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ABSTRACT

MkIIGB divertor tiles exposed in JET for the 1998-2001 and 1999-2001 campaigns have been used to assess the amount of beryllium and carbon deposited at the inner divertor wall. Deposited ˚lms at the inner wall of the divertor tiles have a duplex nature. There is an inner layer rich in carbon and beryllium, whereas the outer layer is rich in deuterium and carbon. Total amount of beryllium at the inner divertor tiles was determined and integrated toroidally. Results were compared with spectroscopic data and good agreement was obtained. The amount of carbon was computed assuming that the ratio of carbon to beryllium arriving in the divertor is the same as the observed source ratio in the main chamber. On the basis of this analysis we would expect there to be ~0.4kg of carbon deposited. This gives an average carbon deposition rate lower than during the MkIIA phase.

1. INTRODUCTION

Previous analyses of plasma-facing tiles removed from JET have shown that material is eroded from outer divertor and vessel walls [1-5]. The material is mostly carbon from the tiles, plus beryllium (evaporated over the tiles and other in-vessel surfaces periodically from sources within the vessel), and metals from the nickel-based vessel walls. Eroded material flows round the Scrape-Off Layer (SOL) from outboard to inboard and deposits in the inner divertor together with hydrogen isotopes in the plasma. Carbon is chemically sputtered from the inner divertor deposits, leaving behind films rich in beryllium and other metals that cannot be chemically sputtered, and is transported to regions shadowed from the plasma, such as the inner louvres. From the fact that the layers deposited in remote areas contained vast amounts of hydrogen isotopes and carbon, but no beryllium, one concluded that their presence was associated with chemical erosion of carbon and long-range transport of hydrocarbons [1]. No re-erosion of Be and metals occurs due to the low electron temperature in the inner divertor. For the JET Mk IIA campaign the total amount of deposition in the divertor (~1kg) was obtained from the long term tritium retention (resulting from the DTE1 campaign) and the tritium content of the flakes recovered from the inner pump duct [1]. However, for the Mk IIGB divertor it is difficult to get a reasonable estimate of the re-deposited carbon and therefore an attempt has been made to estimate the overall deposition from the amount of beryllium left on the inner divertor wall tiles [6].

2. EXPERIMENTAL

The Gas Box divertor (Mk IIGB) was first installed in 1998 in exchange for the Mk IIA divertor tiles that were in use 1996-1998. During the period with the MkII GB configuration there was an intervention in 1999 when a poloidal set of divertor tiles was removed and replaced with a special set of marker tiles. Operations with the MkII GB were completed in February 2001. The special marker tiles installed in 1999 were retrieved for analysis during this shutdown. Another full set of divertor tiles which was exposed from 1998 to 2001 also became available for surface analyses. JET is operated with Plasma Facing Components (PFC) made of carbon fibre composite (Concept I manufactured by Dunlop Ltd). The special marker tiles consisted of two layers (prepared by Plansee

AG, Austria); firstly, a thin layer (0.5 μm) of rhenium (Re), and secondly, a layer of a 90% carbon/ 10% boron mixture 2.5 μm thick. The carbon/boron layer is designed to be similar in composition to the in-vessel tile surfaces so that there is minimal distortion of plasma transport processes, yet allows erosion of the layer to be assessed from the amount of boron detected.

A set of samples each of 17 mm in diameter was cut from the divertor tiles using a coring technique [7,8]. The poloidal positions of the samples are shown in Fig.1 Tiles 1 and 4 have, however, been analysed by Rutherford Backscattering Spectrometry (RBS) without cutting. Surface analysis of the tiles has been discussed earlier [2] and will be discussed only briefly here. RBS measurements were carried out using the 3MeV Van de Graaff accelerator of the University of Sussex. A 2.5MeV proton beam was used in the measurements and backscattered particles were detected with a Si detector placed at a scattering angle of 170º. Secondary Ion Mass Spectrometry (SIMS) analysis of core samples was made with a double focussing magnetic sector instrument (VG Ionex IX-70S) at VTT. The current of the 5 keV O_2^+ primary ions was typically 500 nA during depth profiling and the ion beam was raster-scanned over an area of $300 - 430 m^2$. Layer thicknesses were calculated using sputter rates determined with a profilometer (Dektak 3030ST) from redeposited layers on tile 1 and 3 separately for the carbon and beryllium-rich layers. Some selected SIMS samples were measured with TOF-ERDA to obtain elementary concentrations at the near surface region. In the measurements, the 5MV tandem accelerator EGP-10-II with a 53 MeV beam of $^{127}I^{10+}$ ions of the University of Helsinki was used. The detector angle was 40^o with respect to ion beam and the samples were tilted relative to the beam direction by 20^o. TOF-ERDA gave quantitative amounts of elements up to a depth of about 1μm.

