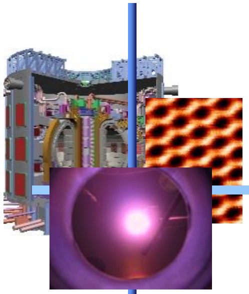


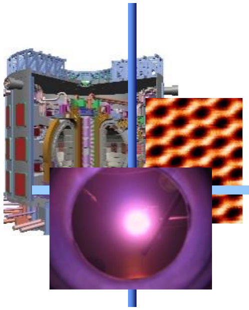
6th NIBS, NIBS, Novosibirsk, 3rd-7th September 2018

H^-/D^- negative-ion surface production on Nanoporous 12 CaO. 7 Al_2O_3 (C12O7) electride surface in low-pressure Cs-free plasmas

R.Moussaoui

Supervisors: G. Cartry
Aix-Marseille Université, CNRS, PIIM, UMR 7345, Centre de Saint Jérôme, 13013 Marseille, France

Daishuke Kuwahara (Doshisha University)
Masumi Kobayashi (Doshisha University)
Takayuki Eguchi (Doshisha University)
Mamiko Sasao (Doshisha University)
Motoi Wada (Doshisha University)



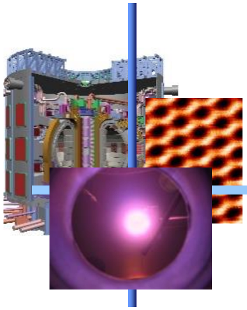
Introduction

High current H$^-$/D$^-$ negative-ion sources are required for fusion (40A D$^-$ @ 1MeV)

The ITER caesium-seeded negative-ion source is meeting these requirements

However, caesium is causing many issues: pollution, consumption, stability of long pulse operation…

Reduction of caesium consumption or its elimination would be highly valuable for a fusion reactor (DEMO)



Introduction

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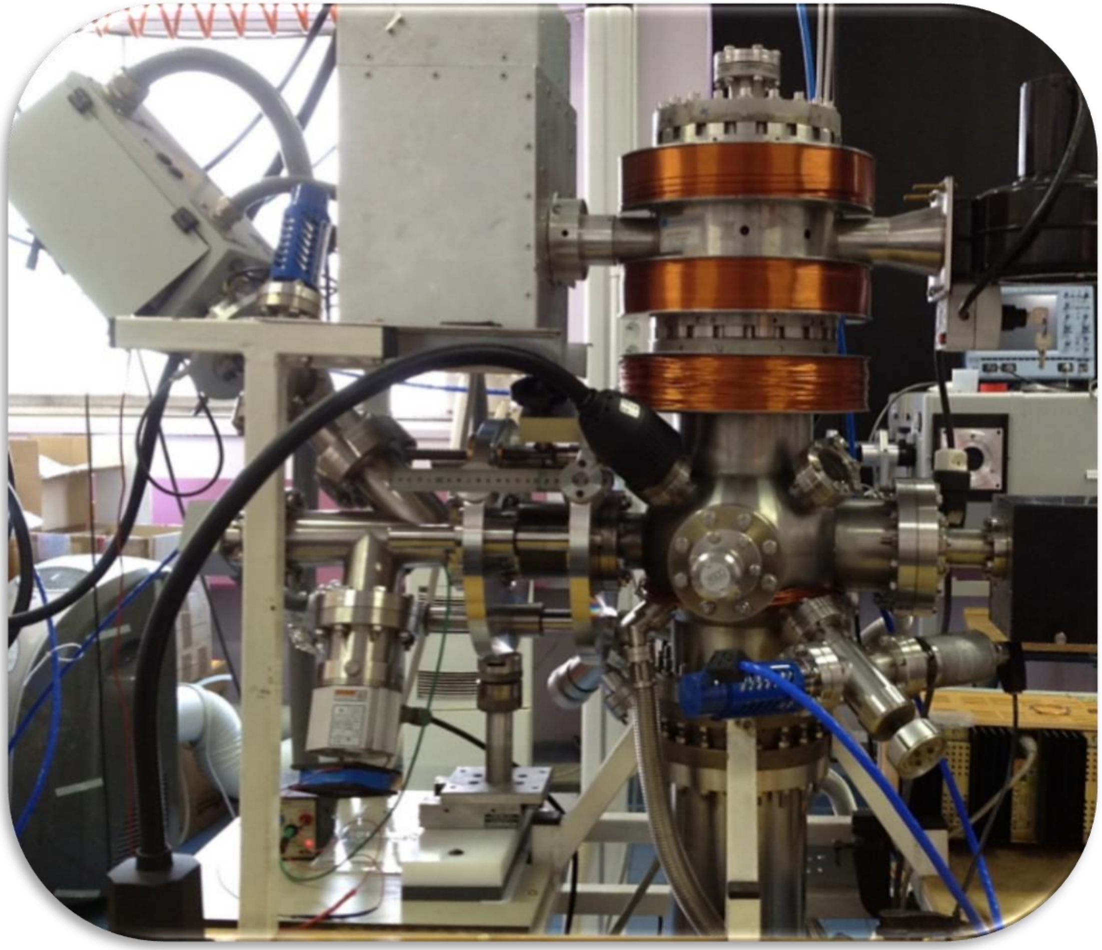
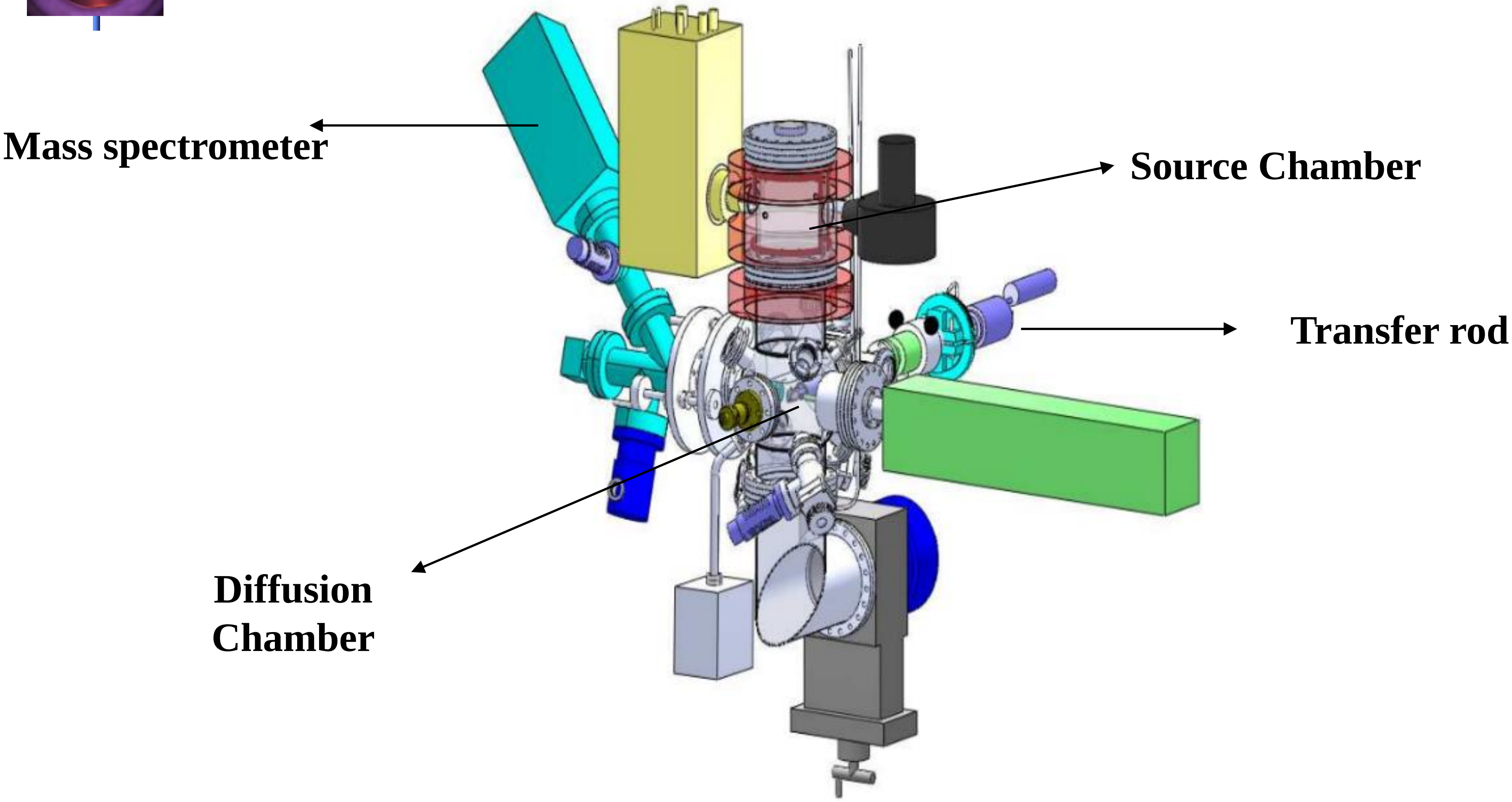
The ITER caesium-seeded negative-ion source is meeting these requirements

Study of negative-ion enhancer materials (other than Cs) in H_2 plasmas

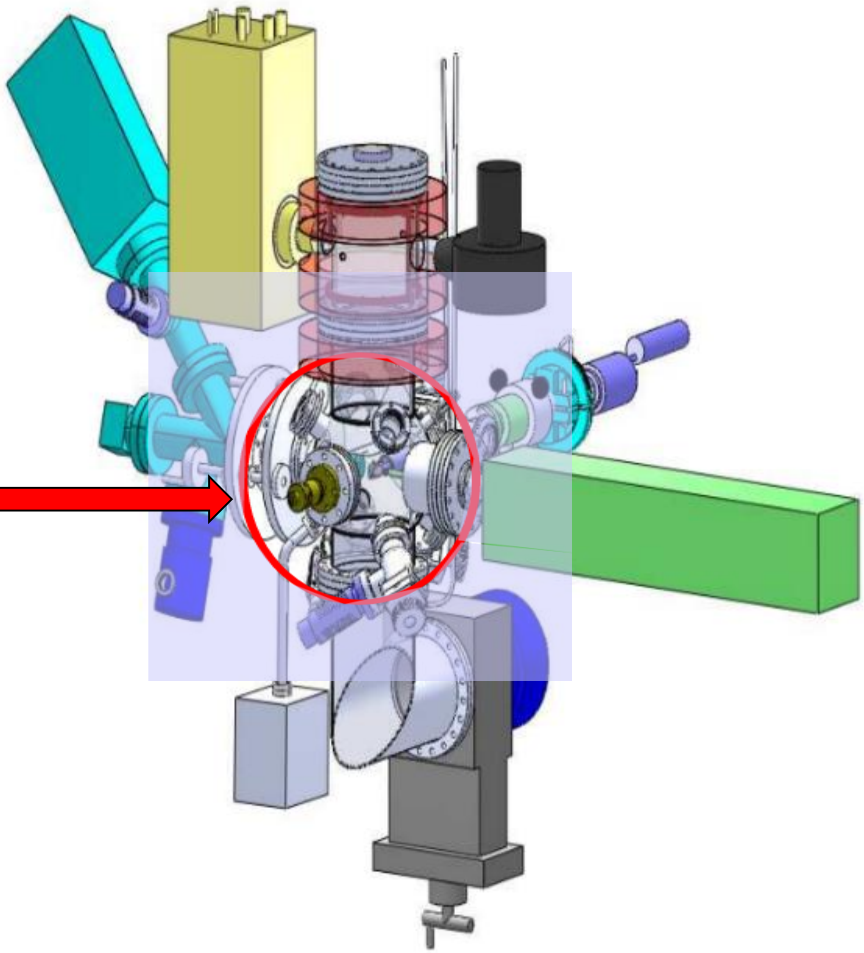
Experimental setup: PHISIS



PHISIS

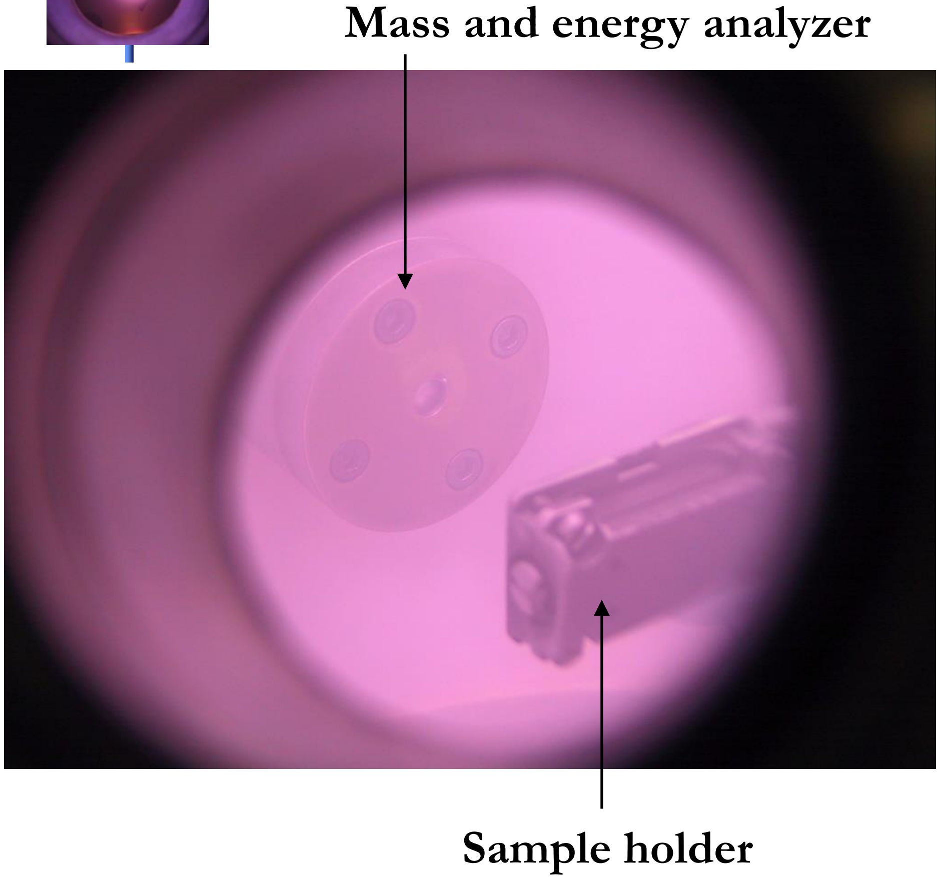

Mass spectrometer
Source Chamber
Transfer rod
Diffusion Chamber

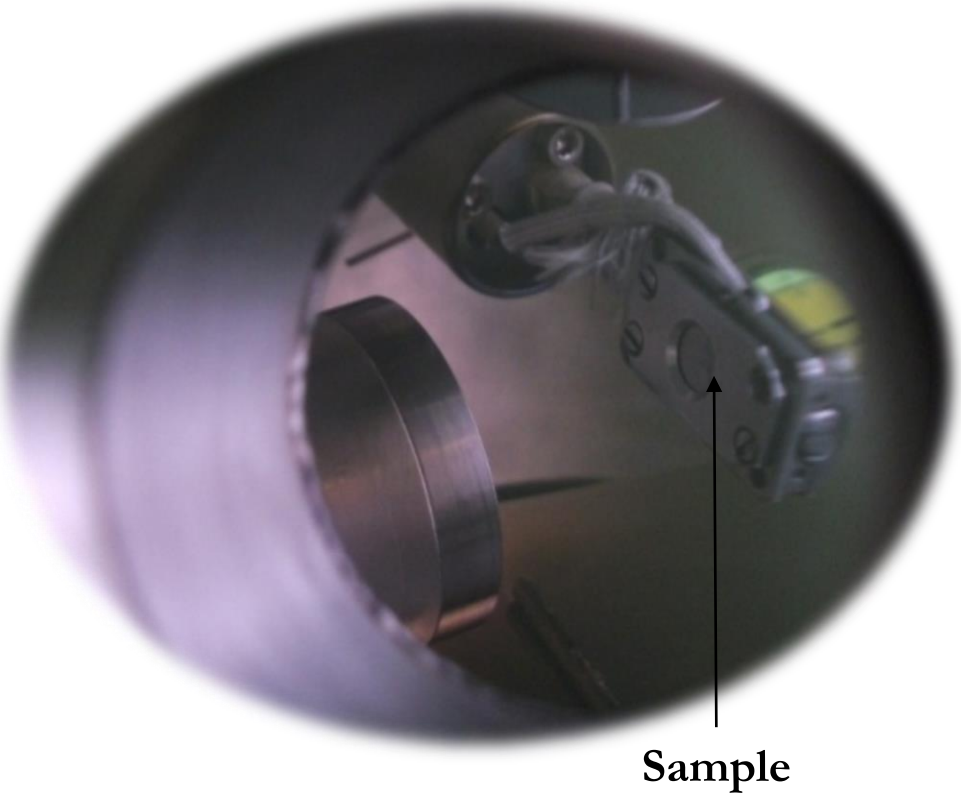
Study method



Zone of interaction plasma- Sample

Experimental set-up





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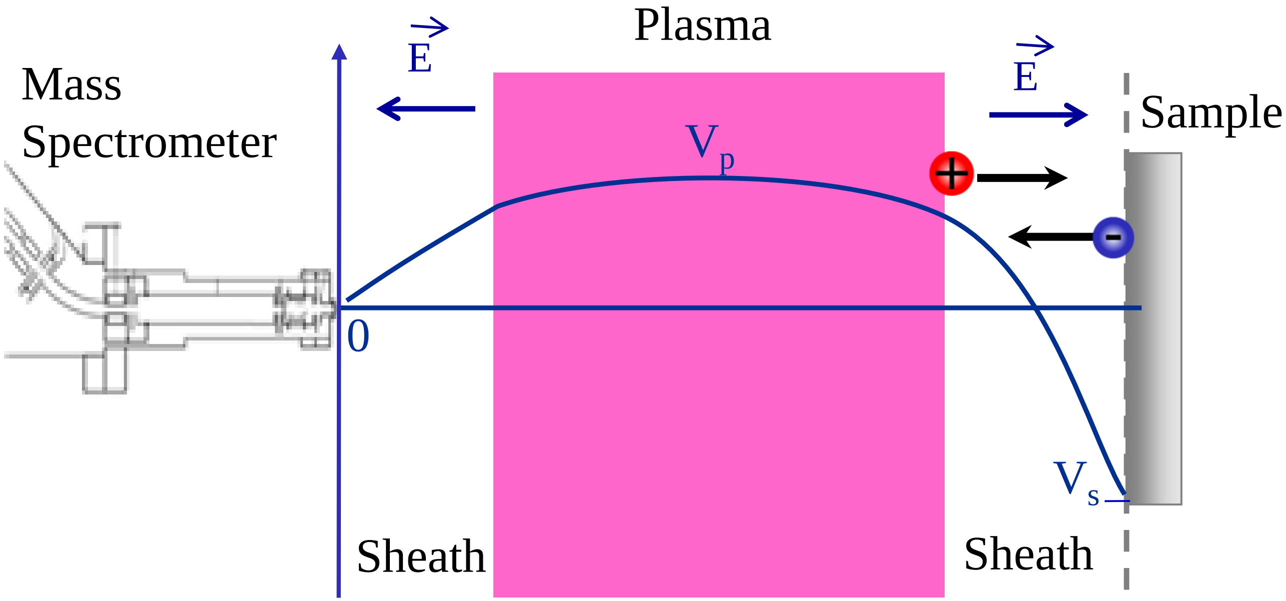

