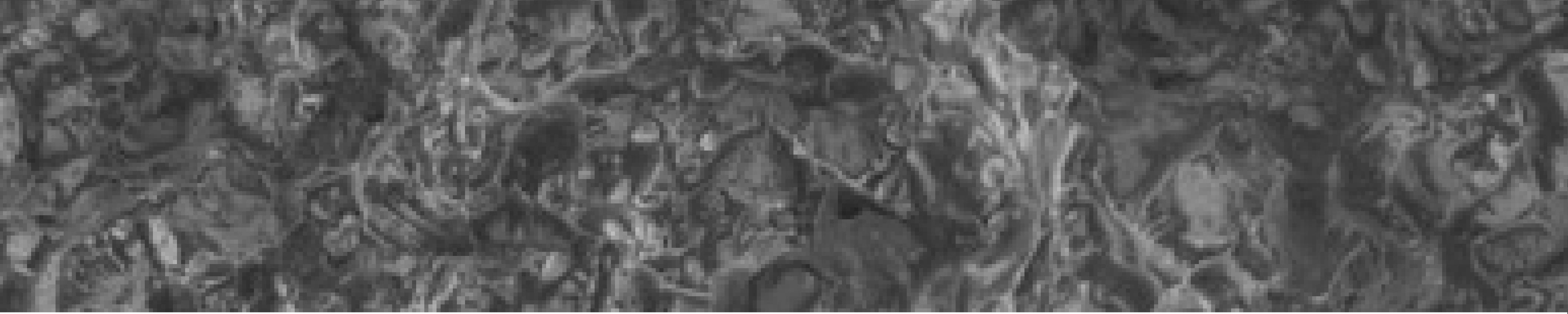
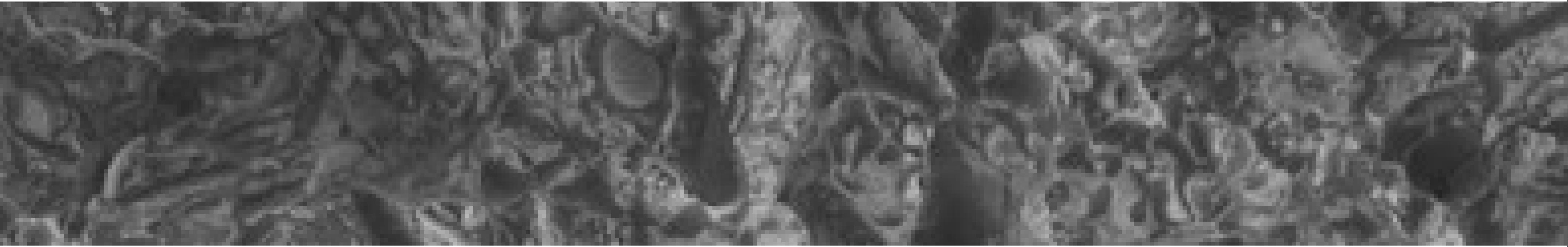


Detection of hydrogen in high strength steel with an N-15 nuclear resonance.

Presented by : Jean-Simon Larochelle

supervisor: Sjoerd Roorda
co-worker: A. Desilets Benoit, G. Borduas, E. Martel

Université de Montréal, CRIAQ project (DPHM-601)



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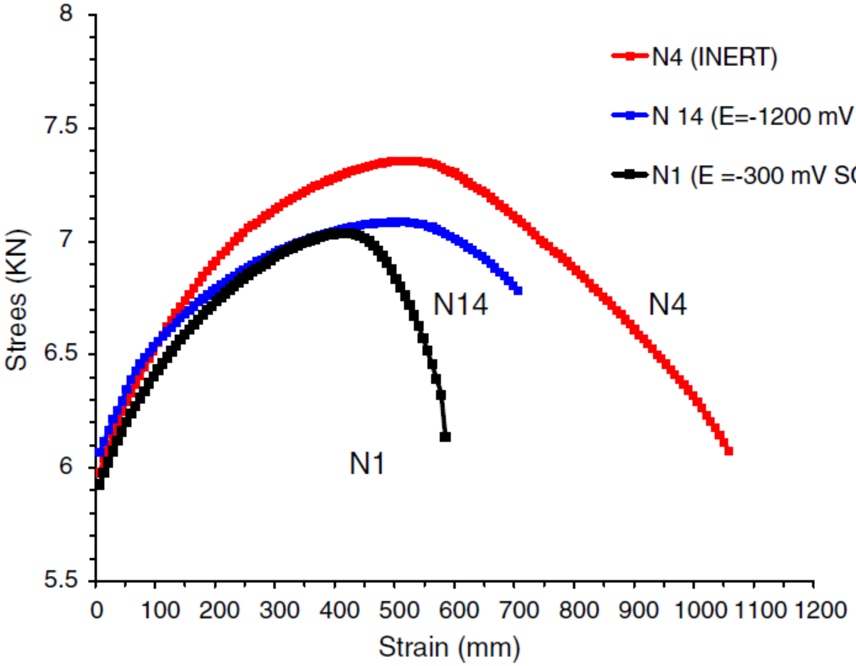
The problem of hydrogen in steel

Hydrogen Embrittlement

Hydrogen in steel decreases its mechanical strength dramatically.

To protect steel pieces, they use protective coating.

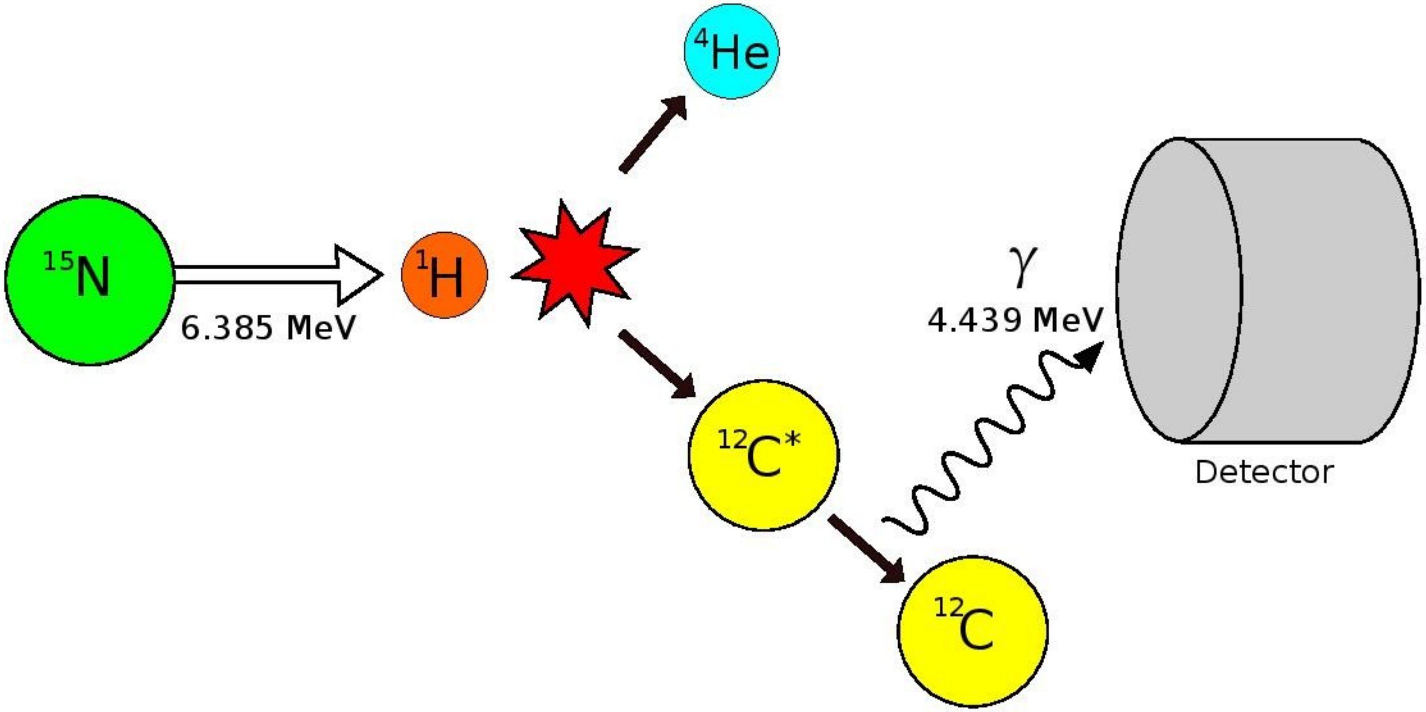
The coating can also introduce hydrogen.

