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# Deuterium retention in the divertor tiles of JET ITER-Like Wall


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## Abstract

Divertor tiles removed after the second JET ITER-Like Wall campaign 2013-2014 (ILW-2) were studied using Secondary Ion Mass Spectrometry (SIMS). Measurements show that the thickest beryllium (Be) dominated deposition layers are located at the upper part of the inner divertor and are up to ~40 µm thick at the lower part of the High Field Gap Closure tile exposed in 2011-2014. The highest deuterium (D) amounts (> $1 \cdot 10^{19}$ at./cm$^2$), in contrast, were found on the upper part of the tile 1, where the Be deposits are ~10 µm thick. D was mainly retained in the near-surface layer of the Be deposits but also deeper in tungsten (W) and molybdenum (Mo) layers of the marker coated tiles, especially at W-Mo layer interfaces. Results for the ILW-2 inner divertor tiles are higher than for the first campaign 2011-2012 (ILW-1) and one probable reason for the difference is that SIMS measurements for the ILW-2 samples were done deeper than for the ILW-1 samples. One can not rule out the differences between in ILW-1 and ILW-2 campaigns e.g. in strike point distributions and injected powers.



Keywords: JET, fuel retention, erosion, deposition



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## 1 Introduction

Retention of the plasma fuel in the plasma facing components (PFC) has a key role affecting economical and safe operation of a fusion reactor. Due to the radioactivity of tritium (T), its accumulation in the PFCs has to be restricted below a pre-determined safety limit, e.g., 700 g in ITER. When the T amount in the vessel structures is too high, they need to be cleaned and that will cause breaks to the operation.

Fuel retention in an ITER-like environment has been studied at JET since 2011 in connection with the JET ITER-Like Wall (ILW) project [1] where the same metallic first wall material mix as in ITER is used: tungsten (W) in the divertor and beryllium (Be) in the main chamber walls. The first JET-ILW operational period in 2011-2012 (ILW-1) was followed by a shutdown in 2012 during which a poloidal selection of first wall and divertor tiles were removed for post-mortem analyses. In the global gas balance experiments during the campaign and post-mortem analyses after the campaign, fuel retention was observed to be reduced by a factor of 10-20 compared to the previous JET operation with the all-carbon wall (JET-C) [2-4]. The highest deposition of Be and the largest amounts of retained fuel were found on Tiles 0 and 1 in the upper inner divertor [3, 4].

In the next JET-ILW campaign in 2013-2014 (ILW-2), the injected power to the plasma discharges was higher and strike point positions different, which might have altered material migration, the composition of the deposited layers and the amount of retained fuel. In the 2014 shutdown, a number of tiles were removed for post-mortem analyses to study these effects. This work focuses on the poloidal retention profile of deuterium (D) on the divertor tiles removed during the 2014 shutdown.

## 2 Experimental methods

The deuterium concentrations were determined from the selected JET-ILW divertor tiles removed during the 2014 shutdown. The analyzed tiles and the so-called S-coordinate system are shown in the poloidal cross section of the divertor in Figure 1. Tiles 0, 3, 7 and 8 were exposed during both ILW-1 and ILW-2 campaigns while Tiles 1, 4 and 6 were exposed only during the ILW-2 period. The ILW-2 (ILW-1) campaign comprised of 19.4 (19) hours of plasma time from which 14.2 (13) hours were in the divertor phase and 5.2 (6) hours in the limiter phase. During the ILW-2 (ILW-1) the inner strike point was on Tiles 3 and 4 (Tile 3) and the outer strike point predominantly on Tile 6 (Tile 5). At the end of the ILW-2 campaign, there was a hydrogen campaign that comprised of about 300 plasma discharges. Hydrogen campaign reduces the activity and the fuel retention due to the isotopic exchange effects.

Tiles 1, 3, 4, 6, 7 and 8 had special marker coatings on top of a carbon fibre composite (CFC) substrate for erosion/deposition studies. Tiles 1, 3, 6, 7 and 8 had a CFC (bulk)/Mo (3 µm)/W (12 µm)/Mo (4µm)/W (4 µm) layer structure and Tile 4 had molybdenum as the plasma-facing material with a CFC (bulk)/Mo (3 µm)/W (12 µm)/Mo (4 µm) layer structure. Tile 0 was a regular tile with a CFC (bulk)/Mo (3µm)/W (10-15 µm) structure.

