HDES

High Density Energy Systems



### Final review

CGG for EP De-Risk project 6 September 2024

### **HDES** Agenda of the Final review

High Density Energy Systems

#### •10.00 Opening

- •10:15 Presentation of the project results of the third and last phase
  - •Introduction of the battleship development phase
  - • Design of the improved test set-up
  - • Experimental results and analysis
  - •System study results
  - •Questions / discussion
- •11:00 10 minute Break
- •11:10 Presentation of the follow on activities for the battleship phase
  - Development Plan
  - Technical Achievement Summary
  - Questions / discussion
- •11.30 Presentation to the Electric Propulsion Section of ESTEC
- •12.00 Lunch
- •13.00 Comments on the deliverables / technical data Packages
- •13:20 Contract Close out activities
- •13:40 Actions / Modifications of the documents
- •13:50 Any Other Business / Minutes of Meeting
- •14:00 Closing





- Goal: to develop a xenon-producing cool gas generator
- WP 300 objectives:
  - To test the grain in a battleship CGG test setup
- The battleship was an improved version of the setup issued in the grain development phase.



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- In the grain development phase, feasibility of the Xenon CGG was proven, however no independent working was realised
  - External heating
  - Hot nitrogen flow help the flow inside the test set-up
- The goal of the battleship phase was to improve the set-up used in the grain development phase.
- Main improvement was reduction in thermal inertia
- Main goals for the grain development phase:
  - Achieve independent xenon production
  - Test filters
  - Improve reaction chamber
- The first goal was achieved, the other two only partly





### Improvements of the test set-up

- From a number of ideas for improvements, the following were selected
- 1) Remove the valves, as the reaction is slow there is minimal risk in removing the valves.
- 2) Reduce the mass and size of the setup to a large degree
- 3) Improve the use resistance wire configuration (more and better placed) and mount them up to the reaction chamber
- Before the improvement were implemented, a thermal model was set-up to chain their effectiveness

# HDES Preliminary design for improved set-up

- High Density Energy Systems
- From a number of ideas for improvements, the following were selected
- 1) Remove the valves, as the reaction is slow there is minimal risk in removing the valves.
- 2) Reduce the mass and size of the setup to a large degree
- Improve the use resistance wire configuration (more and better placed) and mount them up to the reaction chamber
- Before the improvement were implemented, a thermal model was set-up to chain their effectiveness





## (simplified) thermal model

- Considers main parts as nodes with thermal properties
- Used to compute expected heating energies and times
- Confirmed the modifications of the set-up

simplified therma	l model								Specific he	at	heating the se	t-up from 1
item	material	inner diameter (mm	outer diameter (mm)	length (mm)	volume (CC)	density (gr/cc)	mass		J/gr/K		T (degrees C)	E (J)
Tube	Teflon	7	8	150	1,767145868	2,2	3,887721		1,5		40	145,7895
XeF2						4,3	2	given	0,44		40	22
Teflon plug	Teflon	7	7,5	10	0,3848451	2,2	0,846659	left end	1,5		40	31,74972
Teflon plug	Teflon	7	7,5	10	0,3848451	2,2	0,846659	right end	1,5		15	0
Resistance wire	Ni-Chr steel	1	1	TBD		10,22	2				NA	
Reaction materia	l Molybdenum				0,2-0,8		2	25% porosity	4,18		NA	
Collection bag	PTFE	50	50,2	50	6,308351559	2,2	13,87837		1,5		NA	
						total mass	25,45941					199,5393
								sublimation en	ergy of XeF2	304	J/gr	608



- TNO modified the test set-up on the basis of the design of HDES.
- HDES has produced a test plan based on the test set-up design. This plan is input for the TNO detailed experimental procedures
- Plan was to first test the improvements
- Then test different configurations of reaction chamber and filters



### **Battleship setup**

- The sublimation chamber and the reaction chamber have been integrated into a single chamber made of a short PFTE tube.
- A resistance wire heats the complete tube, so both the sublimation and reaction chamber sections are heated.
- The gas collection bag is situated directly after the reaction chamber, thereby reducing all unnecessary thermal mass.



