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Ministero degli Affari Esteri
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INFN
Istituto Nazionale di Fisica Nucleare

中华人民共和国科学技术部
Ministry of Science and Technology of the People's Republic of China

ALLIFLU: a new material for neutron beams tailoring
A new sintering process and device

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Rome – 7 December 2018

NEU_BEAT Workshop

INFN Headquarters

Istituto Nazionale di Fisica Nucleare

BEAT_PRO
(CNS5 – 30 kEuro

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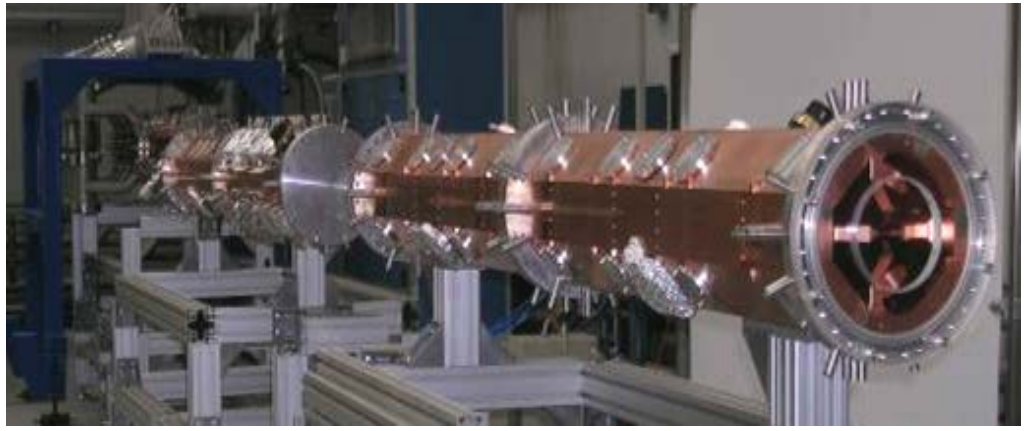
NEU_BEAT
(150 kEuro)

Beam Shaping Assembly



TARGET

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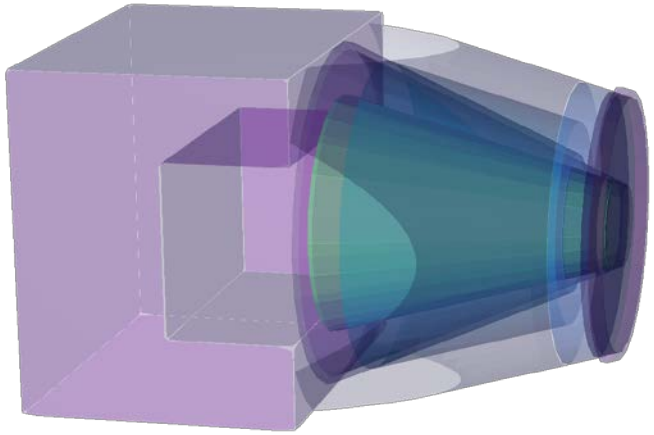
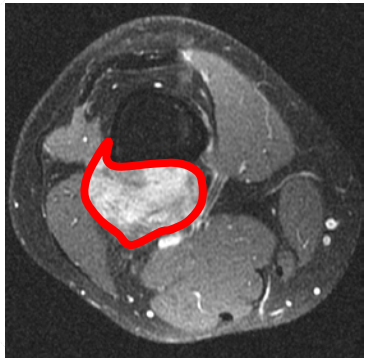
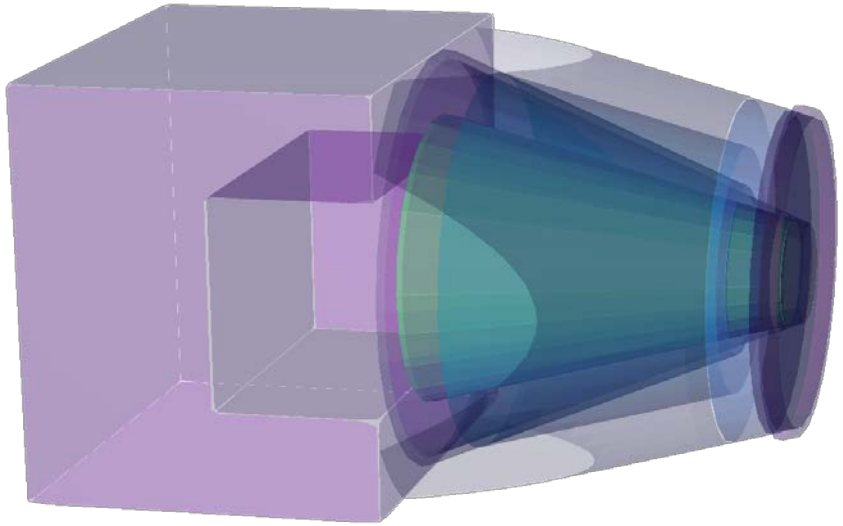


