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The effects of post-deposition ion-beam bombardment with oxygen on the Co surface in modifying the magnetic properties of Co thin films



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ARTICLE INFO

Article history: Received 17 July 2015 Received in revised form 18 December 2015 Accepted 28 December 2015 Available online 30 December 2015

Keywords: Cobalt Ion-beam bombardment Magnetic property

ABSTRACT

Ion beam bombardment is a useful surface modification method in tailoring the microstructure and magnetic properties of ferromagnetic thin films. In this paper, Co thin films were bombarded by Ar^+ ion beam with oxygen and then capped with Al (to prevent oxidation). The oxygen content was altered from 0 to 41% to investigate the ion-beam bombardment effect. An oxide layer containing Al_2O_3 , Co_3O_4 and CoO was formed after the ion-beam bombardment with oxygen, as characterized by x-ray photoelectron spectroscopy, high-resolution transmission electron microscopy, selected area electron diffraction, and x-ray diffraction. Enhanced coercivities (H_c's) (from 4.14 kA/m to 7.96 kA/m) and loop squareness (M_r/M_s from 0.5 to 0.8) was induced by pure Ar^+ ion-beam bombardment that resulted in domain wall pinning due to bombardment-induced defects and Co spin reordering. H_c exhibited a strong dependence on the oxygen content in the ion beam, due to the nature of the cobalt oxide formation. Field cooling to 180 K resulted in increased squareness ($M_r/M_s = 0.9$) and reduced H_c. The low-field magnetization measured during zero field and field cooling to further investigate the exchange bias magnetism identified an irreversibility temperature at 320 K in a film treated by ion-beam bombardment with an O_2/Ar ratio of 21%.

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1. Introduction

Controlling and modifying the magnetic properties of ferromagnetic (FM) thin films have been drawing significant research interest for tailoring the performance of spintronic devices such as giant magnetoresistance sensors [1] and magnetic random access memories [2]. The magnetic properties of ferromagnetic (FM) thin films could be tailored by conducting surface modification or post treatment. Ion-beam bombardment is a frequently used surface modification technique — ionbeam bombardment can modify the orientation of FM spins, resulting in altered coercivity (H_c) and exchange bias (H_{ex}) [3]. Also, the chemical composition of the FM layer can also be changed by introducing a reactive gas (such as oxygen) into the ion beam [4]. The formed compounds and the FM layer will establish interactions (such as exchange coupling [5]) and modify the magnetic properties. Post treatment is another effective method for altering both the microstructure and magnetic

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properties of magnetic thin films. Field cooling (FC) and zero-field cooling (ZFC) is used to change the magnetic properties of FM thin films. The orientations of FM spins are frozen when cooling through the Curié temperature (T_c), maintaining the state favored by the internal magnetic anisotropy energy (ZFC) or the external Zeeman energy (FC) [6]. Thus the orientations of FM spins are influenced strongly by the cooling field.

Cobalt is a ferromagnetic material with high T_C (~1388 K [7]) and large anisotropy energy ($4.26 \times 10^6 \text{ erg/cm}^3$ for α -Co [8]), thus having broad applications in magnetic recording [9] and magnetic multilayers with perpendicular magnetic anisotropy [10,11]. Certain cobalt oxides are antiferromagnetic at low temperatures, such as CoO (T_N ~ 291 K [12]) and Co₃O₄ (T_N ~ 40 K [13]). Ever since the discovery of the exchange bias effect in CoO coated Co particles [14], various surface modification techniques have been introduced to alter the magnetic properties of Co thin films. The CoO could be formed as a result of natural oxidation in air or oxygen [15–17], in-situ oxidation during Co deposition [18–22], and oxygen ion implantation [23,24]. Ion-beam bombardment was also reported in modifying the magnetic properties of Co thin films. In-situ Ar-ion-beam bombardment with oxygen during Co deposition resulted in pillar like CoO structures in the Co layer [25]. However, the effect of post ion-beam bombardment after the Co deposition on modifying the microstructure and magnetic properties of Co thin film remained unclear, and the influence of oxygen content in the ion beam was not yet investigated. In this paper, the composition, microstructure, and magnetic properties of a Co layer treated by post ion-beam bombardment with oxygen was investigated. The changes in the structural and magnetic properties induced by ion-beam bombardment were tracked by changing the oxygen content in the ion beam from 0% to 41%. The comparison between field-cooling (FC) and zero-field-cooling (ZFC) the film for magnetization measurements further revealed the applied field effect in the Co-O/Co structures' magnetism.