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) was used for chemical analysis of beryllium in the inner divertor tiles 1 and 3. The samples cut from tiles 1 and 3 were about 2 mm thick with a surface area of $2cm^2$. These samples were dissolved using both nitric and sulphuric acid.

3. RESULTS

SIMS depth profiling has been made from a number of samples on inner divertor tiles 1, 3 and 4. Figure 2 shows typical depth profiles from marker tiles which were exposed from 1999 to 2001 at JET. For each of the profiles, the rhenium containing marker layers are clearly visible at the interface between a deposited film and the CFC substrate. The deposit forms two layers on tiles 1 and 3. The outer layers (approx. 2-6μm thick on tile 1 and 10-16μm on tile 3) contain mostly carbon together with deuterium and smaller amount of beryllium. The surface composition is similar to that found in deposits at the sides of limiters in the main chamber, and presumably to that arriving at the inner divertor along the SOL. The films underneath the surface layer are very rich in beryllium. On tile 1 the layer thickness of the inner beryllium rich film varies between 2 and 14μm, and on tile 3 between 12 and 21μm. It thus appears that during the last part of the campaign, the carbon was no longer being chemically sputtered from the films.

The film in the shadowed region on tile 4 (sample 4/10, Fig.1(c)) is relatively pure carbon, with a very high deuterium content, and with a well-marked interface to the CFC substrate. Beryllium

content in the deposit is very low. The film is ~85μm thick, with a similar composition to that previously found for the flaking deposits at the inner louvres [9]. Deposits on the part of tile 4 shadowed by the septum are somewhat thinner (~15μm), and do not have quite as high deuterium content. The divertor inner wall tiles 1 and 3 exposed in 1998-2001 contain similar deposits with a double layer structure. The layer thicknesses of the outer carbon rich and inner beryllium rich layer are, however, greater than on the marker tiles exposed in 1999-2001.

For ion beam analyses tile 1 is most interesting because the deposit is thin enough to analyse most of it. In the case of tile 3 the deposit is too thick and the outermost few microns only will be detected. Figure 3 shows a RBS spectrum measured from the bottom of tile 1 (sample 1/1). Spectrum was simulated using SIMNRA program [10]. Best agreement with the experimental spectrum was obtained by using three different layers in the simulations. There are clearly three identifiable layers from SIMS data; a thin outer layer containing the ^{13}C, and the two parts of the main film. The thin ^{13}C layer on the surface is due to $^{13}CH_4$ which was injected into the plasma boundary in the last day of discharges prior to the 2001 shutdown [8]. The thickness and ^{13}C content of the outer layer is determined by the fit to the ^{13}C feature indicated in Fig.3, whilst the second layer thickness and ^{12}C content is first estimated by getting a reasonable fit to the ^{12}C feature arrowed in the spectrum. The thickness of the third layer is first estimated by fitting the width of the beryllium feature in the figure, then the beryllium and carbon contents of the various layers are adjusted iteratively until a best fit to the overall spectrum is obtained. Composition of the deposit is given in Table 1.

Ion fluxes measured with divertor Langmuir probes seem to be higher for tile 1 during 1999-2001 campaign than for 1998-1999 campaign. Langmuir probe measurements indicate that the 1998-1999 campaign contributes only 14% of the total flux to tile 1 for 1998-2001, but for tile 3 the contributions of the 1998-1999 and 1999-2001 campaigns are almost equal. This may imply that the thickness of the deposit on tile 1 formed during 1998-2001 and 1999-2001 campaigns should be comparable, whilst tile 3 should have twice the thickness of beryllium rich film seen on the equivalent 1999-2001 tile if films are additive. SIMS, RBS and ICP-MS analyses show that the films are thicker on the tiles exposed in 1998-2001, but SIMS measurements show that both the carbon rich outer layer and the carbon and beryllium oxide rich inner layers are similarly increased. In Table 2 ratios of total beryllium amounts on -98 tile to that on -99 tile are given from RBS and ICP-MS measurements, plus the ratio of thickness of the beryllium rich layers from SIMS. The deposited films are too thick on sample 1/8 and on tile 3 for RBS to determine the ratio, as the analysis depth is about 8-10 μm. All the results indicate that the deposits are thicker on tile 1 and the results do not quite correlate with Langmuir probe results, especially in the case of samples 1/4 and 1/8. This suggests that the impurity profile within the SOL differs from the ion flux profile which is dominated by deuterium. There is some scatter in the results obtained with the different techniques. There could be some errors in the determination of sputter rates in SIMS analyses. RBS results are based on a certain density value for the deposit which may not be correct, and in ICP-MS analyses there is always the possibility that not all beryllium is dissolved.

