Surface modification of magnetic recording media by filtered cathodic vacuum arc

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Abstract

Surface modification of a magnetic recording medium was accomplished by filtered cathodic vacuum arc (FCVA) treatment. The carbon overcoat of thin-film disks was removed by Ar^+ ion sputter etching in vacuum to prevent oxidation of the exposed magnetic medium, which was then modified by FCVA carbon plasma under conditions of 0 and –100 V pulsed substrate bias. Monte-Carlo simulations performed with the T-DYN code, x-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), and surface force microscopy (SFM) studies provided insight into carbon implantation profiles, surface chemical composition, roughness, and nanomechanical properties of the modified magnetic medium surface. The dependence of the surface modification on the FCVA treatment conditions are discussed in the context of T-DYN, XPS, AFM, and SFM results. The findings of this study illustrate the potential of the FCVA method to provide overcoat-free magnetic recording media exhibiting oxidation resistance and enhanced nanomechanical properties.

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I. INTRODUCTION

Magnetic recording relies on the storage of information in the form of bits in a magnetic layer (normally a Co-based alloy) of a thin-film (hard) disk[1–5] coated with a thin carbon film for protection against mechanical wear and corrosion. A lubricant monolayer adsorbed at the carbon film surface provides an additional corrosion barrier and lowers friction (adhesion) during intermittent asperity contact between the magnetic head and the hard disk. Because the data storage density increases exponentially with the decrease of the magnetic spacing, i.e., the distance between the magnetic write/read element at the trailing edge of the head and the magnetic layer of the hard disk,[2–8] reducing the thickness of the carbon overcoat while preserving the corrosion resistance and mechanical/tribological properties at the head/disk interface is of paramount importance. Despite the remarkable increase in the magnetic recording density to a few hundreds of Gbit/in^2 in current disk-drives,[3–6] even higher densities approaching the 10 Tbit/in^2 level are projected for the foreseeable future, implying a protective overcoat thickness of less than 1 nm. However, continuous carbon overcoats of such small thickness cannot be obtained with traditional coating techniques such as sputtering. Therefore, new surface treatments must be developed to achieve magnetic recording densities on the order of a few Tbit/in^2.

Filtered cathodic vacuum arc (FCVA) is a plasma-treatment method that enables surface modification by pure ion implantation under controlled ion energy conditions.[4,7,9–13] Because the film precursors produced by FCVA are ions (as opposed to atoms or clusters of atoms in other deposition techniques such as sputtering), the ion energy can be varied by applying a bias voltage to the substrate. Earlier studies have shown that the highest fraction of tetrahedral (sp^3) carbon hybridization and strength of carbon films deposited by the FCVA method were obtained for a pulsed substrate bias of –100 V.[12–15] However, zero-bias FCVA treatment produces shallow

implantation profiles that are of particular interest when only the outermost surface layer must be modified as in the case of the magnetic medium of hard disks.[3,16,17]

The objective of this study was to examine the modification of the near-surface region of the magnetic medium of hard disks by energetic C^+ ion bombardment under controlled FCVA conditions. Monte-Carlo simulations, x-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), and surface force microscopy (SFM) studies were performed to determine the dependence of the surface topography, structure, and nanomechanical properties of the modified magnetic medium on FCVA carbon plasma conditions of 0 and –100 V pulsed substrate bias. Representative results from these studies demonstrate the efficacy of the FCVA method to produce overcoat-free magnetic media with increased mechanical strength and good oxidation resistance.

II. EXPERIMENTAL PROCEDURES

A. Sample preparation

Unlubricated hard disks of diameter equal to 3.5 in. were cut into 10 × 10 mm^2 pieces. To prevent oxidation of the magnetic medium, the hard-disk pieces were loaded onto the sample stage of the FCVA system, described in detail elsewhere,[10] and the ~4-nm-thick carbon overcoat was removed by Ar^+ ion sputter etching under a working pressure of ~2.4×10^{-4} Torr. The Ar^+ ion etching time to completely remove the carbon overcoat was determined from simulation results of the carbon sputter rate and profilometry measurements of the etch thickness. A 64-mm Kaufman ion source (Commonwealth Scientific) that produces a 500-eV Ar^+ ion beam of constant ion flux was used for *in situ* sputter etching. During sputter etching and FCVA treatment, the sample holder was rotated at 60 rpm for uniform surface modification.

B. Surface modification by FCVA treatment

Samples with the exposed magnetic medium were subjected to FCVA treatment with carbon plasma in vacuum. Direct-current carbon plasma was generated by striking the high-purity (99.999%) graphite cathode with a mechanical trigger. The plasma was stabilized by a cusp configuration of the magnetic field around the anode,[10] and guided from the cathode to the substrate by a three-dimensional magnetic filter with an out-of-plane *S*-configuration to prevent macroparticle deposition. All the FCVA treatments were performed under a base pressure of $<3 \times 10^{-7}$ Torr, arc-discharge current of 70 A, and carbon ion flux perpendicular to the sample surface of $\sim 1.5 \times 10^{15}$ ions/cm^2·s. These conditions yielded a C^+ ion energy in the bulk of the plasma equal to ~20 eV.[13,14,18] Both the substrate holder and the cathode were continuously cooled by circulating water. FCVA treatments were performed under 0 and –100 V pulsed substrate bias of 25 kHz pulse frequency.

C. Monte-Carlo simulations

Monte-Carlo simulations were performed with the T-DYN code (version 4.0) to study the removal of the preexisting carbon film by Ar^+ ion sputtering and the surface modification of the exposed magnetic medium which was assumed to consist of cobalt. The T-DYN dynamic code is based on the trajectory of ions in matters (TRIM) code, and can be used to simulate energetic atom displacement in solids.[19–22] All the T-DYN simulations were performed for ion impingement perpendicular to the medium surface, constant ion energy, and no Ar^+ ion implantation. Details of the computational algorithms of the T-DYN code can be found elsewhere.[11,22] The surface binding energies of carbon and cobalt were set equal to 7.41 and 4.43 eV and their binding energies equal to 2.27 and 8.80 eV, respectively. These values are typical of solid-state graphite and cobalt.

D. Surface characterization

Surface chemical analysis was performed with an XPS (PHI 5400, Physical Electronics) system equipped with a monochromatic x-ray source of Al–Kα (1486.6 eV). Survey scans were acquired in 1-eV energy steps and pass energy of 178.95 eV. Each XPS survey spectrum was obtained as an average of four survey scans. For high-resolution window scanning, the energy step was set at 0.05 eV and the pass energy at 35.75 eV. Each high-resolution XPS window spectrum was obtained as an average of 20 scans.

A mechanical stylus profilometer (3030 Surface Profiler, Dektak) with a height resolution of 0.1 nm was used to measure the height difference between treated and untreated (covered during treatment) surface regions of each sample. The root-mean-square (rms) roughness of each sample was determined from 1×1 μm^2 surface images obtained with an AFM (Dimension 3100, Veeco Digital Instruments) operated in the tapping mode at a drive frequency of 259.332 kHz and a scan rate of 2 Hz.

