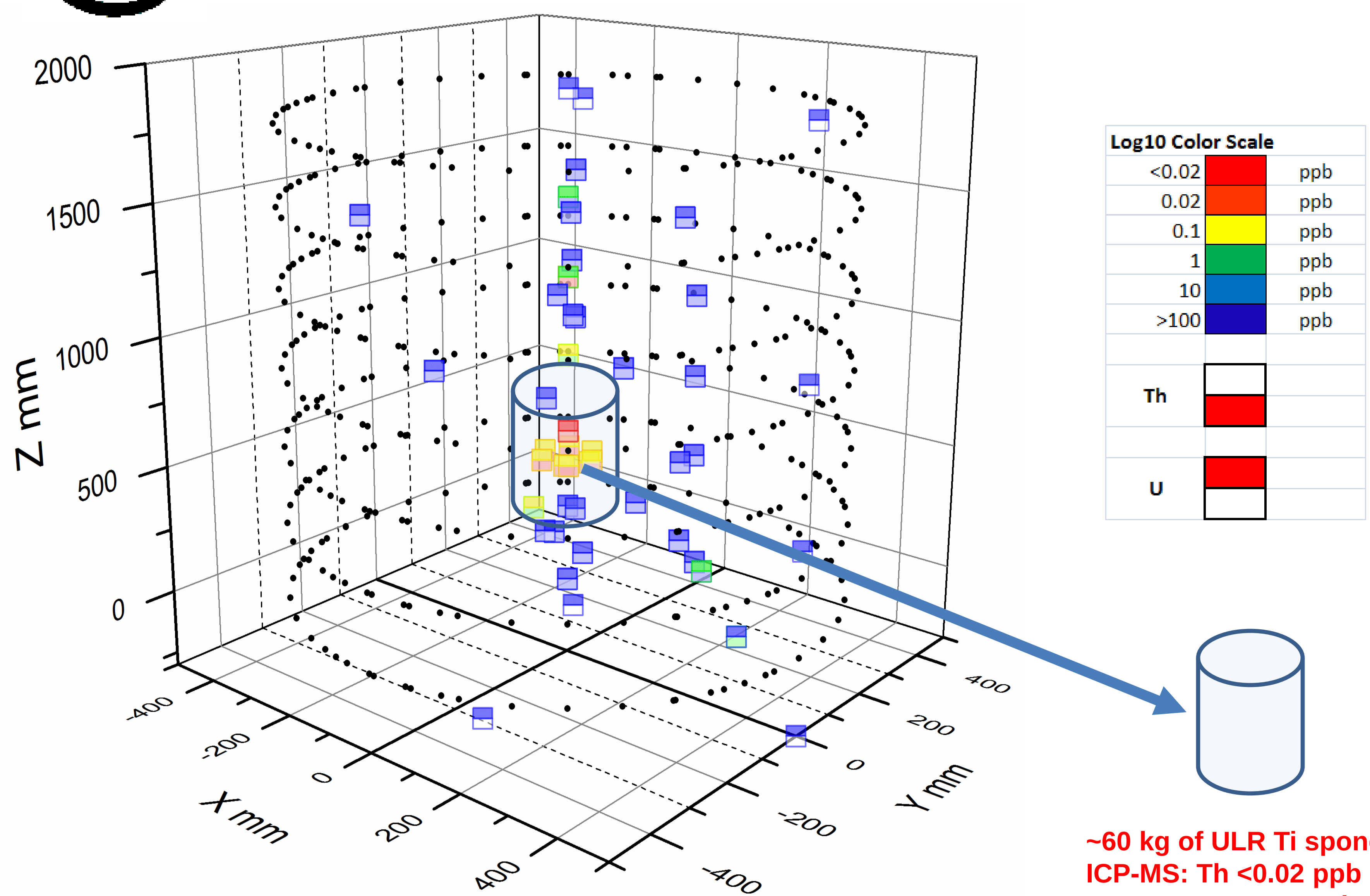
Important practical facts：

1) Titanium sponge is produced from two simple substances – titanium tetrachloride and magnesium.
2) $TiCl4$ is a liquid so could be distilled deeply
3) Mg is a metal with relatively low melting point so, could be purified via vacuum distillation also
4) Titanium shavings and sponge are good getters
5) GRADE-1 (VT-00/VT-01) titanium base alloys are chemically pure titanium with controlled gas impurities
6) Due to the critical application area of titanium alloys, the relevant industry is extremely well organized, and the material life cycle is traceable from raw materials to mechanical parts.