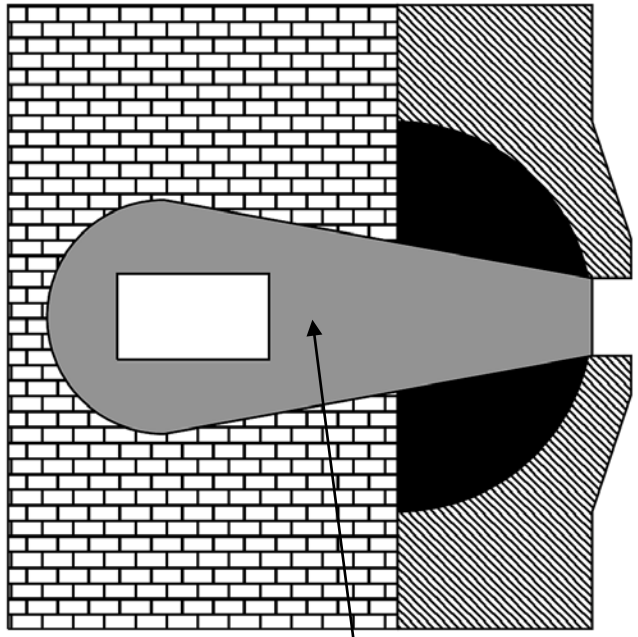
PATIENT

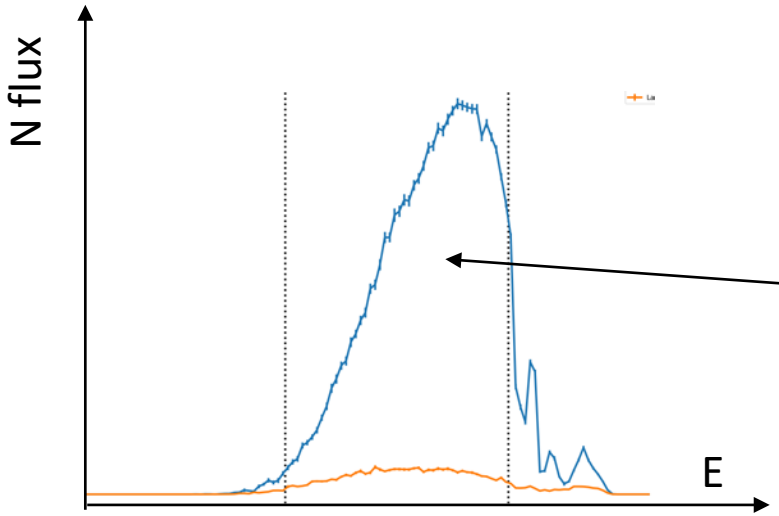


Proton accelerator 5 MeV, 30 mA

BSA Design: test of geometries and materials to obtain a beam suitable for clinical application (deep-seated tumours)