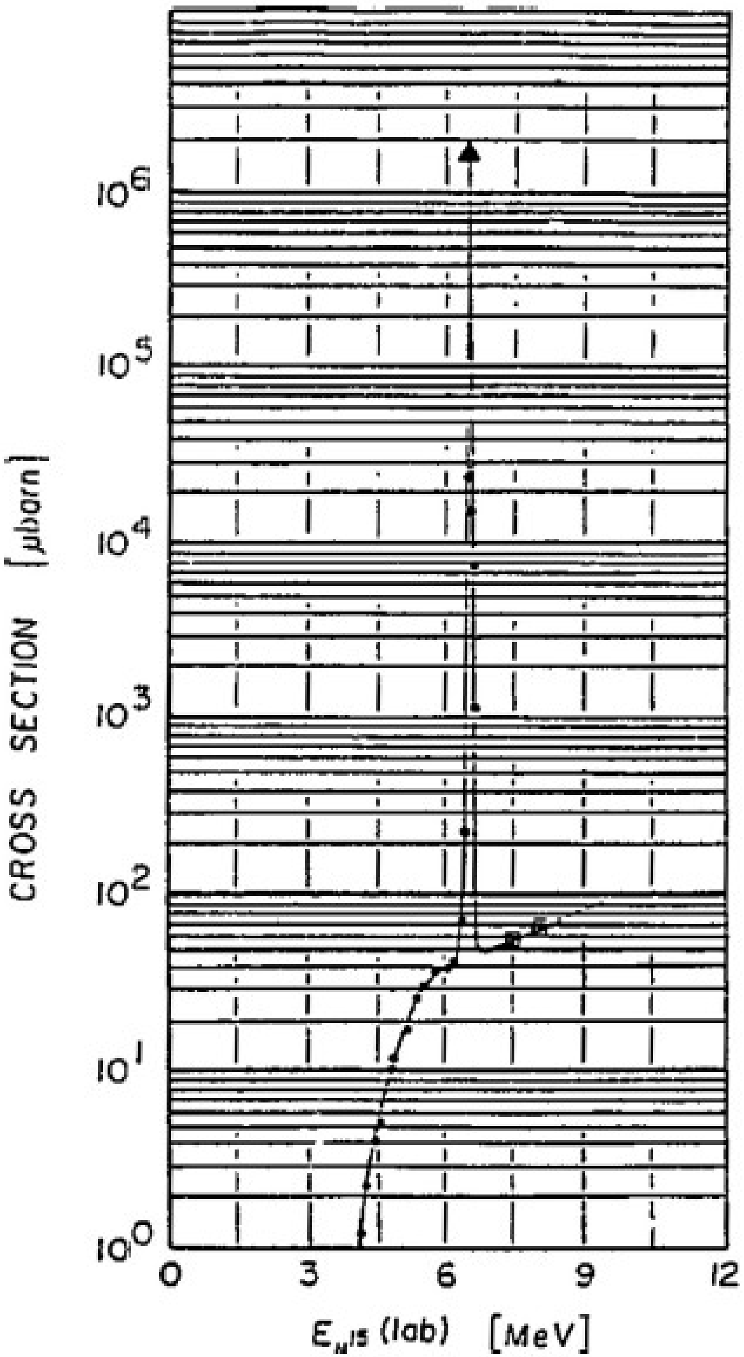


SSRT curve for normal and embrittled pearlitic steel *M. Castellote, 2007.*

Nuclear Reaction Analysis

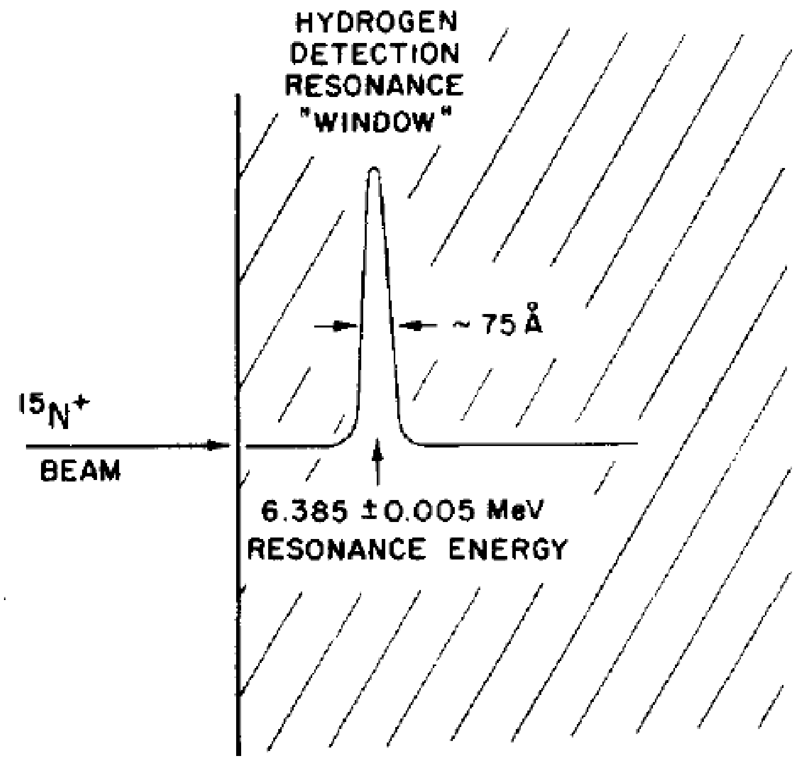
We use the $^{1}H(^{15}N,\alpha\gamma)^{12}C$ reaction and variable ^{15}N ion energy to determine the concentration of hydrogen as a function of depth inside different materials.





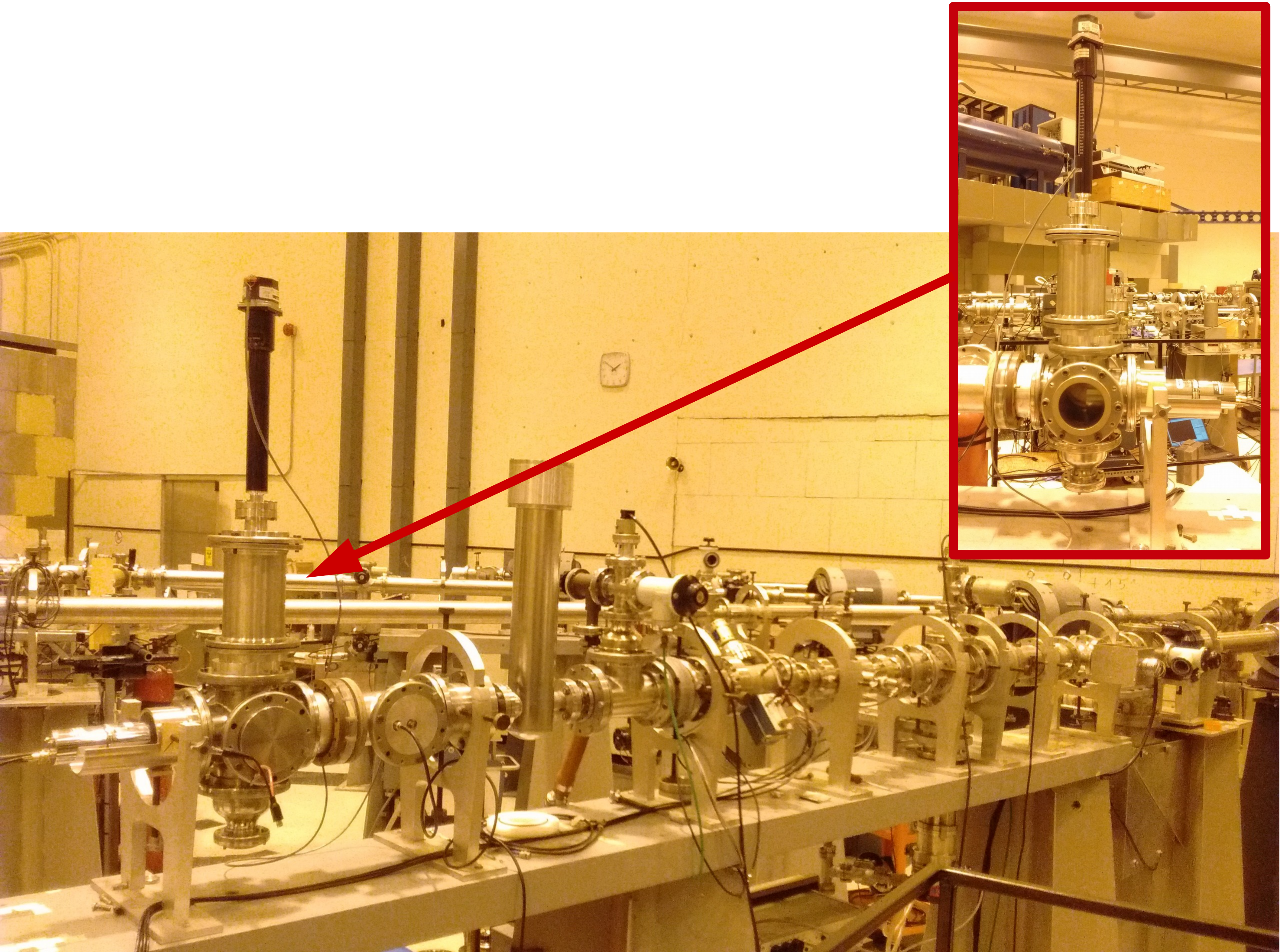
K.M. Horn and W.A. Lanford, 1988

The small energy width (Γ_R = 1,8 keV) of the reaction give us a good depth resolution.



W. Lanford and al. 1976

Stopping Power and range is evaluated with SRIM simulation.

