| ESRF | **Experiment title:** Time-resolved x-ray spectroscopy of iron and iron alloys in the Warm Dense Matter (WDM) regime. | **Experiment number**: HC-2262 |
|---|---|---|
| **Beamline**: ID24 | **Date of experiment**: from: 17/09/2015 to: 21/09/2015 | **Date of report**: 27/11/2015 |
| **Shifts:** 12 | **Local contact(s)**: Dr. MATHON Olivier | *Received at ESRF:* |
| **Names and affiliations of applicants** (* indicates experimentalists): Dr. OCCELLI Florent*, CEA, DAM, DIF, F-91297 Arpajon, France. | | |

## Report:

### 1- Overview

This experiment consisted of performing preliminary tests related to our next laser driven schock experiment at the ESRF that is scheduled in May 2016. This consisted of doing “cold” measurements on our targets (that is without laser drive). Which means

1- Measuring the XANES & EXAFS signal quality (signal-noise ratio) that can be obtained with one x-ray bunch on Fe (5µm thick foil) and $Fe_{64}Ni_{36}$ (8µm thick foil) at the Fe K-edge (7.112 keV), Ni and $Fe_{64}Ni_{36}$ (8µm thick foil) at the Ni K-edge (8.333keV), Ta (3, 4 and 5µm thick foils) near the Ta $L_3$-edge (9.880keV), and Mo foils (10 µm thick) at the Mo K-edge (20.000 keV).

2- Measuring the influence of the diamond windows (25, 40 and 100µm thick) we use as shock confinement media on the XANES & EXAFS signal quality (signal-noise ratio modification, bragg peaks implementation on the spectra); comparing the influence of the diamond windows provided by two different companies (Almax-EasyLab and Applied Diamond).

In addition, we needed to

1- Test and debug the SPEC macros specifically designed for this project.
2- Evaluate the time necessary to go from one energy range to another.
3- Test the overall beam stability at each energy range.
3- Evaluate the necessity of using vacuum tubes along the x-ray path.

### 2- Fe K-edge (7.112 keV)

2-1 Experimental conditions

Fe (ρ=7.874g/cm3) ; ideal thickness = 3.5µm ; ideal jump = ??
$Fe_{64}Ni_{36}$ (ρ=8.1g/cm3) ; ideal thickness = 6µm @ Fe K-edge ; ideal jump = ??

We have used 5µm-thick Goodfellow pure iron (ref. FE000100) and 8µm-thick Goodfellow $Fe_{64}Ni_{36}$ (ref. FE020210).

Spotsize=??µm(H)x??µm(V) ; spectral range ~??eV

We used vacuum tubes along the x-ray path.

## 2-2 Iron and $Fe_{64}Ni_{36}$ without confinement

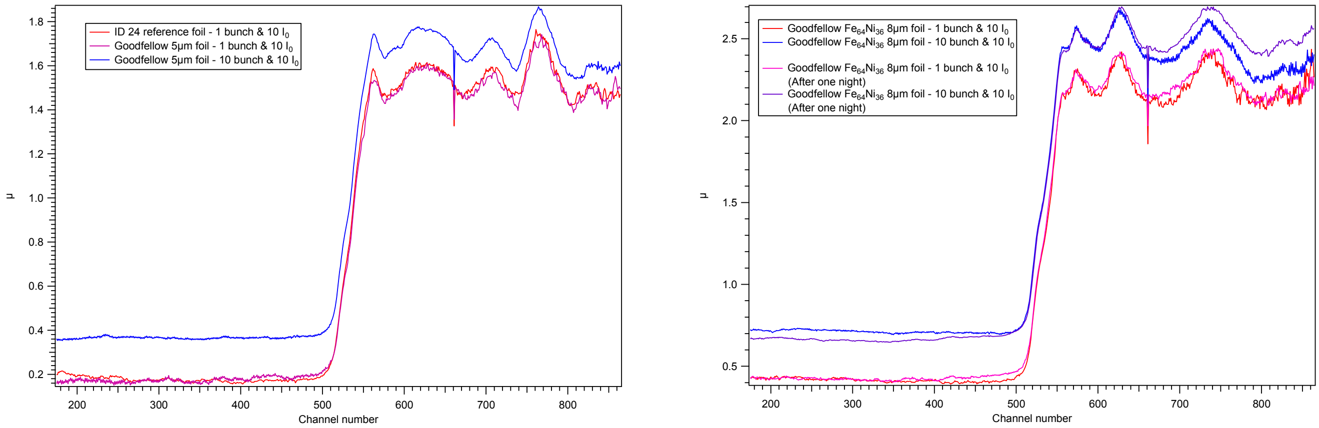



**Fig. 1:** Left, 5µm thick pure Fe foil without confinement – Right, 8µm thick $Fe_{64}Ni_{36}$ foil without confinement.

## 2-3 Iron and $Fe_{64}Ni_{36}$ in between two 25µm-thick Applied Diamond (AD) windows

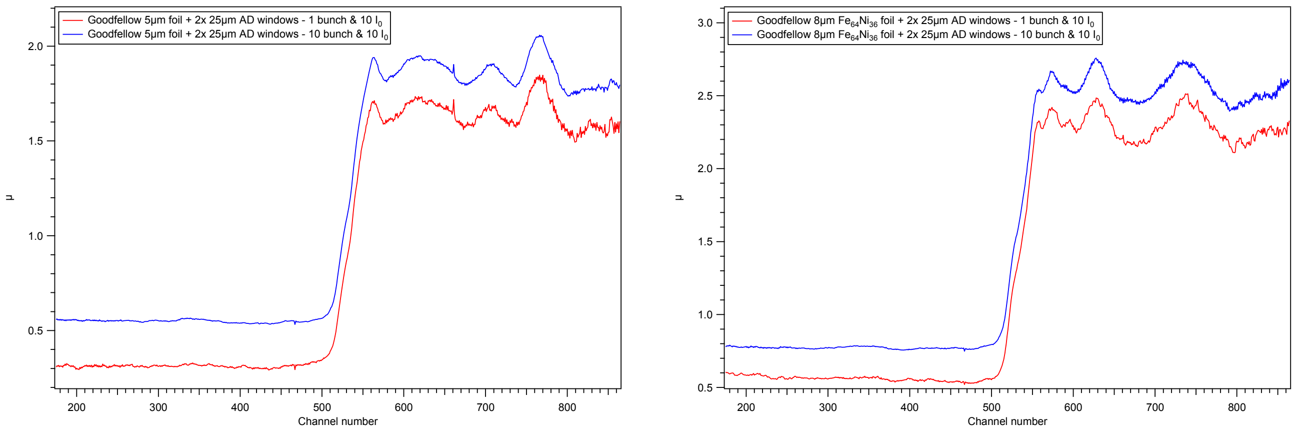



**Fig. 2:** Left, 5µm thick pure Fe foil between two 25µm windows – Right, 8µm thick $Fe_{64}Ni_{36}$ foil between two 25µm windows.