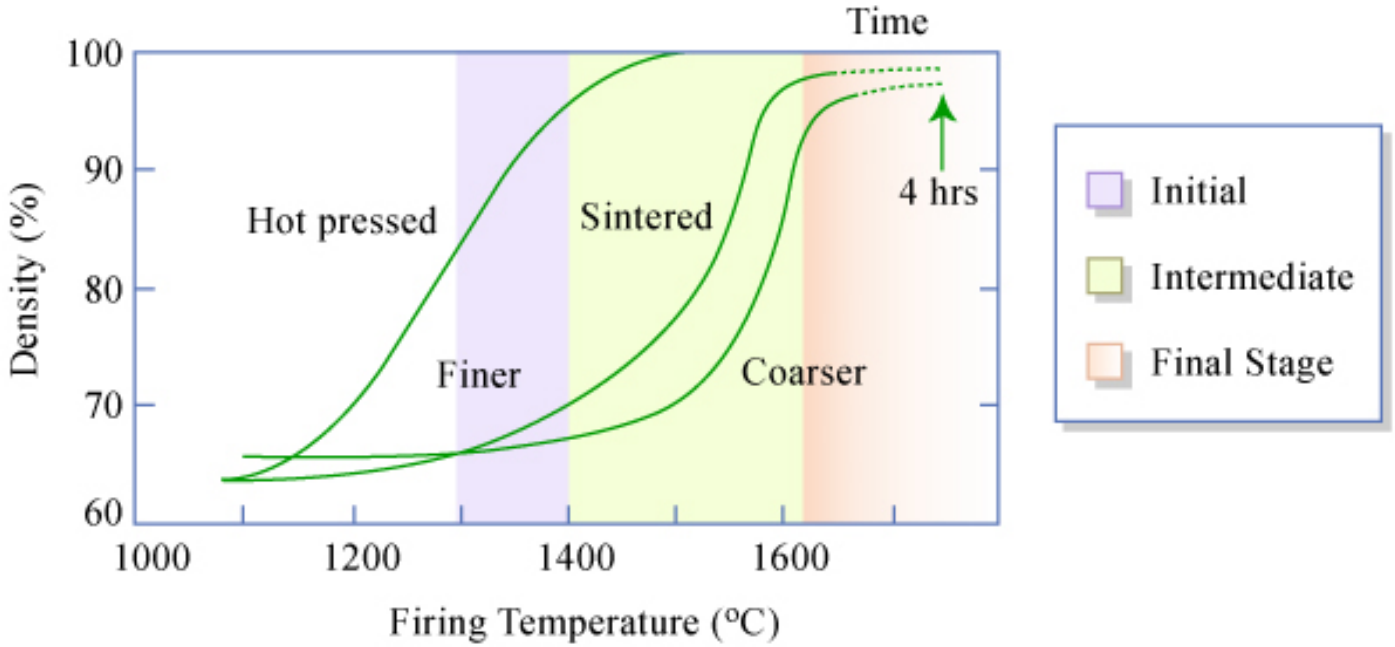


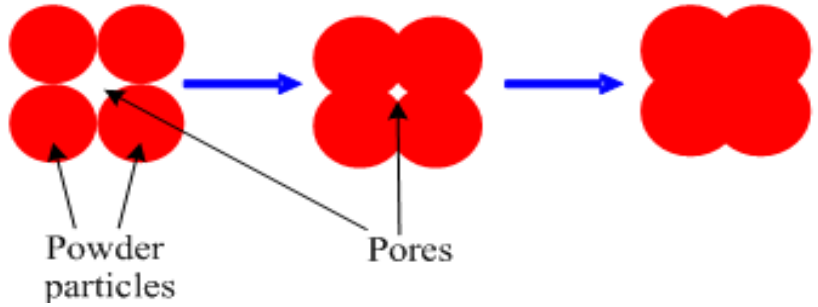




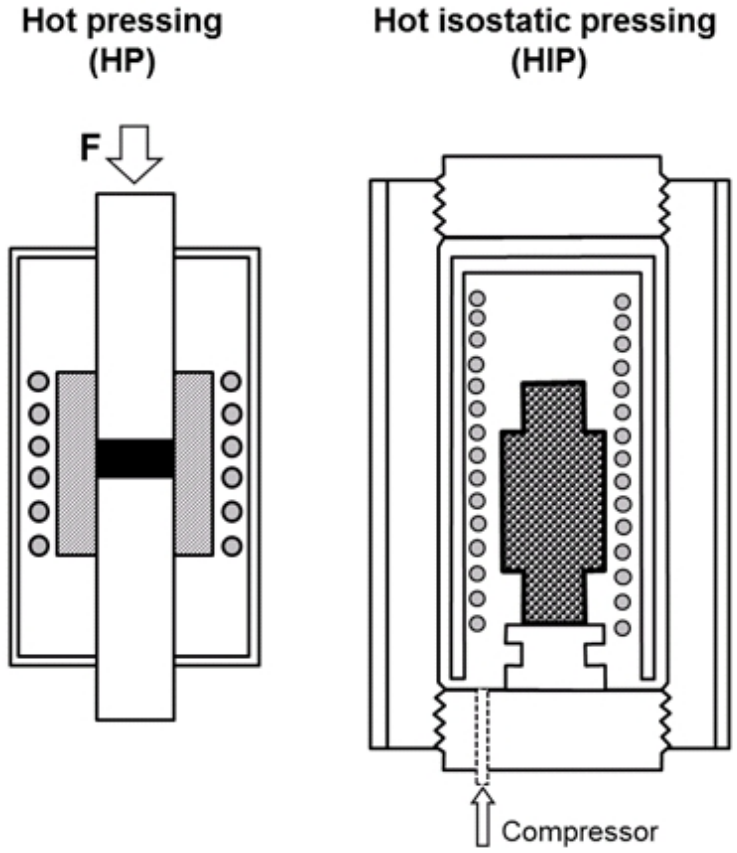
To obtain a neutron spectrum peaked between 1 and 10 keV, with low contamination, the most performing material was proven to be **aluminium fluoride with small % of Li.**

- Sintering of AlF3 powders is difficult due to its high temperature volatility and because Fluorine is the most electro-negative element
- Sintering requires mass transfer through diffusion and generally involves long high temperature annealing





More efficient residual porosity removal can be obtained adding uniaxial or isostatic pressing.