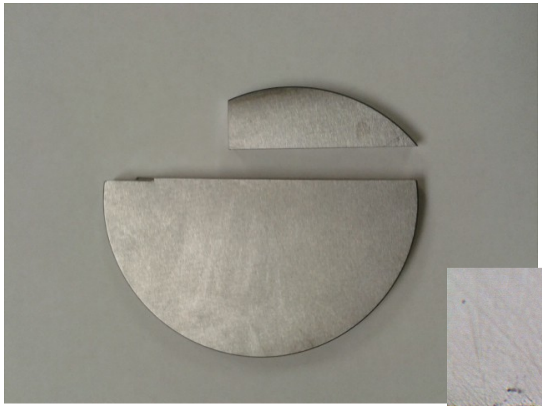


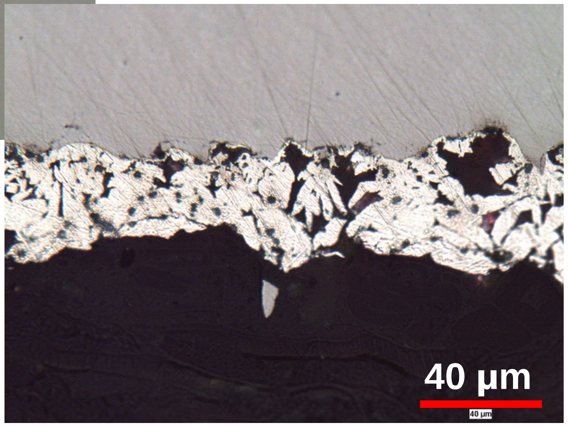
Experimental details

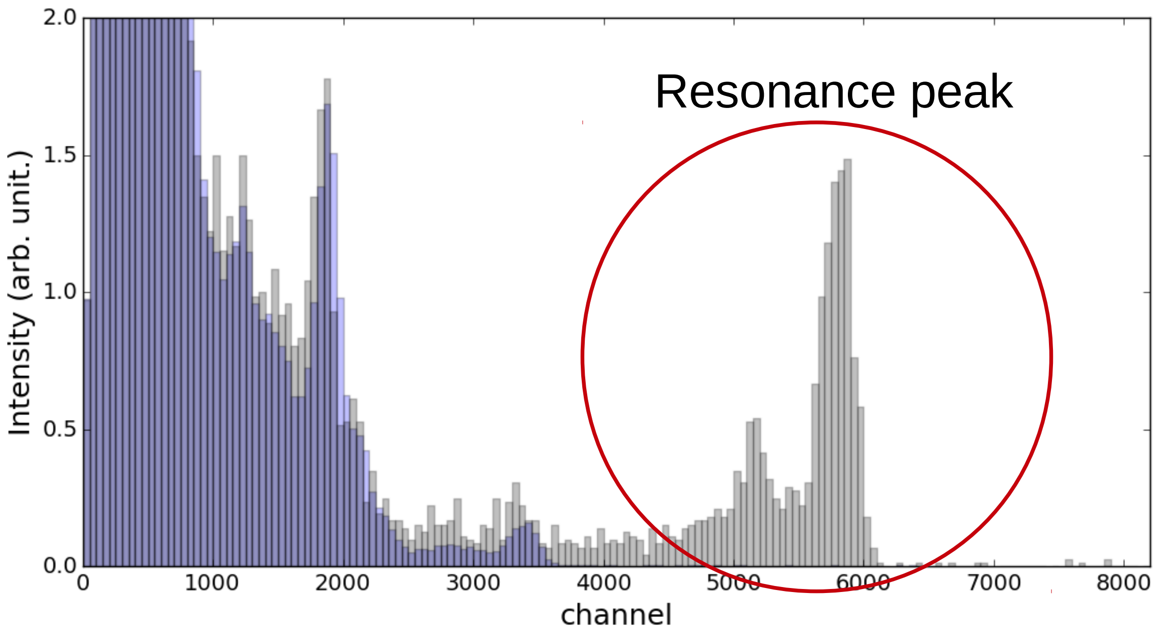
Samples

High strength steel 4340 as per AMS6414

- Standard: non-plated.
- Plated: plated with cadmium then stripped.





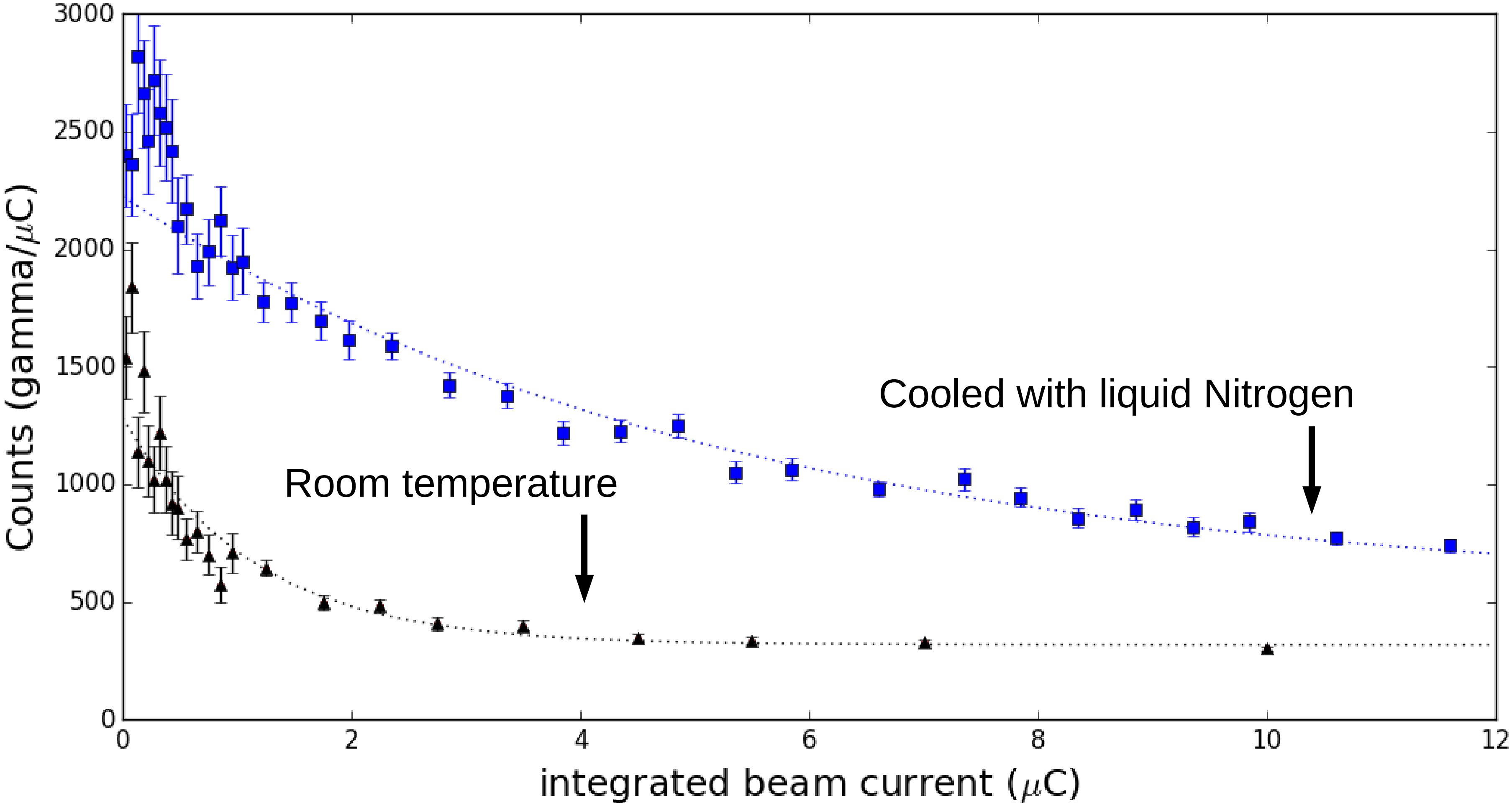


Calibration

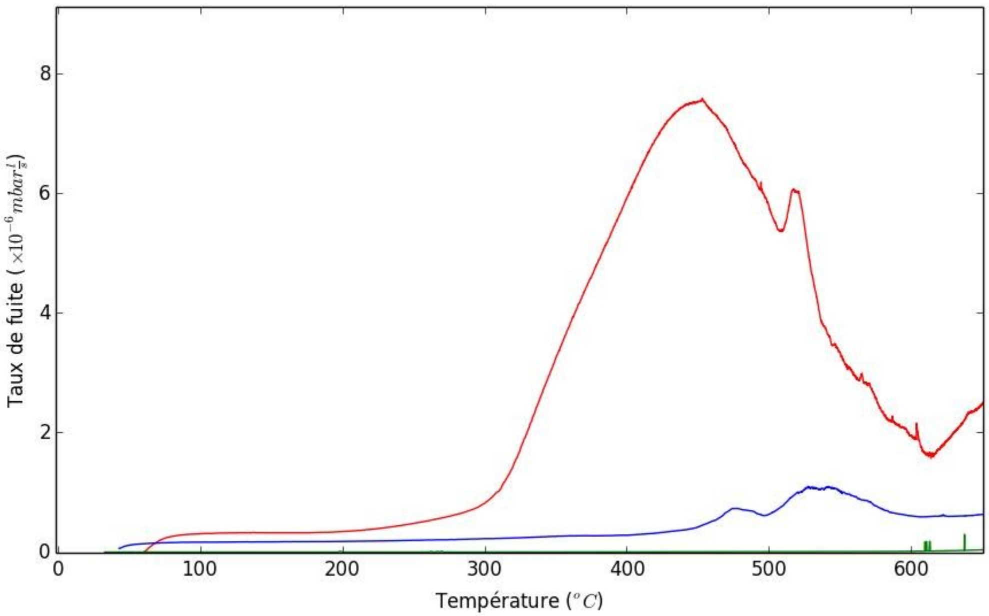
Thin film of diamond-like carbon (DLC) analyzed by RBS, ERD and ellipsometry.