2-4 Iron and $Fe_{64}Ni_{36}$ in between two 40µm-thick ALMAX easyLab or Applied Diamond (AD) windows

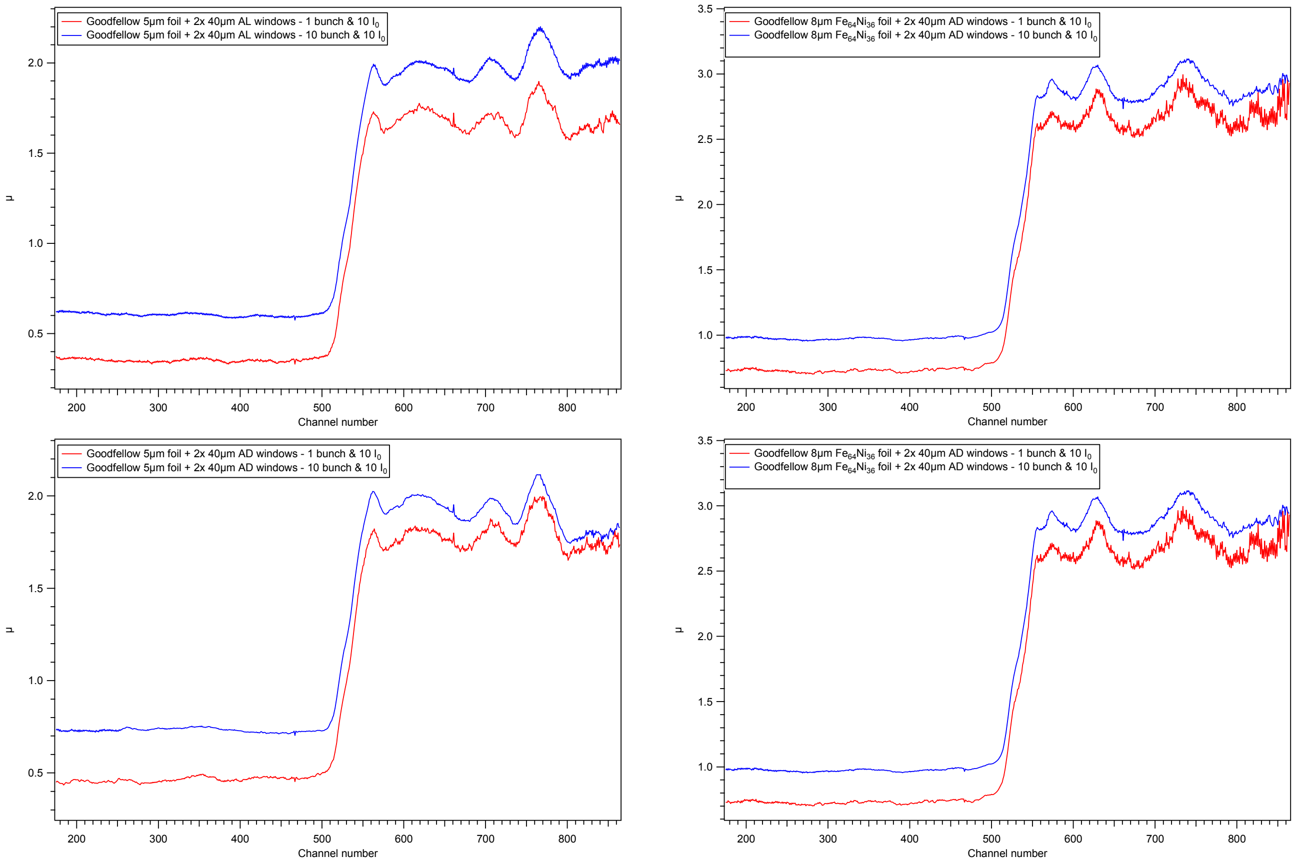



**Fig. 3:** Top-Left, 5µm thick pure Fe foil between two 40µm Almax windows – Top-Right, 8µm thick $Fe_{64}Ni_{36}$ foil between two 40µm Almax windows - Bottom-Left, 5µm thick pure Fe foil between two 40µm AD windows – Bottom -Right, 8µm thick $Fe_{64}Ni_{36}$ foil between two 40µm AD windows.

2-5 Iron and $Fe_{64}Ni_{36}$ in between two 100µm-thick Almax windows

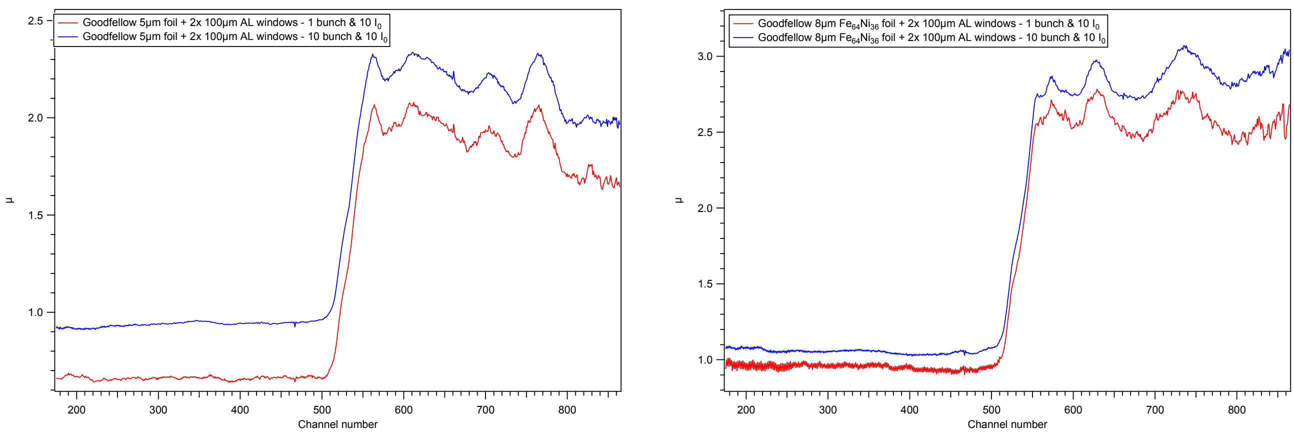



**Fig. 4:** Left, 5µm thick pure Fe foil between two 100µm windows – Right, 8µm thick $Fe_{64}Ni_{36}$ foil between two 100µm windows.

## 3- Ni K-edge (8.333 keV)

### 3-1 Experimental conditions

Ni (ρ=8.908g/cm3) ; ideal thickness = ??µm ; ideal jump = ??
$Fe_{64}Ni_{36}$ (ρ=8.1g/cm3) ; ideal thickness = ??µm @ Ni K-edge ; ideal jump = ??

We have used 5µm-thick Goodfellow pure nickel (ref. NI000224) and 8µm-thick Goodfellow $Fe_{64}Ni_{36}$ (ref. FE020210).

Spotsize=8µm(H)x110µm(V) ; spectral range ~500eV

Time to shift from Fe K-edge to Ni K-edge : ?? hours

We used vacuum tubes along the x-ray path.

