



HOKKAIDO UNIVERSITY

Title	Helium retention and surface morphology of oxidized vanadium alloy
Author(s)	Yamauchi, Y.; Oku, D.; Hino, T. et al.
Citation	Journal of Nuclear Materials, 417(1-3), 327-329 https://doi.org/10.1016/j.jnucmat.2010.12.103
Issue Date	2011-10-01
Doc URL	https://hdl.handle.net/2115/47978
Type	journal article
File Information	JNM417-1-3_327-329.pdf



[ID code] 00048

[Paper Title] Helium Retention and Surface Morphology of Oxidized Vanadium Alloy

[Authors and Affiliations]

Y. Yamauchi^{1*}, D. Oku¹, T. Hino¹, T. Shibayama², Y. Nobuta¹, T. Nagasaka³ and T. Muroga³

¹ Laboratory of Plasma Physics and Engineering, Hokkaido University, Kita-13, Nishi-8, Kita-ku, Sapporo, Hokkaido, 060-8628 Japan, yamauchi@qe.eng.hokudai.ac.jp

² Center for Advanced Research of Energy Conversion Materials, Hokkaido University, Kita-13, Nishi-8, Kita-ku, Sapporo, Hokkaido, 060-8628 Japan

³ National Institute for Fusion Science, 322-6, Oroshi-cho, Toki, Gifu, 509-5292 Japan

[Corresponding Author]

Name : Yuji Yamauchi

Postal address : Laboratory of Plasma Physics and Engineering, Hokkaido University, Kita-13, Nishi-8, Kita-ku, Sapporo, Hokkaido, 060-8628 Japan

Telephone number : +81-11-706-7108

Fax number : ditto

E-mail address : yamauchi@qe.eng.hokudai.ac.jp

Helium Retention and Surface Morphology of Oxidized Vanadium Alloy

Y. Yamauchi¹, D. Oku¹, T. Hino¹, T. Shibayama², Y. Nobuta¹, T. Nagasaka³ and T. Muroga³

¹ Laboratory of Plasma Physics and Engineering, Hokkaido University, Kita-13, Nishi-8,

Kita-ku, Sapporo, Hokkaido, 060-8628 Japan, yamauchi@qe.eng.hokudai.ac.jp

² Center for Advanced Research of Energy Conversion Materials, Hokkaido University,

Kita-13, Nishi-8, Kita-ku, Sapporo, Hokkaido, 060-8628 Japan

³ National Institute for Fusion Science, 322-6, Oroshi-cho, Toki, Gifu, 509-5292 Japan

Abstract

The effects of the surface oxidation on the helium desorption and retention behaviors of vanadium alloy were investigated. V-4Cr-4Ti alloy, which was thermally oxidized at 873K for 15 minutes with 0.05 Pa, was irradiated by helium ions with energy of 5 keV at room temperature. Then, the helium desorption and retention behaviors were evaluated using a technique of thermal desorption spectroscopy. The changes in surface structures by the irradiation were also evaluated. Helium desorption behavior in low temperature region were significantly changed by the oxidation. Helium desorption around 700K became large for the oxidized sample. The amount of retained helium and the blister size in the oxidized sample were smaller than of those in the non-oxidized one. These results indicate that the surface oxidation significantly affects the helium retention and desorption behaviors of the vanadium alloy.

Keywords: Helium retention, oxidation, vanadium alloy, thermal desorption, blister formation

1. Introduction

A vanadium alloy, V-4Cr-4Ti, is one of candidate materials for blanket structure and first wall in fusion reactors because of its low induced radioactivity, and good thermal and mechanical properties at high temperature [1]. Various studies on the vanadium alloy such as the mechanical properties have been reported previously [2]. The first walls made of the vanadium alloy receive helium ions produced by fusion reactions and then retain the helium, if the vanadium alloy is used as the first wall material in the reactor. The re-emission of the retained helium from the wall due to the heat load and the particle bombardment leads to the fuel dilution, so that it is necessary to evaluate the helium retention and desorption behaviors for the material used as the first wall. In addition, it is known that some vanadium alloys are rather oxidized [3]. The surface oxidation affected the deuterium retention and desorption properties of alloys such as reduced activation ferritic steel [4]. The helium retention and desorption behaviors might depend on the oxidized state for the surface of the vanadium alloy. Also, the surface structure might be changed by the oxidation, which would result in the change in the helium retention properties. In the present study, the effects of the surface oxidation on the helium desorption and retention behaviors for the V-4Cr-4Ti alloy were investigated. The changes of the surface microstructures after the helium ion irradiation were evaluated for the non-oxidized and the oxidized samples, and then the effects of microstructure evolution on the helium desorption and retention properties were also discussed.