**TNO** innovation for life

#### **Molybdenum reactor test:**

- Heating wire:
  - Ø: 0.25 mm, L: 37.0 cm, Power: 5.5 Volt & 0.5 Amp → T: 80 °C
- Reactor: Molybdenum
  - Ø: 0.20 mm, spiral form
- XeF<sub>2</sub>:
  - 0.5 g

#### **Observations:**

- Slow sublimation of XeF<sub>2</sub>
- Ripening of XeF<sub>2</sub> between the windings of the heater
- In reactor yellowish liquid formed
- No inflation of gas bag





#### **Conclusions Molybdenum reactor test:**

- Reaction takes place between XeF<sub>2</sub> and molybdenum
- No detectable formation of xenon observed

- Insufficient XeF<sub>2</sub> decomposed or
- Another Xe containing solid is formed
  - XeF<sub>4</sub>
- $[C_6F_5]_2Xe, C_6F_5-Xe-C\equiv N, C_6F_5-Xe-F$

#### Lit. THE CHEMISTRY OF XENON', J. Malm, H. Selig, J. Jortner. S. Rice, Sept. 1964:

'XeF<sub>2</sub> has been prepared by a variety of methods, nearly all of which depend on the rapid removal of XeF<sub>2</sub> from the reaction system to prevent its further reaction resulting in the production of  $XeF_4$ '

F comes from XeF2 then: $2 XeF2 \rightarrow 2 Xe + 2 F2 \rightarrow Xe (gas) + XeF4 (solid)$ : Gaseous Xe formed however 50% yield $12 C + 5 XeF2 \rightarrow Xe + [C_6F_5]2Xe (solid) + 4 Xe (gas)$ : Gaseous Xe formed however 80% yield

To form 'possible' Xe containing molecules always Xe is released from XeF<sub>2</sub> the Xe yield however is reduced

No detectable formation of xenon observed:

most likely insufficient XeF<sub>2</sub> is decomposed to noticeably inflate the gas collection bag



# Molybdenum improved reactor test:

- Heating wire:
  - Ø: 0.25 mm, L: 95.0 cm, Power: 9.5 Volt & 0.4 Amp → T: 110 °C
- Reactor: Molybdenum
  - Ø: 0.20 mm, folded many times, hold together by Teflon tape
- XeF<sub>2</sub>:
  - 0.5 g

#### **Observations:**

- Sublimation of XeF<sub>2</sub>
- Tape of reactor turned black with some blueish spots
- In reactor yellowish liquid formed at the stream upward side
- No inflation of gas bag







#### **Conclusions Molybdenum reactor test:**

- Reaction takes place between XeF<sub>2</sub> and molybdenum
- No detectable formation of xenon observed

- Insufficient XeF<sub>2</sub> decomposed or
- Another Xe containing solid is formed
  - XeF<sub>4</sub>
- $[C_6F_5]_2Xe, C_6F_5-Xe-C\equiv N, C_6F_5-Xe-F$

To form 'possible' Xe containing molecules always Xe is released from XeF<sub>2</sub> the Xe yield however is reduced

No detectable formation of xenon observed however:

• yellowish liquid at the upstream side reactor, most likely  $MoF_5 \rightarrow XeF_2$  is decomposed, where is the Xe?

Mo fluorides	Physical form	mp (°C)	bp (°C)	Density (g/cm <sup>3</sup> )	Vapor pressure (1kPa @ °C)
Mo	Gray cubic	2623	4639	10.2	
MoF3	Brown hexagonal crystal	>600		4.64	
MoF4	Green crystal	D			
MoFs	Yellow monoclinic crystal	67	213	3.5	86.6
MoFs	White cubic crystal Colorless liquid at RT	17.5	34	2.54	41.2
MoOF4	Volatile solid	98			

mp: melting point, bp: boiling point, RT: room temperature, D: decomposes 1Pa = 7.5 mTorr



#### **Hypothesises Molybdenum reactor test:**

Most likely insufficient XeF<sub>2</sub> is decomposed to noticeably inflate the gas collection bag why?

