

Wednesday Afternoon Poster Sessions, December 14, 2016

Thin Films

Room Mauka - Session TF-WeP

Thin Films Posters Session II

TF-WeP1 High Hardness and Oxidation Resistance of Ti–Al–Si–N Nanocomposite Coatings for Machining of Aircraft Mechanical Parts, Wang-Ryeol Kim, Korea Institute of Industrial Technology, Korea; T.H. Nam, University of Ulsan, Republic of Korea; I.-O. Baek, E.Y. Choi, J.B. Jeon, Korea Institute of Industrial Technology, Korea

Superhardness Ti–Al–Si–N nanocomposite coatings were deposited on SKD 61 and inconel substrates by hybrid coating system (AIP+Sputtering) in various Si target powers for machining of aircraft mechanical parts. The relationship among microstructures, mechanical properties, and tribological properties was investigated. The synthesized Ti–Al–Si–N coatings were characterized using x-ray diffraction (XRD) and x-ray photoelectron spectroscopy (XPS). These analyses revealed that the Ti–Al–Si–N coatings are nanocomposites consisting of solid-solution (Ti,Al)N crystallites distributed in an amorphous Si_xN_y matrix. The addition of Si into the Ti–Al–N coating led to percolation of amorphous Si_xN_y and BN phases. The Ti–Al–Si–N coatings exhibited high hardness and H/E values, indicating high fracture toughness, of approximately 42 GPa and 0.097, respectively. Furthermore, the minimum friction coefficient of the Ti–Al–Si–N coatings was approximately 0.25 at low Si target powers. A systematic investigation on the microstructures, mechanical properties, and tribological properties of Ti–Al–Si–N coatings prepared from three Ti–Al composite targets and pure Si target is reported in this study.

TF-WeP2 Microstructure, Mechanical Properties and Tribological Properties of ZrN Based Nanocomposite Hard Coatings Deposited by a Hybrid Coating System, Yeongha Song, Korea Institute of Industrial Technology (KITECH), Pusan National University, Republic of Korea; H.D. Kim, University of Ulsan, South Korea; K.-S. Kim, S.M. Shin, Korea Institute of Industrial Technology (KITECH); J.-H. Kim, Korea Institute of Industrial Technology (KITECH), Republic of Korea

Zr–Si–N coatings with various Si contents were synthesized by a hybrid coating system combining arc ion plating and d.c. magnetron sputtering techniques. The influence of the Si content on the microstructure, mechanical properties and tribological properties of the Zr–Si–N coatings were investigated. The results showed that as the Si content increased from 0 to 5.8 at.%, the coatings transformed from a columnar ZrN to nanocomposite structure with a nano-sized ZrN crystallites embedded in amorphous Si₃N₄ matrix and further increases of Si content lead to amorphous-like coatings. The maximum hardness (H) of 33 GPa, Young's modulus (E) of 265 GPa, and H/E* value of 0.12 were obtained at Si content of 5.8 at.% in the Zr–Si–N coatings, respectively. The coating with the lowest friction coefficient and the best wear resistance was also obtained at Si content of 5.8 at.%, which was mainly contributed to high value of H/E* and formation of SiO₂ or Si(OH)₄ self-lubricating layer due to the interface tribo-chemical reaction.

TF-WeP3 Advanced High Toughness Hard Coatings for Tribological Applications, Sung-bo Heo, U.C. Jung, Korea Institute of Industrial Technology (KITECH), Republic of Korea; H.J. Son, University of Ulsan, Republic of Korea; I.W. Park, Korea Institute of Industrial Technology (KITECH), Republic of Korea

A number of multi-component, nanostructured coatings have been produced for a range of tribological applications. The paper will discuss four such nanocomposite coating systems: (i) Ti–B–C–N; (ii) Ti–Al–Si–N; (iii) Cr–Al–Si–N; and (iv) Zr–Al–Si–N produced by hybrid coating deposition system in which the deposition parameters have been varied to control the structure and properties of the films. In each case, the coating system needs to exhibit a range of tribological properties to meet the required application, such as high wear resistance, low coefficient of friction, self-lubrication, high oxidation and/or corrosion resistance. In particular, the effect of the film chemistry, pulsing the magnetron and utilizing a closed field configuration will be discussed as suitable control parameters in tailoring the structure and properties of the coatings to meet specific tribological applications.

TF-WeP5 Migration-enhanced Epitaxy using Ga Droplets of GaAsNSe Thin Films on Si(110) Substrates, Katsuhiko Uesugi, T. Ozawa, Y. Igarashi, Y. Shimomura, S. Kimura, K. Obara, Muroran Institute of Technology, Japan

The development of strain relaxation buffer layers between GaAs and Si(110) that controlled large lattice mismatch and surface orientation influences are expected to integrate GaAs-based optical-fiber communication devices to Si(110) wafers. The growth of Ge and GeSi layers is mainly studied on Si(001) substrates, but absorb light signal of 1.55 μm wavelength due to the narrow bandgap energy of ~0.67 eV. A two-dimensional growth technique of GaAs(110) films on Si(110) surfaces is also necessary. In this paper, we report the migration-enhanced epitaxy of n-type GaAsNSe thin films with Ga droplets prepared on Si substrate surfaces, as the strain relaxation layers between GaAs and Si(110).

GaAsNSe films were grown by using metal-organic molecular beam epitaxy (MOMBE) equipped with the nitrogen radio-frequency discharge plasma system. MO precursors used were triethylgallium, trisdimethylaminoarsenic (TDMAs), trisdimethylaminoantimony (TDMASb), and ditertiarybutylselenide. The Si(110) substrate surfaces were thermally cleaned at 550 °C with the simultaneous supply of TDMAs. Ga droplets were formed on Sb-terminated Si surfaces which prepared by the supply of TDMASb at the substrate temperature of 470 °C. Then GaAsNSe films were grown at the temperature of 370–510 °C. The growth process of the films was characterized by using RHEED, AFM, and x-ray diffraction methods.