2. Experiment

The cold-rolled V-4Cr-4Ti alloy (NIFS-HEAT-2) [5] was used as a sample. The sample was mechanically and electrolytically polished and then degassed at 873K for 30 minutes in a vacuum. The thermal oxidation was conducted for the degassed sample with the oxidation temperature of 873K, the oxygen pressure 0.05 Pa and the oxidation duration 15 minutes. The helium ion irradiations were conducted for the oxidized and the non-oxidized samples in an ECR ion irradiation apparatus [6] at room temperature. The energy of the helium ion was 5 keV and the fluence was taken in the range from 5×10^{20} to 10^{22} He/m², corresponding to the surface damage of 4.5-90 dpa. After the irradiation, the helium desorption and retention behaviors were evaluated by using a technique of thermal desorption spectroscopy in the same apparatus. The irradiated sample was linearly heated up to 1673K with a heating rate of 1K/s. During the heating, the desorption rate of helium was quantitatively measured by a quadrupole mass

spectrometer. The depth profiles of the surface compositions were evaluated by using a technique of Auger electron spectroscopy with 3 keV Ar^+ etching. The surface morphologies after the irradiations were observed by an atomic force microscope, AFM. The microstructure in the oxidized sample after the helium ion irradiation was also observed by a transmission electron microscope, TEM (JEM-2010F, JEOL Ltd.), after the preparation of the cut sample with a focused ion beam apparatus. The bright field image was obtained under over-focus condition (bubble contrast) with an electron accelerating voltage of 200 kV.

3. Results and discussion

Depth profiles of atomic compositions for the non-oxidized and the oxidized samples are shown in figs.1 (a) and (b), respectively. The non-oxidized sample had an oxide layer at the surface with a thickness of 10 nm or less. For the oxidized sample, the oxygen concentration was approximately 55 at.% at the surface layer, which was almost constant down to the depth of approximately 30 nm. This result indicates a firm oxide layer with a thickness of 30 nm was formed in the surface by the oxidation. The decrease in the oxygen concentration with the increase of the depth under the layer was attributed to fast oxygen diffusion for the bulk region. It is noted that the thickness of the oxidized layer was thicker than the projected range of 5 keV He^+ ion, 27 nm.

Figures 2 (a) and (b) show thermal desorption spectra of helium for the non-oxidized and the oxidized samples, respectively. For the non-oxidized sample, the desorption peaks appeared around 500, 1100 and 1300K. Both for the non-oxidized and oxidized samples, the peak intensity around 1300K increased with the ion fluence and then the peaks in low temperature region gradually increased. While no obvious difference was observed between the spectra of the non-oxidized and the oxidized samples at the low fluence, the comparison of the spectra at the high fluence clearly shows the change in the helium desorption by the surface oxidation. Namely, large desorption around 700K and little desorption around 500 K for the oxidized sample was observed, compared with the case for the non-oxidized sample.

Figure 3 shows amounts of retained helium as a function of the helium ion fluence for the non-oxidized and the oxidized samples. While the fluence for the saturation of the amount of retained helium for the oxidized sample was similar to that for the non-oxidized sample, the saturated amount of retained helium in the oxidized sample was smaller than that of non-oxidized one. These results clearly indicate that the formation of trapping sites for the implanted helium during the helium ion irradiation was

changed by the prior oxidation. Fedorov et al have reported that the impurity such as oxygen acts the core for the helium trapping site resulting from the formation of helium-impurity-vacancy cluster for various vanadium alloys [7]. One may think that this fact leads to large helium retention for the case of the oxidized vanadium alloy, which is not consistent with the results obtained in the present study. The ion fluence in the present study was considerably high, compared with the case in ref. [7], so that the formation process of the blister might be changed with the oxide layer at the surface.