2. Experimental methods

26-nm-thick Co single layers were deposited on thermally oxidized silicon substrates by dual-ion-beam deposition technique [26]. A Kaufman deposition source operated at $V_K = 800$ V was engaged to sputter the Co target at base pressure of 4.0×10^{-7} Torr and working pressure of 3.0×10^{-4} Torr. After the Co deposition, Ar⁺ ion-beam bombardment with oxygen (O₂/Ar ratio ranging from 0 to 41%) was conducted on Co surface for 10 min by an End hall ion source operated at V_{EH} = 70 V. 6-nm-thick Al layers were deposited subsequently to prevent surface oxidation.

The composition and binding energies of the Al 2p, Co 2p and O 1s transitions of the Al/Co–O (15% O₂/Ar)/Co and Al/Co–O (41% O₂/Ar)/Co films were characterized by a commercial ULVAC-PHI (PHI 5000 Versa Probe) x-ray photoelectron spectroscopy (XPS) equipped with a X-ray source (Al K α radiation) and a 5 kV Ar ion gun. The surface and cross-sectional morphologies of both ion-beam bombarded and

un-bombarded Co layers were characterized by a JOEL-2100F highresolution transmission electron microscope (HRTEM). The crystalline structures were characterized from selected area electron diffraction (SAED) by HRTEM and x-ray diffraction (XRD) patterns by a Bruker-AXS D8 Advance. The magnetic hysteresis loops of all the samples were measured by an ADE-DMS 1660 vibrating sample magnetometer (VSM) at room temperature and 180 K after cooling in a 1.2 T magnetic field. The temperature dependent magnetization was measured with 10 mT between 50 K and 400 K by a Quantum Design VSM after FC and ZFC processes.

3. Results and discussions

The depth profile XPS of Al/Co-O (15% O₂/Ar)/Co and Al/Co-O $(41\% O_2/Ar)/Co$ films are shown in Fig. 1 and Fig. 2, respectively. The samples were sputter etched at ~13.3 nm/min, and the bonding energy spectra and concentrations of Co, Al, O and Si were characterized at 0.1 min or 0.08 min intervals. In the depth profile of the elemental concentration in the Al/Co-O (15% O₂/Ar)/Co film (Fig. 1(a)), a clear oxide layer (corresponding to sputtering time (T_{sp}) ranging from 0.4 min to 0.8 min) is shown. In addition, the high concentration of Al in the oxide layer (30%) evidences strong Al diffusion. Both metallic Al and alumina exist at the Al/oxide interface, as shown in the double peak at 72.2 eV and 74.6 eV in the Al 2p spectrum (Fig. 1 (b), $T_{sp} = 0.4 \text{ min}$). Al atoms in the form of Al₂O₃ and a CoAl_x alloy were detected at the oxide/Co interface, which is indicated by the broadened Al 2p peak at around 74.8 eV (Fig. 1 (b), $T_{sp} = 0.7$ min). The Al atoms in the Co layer are likely due to the Al diffusion. Another phase at the oxide/ Co interface is cobalt oxide, as shown by the sub-peak at 780.3 eV (Co₃O₄) [27] and 780.9 eV (CoO) [28] in Co 2p_{3/2} spectra (Fig. 1 (c), $T_{sp} = 0.7$ min). The formation of a CoAl_x alloy was also indicated by



Fig. 1. (a) The depth profile of the chemical concentrations in the Al/Co–O (15% O₂/Ar)/Co and (b) the Al 2p, (c) Co 2p, and (d) O 1s spectra at each sputter etching time measured by XPS.