The Si- and As-terminated Si(110) surfaces were not covered with Ga layers, and Ga droplets of 40–75 nm height were formed at a density of 1×10⁸ cm⁻². On the other hand, small Ga droplets of 2 nm height were formed on the Sb-terminated Si surfaces at a high density of 2×10¹⁰ cm⁻², which suggest the anti-surfactant effect of Sb surface atom. The growth of a GaAsNSe ring structure occurred by the isotropic Ga diffusion from the droplet at low temperature. The step-flow growth mode of GaAsNSe was promoted as the increase of growth temperature. The activation energy of the Ga diffusion from the droplet during the growth was estimated to be 0.46 eV. At a high growth temperature of 510 °C, the two-dimensional growth of GaAsSe(110) with a large lattice mismatch of 3.5% was promoted considerably by using the effect of the surface Ga diffusion from the droplets. The formation of the pits and three-dimensional islands was suppressed in comparison with the films prepared by normal MOMBE growth. The migration enhanced MOMBE using surface Ga nano-droplets is a very useful technique for the fabrication of the flat GaAsNSe lattice relaxation layers on Si(110) substrates.

TF-WeP6 Formation of Low Friction Interface Utilizing Diamond-like Carbon Coating Containing Molybdenum Disulfide in Vacuum, Oxygen and Nitrogen, Junko Masuda, K. Adachi, T. Takeno, Tohoku University, Japan

Molybdenum disulfide (MoS₂) is widely used as a solid lubricant in vacuum. It produces very low friction due to its lamellar structure. However, one of the drawback of MoS₂ is weak for oxidation. Recently, the coating that MoS₂ nanoparticles are dispersed into diamond-like carbon (DLC) host matrix is proposed to overcome this problem utilizing gas barrier effect of DLC in addition, its hardness increases. Such coating is called MoS₂-containing DLC (MD). The coating shows lower friction, $\mu \sim 0.01$, than MoS₂ in vacuum when the coating is prepared under specific condition. When it shows low friction, unique transfer layer onto counter surface is formed. The structure of the transfer layer is consisted of 5 nm carbon layer on counter surface and mixed layer composed of MoS₂ and C on it. These facts clearly indicate that such specific structure is organized thanks to the coating. We have also demonstrated that low friction can be achieved depending not only on MoS₂ concentration but also bias voltage during deposition. Carbon matrix is important for achieving low friction. It is indicated that in order to form unique transfer layer, carbon has something potential to support MoS₂ to arrange lamellar structure. The purpose of this study is to clarify mechanisms of low friction.

Friction tests were conducted with ball-on-disk type apparatus in well-controlled environmental conditions. Dry nitrogen, dry oxygen and humid nitrogen gases are used to change atmospheric condition during friction tests. Various observation techniques including scanning electron microscope (SEM) and transmission electron microscope (TEM) equipped with energy dispersive X-ray spectrometer (EDS) are employed in this study.

Under nitrogen gases, both MoS₂ and MD coatings show low friction with vacuum. Actually, it is natural that MD coating shows low friction in nitrogen because MoS₂ and DLC give low friction in this condition. In case

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of oxygen condition, MoS₂ shows higher friction coefficient, ~ 0.2, in oxygen condition than in vacuum. On the other hand, friction coefficient of 0.02 is achieved utilizing MD coating, and very thin transfer layer was formed on the counter surface, which clearly shows the potential for low friction utilizing MD coating. A cross-sectional TEM image of the layer shows that structure of the transfer is also same as the one obtained in vacuum. In addition, EDS mapping on the TEM image revealed that oxygen from atmosphere is included in the whole transfer. It suggests that composite structure of MD coating can contribute the formation of a thin transfer layer.

TF-WeP7 Stability on O₂ Plasma Treatment of DLC Coating as Biocompatibility, Kyoichi Terai, Tokyo Denki University, Japan

Diamond-like carbon (DLC) films have been investigated in domestic fields because of their many attractive properties. In especially, mechanical high hardness, low wear resistance and gas barrier property of DLC films can be mentioned as the dominant advantages. DLC coating has already applied to surface finishing technology of metallic molds, cutting tools and polyethylene terephthalate (PET) bottles. In recent, many researchers have reported the characteristics of chemical stabilities, blood compatibilities and cell affinities on the DLC films. Modification of the surface condition of DLC films has been rapidly expected to demand of the advanced medical care.

In order to improve the biocompatibility of DLC films, plasma treatment of oxygen (O₂) and nitrogen (N₂) gases are proposed on the DLC surfaces¹. Surface conditions of the DLC films are delicate because bonds between carbon and oxygen are not so strong. The stability of effect of plasma treatment for DLC films has to investigate by systematic approach. In this study, we investigate that stability of the surface characteristics for time of O₂ plasma treatment. DLC films were prepared on Si substrate by using r. f. plasma chemical vapor deposition (r. f. plasma CVD) in methane (CH₄) gas. After DLC films deposition, surface modification was carried out by O₂ plasma. To evaluate stabilities of the plasma treatment on surface characteristics, surface conditions were analyzed by contact angle measurement and X-ray photoelectron spectroscopy (XPS). The cell affinity is estimated by *in-vitro* examination with a cell proliferation test using mouse-derived fibroblasts.

In case of contact angle measurement, DLC films without plasma treatments have water repellency and are almost constant. Hydrophilicity of surfaces immediately O₂ plasma treatments is stables for several days, after that their change to the saturation value.

From the result of XPS, intensity ratio of oxygen and carbon peak on the surfaces of the DLC films without plasma treatment is increased as function of time. In contrast, the intensity ratio between oxygen and carbon is decreased with time in case of sample with O₂ plasma treatments. The results revealed that adsorption and desorption of oxygen play important role to shift of the wettability in the plasma treatment on DLC films.