### 3-2 Ni and $Fe_{64}Ni_{36}$ without confinement

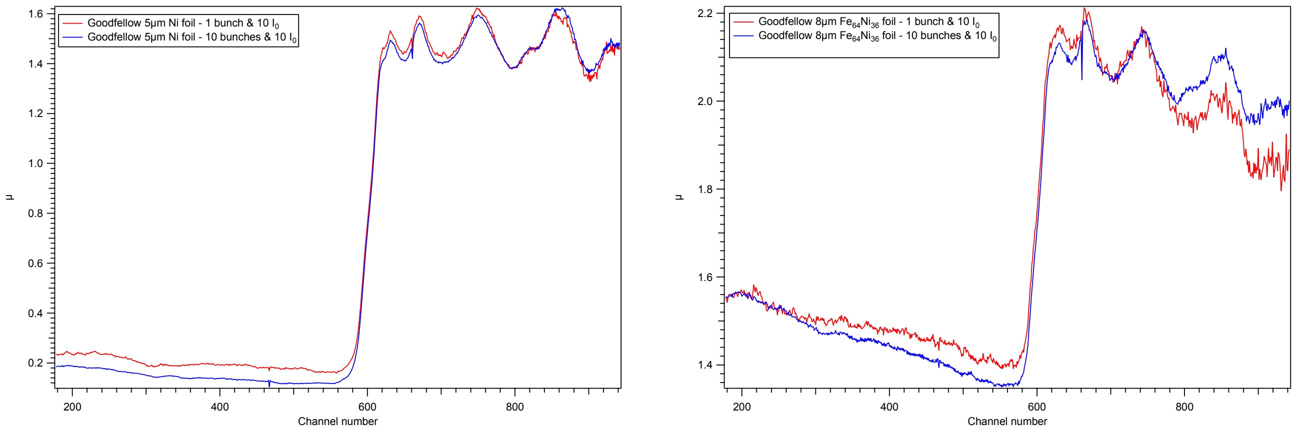



**Fig. 5:** Left, 5µm thick pure Ni foil without confinement – Right, 8µm thick $Fe_{64}Ni_{36}$ foil without confinement.

### 3-3 $Fe_{64}Ni_{36}$ in between two 40µm-thick Applied Diamond windows

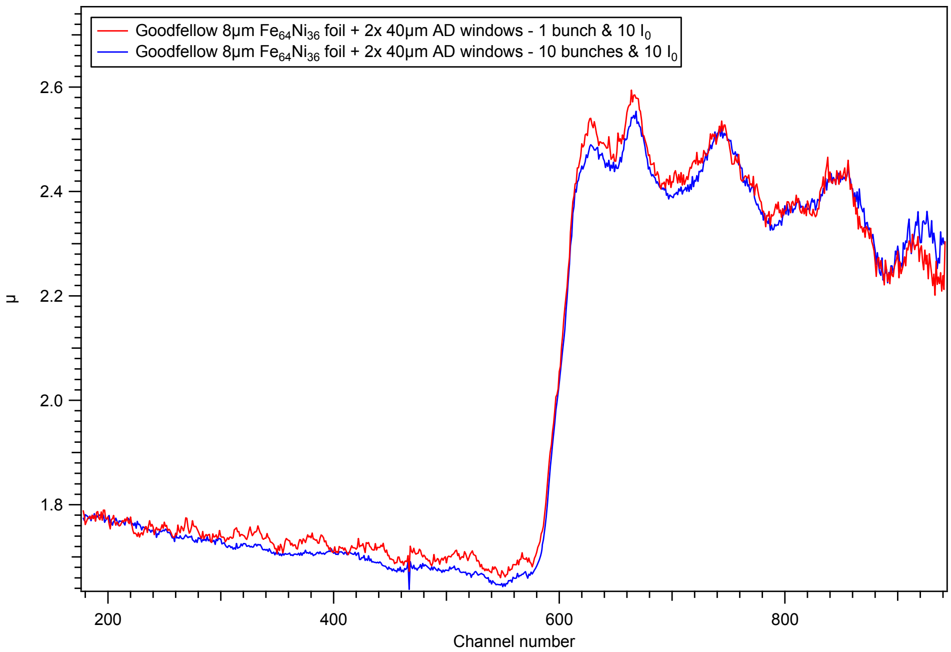



**Fig. 6:** 8µm thick $Fe_{64}Ni_{36}$ foil between two 40µm AD windows.

3-4 $Fe_{64}Ni_{36}$ in between two 100µm-thick ALMAX easyLab windows

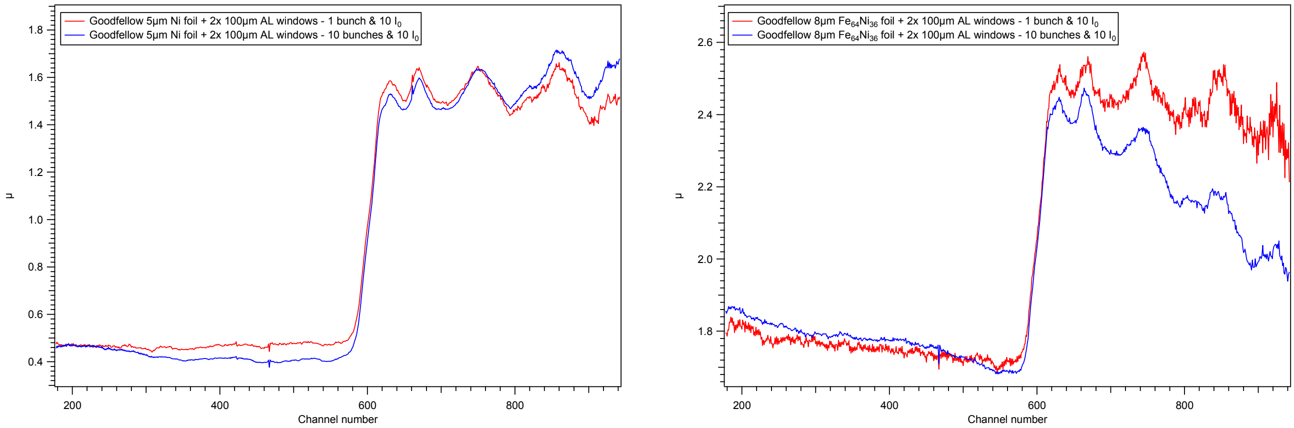



**Fig. 7:** Left, 5µm thick pure Ni foil between two AL 100µm windows – Right, 8µm thick $Fe_{64}Ni_{36}$ between two AL 100µm windows.

## 4- Ta $L_3$-edge (9.880 keV)

4-1 Experimental conditions

Ta (ρ=16.650g/cm3) ; ideal thickness = 3.5µm ; ideal jump = 0.83

We have used 3, 4 and 5µm-thick Goodfellow pure tantalum (respective refs TA000120, TA000130 and TA000140).

Spotsize=??µm(H)x??µm(V) ; spectral range ~??eV

Time to shift from Ni K-edge to Ta $L_3$-edge : 2 hours.

We used vacuum tubes along the x-ray path.