In an attempt to calibrate Be/C signal ratios for SIMS, RBS spectra for both tiles 1 exposed in 1998-2001 and 1999-2001 were simulated with SIMNRA, and the compositions of the deposits were calculated. SIMS Be/C signal ratios both for the inner and outer layers were plotted as a function of beryllium amount measured with RBS and a linear fit was made (see Fig.4). Beryllium amounts on analysed samples from tiles 1 and 3 exposed in 1999-2001 were calculated using layer thicknesses determined with SIMS and compositions of layers from RBS, TOF-ERDA and SIMS analyses, and then integrated over the tile surface areas. A value of 1.6 g/cm^3 was used as a density of the deposits. The total amount of beryllium at the inner divertor can then be obtained by assuming uniform concentrations toroidally.

The amounts of beryllium on tiles 1 and 3 integrated around the torus are thus estimated as 7 and 15 g, respectively. Therefore, the estimate for the total amount of beryllium at the inner divertor deposited during the 1999-2001 campaign is 22 g. The uncertainty of the beryllium amount on tiles 1 and 3 is estimated to be 40%. This does not include possible toroidal asymmetry.

4. DISCUSSION

According to the analyses the films on inner divertor wall tiles 1 and 3 are thicker on the tiles exposed from 1998-2001 than on tiles exposed from 1999-2001, but both the carbon and deuterium rich outer layer and the carbon and beryllium oxide-rich inner layers are similarly increased. It could be that the outer region represents a modification of a film which was previously entirely beryllium rich. During some operation phase at the end of the campaign (e.g. the change in temperature, or operation in helium) chemical erosion was reduced at the inner divertor.

It seems reasonable that at deposition dominated areas in the inner divertor, the films on tiles exposed from 1998-2001 should be a composite of the film found on the 1999-2001 tiles on top of the film deposited during the 1998-1999 campaign. However, another possible scenario is that there is a certain amount of erosion and re-deposition of elements at the inner divertor walls before they are finally buried within the surface films. Each erosion/re-deposition step involves toroidal transport of the order of centimetres, so the clean tiles installed in 1999 may end up with a typical tile analysis for that poloidal location, not an analysis characteristic only of its period in the vessel. On the other hand, the floor tile 4 gives rather different analyses in the cases of campaigns 1998-2001 and 1999-2001, which cannot be explained by exposure time. Another possibility is therefore a toroidal variation in tile analysis. Thus, further post-mortem surface analyses are required in the future in order to get more information on material transport at JET.

The picture we have of material migration in JET is erosion of carbon and beryllium from the main chamber, ionisation in the SOL and transport mainly to the inner divertor driven by parallel flows. The primary source of beryllium in JET is thought to be the periodic evaporation. Carbon is subsequently eroded due to chemical sputtering. Spectroscopic measurements show that carbon is eroded and re-deposited about ten times before being finally deposited at remote areas in the divertor [6]. Beryllium does not, however, appear to migrate significantly within the divertor. The amount of carbon deposited

at the inner divertor in Mk IIA from 1996-1998 was estimated as ~1kg, from the amount of tritium co-deposited with the carbon [1]. Since there was no tritium used during the MkIIGB phase in 1998-2001, the amount of deposition is being estimated from the beryllium build-up found on tiles 1 and 3 which are the only sinks for beryllium. This result is combined with the assumption that on average the beryllium arriving at the divertor is the same fraction of the average impurity flux as that in the plasma. For the first pulse immediately after a beryllium evaporation there is a large beryllium plasma impurity fraction, but this rapidly drops to just a few per cent of the carbon, and the average beryllium level is ~7% of the carbon [6]. As mentioned in the previous section, the total amount of beryllium deposited during the MkIIGB phase (1999-2001) is estimated to be 22g at the inner divertor wall tiles 1 and 3 integrated over the torus. If we then take into account the beryllium impurity level of ~7% of the carbon in the plasma, we obtain ~ 400g of deposited carbon.