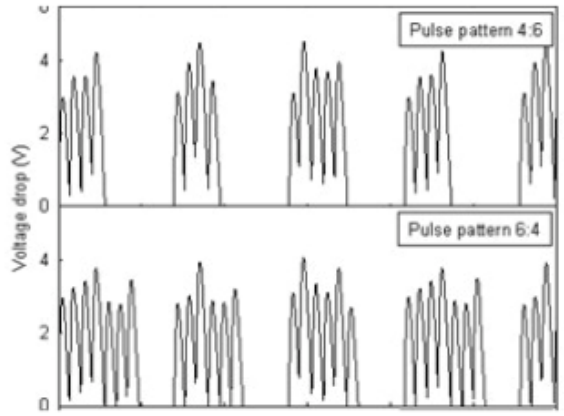
These methods, however, involve the use of large, expensive machines with very long heating and cooling cycles

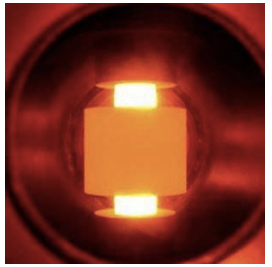
Field Assisted Sintering (FAST) / Spark Plasma Sintering (FAST/SPS)

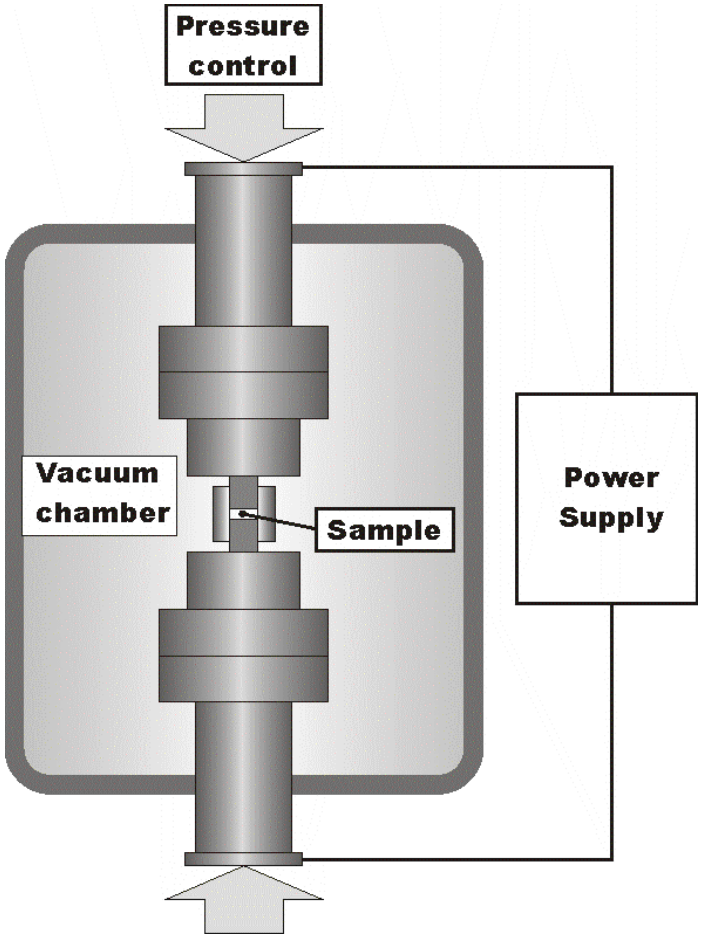
It has recently become the technique of choice for the hard to sinter materials

- High heating rates (up to 1000°C/min)
- Very short sintering times (minutes instead of hours)
- Low sintering temperatures

Typical experimental conditions:
Voltage 5-10 V
Current intensity 10^3-10^4 A
Pressure 5-100 MPa

