







# CNAO tests with NIT emulsions

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XVII FOOT General Meeting, Cherasco, 17/12/2024

# Outline

- Short summary of NIT activities
- NIT sensitivity to protons: the results so far
- New sensitization tests in Laboratori Nazionali del Gran Sasso (LNGS)
  - 1. Sulphur-plus-gold (Au-S)
  - 2. Triethanolamine (TEA)
- November 2024 test beam in CNAO
  - Measurement strategy and goals
  - Expectations of NIT Fading
- NIT measurements in Japan
  - HIMAC test beam (December 2024)
  - Future measurement campaign in Japan (January-March 2025)

# Nano Imaging Trackers (NIT)

- Nano Imaging Trackers (NIT) are a novel kind of nanometric nuclear emulsion films that was designed to achieve a directional direct detection of WIMP-induced nuclear recoils
- The expected nuclear recoil track lengths in NIT are of the order of **100 nm** → extremely high spatial resolution required
- New production method: finer AgBr crystals (tunable from 20 nm to 80 nm) and dedicated low temperature development
- NIT production facilities in Nagoya (Japan) and Gran Sasso (LNGS, Italy)



#### LNGS Gel Production Machine



#### Undeveloped NIT sample





# DAMON: A new approach to Target Fragmentation

- The DAMON (Direct meAsureMent of target fragmentation) project (PRIN 2022) aims at measuring for the first time proton-induced target fragmentation in direct kinematics
- Direct detection of short fragments made possible by NIT **acting both as target and tracking devices**
- The estimated interaction probability for 200 MeV protons in a detector with 20 NIT is  $\sim 1\%$
- Among all interactions (Geant4 Simulation):
  - ~38 % occur in the emulsion gel (C, O, H, N, Ag, Br)
  - ~62% occur in the plastic support (Polystirene,  $(C_8H_8)_n$ )
  - Less than 10% of interactions on Ag, Br
- Typical energies of fragments, of the order of MeV, make them travel at least 300 nm → detectable!



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#### Sensitivity Issues: Test with Trento Data (1)

- The recorded sensitivity to primary protons (211 MeV) was extremely low
- The film was tilted by ~ 15° with respect to the beam direction so one expects approximately sin(15°)\*(180)\*0,12 ~ 6 protons per view (40x objective, 400x300 µm<sup>2</sup> views)





#### Most Views are Empty!

From XV FOOT General Meeting

### NIT Exposure at CNAO (November 2023)

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- The samples exposed at CNAO are aimed at:
  - Testing NIT sensitivity to protons at 70 MeV (exposure with a single spot of 10<sup>7</sup> primaries)
  - Testing NIT-OPERA double coating and tracking with thin OPERA layers (exposure with a single spot with 10<sup>5</sup> primaries)
  - Mechanical test with double side pouring on 170 µm thick cover glasses
- For this purpose, NIT gel from two separate batches was poured on 2 mm thick slide-glasses
- The samples have been developed in LNGS and they need to be scanned (analysis on-going, more details will be given in future meetings)





## First Results from 2023 CNAO Test Beam

- For the sensitivity test, NIT films were exposed to a high intensity spot of 10<sup>7</sup> protons @70 MeV
- NIT samples were produced in LNGS and kept in a refrigerated box during transport to minimize thermal noise
- Usual NIT development was perfomed in LNGS and the samples have been brought to Naples



**OPERA-like and NIT layers** 

together! More tests needed

From XVI FOOT General Meeting



CNAO Exposure: single high intensity spot  $(10^7 \text{ protons})$ 







# MC Study of Proton Sensitivity



XVI FOOT General Meeting, Napoli 26/6/2024

<sup>10</sup> μm AgBr Box in Air

# New Sensitization Approaches

- The 2023 test beam in CNAO showed that NIT with HA sensitization and MAA (5°C) development are not sensitive to protons @70 MeV
- HA sensitization uses sodium sulfite (halogen acceptor) to reduce the recombination due to Br at the surface of AgBr(I) crystals, without modifying the structure of the crystals
- DAMON needs a more aggressive approach with sensitization  $\rightarrow$  new tests performed in LNGS



#### **Unsensitized vs TEA sensitized**

#### Sulphur-plus-gold (Au-S)

#### Triethanolamine (TEA)

Acts by forming  $Ag_2S$  sensitization center on the surface of the AgBr(I) crystals. Gold helps the  $Ag_2S$  sensitization center and the development of latent image Extensive experience with OPERA-like emulsions (sensitive to MIP), but no experience with NIT