Fig. 2. (a) The depth profile of the chemical concentrations in Al/Co-O (41% O₂/Ar)/Co and (b) the Al 2p, (c) Co 2p, and (d) O 1s spectra at each sputter etching time measured by XPS.

the major peak at 783 eV in Fig. 1 (c). The O 1s peak at around 531.5 eV indicates that the major oxide in the oxide layer is Al₂O₃. However, the asymmetric O 1s peak at $t_{sp.} = 0.7$ min contains a sub-peak at 531 eV, which corresponds to the binding energy of Co-O in Co₃O₄ [29]. The chemical composition of the Al/Co-O $(41\% O_2/Ar)/Co$ (Fig. 2 (a)) is similar to that in the Al/Co-O (15% O₂/Ar)/Co. However, the increased oxygen content in the ion beam used during deposition resulted in more oxidation of Al and Co. A new Al 2p peak at 75.4 eV in the Al/oxide interface was observed in Fig. 2 (b) ($t_{sp} = 0.48 \text{ min}$), indicating the changes in stoichiometry of AlO_x. In addition, the formation of cobalt oxides was greatly promoted in the oxide/Co interface. The asymmetric Co 2p peak at the oxide layer contains a sub-peak at around 781 eV (Fig. 2 (c), $t_{sp.} = 0.74$ min), which possibly derived from the Co-O band in Co₃O₄ (780.3 eV [27]) and in CoO (780.9 eV [28]). The peak shifting from 532 eV (Fig. 2 (d), $T_{sp} = 0.48$ min) to 531 eV (Fig. 2 (d), $T_{sp} = 0.64$ min and 0.74 min) also indicates the composition transition from the Al/oxide interface to the oxide/Co interface is Al₂O₃ to CoO/Co₃O₄. It should be noted that the concentration of Al (~20%) in the Co layer of the Al/Co-O (41% O₂/Ar)/Co film is slightly greater than that of the Al/Co-O (15% O₂/Ar)/Co film (10%-15%). It is possible that the measured Al content is partially contributed by the mixing during sputter etching. However, since the same sputtering condition was used during the characterization of the two samples, the contribution of the mixing should be the same. So Al diffusion was more pronounced in the Co layers bombarded under higher O₂/Ar ratio, which was interpreted as follows. The higher O₂/Ar ratio resulted in more oxides on the surface of Co, as relatively higher O content was observed in the oxide layer of Al/Co-O $(41\% O_2/Ar)/Co (67\% in Fig. 2 (a))$ than that in Al/Co-O (15% O₂/Ar)/Co (63% in Fig. 1(a)). The grain boundaries and dislocations between the oxides and adjacent phases provided diffusion path for the subsequently deposited Al atoms. So the higher content of oxides at higher O_2/Ar ratio resulted in stronger Al diffusion into Co layer.

In order to investigate the effect of ion-beam bombardment on the crystalline structure, the cross-sectional morphology of the Al/Co-O $(30\% O_2/Ar)/Co$ film was characterized by HRTEM, as shown in Fig. 3. Unlike the in-situ bombarded Co/CoO bilayer, which generated spikelike CoO structures penetrating into Co [30], the post-bombardment only resulted in a 4-nm-thick disordered oxide layer on the surface of Co. This is because the relatively low kinetic energy of O_2^- ions (70 eV) resulted in small penetration depth inside Co. To compare the interplanar lattice spacing of the crystalline structures depicted in Fig. 3, a face-centered cubic (FCC) Co (space group: 225 fm-3m), hexagonal close-packed (HCP) Co (space group: 194 P63-mmc), rocksalt CoO (space group: 225 fm-3m), and spinel Co₃O₄ (space group: 227 Fd-3m) lattices were investigated by conducting first-principles density functional theory (DFT) calculations. We employed the Cambridge Serial Total Energy Package (CASTEP) [31] based on spin polarized GGA-PW91 functionals [32,33]. The interlayer spacings in regions (1) and (2) of Fig. 3 are 2.05 Å and 2.08 Å, respectively, in excellent agreement with the corresponding calculated values, 2.05 Å, determined from the interplanar spacing of the (111) plane in the FCC Co. The interlayer spacings in regions (3) and (4) of Fig. 3 are 2.12 Å and 2.11 Å, respectively, which are approximately 3% smaller than the calculated values of 2.18 Å, determined from the interplanar spacing of the (100) plane in the HCP Co. This smaller interlayer spacing is possibly due to the lattice mismatch with the adjacent heterogeneous phases. In region (5) of the oxide layer of Fig. 3, the interlayer spacing is 2.13 Å, which closely matches the value 2.14 Å found for the interplanar spacing of the (200) plane in rocksalt CoO. On the other hand, we find that the interlayer spacing in region (6) of the oxide layer of Fig. 3 is 2.43 Å, which is in good agreement with the value of 2.47 Å calculated from the



Fig. 3. The cross-sectional morphology of the Al/Co–O (30% $\rm O_2/Ar)/Co$ film characterized by HRTEM.

interplanar spacing of the (311) plane in the spinel Co_3O_4 . It should be noted that bombardment-induced Co-oxide grains are not likely to be the highly ordered structures since they are small in size and are surrounded by amorphous phases. This may result in deviations in lattice parameters and magnetic properties from the ordered crystalline phases.