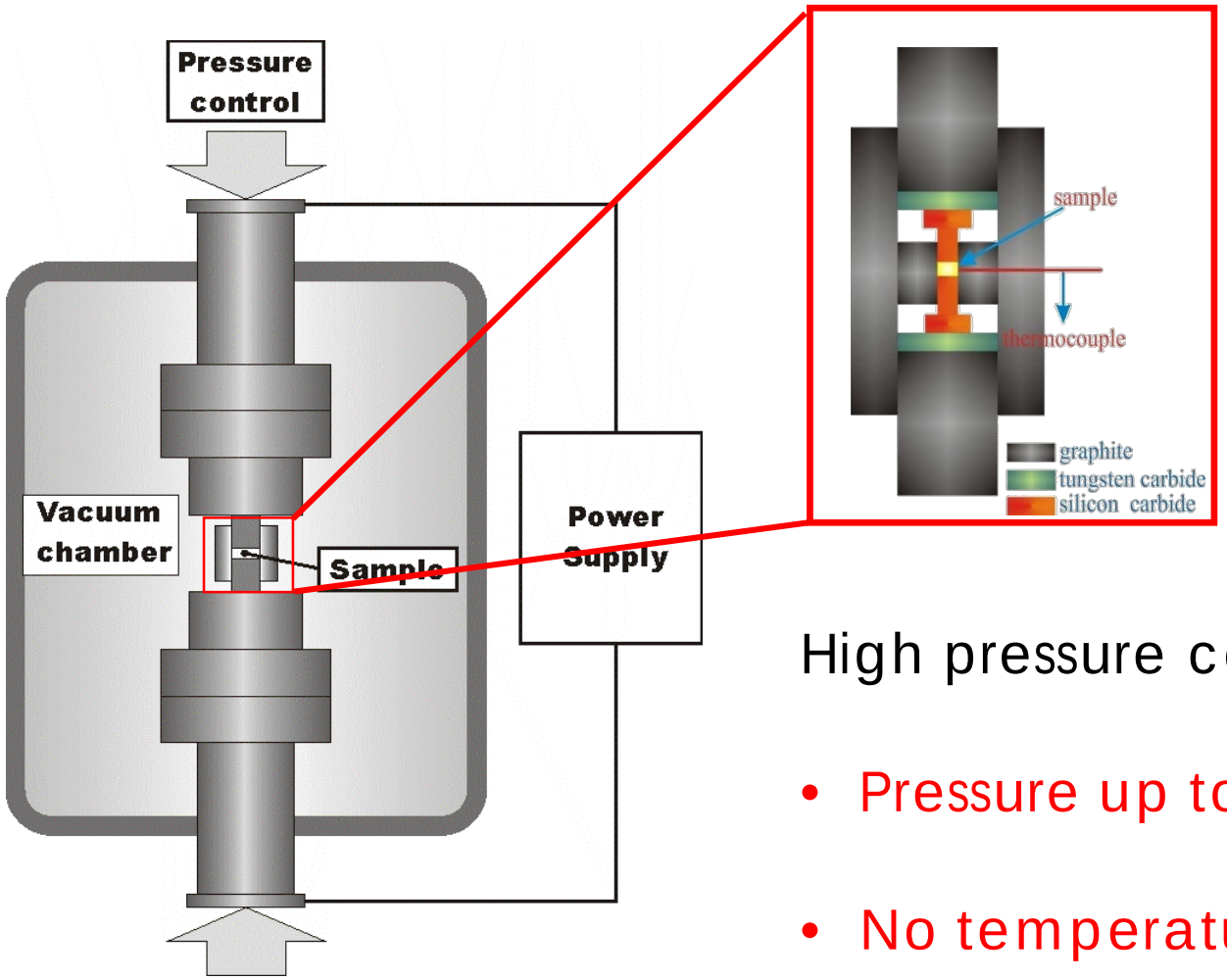






However, in most cases, costly power supply of pulsed DC current is used

High-pressure FAST/SPS (HP-FAST/SPS)

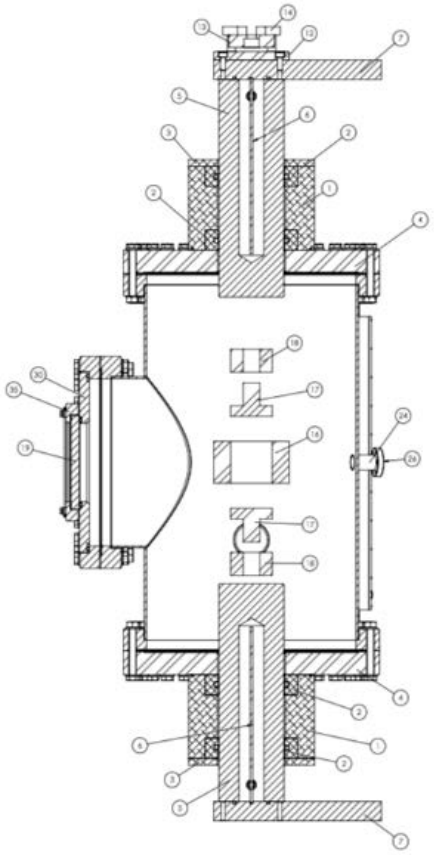


High pressure components realized in *SiC*

- Pressure up to 1 GPa
- No temperature limitation

Design and construction of a sintering machine prototype @INFN workshop Pavia in collaboration with University of Pavia