- Test with syringe shows that from 1 ml gas insertion, the gas collection bag starts to show signs of inflation.
  From this it is calculated that less than 7.6 mg (<1.5% of the XeF<sub>2</sub>) generated Xe why?
  - Molybdenum is oxidized at the surface area of the wire and for this not effective as reactor?:
    - Pro: Free metallic molybdenum does not occur naturally on Earth (Wikipedia) it is found only in oxidized states
    - Con: Oxidation of Molybdenum starts at 300 °C (Wikipedia)
  - The surface area of the reactor is too small or the residence time in the reactor is too short?
    - Con: no inflation of the bag, if the residence time was too short, the velocity at which XeF<sub>2</sub> travels through the reactor would have been high and unreacted XeF<sub>2</sub> should ripen after the experiment down stream of the reactor → this is not observed



#### **Hypothesises Molybdenum reactor test:**

- Within the reactor XeF<sub>2</sub> ripens and is blocking the flow through the reactor
  - Pro: only a reaction is observed upstream of the reactor (formation yellow liquid) no reaction products or XeF<sub>2</sub> observed down stream of the reactor
  - Con: no XeF2 was found in the catalyst, but that is due to the passivation procedure before the setup could be taken apart
  - Con: the Teflon tape is changed from white to black over the total length of the test and coloration started upstream and traveled downstream in time (something flowing?)

May be possible, the heat added to the reactor comes from the outside and has to be transferred to the core of the reactor, the contact surface between the wires is small. Significant heat is required to do heat the reactor down to its core. It is believed that only a very small part of the reactor is warm and capable decomposing XeF<sub>2</sub>, the core of the reactor is cold and gaseous XeF<sub>2</sub> ripens between the Mo wires, and may there react very slowly.

Note: no conclusive proof for any of the hypothesis



### **Activated charcoal reactor**

- Fairly similar to the molybdenum setup, the only difference is that the activated charcoal version has a bit longer reaction chamber .
- The number of windings of the heating wire on the reaction chamber were increased to prevent the xenon difluoride from ripening between the windings of the heating wire
- To boost the sublimation reaction by increasing the temperature of the heating wire to 140°C.





#### **Activated charcoal reactor test A:**

- Heating wire:
  - Ø: 0.25 mm, L: 74.0 cm, Power: 8.0 Volt & 0.4 Amp → T: 140 °C
- Reactor: activated charcoal
  - 0.56 g
  - thermo scientific Carbon, activated, -4+8 mesh, cat.no 043118.36 lot:Y13J028
  - XeF<sub>2</sub>:
    - 0.50 g

#### **Observations:**

- Sublimation of XeF<sub>2</sub> more quickly than previously
- Ripening of XeF<sub>2</sub> at the fixation clamp
- No inflation of gas bag



Ripening of XeF<sub>2</sub> at the clamp





#### **Activated charcoal reactor test B:**

- Heating wire:
  - Ø: 0.25 mm, L: 115.0 cm, Power: 14.5 Volt & 0.5 Amp
    → T: 140 °C
- Reactor: activated charcoal
  - 0.56 g
  - thermo scientific Carbon, activated, -4+8 mesh, cat.no 043118.36 lot:Y13J028
  - XeF<sub>2</sub>:
    - 0.50 g

#### **Observations:**

- Sublimation of XeF<sub>2</sub>, some ripening of XeF<sub>2</sub> at the steering bean
- No inflation of gas bag
- Mass reactor increased to 0.61 g
- White deposits on the activated charcoal





#### **Decomposition of XeF<sub>2</sub> in the activated charcoal reactor:**

• sample of the reactor was taken for analyses means of FTIR (Fourier-transform infrared spectroscopy



- Because the concentration of the white residual is low, only small deviation in the spectra between before and after reaction is visible.
- Despite this, the deviation between both spectra's starts exactly at the location of the PTFE absorption peak

White residual is most likely PTFE, and is proof that decomposition of XeF<sub>2</sub> has taken place

#### **Conclusions activated charcoal reactor test:**

- Reaction takes place between XeF<sub>2</sub> and charcoal
- No detectable formation of xenon observed

- Insufficient XeF<sub>2</sub> decomposed or
- Another Xe containing solid is formed or
- Xe is adsorbed by the activated charcoal

Lit. Adsorbed xenon propellant storage: are nanoporous materials worth the weight? M. Huynh et all: Activated Carbon is studied for Xe storage due to its ability to adsorb Xe

 $\rightarrow$  Most likely, in the gas generator the gaseous Xe is trapped in the reactor

The activated charcoal adsorbs the xenon that is being produced and for this, it is not a viable material for a xenon gas generator reactor.