Nothing from above is true for stainless steel production !

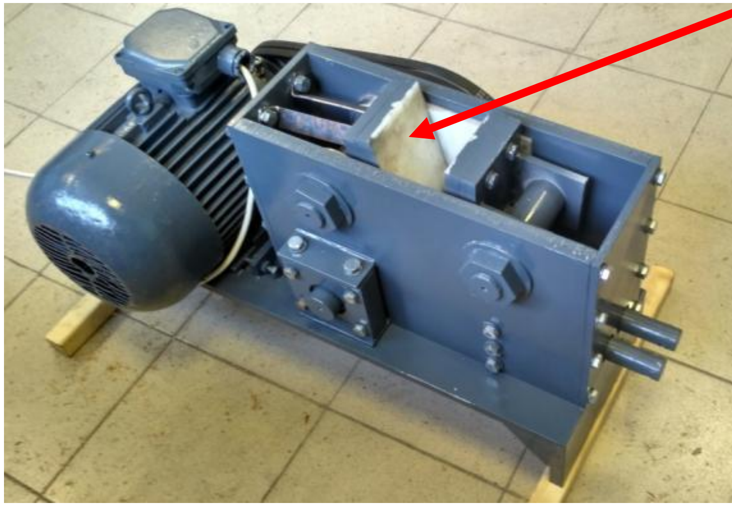
First stage experiment at JPSC "SMZ"

Confirmation of principal hypothesis about ULR-area of titanium 6t sponge block produced at JPSC "SMZ"



~60 kg of ULR Ti sponge
ICP-MS: Th <0.02 ppb
U <0.06 ppb

Special "ULR-Mill" lined with ULR titanium

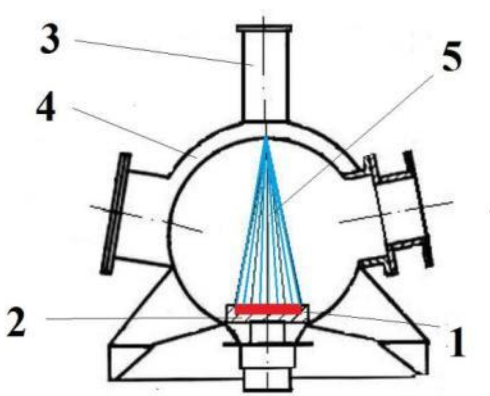


~ 50x200 mm

Isostatic compacting of the ULR titanium sponge

Electron Beam Vacuum Melting

Mass of each ingot is ~5 kg thickness - 18-20 mm, material losses during the melting up to 15-20% of the load. **Th <0.02ppb, U <0.06ppb**



1-liquid metal,
2-mould,
3-EB axial gun,
4-vacuum vessel,
5-electron beam

&

Cylindrical consumable electrodes are melted by electric arc into a water-cooled mold. The weight of each ingot is 1.9 kg.

	38-3	39-3	40-3
	ppb	ppb	ppb
Th	<0.02	<0.03	<0.02
U	<0.01	<0.02	0.005

Vacuum Arc Melting

Ultra Low Radioactive titanium samples manufactured from industrial preselected titanium sponge

Step 1. Production of low-radioactive sponge.
Step 2: Crushing sponge. Step 3: Isostatic compacting of the sponge.
Step 4: Vacuum arc- melting or Step 5: Vacuum e-beam-melting.
Step 6: Rolling with annealing. Step 7: Laser welding.

	ppb
Th	<0.02
U	<0.03

Mechanical and U/Th properties

Rolling processes have been developed and resulted materials mechanically tested. Optimal rolling process which gives necessary mechanical and keeping ULR properties was selected.