Collaboration with SIRAS and TECNEL Pavia Companies



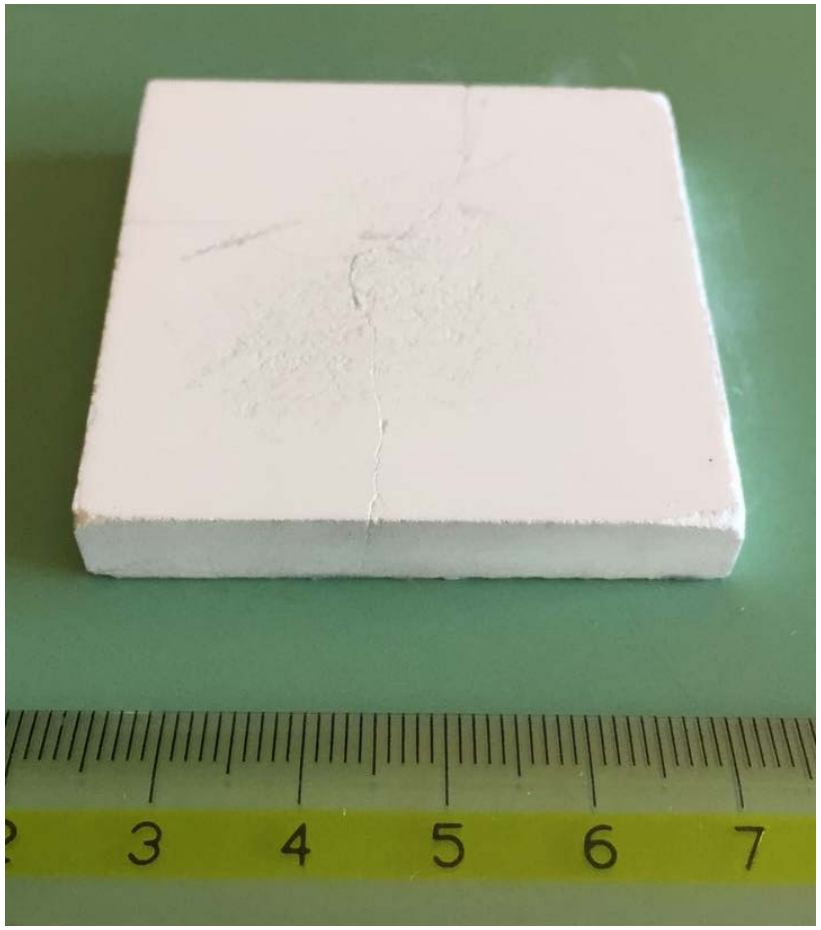
Powder

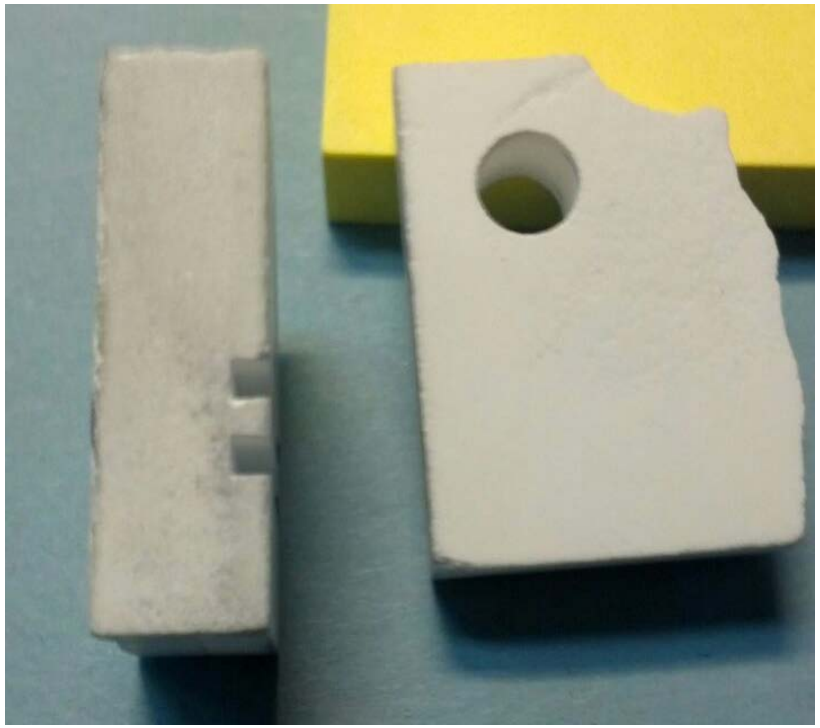
Solid AlF_3 + LiF

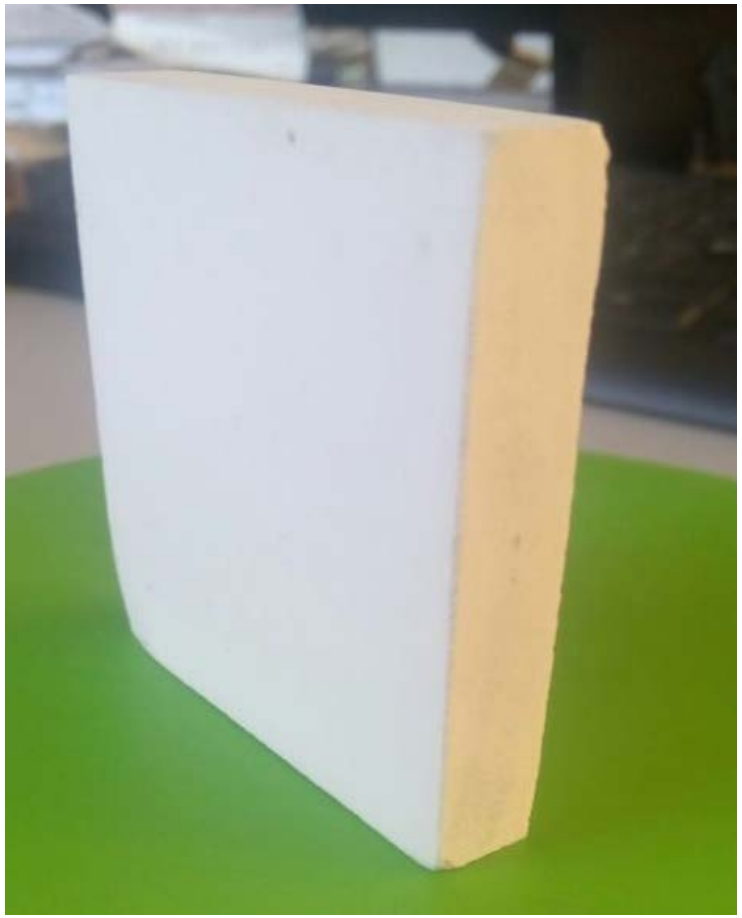
ALLIFLU

ALLIFLU

Good mechanical properties
High density (~ 100%)
Resistance
Can be machined







Patent application deposited by INFN TT office

Ministero dello Sviluppo Economico

Ricevuta di presentazione

per

Brevetto per invenzione industriale

Domanda numero: 102018000005315

Data di presentazione: 14/05/2018

CLAIMS

1. A method for manufacturing a sintered compact of lithiated aluminum fluoride, the method comprising:

providing a mixture of powders of aluminum fluoride and of a compound containing fluorine and lithium,

loading the mixture in a mold (10),

heating the mixture by means of application of electrical current through the mold (10) and applying a uniaxial compression to the mixture to obtain a sintered compact of lithiated aluminum fluoride,

wherein heating the mixture comprises

- increasing the temperature of the mixture from an ambient temperature to a maximum sintering temperature, with a heating ramp equal to or greater than 10°C/min, and

- holding the mixture at the sintering temperature for a holding time comprised between 5 and 15 min.

2. A method according to claim 1, wherein the heating ramp is equal to or greater than 20°C/min.

3. A method according to claim 1 or 2, wherein the powders of aluminum fluoride have a purity comprised between 90% and 99,99% m/m.

4. A method according to any of the preceding

claims, wherein the mold (10) comprises a central body (11) and two opposing dies (13) capable of sliding in a through bore (11a) formed in the central body (11), the central body (11) of the mold (10) cooperating with the dies (13) to define a closed cavity adapted to be loaded with the mixture, wherein the central body (11) and the dies (13) are of a material capable of being heated to the maximum sintering temperature by Joule effect, and wherein the uniaxial compression is applied to the mixture through the dies (13).