E. Nanomechanical testing

The nanomechanical properties of the FCVA-modified magnetic medium were examined with an SFM consisting of an AFM (Nanoscope II, Digital Instruments) retrofitted with a capacitive force transducer (Triboscope, Hysitron). All the SFM experiments were performed with a pyramidal diamond tip of nominal radius equal to ~75 nm.[23] A normal force of ~3 μN was used to engage the tip with the sample surface. A triangular loading function with loading and unloading times both equal to 2 s was used in all the nanoindentation experiments. The shape function of the diamond tip (a polynomial function of indentation depth) was determined by indenting an ultrasmooth fused quartz sample with an in-plane elastic modulus $E/(1-\nu^2) = 69.6$ GPa, where E is the elastic modulus and ν is the Poisson's ratio. The maximum contact pressure was obtained as the ratio of the applied maximum load to the projected contact area, determined

from the polynomial tip-shape function at the corresponding indentation depth. The in-plane elastic modulus (referred to as the reduced modulus) was calculated from the stiffness estimated at the maximum tip displacement point of the unloading curve.[23–25]

III. RESULTS AND DISCUSSION

A. Sputter-etching of preexisting carbon overcoat

Figure 1 shows T-DYN simulation results of the etch thickness of carbon as a function of incidence angle for an Ar^+ ion dose of 1×10^{16} ions/cm^2. The data show that the maximum etch thickness corresponds to an incidence angle of ~70^o. Therefore, to minimize the time to remove the preexisting carbon overcoat from the hard-disk samples, the incidence angle of the Ar^+ ion beam was set at 60^o. Surface profilometry and XPS measurements confirmed the removal of the ~4-nm-thick carbon overcoat. For 4, 6, and 8 min of Ar^+ ion sputtering, the measured etch thickness was equal to 3.3, 4.5, and 7.3 nm, respectively. In view of the lower binding energy of cobalt than that of carbon, a higher etch rate was observed after the removal of the carbon overcoat. XPS results provided additional evidence of the complete removal of the carbon overcoat by sputter etching. Figures 2(a) and 2(b) show XPS survey spectra obtained before and after Ar^+ ion sputter etching for 8 min, respectively. The O1s peak is attributed to the adsorption of oxygen upon the sample exposure to the ambient. The Co2p, Cr2p, Pt4d, and Pt4f peaks and the significant decrease of the C1s peak intensity in the XPS spectrum shown in Fig. 2(b) indicate the exposure of the magnetic medium due to the removal of the carbon overcoat. The low-intensity C1s peak in Fig. 2(b) is also due to carbon adsorbents from the ambient.

B. FCVA treatment of magnetic medium

Figure 3 shows carbon implantation profiles in cobalt medium obtained from a T-DYN analysis for 0 and –100 V substrate bias and C^+ ion dose in the range of (0.9–13.5) × 10^{15} ions/cm^2 (i.e., treatment time in the range of 6–90 s). The results presented in Fig. 3, as well as in following figures, are for a C^+ ion flux perpendicular to the medium surface of ~1.5 × 10^{15} ions/cm^2·s. The impinging ion energy was set equal to the sum of the initial ion energy (~20 eV for 0 V substrate bias) and the energy due to the substrate bias voltage. The trend is for substrate biasing to decrease the carbon fraction at the surface and increase the thickness of the implantation profile.

Figure 4 shows the surface elevation as a function of treatment time with carbon plasma for 0 and –100 V substrate bias. An etch thickness of 7.3 nm was subtracted from all the measurements shown in Fig. 4. The very small or slightly negative values obtained for short treatment time (i.e., <20 s) are due to the resputtering effect of energetic carbon ions, especially in the case of the –100 V substrate bias. The surface elevation values for treatment times longer than 20 s are in fair agreement with the thickness of the carbon implantation profiles obtained from the T-DYN simulations (Fig. 3).

C. Surface chemical analysis

The oxidation resistance and structure of the FCVA-treated magnetic medium can be interpreted in the context of the XPS results shown in Figs. 5–8. XPS window spectra of the Co2p core-level peak obtained before and after the treatment of the magnetic medium are shown in Fig. 5. The broad $Co2p_{3/2}$ peak in the spectrum of the untreated medium [Fig. 5(a)] reveals the oxidation of cobalt.[2,26] The much narrower $Co2p_{3/2}$ peak in the spectrum of the treated medium [Fig. 5(b)] suggests that 6-s FCVA treatment of C^+ ion dose equal to ~0.9 × 10^{16} ions/cm^2 enhanced the oxidation resistance of the magnetic medium.

Figure 6 shows the XPS window spectrum of the C1s peak of the FCVA-treated magnetic medium. Although this spectrum is for treatment time of 12 s and 0 V substrate bias, it is representative of the C1s XPS spectra obtained for all the FCVA conditions of this study. After inelastic background subtraction,[27] the C1s spectrum was fitted with six Gaussian distributions at characteristic binding energies, as shown in fig. 6. The details of the deconvolution method can be found elsewhere.[28–30] Distributions referred to as C1s-1, C1s-2, and C1s-3 correspond to sp^1, sp^2, and sp^3 carbon hybridizations, respectively. The fraction of each type of atomic carbon bonding was estimated from the deconvolution of the C1s XPS spectrum, as suggested in previous studies.[31,32] Distributions denoted by C1s-4, C1s-5, and C1s-6 are assigned to atomic carbon bonding with surface adsorbents from the ambient,[11,28–30] hereafter referred to as satellite peaks. Thus, the sum of satellite peak areas indicates the fraction of carbon bonding with surface adsorbents.

Figures 7 and 8 respectively show the binding energies and fractions of sp^1, sp^2, and sp^3 carbon hybridization as functions of treatment time. It has been reported that the binding energies of carbon species depend on the stress state.[11,29,33] The relatively constant sp^1, sp^2, and sp^3 binding energies seen in Fig. 7 indicate a constant stress in the carbon species for treatment time in the range of 6–90 s. Therefore, the nanomechanical properties of the FCVA-treated magnetic medium presented later were not affected by variations in internal stress. The lower binding energies for –100 V [Fig. 7(b)] than 0 V [Fig. 7(a)] substrate bias may be attributed to a higher compressive stress due to the increased energy of the impinging C^+ ions that resulted from substrate biasing.[29] Figure 8 shows the variations of sp^1, sp^2, sp^3, and satellite fractions with the treatment time. For both 0 and –100 V substrate bias, the curves of the sp^2 and sp^3 fractions intersect at a point corresponding to a treatment time of ~24 s. The relatively low sp^3 fraction for treatment time less

than 20 s is in qualitative agreement with sp^3 fraction data for shallow implantation thickness.[8,11] According to the subplantation model, [16,17,19] energetic ions penetrate the substrate to a certain depth, resulting in material densification that is conducive to sp^3 hybridization.[17,34] This implies that sp^3 hybridization was affected by the formation of a carbon-rich surface layer. However, carbon hybridization under FCVA conditions differs from that resulting from conventional implantation because the low ion dose in FCVA affects significantly the carbon concentration at the surface. Although zero substrate bias yielded a higher sp^3 fraction for short treatment time (e.g., 6 s), an opposite trend was observed for treatments longer than ~24 s. This may be related to the combined effects of recoil implantation and deeper ion implantation under substrate biasing conditions. The higher satellite fractions for shorter treatment times are attributed to the availability of more surface sites for ambient adsorbents as a result of the lower implantation dose.

D. Surface roughness

Figure 9 shows the roughness of the treated magnetic medium as a function of treatment time. The rms roughness of the carbon-coated hard disk was equal to ~0.19 nm. The roughness for zero treatment time corresponds to the magnetic medium surface exposed after sputter etching for 8 min to ensure the complete removal of the preexisting carbon overcoat. Although Ar^+ ion etching induced significant surface roughening (rms ≈ 0.72 nm), FCVA treatment with carbon plasma for 12 s restored the original surface smoothness (rms ≈ 0.2 nm). It has been reported that the exposure of smooth Si wafers to FCVA carbon plasma resulted in the deposition of ultrathin carbon films of rms <0.1 nm.[11] Therefore, the ~0.2 nm roughness values in Fig. 9 are attributed to the rougher morphology of the magnetic medium produced from Ar^+ ion sputtering. Figure 9 also shows that a substrate pulsed bias of –100 V resulted in smoother topographies, presumably due to the enhancement of resputtering that promoted surface smoothening. Carbonaceous adsorbents

from the ambient might have also contributed to surface smoothening, particularly for short treatment times, resulting in higher fractions of carbon bonding attributed to ambient adsorbents (satellites), as shown in Fig. 8. Thus, the lowest surface roughness for a treatment time of 12 s may be related to the smoothening due to resputtering (primary effect) and adsorption of carbonaceous substances (secondary effect). The effect of both mechanisms decreased with the increase of the treatment time due to the formation of a more etch-resistant carbon-rich surface layer possessing a slightly rougher topography of rms ≈ 0.2 nm (Fig. 9).