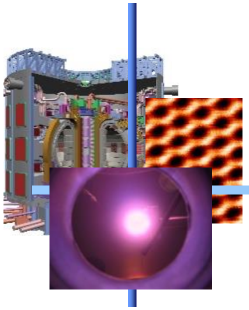

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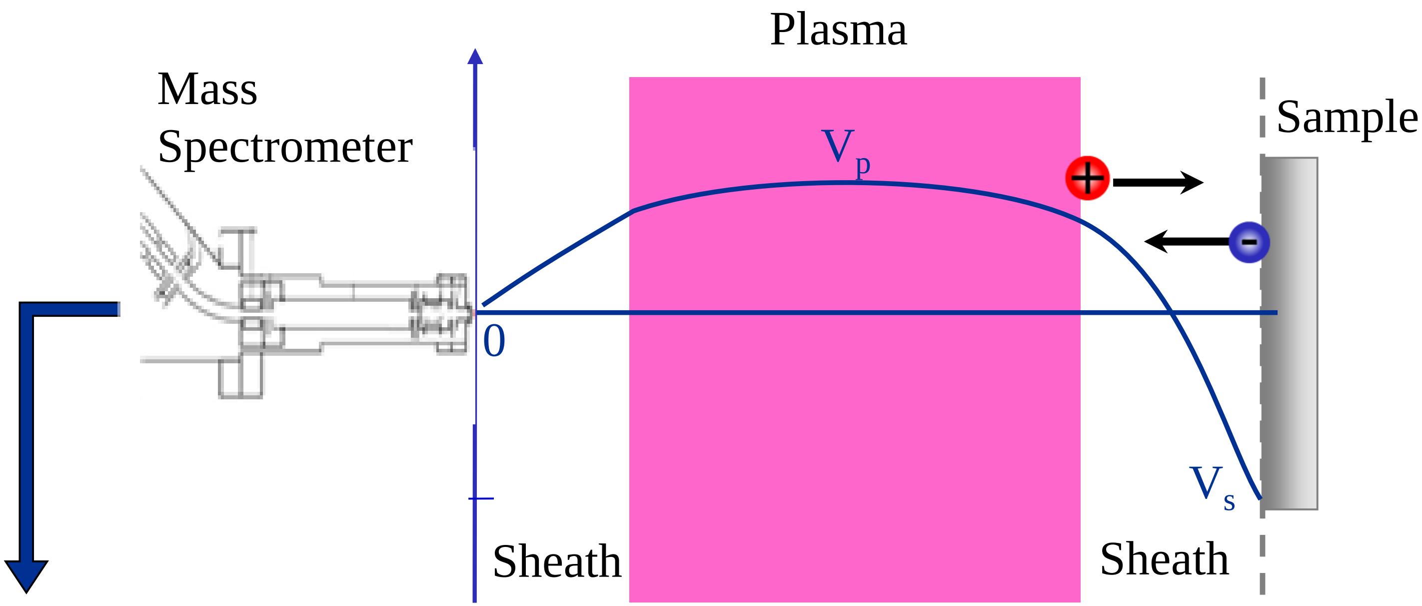
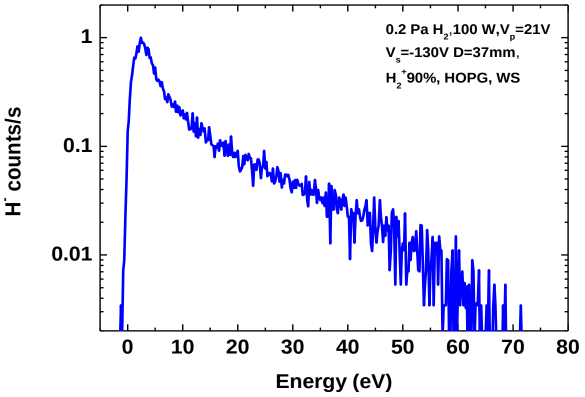
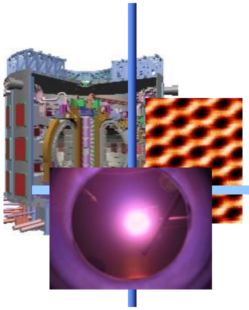




Negative ion energy distribution function (NIEDF)



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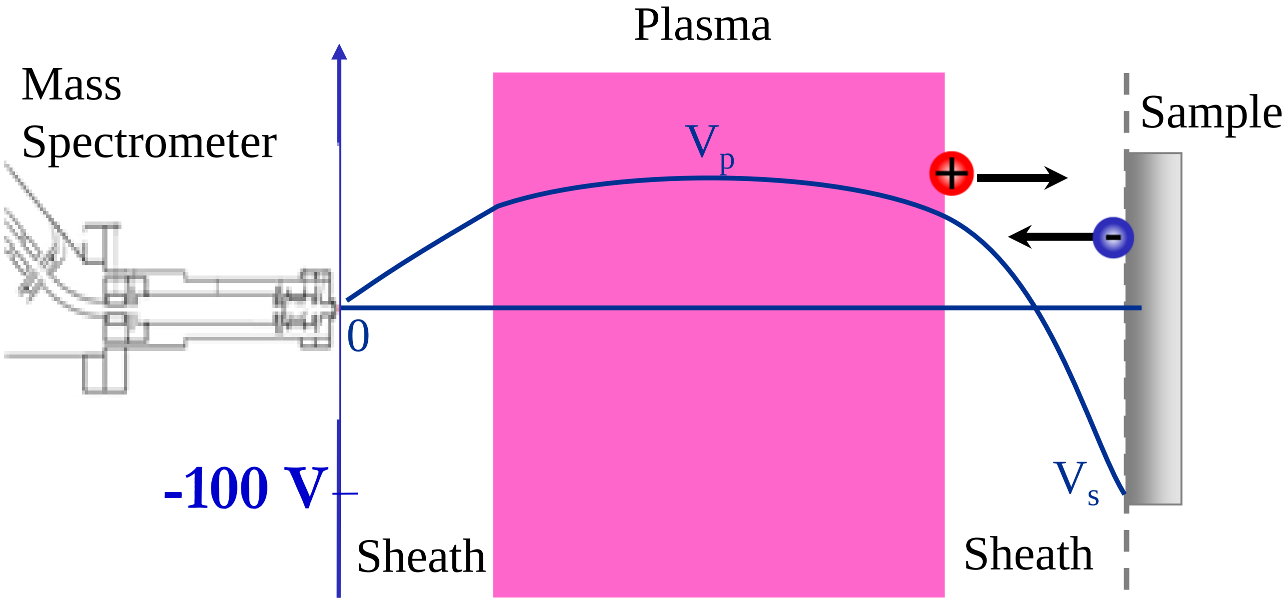
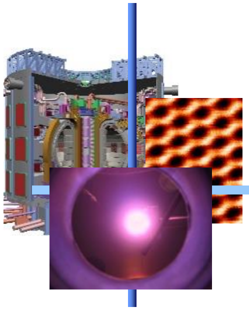


Negative-ions (i^-) are self-extracted:

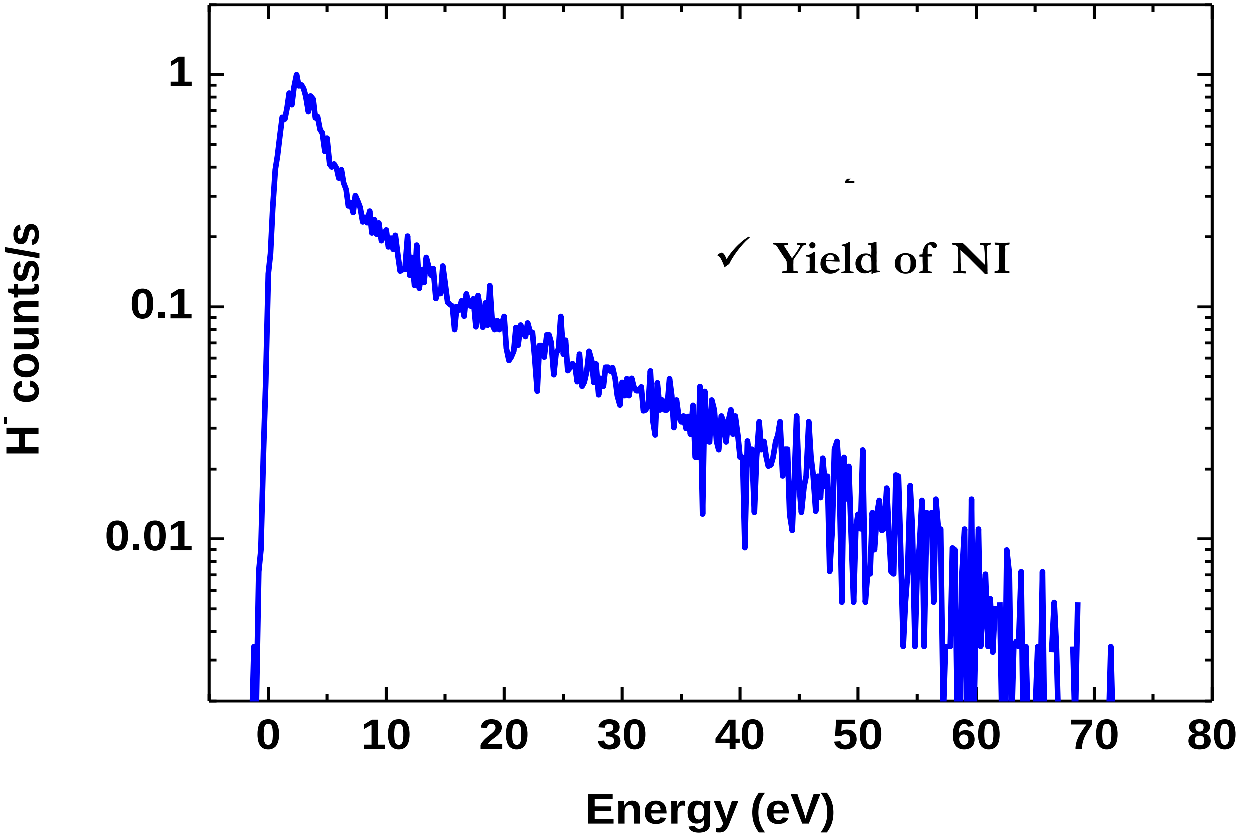
Advantages: simple extraction, sample materials can be changed easily

Drawback: Bad control of surface state. It is strongly dependant on plasma conditions and ion bombardment



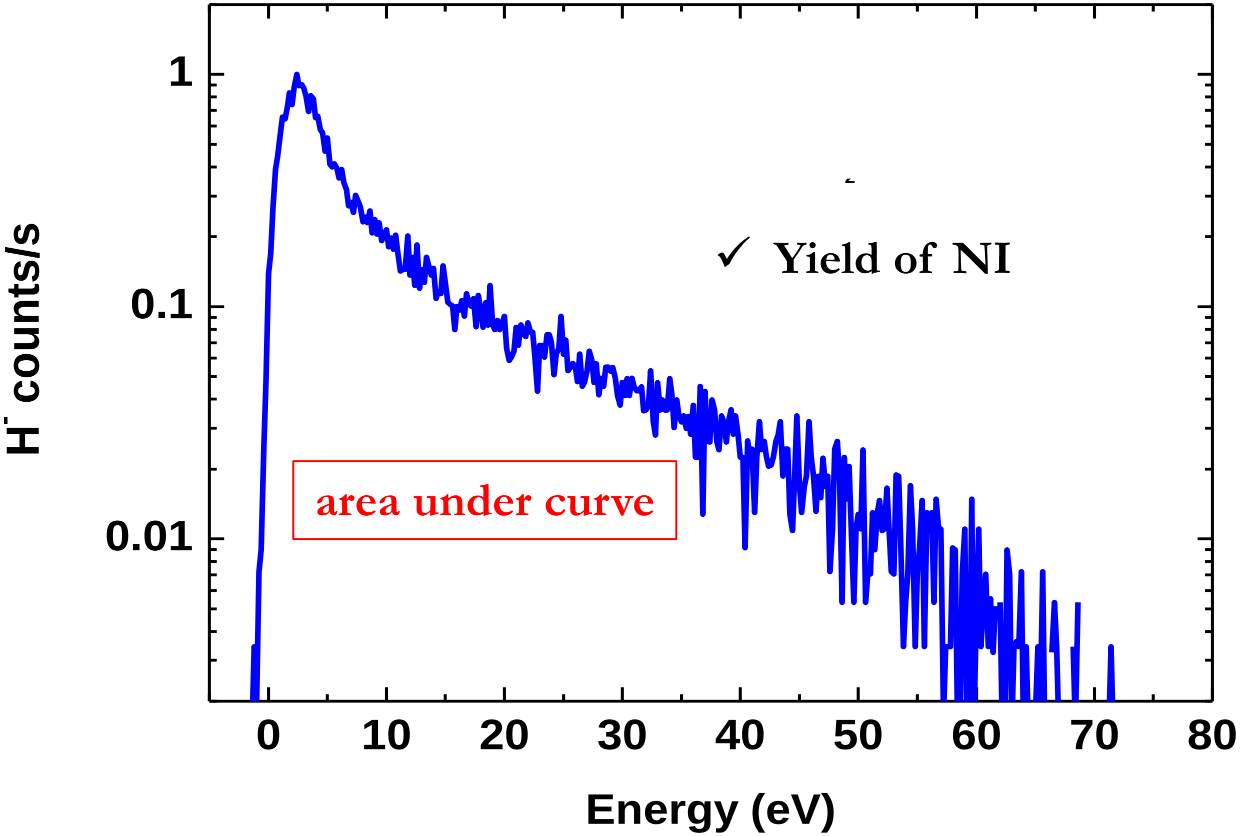
NIEDF

Negative Ions Energy Distribution Function



✓ Yield of NI

Negative Ions Energy Distribution Function

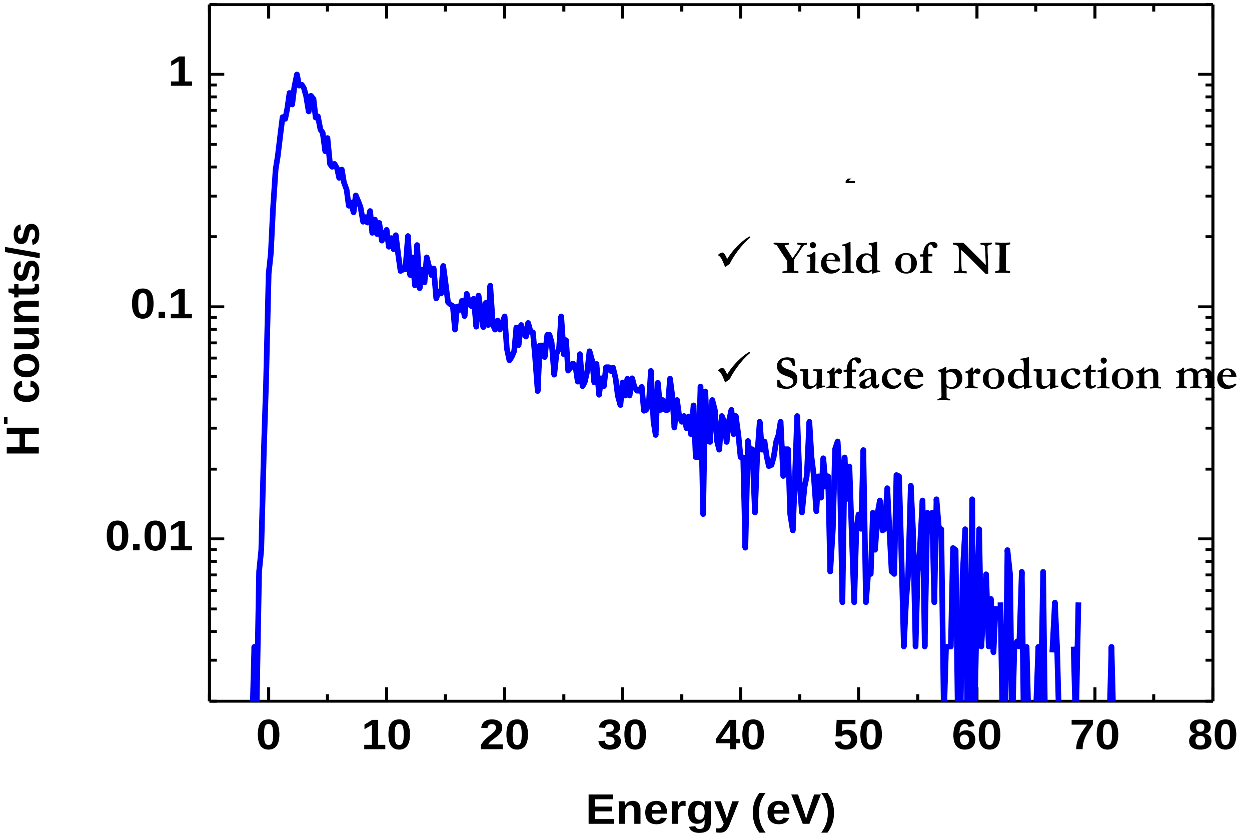


✓ Yield of NI

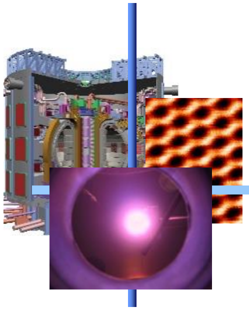
area under curve

NIEDF

Negative Ions Energy Distribution Function

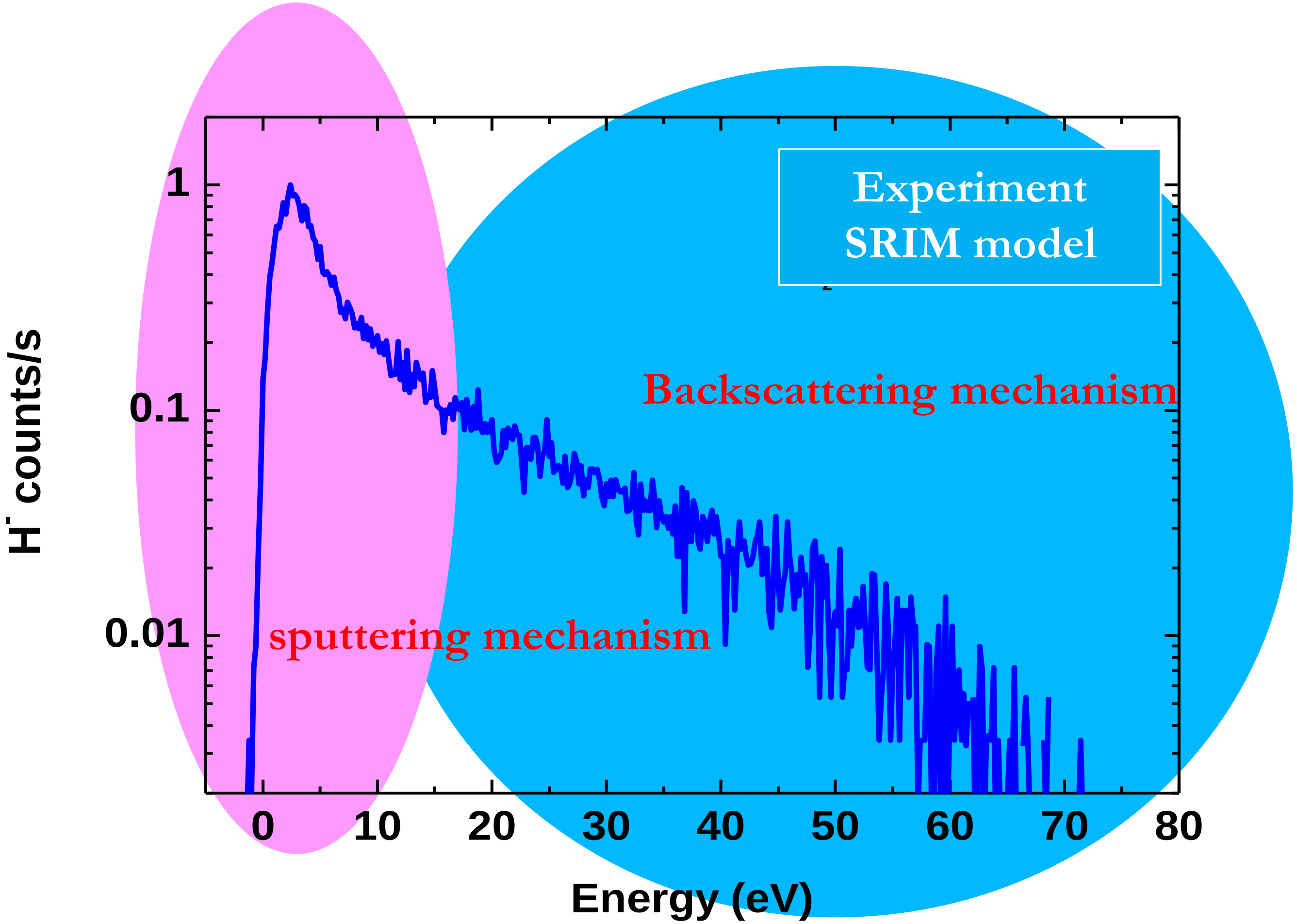


- ✓ Yield of NI
- ✓ Surface production mechanisms of NI

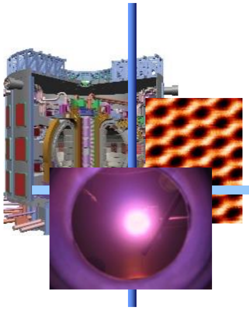


NIEDF

Negative Ions Energy Distribution Function



NIEDF shape is determined by the relative contribution of two production mechanisms sputtering and backscattering.