Beryllium amounts from post-mortem tile analysis have earlier been correlated with calculations of beryllium flux from spectroscopic measurements for Mk IIGB [6]. According to spectroscopy data the wall carbon source was estimated to be 390-480g and the beryllium wall source 20g. Previous tile analyses predicted that the net carbon and beryllium deposition in the JET divertor is 2 to 4 times greater than the spectroscopy data for the main chamber sources. It was suggested that there may some other source mechanisms such as material transport due to ELMs, or erosion of the Be ICRH antenna screens, that are not correctly accounted for spectroscopically. However, the earlier estimates were made before proper calibration of SIMS Be/C ratios was available, which resulted in over-estimates of the amounts of beryllium and carbon deposited at the inner divertor. After calibration of SIMS, the results are now much more in line with the spectroscopy data. Both surface analyses and spectroscopy data now indicate that the average deposition rate during the MkIIGB phase (1999-2001) is somewhat lower than during MkIIA phase (1996-1998) when the total deposition was estimated from tritium measurements to have been 1kg.

CONCLUSIONS

A double layer structure, with a carbon-rich outer layer and a carbon- and beryllium-rich inner layer, has been observed on JET inner divertor tiles 1 and 3 removed in 2001 by post-mortem surface analyses. The amount of beryllium deposited on the inner divertor wall tiles has been calculated, and by assuming that the ratio of carbon to beryllium arriving in the divertor is the same as the ratio in the main chamber, the amount of carbon deposited has been computed. On the basis of this analysis, the amount of carbon deposited is ~0.4kg. This gives an average deposition rate which is somewhat lower than that during the MkIIA phase at JET.

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REFERENCES

[1]. J.P. Coad, N. Bekris, J. D. Elder, S.K. Erents, D.E. Hole, K.D. Lawson, G.F. Matthews, R. -D. Penzhorn and P. C. Stangeby, J. Nucl. Mater. **290-293** (2001) 224.

[2] J.P. Coad, P. Andrew, D.E. Hole, S. Lehto, J. Likonen, G.F. Matthews, M. Rubel, J. Nucl. Mater. 313-316 (2003) 419.

[3]. R.-D. Penzhorn, N. Bekris, U. Berndt, J.P. Coad, H. Ziegler and W. Nägele, J. Nucl. Mater. 288 (2001) 170.

[4]. N. Bekris, C.H. Skinner, U. Berndt, C. A. Gentile, M. Glugla and B. Schweigel, J. Nucl. Mater. **313-316** (2003) 501.

[5]. M. Rubel, J.P. Coad, N. Bekris, S. K. Erents, D. Hole, G. F. Matthews, R. -D. Penzhorn J. Nucl. Mater. 313-316 (2003) 323.

[6]. G.F. Matthews, M.F. Stamp, P. Coad, J. Likonen, V. Philipps, M. Rubel, J.D. Strachan, Proceedings of the EPS Conference on Controlled Fusion and Plasma Physics, St. Petersburg, Russia, 7-11 July 2003.

[7]. R.-D. Penzhorn, N. Bekris, P. Coad, L. Dörr, M. Friedrich, M. Glugla, A. Haigh, R. Lässer and A. Peacock, Fusion Engineering and Design, **49-50** (2000) 753.

[8]. J. Likonen, S. Lehto, J.P. Coad, T. Renvall, T. Sajavaara, T. Ahlgren, D.E. Hole, G.F. Matthews, J. Keinonen, Fusion Engineering and Design 66-68 (2003) 219.

[9]. J.P.Coad, P.L.Andrew, et al., 26th EPS Conference on Controlled Fusion and Plasma Physics, Maastricht, June, 1999.

[10]. M. Mayer, SIMNRA User's Guide, Report IPP 9/113, Max-Planck-Institut für Plasmaphysik, Garching, Germany, 1997.

Layer	D, at.%	Be, at.%	^{12}C, at.%	^{13}C, at.%	O, at.%	Cr, at.%
1	11	5	27	30	27	0
2	30	5	53	0	10	2
3	5	31	33	0	28	3

Table 1. Composition of the deposit at the bottom of tile 1 exposed in 1999-2001 measured with RBS.