E. Nanomechanical properties

Figure 10 shows results illustrative of the nanomechanical behavior of the FCVA-modified magnetic medium for 48 s treatment time and 0 V substrate bias. Nanoindentation load-displacement responses, such as that shown in Fig. 10(a), were used to determine the maximum contact pressure and reduced modulus as functions of the maximum tip displacement shown in Fig. 10(b). The initial increase in the maximum contact pressure with the maximum displacement is attributed to the gradual evolution of plasticity under the indenting diamond tip.[10,11,24] After reaching a peak value, the maximum contact pressure decreased with the increase of the maximum displacement due to the much softer and compliant magnetic medium and other sublayers comprising the hard disk. By definition, the peak value of the maximum contact pressure represents the "effective" hardness, which indicates the material resistance to plastic deformation due to indentation.[10,11,25,35] The effective depth is defined as the maximum displacement corresponding to the effective hardness. As shown in Fig. 10(b), the reduced modulus exhibited a slight decrease with the increase of the maximum displacement above the effective depth. This is attributed to the greater contribution of the more compliant magnetic medium and other sublayers to the elastic contact deformation of the hard disk.

Figure 11 shows representative results of the nanomechanical properties of the FCVA-treated magnetic media for 0 and –100 V substrate bias. The nanomechanical properties for short treatment time (i.e., low ion dose) are close to those of the unmodified (original) magnetic medium. The increase in the effective hardness with treatment time [Fig. 11(a)] may be related to the greater amount of implanted carbon that enhanced the surface deformation resistance of the magnetic medium. The higher hardness for –100 V pulsed substrate bias is attributed to the higher sp^3 content [Fig. 8(b)] due to the stronger effect of energetic C^+ ion bombardment and, presumably, the increase in surface densification that enhanced the penetration resistance of the magnetic medium, as evidenced by the lower effective depth in the case of substrate biasing [Fig. 11(c)]. In contrast to the effective hardness, the reduced modulus did not show a clear dependence on the substrate bias and treatment time [Fig. 11(b)]. Thus, FCVA treatment resulted in surface hardening of the magnetic medium without altering significantly the elastic behavior.

IV. CONCLUSIONS

Surface modification of the magnetic medium of hard disks by FCVA treatment was examined in the context of analytical and experimental results. The magnetic medium of hard disks, exposed in high vacuum by sputter etching the preexisting carbon overcoat to prevent oxidation, was subjected to energetic C^+ ion bombardment under 0 and –100 V pulsed (25 kHz) substrate bias conditions. T-DYN, XPS, AFM, and SFM results provided insight into the effect of FCVA treatment conditions on carbon implantation profiles, carbon atom hybridization, surface roughness, and nanomechanical properties of the surface-modified magnetic medium. An enhancement of the oxidation resistance was observed for a C^+ ion dose of ~0.9×10^{16} ions/cm^2 and a treatment time of 6 s. Higher sp^3 fractions and shallower modification of the magnetic medium were found for relatively short treatment time (<24 s) and 0 V substrate bias. However,

substrate biasing with –100 V pulsed voltage resulted in surface smoothening of the magnetic medium. FCVA treatment increased the surface hardness of the magnetic medium.

The presented results illustrate the effectiveness of the FCVA method to modify the surface structure, texture, and nanomechanical properties of magnetic recording media. These findings provide impetus for further investigations of FCVA plasma treatment aimed to produce overcoat-free magnetic media with suitably modified surface properties for ultrahigh-density magnetic recording applications.

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FIG. 9 Surface roughness (rms) vs treatment time for 0 and –100 V (25 kHz pulse frequency) substrate bias and ~1.5×10^{15} ions/cm^2·s ion flux.

FIG. 10 (a) Nanoindentation load vs displacement response and (b) maximum contact pressure and reduced modulus vs maximum displacement of FCVA-treated magnetic medium for 0 V substrate bias, ~1.5×10^{15} ions/cm^2·s ion flux, and 48 s treatment time.

FIG. 11 (a) Effective hardness, (b) reduced modulus, and (c) effective depth vs treatment time of FCVA-treated magnetic medium for 0 and –100 V (25 kHz pulse frequency) substrate bias and ~1.5×10^{15} ions/cm^2·s ion flux.

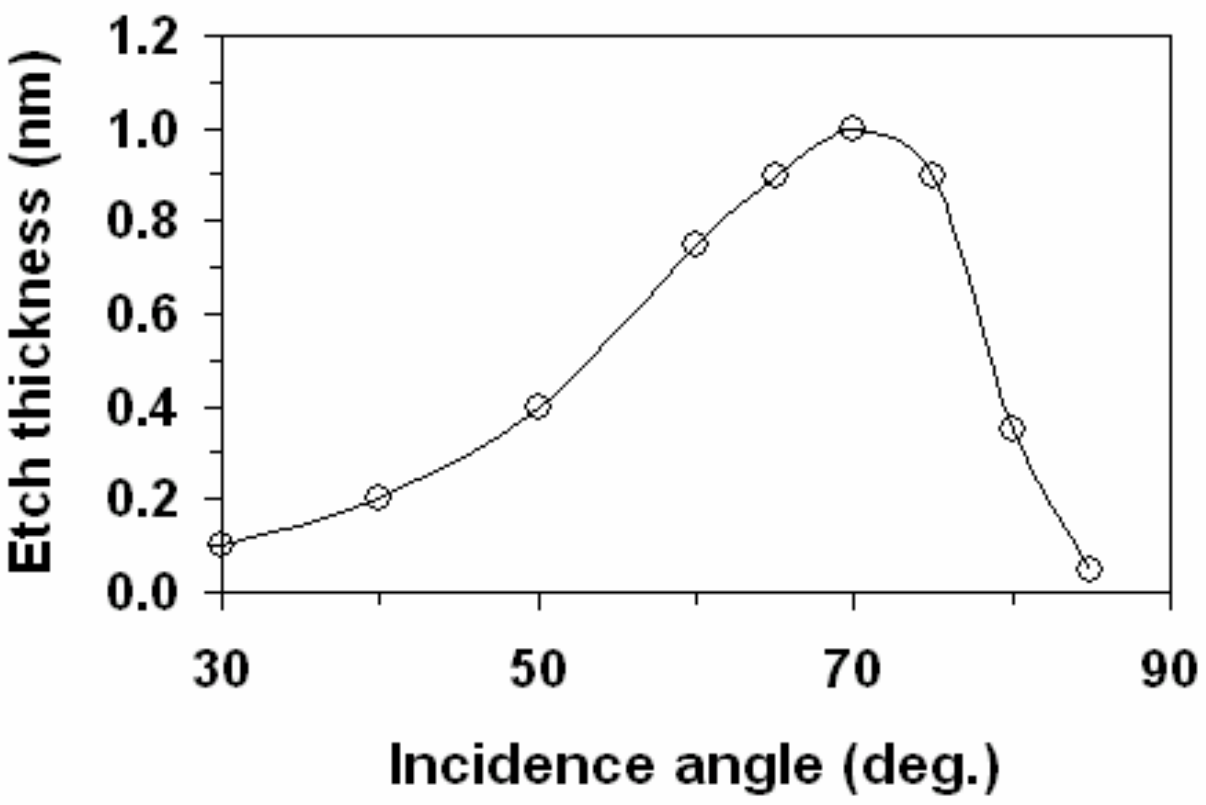
Etch thickness (nm)
1.2
1.0
0.8
0.6
0.4
0.2
0.0
30
50
70
90
Incidence angle (deg.)