Experimental conditions

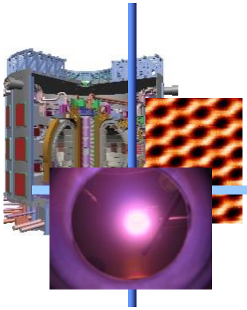
Samples:

- HOPG : Highly Oriented Pyrolitic Graphite
- MCBDD : Micro Cristalline Boron Doped Diamond
- NCD : Nano Crystalline Diamond
- MCD : Micro Crystalline Diamond
- NDD: Nitrogen Doped Diamond
- Nanoporous $12CaO\cdot 7Al_2O_3$ (C12A7)

Plasma Conditions :

- H_2 and D_2 plasma
- P = 30-900 W

- No magnetic field
- Pr = 0.2 – 2 Pa



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Collaboration with Doshisha university

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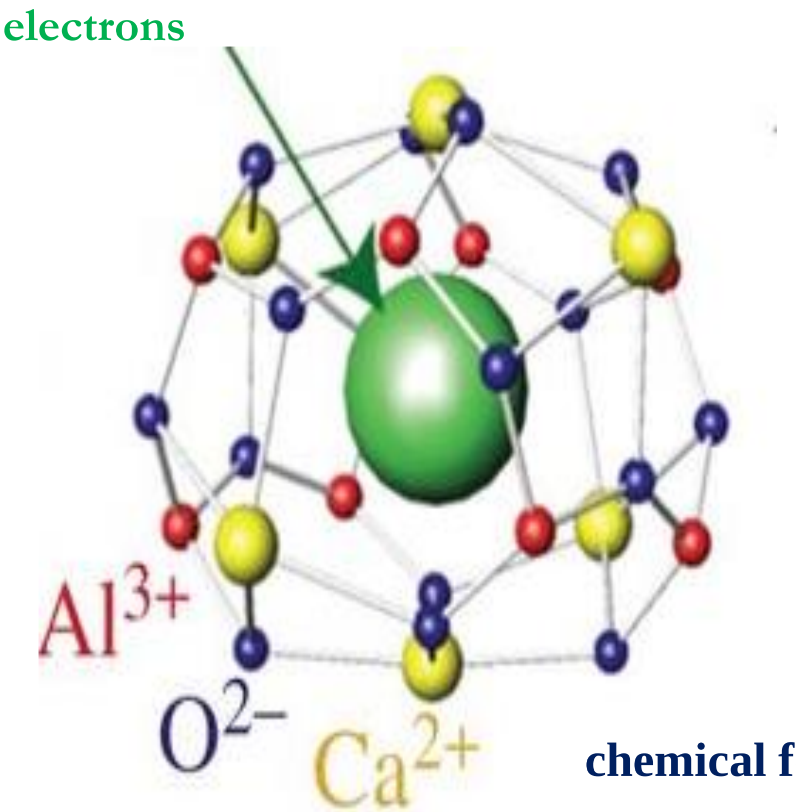
C12A7 electride surface

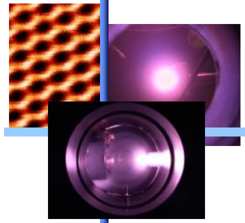


chemical formula is $[Ca_{24}Al_{28}O_{64}]^{4+} + 4e^{-}$

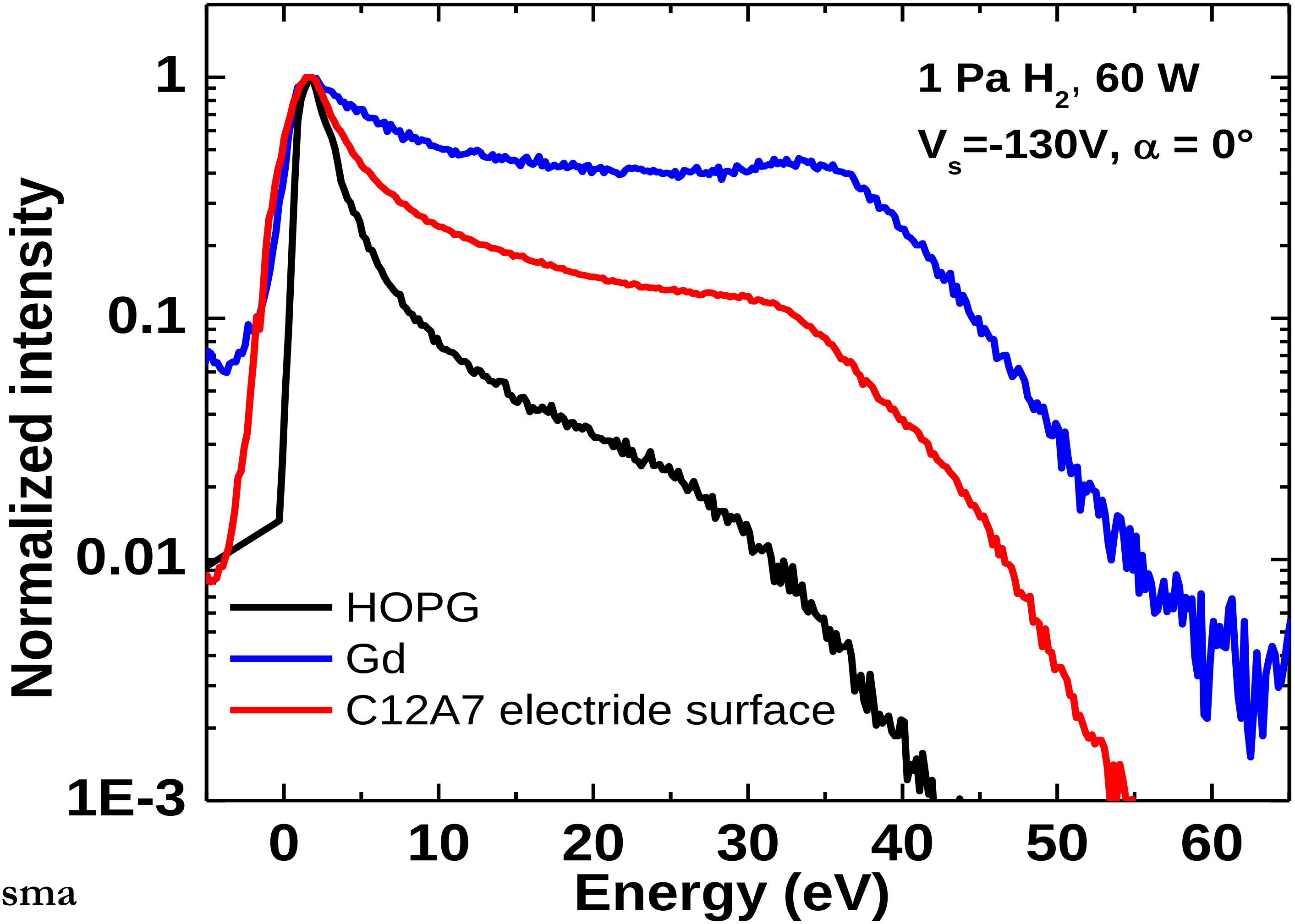
Kitano et al., NATURE CHEMISTRY | VOL 4 | NOVEMBER 934 2012

Conductivity: 1000 Siemens/cm
Low work function
Thermal stability
Chemical stability

Favorable proprieties for negative ion production

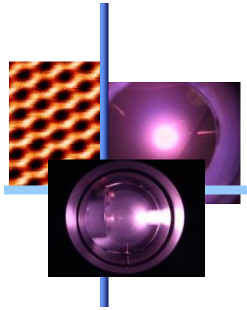


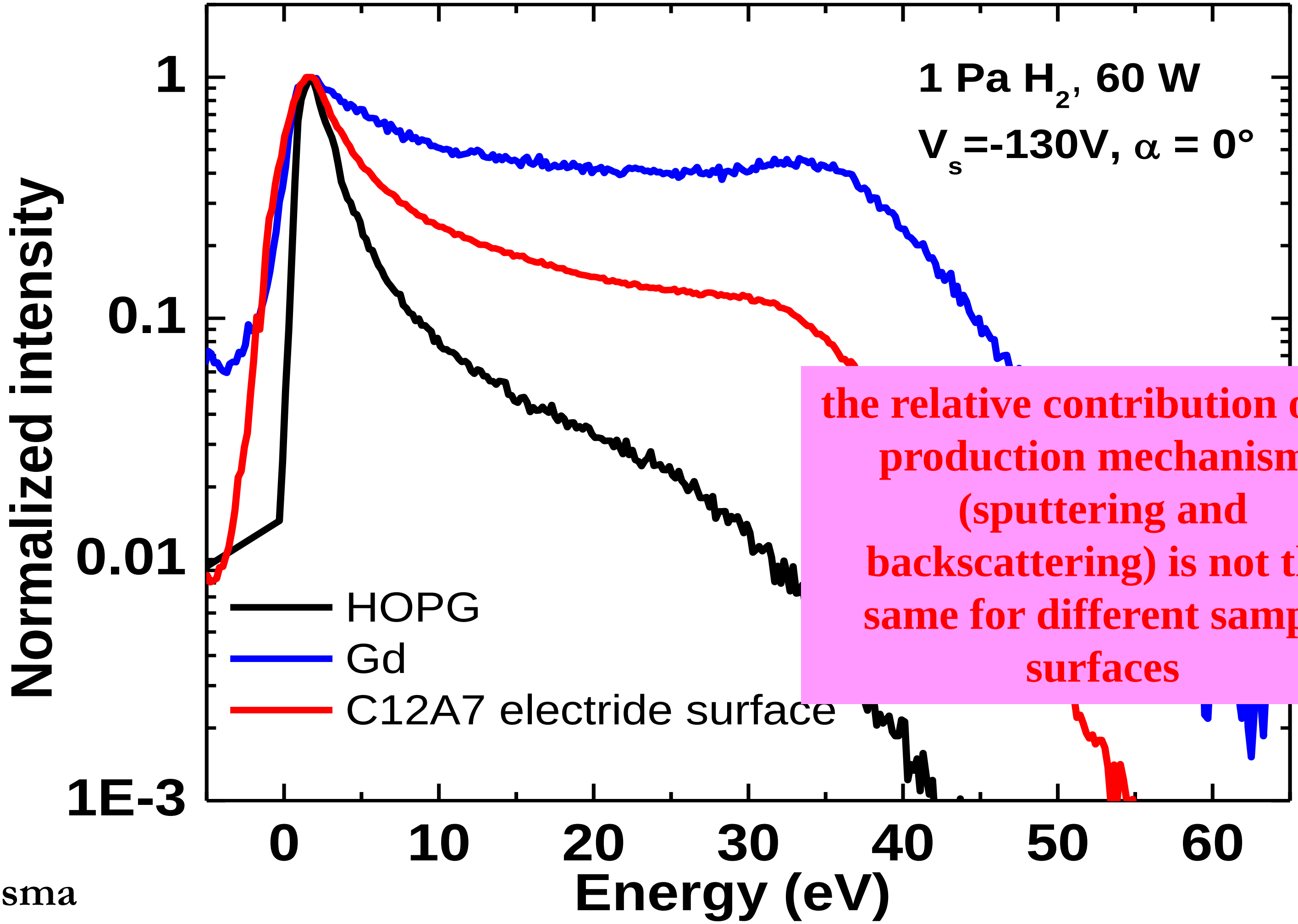
NI surface production mechanisms on C12A7 electride surface



ECR H_2 plasma
ne= 10^9 /cm3
Te= 1 eV

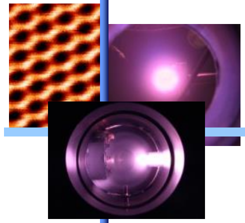
NI surface production mechanisms on C12A7 electride surface





the relative contribution of NI production mechanisms (sputtering and backscattering) is not the same for different sample surfaces

ECR H_2 plasma
ne= 10^9 /cm^3
Te= 1 eV



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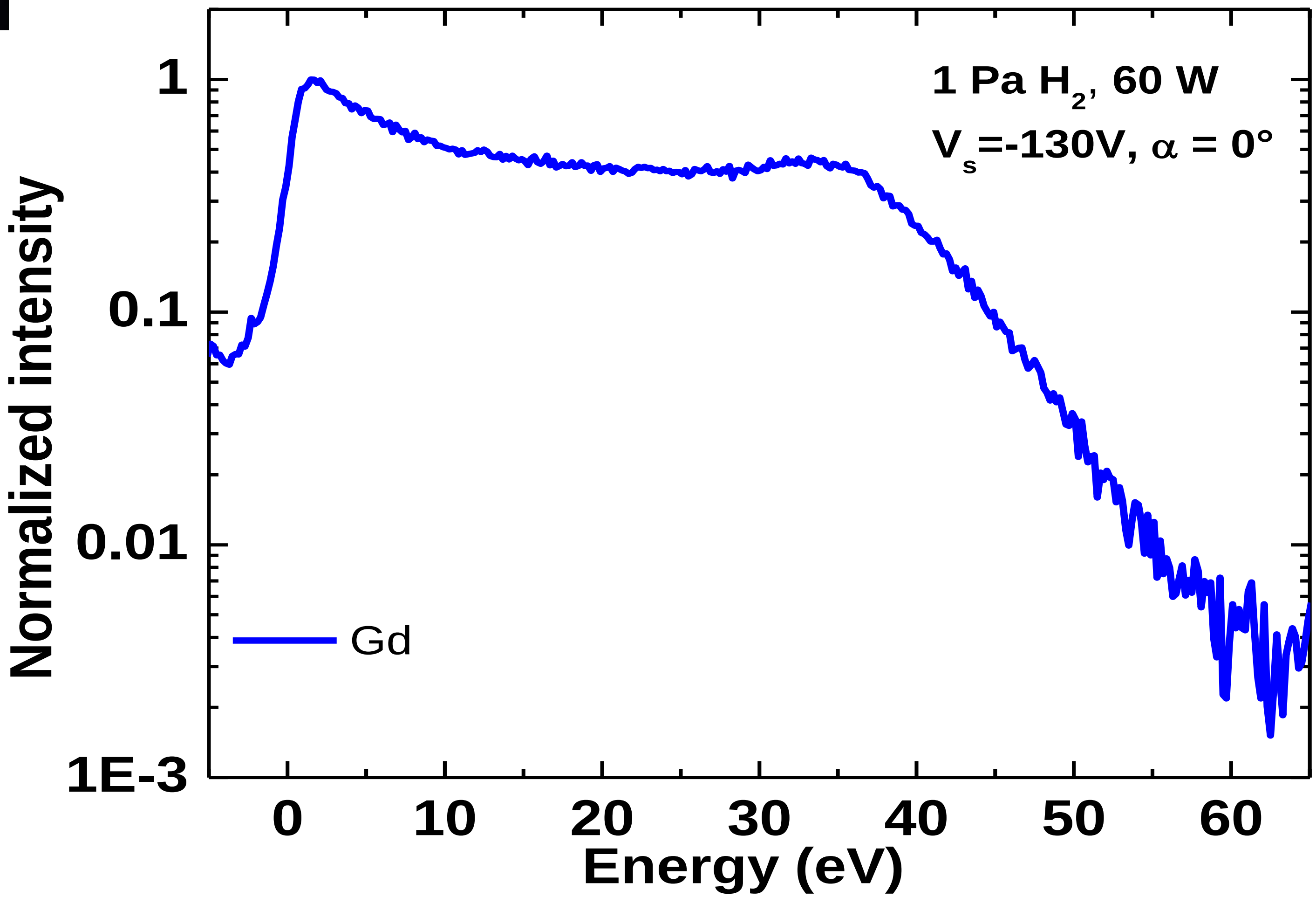
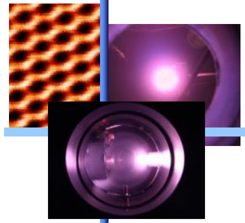
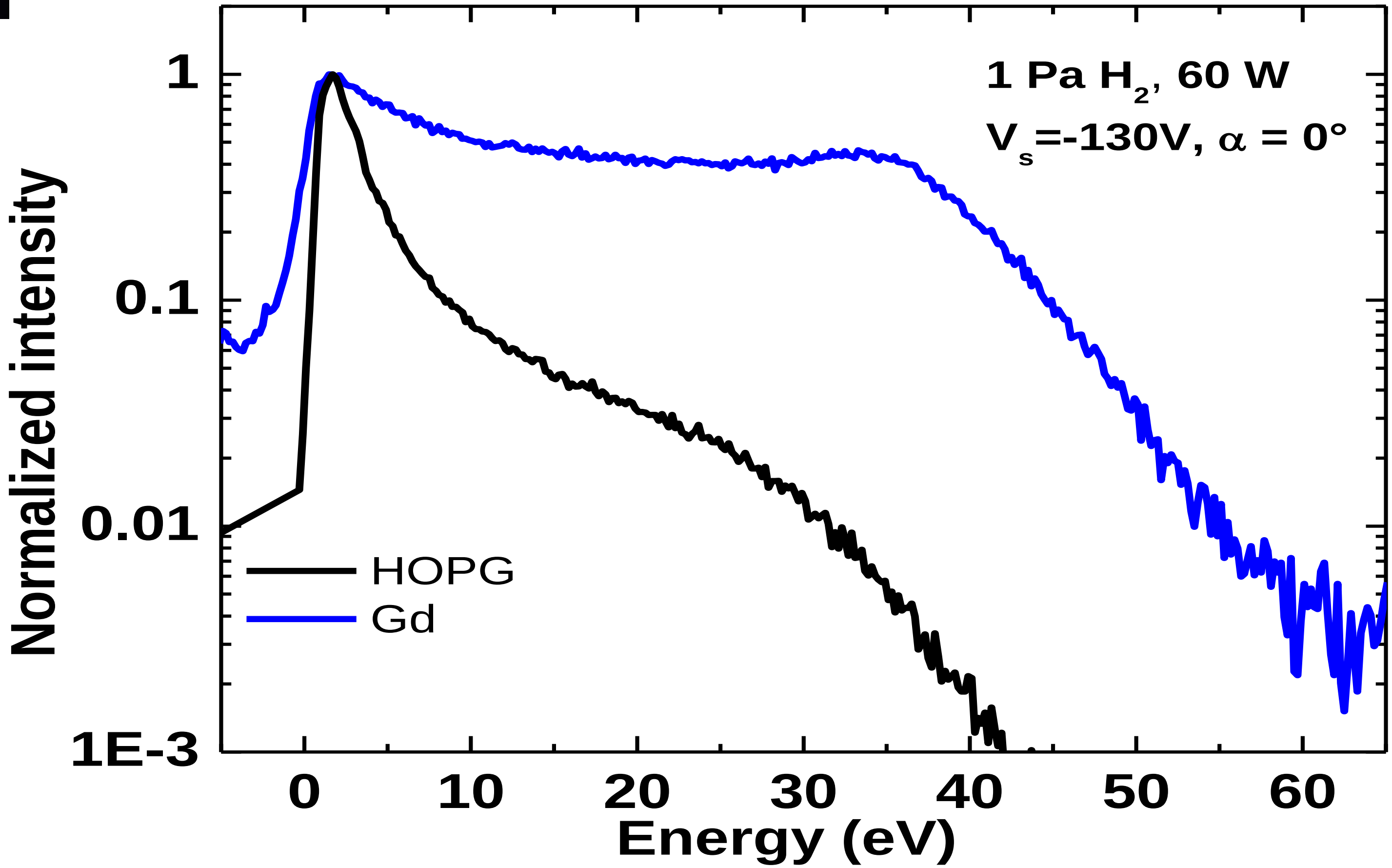


Gd the major process is backscattering of ions with a peak of production at 36 eV

D. Kogut et al., ‘Reconstruction of Energy and Angle Distribution Function of Surface-Emitted Negative Ions in Hydrogen Plasmas Using Mass Spectrometry’, Plasma Sources Science and Technology 26, no. 4 (7 March 2017): 045006.