4-2 3µm-thick Ta without confinement

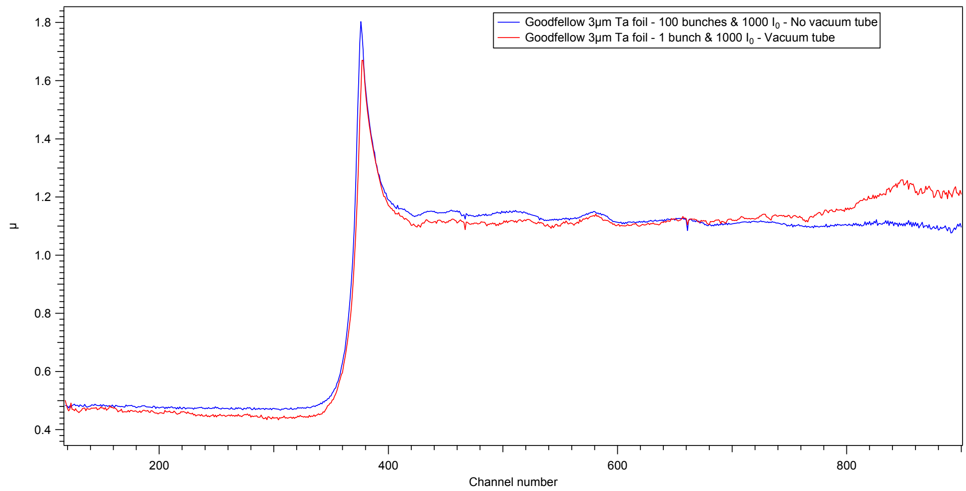



**Fig. 8:** 3µm thick Ta foil without confinement. A vacuum tube was monted before recording the 1-bunch spectrum

4-3 3µm-thick Ta in between two 25µm-thick Applied Diamond windows

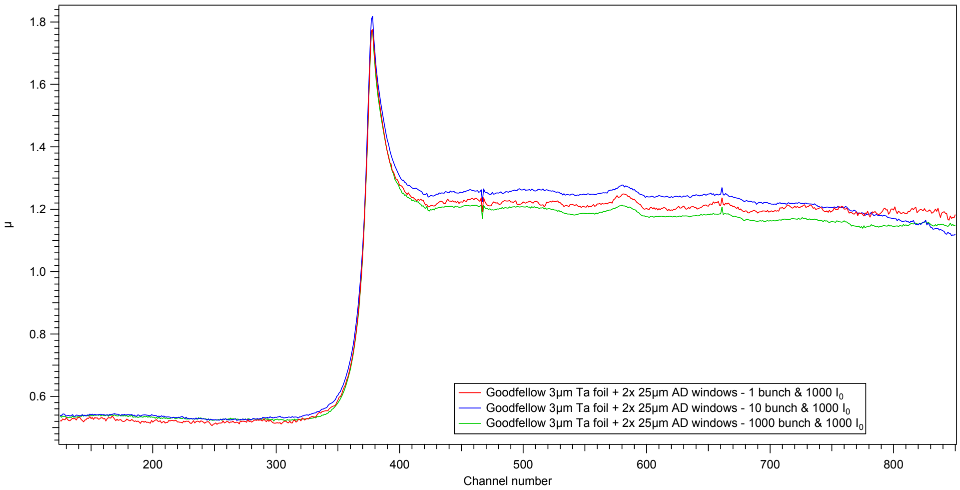



**Fig. 9:** 3µm thick Ta foil between two AD 25µm windows.

4-4 3µm-thick Ta in between two 40µm-thick Almax or Applied Diamond windows

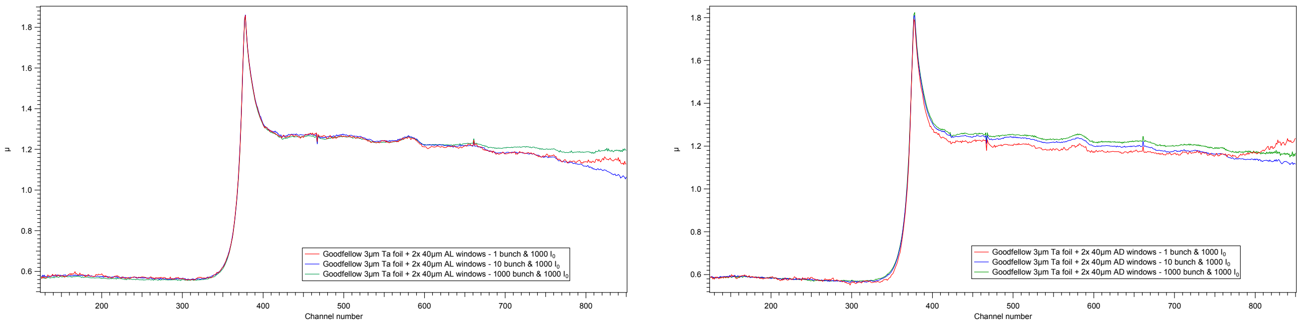



**Fig. 10:** Left, 3µm thick Ta foil between two 40µm Almax windows – Right, 3µm thick Ta foil between two 40µm Applied Diamonds windows.

4-5 3µm-thick Ta in between two 100µm-thick Almax windows

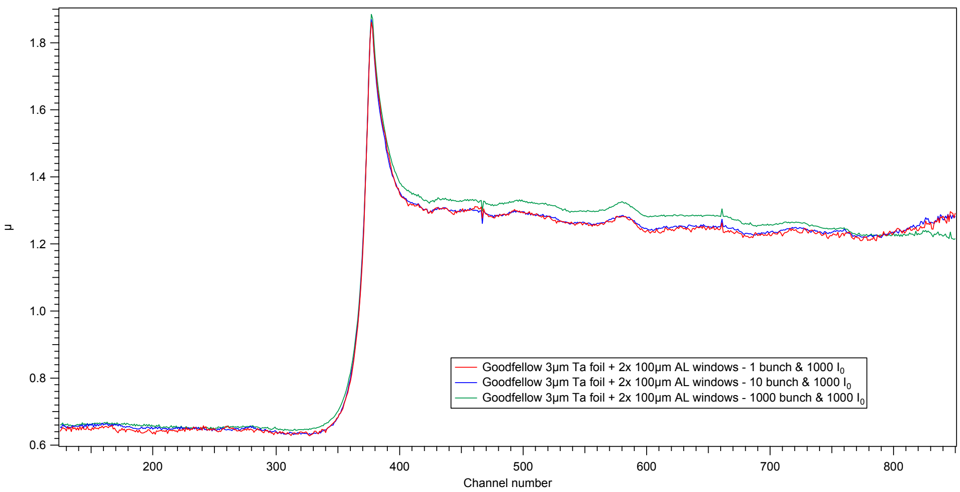



**Fig. 11:** 3µm thick Ta foil between two Almax 100µm windows.

4-6 4µm-thick Ta in between two 40µm-thick Almax windows ;  comparison with 3µm-thick Ta in the same conditions.

The spectra quality of 4µm-thick Ta is way better than that of 3µm-thick Ta. This may be due to a poor material quality of the 3µm-thick foil we used.

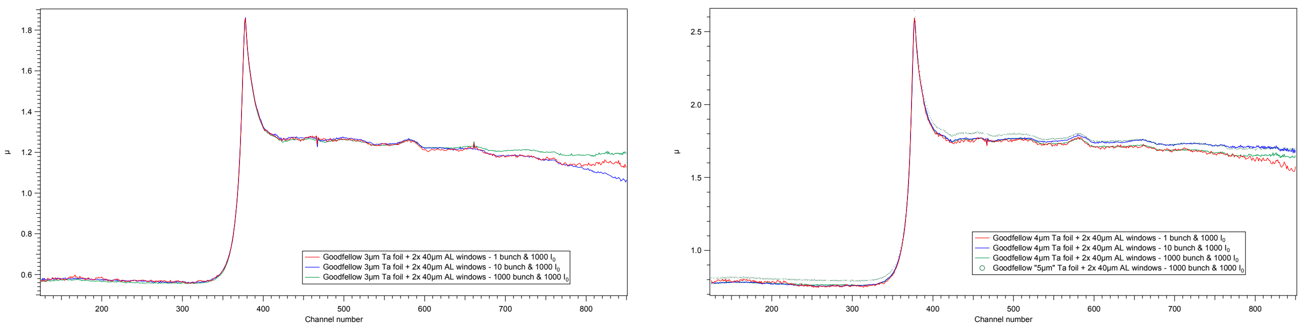



**Fig. 12:** Left, 3µm thick Ta foil between two Almax 40µm windows.– Right, 4µm thick Ta foil between two Almax 40µm windows. The Ta foil sold by Goodfellow as “5µm-thick” turns out to be 4µm-thick

4-7 Miscellaneous: Test with or without vacuum tube ; Change of detector position @ 194cm

We have removed the vacuum tubes. By testing on a 4µm Ta target in between two 40µm-thick Applied Diamond windows, we see a decrease on I by a factor of 3. The XH detector capacitance is set to 5pF.