Hydrogen Loss

We observed a decrease in the signal. (second order fit at fixed energy)



Desorption spectroscopy

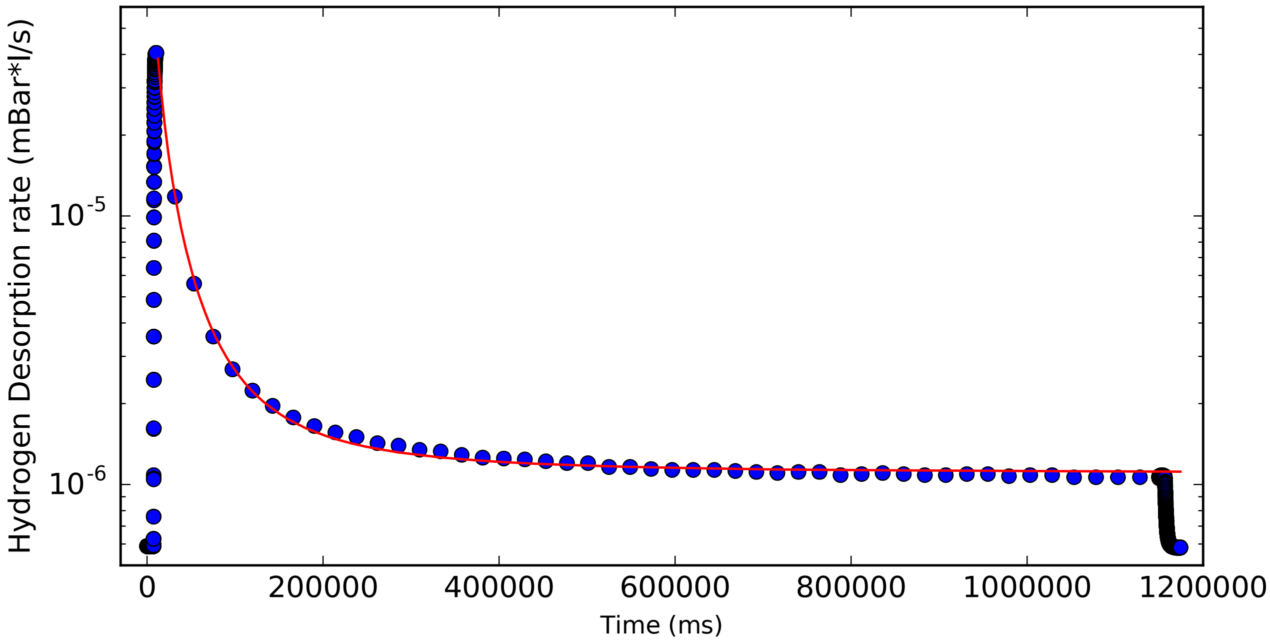


Thermal desorption spectrum of stainless steel for a first run (in red), background (in green) and after re-exposure (in blue).

E. Martel
2015

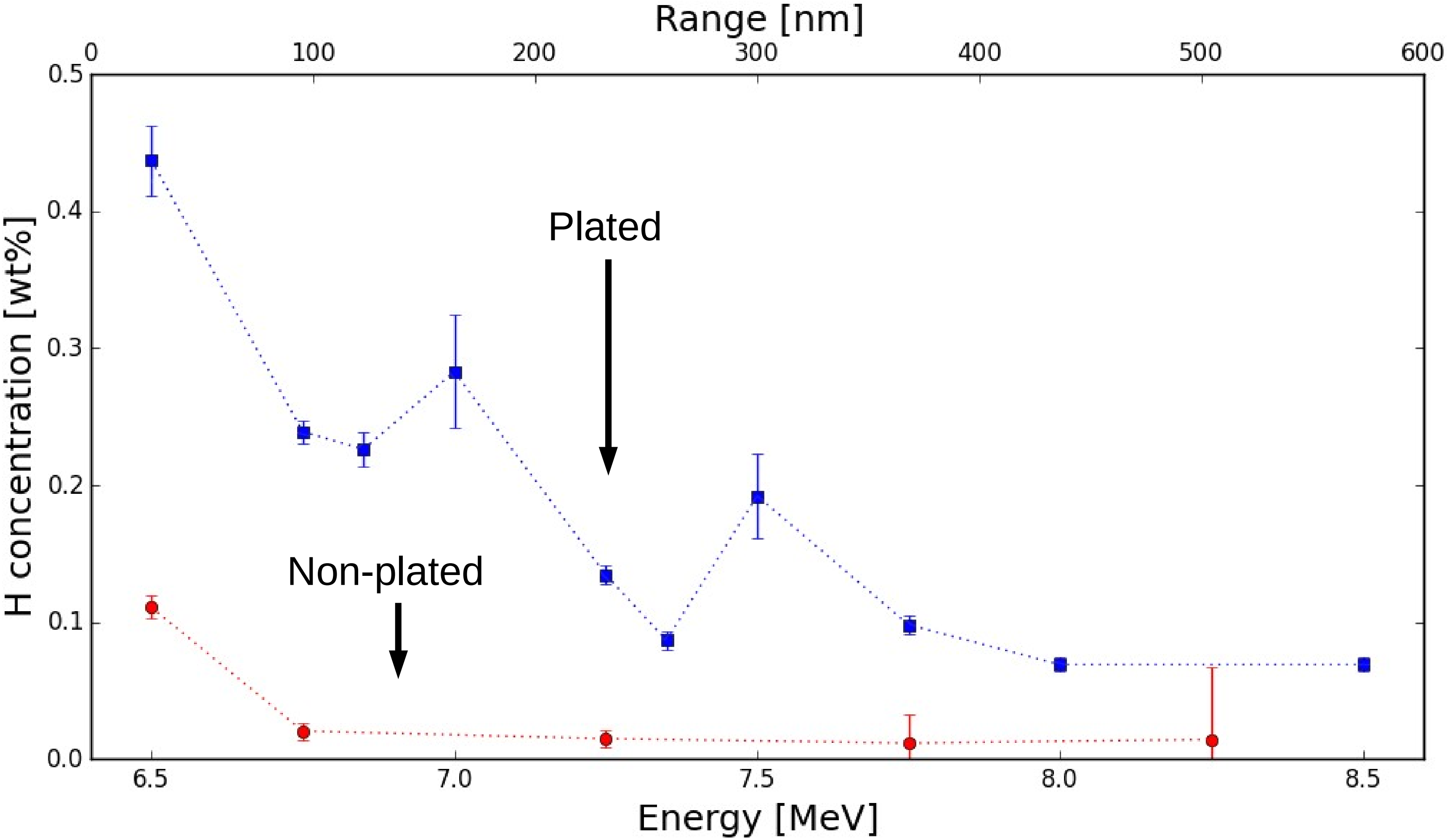
Ion beam induced desorption.