Sample	SIMS (98/99)	RBS (98/99)	ICP-MS (98/99)
1/1	1.1	1.7	1.3
1/4	2.6	2.5	2.0
1/8	1.6		1.1
3/1	1.7		1.3
3/4	1.5		1.5
3/8	1.3		2.4

Table 2. Ratio of Be rich layer thickness in -98 samples to that in -99 samples measured with SIMS and ratio of Be amount in -98 samples to that in -99 samples measured with RBS and ICP-MS.

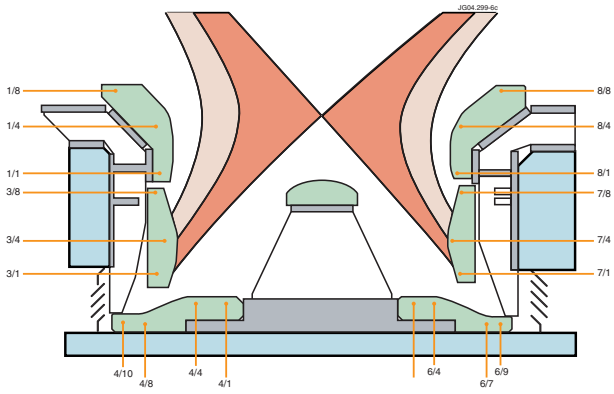

Figure 1. The JET MkIIGB divertor tile set. The samples for SIMS and IBA measurements are indicated with numbers. The first number in the sample code refers to divertor tile and the second one to the position in the tile.