We have also moved the detector to 194 cm so as to increase the resolution. But the vacuum tube had to be put back in place.

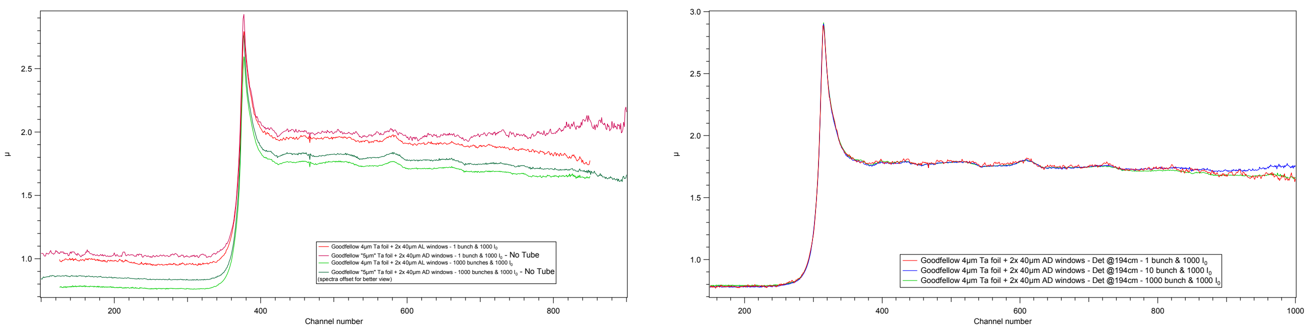



**Fig. 13:** Left, Differences between spectra recorded with or without vacuum tube.– Right, Change of detector position @ 194cm

## 5- Mo K-edge (20.000 keV)

### 5-1 Experimental conditions

Mo (ρ=10.280g/cm3) ; ideal thickness = 16.7µm ; ideal jump = 1.17

We have used 10µm-thick Goodfellow pure molybdenum (ref. MO000200)

Spotsize=??µm(H)x??µm(V) ; spectral range ~??eV

Time to shift from Ta $L_3$-edge to Mo K-edge : ?? hours

Here, we **didn't** use vacuum tubes along the x-ray path.

### 5-2 10µm-thick Mo without confinement

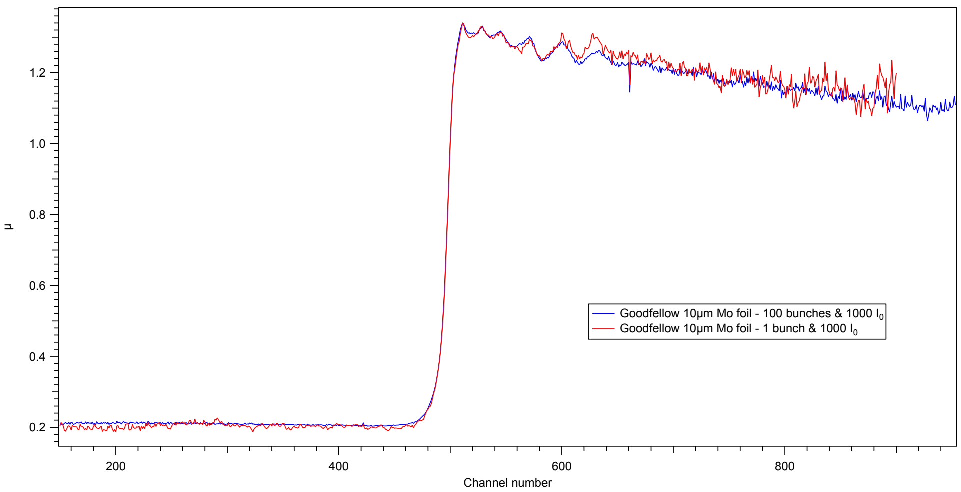



**Fig. 14:** 10µm thick Mo foil without confinement.

### 5-3 10µm-thick Mo in between two 25µm-thick Applied Diamond windows

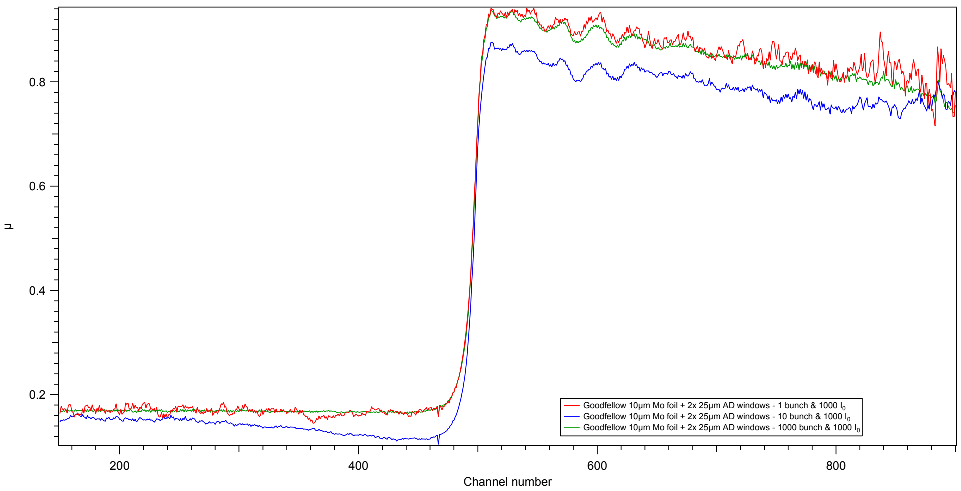



**Fig. 15:** 10µm thick Mo foil between two AD 25µm windows.

5-4 10µm-thick Mo in between two 40µm-thick Almax or Applied Diamond windows

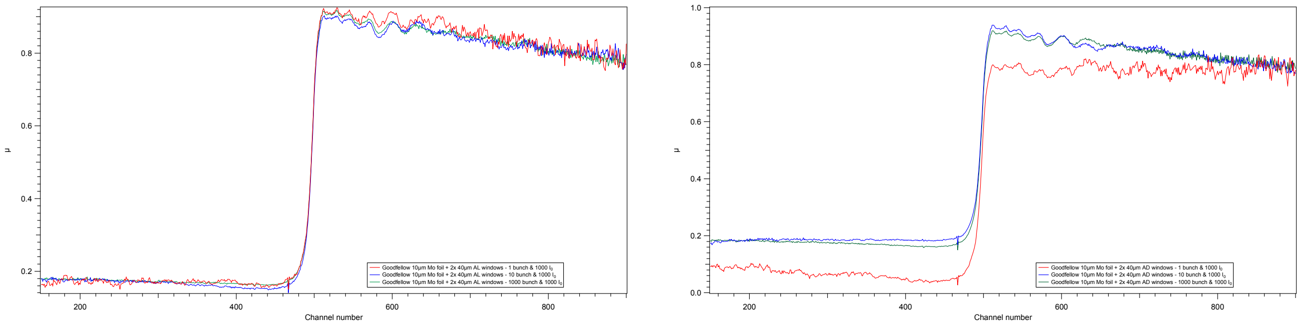



**Fig. 16:** Left, 10µm thick Mo foil between two 40µm Almax windows – Right, 10µm thick Mo foil between two 40µm Applied Diamonds windows.