Reference

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TF-WeP8 Characterization of Compound Thin Films of CeO₂ and SiO₂ deposited by MOCVD, Tomonari Furuya, Hosei University, Japan; T. Matsumura, TOSHIBA Corporation, Japan; K. Kikuchi, Hosei University, Japan; K. Ishibashi, S. Suzuki, COMET Inc., Japan; Y. Yamamoto, Hosei University, Japan

Chemical states and electrical properties of the CeO₂ based compound oxide doped with SiO₂ as the promising gate stack material for MOS devices were investigated, based on the consideration that the crystallization could be suppressed by mixing materials having different crystalline structures. The X-ray photoelectron spectroscopy (XPS) analysis revealed that the compound oxide was successfully prepared on p-type Si (100) substrates by pyrolytic MOCVD using Ce(OC(Et)₂Me)₄ at the substrate temperature of 350 °C for 30 min with the intermittent introduction of TEOS (TetraethoxyOrthoSilicate) for 10 sec every 3, 5, or 10 min. The decomposition temperature of TEOS was lowered by the hydrolysis utilizing H₂O generated from Ce source decomposition. In the X-ray diffraction (XRD) patterns, the pure CeO₂ films represented the distinct cubic CeO₂ peaks, while CeO₂ peaks decreased to the trace level for the samples with TEOS introduction; the CeO₂ films with TEOS introduction were essentially amorphous.

From the X-ray photoelectron spectroscopy (XPS) spectra of Ce3d, Si2s and O1s, the average molar concentrations of SiO₂ in the films with the introduction of TEOS for 10 sec every 3, 5, or 10 min were determined to

be 15%, 6% and 6%, respectively. Although TEOS was intermittently introduced during CeO₂ deposition, the distribution of Si in CeO₂ films was uniform and the amount of incorporated Si was not directly related to the TEOS supply rate. Cerium silicate formation in the film prepared with TEOS introduction was confirmed from Si2s peaks at 153.5 eV and O1s spectra appearing with the shoulder at the higher binding energy.

The electrical properties after annealing at 500°C for 30 min in the ambient of O₂ were analyzed by I-V and C-V measurements using the Pt dot electrodes. The sample with TEOS introduction for 10 sec every 3 min represented the lowest leakage current around 1.0×10⁻⁵ A/cm² at 1.5 MV/cm, which was one order of magnitude lower than that of the pure CeO₂ films. The relative dielectric constant ranged between 16.6 and 21.6 depending on the TEOS introduction interval. These values were rather higher than that of pure CeO₂ films without Si doping which was usually lower than that of bulk relative dielectric constant of 26. These results suggest that Si doping brought about the change in the interface structure between Si substrates and deposited films such as the disappearance of the interfacial layer with lower dielectric constant. The flat band voltage shifted toward lower gate voltages for the samples prepared with TEOS introduction probably due to the introduction of fixed positive charge by silicate formation.

TF-WeP9 Development of Visualization System of Permeated Hydrogen Map on Stainless Steel Membrane, Naoya Miyauchi, Toho University, Japan; Y. Murase, National Institute for Materials Science, Japan; H.A. Sakaue, National institute for fusion Science, Japan; A.N. Itakura, National Institute for Materials Science, Japan; S. Takagi, Toho University, Japan

Hydrogen outgassing from an inside wall is the most important issue for vacuum chambers in ultrahigh vacuum (UHV) and extremely high vacuum (XHV). In addition, the behavior of hydrogen in metals should be made clear to understand the mechanisms of hydrogen embrittlement and storage. We have observed the behavior of hydrogen in metals by visualizing sequentially spatial distributions of permeated hydrogen on the surface of stainless steel membrane. The distributions of surface hydrogen were obtained using ions emitted by the method of desorption induced by electronic transition (DIET) process with the scanning electron microscope (SEM). The diffusion pass of hydrogen can be revealed from comparison of hydrogen maps obtained by this technique with the surface grain structure. In addition, physical information on permeation, which contains the processes of solution, diffusion, adsorption, is obtained by conjunction with a measurement on time dependence of permeated hydrogen pressure in the vacuum. The experimental setup is shown in Figure 1. We equipped the SEM (JEOL JAMP10) to the sample holder with hydrogen supply system and collecting electrode of DIET ions, the detection system of DIET ions and the quadrupole mass analyzer (Pfeiffer Vacuum QMG220). The collecting electrode is attached due to focusing DIET ions on the detector system. The DIET signals are measured in two-dimensional pulse counting system constructed by LabVIEW. This two-dimensional pulse counting system is synchronized with the scanning electron beam. The sample is SUS304 stainless steel, which has austenite structure with martensite dislocations caused by cold working of 20 %. The sizes of austenite grains are about 100 μm. The thickness of membrane is 200 μm. After outgassing of hydrogen in the sample (573K: 48 hours) the following experiments were performed under the outgassing temperature. The back side of SUS membrane was exposed to hydrogen (2.7×10⁵ Pa) and the permeated hydrogen on the opposite observation side was observed by DIET method. The vacuum chamber was evacuated by the sputter ion pump (100 l/s) under the experiments. The pressure was 1×10⁻⁷ Pa under the experiment. Both H₂ and D₂ gases were used to investigate an isotope effect for permeation.

Figures 2 and 3 are the secondary electron image and the permeated hydrogen map, which is obtained by accumulating DIET ions for 50 hours at 473 K, respectively. A comparison of two kinds of image suggested that the hydrogen permeation from the inside of grain and more permeation in austenite grains than martensite grains.

TF-WeP10 Hydrogen Diffusion in Stainless Steel and the Surface Stress Due to the Hydrogen, Akiko N. Itakura, Y. Murase, T. Yakabe, M. Tosa, National Institute for Materials Science, Japan; N. Miyauchi, S. Takagi, Toho University, Japan

Hydrogen in metals causes hydrogen embrittlement, which is the process of various metals to become brittle and fracture. Surface stress measurement is an effective technique for gas detection. In this study we focused on the stress of hydrogen contained in metals. We have detected a surface stress of stainless steel coated micro-cantilever under hydrogen

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irradiation. Meanwhile, it is not known the existing position of hydrogen in an alloy attributed to the complex structure of the alloy.

Metal thin films with the thickness of 20-50nm were deposited by RF magnetron sputtering on silicon micro cantilever (MCS: thickness of 1 micron and length of 500 micron). We found both of austenite and martensite structure in the film. The stress under hydrogen plasma irradiation was measured in-situ using a bending of cantilever beam [1]. MCS bended toward compressive surface stress and saturated under the irradiation. Probably it was a stress change due to hydrogen absorption although the bending might include temperature effect. After the irradiation stop, the bending curve decreased gradually. In 2nd experiment running, the saturated value of the bending was smaller than the 1st. After 10 times of the irradiation experiments (10 h irradiation) the bending did not decrease more.