In addition, the morphologies of all the samples were characterized by bright field image HRTEM, and the crystalline structures were analyzed from their SAED patterns, shown in Fig. 4. The Al single layer exhibited amorphous structures in the films, as shown by the extended SAED rings in the inset of Fig. 4(a). The un-bombarded Al/Co bilayers, on the other hand, possessed polycrystalline structures with grain sizes ranging from 5 nm to 15 nm (Fig. 4(b)). (002) plane of HCP Co can be observed in the electron diffraction rings in the inset of Fig. 4(b). CoAl alloy was observed in the un-bombarded Al/Co bilayers, shown by the innermost SAED ring, which corresponds to the (100) and (111) planes in B2 CoAl. The ion-beam bombardment altered the grain sizes, crystalline phases, and preferred orientations of the bilayers, resulting in large changes in surface morphologies as shown in Fig. 4(c) to (h). Broadened distributions of grain sizes, and nonuniform surface textures were observed on the surface of the ion-beam bombarded samples. The pure Ar⁺ ion beam changed the preferred orientation of the grains in Co layer, as shown by the electron diffraction rings corresponding to HCP Co (100), (102) and (112) planes in the inset of Fig. 4(c). The CoAl (210) plane was observed in all the ionbeam bombarded films. The preferred orientations of the spinel Co₃O₄ phase formed by the ion-beam bombardment with oxygen was (511) (insets of Fig. 4(d) to (h)). This is also different from that of the naturally oxidized Co₃O₄ (311). The samples bombarded by the ion beam with oxygen also exhibited the rock-salt CoO phase as shown by the CoO (331) and (111) electron diffraction ring in the insets of Fig. 4(d) to (h).

The changes in crystalline structure among films were also observed in their respective XRD patterns, presented in Fig. 5. A broadened major reflection at $2\theta = 44.3^{\circ}$ was observed in the XRD patterns of all the samples. This broadened reflection resulted from the overlay of ordered B2 CoAl (110) ($2\theta = 44.741^{\circ}$), and HCP Co (002) ($2\theta = 44.762^{\circ}$) reflections. Co atoms form HCP Co (a = 2.507 Å, c = 4.07 Å) and B2 CoAl ($a \sim 2.83$ Å) lattices in the ion-beam bombarded Co layers, as observed



Fig. 4. The morphologies and SAED patterns of the un-bombarded and ion-beam bombarded Co with Al capping layer characterized by TEM.

in the SAED patterns (Fig. 4) and XPS spectrum (Fig. 1 and Fig. 2). In addition, rock-salt CoO (a ~ 4.24 Å) and spinel Co₃O₄ (a ~ 8.09 Å) was formed as a result of the oxidization of O₂/Ar ion beam, as proved by the CoO (200) peak at $2\theta = 42.6^{\circ}$ and Co₃O₄ (222) peak at $2\theta = 38.6^{\circ}$ in the XRD pattern.

The compounds that formed at the interface can be interpreted by way of the thermodynamics point of view. During the ion-beam bombardment with oxygen, cobalt oxides including CoO and Co_3O_4 could be formed. However, in the initial stage of the bombardment, oxygen atoms preferred to form Co_3O_4 since Co_3O_4 (-891 KJ/mol [34]) has more negative enthalpy of formation than CoO (-237.9 KJ/mol [34]). As the amount of oxygen on the Co surface increases, CoO content could also be formed although the content is relatively low. This is proved by the XPS results which indicate that the content of cobalt



Fig. 5. The XRD pattern of the Co layers bombarded by ion beam with O_2/Ar ratio ranging from 0 to 41% and capped with Al layer.

oxides increased remarkably in samples bombarded by the ion beam with higher O_2/Ar ratio. The abundant oxygen atom at the surface of the Co layer reacted with the subsequently deposited Al since Al_2O_3 has the most negative enthalpy of formation (-1669.8 KJ/mol [34]). Since no evidence of Al_2O_3 grains was observed in the SAED and XRD analyses, the alumina more likely possesses an amorphous structure. Despite its small enthalpy of formation (-110 kJ/mol [35]), CoAl was also formed at the Al/Co interface where both Co and Al were in supersaturated state [35], as shown in the XRD patterns. The above analyses in chemical compositions and crystalline structures reveal that the chemical contents and microstructures were altered by the ion-beam bombardment. These changes influenced their magnetic properties to a great extent, which were characterized by VSM measurements.