NI surface production mechanisms on C12A7 electride surface



Gd the major process is backscattering of ions with a peak of production at 36 eV

HOPG the sputtering contribution due to adsorbed H on the surface is also important and there is production peak at low energy

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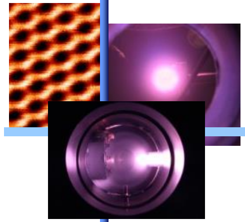
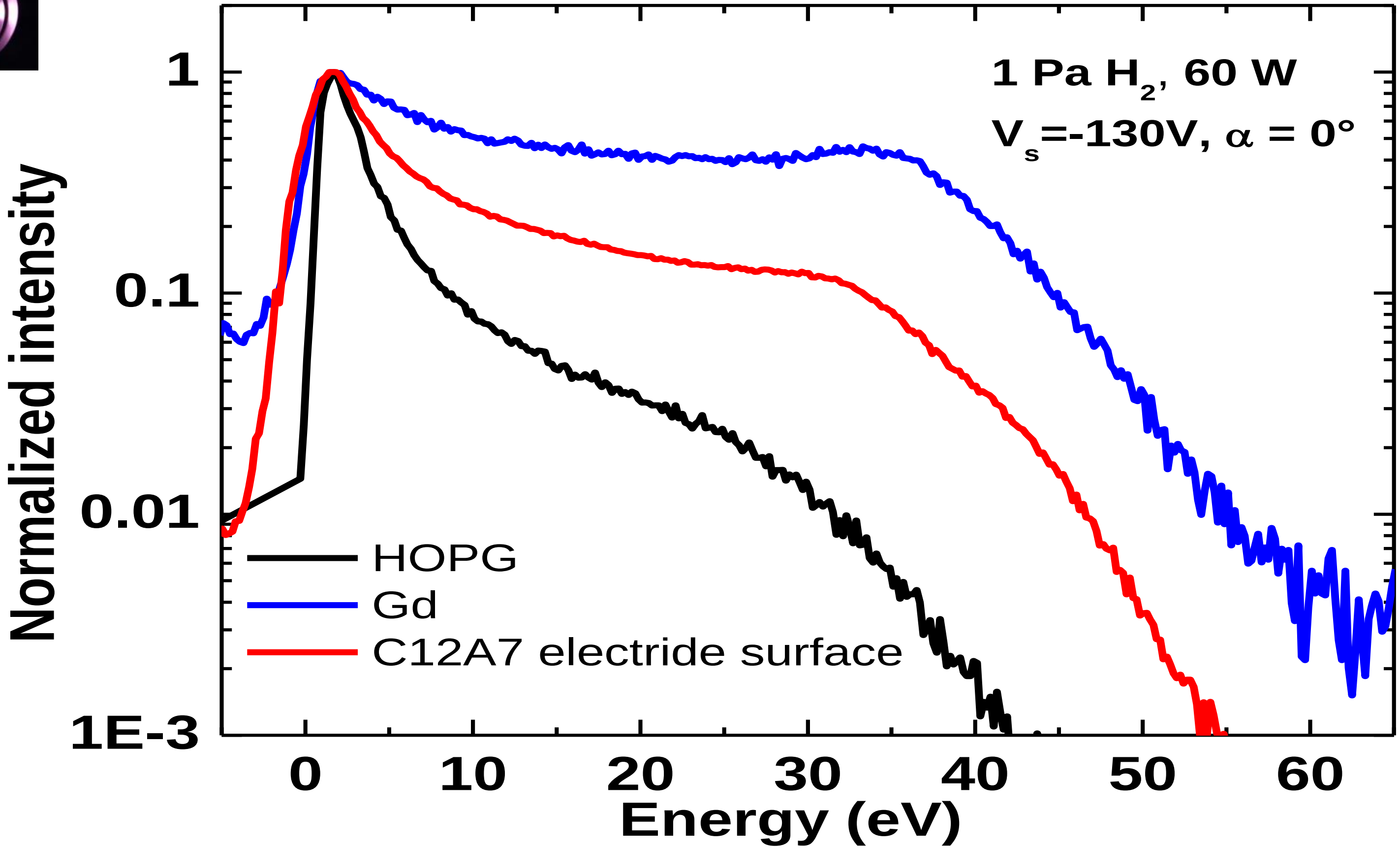


NI surface production mechanisms on C12A7 electride surface



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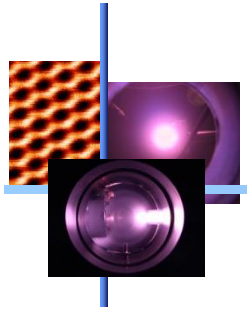
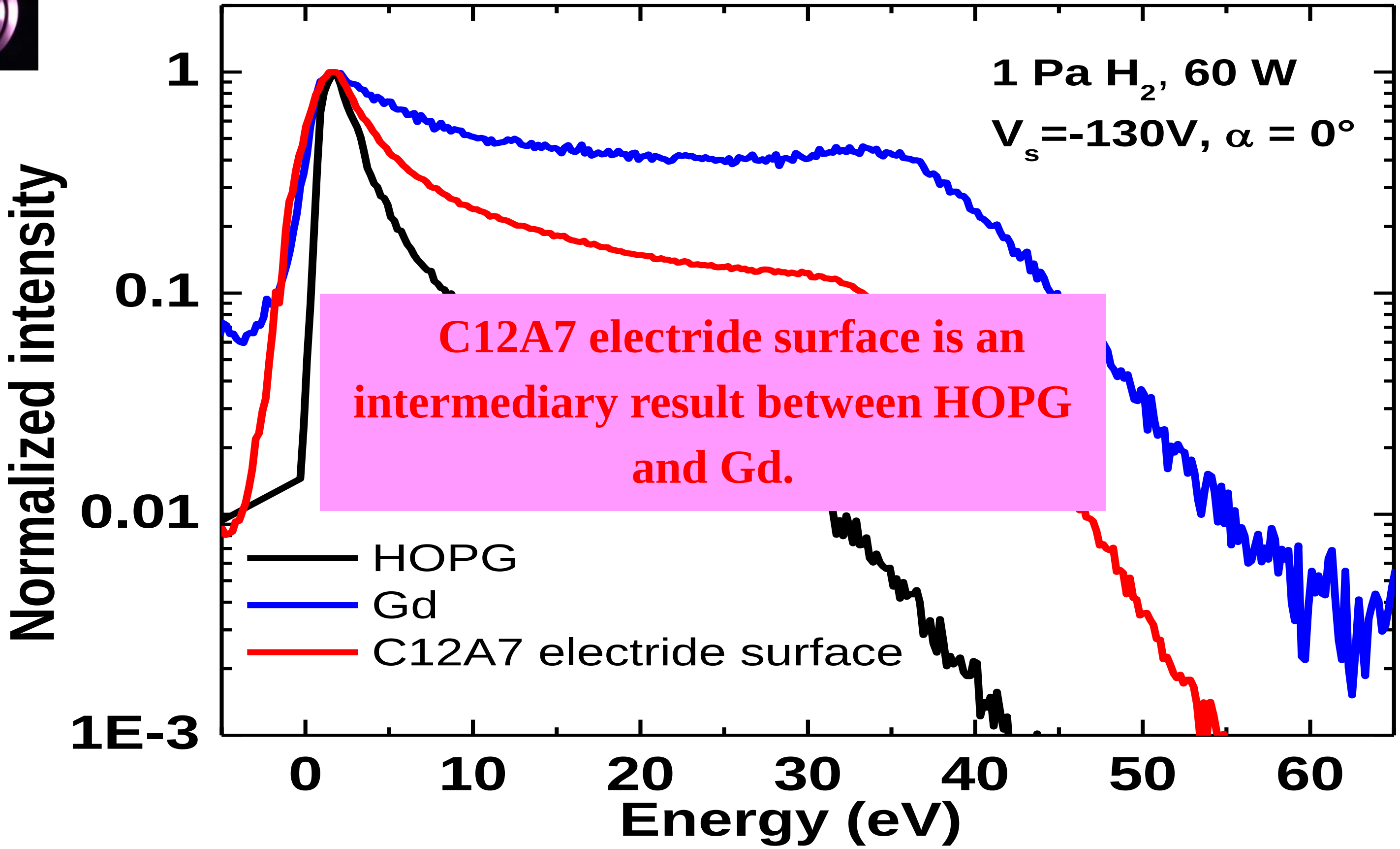
C12A7 the relative contribution of sputtering to NI production is higher than on Gd and lower than on HOPG



NI surface production mechanisms on C12A7 electride surface



C12A7 electride surface is an intermediary result between HOPG and Gd.

Gd the major process is backscattering of ions with a peak of production at 36 eV

HOPG the sputtering contribution due to adsorbed H on the surface is also important and there is a production peak at low energy

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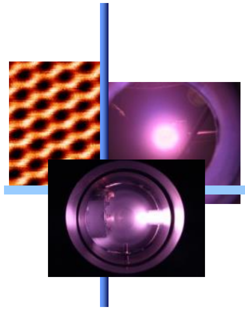
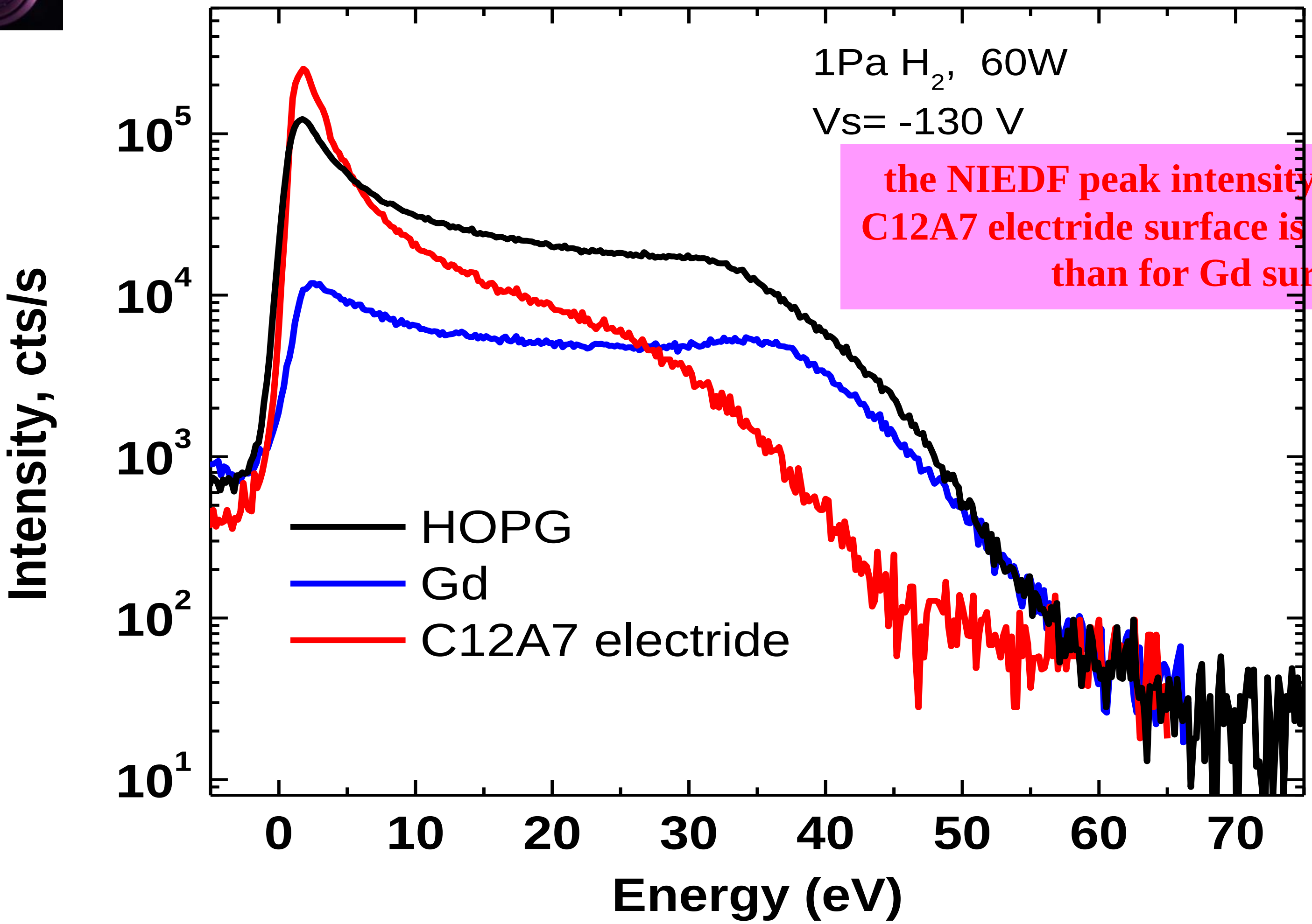
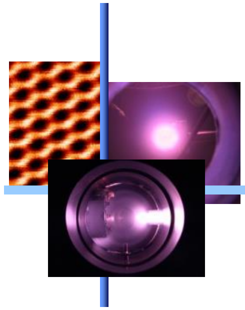


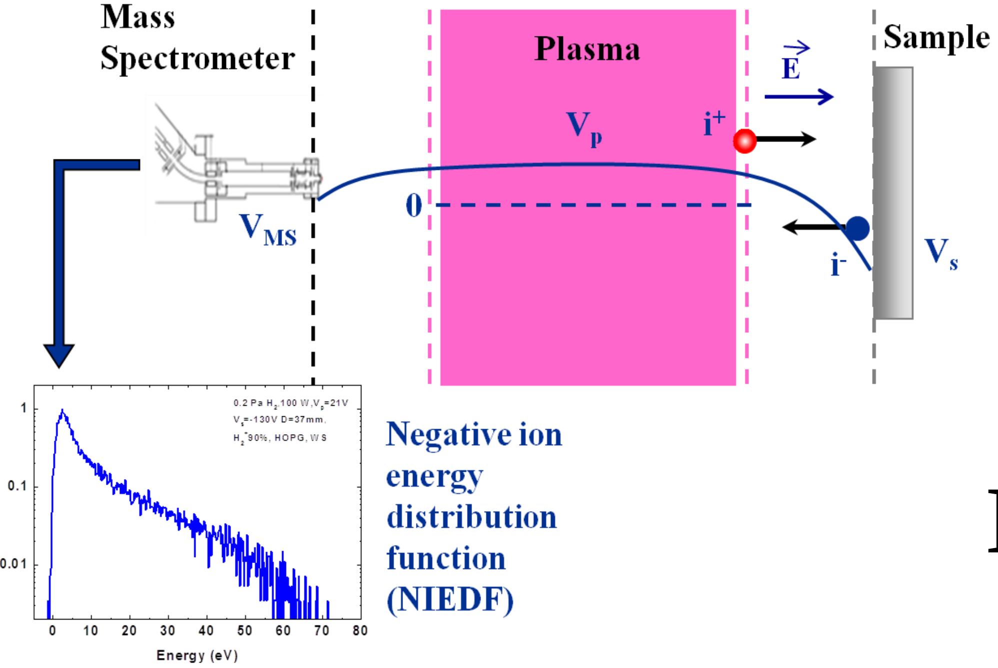
NI surface production efficiency on C12A7 electride surface



the NIEDF peak intensity measured for C12A7 electride surface is 21 times higher than for Gd surface

Effect of positive ion impact energy on NI production efficiency on C12A7 electride surface





Negative ion energy distribution function (NIEDF)

1 Pa H_2, 60 W ECR source

$V_p = 8$ V

H_3^+ is the dominant ion species

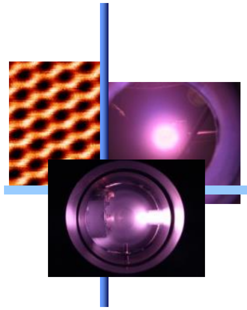
$$E_{H3^+} = e\,(V_s - V_p)$$

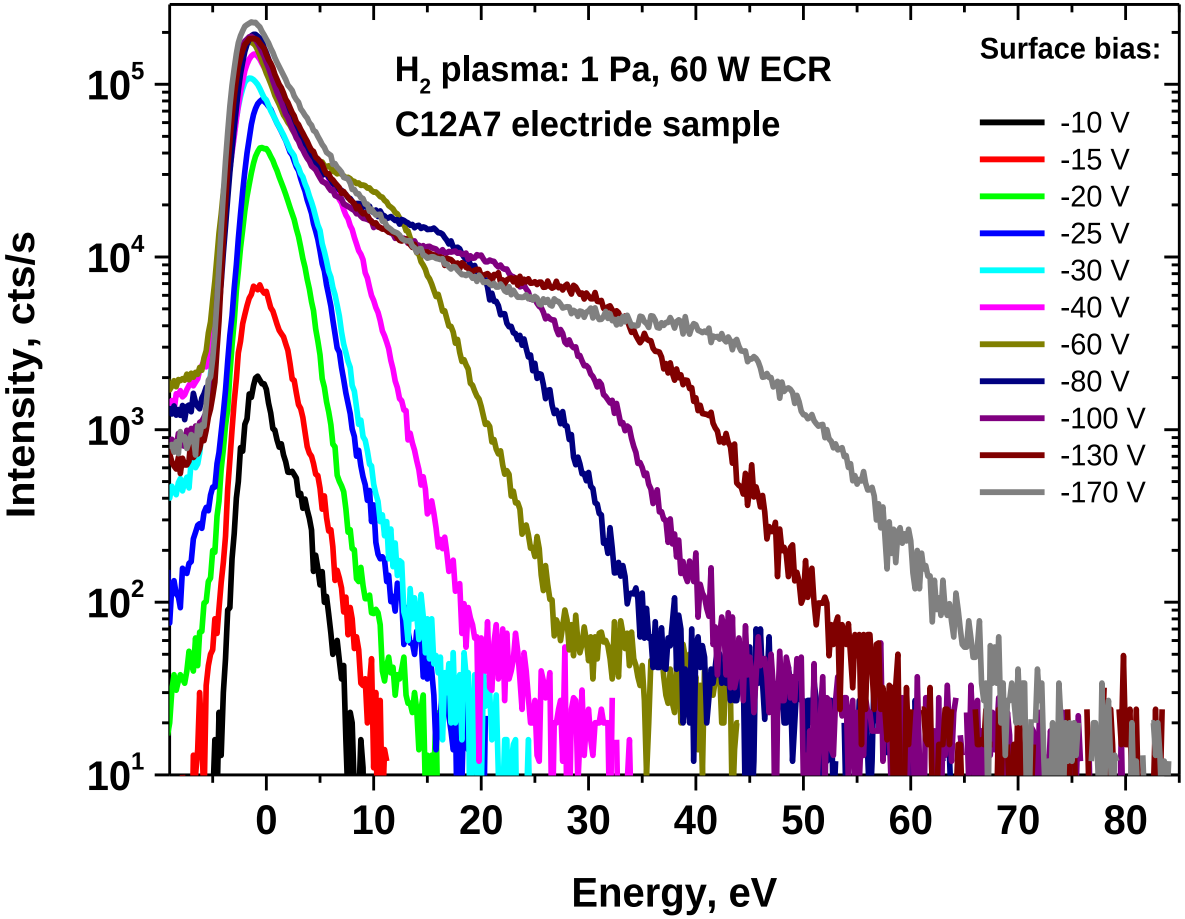
$$E_{impact} = e\,(V_s - V_p)/3$$

Vs is varied from -10 V to -170 V

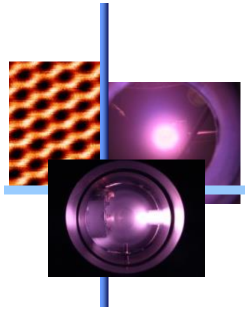
E_{impact} is varied from 6 eV to 60 eV

Effect of surface bias parameters on NI production efficiency on C12A7 electride surface