Cylindrical samples with a diameter of 17 mm were cut from the tiles using a hollow drill. The deuterium concentrations of the samples were studied with Secondary Ion Mass Spectrometry (SIMS) using a double focusing magnetic sector instrument VG Ionex IX-70S at VTT Technical Research Centre of Finland (VTT). A 5 keV $O_2^+$ primary beam was used and the intensities of the positive secondary ions at $m/z$ values of 1 (H), 2 (D), 9 (Be), 12 (C), 58 (Ni), 98 (Mo) and 183 (W) were measured as a function of time. The current of the primary beam was 500 nA and the beam was raster-scanned over an area of 300×400 µm$^2$. A 10 % electronic gate was used to avoid crater wall effects. After the SIMS measurements, the depths of the craters were measured with a profilometer. D-implanted Be, Mo and W samples were used for quantification of the SIMS measurements.

## 3 Results and discussion

### 3.1 Inner divertor

A SIMS depth profile from the lower part of Tile 0 (S-coordinate 135 mm) is shown in Figure 2a. The depth profile shows a ~40 µm thick Be dominated deposition layer on top of the W coating. A strong H peak and depletion of D is seen at the surface which is probably due to the H phase at the end of the ILW-2 campaign. The main D peak is located near the surface at about a depth of 2 µm. After the main peak, the intensity of the D signal remains constant throughout the deposition layer and there is no significant change in its intensity when moving to the W, Mo and C layers. Nickel originating from the Inconel-steel components of the vessel is also present in the layers. The CFC/Mo/W layer structure of Tile 0 can be seen at the SIMS depth profile: after the Be deposition layer there is ~10 µm thick W layer and thin Mo interlayer before the CFC substrate. SIMS depth profiles do not show sharp interfaces between the layers due to the surface roughness, crater wall effects, non-uniform deposition and mixing of the layers. This is best observed from the slow decay of the Be signal when proceeding deeper into the W and Mo layers. Penetration of Be to W/Mo structure has also been observed with GDOES analyses [5].

The thickness of the Be deposition layer is the largest in the lower part of Tile 0 and in the upper part of Tile 1. Tile 1 was exposed only during 2013-2014 and the thickest measured Be deposits there are about 10 µm. The thickness of the deposit decreases to ~2 µm when moving to the lower part of Tile 1. In addition to the regions with strong deposition, both Tiles 0 and 1 have areas where Be deposition is very low and the thickness of the deposition layer is almost zero. The intensity of the near-surface D peak is higher in the lower part of Tile 1 but after the peak the intensity decreases faster than in the upper part of the tile. In Tile 0, the intensity of the near-surface D peak is about the same in all the measurement points. In the regions with thick deposits, the D peak occurs at around 2 µm and the intensity stays at a high level deep inside the material, whereas in the low deposition regions, the D peak can be seen already at ~0.5 µm and the intensity decreases faster after the peak.

Figure 2b shows a SIMS depth profile from the center of Tile 3 (S=524 mm). The Be deposition layer on top of W is ~1 µm thick and the intensity of the Be signal decreases slowly, indicating rough surface and mixing of Be with W. Due to the H campaign, the H peak is again seen at the surface while the D peak now emerges at ~0.4 µm. The intensity of the D signal decreases slowly after the peak but the behavior of the D intensity in the deeper material can not be deduced because of the short, under 10 µm, measurement depth. The intensities of C and Ni show peaks at the surface and after that intensities decrease slowly to a low level. The W/Mo/W layer structure of the tile can be seen from the depth profiles.

On Tile 3, the thickness of the Be deposition layer is the highest, about 3 µm, in the upper part of the tile and decreases to about 1 µm at the lower part of the tile. Measurements from the upper part of Tile 3 show erosion of the plasma-facing W layer. For a measurement point from the S-coordinate 445 mm, the original 4 µm thick W layer is almost completely eroded. Optical microscopy from cross-sectional samples prepared for the upper part of Tile 3 also indicates only small remnants of the topmost W layer and also the Mo layer has partly been eroded in some areas [6]. The intensity of the near-surface D peak is about the same in all measurement points from Tile 3, but stays at a high level deep in the samples towards the upper part of the tile.