G. Borduas
2016



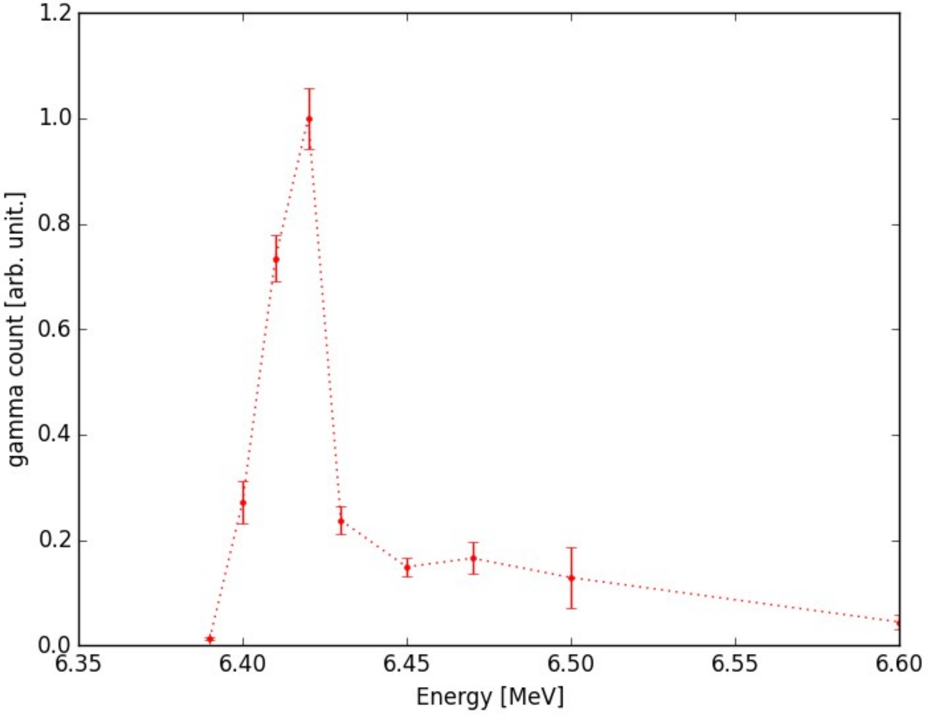
Depth profile

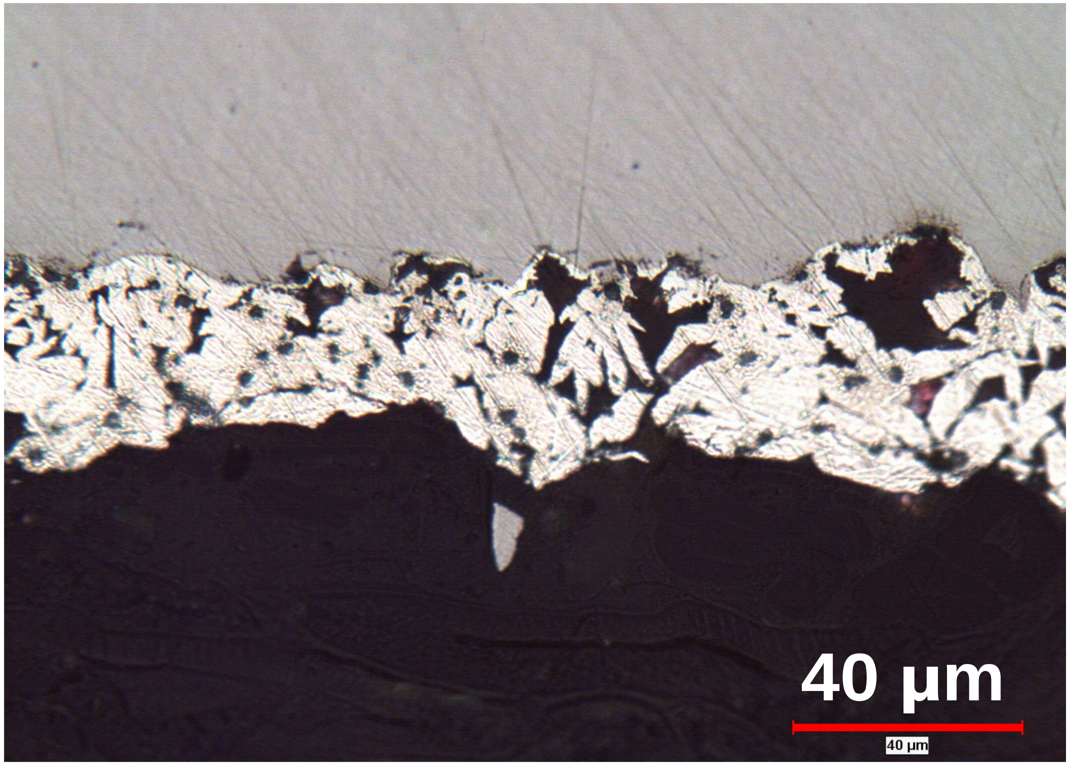
In spite of hydrogen loss during the measurement we can evaluate the concentration of hydrogen before ion beam interaction.

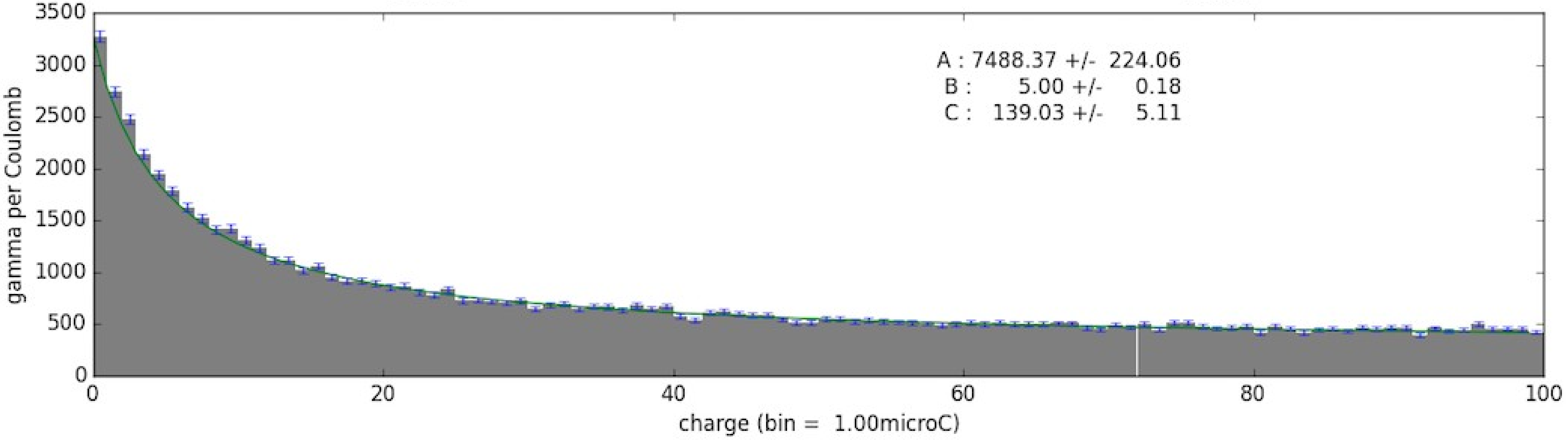


Depth profile of initial hydrogen concentration.