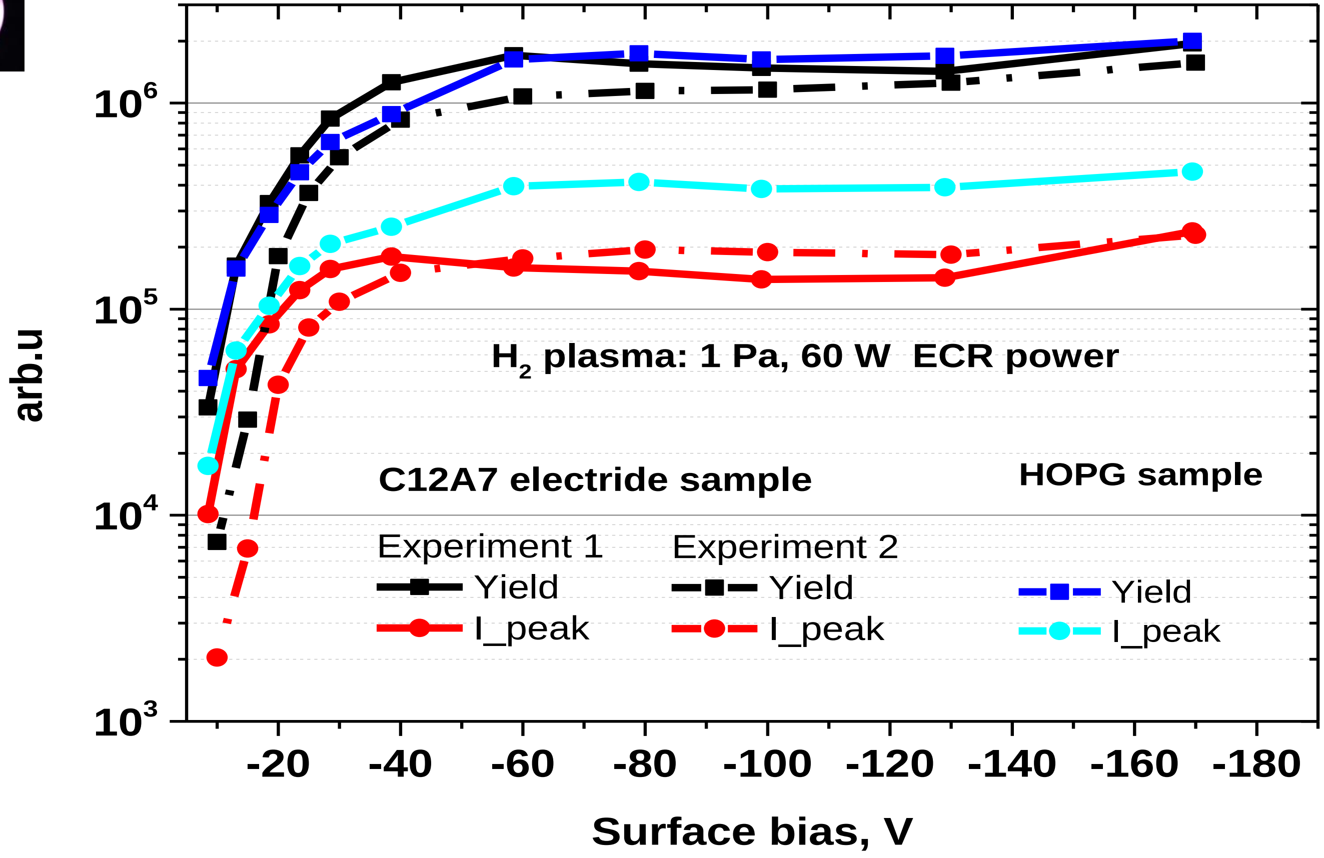




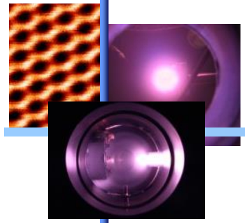
- **All measurements presented are performed at steady state**
- **The NIEDF peak intensity increase with bias (10^3 cts/s at -10 V to 2.10^5 cts/s at -170 V)**



Effect of surface bias parameters on NI production efficiency on C12A7 electride surface



- **The influence of surface bias variation on NI surface production in the case of C12A7 electride surface is similar to the case of HOPG surface**
- **The highest NIEDF peak intensity has been measured at -170 V for HOPG surface, same result is found for C12A7 electride surface where NIEDF peak intensity measured for C12A7 electride surface is 120 times higher than the one measured at -10 V.**



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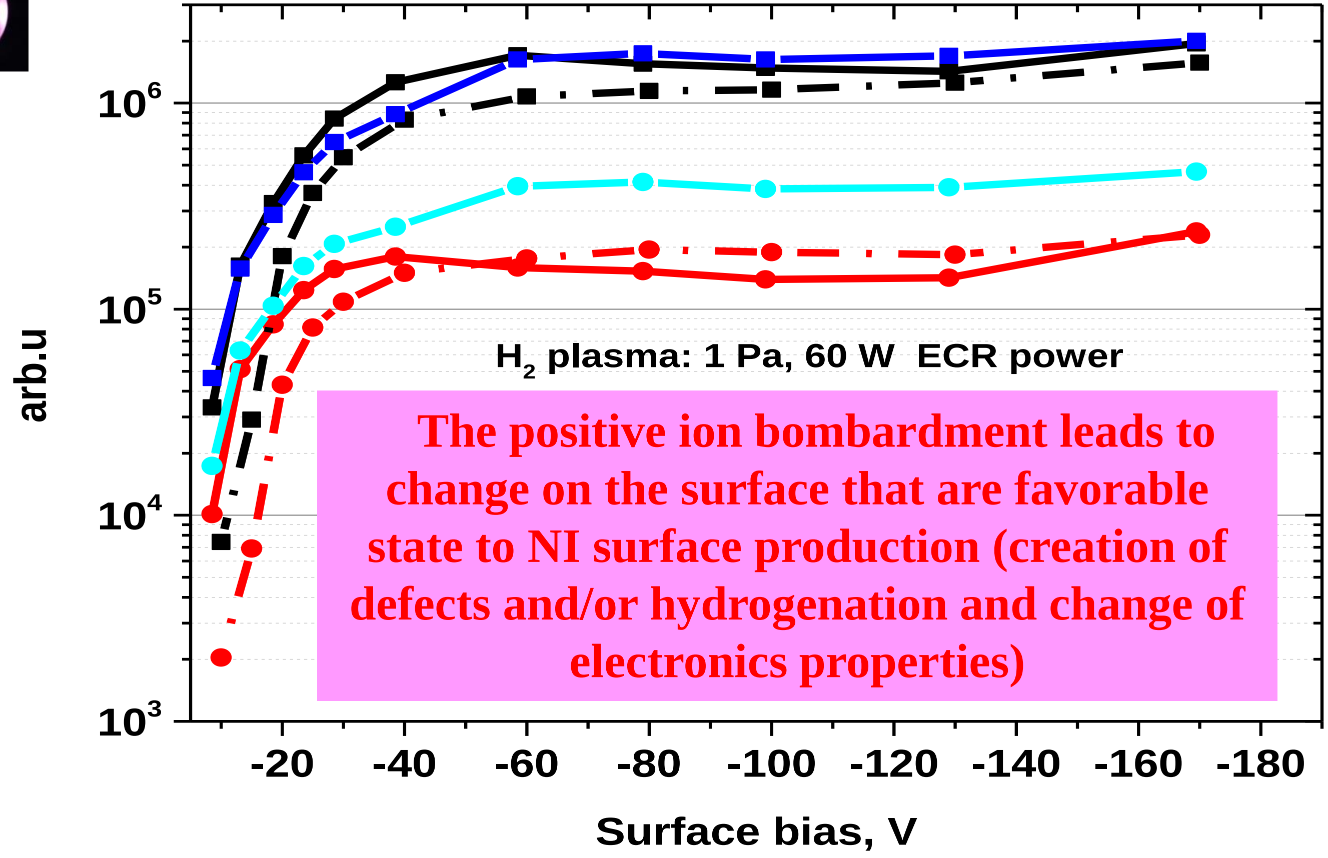
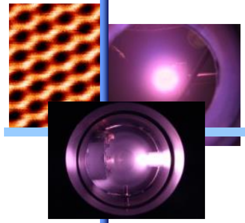


The positive ion bombardment leads to change on the surface that are favorable state to NI surface production (creation of defects and/or hydrogenation and change of electronics properties)

- The influence of surface bias variation on NI surface production in the case of C12A7 electride surface is similar to the case of HOPG surface
- The highest NIEDF peak intensity has been measured at -170 V for HOPG surface, same result is found for C12A7 electride surface where NIEDF peak intensity measured for C12A7 electride surface is 120 times higher than the one measured at -10 V.



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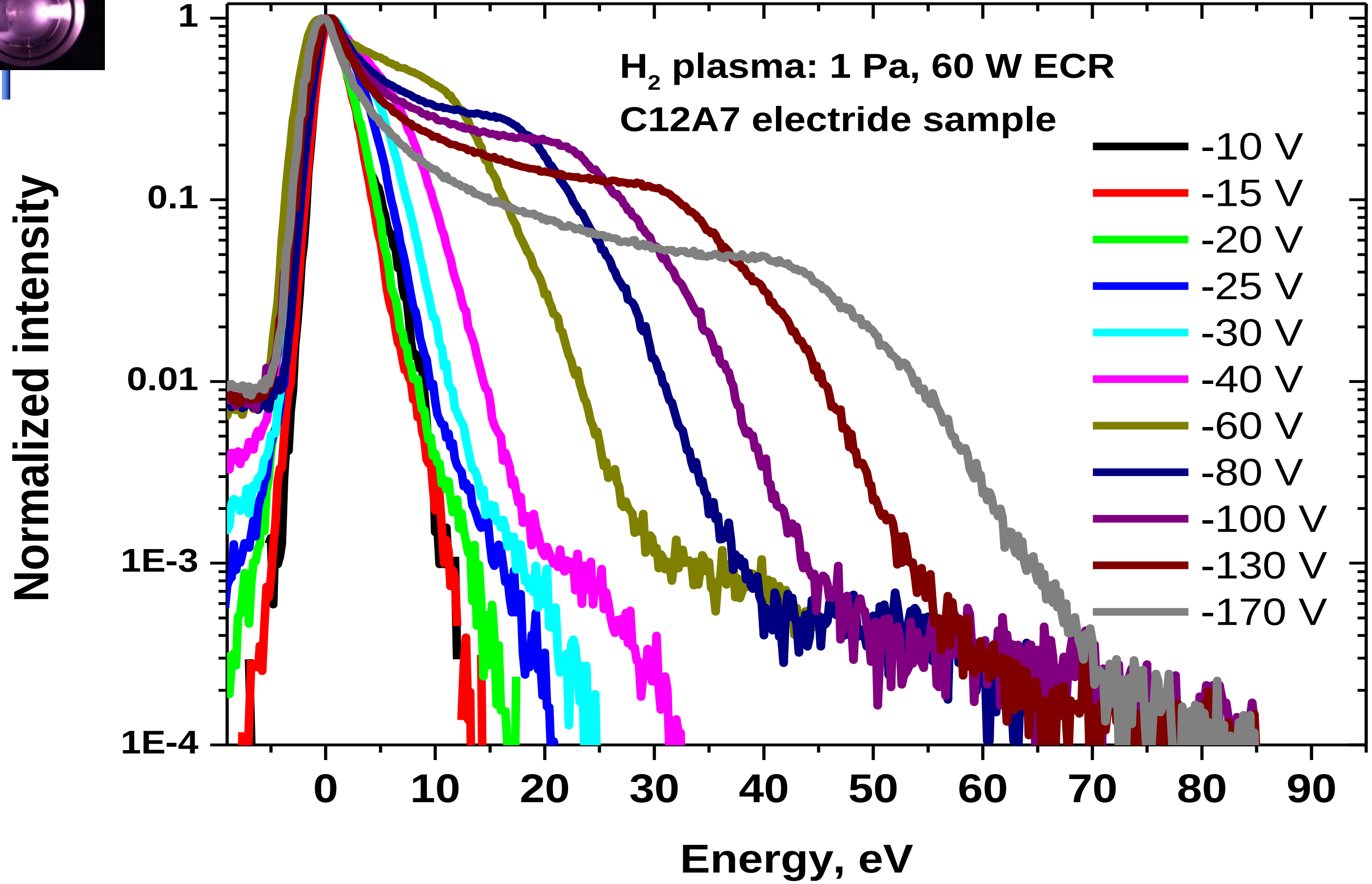
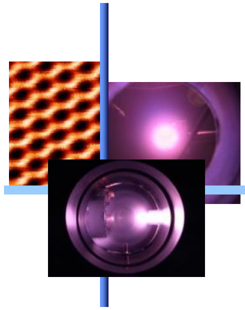


- The increase of the NIEDF tail means that mean energy of produced NI increases
- NI surface production on low work function materials is expected to increase with increasing perpendicular velocities of outgoing particles



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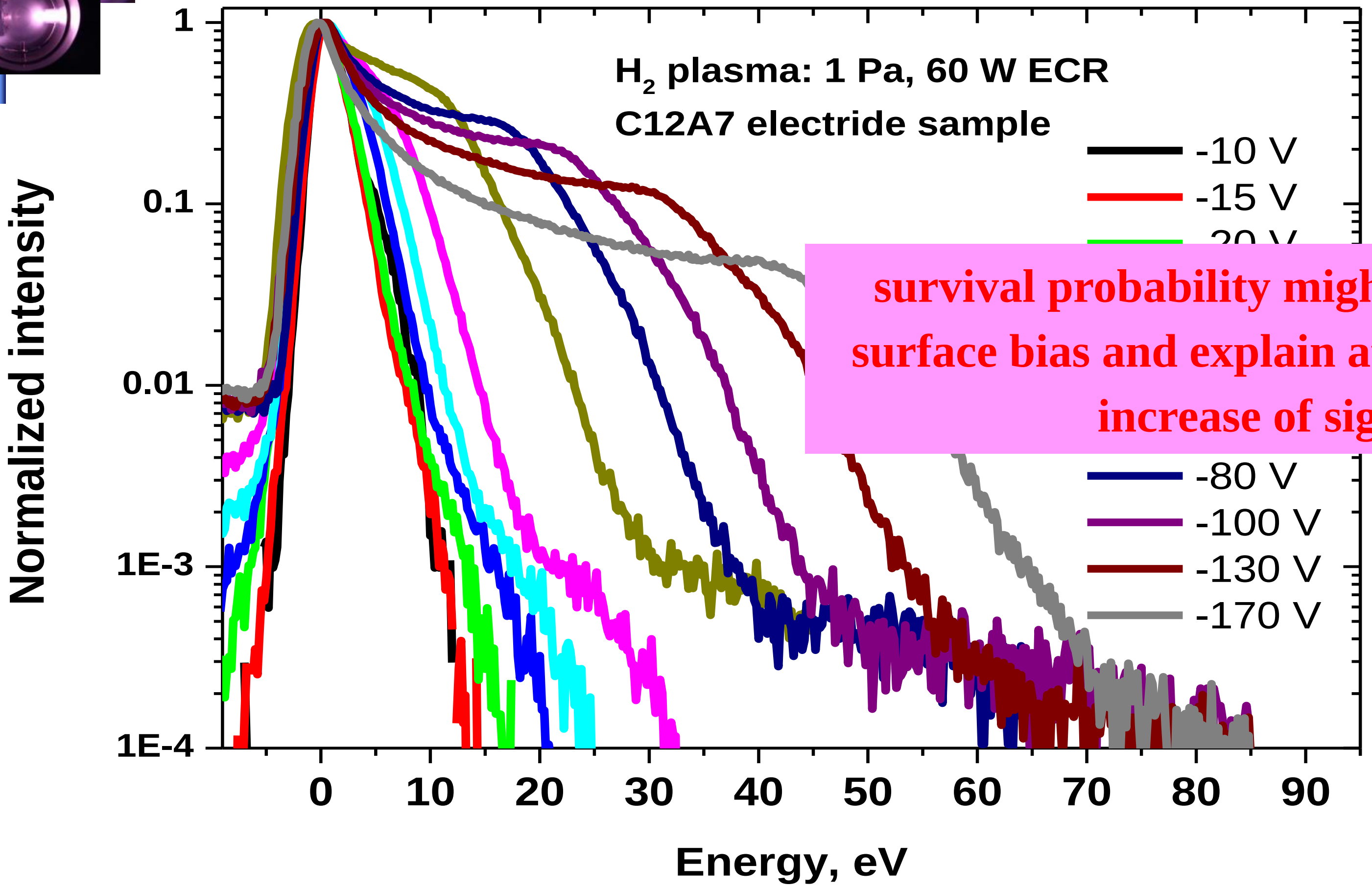

survival probability might increase with surface bias and explain at least partly the increase of signal.

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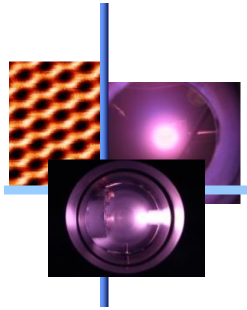

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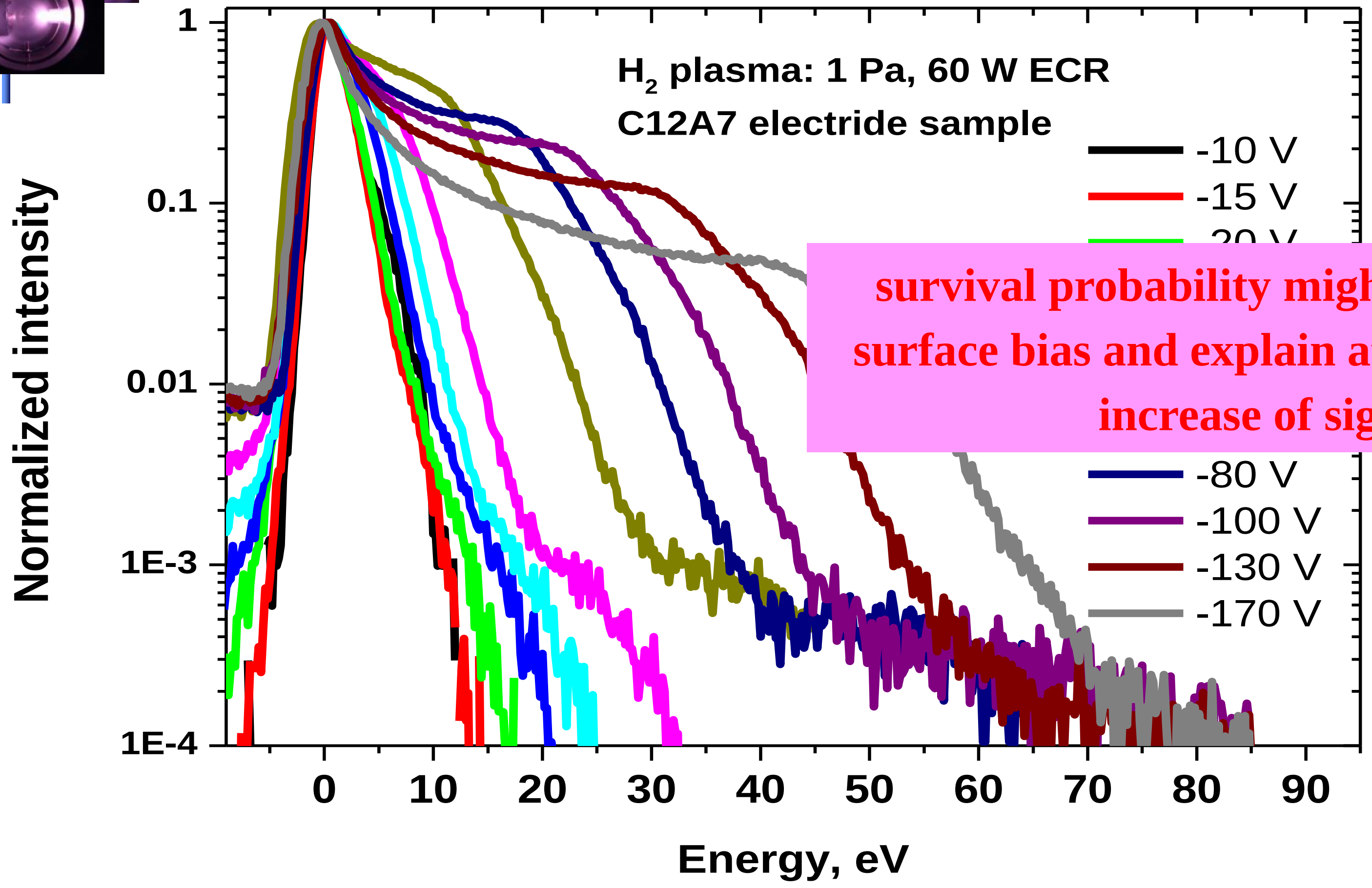