Acts by partially melting AgBr(I) crystals thus leading to the formation of AgOH which becomes  $Ag_2O$  and  $H_2O$  $Ag_2O$  can then be reduced to Ag thus **increasing the amount of silver** in the development centers

## Sensitization Tests in LNGS

Sensitizer	Condition	Exposure
Au-S	OPERA recipe	$\alpha, \gamma, \beta$ + ref
Au-S	OPERA recipe adjusted by surface ratio (70 nm vs 200 nm)	$\alpha, \gamma, \beta$ + ref
TEA	5 g/L solution, dip for 15 minutes	α,γ + ref
TEA	5 g/L solution, dip for 10 minutes	$\alpha,\gamma$ + ref
TEA	24.5 g/L solution, dip for 15 minutes	α,γ + ref
TEA	24.5 g/L solution, mix to gel (same proportion as HA)	α,γ + ref
HA	Standard recipe	$\alpha,\gamma$ + ref

### Radioactive Sources in LNGS

Source	Emission	Activity	Exposure time
LNGS-150	Mainly $\gamma$ at 60 keV from $^{241}Am$	402 <i>kBq</i> (08/11/24)	3 min
LNGS-160	<sup>241</sup> Am with thin window to for α radiation (~ 5.4 MeV)	73.9 <i>kBq</i> (07/11/24)	4 min
LNGS-036	$\beta$ from Sr-90	231 <i>kBq</i> (07/11/24)	30 s

**LNGS-150** 



#### LNGS-71



~3 *mm* 

### Au-S and TEA: Very First Results

- In general, an increase in sensitivity comes with an increase in noise (fog for NIT)
- For Au-S sensitization, the second condition (adjusted ratio) proved to be **too** aggressive (gray but transparent emulsions with MAA development)
- For TEA, there was an issue keeping the samples too long in the sensitizer solution (probably the interaction of TEA with the undercoat led to emulsions detaching at the sides)





Sensitizer	Condition
TEA	Dip for longer than 7-8 minutes (5 g/L, 24.5 g/L)

# Sensitivity to Gamma Radiation

- Photo-electrons from the gamma source have too low ionization to be detectable in HA-sensitized NIT, except for the very last part of their range
- Therefore, HA-sensitized NIT only show isolated grains coming from photo-electrons when exposed to the gamma source
- A simple way to quantify the relative sensitivity of different batches (or different sensitization) consists of counting the number of sensitized grains in the central part of the irradiated region





#### Measurement of $\gamma$ sensitivity

- 1. Line scan (200 views) along Y direction
- 2. Line scan (200 views) along X direction
- 3. Line scan along the central part of the irradiated region (200 views, Y direction)
- Compare the height of the peak (~ number of grains) after an exposure to the same source, for the same time and at the same illumination conditions

### Visual Inspection: Au-S



- Detail from 1 microscope view (~ 70  $\times$  50  $\mu m^2$ ) near the gamma peak
- Many short tracks (2 to 3 grains) visible, a few even longer ones
- Appreciable increase in sensitivity but also in fog



### Relative Sensitivity: HA vs Au-S

Line scan (Y direction, 200 views) along the peak of the gamma source (3 minutes standard exposure) HA

fitYline gr.y+y {(((gr.z>0.000000&&gr.z<20.000000&&vid>=0&&vid<=199)&&(ngr<3000))&&(abs(gr.x)<25&&abs(gr.y)<25))&&(flad==0) 49474 Entries 2400F 3092 Mean RMS 2184 2200 χ<sup>2</sup> / ndf 475.5/44 2000F 3.371e+009 ± 1.171e+008 p0  $2977 \pm 10.7$ p1 1800F p2 -1301 ± 24.7 1600  $\textbf{300.2} \pm \textbf{9.6}$ p3 1400F 1200 1000E 800F 600E 400E 200

-600040002000

Au-S



## Visual Inspection: TEA (mix)



- Detail from 1 microscope view (~ 70  $\times$  50  $\mu m^2$ ) near the gamma peak
- Many short tracks (2 to 3 grains) visible, a few even longer ones
- Similar conclusions to Au-S samples from eye-check



## Relative Sensitivity: HA vs TEA (mix)

 Line scan (Y direction, 200 views) along the peak of the gamma source (3 minutes standard exposure)