In this study we tried to investigate hydrogen behavior in metal alloys from observation of two-dimensional hydrogen distribution on metal surface using a method of DIET (Desorption Induced by Electronic Transition). For the experiment we divided a vacuum container into two rooms. A part of a vacuum wall, between the two rooms, was a sample. Surface of the sample membrane thickness of 200 nm, had ring shape lines of 40micron pitch. The lines, which is a trace of the blade, were formed when the surface was flattened by a lathe. Cutting blade made un-uniformity of dislocations, too. H₂ gas was supplied from the backside room, H atom (ion) permeated through a sample and was desorbed into the vacuum room by electron irradiation. DIET mapping shows the hydrogen concentration reflects a density of dislocations, which has 40micron pitch [2]. It should be considered more by the comparing of DIET experiments with the structure measurement, grains, grain boundaries and dislocations [3]. Schematic diagram of the new experiment is shown in fig1. In the presentation we will show higher resolution images of DIET mapping, which can be compared with grains in SEM picture.

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TF-WeP11 Tight-Binding Quantum Chemical Molecular Dynamics Simulations on Tribochemical Reaction Dynamics of Carbon Nitride Thin Films, Miho Nakamura, Tohoku University, Japan; S. Sato, Tohoku University; Y. Ootani, Y. Higuchi, N. Ozawa, K. Adachi, M. Kubo, Tohoku University, Japan

Recently, carbon nitride (CN_x) has gained much attention as solid lubricant in automotive engines, aerospace instruments, etc. Experimentally, one of the authors, Adachi, discovered that CN_x gives super-low friction coefficient. However, the super-low friction mechanism of CN_x has not been clarified experimentally. It is experimentally pointed out that CN_x shows lower friction coefficient than diamond-like carbon under specific condition. It means that the role of nitrogen in CN_x is very important. However, the function of nitrogen in CN_x has not been elucidated. Therefore, the theoretical approach is desired to reveal the role of nitrogen in CN_x. In the present study, we employed our original tribochemical reaction simulator based on the tight-binding quantum chemical molecular dynamics method [1-3]. The friction dynamics of H-terminated CN_x is simulated under 1 GPa pressure. The simulation result shows low friction coefficient of 0.05. We found that hydrogen-hydrogen repulsion is source of the super-low friction. However, this reason is same with that of H-terminated diamond like carbon films. It means that the role of nitrogen has not been clarified. Then, we investigate the effect of water molecules on the friction dynamic of H-terminated CN_x. It is very interesting to see the generation and evaporation of NH₃ molecules by the tribochemical reaction of CN_x and water. The generation and evaporation of NH₃ molecules give low density diamond-like carbon thin films. Therefore, we propose that low density diamond-like carbon generated by the tribochemical reactions of CN_x and water gives super-low friction coefficient.

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TF-WeP12 Detection of 2-nitrotoluene – Comparison of Thin Film Sensors and Ion Mobility Spectrometry, Premysl Fitl, D. Tomecek, M. Pajkova, J. Vleck, L. Fiser, E. Maresova, S. Havlova, P. Hozak, M. Vrnata, University of Chemistry and Technology Prague, Czech Republic

Our work deals with comparison of detection abilities of thin film chemiresistors based on silver phthalocyanine with metallic nanoparticles and ion mobility spectrometry. In our study we focus to the detection of widely used taggant in explosives - 2-nitrotoluene.

The sensing layers of chemiresistors were deposited by vacuum evaporation (AgPc) and DC magnetron sputtering (metallic nanoparticles) on ceramic substrates with platinum interdigital electrodes. We varied type of metallic nanoparticles (Au, Pd and Ag), their amount (equivalent thickness in the range of 1 to 50 nm) and also the deposition sequence of used techniques (bottom AgPc + top nanoparticles vs top AgPc layer - bottom nanoparticles). For underlying metal nanoparticles the substrates were heated during deposition (temperature range of 20 - 600 °C) to investigate and tailor nanoparticle shape and conductivity properties.

Growth of metallic nanoparticles was continuously monitored by in-situ resistance measurements during sputtering and annealing operations. These measurements enable also detection of percolation threshold. The morphology of prepared layers was investigated by electron microscopy.

For as-deposited layers of metal on AgPc it was found that when the amount of metal is relatively low (i.e. less than layer with equivalent thickness of 4 nm for Pd, 5 nm for Au and 8 nm for Ag) metal clusters on organic surface are created. Continuous but incompact layers are formed for slightly greater amounts of sputtered metals. Finally, continuous and compact layers were observed when equivalent thicknesses achieved ≈ 15 nm. When the layers are annealed the percolation threshold is in general shifted to greater amounts of sputtered metal.

Optimized sub-threshold layers containing metallic cluster arrays on chemiresistor substrates were used for detection of 2-nitrotoluene. The taggant vapors were detected in two modes: without or with photoactivation (λ = 266 nm). While the dc-response of AgPc/Au(10 nm) sensor to 189 ppm of non-activated 2-nitrotoluene vapors was negligible, after photoactivation the dc-response rose to 373.

The sensing properties of thin films were compared with sensitivity of commercially available GC-IMS system. The detection limit of our thin film sensors is in range of hundreds of ppb. The ion mobility spectrometer is cable to reach lower detection limits (ppt). Although the ion mobility spectrometry shown the higher sensitivity, the possibility of overexposure and contamination of sampling valve and drift tube may cause fatal ion mobility spectrometer failure. Our phthalocyanine thin film sensors can be used as low cost alternative for detection of 2-nitrotoluene.

TF-WeP13 Development of SiC-DLC cOatings for Super Low Friction Mechanical Systems in Water, Takanori Takeno, T. Hayase, K. Adachi, Tohoku University, Japan

Water is environmentally friendly liquid. When we used it as a lubricant, it gives very low friction. Silicon carbide and silicon nitride can produce very low friction, < 0.01, under water lubricating condition. However, one of the drawback is low load carrying capacity due to low viscosity of water. With increase of load, direct contact between the materials occurs, which leads to seizure. Some researchers have shown increase of load-carrying capacity thanks to DLC that can reduce friction under direct contact condition. Although it works as protective coating to avoid seizure, friction under such severe condition is still high, ~0.1-0.2. So, the target of this study is to produce the coating to show low friction and high load carrying capacity under water lubricating condition. To achieve the goal, we are developing SiC-containing DLC coatings.