Figure 1

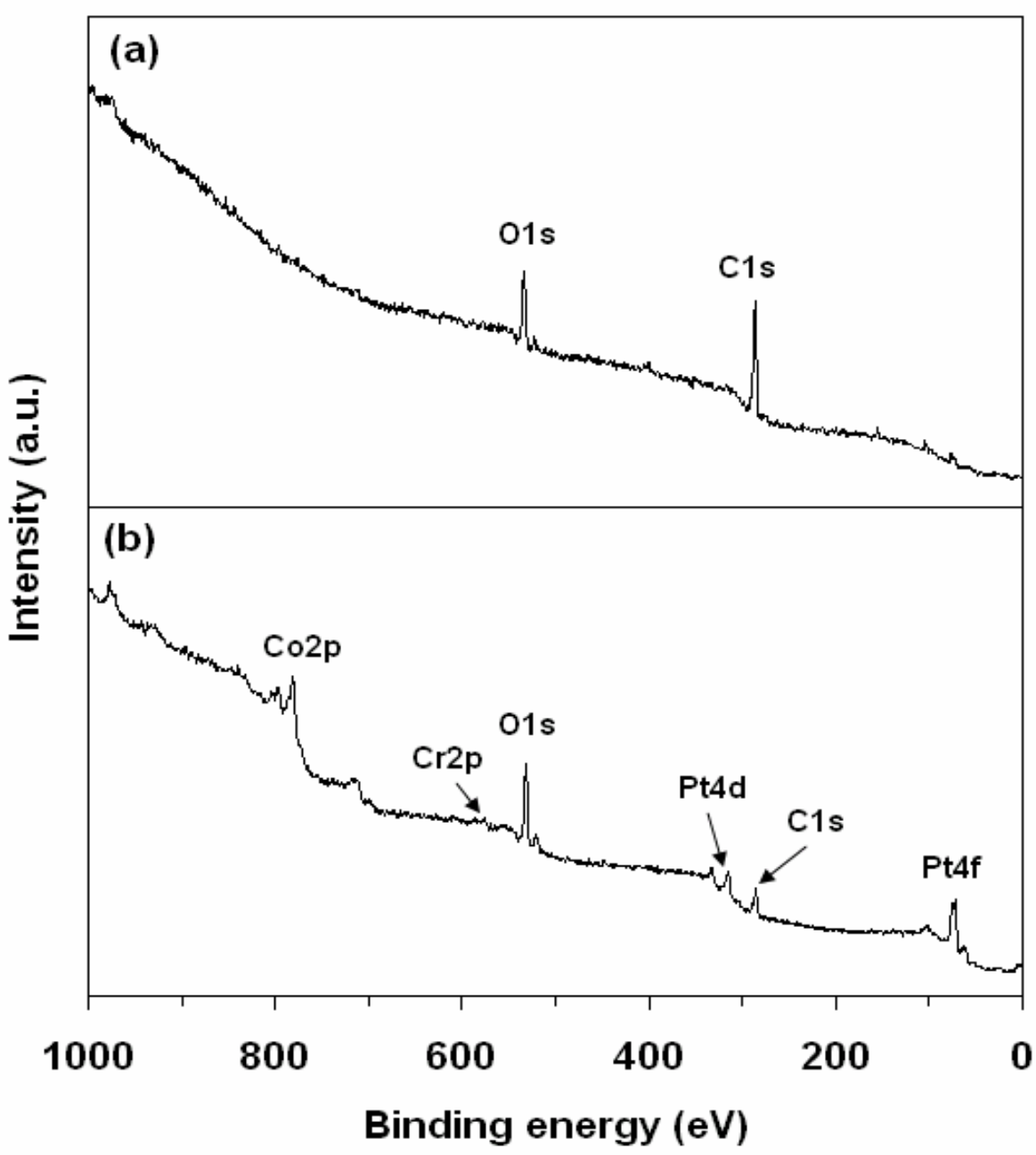
(a)
O1s
C1s
(b)
Co2p
Cr2p
O1s
Pt4d
C1s
Pt4f
Intensity (a.u.)
1000
800
600
400
200
0
Binding energy (eV)

Figure 2

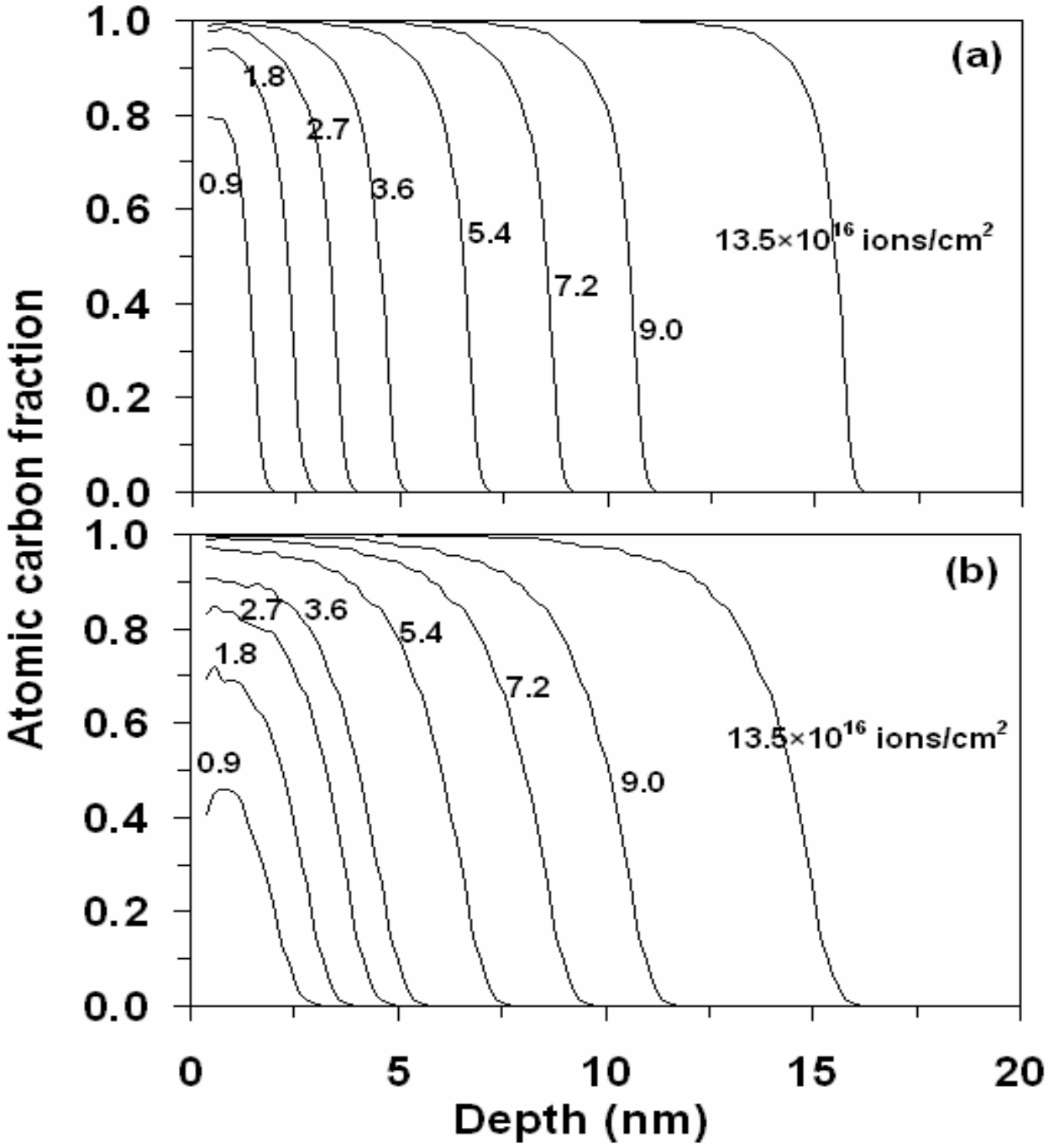
(a)
(b)
0.9
1.8
2.7
3.6
5.4
7.2
9.0
13.5×10^16 ions/cm^2
Atomic carbon fraction
Depth (nm)
0.0
0.2
0.4
0.6
0.8
1.0
0
5
10
15
20