5-5 10µm-thick Mo in between two 100µm-thick Almax windows

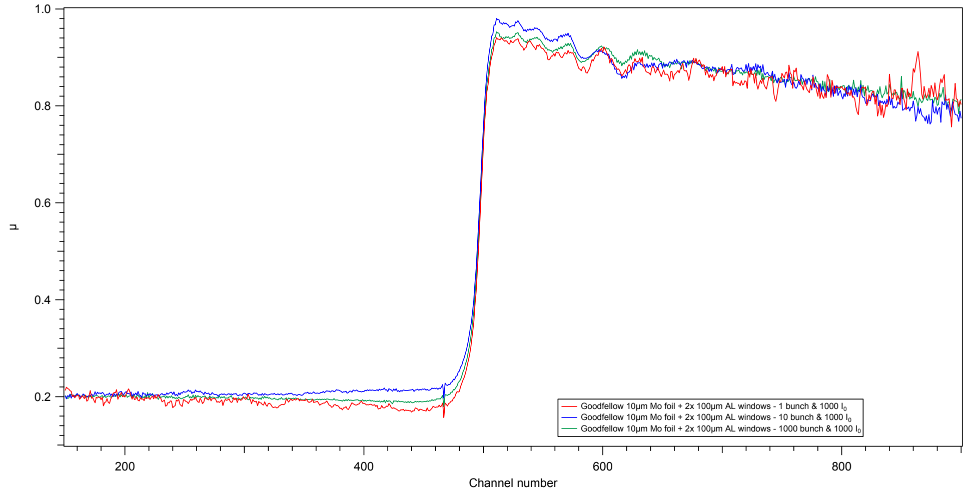
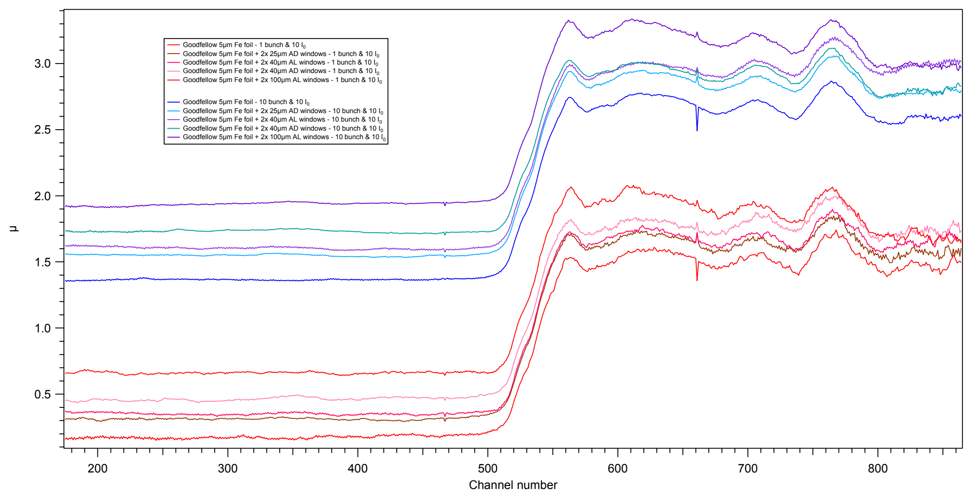



**Fig. 17:** 10µm thick Mo foil between two Almax 100µm windows.

## 6- Influence of the diamond windows

6-1 Fe case



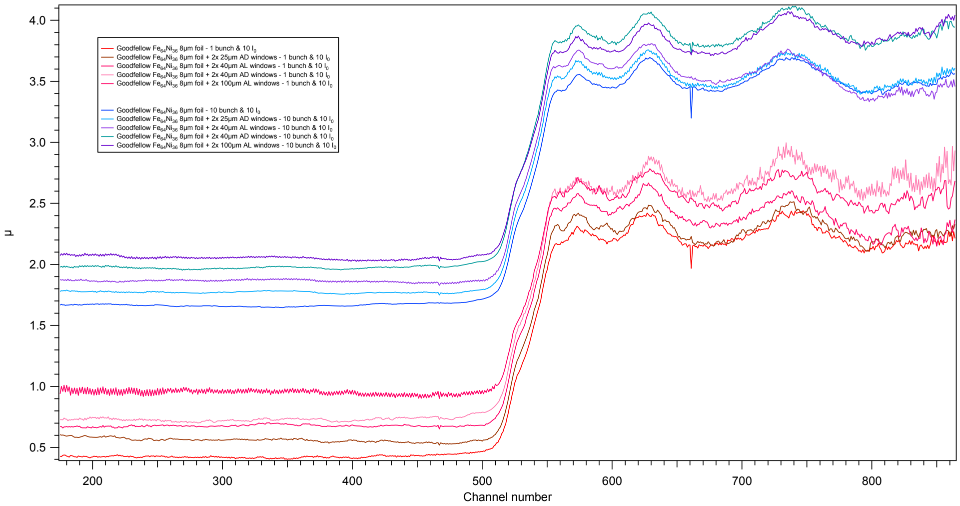



**Fig. 18:** Influence of the different diamond thicknesses on the Fe (Top) and on the $Fe_{64}Ni_{36}$ (Bottom) spectra qualities. The 10 bunches spectra (bluish curves) are offset by +1 for clarity.