5. A method according to claim 4, wherein the electrical current is conducted through the mold (10) by entering to the mold (10) through one of the dies (13) and exiting from the mold (10) through the other of the dies (13).

6. A method according to any of the preceding claims, wherein the maximum sintering temperature is comprised between 630°C and 650°C.

7. A method according to any of the preceding claims, wherein the sintered compact of lithiated aluminum fluoride is free of metallic aluminum.

8. A neutron moderator comprising a sintered compact of lithiated aluminum fluoride, manufactured with a method according to any of the preceding claim.

How can this be useful for other needs?

This technology, born to solve a problem in BNCT, can be transferred to other applications, after proper engineering

FastSinter

R4I project (funded by INFN-TT) and Proof Of Concept (submitted to Vertis) to make a new machine based on the prototype with the following improvements:

- Compact the design
- Re-think the vacuum chamber and the cooling
- Certify
- Exploring the market

- Commercial sintering machines are generally designed for large industrial applications
- Use DC or pulsed DC current
- Typically priced between 250-500 k€

Thermal technology LLC

FCT Systeme GmbH

Fuji-SPS

Re-design and assembly of a sintering machine whose characteristics are:

- Simplifed layout
- Use of AC power supply
- Smart design of molds
- Off-the-shelf hydraulic and power components and controllers

These features ensure:

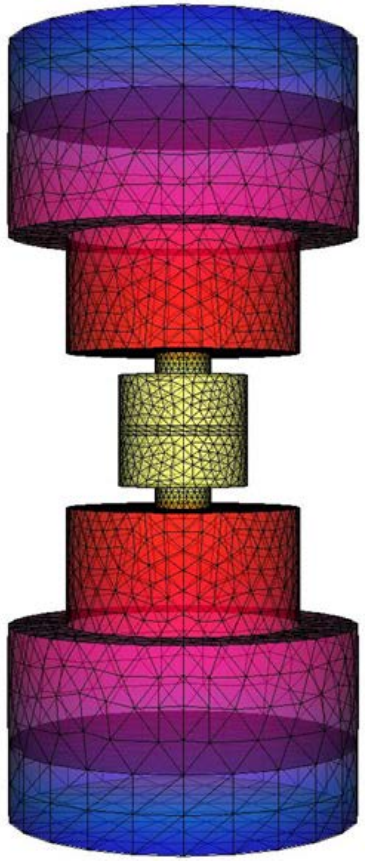
- Very fast process cycle
- Customizable tooling
- Easy to use and maintain
- Affordability