Surface peak





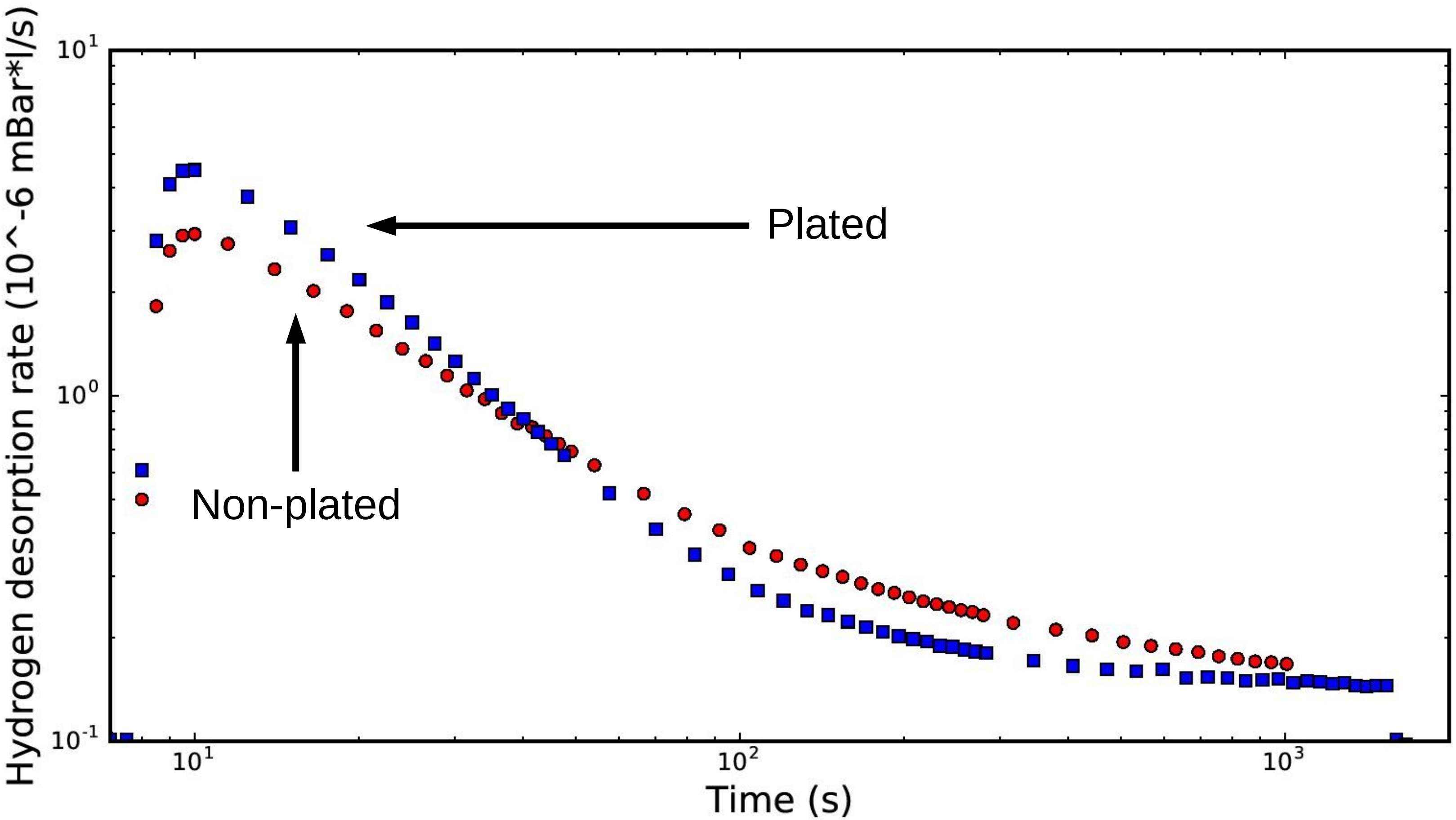


Conclusion

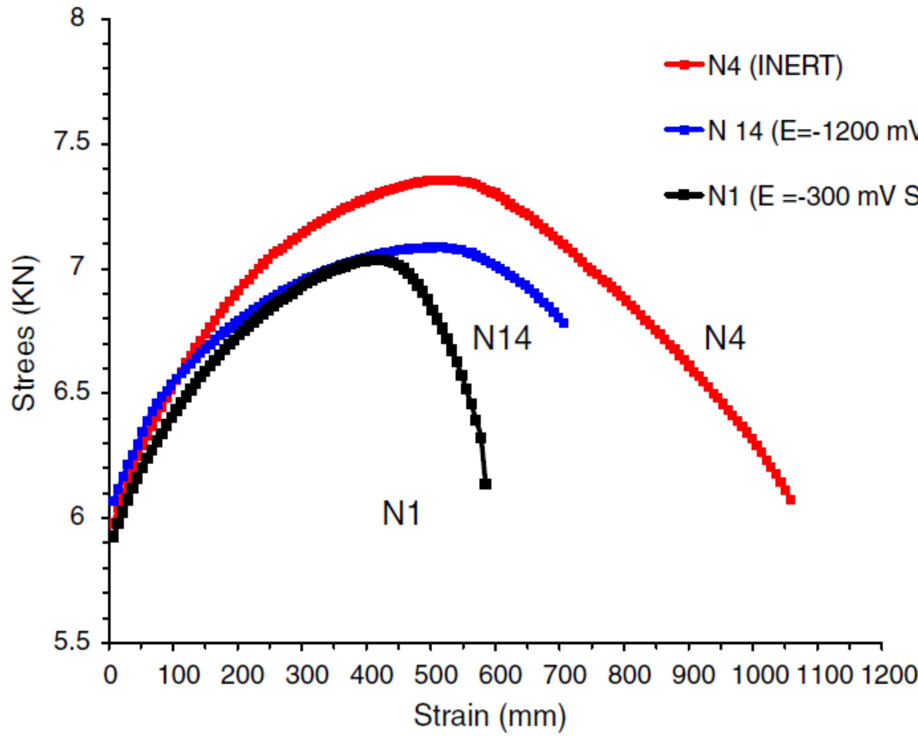
-It’s clear that the loss of sgnal is induced by the beam.

-The plated steel contains an amount of hydrogen significantly higher than the non-plated one. More plating parameters will be analysed.

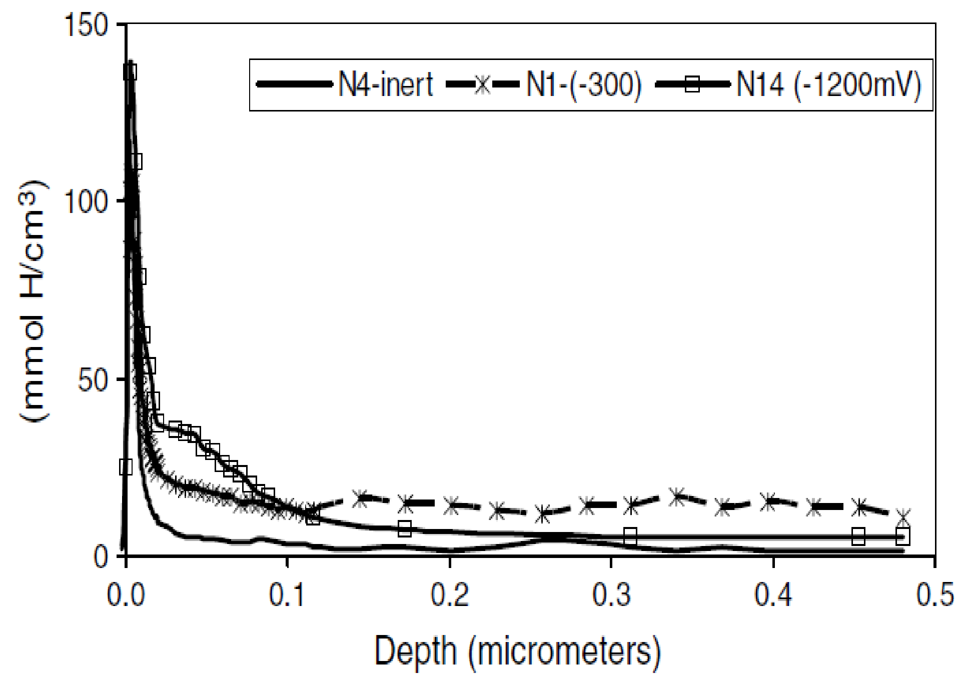
-We can have a resolution of 10 ppm. We are now improving reproducibility and extending the depth range.



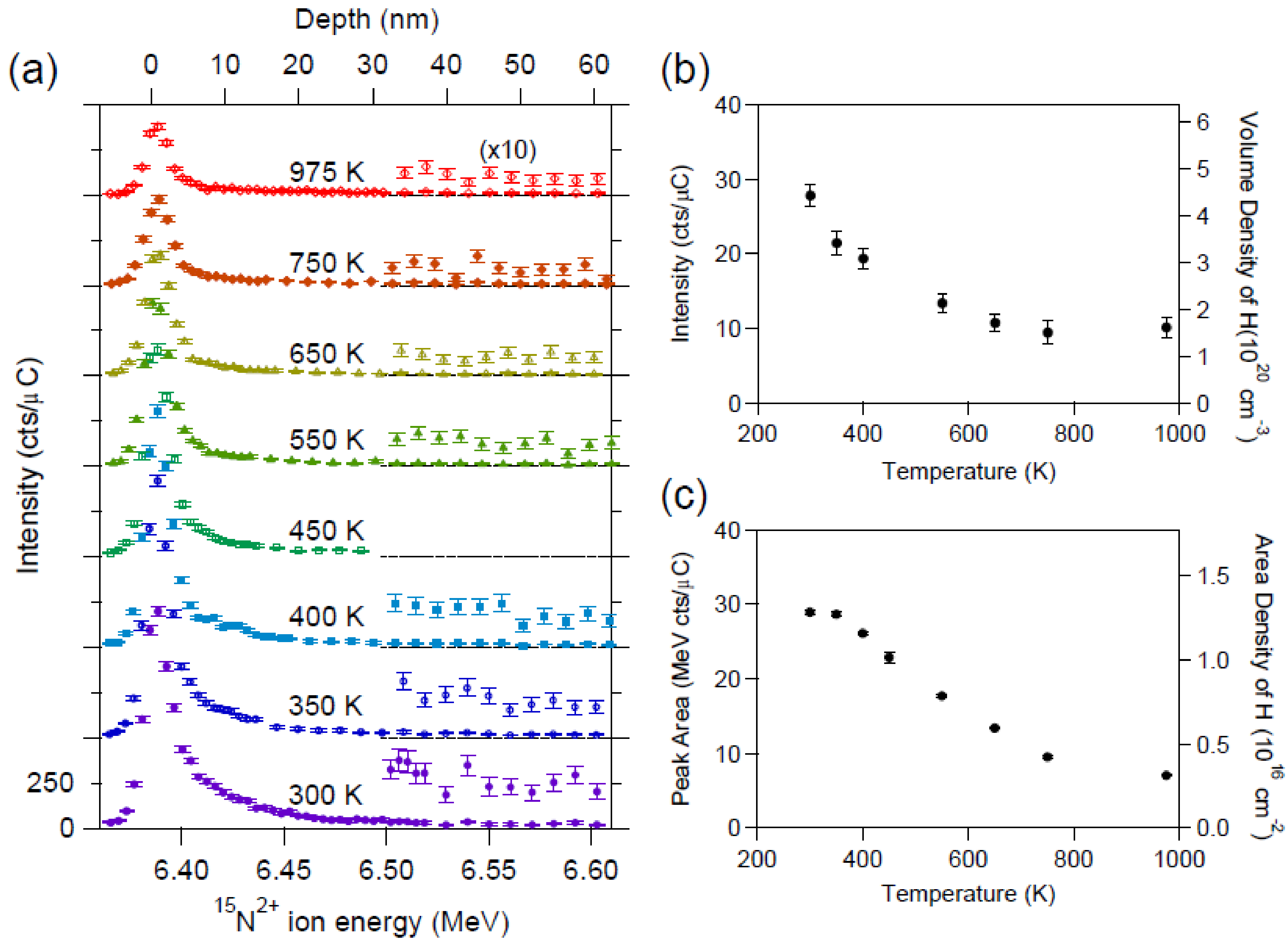
Hydrogen desorption rate (10^-6 mBar*l/s)
10^1
10^0
10^{-1}
10^1
10^2
10^3
Time (s)
Plated
Non-plated



Courbe de SSRT (Slow Strain Rate Test) pour trois échantillons d'un acier perlitique dans des milieux différents ($NaHCO_3$).