Figure 3

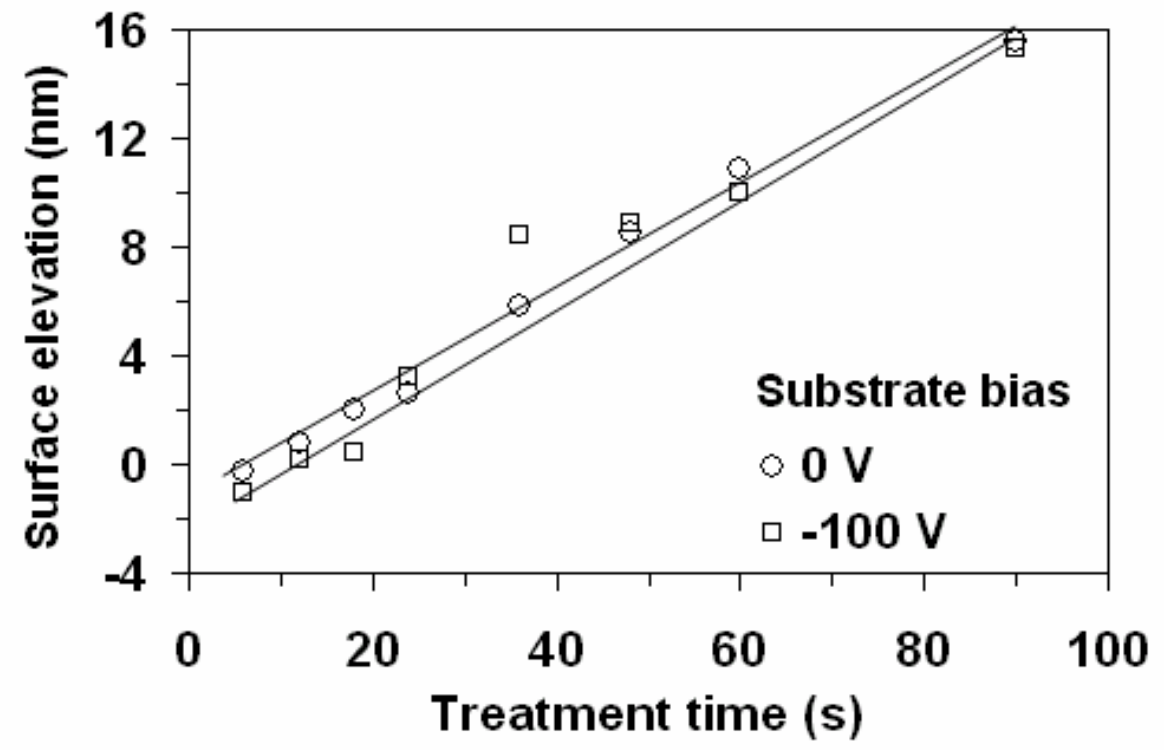
Surface elevation (nm)
16
12
8
4
0
-4
0
20
40
60
80
100
Treatment time (s)
Substrate bias
0 V
-100 V

Figure 4

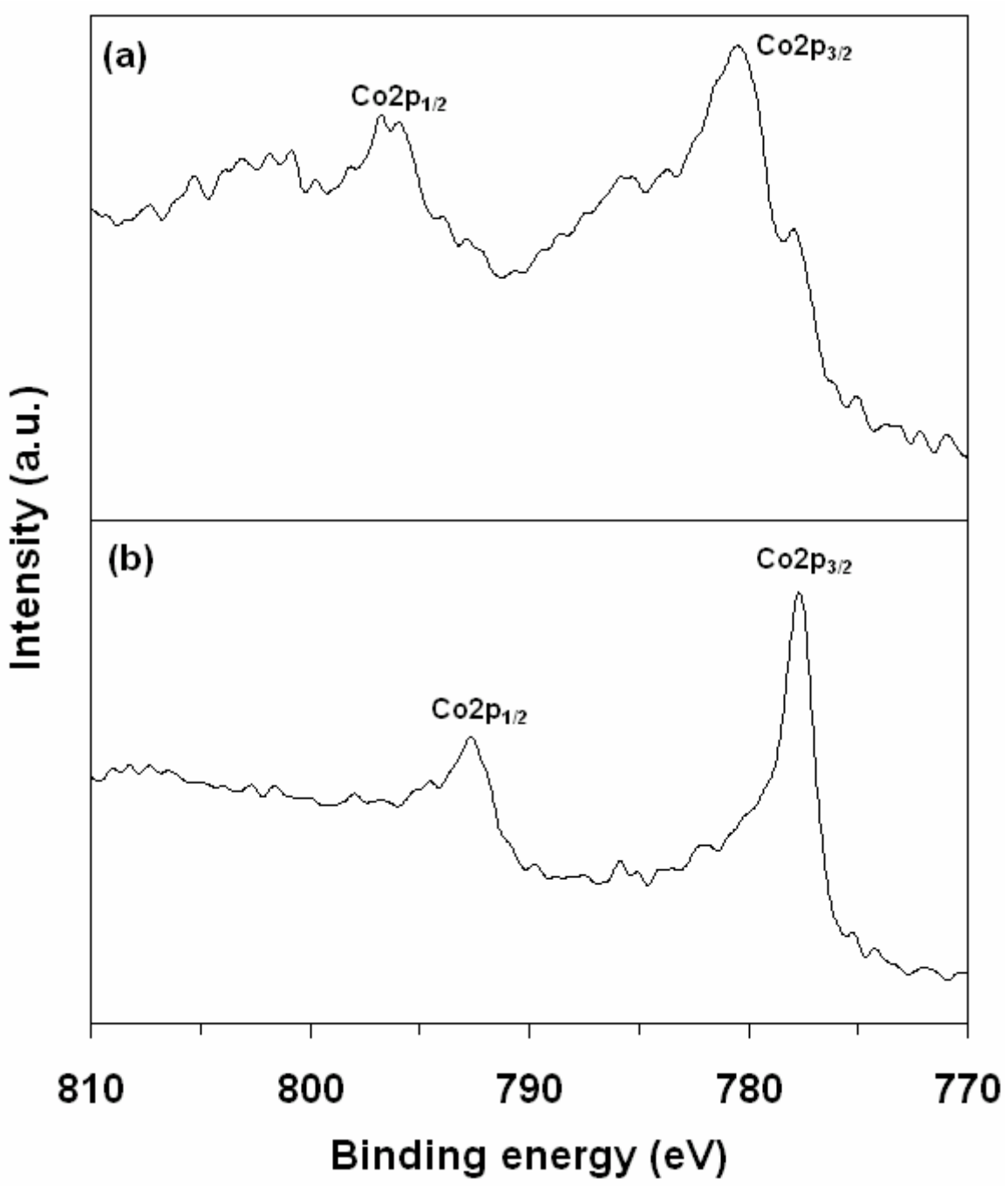
(a)
Co2p1/2
Co2p3/2
(b)
Co2p3/2
Co2p1/2
Intensity (a.u.)
810
800
790
780
770
Binding energy (eV)