The magnetic hysteresis loops measured at 298 K and 180 K after field cooling with 1.2 T magnetic field are shown in Fig. 6(a) and (b). At 298 K, the un-bombarded Co layer presented soft magnetism (low H_c of 4.14 kA/m) with a concomitant rounded hysteresis loop $(M_r/M_s = 0.5)$. The pure Ar⁺ ion-beam bombardment resulted in enhanced H_c (7.96 kA/m) and squareness ($M_r/M_s = 0.8$). This is likely due to the formation of defects (vacancies, dislocations, and implanted Ar atoms) and the reordering of the surface Co spins due to the ionbeam bombardment, which act as pinning centers for domains during the magnetic reversal process. After the ion-beam bombardment with oxygen, the film's hysteresis loops maintained similar squareness (~0.8) with H_c ranging from 4.32 kA/m to 8.36 kA/m. The field cooling to 180 K resulted in a reduced H_c (2.39 kA/m to 5.97 kA/m) in the ionbeam bombarded samples. This is possibly due to the competition between the magnetocrystalline anisotropy of different phases. When the films were cooled to below the T_N or T_C of one (or more) phase, it (or they) starts to contribute to and gradually dominates the magnetization reversal processes of the films at low temperature. Squarer hysteresis loops $(M_r/M_s \sim 0.9)$ was observed at 180 K, indicating an enhanced magnetic anisotropy at lower temperatures after field cooling [36]. Unlike the bilayers after in-situ bombardment where large H_{ex} (~50 kA/m) was measured [30], only small loop shifts ($H_{ex} < 477$ A/m) were observed in the hysteresis loops of all the post-bombarded samples. This is because Co was more readily oxidized into Co-oxide by in-situ oxidization, while relatively small amount of Co-oxide was formed after post bombardment, as inferred from the composition analyses above. This weak exchange coupling was unable to pin Co spins during magnetization reversal process. In order to further analyze the influence of oxygen content in the ion beam, the H_c and H_{ex} measured at 298 K and



Fig. 6. Magnetic hysteresis loops measured at 298 K (a) and 180 K (b) after field cooling at 1.2 T magnetic field. (c) Magnetic coercivity (H_c) and exchange bias (H_{ex}) of Co bombarded by ion beam with O_2/Ar ratio ranging from 0 to 41% and capped with Al layer.

180 K were plotted against the O₂/Ar ratio, as shown in Fig. 6(c). Assuming $H_{ex}(T)$ was similar between films (i.e. comparable blocking temperatures), at lower O₂/Ar ratios, H_c decreases with increasing oxygen content. When the O₂/Ar ratio is higher than 15%, an enhanced H_c was observed with the higher oxygen concentrations. Clearly, the ionbeam bombardment with oxygen plays an important role in Co anisotropy as well as magnetization reversal processes. Compared with the