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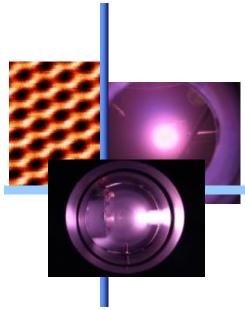
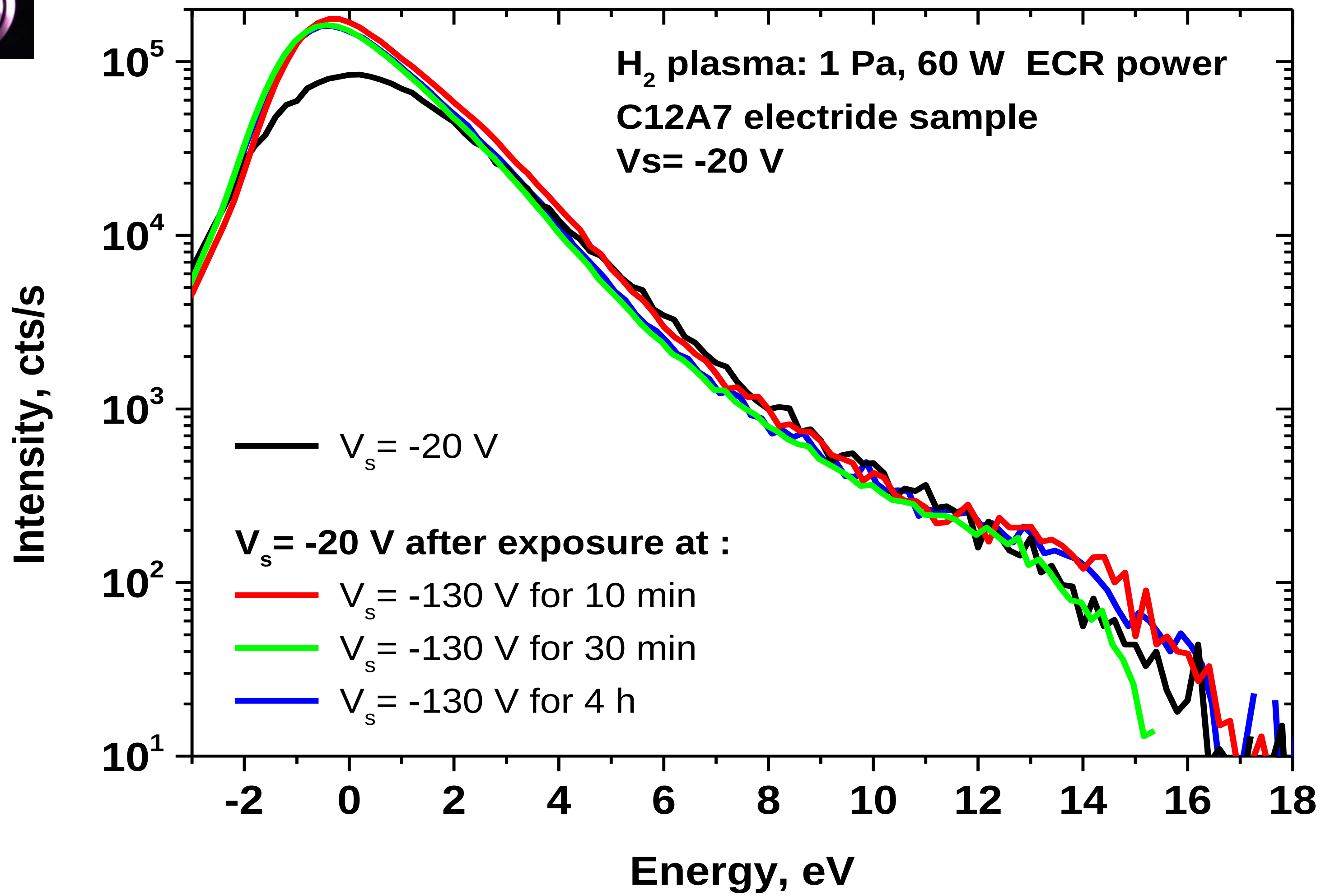
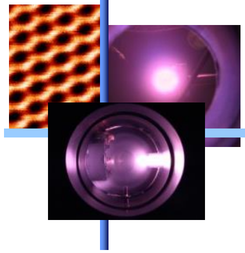
NIEDF measurements at low bias just after high bias exposure

Effect of surface bias parameters on NI production efficiency on C12A7 electride surface





- ✓ **The signal is increased by a factor 1.5 after 10 min of bombarding at -130 V**
- ✓ **10 min is enough to change surface properties and increase NI surface efficiency.**

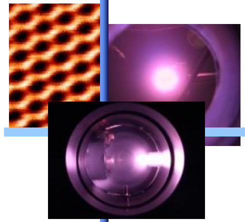


Effect of surface bias parameters on NI production efficiency on C12A7 electride surface

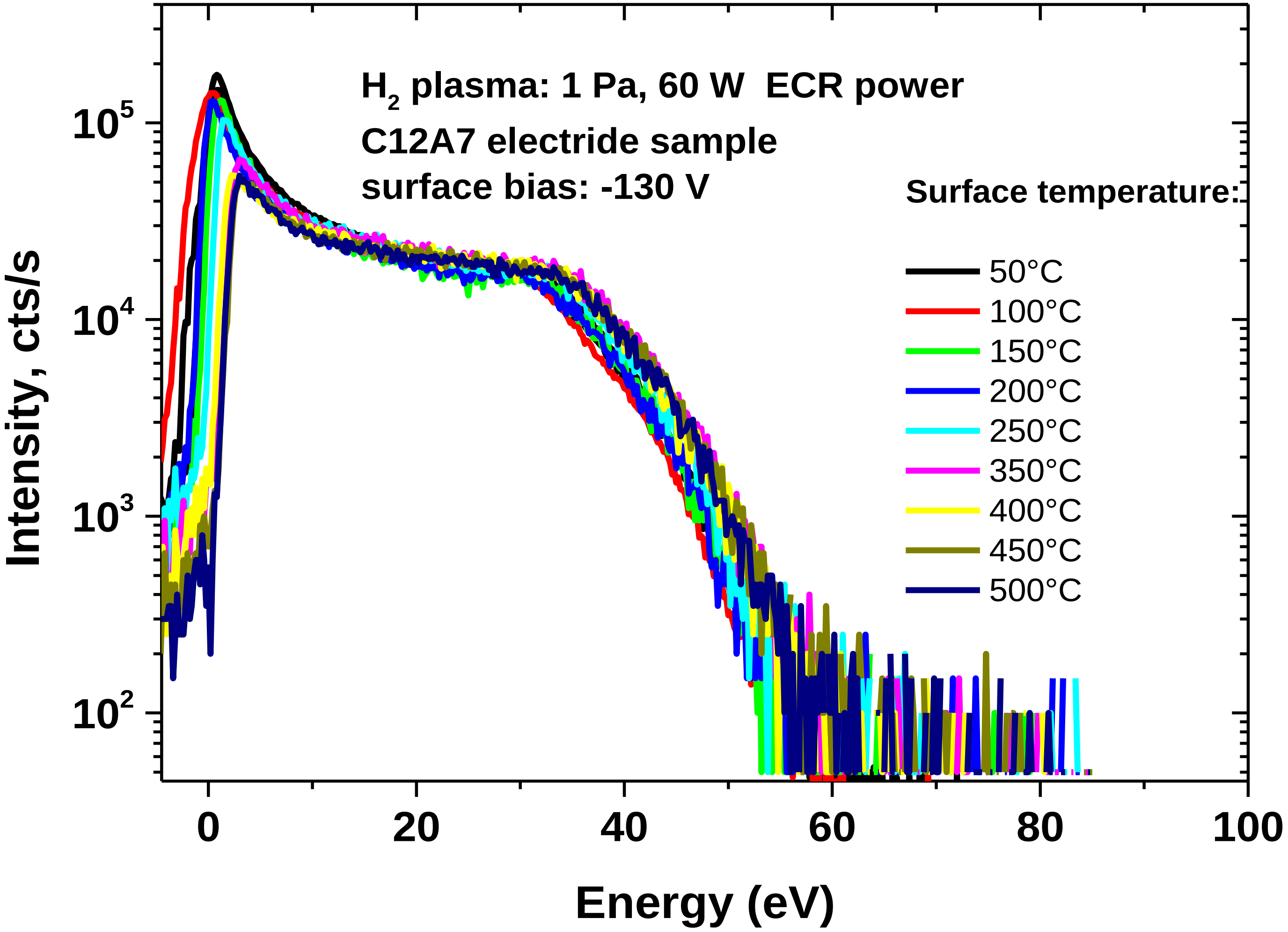
The increase of sputtering mechanism contribution to NI surface production can be related to the variation of hydrogen surface coverage

after a high energy bombardment we obtain a C12A7 electride surface with a higher hydrogen coverage than the pristine one.

heating experiment

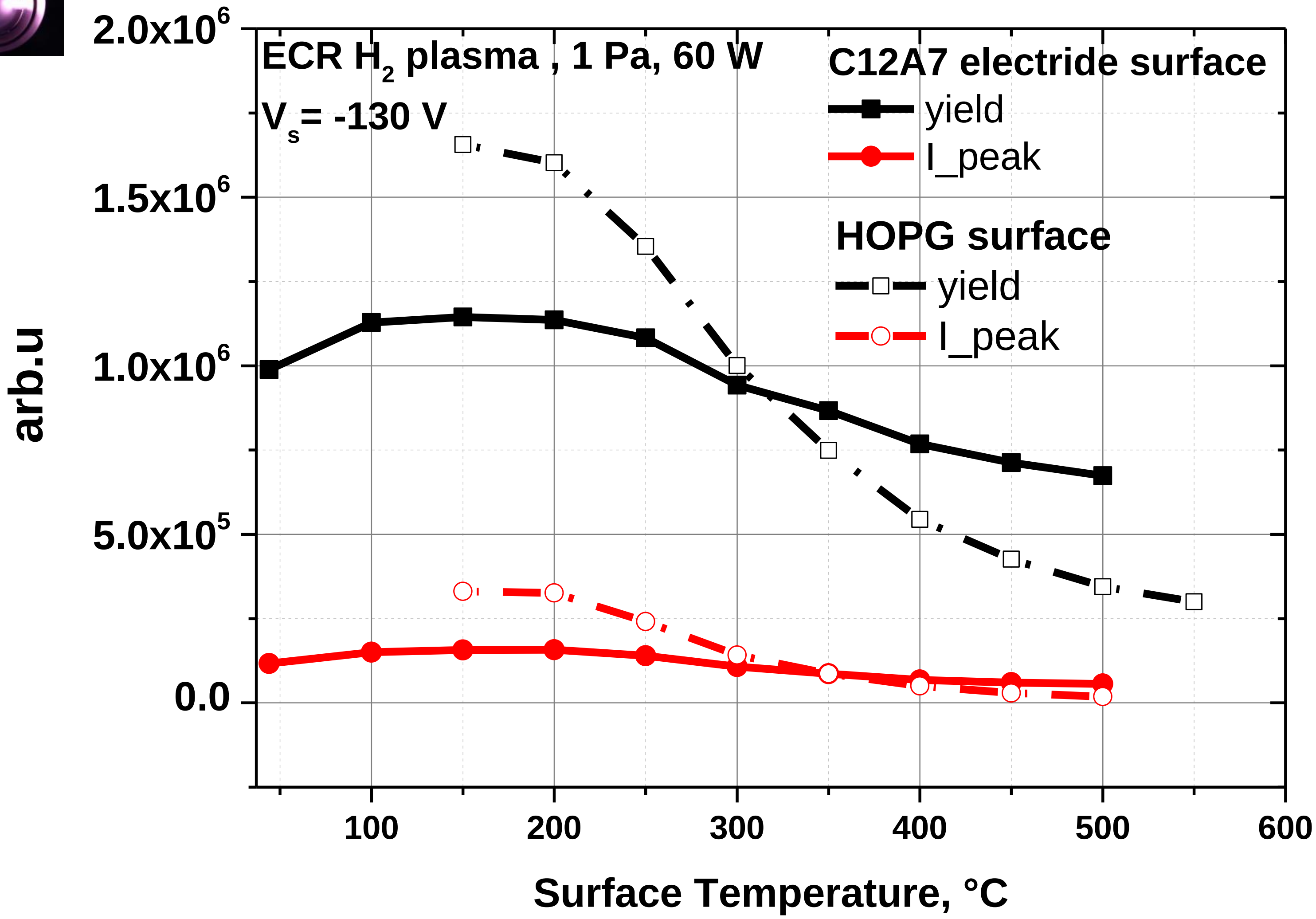


Effect of surface temperature on NI production efficiency on C12A7 electride surface

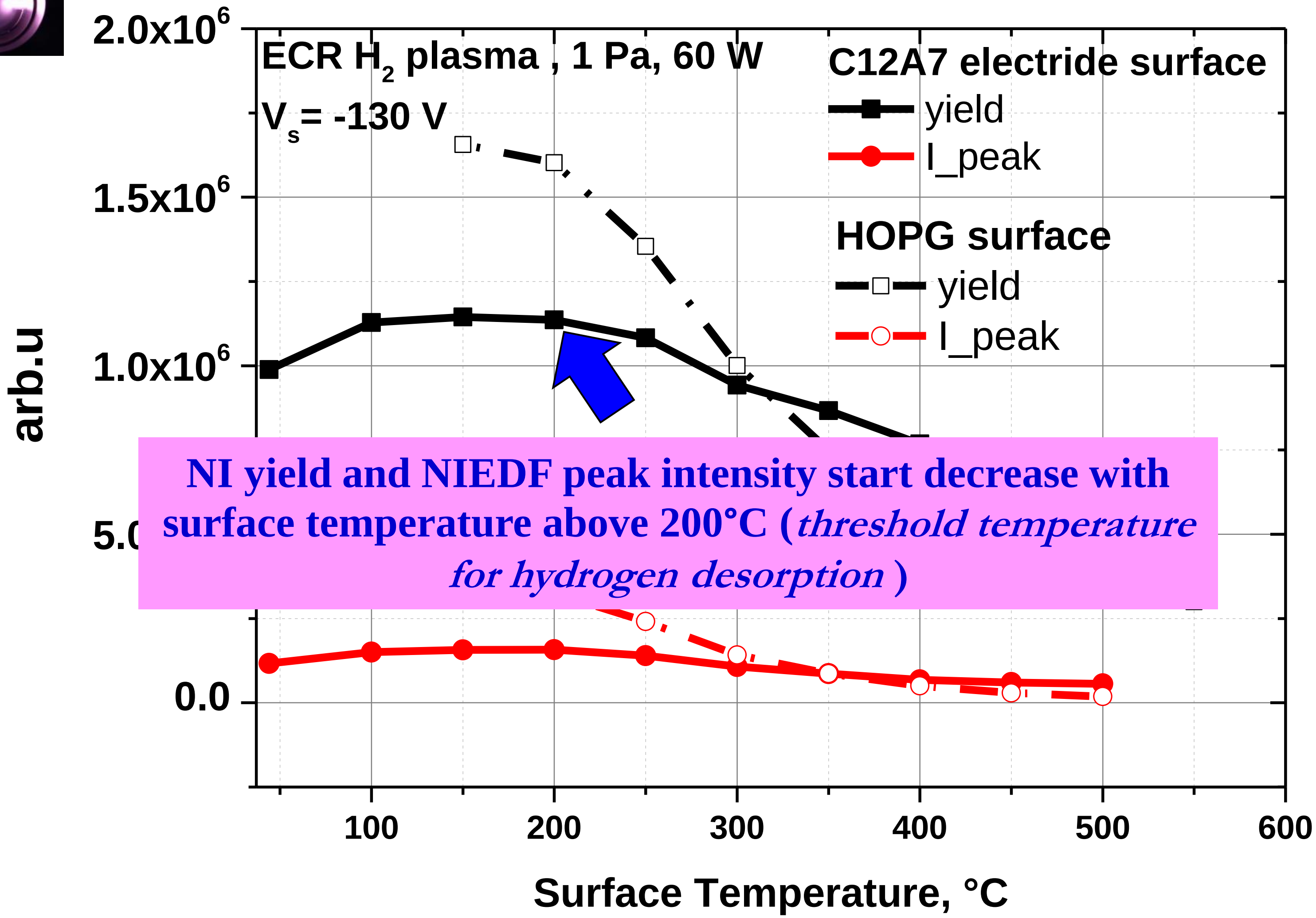


Temperature is scanned from 50° to 500°C

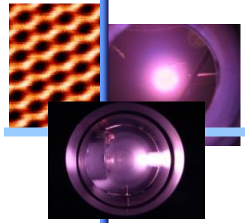
Effect of surface temperature on NI production efficiency on C12A7 electride surface



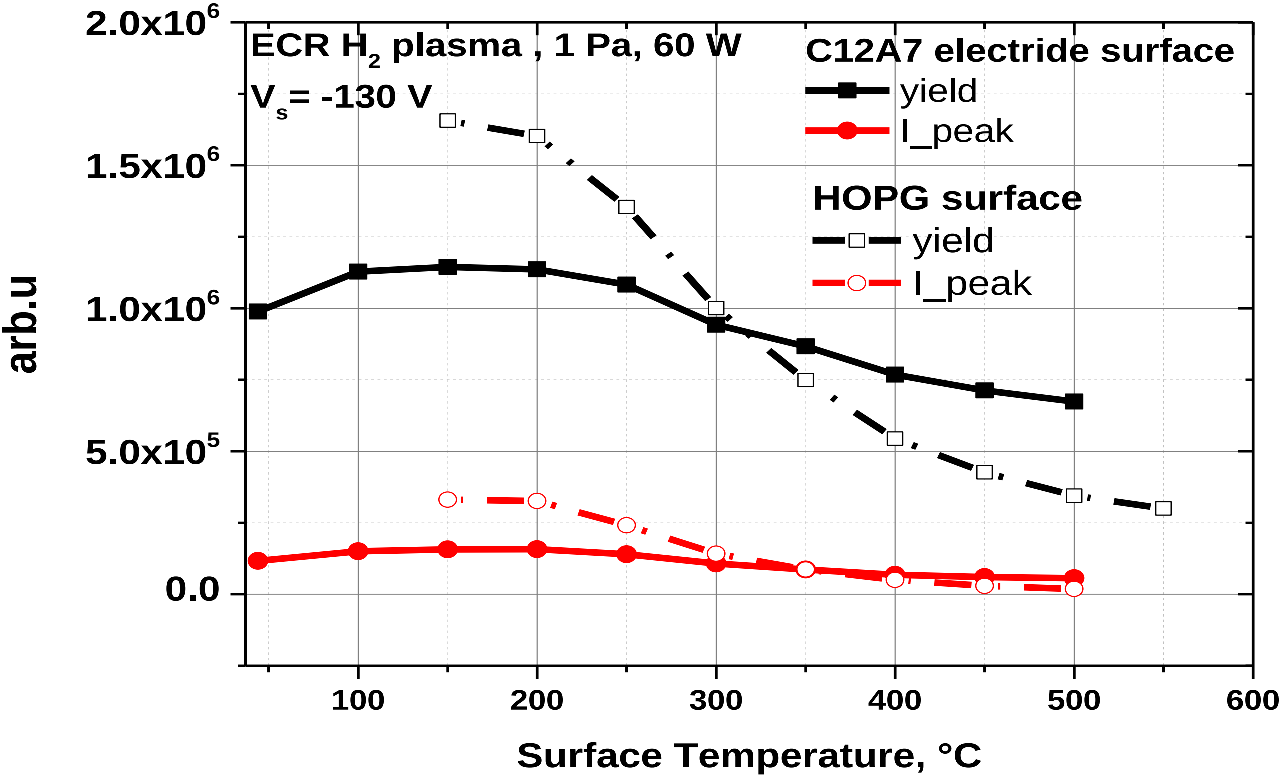
Effect of surface temperature on NI production efficiency on C12A7 electride surface



NI yield and NIEDF peak intensity start decrease with surface temperature above 200°C (*threshold temperature for hydrogen desorption*)