Figure 5

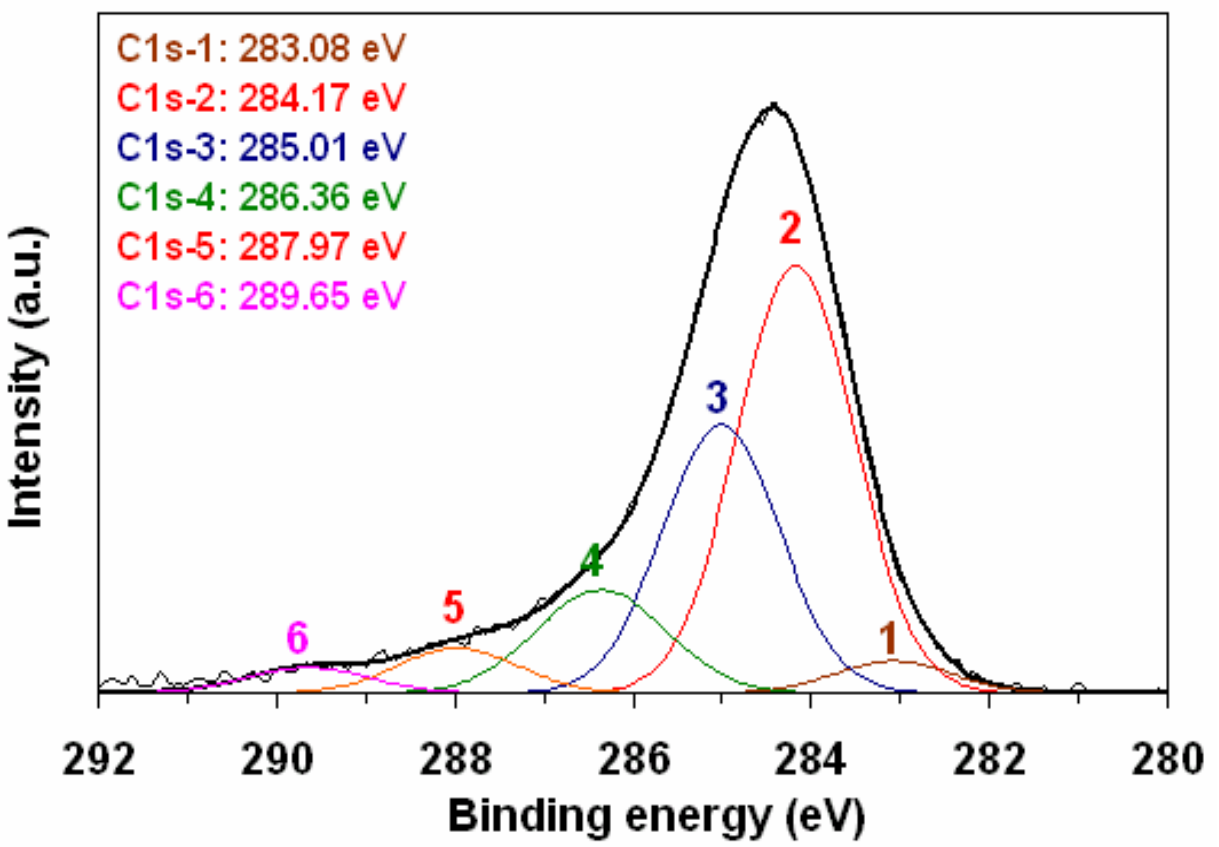
C1s-1: 283.08 eV
C1s-2: 284.17 eV
C1s-3: 285.01 eV
C1s-4: 286.36 eV
C1s-5: 287.97 eV
C1s-6: 289.65 eV
Intensity (a.u.)
1
2
3
4
5
6
292
290
288
286
284
282
280
Binding energy (eV)

Figure 6

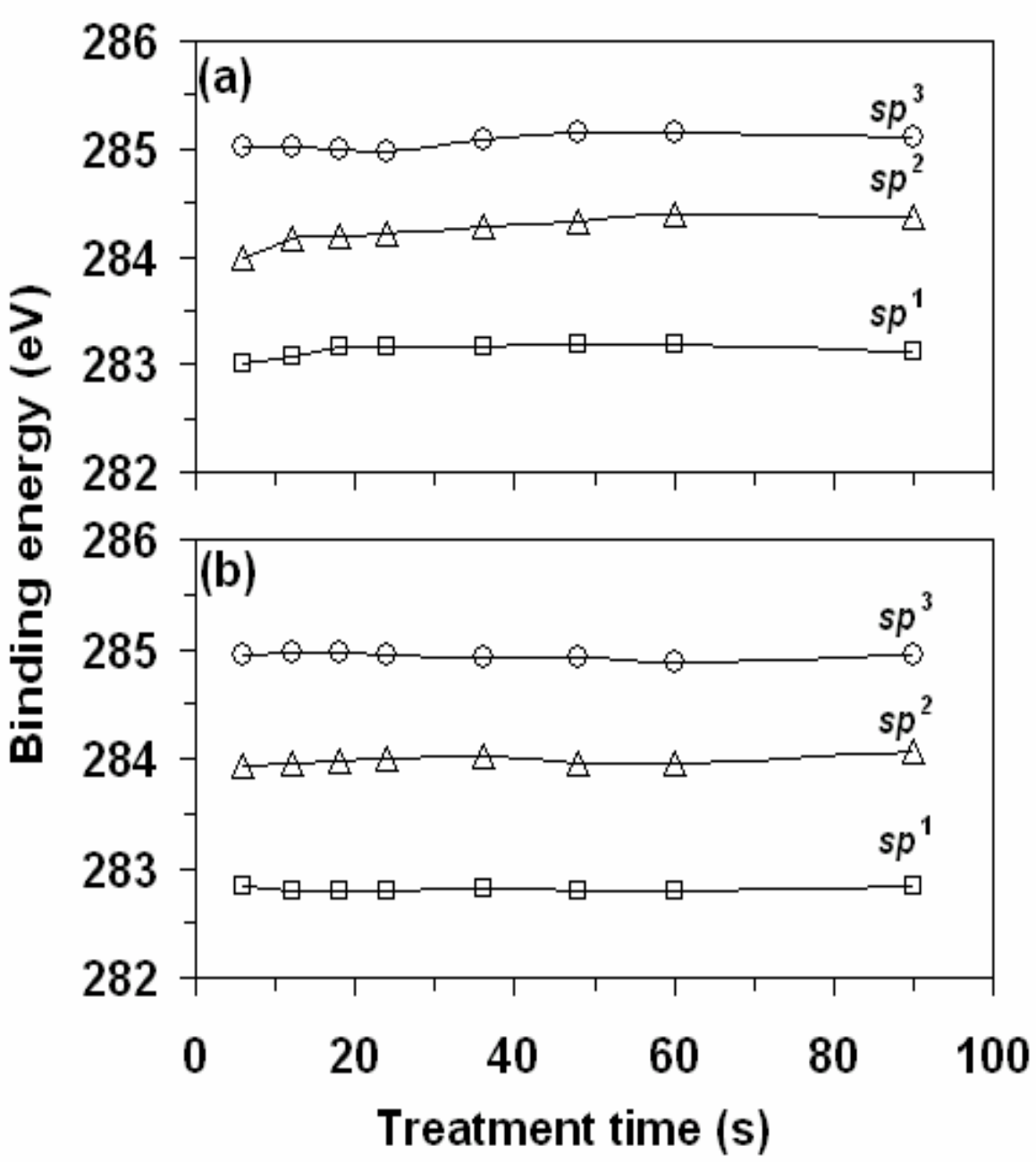
(a)
(b)
sp3
sp2
sp1
Binding energy (eV)
286
285
284
283
282
0
20
40
60
80
100
Treatment time (s)