Material	HV (hardness)	$\sigma_{0,2}$, Mpa (conventional yield limit)	$\sigma_{\text{в}}$, Mpa (tensile strength)		
Industrial materials					
AISI 304		210	510		
VT1-00 / GRADE 1	**110-140**	**250-380**	**300-450**		
AISI 316	180-250	250-450	450-800		
VT16 (Al, V)	170-400	600-1000	1000-1500		
Ingots/sheet from pure Ti-sponge				U, ppb	Th, ppb
Original ingot from pure Ti-sponge	140 std.dev 30	150	220	**<0.003**	**< 0.05**
Sample №2	160 std.dev 53	410	565	**0.83**	**0.67**
Sample №3	**165 std.dev 10**	**420**	**590**	**<0.003**	**< 0.05**
Sample №4	155 std.dev 15	250	390	**0.16**	**0.14**
Sample №5	160 std.dev 45	280	400	**<0.003**	**0.017**

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THIS MATERIAL COULD BE USED FOR PRESSERUSED APPARATUS SUCH AS CRYOSTATS

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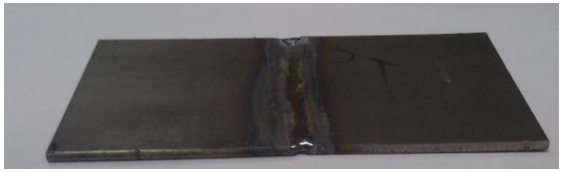
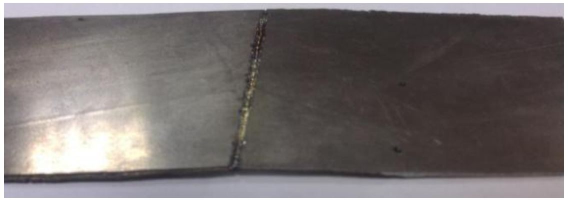
Study of ULR titanium welding

An essential step in the manufacture of the cryostat is welding. The welding process should not lead recontamination by radioactive impurities, and the mechanical properties of the welds should not impair the mechanical strength of the assembly. To investigate the possibility of creating a weld sheets radiation pure titanium welding process three were selected:

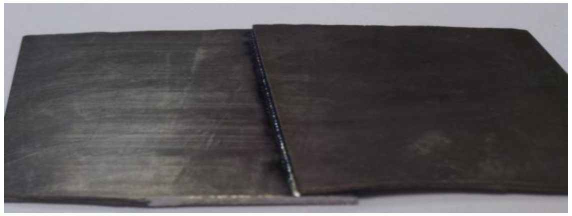
1. Argon arc welding of non-consumable tungsten electrode with the location details of the butt, without the use of additives, with double-sided weld penetration.



2. Laser welding arrangement butt parts without using additives with bilateral weld penetration.



3. Laser welding with overlapping, without the use of additives with a bilateral joint penetration.



Mechanical properties and U/Th of the welds

Mechanical properties show that the laser and argon welding allow to use UHP Ti as a structural material in the manufacture of a cryostat. The strength of the welded joint was about 400MPa. An advantage of laser welding is a small heat-affected zone.

Samples	Max strength MPa
Laser welding (butt)	450
Laser welding (overlap)	355
Argon arc welding (butt)	390
Original material	590

Ti 1-X - argon arc welding;
Ti 2-X - laser welding.

Task		Ti 1-1			Ti 1-2	Ti 2-1	Ti 2-2
ppb		ppb	St dev ppb	St dev %	ppb	ppb	ppb
<0.25	Th	<0.02			<0.02	**<0.01**	**<0.01**
<0.10	U	0.69	0.04	6.4	<0.03	**<0.01**	**<0.01**

Conclusion: The **laser welding keeps U/Th level low**. Contamination with argon arc welding by U requires further study and may be associated with a contamination by the tungsten electrode.