### LNGS Test Results

Sensitizer	Condition	Exposure	
Au-S	<b>OPERA</b> recipe	$\alpha, \gamma, \beta$ + ref	<ul> <li>Both Au-S and TEA consistized</li> </ul>
Au-S	OPERA recipe adjusted by surface ratio (70 nm vs 200 nm)	$\alpha, \gamma, \beta$ + ref	NIT showed a significantly
TEA	5 g/L solution, dip for 15 minutes	$\alpha, \gamma$ + ref	than HA samples
TEA	5 g/L solution, dip for 10 minutes	α,γ + ref	<ul> <li>Fog is larger in Au-S samples (and preparation)</li> </ul>
TEA	24.5 g/L solution, dip for 15 minutes	α,γ + ref	is more complex)
TEA	24.5 g/L solution, mix to gel (same proportion as HA)	<i>α,γ</i> <b>+ ref</b>	<ul> <li>TEA sensitization seems to be the</li> </ul>
HA	Standard recipe	<i>α</i> , γ <b>+ ref</b>	best approach

#### NIT Exposures in CNAO 2024







Sensitizer Recipe	Support Material	Exposures	Purpose
Au-S with OPERA recipe HA TEA (mix)	Plastic	<sup>12</sup> C at 200 MeV/n and 400 MeV/n	Alignment + Tracking
Au-S with OPERA recipe HA TEA (mix)	Slide Glasses (1 mm) Plastic	p at 15 MeV, 30 MeV, 45 MeV, 55 MeV, 70 MeV, 200 MeV	Sensitivity Test Alignment + Tracking (with plastic if sensitivity is





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high enough)

## Results from CNAO 2024

- New RP rules → exposed NIT samples are still in CNAO
- Discussion between RP experts (Napoli & CNAO) on-going, updates expected at the start of January 2025
- No results before
   2025 Japan campaign



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# NIT Fading: a quick look

A/B

- NIT exposed at the 2024 test beam in CNAO are currently stored at -20°C
- No previous experience with protons, but NIT fading test with alpha and gamma sources were performed in the past

Exposure A Store in each place Exposure B development -20°C Control to room temperature

Fading time

B / Oday



#### Relative Fading @-15 °C

140

## Results from CNAO 2024

- Only educated **guess** of the sensitivity to protons possible
  - 1. Eye checks of Au-S and TEA samples have shown short tracks (~ 1  $\mu m$  in length)
  - 2. At 30 keV electrons are expected to have ~  $1 \mu m$  CSDA range in emulsion (NIST database)
  - 3. The total stopping power for 30 keV electrons in water is equal to  $\sim 9.6 MeV/cm$
  - 4. The stopping power is comparable to protons at around ~ 70 MeV
- Results from reference samples show comparable levels of background (no major issues during transport)



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# NIT Measurements in Japan (HIMAC)

- TEA sensitization is also being investigated by T. Naka and T. Asada in Japan
- Latest measurement: test beam at HIMAC, Chiba (13/12/2024) exposing NIT to 290 MeV/n  $^{12}C$



- Purpose: testing crystal efficiency with horizontal exposures to carbon ions (several TEA conditions tested)
- Analysis on-going at Toho university





12C 290 MeV/n @HIMAC HA

Pictures by T. Asada

# Upcoming NIT Measurements in Japan

- Many activities planned from ~ January 2025 to March 2025
- Machine time booked at Nagoya proton therapy center (3 exposure slots are expected to be available)



- Define <u>optimal sensitization and support material</u> (overlap with 2024 CNAO campaign?)
  - Use of alternative plastic materials like PMMA (first checks show promising optical properties)
  - Testing reversal development for NIT (70 nm) to improve contrast
- <u>Physics measurement</u>: expose a NIT brick to protons to reconstruct vertices
- Work to be done between Nagoya and Toho (Vincenzo, Giuliana + T. Naka, T. Asada)



## Conclusions

- New NIT sensitization approaches tested in LNGS and at the 2024 CNAO test beam
- Proton sensitivity still unknown (December 2024)
- Additional measurements to characterize TEA sensitization on-going at HIMAC
- Future
  - TEA sensitization test with more conditions in Japan (January-February 2025)
  - Physics measurement in Japan (March 2025)













# Au-S: Background increase over time

- The first tests with Au-S were performed last September
- In November, we sensitized more gel and compared the fog level with the previous batch

#### Au-S (Sep24, developed in Nov24)

#### Au-S (Nov24)



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