Figure 7

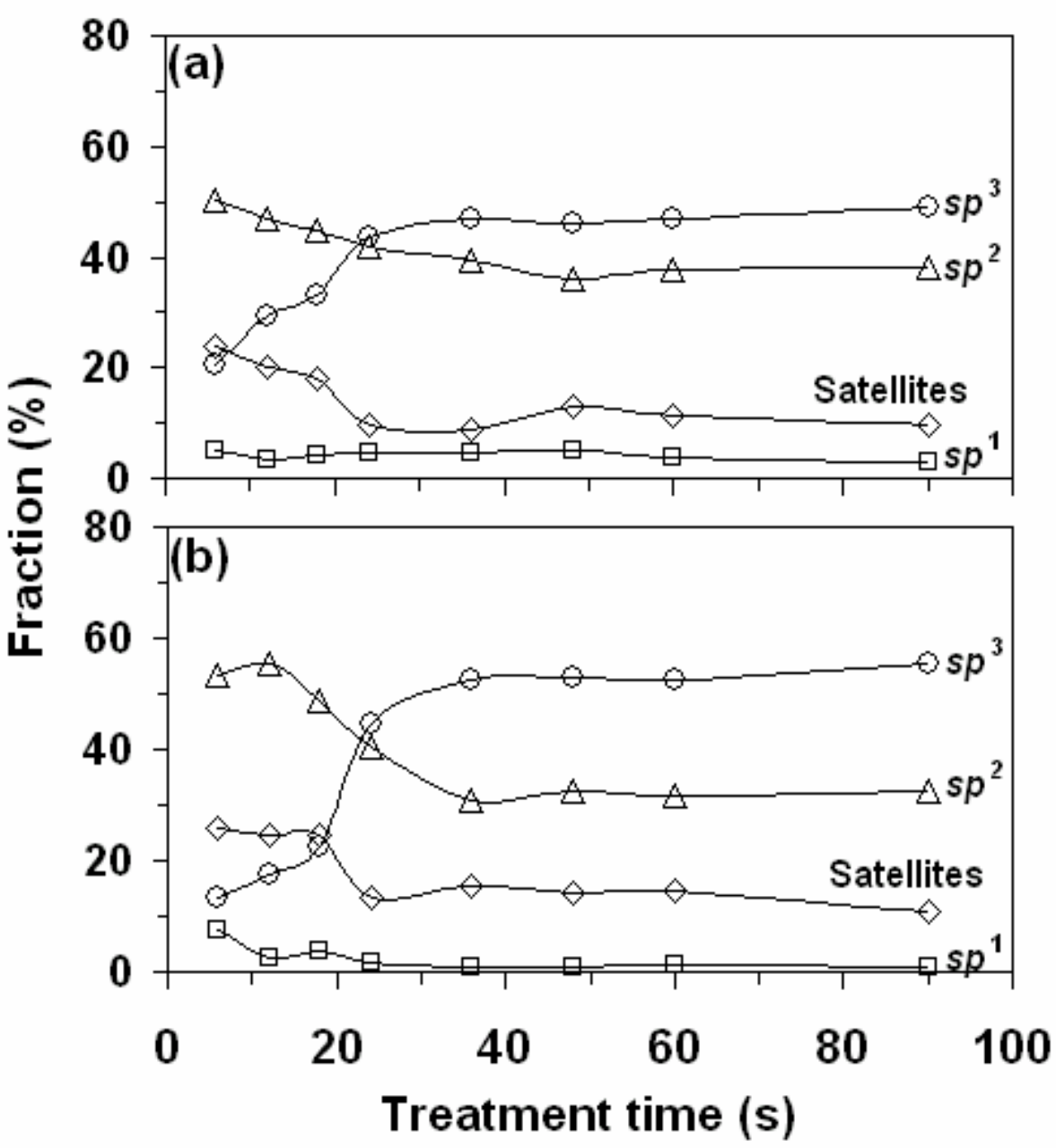
(a)
(b)
Fraction (%)
Treatment time (s)
Satellites
Satellites
sp 3
sp 2
sp 1
sp 3
sp 2
sp 1
0
20
40
60
80
100

Figure 8

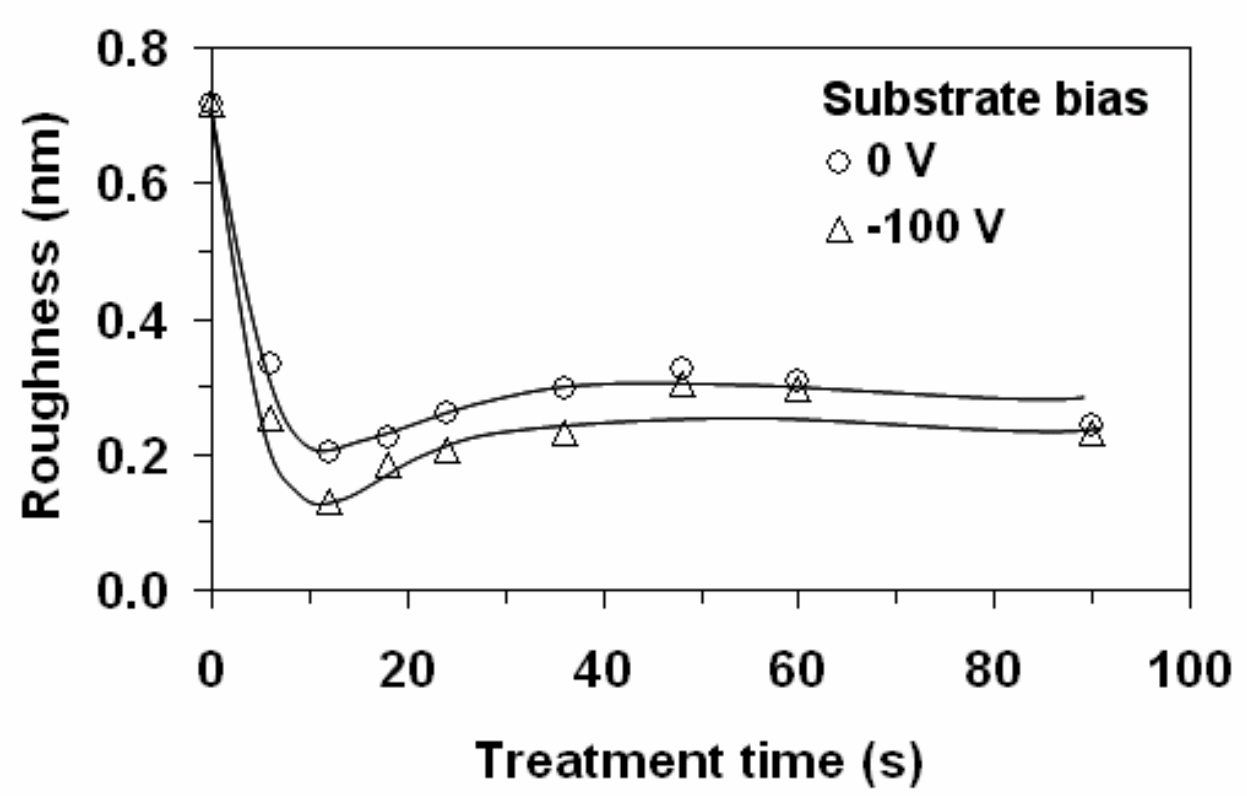
Roughness (nm)
0.8
0.6
0.4
0.2
0.0
0
20
40
60
80
100
Treatment time (s)
Substrate bias
0 V
-100 V