Low radioactivity of the original material was additionally confirmed.

Laboratory stage conclusions:

1. **Confirmed purity of samples is equal to < 0.1 mBq/kg**

   (better than the 1 mBq/kg Target Values)

2. Bulk radiopurity confirmation was not completed

	U, ppb	Th, ppb
Sample №3	**<0.003**	**< 0.05**

Ti sponge production

3. Preselection and analysis of initial raw material for titanium sponge production is mandatory.

4. **Magnesium purity and cleanliness of the vacuum separation process are two key points of ULR Ti sponge production.**

Ti sheets, rods production

5. All adding and scrap should be avoided at the stage of metal titanium production.

So, **VT1-00 / GRADE 1** only could be used

6. Arc and e-beam ingot melting as acceptable.

7. Cold forging should be used instead of hot forgingz

8. Recontamination during forging and rolling could be removed by etching. Core material stays pure.

Ti vessel manufacturing

9. **Laser welding should be used** for vessel manufacturing

Russia-Italy R&D grant :

The main goal was to develop and test industrial scale technology of ULR titanium production

Ministero degli Affari Esteri
e della Cooperazione Internazionale

1st CALL FOR PROPOSALS
OF JOINT RESEARCH PROJECTS WITHIN THE JOINT PROGRAM FOR SCIENCE AND TECHNOLOGY COOPERATION AND IMPLEMENTATION OF JOINT CALL FOR RUSSIAN-ITALIAN PROJECTS FOR 2021-2023

quality confirmation (+-)

3. To develop and test methods how to prevent the secondary recontamination during welding and transportation (+-)
4. To test these methods and procedures on the samples produced by the industrial methods (+-)
5. To develop semi industrial scale apparatus for ULR Ti sponge production (++)

   Apparatus was constructed in cooperation with LLC «MODERN TECHNOLOGIES» (Solikams)

6. To supervise the manufacturing of 20 t of ULR titanium for DS-20k inner vessel at PJSC VSMPO-AVISMA (--)

Russia-Italy R&D grant :

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Industrial scale of any High Tech projects needs :

- Customer who is ready to place an order and pay (INFN – DS-20k)
- Producer who is ready to produce the goods (JSC VSMPO-AVISMA)

A joint Russian-Italian grant was used to complete the semi-industrial stage of the project and launch the industrial stage of the project, which includes the following stages :

1. To develop methods and procedures of precise measurements of impurities in titanium and titanium production –related products for industrial use (++)
2. To develop methods and procedures of direct radioactive impurities detection (HP-Ge and surface alpha counting) in different titanium product during the titanium parts production cycle for final quality confirmation (+-)
3. To develop and test methods how to prevent the secondary recontamination during welding and transportation (+-)
4. To test these methods and procedures on the samples produced by the industrial methods (+-)
5. To develop semi industrial scale apparatus for ULR Ti sponge production (++)