Investigated coatings are prepared by RF generated methane plasma and DC magnetron co-sputtering of silicon carbide target (Purity: 99.99%). Silicon carbide disk is used as a substrate material. Various coatings are deposited by changing methane and argon flow rate (CH₄/Ar). Composition, structural analyses and hardness are measured by Rutherford backscattering spectroscopy (RBS), X-ray photoelectron spectroscopy (XPS) and nanoindentation technique. Friction tests are performed using ball-on-disk tribometer. Silicon carbide ball is used as a counter material. Ball and disk are immersed in water and friction tests are carried out.

By changing flow rate of methane and argon, various coatings with different composition are achieved. Hardness of the coatings decreases with increase flow rate. Friction properties also vary with composition of the coatings. With CH₄/Ar = 0.1, coating is removed at very beginning of the friction test. When the coating is deposited with CH₄/Ar = 0.2, very low

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friction of 0.05 can be achieved. Almost similar friction curve is obtained with further increase of the ratio but fluctuation of the friction coefficient is also observed with $\text{CH}_4/\text{Ar} = 0.3$. It suggests that these is optimum structure or composition of the coating. We also changed speed and load for friction tests. Surprisingly, friction coefficient of the coating with $\text{CH}_4/\text{Ar} = 0.2$ decreases with increase of load and decrease of speed condition, meaning lower friction is obtained with more severe contact condition. According to the literature, lowering friction coefficient is due to the termination of OH bonds by reacting SiC surface with water. Thanks to the hydrogen bonding between O from OH and H from H_2O , water is captured on the frictional surface and friction force can be derived from shearing the low viscos water.

TF-WeP14 Atomic Layer Deposition of 2D MoS_2 on Si/ SiO_2 and Quartz Substrates, A. Valdivia, John Conley, Jr., Oregon State University, USA

The synthesis of high quality single layer 2D MoS_2 on large area substrates remains challenging. Mechanical exfoliation is capable of producing high quality material, but it is limited to small areas, requires transfer to the device substrate, is not scalable, and is impractical for manufacturing. Chemical vapor deposition (CVD) has been shown to yield MoS_2 on a variety of substrates, but is limited by non-uniform electrical properties, poor process stability, and high deposition temperature ($>600^\circ\text{C}$). A natural method for the synthesis of 2D materials is atomic layer deposition (ALD) in which alternating, purge separated, self-limiting surface reactions allow for precise thickness control, high conformality, and scalability to large surface areas. Although reports of ALD MoS_2 are beginning to emerge, ALD of single layer MoS_2 has typically required specialty sapphire substrates and high temperature (800°C) post deposition anneals and/or has been limited to small diameter wafers.

In this work, we demonstrate low temperature ALD of single to few monolayer MoS_2 uniformly across 150 mm diameter patterned SiO_2/Si and quartz substrates.¹ Purge separated cycles of MoCl_5 and H_2S precursors were used at ALD reactor temperatures ranging from 375°C to 475°C . Raman scattering measurements show clearly the in-plane (E_{12g}) and out-of-plane (A_{1g}) modes for ALD films deposited at 375°C or 475°C , indicating the presence of mono- to few layer MoS_2 . The separation of the E_{12g} and A_{1g} peaks is a function of the number of ALD cycles, shifting closer together with fewer layers. Films deposited at 475°C are of much higher quality than films deposited at 375°C . While the E_{12g} - A_{1g} peak separation of the 375°C film corresponds to bulk-like MoS_2 , the smaller peak separation of the 475°C film suggests a thickness of approximately two monolayers. Raman polarization tests confirm the MoS_2 crystals have the desired orientation parallel to the surface. High temperature H_2S and sulfur annealed films produce a sharpening of the E_{12g} and A_{1g} peaks as well as the appearance of the band edge PL and spin orbit splitting peaks, a further indication of the presence of monolayer MoS_2 . High resolution transmission electron microscopy images confirms the presence of monolayer to bilayer MoS_2 films. X-ray photoelectron spectroscopy indicates that a sub-stoichiometric sulfur ratio in the as-deposited films is increased to the stoichiometric S/Mo ratio after annealing in H_2S at 600°C and above. Results suggest that ALD may be a promising method for production of 2D MoS_2 .

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TF-WeP16 Optical and Structural Study of GaN Films Grown by Pulsed Laser Deposition in Nitrogen Atmosphere, Luis Arturo Martínez-Ara, P. Maldonado-Altamirano, R.Y. Ponce-Cano, J.R. Aguilar-Hernandez, ESFM-IPN, Mexico; M.A. Hernandez-Perez, ESQIE-IPN, Mexico; G.S. Contreras-Puente, ESFM-IPN, Mexico

Results about processing and characterization of gallium nitride (GaN) films grown by pulsed laser deposition technique are presented. The films were grown on sapphire (0001) and silicon (111) substrates, under the following conditions: substrate temperature of 850°C , time deposition of 60 minutes and pressure of 4.2×10^{-6} torr. A Nd: YAG laser was used with wavelength of 1064 nm, repetition frequency of 50 Hz and power of 2.8 W. To study the GaN films structural properties, X-ray diffraction was used obtaining peaks around 34.5° corresponding to GaN. To study the optical properties, UV-Vis absorption spectroscopy, photoluminescence (PL) and Raman spectroscopy were used. From the UV-Vis spectroscopy a band-gap value of 3.2 eV was obtained. Photoluminescence at room temperature was observed, the PL spectra shows two bands, one of these associated to yellow band of GaN at 2.2 eV, and the other one around 3.0 eV associated with recombination

centers, such as interstitials Gallium or Nitrogen atoms. Raman shift in 722 cm^{-1} was obtained corresponding to GaN in its wurtzite structure.