Figure 9

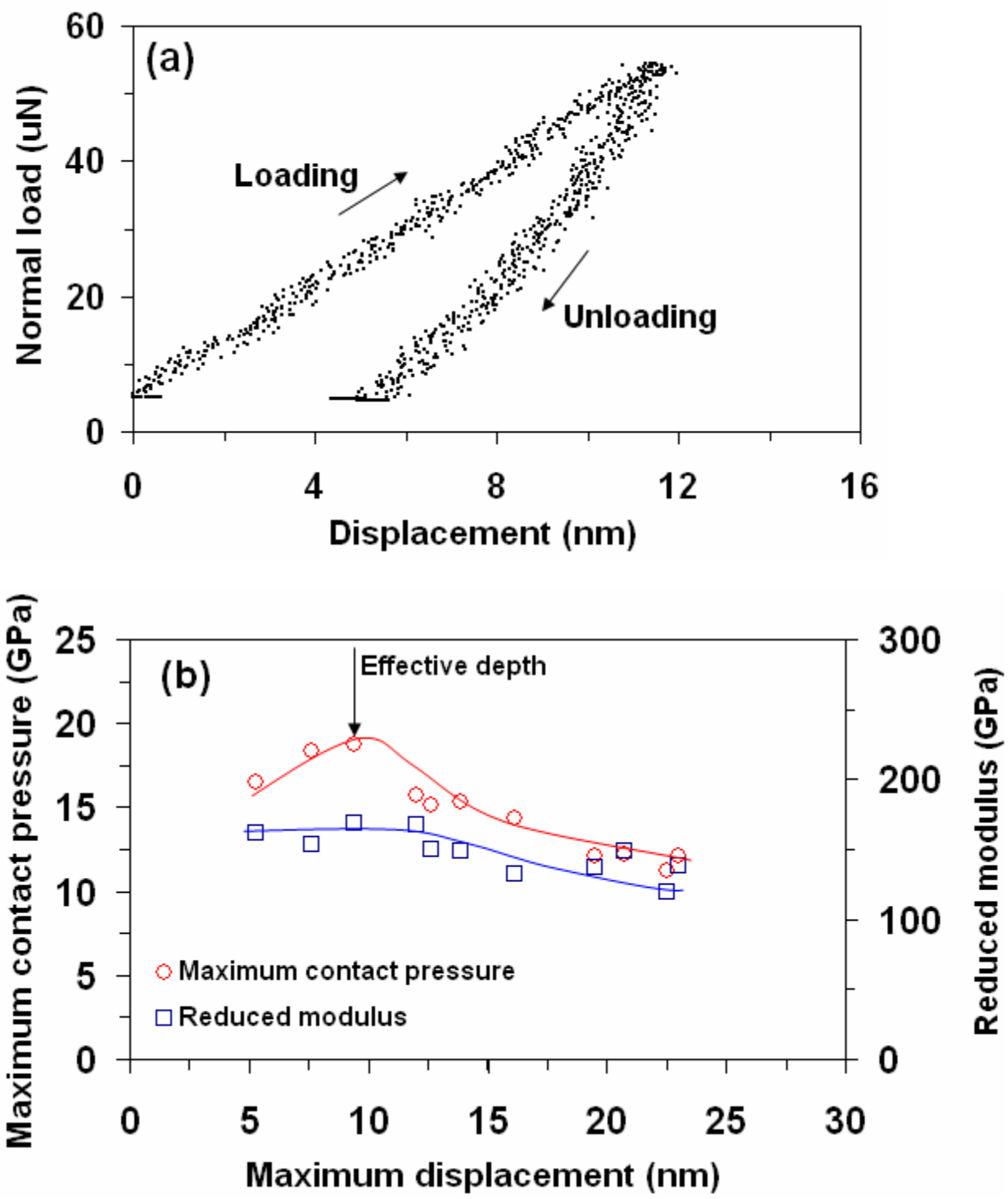
(a)
Loading
Unloading
Normal load (uN)
Displacement (nm)
0
20
40
60
0
4
8
12
16
(b)
Effective depth
Maximum contact pressure
Reduced modulus
Maximum contact pressure (GPa)
Reduced modulus (GPa)
Maximum displacement (nm)
0
5
10
15
20
25
0
100
200
300
0
5
10
15
20
25
30

Figure 10

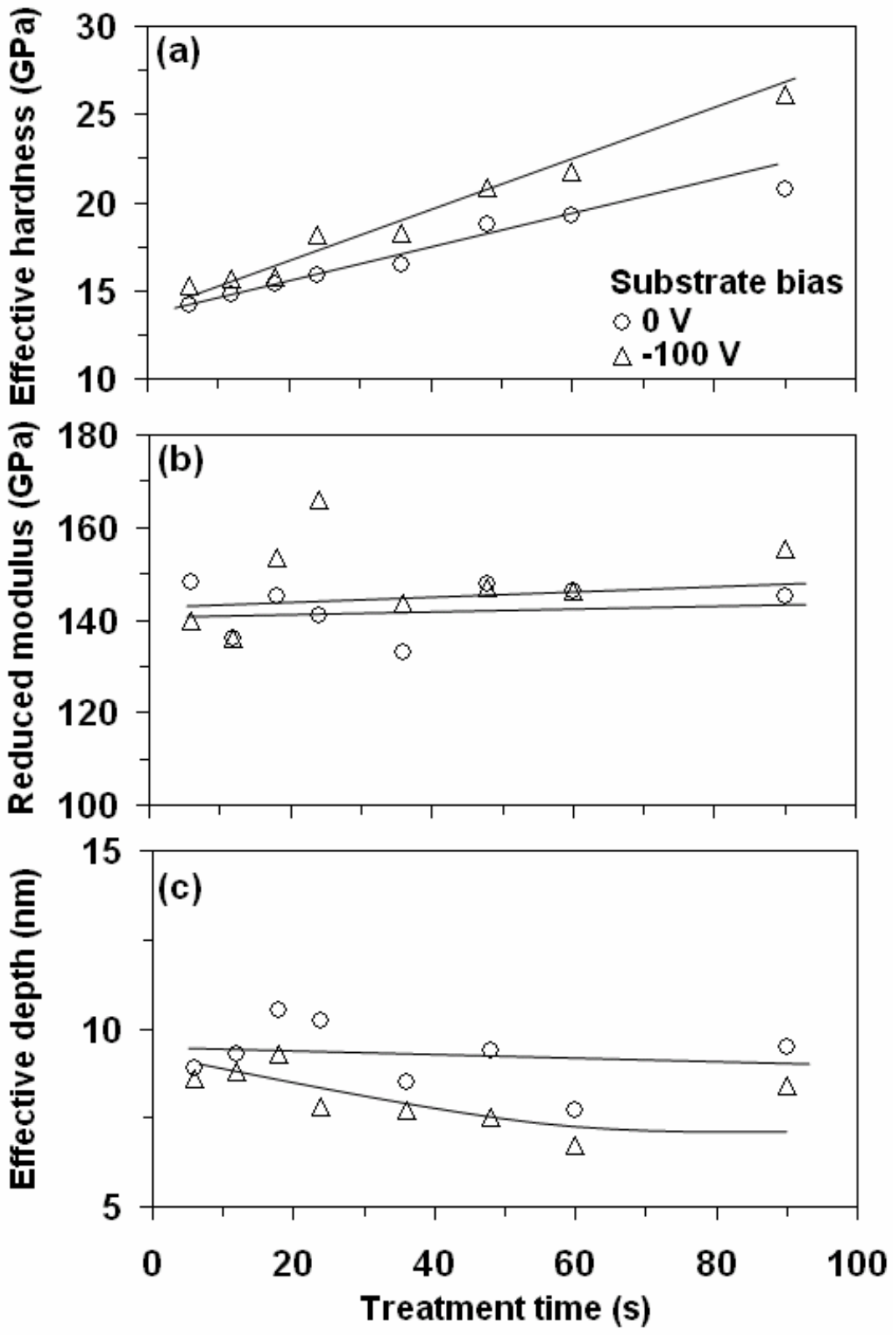
(a)
(b)
(c)
Effective hardness (GPa)
Reduced modulus (GPa)
Effective depth (nm)
Treatment time (s)
Substrate bias
0 V
-100 V
30
25
20
15
10
180
160
140
120
100
15
10
5
0
20
40
60
80
100

Figure 11