Figures 4 (a) and (b) show AFM images of the non-oxidized and the oxidized samples after the helium ion irradiation, respectively. The helium ion fluence was 5×10^{21} He/m². After the irradiation both for the non-oxidized and the oxidized samples, many blisters were formed at the surface. The average lateral diameter of the blisters for the oxidized sample was similar to that for the non-oxidized sample, ~200 nm. The average height of the blister for the oxidized sample (~10 nm), however, was approximately a half of that for the non-oxidized sample. The formations of small blisters might be associated with small helium retention for the oxidized sample.

Figure 5 shows TEM image of the cross section of the oxidized sample after the helium ion irradiation. The helium ion fluence was 5×10^{21} He/m². The formations of the helium bubble clusters (Typical contrasts of them were indicated by arrows in fig. 5) with various sizes were clearly observed at the sub-surface region (marked by white circle in fig.5). This suggested the blister-like protuberance, shown in fig. 4, might contain plenty of helium bubble clusters. The helium desorption might result from the fractures of these bubbles or the blisters. The recrystallization of V-4Cr-4Ti alloy started around 900K [8] and the fine precipitate at the surface disappeared by the annealing at 1373K for 1 hr [9]. So, the desorption at around 1300 K, which increased by the irradiation with the low fluence, might be attributed to the fractures of the helium bubbles accompanied with the dissolutions of the impurities from the helium-impurity-vacancy cluster or the recrystallization. In addition, it was reported that the increase of oxygen concentration in the vanadium alloy caused the decrease in its ductility [10]. This suggest that the blister at the surface of the oxidized sample easily fractured, which might lead to large desorption around 700K, compared with the case of the non-oxidized sample.

4. Conclusions

The effects of the surface oxidation on the helium desorption and retention properties of

V-4Cr-4Ti alloy were evaluated. The structural modifications at the surface and the sub-surface region after the helium ion irradiation were also observed to discuss the change in the desorption/retention properties. For the low fluence, helium desorption spectrum of the oxidized sample was similar to that of the non-oxidized sample, which had a peak around 1300K. For the high fluence, the desorptions in low temperature region rapidly increased both for the cases of the non-oxidized and the oxidized sample. The large desorption around 700K was observed for the oxidized sample. This might be attributed to the decrease in ductility of the surface region by the oxidation, which resulted in easy fractures of the blisters. The saturated amount of retained helium for the oxidized sample was smaller than that of the non-oxidized sample. The average height of the blisters in the oxidized sample was also smaller than of that of non-oxidized one, which might lead to small helium retention for the oxidized sample. These results indicate that the surface oxidation significantly affects the helium retention and desorption behaviors of the vanadium alloy.

References

- [1] For example, D.L. Smith, M.C. Billone, K. Natesan, International Journal of Refractory Metals and Hard Materials, Volume 18 (2000) 213-224.
- [2] For example, K. Fukumoto, M. Narui, H. Matsui, T. Nagasaka, T. Muroga, M. Li, D.T. Hoelzer, S.J. Zinkle, Journal of Nuclear Materials, 386-388 (2009) 575-578.
- [3] M. Fujiwara, K. Takanashi, M. Satou, A. Hasegawa, K. Abe, K. Kakiuchi, T. Furuya, Journal of Nuclear Materials, 329-333 (2004) 452-456.
- [4] Y. Yamauchi, Y. Hirohata, T. Hino, Journal of Nuclear Materials, 363-365 (2007) 984-988.
- [5] T. Muroga, T. Nagasaka, K. Abe, V.M. Chernov, H. Matsui, D.L. Smith, Z.-Y. Xu and S.J. Zinkle, Journal of Nuclear Materials, 307-311 (2002) 547-554.
- [6] T. Hino, Y. Yamauchi, Y. Hirohata, Journal of Nuclear Materials, 266-269 (1999) 538-541.
- [7] A.V. Fedorov, A. van Veen, A.I. Ryazanov, Journal of Nuclear Materials, 233-237 (1996) 385-389.
- [8] N.J. Heo, T. Nagasaka and T. Muroga, Journal of Nuclear Materials, 325 (2004) 53-60.
- [9] N. J. Heo, T. Nagasaka, T. Muroga and H. Matsui, Journal of Nuclear Materials, 307-311 (2002) 620-624.
- [10] K. Natesan, M. Uz, Fusion Engineering and Design, 51-52 (2000) 145-152.