TF-WeP17 Electrical and Chemical Properties of $\text{HfO}_2/\text{Al}_2\text{O}_3$ on $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ Annealed at Different H_2 Pressures, Sungho Choi, Y. An, C. Lee, J. Song, Sungkyunkwan University, Republic of Korea; M.-C. Nguyen, Inha University, Republic of Korea; Y.-C. Byun, J. Kim, University of Texas at Dallas, USA; R.C. Choi, Inha University, Republic of Korea; H.S. Kim, Sungkyunkwan University, Republic of Korea

Passivation of a high- k /III-V interface is the most important process to be developed for future III-V-based transistors. Recently, there have been several attempts to passivate the interface defects by performing H_2 annealing after the high- k deposition on $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$: forming gas annealing [1] or H_2 high pressure annealing [2].

In this study, we deposited a $\text{HfO}_2/\text{Al}_2\text{O}_3$ gate stack using atomic layer deposition on the $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ substrates with different doping types and carried out H_2 high pressure annealing (400°C) at different pressures (10 bar and 30 bar). According to the time-of-flight secondary ion mass spectrometry and X-ray photoelectron spectroscopy measurements, out-diffusion of In and Ga atoms toward the high- k film was escalated as the H_2 pressure increased. The relationship between the observed H_2 -induced out-diffusion and the resulting electrical properties will be discussed.

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TF-WeP18 In-situ Visualization of Graphene Grain Boundary by Optical Microscopy, K.-P. Hong, S.-H. Park, D.-J. Oh, Yekyung Kim, Korea Electronics Technology Institute (KETI), Republic of Korea; K.-S. Kim, Sejong University, Republic of Korea; K.-H. Kim, J.-B. Choi, Sungkyunkwan University, Republic of Korea; H. Kim, Korea Electronics Technology Institute (KETI), Republic of Korea

The graphene consisting with carbon atoms to honeycomb structure has the excellent properties about mechanical, chemical and thermal and it has been research in a variety of fields. In order to commercialize graphene with these outstanding properties, as well as a large area, mass production is essential for precise and fast measurement. In this study, for measuring the shape and size of the oxidation graphene fast and precisely, we configured the measuring system with an optical microscope, a heating stage and 3 W level laser. Through the configured system we can keep the graphene in a vacuum and wet condition to 200°C and measure the grain boundary of the oxidation graphene by irradiating the laser to locally. Later by proceeding the additional experiment and analyzing the correlation between the degree of graphene oxide and the laser irradiation time, through this process we develop the optimized visualization method of the graphene defects.

TF-WeP21 Thin Films of Sustainable Materials by UV Photo-transformation, Lopamudra Das, M.J. Kelley, College of William & Mary, USA

The expanding availability of narrow-wavelength deep UV lamps and powerful tools for numerical modelling afford growing opportunity to create precisely tailored organic thin films by transformation of an original substrate. Having begun with synthetic polymers (polyesters), application to natural materials (cellulose) is now proving successful, as revealed by surface spectroscopies (XPS and ToF-SIMS), AFM and wetting.

TF-WeP22 Nanostructured Oxide Thin Films as an Active Layers of Chemiresistive Sensors for Detection of Chemical Warfare Agents and Their Simulants, Jan Vlcek, P. Hozak, D. Tomecek, E. Maresova, P. Fítl, L. Fiser, University of Chemistry and Technology Prague, Czech Republic; T. Rozsypal, M. Haskovcova, Z. Koblíha, Z. Skalican, University of Defense, Czech Republic; J. Lancok, L. Fekete, Institute of Physics, Academy of Science of the Czech Republic; M. Vrnata, University of Chemistry and Technology Prague, Czech Republic

The aim of this work is to investigate the response of nanostructured chemiresistive gas sensors with an active layers based on nanostructured metal oxides (nanoporous structures) to a significant group of chemical warfare agents (CWAs) - (G-series: sarin, cyclosarin, soman, choking agent: diposgene) and their simulants - (diethyl-malonate, ethyl-chloroacetate and difenyl-phosphochloridate). The active layers of sensors (based on SnO_2 , ZnO , PdO) with thicknesses of 100 - 400 nm were prepared on sensor substrates by (i) sputtering of source metal and subsequent anodic oxidation, (ii) sputtering of source metal and subsequent thermal oxidation

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in oxygen atmosphere, (iii) thermal evaporation of source material metal and subsequent oxidation in oxygen atmosphere. Sensor platform consists of a planar sensor aluminium oxide substrate with interdigital platinum electrodes.

We present results of morphology investigation of nanostructured oxide films by AFM and SEM microscopy and structural analysis by XRD. Conventional dc-sensitivity of sensors S_{dc} was evaluated as a ratio of their resistance in air containing given concentration of analyte and that in "pure" air respectively. Detection was tested for concentrations of CWAs up to 50 ppm in air. SnO₂-based sensors feature a high sensitivity towards CWAs, where S_{dc} reaches a values of 3 - 10. On the other hand ZnO-based sensors exhibit significant selectivity to CWAs, whereas the responses to a common interferents are negligible. We show the connection between material morphology properties (porosity, crystalline size) and detection ability of CWAs.

TF-WeP23 Effect of Substrate Temperature on the Mechanical Properties and Structure of Sputtered Titanium Nitride Film by a Pure Nitrogen Plasma, C.-H. Park, B.-S. Jang, Chang-Hyun Lee, Catholic University of Daegu, Republic of Korea; S.-Y. Son, Pohang University of Science and Technology, Republic of Korea; B. Kim, H.-M. Kim, Catholic University of Daegu, Republic of Korea

Titanium nitride (TiN) films were prepared by rf magnetron sputtering technique. The depositions were carried out by a pure N₂ plasma sputtering. Their mechanical properties, such as nano-indentation hardness, friction coefficient, and water contact angle have been investigated. XRD studies revealed that TiNx films grown at temperatures more than 360°C exhibits an intense compressive stress as compared with the films grown at temperatures less than 360°C. The orientation of TiNx films changes toward (111) orientation at 360°C due to increased ion bombardment which favors low surface energy at (111) orientation. The increase of Ts, which means the distance between target and substrate, increases the mobility of adatoms promoting closed packing structures in near thermodynamic equilibrium conditions. Thus, for high adatom mobility the TiNx films expected to grow along the (111) orientation corresponding to that with the lowest surface free energy. On the other hand, for low adatom mobility the preferred orientation is the (002) in which the highest number of atoms per unit area can be incorporated at low energy sites. The mechanical properties of TiNx films grown at a pure N₂ atmosphere strongly dependent on the Ts. The highest hardness and the smallest friction coefficient of 26 GPa and $\mu=0.13$, respectively were in a TiNx film deposited at 400°C. This film was found to be accompanied by a water repellent surface.