6-2 Ni case

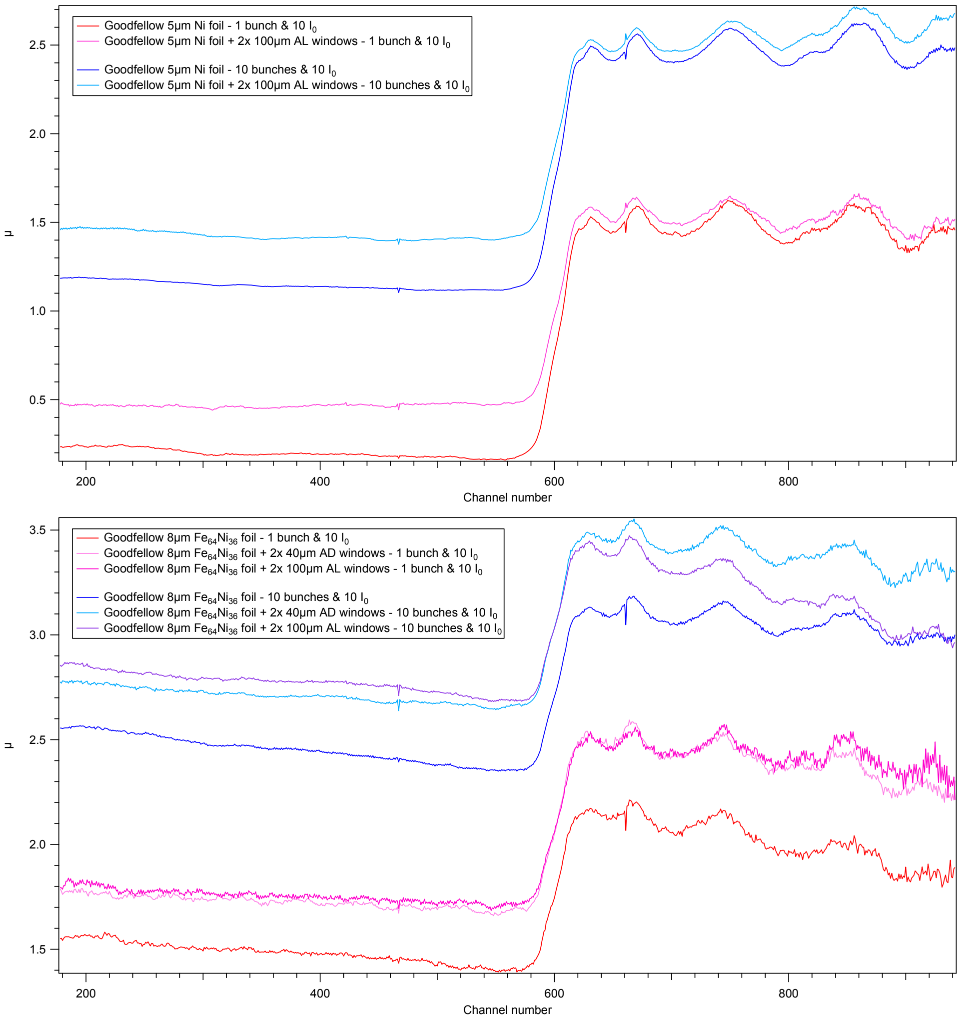



**Fig. 19:** Influence of the different diamond thicknesses on the Ni (Top) and on the $Fe_{64}Ni_{36}$ spectra qualities. The 10 bunches spectra (bluish curves) are offset by +1 for clarity.

6-3 Ta case

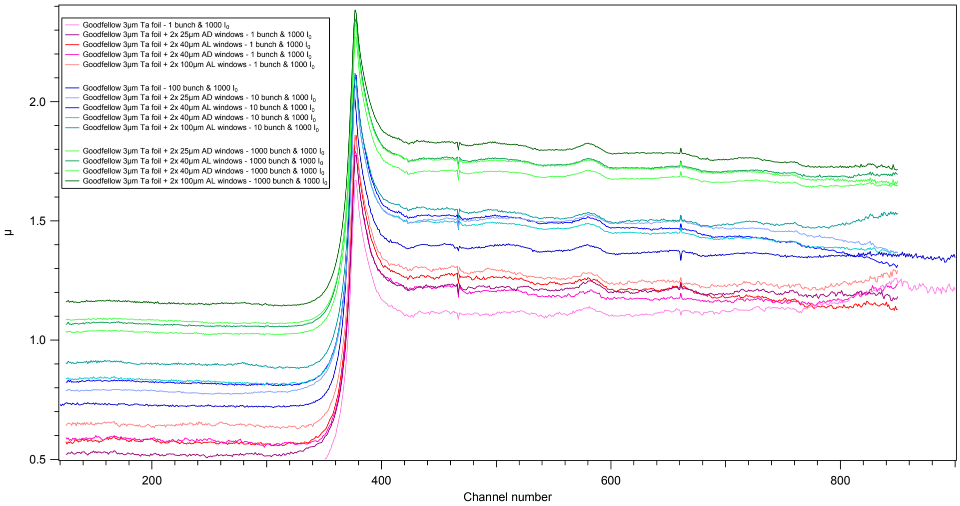



**Fig. 20:** Influence of the different diamond thicknesses on the Ta spectra qualities (3µm –thick foil). The 10 bunches spectra (bluish curves) are offset by +0.25, and the 1000 bunches spectra (greenish curves) are offset by +0.5 for clarity.

6-4 Mo case

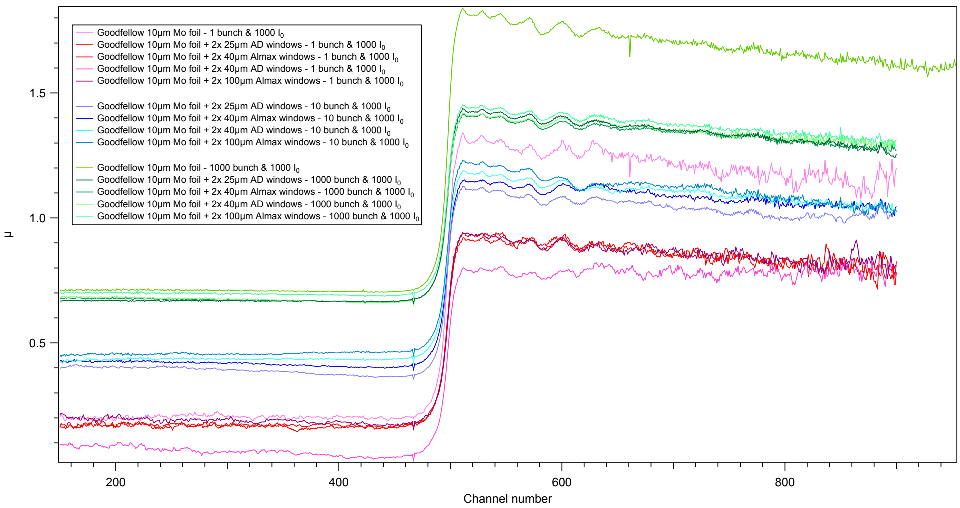



**Fig. 21:** Influence of the different diamond thicknesses on the Mo spectra qualities. The 10 bunches spectra (bluish curves) are offset by +0.25, and the 1000 bunches spectra (greenish curves) are offset by +0.5 for clarity.