Figure 2c shows a SIMS depth profile from the shadowed part of Tile 4 (S=737 mm) in the corner of the inner divertor floor. Here, the Be deposition layer is less than 1 µm thick. The intensity of the Be signal at the surface is lower and decreases faster than in Figures 2a and 2b. Low deposition in the inner divertor corner during the ILW operation is different from the observations made after operations with the carbon-wall when deposits in this region were thick [7]. This is attributed to the lower main chamber source and the reduced chemical erosion of Be compared to C [8]. Similarly to Tiles 0, 1 and 3, the H peak is seen at the surface but now also the main D peak is visible very close to the surface. In addition to the surface H peak, the intensity of the H signal exhibits another peak around the same depth as the main D peak. The intensity of the D signal decreases fast to a low level after the peak. Deposited C and Ni are also seen at the surface. The intensity of Ni is very low and the intensities of Ni and C decrease faster to a low level than in Tiles 0, 1 and 3. Tile 4 has Mo as plasma-facing coating and deposited W is seen at the surface.

Overall, deposition of Be, C and Ni on Tile 4 is low – the thickness of the Be deposition layer is negligibly small in the measurement points taken from Tile 4. The depletion of D in Tile 4 is lower than in Tiles 0, 1 and 3, and the D peak is very close to the surface. There are significant differences in the intensity of the D peak in different measurement points from Tile 4. The intensity is low at the strike point area and high at both sides of the strike point and in the shadowed part of the tile. In the inner strike point area and in the shadowed part of the tile, the intensity decreases fast to a low level. The SIMS depth profiles from the outer end of the tile show also small peaks in intensity of D signal at the Mo-W interface.

The deuterium concentrations (in at./cm$^3$) were calculated from the SIMS depth profiles with the help of D-implanted Be, Mo and W samples, which were analyzed with Heavy Ion Elastic Recoil Detection Analysis (HI-ERDA), and then the D amounts (in at./cm$^2$) in the SIMS profiles were integrated from the D concentration profiles. Uncertainties in the quantification of the measurements are related to matrix effects arising from impurities and mixing of the materials, which might affect the secondary ion yields compared to the D-implanted Be, Mo and W samples.

For the samples from the lower part of Tile 0 and upper part of Tile 1, some of the measurements reached the CFC substrate. We did not have a D-implanted CFC sample and therefore deuterium amount in the CFC substrate could not be quantified. The results for these measurement points are integrated from the metallic layers before the intensity of the C signal starts to increase. The intensity of the D signal stays high in the C region and the real D amounts are thus higher than what is presented here. It is notable that the intensity of the D signal decreases fast to a low level only in some measurement points from the shadowed part of Tile 4 and the inner strike point region on Tile 4. In Tiles 0, 1, 3 and other parts of Tile 4, the intensity of the D signal stays relatively high even deep in the material and measurements could have been continued even deeper.

The results (in at./$cm^2$) for the ILW-2 inner divertor tiles are shown in Figure 3 with the results for the ILW-1 campaign [3, 4]. Results for most of the measurement points are calculated as an average of more than one measurements. The results for the ILW-2 tiles are generally higher than the data obtained after ILW-1, especially for Tiles 1 and 3, but the retention profile is quite similar. In Tile 0, the D amount is lower in the upper part of the tile, where the deposits are thin, and higher in the lower part of the tile where the thickest deposits were formed. For Tile 0, results from both the regions with thick Be deposits and with low deposition are shown in the figure. In Tile 1, the highest D amounts and the thickest deposits are in the upper part of the tile. In Tile 3, the D amounts are higher at both ends of the tile than in the inner strike point region at the central part of the tile. In Tile 4, the lowest D amount was measured from the inner strike point region around the s-coordinate 800 mm and the highest amount, which is clearly higher than for the ILW-1 results for Tile 4, at the inboard side of the inner strike point region, very close to the location of the lowest amount. At the outboard side of the inner strike point region, measured D amounts are also higher than for the ILW-1, whereas at both ends of the tile results are close to the ILW-1 results. Similarly to ILW-1, results show that the co-deposition of D is the main mechanism for the fuel retention in the inner divertor.