Figure captions

- Figure 1 Depth profiles of atomic compositions for the V-4Cr-4Ti alloys before, (a), and after the surface oxidation, (b).
- Figure 2 Thermal desorption spectra of helium for the helium-irradiated V-4Cr-4Ti alloys before, (a), and after the surface oxidation, (b).
- Figure 3 Amounts of retained helium as a function of the helium ion fluence for the non-oxidized and the oxidized V-4Cr-4Ti alloy.
- Figure 4 AFM images of the surface morphologies after the helium ion irradiation for the non-oxidized V-4Cr-4Ti alloy, (a), and the oxidized V-4Cr-4Ti alloy, (b).
- Figure 5 TEM image of the cross section of the oxidized V-4Cr-4Ti alloy after the helium ion irradiation.

Fig. 1 (one column, Y. Yamauchi)

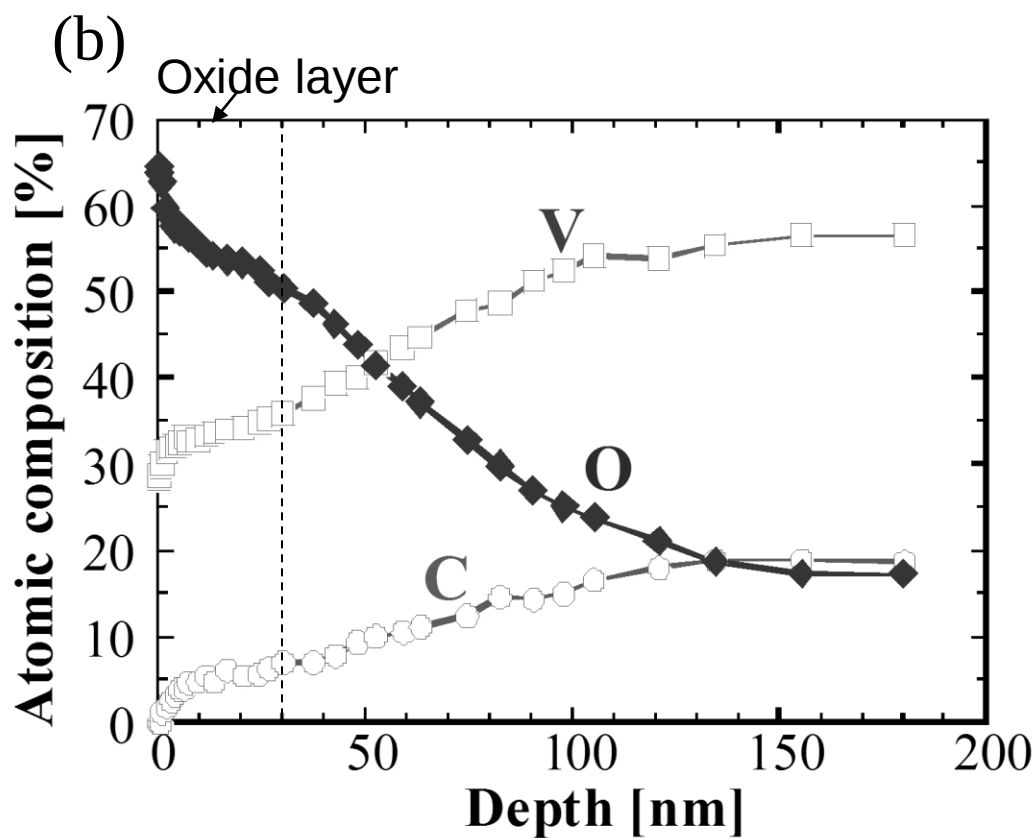
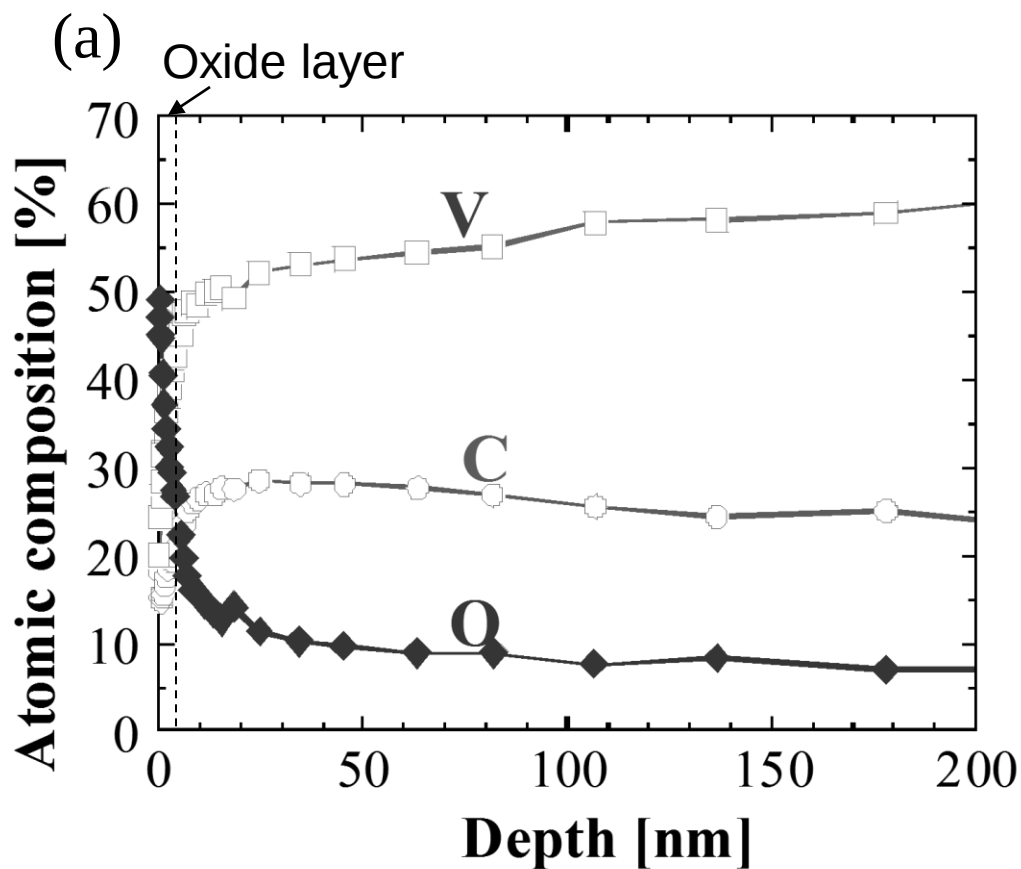


Fig. 2 (one column, Y. Yamauchi)