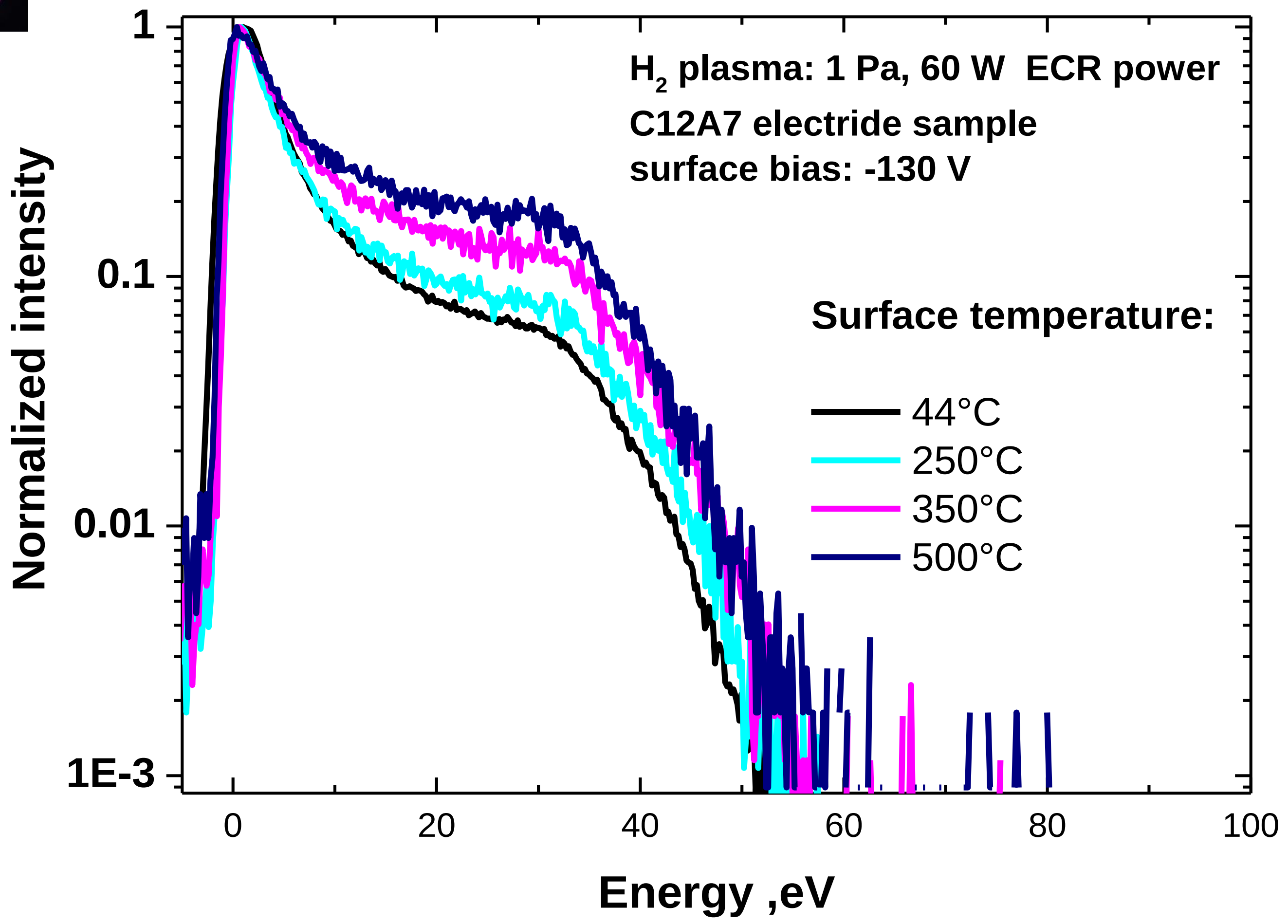
Effect of surface temperature on NI production efficiency on C12A7 electride surface



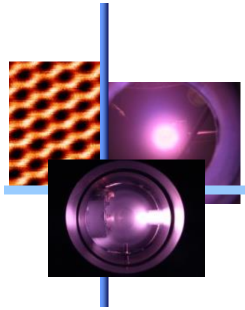
C12A7 electride surface behave in a very similar way to NI surface production on HOPG when increasing surface temperature

significant decrease of NIEDF signal at 300°C is probably caused by the decrease of the amount of hydrogen on the surface

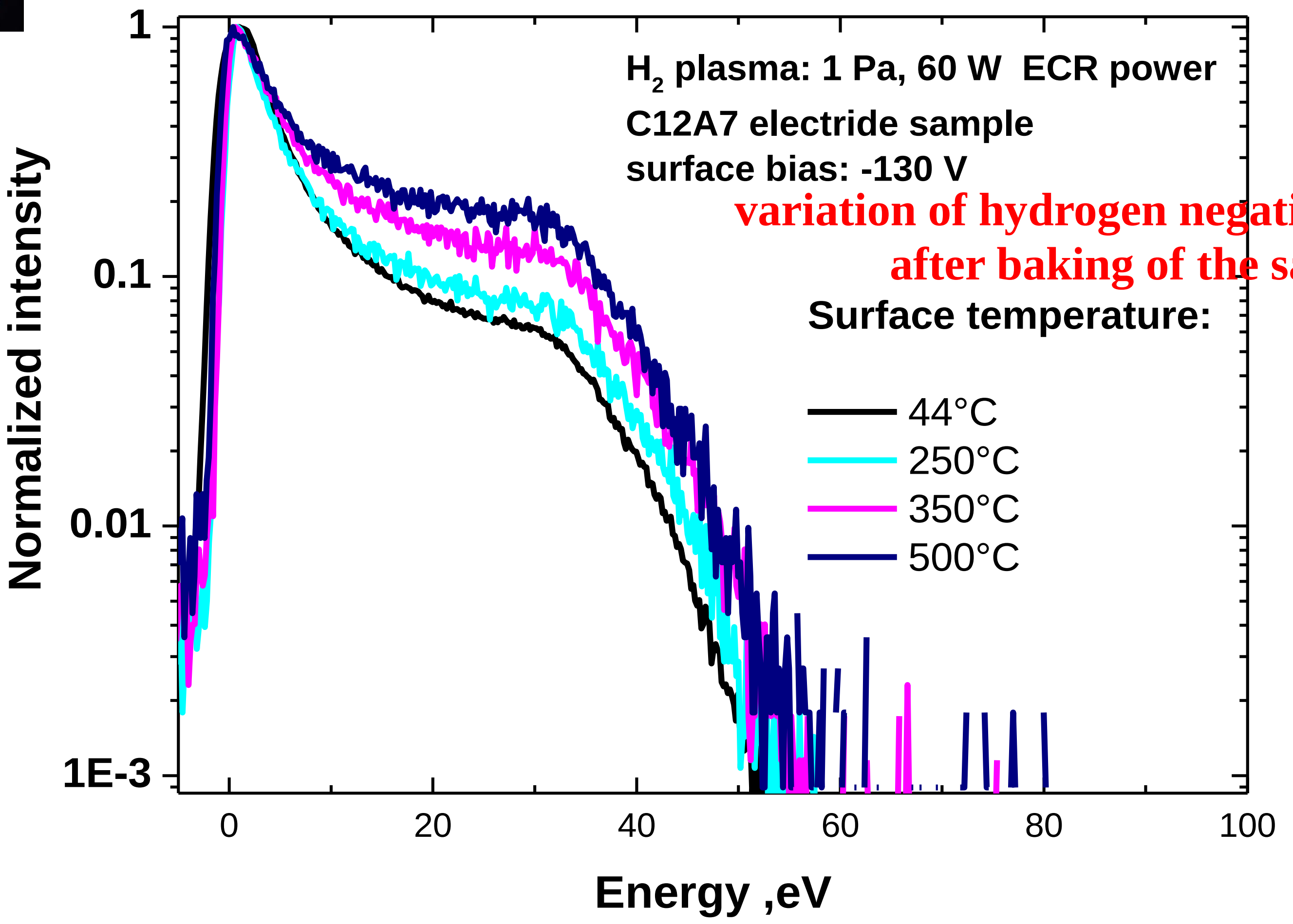
Effect of surface temperature on NI production efficiency on C12A7 electride surface



The normalized NIEDF measured at 500°C has a larger tail than the measured one at 44°C indicating a decrease of sputtering contribution



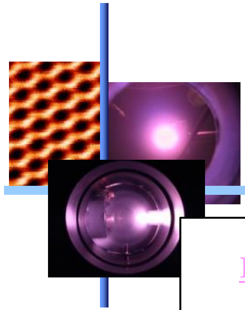
Effect of surface temperature on NI production efficiency on C12A7 electride surface



variation of hydrogen negative ion signals after baking of the sample

The normalized NIEDF measured at 500°C has a larger tail than the measured one at 44°C indicating a decrease of sputtering contribution

Baking experiment



H_2 plasma bombardment

- ECR plasma (1 Pa H2, 60 W)
- Vs= -130 V
- 2 hours

Baking in vacuum

- 10^{-7} mbar
- 520°C
- 50 min

H_2 plasma

- ECR plasma (1 Pa H2, 60 W)
- Vs= -20 V

Time evolution

H_2 plasma bombardment

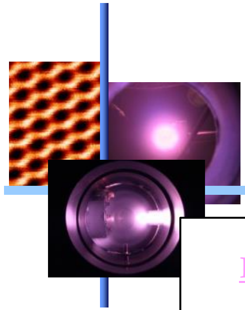
- ECR plasma (1 Pa H2, 60 W)
- Vs= -130 V
- 2 hours

No Baking in vacuum

H_2 plasma

- ECR plasma (1 Pa H2, 60 W)
- Vs= -20 V

Time evolution



Baking experiment

H_2 plasma bombardment

☐ ECR plasma (1 Pa H2, 60 W)
☐ Vs= -130 V
☐ 2 hours

Baking in vacuum

☐ 10^{-7} mbar
☐ 520°C
☐ 50 min

Amount of H_2O increase 24 times
Amount of H increase 2 times

H_2 plasma

☐ ECR plasma (1 Pa H2, 60 W)
☐ Vs= -20 V

Time evolution

H_2 plasma bombardment

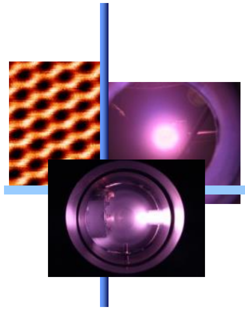
☐ ECR plasma (1 Pa H2, 60 W)
☐ Vs= -130 V
☐ 2 hours

No Baking in vacuum

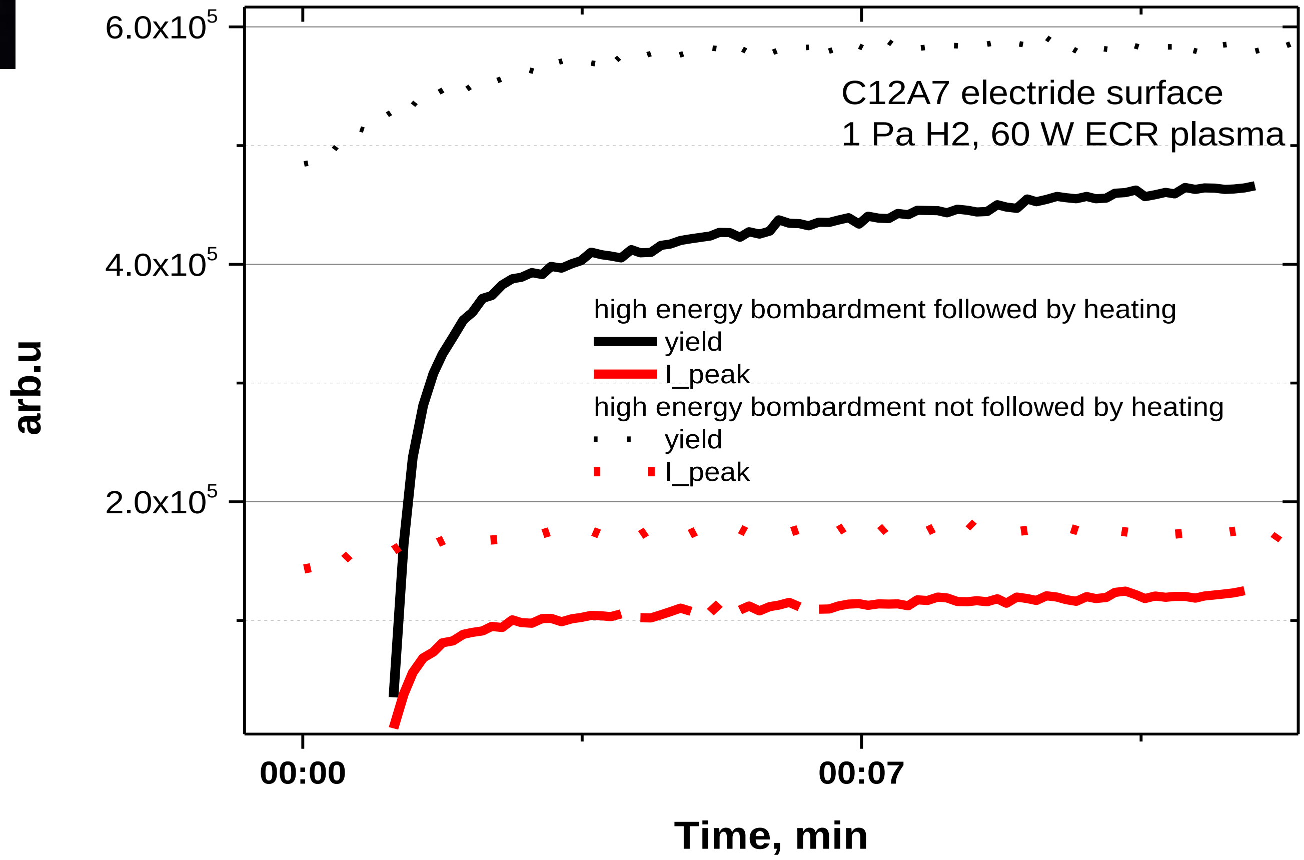
H_2 plasma

☐ ECR plasma (1 Pa H2, 60 W)
☐ Vs= -20 V

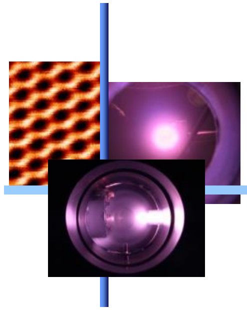
Time evolution



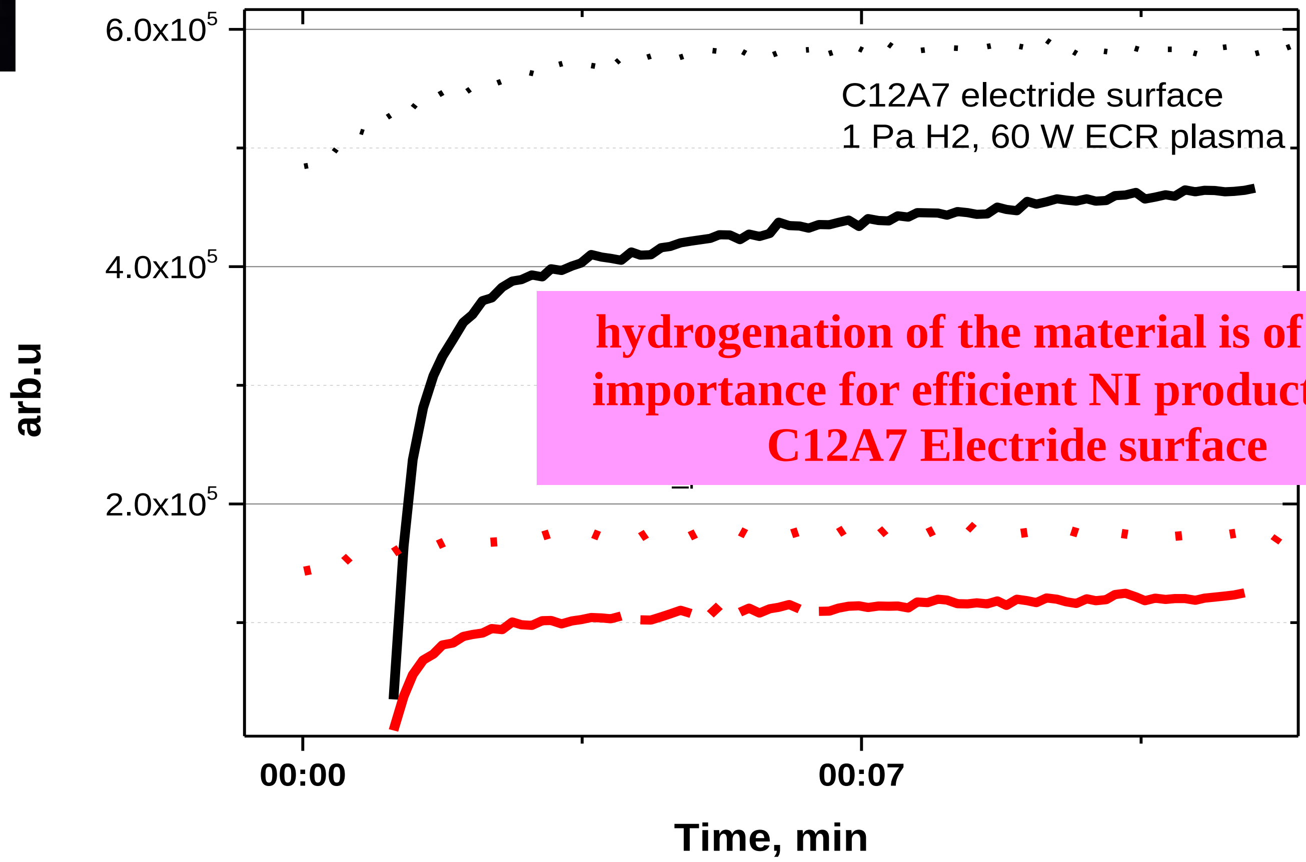
Baking experiment



- **Followed by heating: negative ion peak intensity increased by a factor of 11.7 the first 5 min, and it increased then slowly.**
- **Not followed by heating: the peak intensity increases only by a factor 1.7**

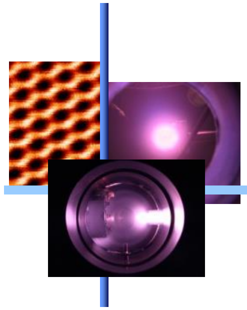


Baking experiment

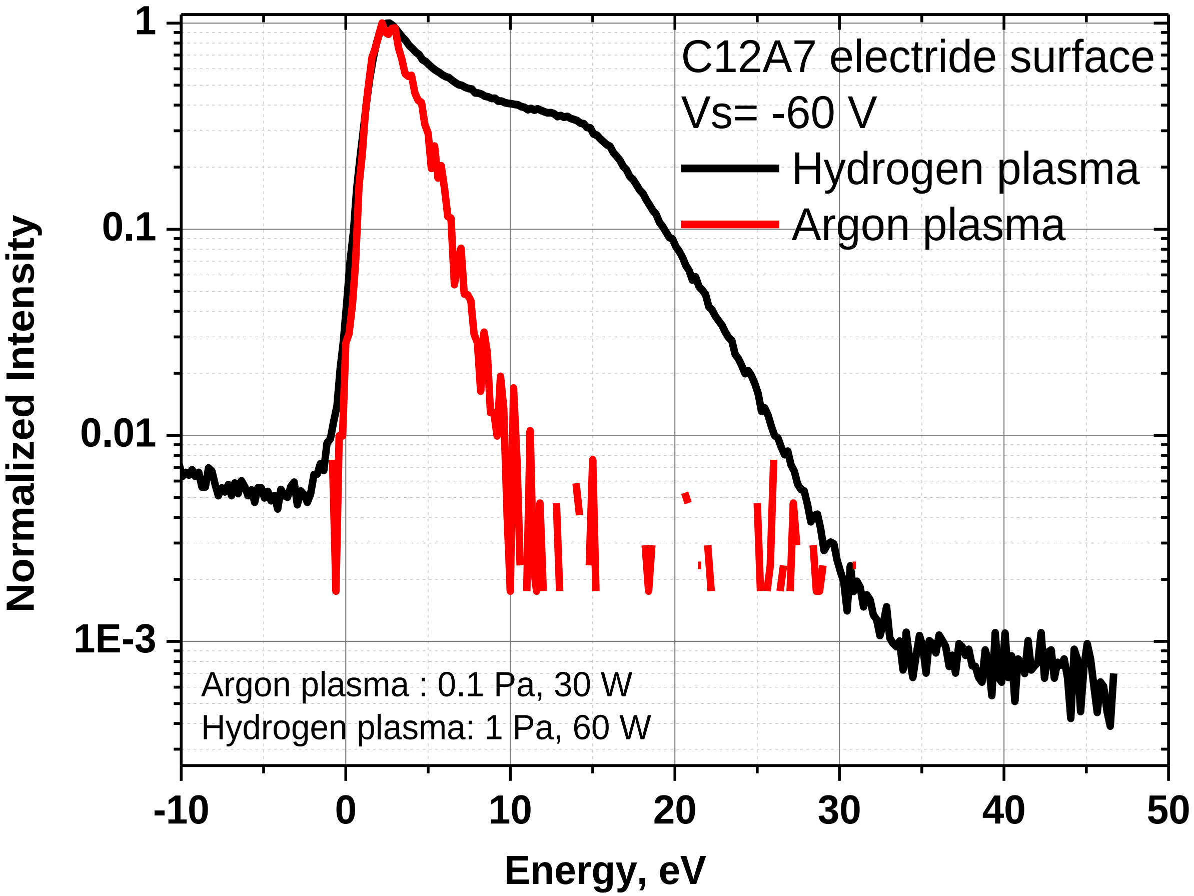


hydrogenation of the material is of major importance for efficient NI production on C12A7 Electride surface