After the ILW-1, the highest deposition was measured on the lower part of Tile 0 and the upper part of Tile 1 [3, 4] and the thickest deposits were found on top of Tile 1 were the thickness was ~15 µm [9]. Change of the inner strike point position from Tile 3 (ILW-1) to Tile 4 (ILW-2) was expected to move ion fluxes downward and therefore the D fluxes and retention of D on Tiles 1, 3 and 4 might have increased compared to the ILW-1. On the other hand, measurements after ILW-2 show that the Be deposition is higher on the lower part of Tile 0 than on Tile 1. Due to the downward movement of the inner strike point, higher deposition on Tile 1 compared to Tile 0 would have been expected during ILW-2. One possible reason for the difference is the surface roughness of the tiles: Tile 1 was a new and had a clean W surface, whereas Tile 0 was already exposed to ILW-1 campaign and therefore had a rough Be dominated deposits on its surface at the beginning of ILW-2. Many studies, e.g. $^{13}C$ tracer experiments in TEXTOR [10], have shown that the surface roughness and material have significant effect to the deposition efficiency – rougher surface leads to higher deposition. The studied Tile 3 was also exposed during both ILW-1 and ILW-2, and longer exposure time possibly partly explains the higher result for Tile 3 after ILW-2.

The higher D amounts measured on ILW-2 samples may also be due to the fact that the SIMS measurements were made deeper than for the ILW-1 samples and as already mentioned, the intensity of the D signal was found to stay relatively high even deep inside the material. The measurement depth was more than 10 µm for most of the measurements and the longest measurements reached depths beyond 60 µm, whereas with ion beam analysis (IBA) the accessible depth for D analysis is typically < 10 µm. Variations caused by a non-uniform deposition were seen between the measurements from the same sample. It was shown after the ILW-1 using µ-IBA methods that D retention concentrated preferentially on cracks, pits and depressed areas on the surface and more than 70 % of trapped D was found in less than 30 % of the surface area [11].

### 3.2. Outer divertor

Figure 4a shows a SIMS depth profile measured close to the outer strike point at the center part of Tile 6 (S= 1475 mm) in the outer divertor floor. Be is seen at the surface mixed with W. The intensity of the Be signal at the surface is about the same as in Figures 2a and 2b, and it decreases slowly. W/Mo/W layer structure of the sample can also be seen in the figure. The thickness of the plasma-facing W layer is ~3.5 µm, so ~0.5 µm of the original W layer has been eroded at this point. Similarly to the inner divertor tiles, H peak can be seen on the surface and the D peak near the surface but the difference between the surface value and the peak value of the intensity is small. The intensity of the D peak is lower than in the SIMS depth profiles from the inner divertor tiles in the Figure 2 and intensity decreases fast to a low level. The intensities of C and Ni are similar to Fig. 2b from Tile 3 at the surface but decrease slowly when moved to deeper.

For most of the samples from Tile 6, the thickness of the Be deposits was less than 1 µm and the intensity of the W signal increased sharply close to the surface. The intensity of the Be signal decreased slowly, indicating rough surface and mixing of the Be with W/Mo structure. The thickest measured Be deposits were over 10 µm thick at S=1516 mm which is 29 mm outward from the predominant outer strike point location at the S= 1487 mm. In the outer strike point area at the central part of the tile, the intensity of the near-surface peak of D is low and intensity decreases fast to a low level. In the other parts of Tile 6, the intensity of the D peak is higher and the intensity decreases slowly.

Figure 4b shows a SIMS depth profile from near the center of Tile 7 (S= 1765.5 mm). The depth profile resembles the one from Tile 3 in the Fig 2b. Be layer on top of W is thinner than in Fig. 2b. The H peak is seen again at the surface and the D peak near to the surface. The near-surface D peak is narrower than in Fig. 2b and similarly to Fig. 4a, the difference between the surface and the peak values of the intensity of D signal is small. The intensity decreases slowly and stays clearly on a higher level than in Fig 4a. The intensities of the C and Ni are similar to Fig. 2b and decrease faster than in Fig. 4a.

Figure 4c shows a SIMS depth profile from the lower part of Tile 8 (S-coordinate 1877 mm). Be is seen at the surface and its intensity decrease fast. The surface H peak is about similar in all the samples and it is likely that besides the H experiments at the end of ILW-2 also air exposure after the removing of the tiles from the vessel has contributed to this. Similarly to figures 4a and 4b, the D peak is very close to the surface and the difference of the peak value and the surface value of the intensity is small. In addition to the near-surface peak, other D peaks are seen at the W-Mo and Mo-W interfaces. The intensities of the C and Ni are similar to Figures 4a and 4b at the surface but decrease faster in Figure 4c.