This research was financially supported by the Ministry of Education (MOE) and National Research Foundation of Korea(NRF) through the Human Resource Training Project for Regional Innovation (No. 2015H1C1A1035619). This work (Grants No. C0350891) was supported by Business for Cooperative R&D between Industry, Academy, and Research Institute funded Korea Small and Medium Business Administration in 2015.

TF-WeP24 Study of a Temperature and Humidity Sensor Constructed by the Multi-layer Film including a TiO₂ Layer, Takahisa Kawaguchi, I. Takano, Kogakuin University, Japan

Currently, commercial temperature and humidity sensors have a low sensitivity for both sides of a high humidity and a low humidity. Moreover, because the main parts of these sensors are manufactured using polymer materials, there is the problem of a short life in environments such as high temperature or high humidity. Therefore, the next-type sensors are required in a longer life and a higher sensitivity.

As a material satisfying some of the above-mentioned function, TiO₂ was adopted. TiO₂ is an n-type oxide semiconductor, and has the stable photo-excited state and causes no autolysis. In addition, TiO₂ shows the hydrophilicity under ultraviolet irradiation. The next-type sensor with a high sensitivity is promised by the suitable junction of between a TiO₂ layer and a Cu₂O (p-type oxide semiconductor) layer.

In this study, the investigation of TiO₂ thin films on sensor characteristics and the improvement of the sensor sensitivity by using the TiO₂/NiO/Cu₂O multi-layer thin film were carried out. The rutile-type TiO₂ thin film (TiO₂-R) with a thickness of 200 nm, the anatase-type TiO₂ thin film (TiO₂-A) with a thickness of 200 nm and the TiO₂/NiO/Cu₂O (200/10/200 nm) thin film

were prepared by reactive magnetron sputtering. The electric resistance of each sample was measured by changing a voltage from 0 V to 10 V. The resistance changes for a temperature and a humidity were measured by changing a temperature from 25 °C to 60 °C by changing a humidity from 30 % to 60 %. As the characteristic relating to the humidity, a water contact angle was measured by the $\theta/2$ method. In these experiments, the photoreaction on a semiconductor characteristic was examined by irradiating the white-LED light or UV-LED light to the sample surface.

The resistances of the TiO₂-R thin film and the TiO₂-A thin film decreased by the photo-excitation under the UV-LED light irradiation. The resistance of the TiO₂/NiO/Cu₂O thin film under the white-LED light irradiation showed the value as well as that of UV-LED. The temperature coefficient of resistance of the TiO₂-R thin film, the TiO₂-A thin film and the TiO₂/NiO/Cu₂O thin film were -8354×10^{-6} [1/K], -5264×10^{-6} [1/K] and -16390×10^{-6} [1/K], respectively. In the case of a humidity characteristic, there was no large change in the electric resistance against the humidity change for any sample. In addition, the improvement of hydrophilicity by light irradiation was not confirmed from the water contact angle examination.

The TiO₂/NiO/Cu₂O thin film showed a superior temperature characteristic in comparison with the TiO₂-R thin film or the TiO₂-A thin film.

TF-WeP25 Improvement of the Photocatalyst Effect by the Intermediate Layer Insertion to TiO₂/Cu₂O Films, Toshiya Souma, I. Takano, Kogakuin University, Japan

In recent years, various characteristics of TiO₂ have attracted considerable attention. One of their characteristics is a photocatalytic effect. The photocatalytic effect of TiO₂ shows antifouling or antimicrobial activity, and has the ability to decompose environmental pollutants. The most important characteristic as a photocatalysis of TiO₂ is well known that the photo-excited state is very stable and does not cause self-decomposition. Therefore, TiO₂ can perform the electrolysis of water by light, however a light reaction of TiO₂ is limited at ultraviolet region corresponded with only about 3 % of sunlight.

In this study, to improve the photocatalytic property of TiO₂ the double layer films were fabricated by the constitution of the TiO₂ layer with 3.0 - 3.2 eV and the Cu₂O layer with 2.2 eV in a band gap energy. In order to prevent the diffusion of Cu, an NiO or TaON layer was inserted between the TiO₂ layer and the Cu₂O layer. NiO has a high melting point, high hardness and has been used as a barrier layer in various fields. TaON is used as electrode materials for a semiconductor and light electronics devices. Each oxide layer was prepared by reactive magnetron sputtering.

The film composition and microstructure were investigated by the X-ray photoelectron spectroscopy and X-ray diffraction, respectively. The chromatic change of a methylene blue solution was applied to the measurement of the photocatalytic property. The light irradiation to the TiO₂/NiO/Cu₂O film or the TiO₂/TaON/Cu₂O film in a methylene blue solution was carried out using a commercial sterilizing lamp as ultraviolet light and an artificial sun lamp as visible light. The transmittance of a methylene blue solution was measured by a spectrophotometer.

It was confirmed that Cu on the TiO₂ surface was not observed by inserting the NiO or the TaON layer. The crystal structure of the TiO₂/Cu₂O film showed a rutile type while the ratio of rutile (110) / anatase (101) changed with the NiO film thickness. In the case of the intermediate layer of NiO, the suitable photocatalytic effect was obtained by the film with the NiO layer of 20 nm thickness, when the transmittance showed about 57 % under an artificial sun lamp and about 77 % under a sterilization lamp. In the case of the TaON layer, the suitable photocatalytic effect was obtained by the TaON layer of 10 nm thickness, when the transmittance showed about 75 % under an artificial sun lamp and about 82 % under a sterilization lamp.

It is considered that the role of the intermediate layer is not only the protection of Cu diffusion from the Cu₂O layer but also the activation of the mobility of carriers.