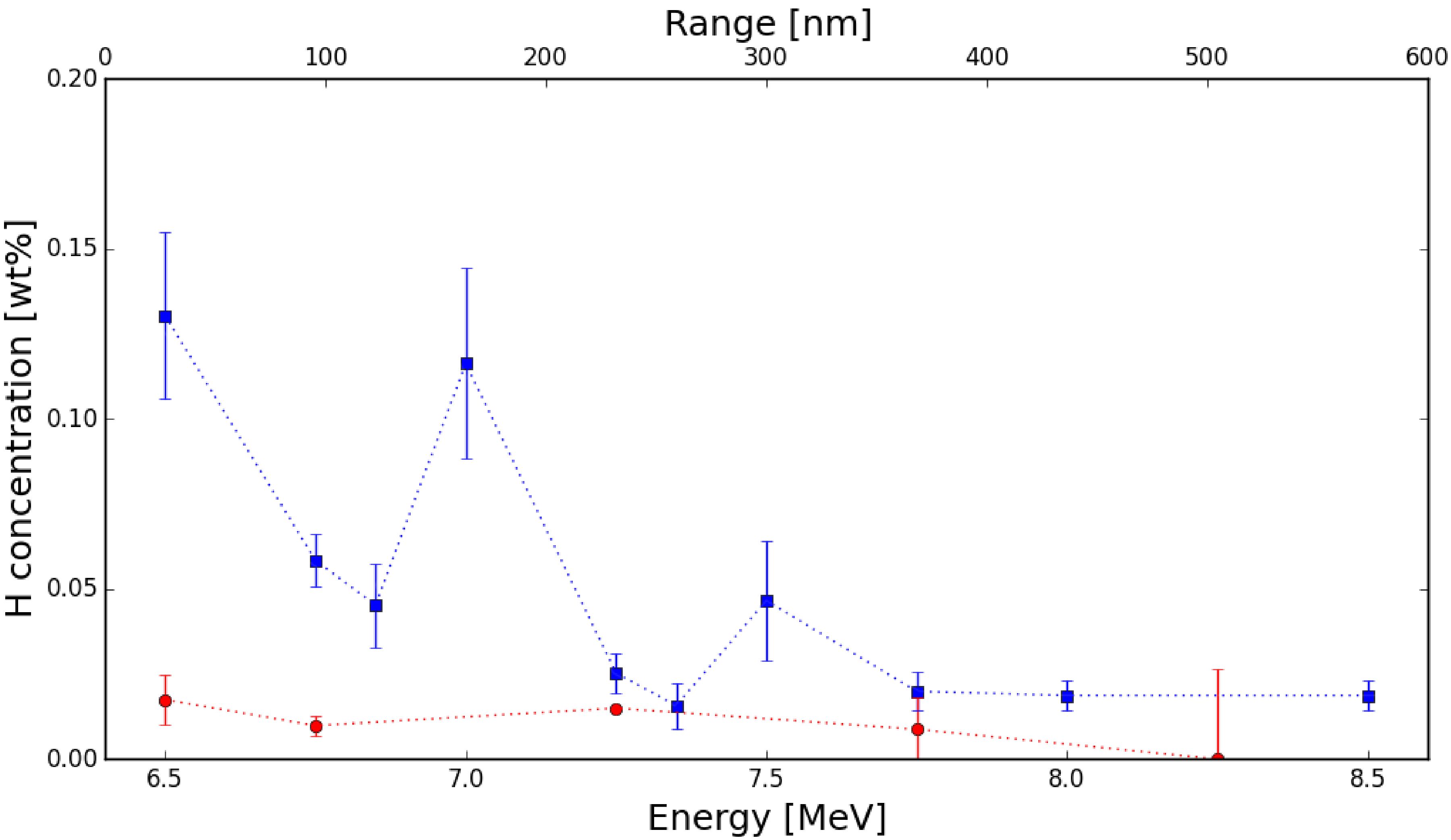


Concentration d'hydrogène en fonction de la profondeur mesurée par NRA.