# 7- Comparison between the diamond windows provided by the two different suppliers

7-1 Fe case

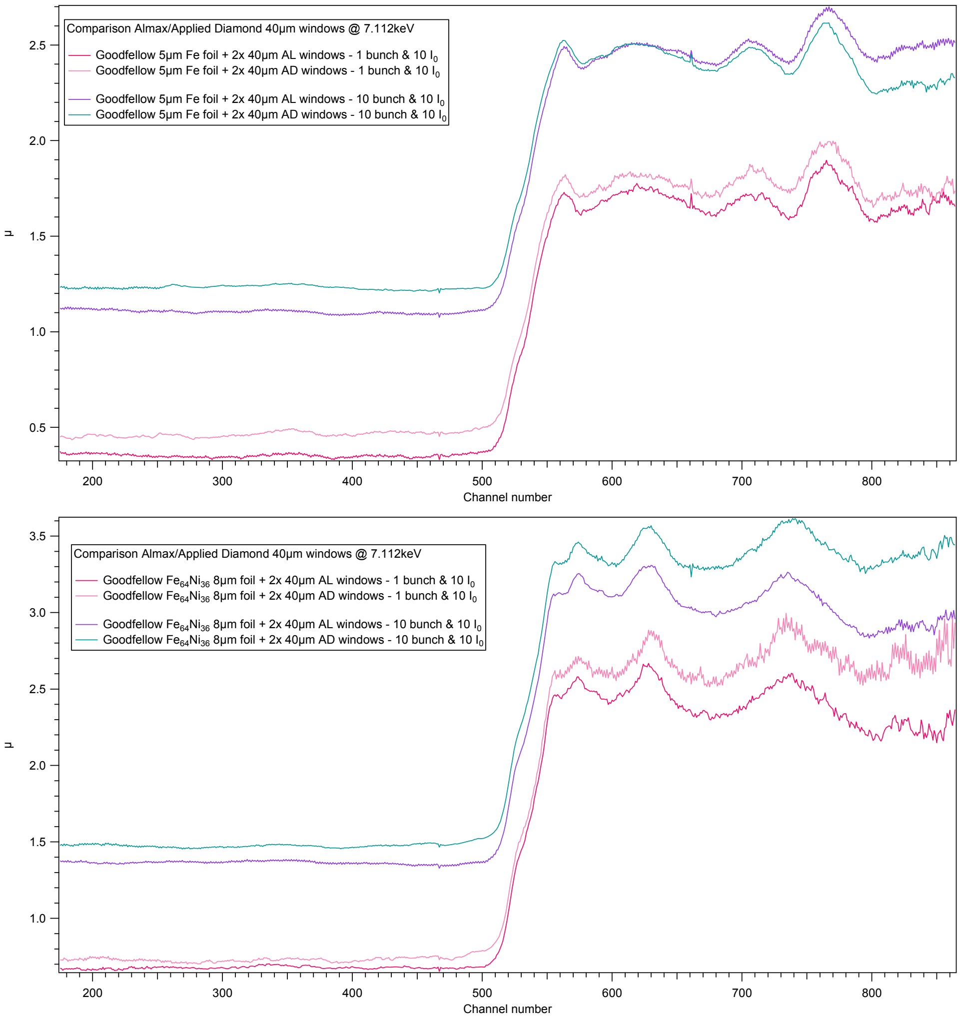



**Fig. 22:** Influence of the Almax and of the Applied Diamond 40µm-thick windows on the Fe (Top) and on the $Fe_{64}Ni_{36}$ (Bottom) spectra qualities. The 10 bunches spectra (bluish curves) are offset by +0.5 for clarity.

7-2 Ni case

This comparison hasn't been performed at the Ni K-edge.

7-3 Ta case

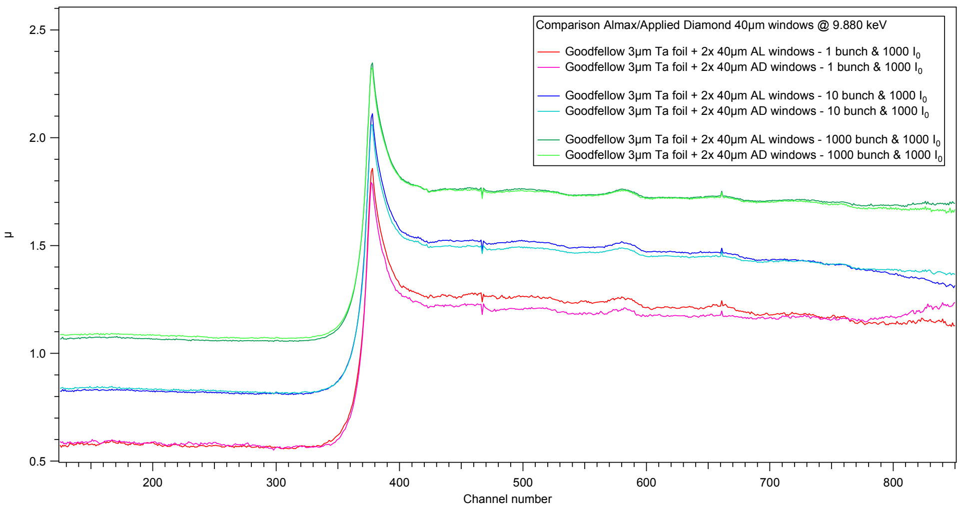



**Fig. 23:** Influence of the Almax and of the Applied Diamond 40µm-thick windows on the Ta spectra qualities. The 10 bunches spectra (bluish curves) are offset by +0.25, and the 1000 bunches spectra (greenish curves) are offset by +0.5 for clarity.

7-4 Mo case

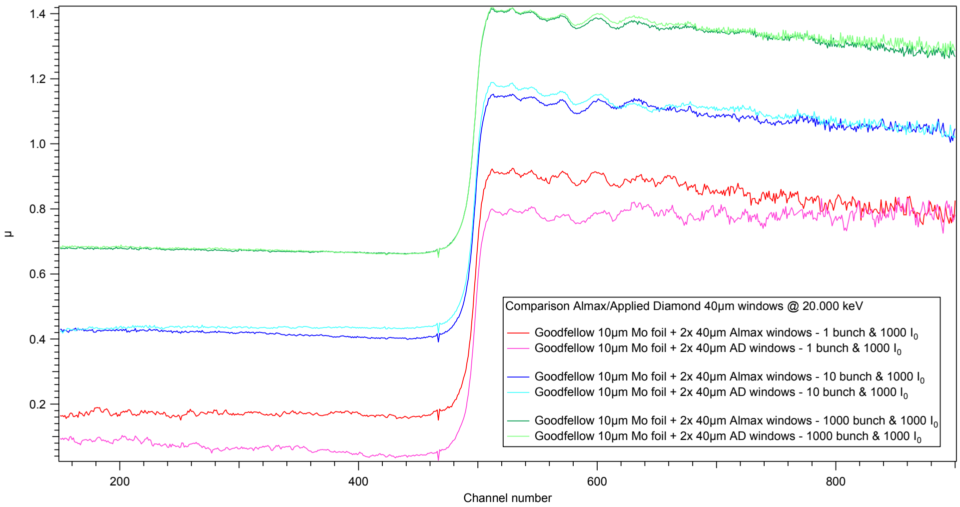



**Fig. 24:** Influence of the Almax and of the Applied Diamond 40µm-thick windows on the Mo spectra qualities. The 10 bunches spectra (bluish curves) are offset by +0.5 for clarity.

## 8- Conclusion

1- We noticed only the macro "rmspectrum" provides a synchronization scheme between the Machine and the XH detector.
2- This study proved it feasible to perform one-shot experiments at the four energy ranges we set up the beamline. In each case, data of good enough quality to extract physical information were recorded. Though, data recordings from one bunch are noisy while those from 10 bunches are perfect. If needed (e.g. if a fine effect needs to be studied), we will have to reproduce 10 times the same experimental conditions in order to be able to average 10 identical spectra.
3- The diamond windows (even the thickest ones) proved altering very little the spectra. Though an effect is seen, the data usability is not at stake. No bragg peaks from the diamond window were seen on any spectrum at any energy range. There was little noticeable differences between the data collected on samples emmbedded in 40µm-thick Applied Diamond windows and in 40µm-thick Almax-EasyLab windows : the spectra are slightly better with Almax windows at the Fe K-edge, and slightly better wit Applied Diamond windows at the other energy ranges. But it may also be due to the ESRF machine conditions, which we didn't keep record of. But overall, the windows provided by the two suppliers can be said of equivalent quality. Though the Applied Diamond windows are less expensive than the Almax windows (75$ vs 100€ for 40µm-thick items), we (at the CEA) decided to purchase Almax windows to pursue this project because of the shorter delivery time, and because we know the Almax windows work fine in the real shock conditions.