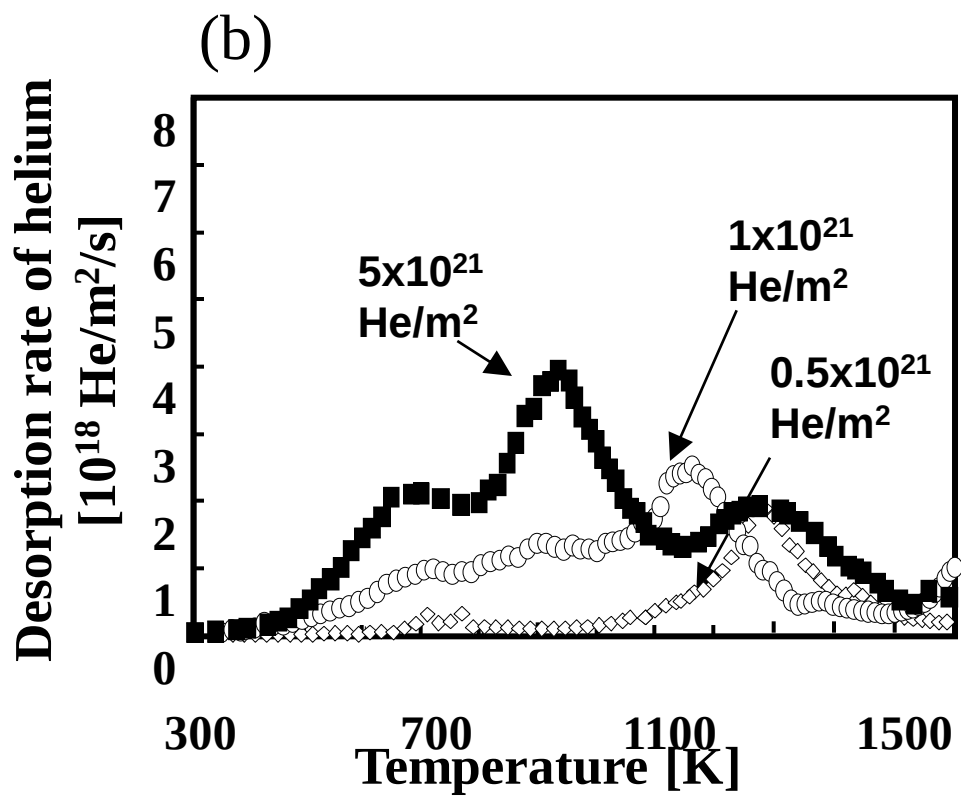
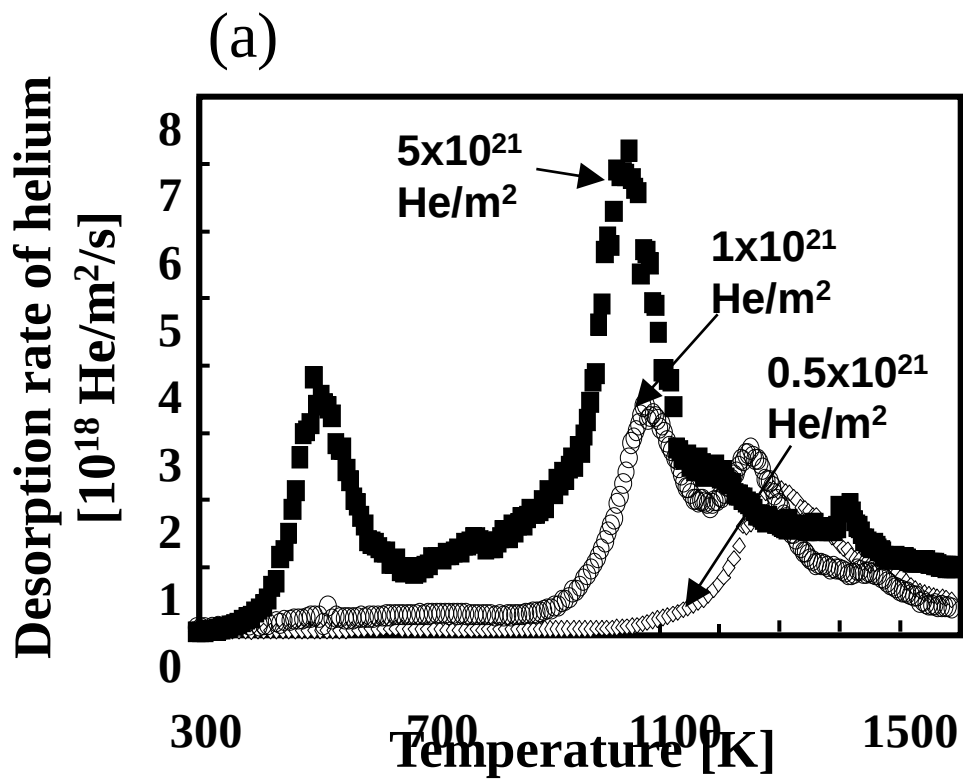