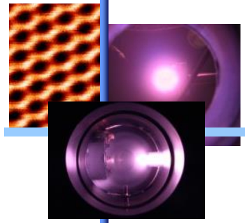
- **Followed by heating: negative ion peak intensity increased by a factor of 11.7 the first 5 min, and it increased then slowly.**
- **Not followed by heating: the peak intensity increases only by a factor 1.7**



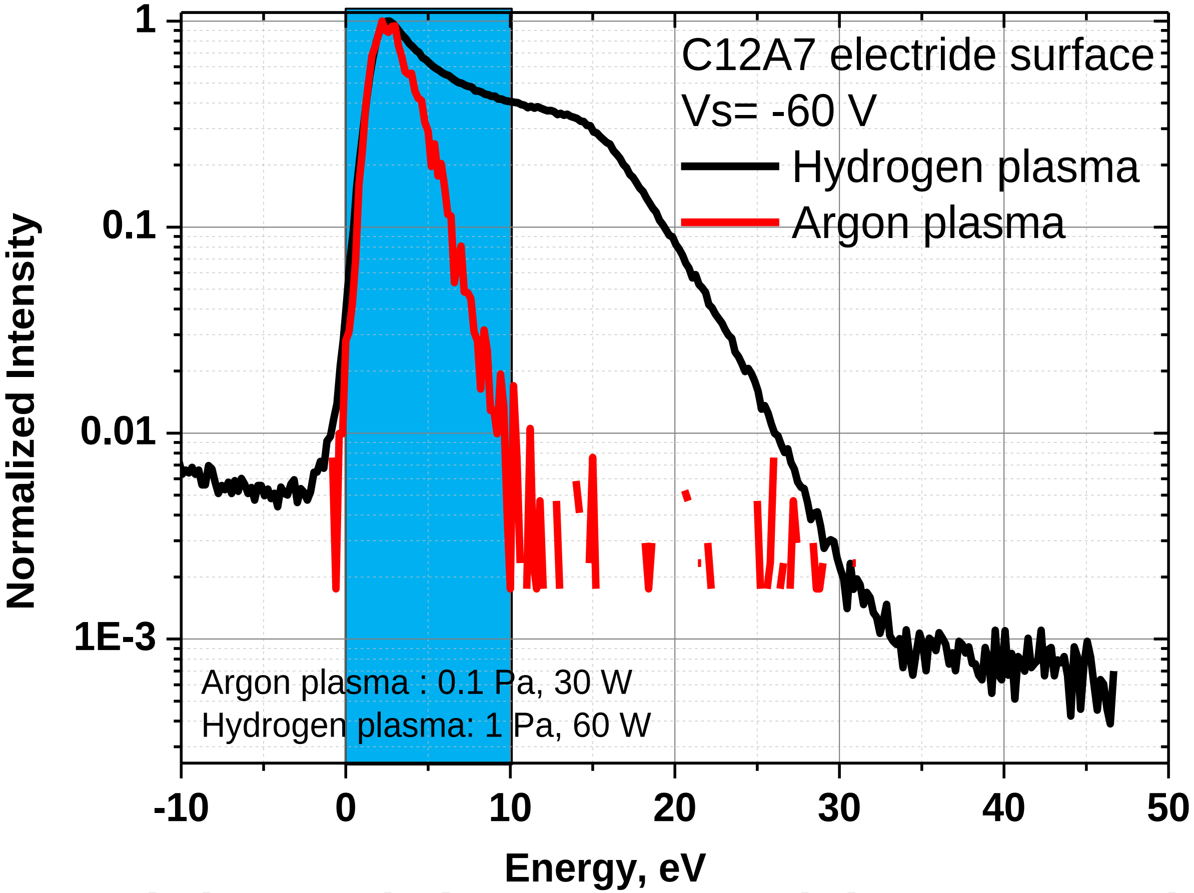
Importance of sputtering mechanism in NI surface production on C12A7 electride surface



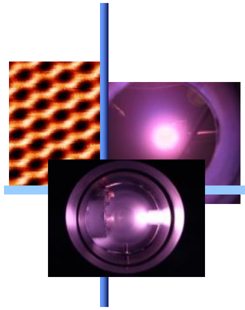
- Normalized energy spectra measured at steady state of hydrogen negative ions from the C12A7 electride bombarded by hydrogen and by argon ions are compared
- In both measurements, the target was pre-treated with an exposure to hydrogen ECR plasma (1 Pa H_2, 60 W) at Vs= -130V for 10 min.



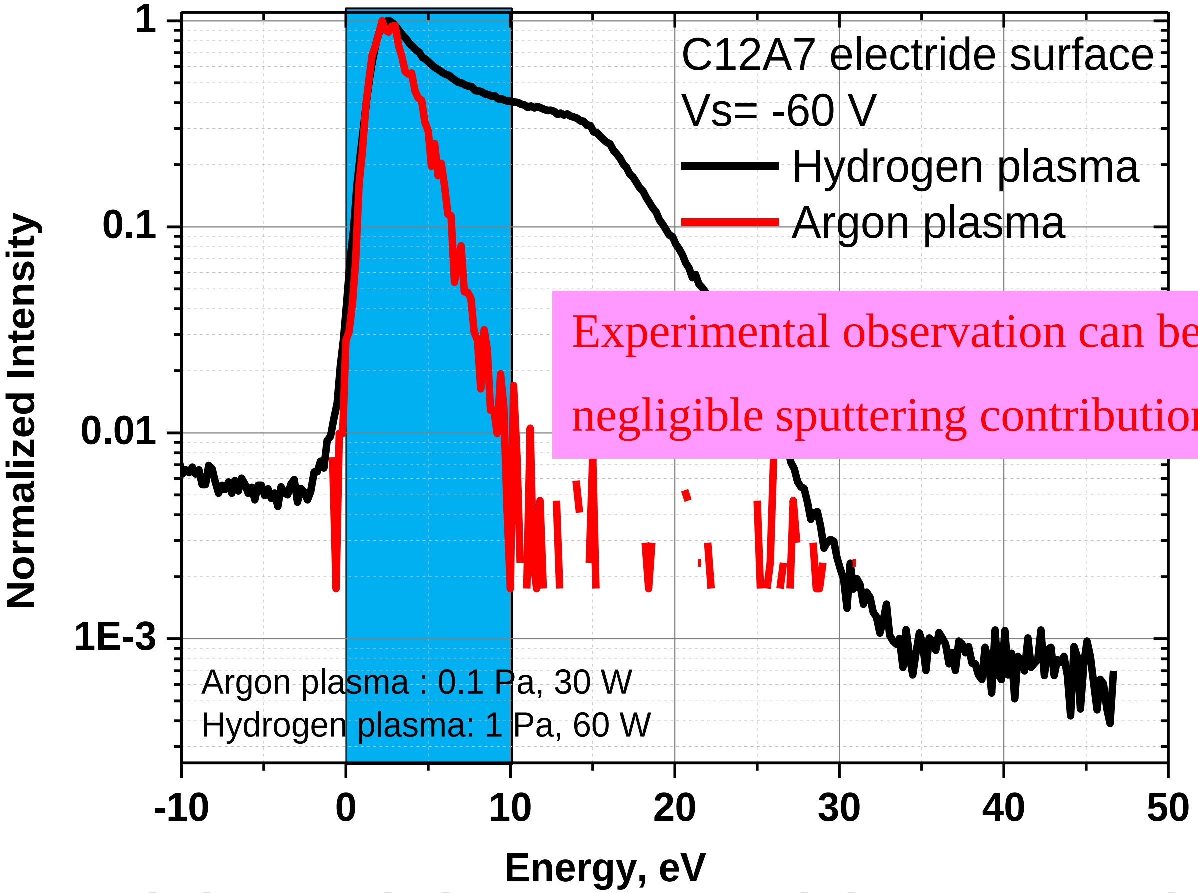
Importance of sputtering mechanism in NI surface production on C12A7 electride surface



- A dominant peak shown in the low energy region below 10 eV was observed for different surface bias.
- The sputtering of pre-implanted hydrogen particles in C12A7 electride surface can be the origin of this dominant peak below 10 eV.



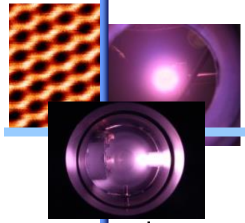
Importance of sputtering mechanism in NI surface production on C12A7 electride surface

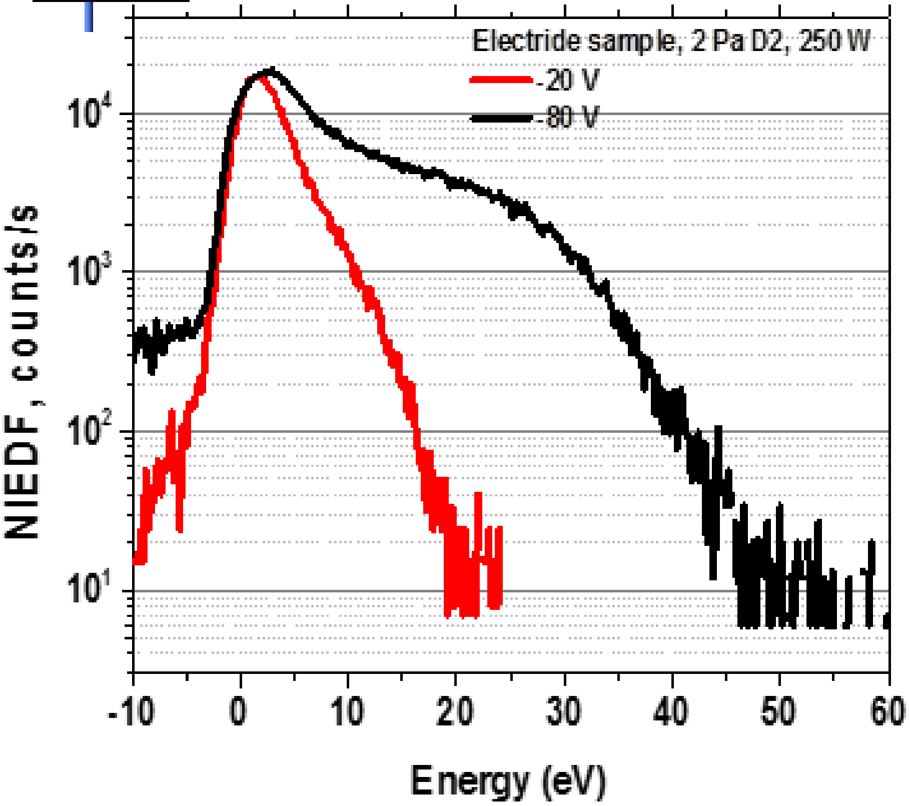


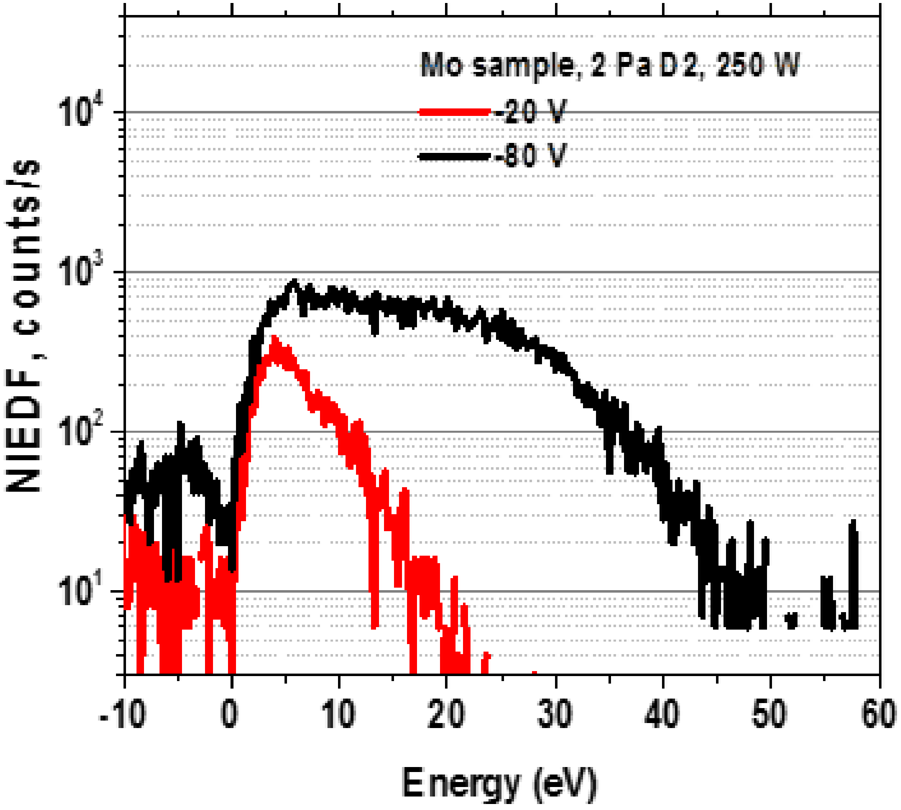
Experimental observation can be a sign of non-negligible sputtering contribution at low bias

- A dominant peak shown in the low energy region below 10 eV was observed for different surface bias.
- The sputtering of pre-implanted hydrogen particles in C12A7 electride surface can be the origin of this dominant peak below 10 eV.

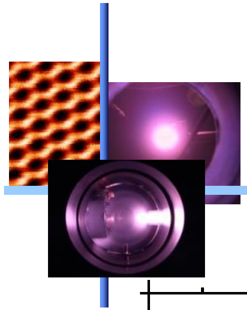
Potential of using C12A7 electride surface in non cesiated NI source



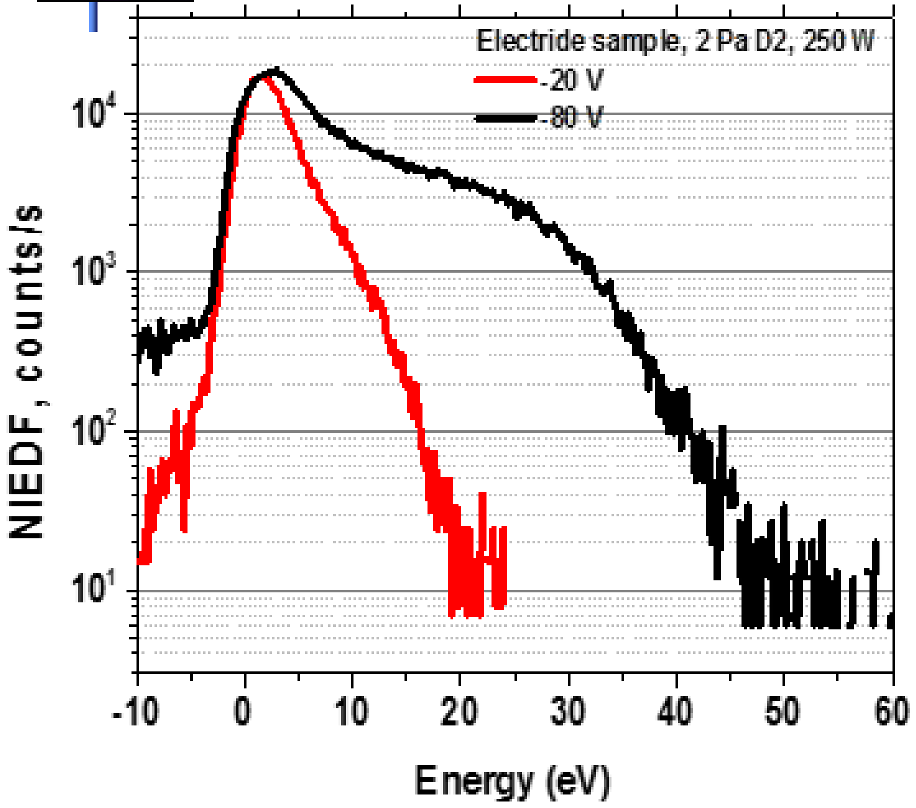


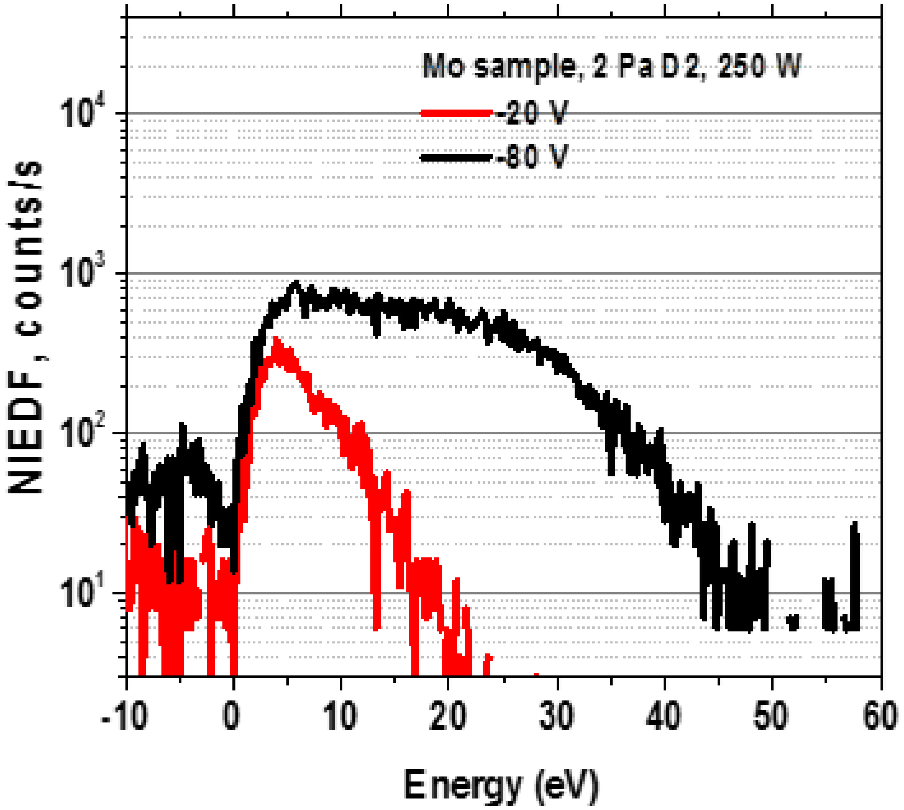


The ratio of the total H^- yield from a C12A7 electride surface bombarded at -80 V to that from a clean molybdenum surface is approximately 10. This ratio increases to 50 at bias voltage Vs= -20 V. This value is consistent to previous indirect measurement done in Doshisha University

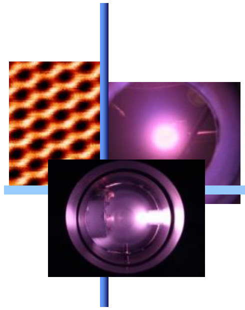


Potential of using C12A7 electride surface in non cesiated NI source





In this experiment the electric current due to negative hydrogen ions leaving the C12A7 surface upon the exposure to atomic hydrogen (H^0) flux was measured. This current was at a similar level to that obtained from a low work function bi alkali covered molybdenum surface.



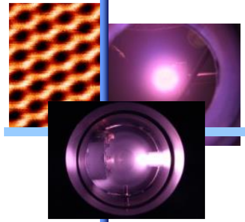
Conclusion

High NI yield production
low work function of this material
Specific nano structure of electride material

C12A7 electride is air-stable, mechanically robust and machinable, and has potential to be used as production surface of cesium-free negative ion sources

Negative Hydrogen Ion Production from a Nanoporous 12CaO・7Al2O3 (C12A7) Electride Surface

M. Sasao,[1,] R. Moussaoui,[2] D. Kogut,[2] J. Ellis,[3] G. Cartry,[2] M. Wada,[4] K. Tsumori,[5] and H. Hosono[6]



Thank you for your attention