### System study results

- In the system study the test results were used for a system design
  - Based on experience of the last
  - Realistic and conservative expectations
- Comparison with existing systems
- Analysis of the business case (market size and economics)
- Main results are:
- There are big advantages in mass and volume for xenon system with up to 5 kg Xe storage







- Specifications:
- Xenon storage: 300 grams
- Mass: 0,7 kg
- Volume: 1 U
- Power: < 5 W
- Flow is controllable
- No filling before launch
- No mech parts





Xenon tank volume vs xenon CGG volume

- Advantage in mass is up to 20% (realistic case)
- Advantage in volume is up to 60% (realistic case)
- Advantage in mass is up to 10% (conservative case)
- Advantage in volume is up to 30% (conservative case)
- Up to 5 kg of xenon storage

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IFZ Business case xenon CGG system

- Market size is estimated to be between 10 and 30 systems/ yr
- A storage system may cost between 5 and 25 **k**Euro
- This price is achievable, but XeF<sub>2</sub> will be 25% of the price
- This assumes production of larger quantities at a lower price
- Total expect turn-over between 400 and 800 kEuro/yr











# **HDES** Take-aways from the de-risk project

- The project went very well
- With a lot of flexibility, a working concept has been established
- Feasibility of a Xenon CGG has been shown
- System study confirmed the expected advantages
- However.....
- Detailed interaction between the physical processes in a Xenon CGG are still poorly understood
- No design optimisation
- Xenon purity has not yet been proven



- On the basis of the successful De-risk a Development Plan has been made
- The plan addresses the problems identified during the de-risk
  - Better characterisation of physical processes and interaction
  - Design optimisation
  - Filtering
- This included budget estimation and planning.
- It uses the end point of the de-risk as the start
- Three phases: Demonstration Model, EM and QM

# **HDES** Proposed development logic





- Demonstration Model phase is needed to perform an extended characterisation
  - Physical processes
  - Chemical processes
  - Thermal processes
- And their interaction
  - With a flexible and modular set-up
  - With up to 100-300 grams of XeF<sub>2</sub>
  - Extensive tests with many variations
- Identification of a first flight opportunity / customer / mission
- EM and QM can be typical for ESA



- Three phases DM, EM and QM
- 2,5 to 3 year development until flight
- Estimated costs
  - DM phase: 445,5 kEuro (to TRL 4/5)
  - EM phase: 178,3 kEuro (to TRL 6)
  - QM phase: 267,3 kEuro (to TRL 7-8)
- Total: 891,1 kEuro
- Programme could start early 2025



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# **DES** Technical achievement summary

- The feasibility of a xenon CGG has been established
- Independent working has been shown
- A large body of knowledge on XeF<sub>2</sub> has been gathered
- XeF<sub>2</sub> is much more benign than expected and good to work with
- System study confirmed the potential gains in mass and volume
- A development plan has been made up to applications
- Benefits
  - Up to 20 % reduction in mass, up to 40% in volume
  - No high pressure, limited mechanical parts
  - Can be filled and prepared in Europe, not pressurised during launch
  - No extensive temperature and pressure control needed





#	Requirement	Verification Method	Status
1	The compound has to be chemically stable (limited or no degradation over time)	Test	Verified by experiment
2	The compound has to be compatible with other ingredients of the CGG grain	Test	Verified by experiment
3	The xenon yield of the CGG grain composition has to be at least 40% (so 40 % of the grain mass needs to exit as xenon)	Test	Verified by analysis,
4	The CGG grain has to be mechanically stable (before and after firing), that is it should stay in one piece during removal from the mould and handling	Test	Verified by experiment
5	The overall volumetric yield has to exceed 80 liters of xenon per liter of gas generator	Analysis	Verified 85 nl/l estimated
6	The overall mass yield has to exceed 80 liters of xenon per kg of gas generator	Analysis	Not yet met: 72 nl/kg estimated
7	The xenon purity should be > 99%	Test	Not yet measured



### Presentation to ESA Electric propulsion Group

• Separate presentation (CEP-HDS-PT-05)







### **DES** Comments on the deliverables

**High Density Energy Systems** 

- Comments of ESA
- Comments of the team
- RIDs
- Actions



#### **Deliverables**

[di-'li-v(ə-)rə-bəlz]

A project management term describing the quantifiable goods or services that must be provided upon the completion of a project.

Investopedia



- What needs to be done before contract close-out?
- MoM of this meeting
- Invoice
- How to jointly working on a continuation?
- Any last points?