Fig. 3 (one column, Y. Yamauchi)

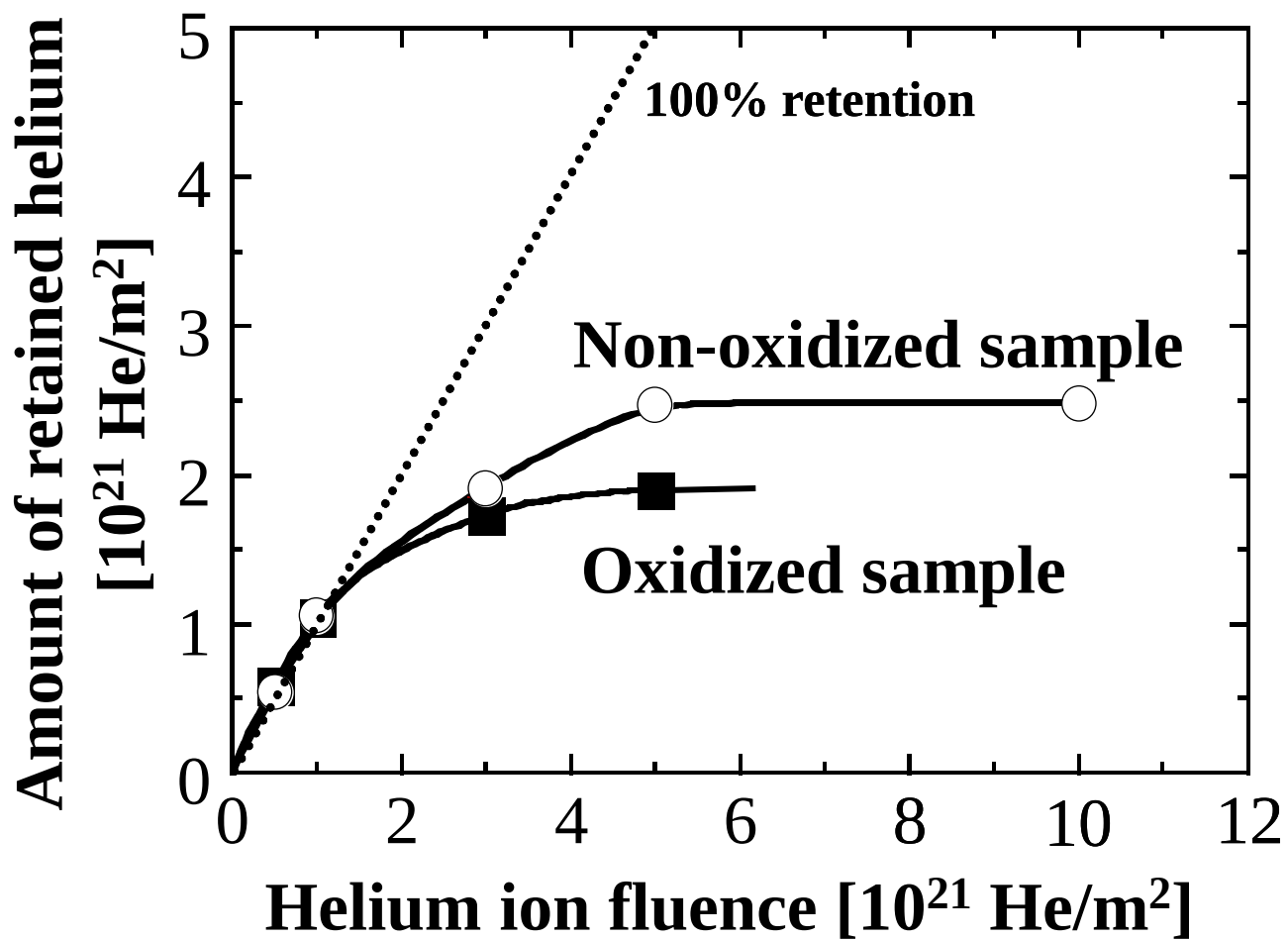


Fig. 4 (one column, Y. Yamauchi)