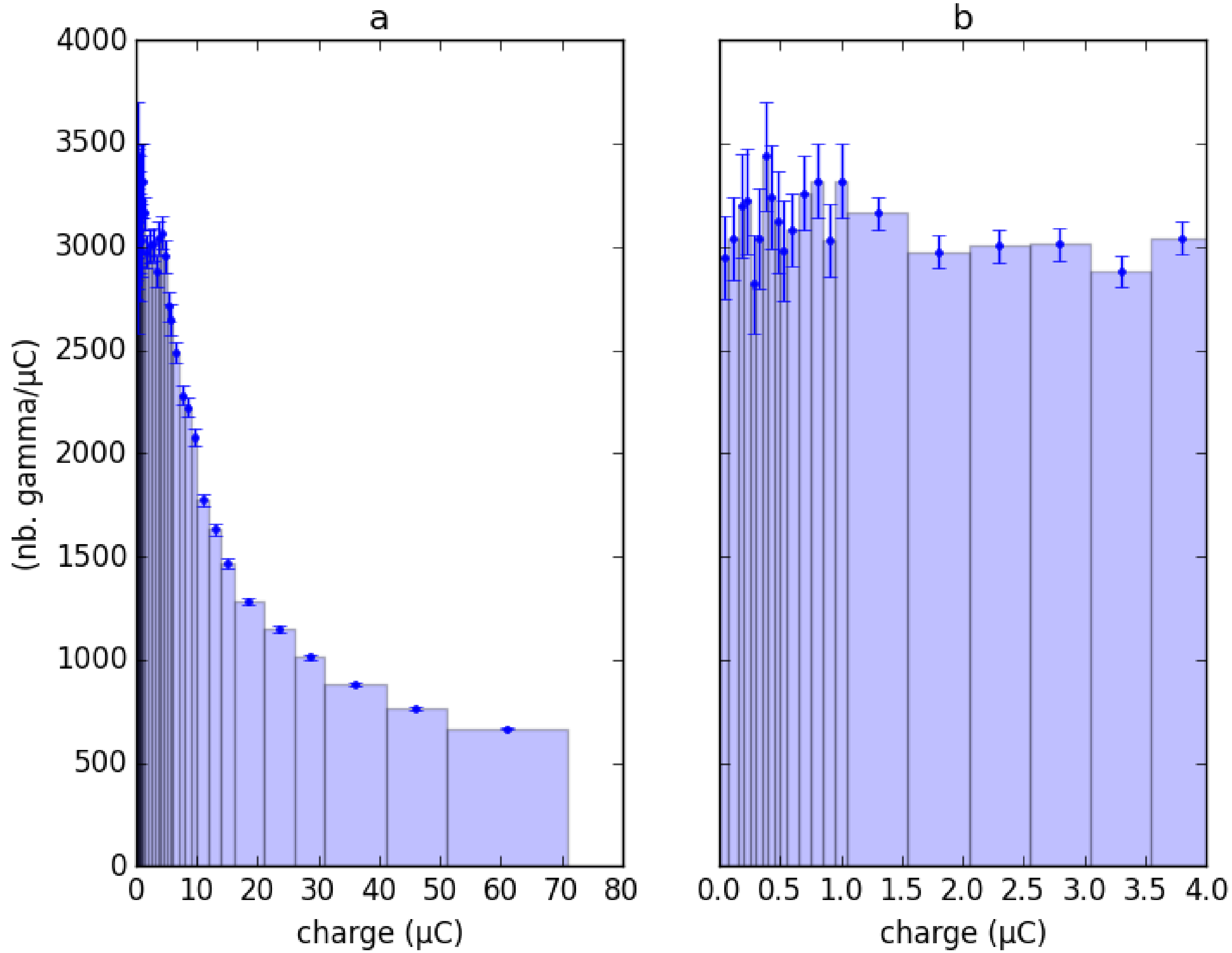


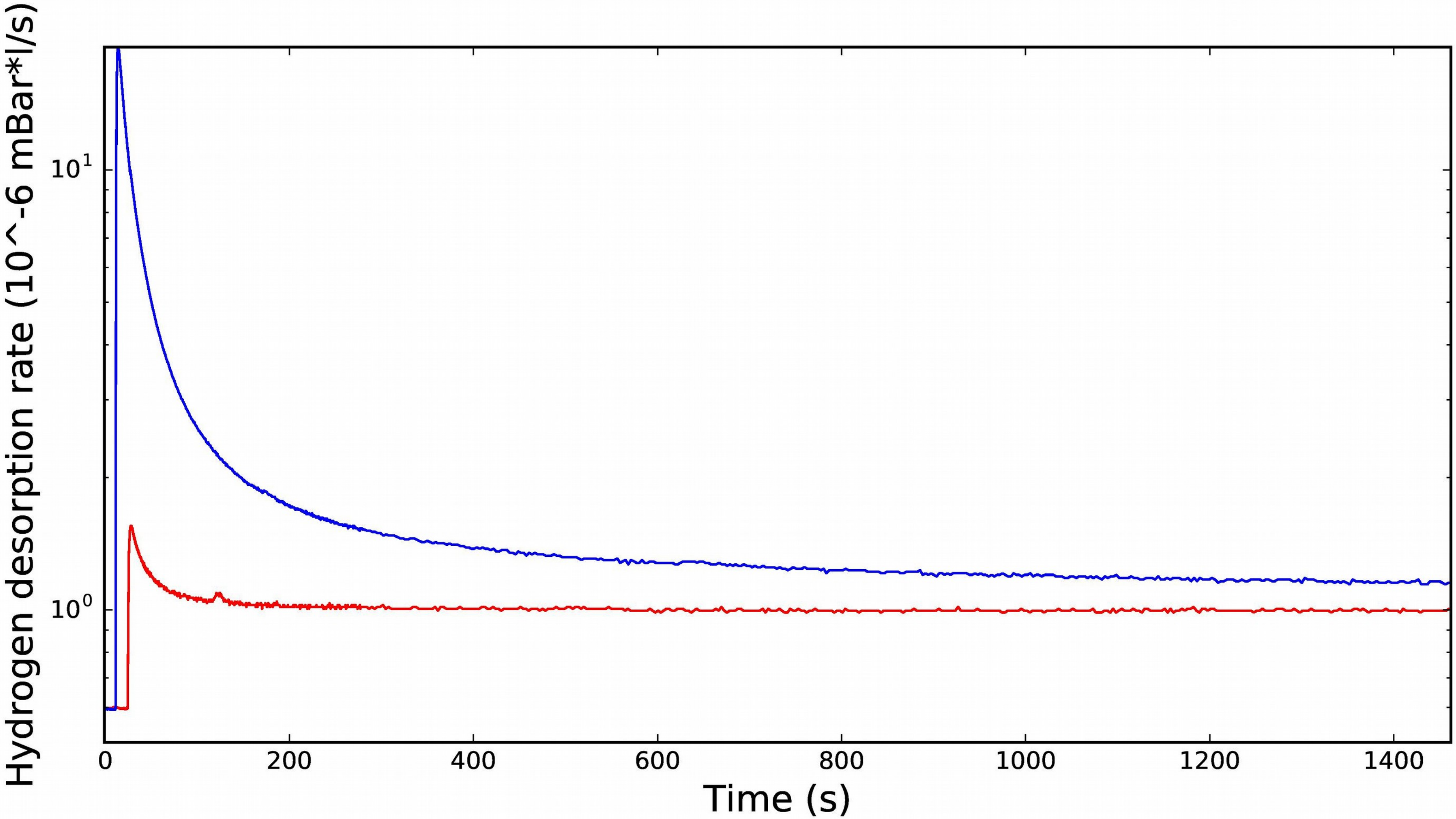
a) Profil de la concentration d'hydrogène dans un acier de type 304 pour différentes températures. b) Intensité du compte de gamma pour la région comprise de 6.50 a 6.60 MeV. c) Intensité du compte de gamma pour le pic de surface. *K. Takeyasu 2014*

Final concentration



$$N_{hydrogen} = \frac{YQ\frac{dE}{dx}}{1 - YQS_{hydrogen}}$$





Hydrogen desorption rate (10^-6 mBar*l/s)
10^1
10^0
0
200
400
600
800
1000
1200
1400
Time (s)

Strip with : Ammonium nitrate

2. Plating process [4]

- Parts are electroplated in a cadmium-cyanide bath and baked.
- Bath characteristics as per ASTM F-519, bath A :
  - 4.5 oz/gal CdO;
  - 14 oz/gal $NaCN$;
  - pH : 12;
  - Temperature : 75 °F;
  - Current density : 10 A/ft^2;
  - Plating time : 30 min.
- Baking parameters :
  - Bake time and temperature : 8h @ 375 °F;
  - Delay between plate and bake is varied (No delay, 8h, 16h).

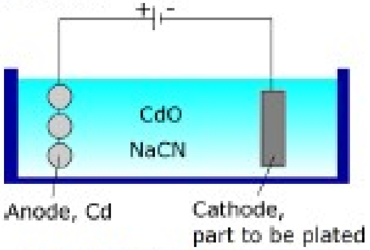
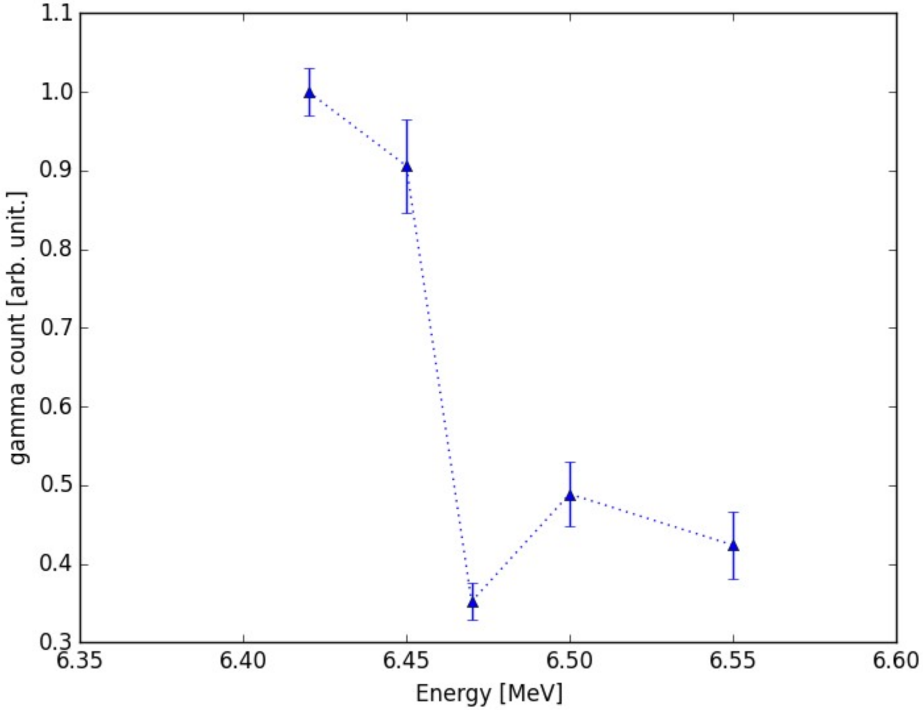
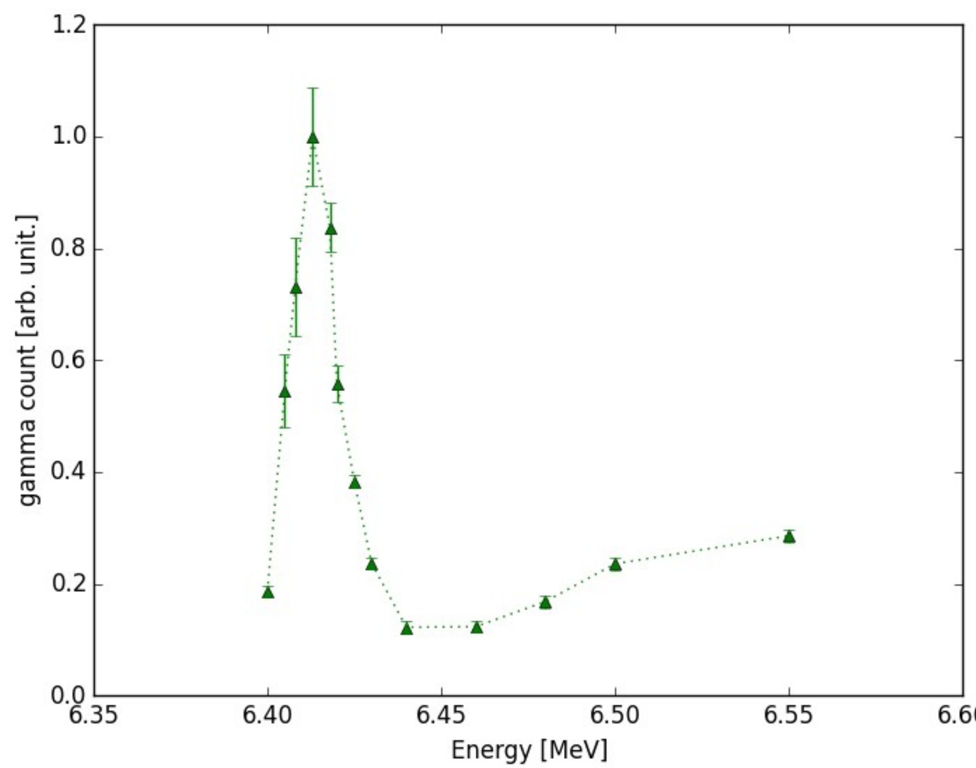


Fig. 4 : Cadmium electroplating bath



gamma count [arb. unit.]
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
6.35
6.40
6.45
6.50
6.55
6.60
Energy [MeV]



gamma count [arb. unit.]
1.2
1.0
0.8
0.6
0.4
0.2
0.0
6.35
6.40
6.45
6.50
6.55
6.60
Energy [MeV]