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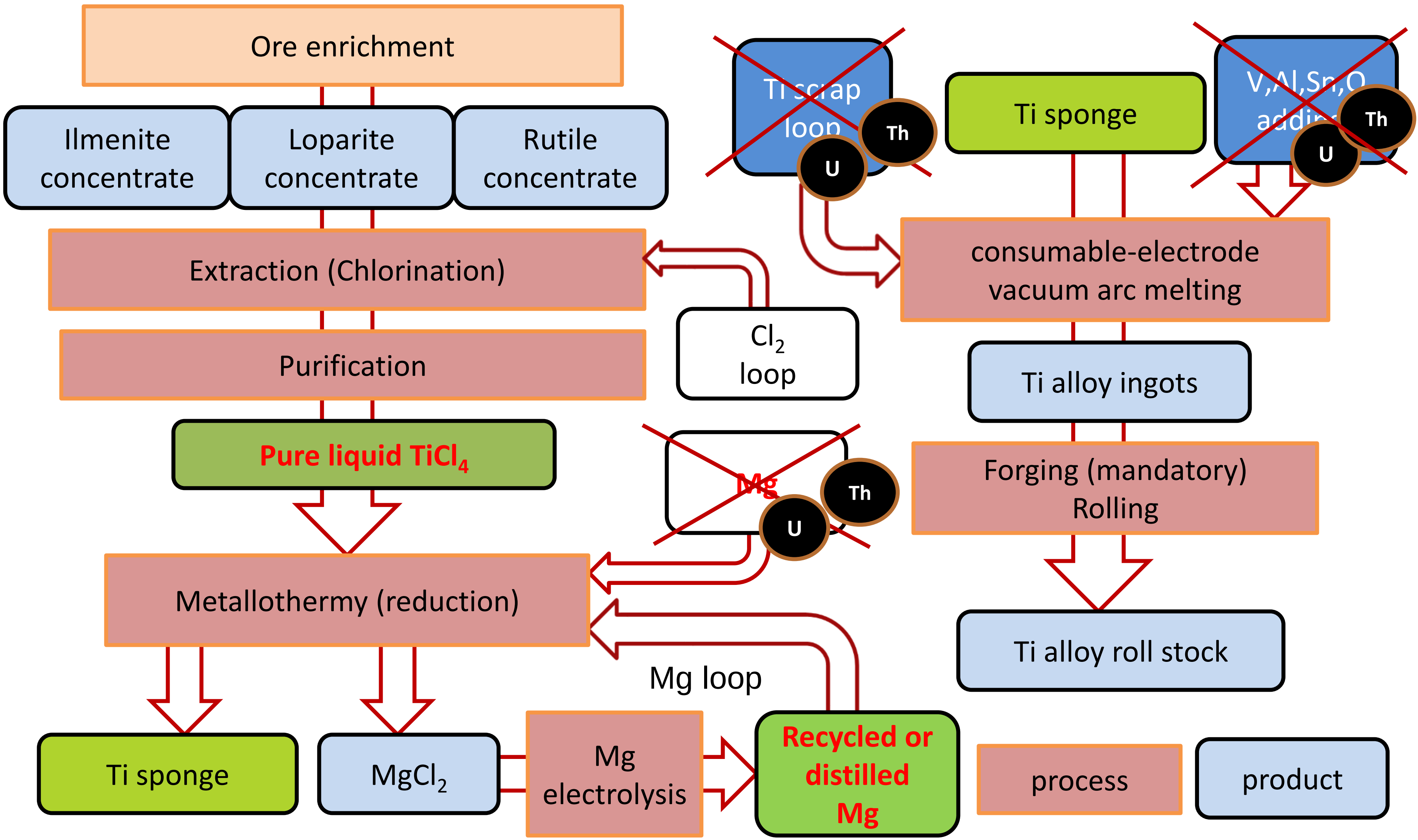
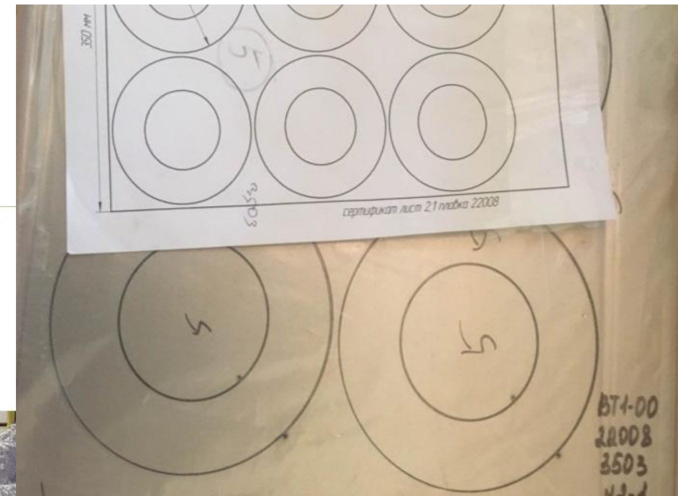
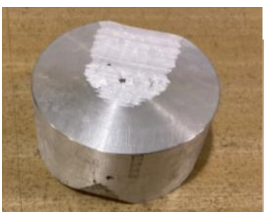
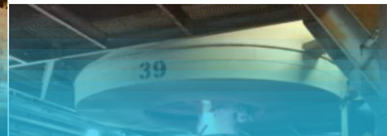
MODIFIED industrial titanium production cycle "Kroll-process" in cooperation with VSMPO-AVISMA