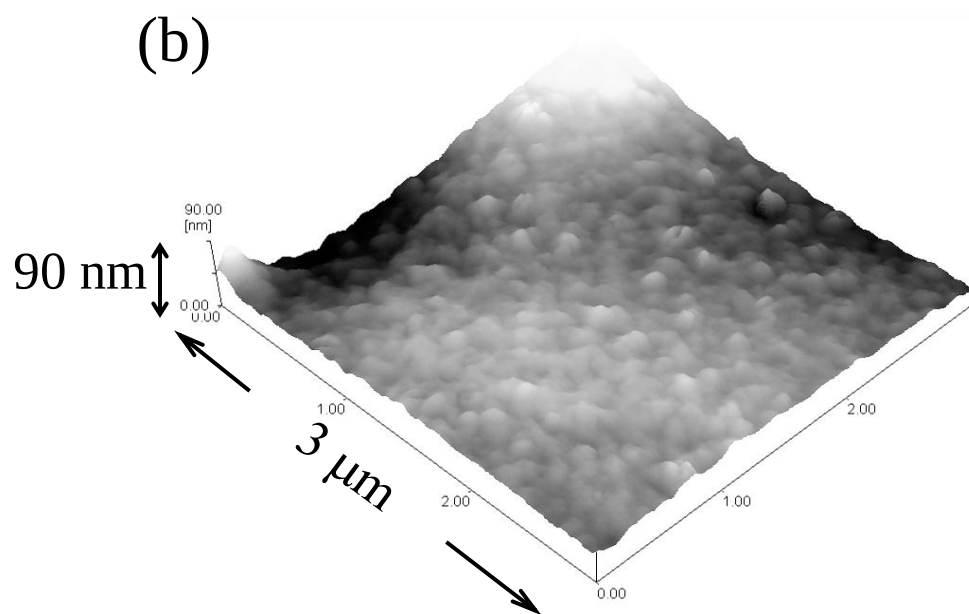
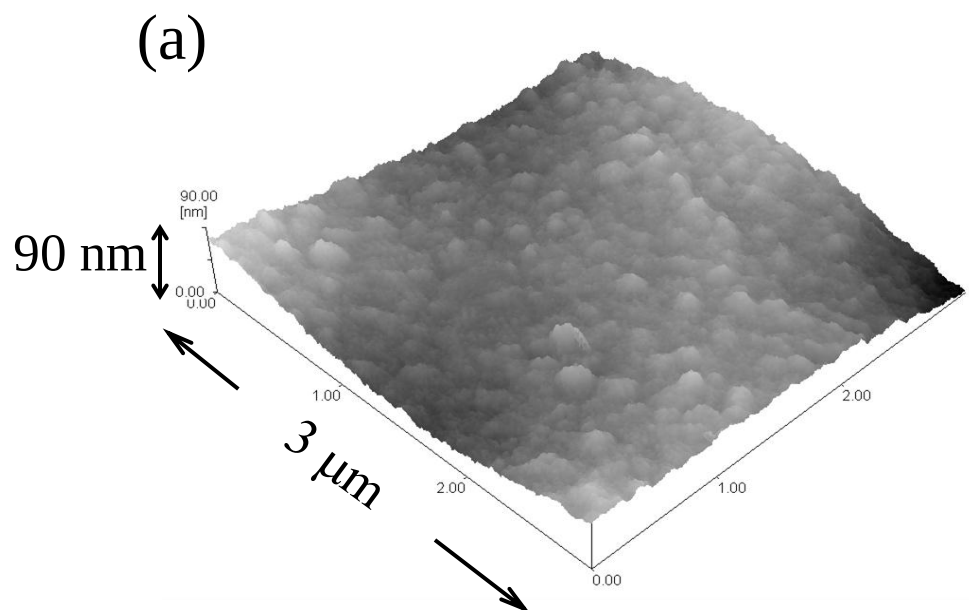


Fig. 5 (one column, Y. Yamauchi)