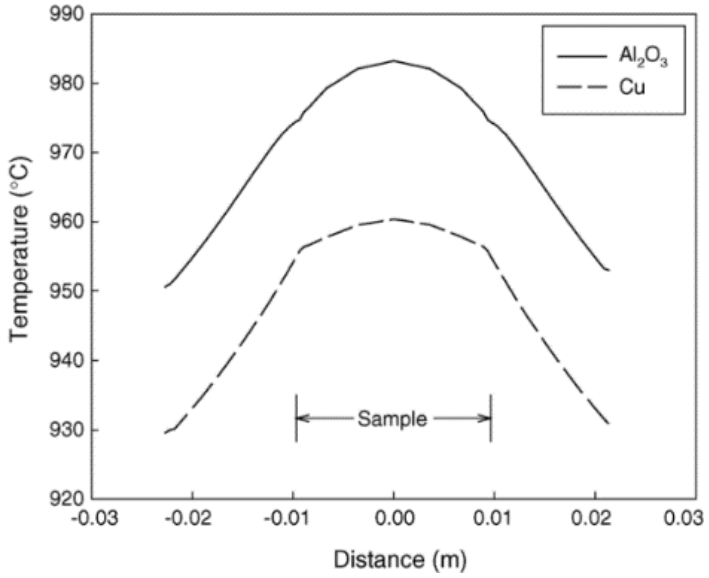
10 tons - 5000 A

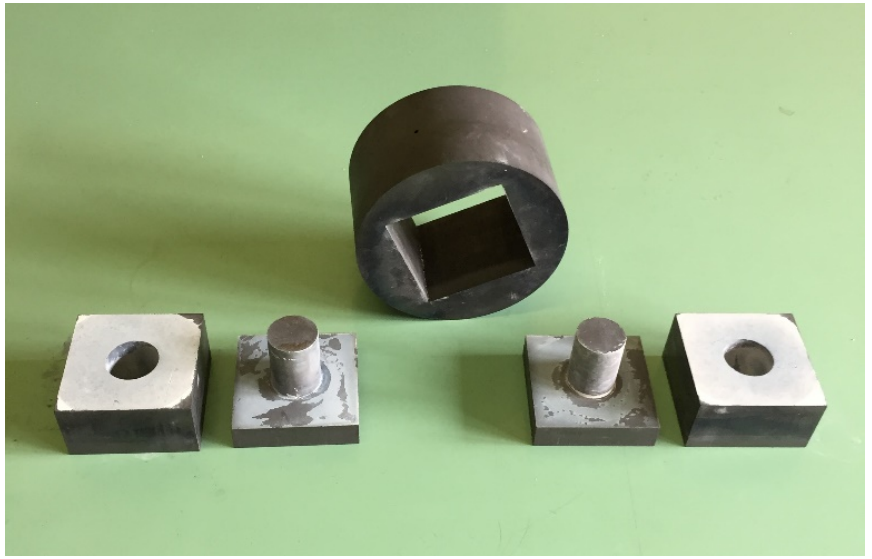
Material cost around 30 kEuro

Numerical modeling plays a crucial role in tooling optimization: molds

Claim in ALLIFLU patent







Target for this technology:

- Research labs for small-medium production but with flexibility and optimization needs for material studies, or for samples that need to be replaced at high rate
- Industries that aim at creating and testing new materials, for fast and reliable process in view of larger production
- Industries that need sintered samples of relatively large dimensions
- Industries that already manage other ways of sintering, but want to cover also this specific strategy
- Sintering of complex, multi-layer materials (e.g. ceramic over metal)

What Next ?

- In the middle of difficulties lies opportunity… ☺
- The path to innovation is definitely non-linear…
- Difficulties:
  - Lack of market knowledge and dynamics
  - People not trained to go commercial
- Opportunity:
  - Enter a market niche potentially expanding
  - Help also other INFN activities (e.g. production of thin targets for Laramed)
- Waiting for POC results
  - 1st phase passed, interview on December 18th

Aknowledgements

- Umberto Anselmi Tamburini (PI of FastSinter), Department of Chemistry, University of Pavia
- INFN Pavia Mechanical Workshop
- INFN TT Office (in particular Ilaria Giammarioli for the Patent scrutiny and Veronica Valsecchi for POC assistance)