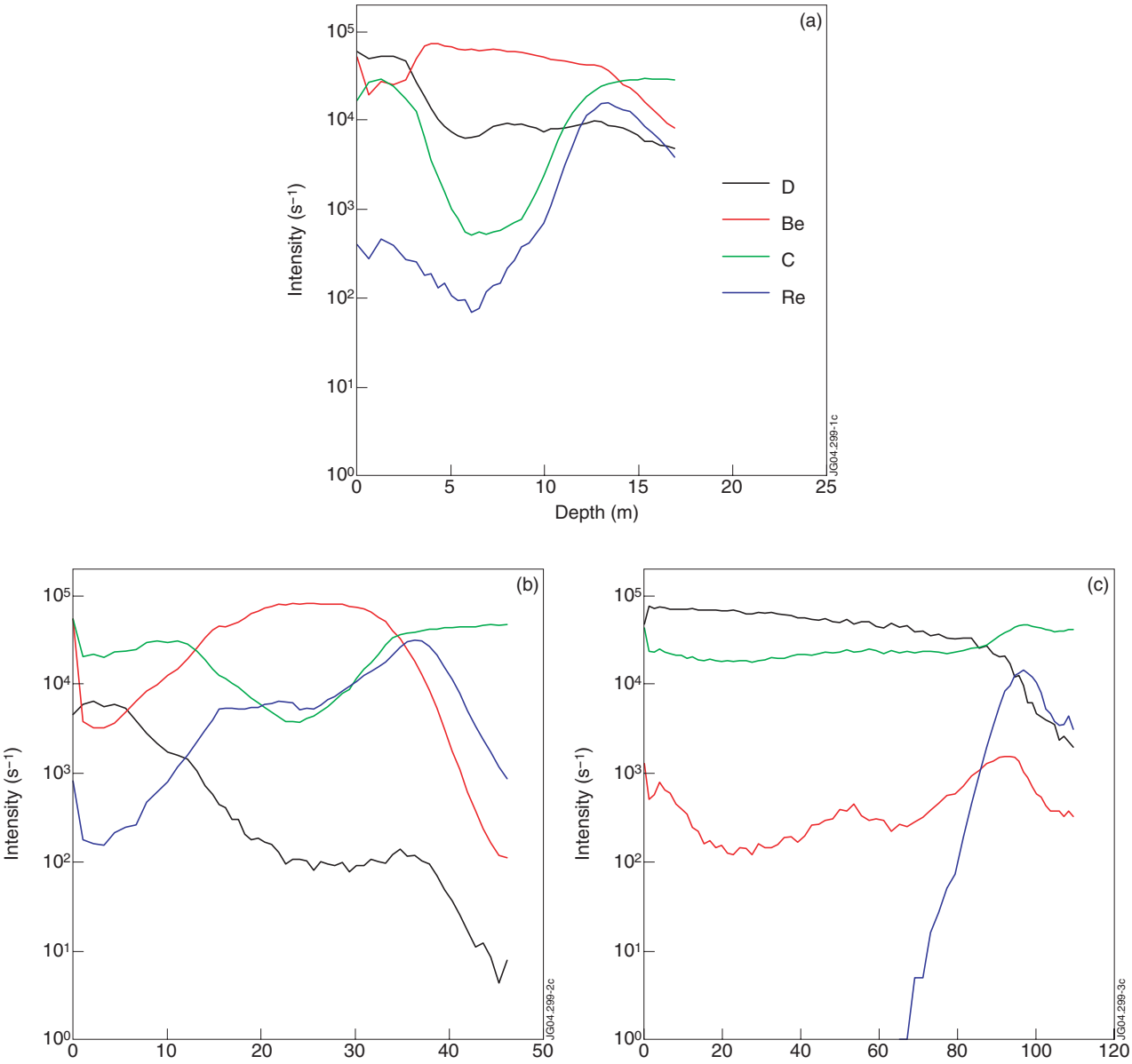

Figure 2. SIMS depth prfiles of D, Be, C and Re from (a) the bottom of tile 1 (sample 1/1), (b) the centre of tile 3 (sample 3/4) and (c) from the shadowed region on tile 4 (sample 4/10). Samples were exposed in 1999-2001.

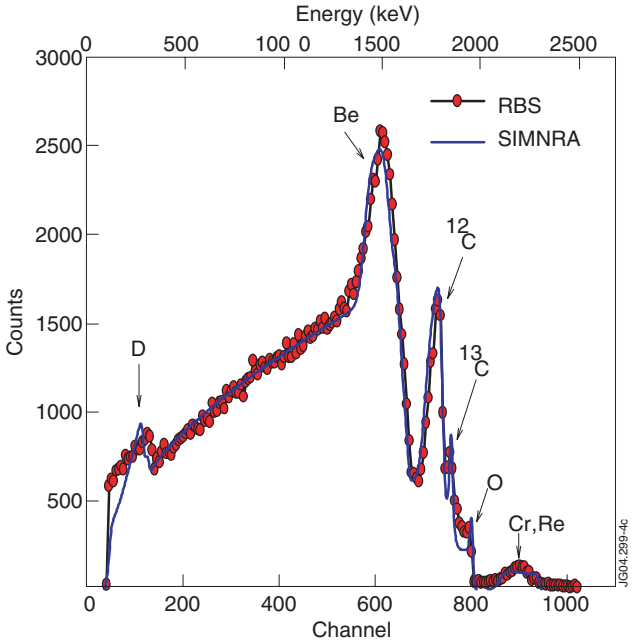

Figure 3. RBS spectrum and SIMNRA simulation from the bottom of tile 1 which was exposed in 1999-2001.

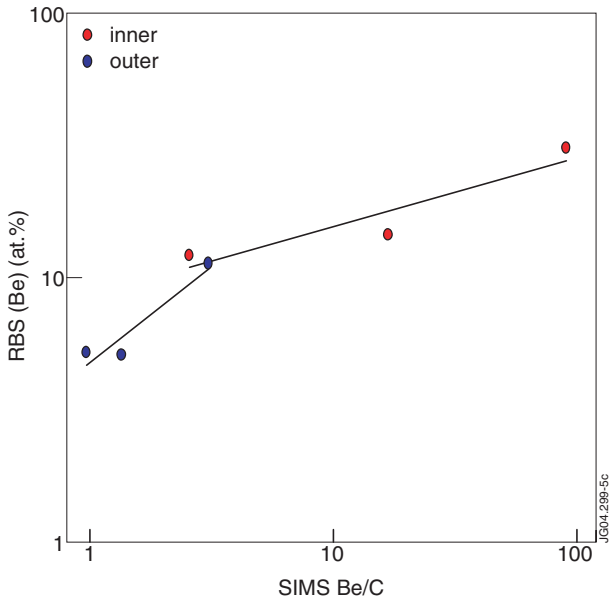

Figure 4. Calibrations curves for SIMS. Be amount measured with RBS is plotted as a function SIMS Be/C signal ratios for inner wall tile 1 exposed from 1999-2001. Be/C ratios calculated separately for outer carbon-rich and inner beryllium-rich layer.