TF-WeP26 Combining XPS and UPS Measurements in Thin Film Depth Profiles, Joseph Robinson, Thermo Scientific, USA; T. Nunney, Thermo Scientific, USA, UK; A. Bushell, J. Treacy, Thermo Scientific; C. Deeks, P. Mack, Thermo Scientific, USA

X-ray Photoelectron Spectroscopy (XPS) depth profiling has been widely utilized to provide detailed elemental and chemical state information of thin films, such as those used in microelectronic devices and protective coatings. These measurements have often been combined with valence electronic information obtained using the related technique of Ultraviolet Photoelectron Spectroscopy (UPS). Such detailed and complementary

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information is essential when attempting to fine-tune specific thin film parameters for best device or coating performance.

While useful information is acquired from XPS and UPS in isolation, a more powerful insight into the structure of a material comes from using these two techniques in conjunction, allowing a more complete material characterisation to be performed. Previously, switching between techniques throughout the course of an experiment has been an involved and often laborious process, discouraging more widespread use. Recently the automation of UPS has allowed acquisition of XPS and UPS data at each level of a depth profile, providing a much sought after insight into the correlation between chemical and electronic structure at within a substrate at various depths.

Of particular interest is the ability to access the valence electronic structure at mixed oxide interfaces using small argon ion gas clusters, which was not previously possible due to the loss of electronic structure in semiconductors or organic materials on exposure to monatomic argon ion beams. This presentation demonstrates the wealth of information that can be acquired by performing combined XPS-UPS depth profiles and the ease with which this information can be acquired and processed, due to recent instrumentation and software developments.

TF-WeP27 Enhancement of Oxidation Resistance of Metal Surface by a Polymer Monomolecular Layer and Its Influence on Electrical Conductivity, T. Ikeda, H.T. Nguyen, J. Takagi, K. Yamakawa, K. Adachi, Yasuhisa Tsukahara, Kyoto Institute of Technology, Japan

Metals like copper are easily oxidized in air to form metal oxides on the surface. This is often problematic for their applications because of reduction of the electrical conductivity. We studied influence of molecular coating of organic thiol compounds on copper plates and fine particles on their oxidation resistance and electrical conductivity.

Copper and nickel surfaces were coated with thiol terminated polystyrenes (PSt-SH)s and alkanethiols by dipping copper into solutions of the thiol compounds. XPS measurements revealed that the molecular thin layer of PSt-SH lead to a resistance against oxidation. Coating with alkanethiols like 1-dodecanethiol (Dod-SH) was also effective. However, only the PSt-SH layer kept the oxidation resistance against heating at high temperature, which was also confirmed by the color change of the metal surface by heating.

We also used cyclic voltammetry (CV) to evaluate the oxidation resistance. The oxidation resistance was determined from the integral value at the anode and cathode regions in the oxidation-reduction curve. We found that the oxidation resistance of copper covered with PSt-SH was excellent at high temperatures under atmosphere and was not decreased even by heating up to 150°C.

The temperature dependences of the contact electrical resistance measured by the four-point method indicated that the electrical conductivity was not reduced for the metal surface coated with PSt-SH even at high temperatures which is consistent with the results of XPS and CV measurements.

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TF-WeP28 Study of the Influence of the Abrasive Slurry Concentration on the Coefficient of Friction of Thin Films submitted to Micro-abrasive Wear, Ronaldo Cozza, G.H.B. Donato, University Center of FEI, Brazil

The purpose of this work is to study the influence of the abrasive slurry concentration on the coefficient of friction of thin films submitted to micro-abrasive wear. Initially, a micro-abrasive wear testing by free rotative ball equipment was designed and constructed, able to measure the coefficient of friction on the tribo-system "thin-film - abrasive slurry - ball". After, experiments were conducted with thin films of TiN, TiC, CrN, TiAlN, HfN, ZrN, TiZrN, TiN/TiAl (multi-layer), TiHfC and TiHfCN, balls of AISI 52100 steel and abrasive slurries prepared with black silicon carbide (SiC) particles + glycerin. All tests were conducted without interruption, and the abrasive slurry was continuously agitated and fed between the ball and specimen. The tangential (T) and normal (N) forces were monitored throughout the tests and the coefficient of friction (μ) was calculated by the equation $\mu = T/N$. The results obtained have shown that the concentration of abrasive

slurry affected the actions of the abrasive wear modes (grooving abrasion or rolling abrasion) and, consequently, the magnitude of the coefficient of friction: high abrasive slurry concentration was related with low coefficient of friction.

TF-WeP29 A Study on Coefficient of Wear and Coefficient of Friction of Thin Films submitted to Micro-scale Abrasion, Fabiano Silva, S.O. Santos, CEETEPS - Centro Estadual de Educação Tecnológica "Paula Souza" - FATEC-Mauá; R.C. Cozza, CEETEPS - Centro Estadual de Educação Tecnológica "Paula Souza" - FATEC-Mauá, Brazil; C.F. Silva, M.O. Gentil, A.J. Moura Jr., CEETEPS - Centro Estadual de Educação Tecnológica "Paula Souza" - FATEC-Mauá

Several works on the coefficient of friction during abrasive wear tests are available in the literature, but only a few were dedicated to the coefficient of friction in micro-abrasive wear tests conducted with rotating ball. This work aims to study the influence of titanium nitride (TiN) and titanium carbide (TiC) coatings hardness on the coefficient of friction and coefficient of wear in ball-cratering micro-abrasive wear tests. A ball of AISI 52100 steel and two specimens of AISI D2 tool steel, one coated with TiN and another coated with TiC, were used in the experiments. The abrasive slurry was prepared with black silicon carbide (SiC) particles and distilled water. Two normal forces and six sliding distances were defined, and both normal and tangential forces were monitored constantly during all tests. The movement of the specimen in the direction parallel to the applied force was also constantly monitored with the help of an electronic linear ruler. This procedure allowed the calculation of crater geometry, and thus the coefficient of wear for the different sliding distances without the need to stop the test. The coefficient of friction was determined by the ratio between the tangential and the normal forces, and for both TiN and TiC coatings, the values remained, approximately, in the same range (from $\mu = 0.4$ to $\mu = 0.9$). On the other hand, the coefficient of wear decreased with the increase in coating hardness.

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