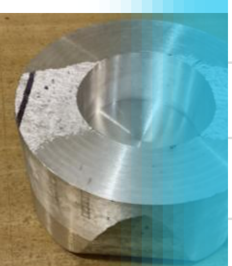




~ 200 kg piece of ULR Ti sponge was "cooked" using standard equipment by nonstandard way

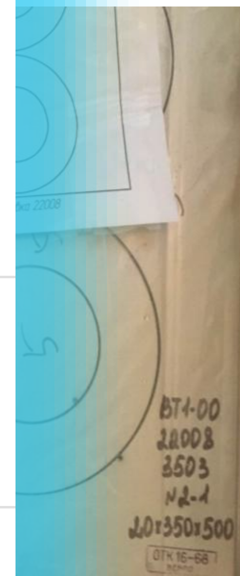



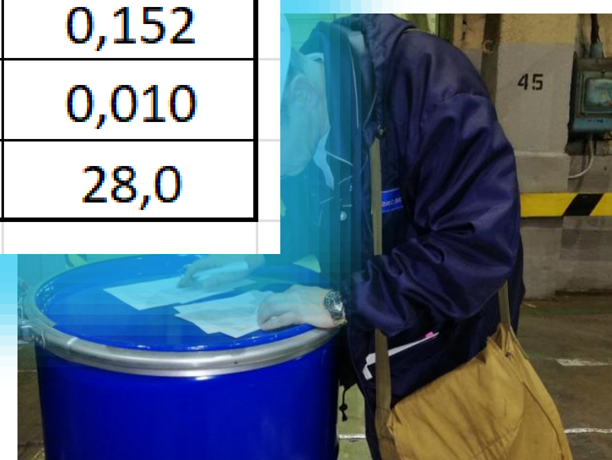


2023 result !

	Ti ingot sheet sample 22008			
		standard deviation	relative standard deviation	Definition limit
	ppb	ppb	%	ppb
Th	<0.152			0,152
U	0,066	0,01	17	0,010
K	<28.0			28,0





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What purity level we need and how to prove it

2012
[fundamental] - 238U < 1 mBq/kg (equivalent to < 0,100 ppb)
[fundamental] - 232Th < 1 mBq/kg (equivalent to < 0,250 ppb)
[less important] - 40K < 1 mBq/kg (equivalent to < 40 ppb) can't be measured by ICP-MS

2017
New target to reach mkBq/kg levels of U/Th was defined at the end of 2017

2021
What target level do we expect to achieve?
The realistic target seems to be ~ < 10-30 mkBq (~ 20 ppt)

ICP-MS and HP-Ge and surface α- screening
The three methods should be used in parallel and at least in two locations according to synchronized methods.

Conclusions

- Lab-scale phase completed and samples demonstrated
- Preliminary industrial scale phase completed. The results are under analysis mainly because of HPGe data analysis delay

Next steps

- To complete the “Preliminary industrial scale phase” and synchronize screening procedures between all interested institutions
- To combine the efforts of all interested institutions (experiments) to complete the process by ordering an producing the industrial batch of titanium parts and mill products

Thank you for your attention !

Titanium monument above
Moscow museum of space research
http://москва-россия.рф/muzey-kosmonavtiki-v-moskve/

Source of ULR Mg is critical for ULR Ti

ULR Mg study program was started in 2018 and it was discovered that all industrial magnesium may have to high level of U/Th due to some features of the industrial processes

so

Magnesium **SHOULD BE REFINED** for Ti-sponge production process

How to do it less expensive way ?

- Mg purification through "Mg loop"
- Preliminary distillation of Mg in special apparatus