un-bombarded films, the structural changes in the ion-beam bombarded Al/Co bilayers include the emergence of (1) bombardment-induced defects (vacancies, dislocations, and implanted ions), (2) diffused Al and O atoms in the grain boundaries, and (3) Co oxides. The first factor is considered to be to the same in all the ion-beam bombarded samples since the same V_{EH} and Ar gas flow was used in the ion-beam bombardment. The diffused Al and O atoms inside the Co layer accumulate in the grain boundaries. Some Al atoms react with Co to form CoAl_x. With the increasing oxygen content in the ion beam, the concentration of the diffused Al increases, as shown in the XPS results. Similarly, it can be inferred that the content of the diffused O also increases with O₂/Ar ratio. These defects lead to lower H_c since the diffused atoms and CoAl_x reduce the overall magnetocrystalline anisotropy of the Co layers [37-39]. Besides, Co oxides were formed in the interfacial layer as a result of the ion-beam bombardment with oxygen. The Co oxides at domain walls acted as the pinning centers during the magnetization reversal, leading to the higher surface anisotropy, and the increased H_c. As the content of Co-oxides increases with the O_2/Ar ratio, this effect tends to increase H_c at higher O_2/Ar ratio. This H_c dependence on the O₂/Ar ratio is believed to be the result of competition between the reduction of magnetocrystalline anisotropy due to Al and O diffusion and the increase in surface anisotropy due to Co-oxide formation. At lower O₂/Ar ratio, the content of Co oxide is relatively low, so the pinning effect at the domain wall is also relatively weak over the whole film's magnetism. However, relatively high Al concentration in Co was characterized by XPS (10-20%). This indicates that the ion-beam bombardment effects on the overall magnetism were defect-induced reductions of the magnetocrystalline anisotropy. This resulted in the lowest H_c (4.32 kA/m at 298 K) for the 15% O₂/Ar sample. The domain wall pinning effects of Co oxides are dramatically increased with increasing O₂/Ar ratio, while the concentration of diffused Al and O gradually approaches saturation. So the overall H_c is dominated by the surface anisotropy enhancement induced by Co oxides at O₂/Ar higher than 15%. This mechanism leads to the increasing H_c with O_2/Ar ratio until the highest H_c (5.97 kA/m) at 41% O₂/Ar.

The temperature dependence of the low field (10 mT) magnetization from ZFC and FC processes are presented in Fig. 7. In all the films, the magnetization decreased monotonically with increasing temperature. FC and ZFC curves exhibited only small variations in most of the samples due to the similar crystallite sizes (resulting in similar onset temperatures of superparamagnetism, which are above 300 K since H_c's are still measurable at that temperature) and the high Curié temperature of Co (T_C ~ 1388 K [7]). However, the separated FC and ZFC curves merge at around 320 K, which is known as the irreversibility temperature (T_{irr}). This is likely due to the magnetic transition of CoO from AF to paramagnetic above T_N ~ 291 K. The uncompensated AF interfacial spins begin to give rise to enhanced moment at lower temperatures [40,41]. The smaller content of CoO in this experiment is also responsible for the little difference in the FC and ZFC curved in Fig. 7. Further characterization down to 5 K to obtain blocking temp or significant separation between FC and ZFC is in progress.

4. Conclusions

The influence of oxygen content used during the post-deposition ion-beam bombardment on Co layers' magnetism was investigated in this paper. Different microstructure and magnetic properties were observed compared with the in-situ treatment reported previously. Naturally oxidized Al₂O₃, B2 CoAl, and HCP Co existed in the unbombarded Al capped Co film. Increasing the oxygen content in the ion beam resulted in the formation of amorphous Al₂O₃, rock-salt CoO, and spinel Co₃O₄ in the Al/Co interface. The magnetic properties of the Co film were also modified due to these compositional and microstructural changes. The Ar⁺ ion-beam bombardment created defects in Co layer and resulted in pinned Co interface spins, resulting in enhanced anisotropy and higher H_c (from 4.14 kA/m to 7.96 kA/m). Both enhanced and reduced H_c were observed after the ion-beam bombardment with oxygen due to the competing effects between the reduced magnetocrystalline anisotropy by Al and O diffusion (which decreased H_c) and the increased surface anisotropy by the pinning effect of Co oxides at domain walls (which increased H_c).The low field magnetization (susceptibility) increased monotonically with decreasing temperature during ZFC and FC measurements, with an irreversibility temperature at around 320 K in the Al/Co-O (21% O₂/Ar)/Co. This work provides an effective method for the modification of the structural, compositional,



Fig. 7. Temperature dependence of magnetic moment of ion-beam bombarded Co capped with Al layer after field cooling (FC) and zero field cooling (ZFC) process.

and magnetic properties of Co layers through ion-beam bombardment with oxygen and argon.

Acknowledgments

This work was supported by the Seed Funding for Basic Research and Small Project Funding from the University of Hong Kong, the RGC-GRF grant (HKU 704911P), ITF Tier 3 Fundings (ITS/104/13, ITS/ 112/12), the University Grants Committee of Hong Kong (contract no. AoE/P-04/08), the Natural Sciences and Engineering Research Council (NSERC) of Canada, the Ministry of Science and Technology (MOST) of Taiwan, and the Norwegian Research Council.

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