In the outer divertor Tiles 7 and 8, the thickness of the Be deposits is less than 1 µm. In addition to the near-surface peak, SIMS depth profiles from Tiles 7 and 8 typically show also peaks in the intensity of D signal at the W-Mo and Mo-W interfaces. The intensity of these interface peaks are the highest in the lower part of Tile 8. The D retention in the W-Mo and Mo-W interfaces was also observed after the ILW-1 and was found to be dominant retention mechanism in the outer divertor tiles [12].

The results for the outer divertor tiles are not calculated yet, but the change of the predominant outer strike point position from Tile 5 (ILW-1) to Tile 6 (ILW-2) possibly increased ion fluxes to outer divertor Tiles 6, 7 and 8 compared to the ILW-1. Notable is also that the D signal decreases fast to a low level only in measurement points from the outer strike point region in Tile 6. In the other parts of Tile 6 and in Tiles 7 and 8, the D signal decreases slowly and stays relatively high deep in the material, which indicates that final results will probably be higher than for the ILW-1 measurements.

## 4 Conclusions

The deuterium amounts (at./cm$^2$) were determined from the selected ILW-2 divertor tiles removed during the 2014 shutdown using SIMS. Similarly to ILW-1, the areas of the highest Be deposition in the inner divertor were found from the lower part of Tile 0 and the upper part of Tile 1. The highest deuterium amounts were > $1\cdot10^{19}$ at./cm$^2$ in the upper part of Tile 1, which was exposed only during ILW-2. Be dominated deposition layers were the thickest, up to ~40 µm, in Tile 0, which was exposed during both the ILW-1 and ILW-2 campaigns. The thickness of the Be deposition layer in inner divertor Tiles 3 and 4 was 0-3 μm and in the outer divertor tiles principally smaller than 1 μm, except in the outer divertor floor Tile 6 where deposits with thicknesses >10 μm were found ~30 mm outward from the predominant outer strike point position.

Deuterium was found to be retained in the near-surface layer, thick Be deposition layers and W-Mo interfaces but the intensity of the D signal stayed relatively high also deeper in the material except some parts of the divertor floor Tiles 4 and 6. Depletion of D was seen at the surfaces of the tiles, likely due to the H experiments at the end of ILW-2. Measured D amounts in the inner divertor tiles were generally higher than the results for the ILW-1 campaign. Possible reasons for the higher results are that measurements of the ILW-2 samples were done deeper compared to the ILW1-samples and changes in the strike point distribution and the plasma conditions between ILW-2 and ILW-1. ILW-2 campaign contained a larger number of high-power plasma pulses than ILW-1 and downward movement of the inner strike point position from Tile 3 to Tile 4 might have increased D fluxes to the inner divertor tiles.

**Acknowledgments**

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**Figure Captions**

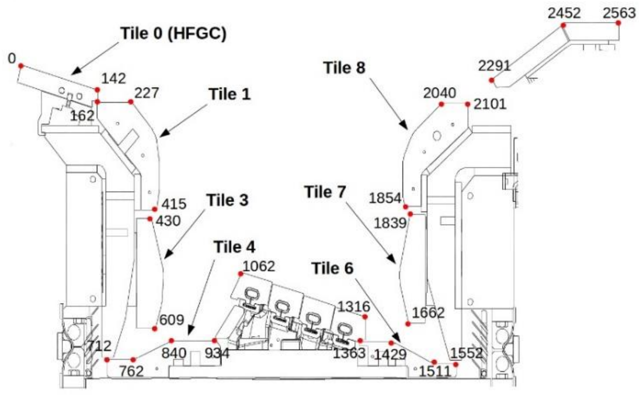
Figure 1. The JET-ILW divertor tile configuration and the S-coordinate system.

Figure 2. SIMS depth profiles from a) Tile 0 (S-coordinate 136 mm), b) Tile 3 (S-coordinate 524 mm) and c) Tile 4 (S-coordinate 737 mm).

Figure 3. D amounts (at./$cm^2$) in the inner divertor tiles for the ILW-2 and ILW-1 campaigns.

Figure 4. SIMS depth profiles from a) Tile 6 (S-coordinate 1475 mm), b) Tile 7 (S-coordinate 1765.5 mm) and c) Tile 8 (S-coordinate 1877 mm).

## Figures

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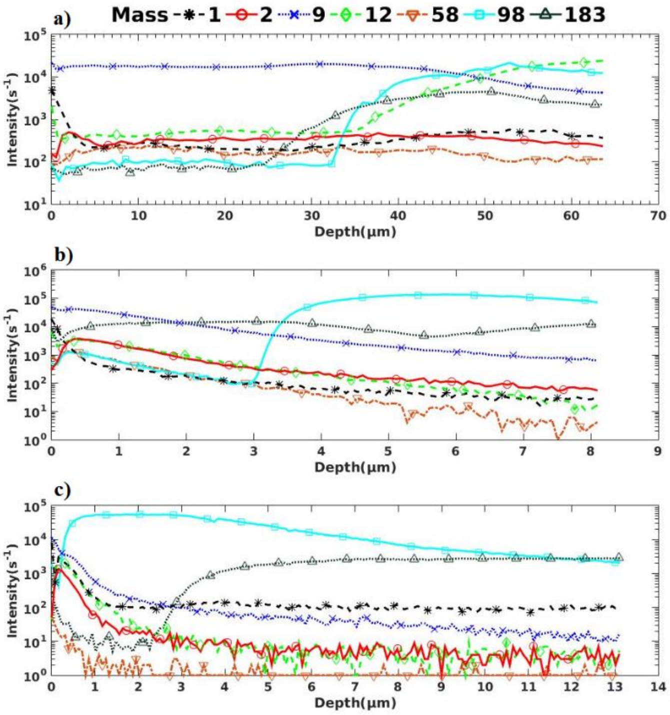


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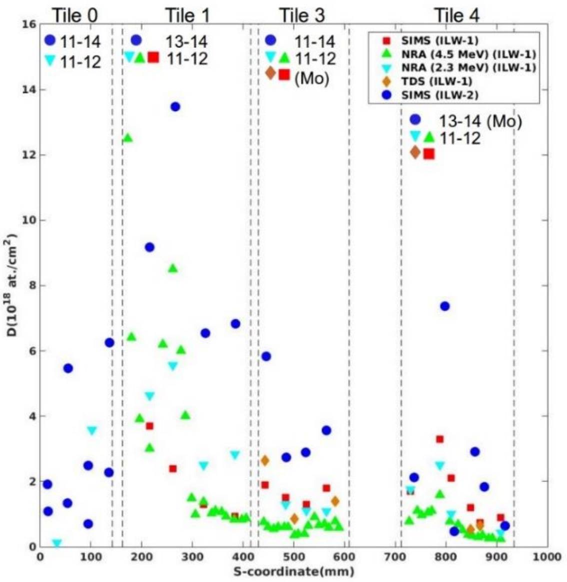


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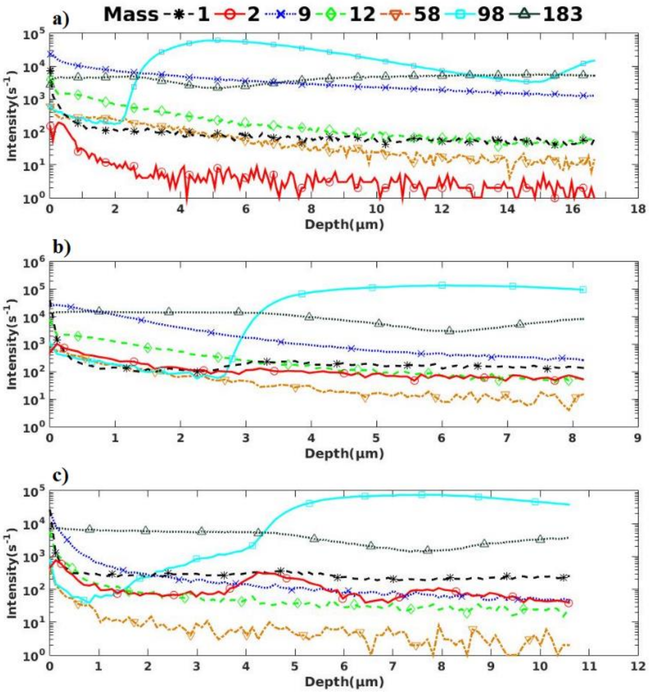


Figure 4. SIMS depth profiles from a) Tile 6 (S-coordinate 1475 mm), b) Tile 7 (S-coordinate 1765.5 mm) and c) Tile 8 (S-coordinate 1877 mm).