Forum M Advanced Preparation and Characterization Technology of Materials

M01 (Invited)

Manipulating Electronic Phase Separation in Complex Oxides

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For strongly correlated systems, it has been known that co-existence of electronic phases is often energetically favored. Investigation of the so-called electronic phase separation (EPS) phenomena is not only important for understanding the strong electronic correlations in these materials, but also very useful for tuning their physical properties. In this work, we push this trend to its limit by developing the capability of manipulating EPS in colossal magnetoresistance (CMR) manganites systems. We demonstrate that it is possible to pattern the EPS in manganites and thus design the physical properties of the systems. By doing so, we are able to gain a much deeper insight of the physical origin of electronic phase separation in these materials.

M02 (Invited)

XAS Study of Low Dimensional Dilute Magnetic Semiconductors

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Low Diluted magnetic semiconductors (DMSs), which possess the magnetic and semiconducting characteristics of materials, are promising candidates for developing the next-generation spintronics devices to use simultaneously the spin and charge characters of electrons. Electronically understanding the microscopic mechanism of mediating the magnetism of the DMS materials is of crucial importance to fabricate spintronics devices controllably through external fields like light and electric fields. The lecturer used X-ay absorption spectroscopy combining first-principles calculations explored the local atomic and electronic structures of numerous low-dimensional semiconductors, revealed the interplay of the dimension, structure and magnetism of magnetic semiconductors, illustrated the influence of the surface structure distortion on the bandgap structure of the semiconductors, and proposed a series of novel methods for tuning the magnetism of low-dimensional magnetic semiconductors via shell-core structure, phase doping, surface structure relaxation, valence alteration and so forth. All of these researches present themselves as a series of significant breakthrough in understanding the magnetic origin of magnetic semiconductors.

M03

Investigation of Two-dimensional Organic/Inorganic Materials Via Synchrotron-based Approaches

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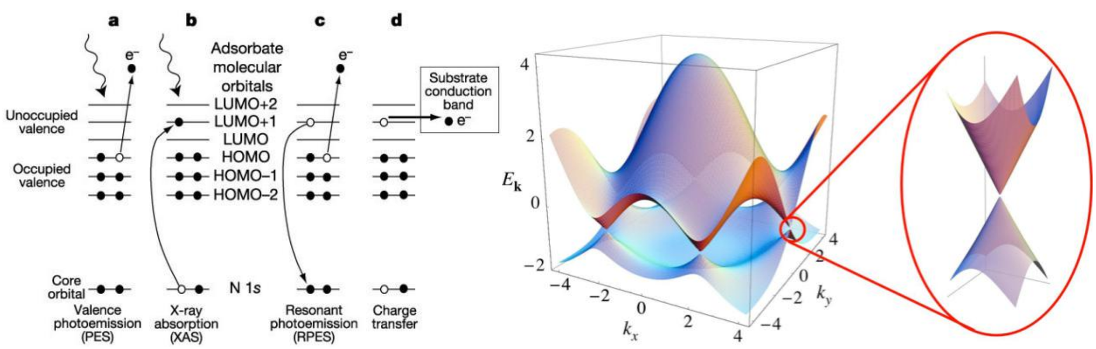
Owing to its unique high surface area, artificially controllable structures and facile tuning of electric/chemical properties, two-dimensional materials have attracted numerous interests in the past decades. As the typical examples, graphene and transitional metal dichalcogenide have already found their promising applications in the field of solar cells, super capacitors, filed effect transistor, nanosensors and catalysis. Based on such promotion, our study herein mostly focuses on: 1) controllable fabrication of functional organic thin films via self-assembly based on diverse organic precursors, as well as the characterization. 2) Investigation of functionalized graphene, metal selenium compound and the tuning of their electronic structures. With a combination of synchrotron-based measures, including X-ray photoemission spectroscopy (XPS), X-ray absorption spectroscopy (XAS) and scanting tuning microscopy (STM), we have been able to visualize uniquely the nanostructures of metal thin films (Bi, Fe, $LaSrMnO_3$) and decorated organic network on various substrates, but also characterize the physical, chemical and electronic properties of such surfaces and interfaces. In the end, one kind of structure-activity relationship was established for certain low dimensional nanostructures.



M04

Effects of PEM Controlled Reaction Atmosphere on Composition and Structure of Carbon Doped Ti-O Films

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Titanium dioxide (TiO_2) has good chemical stability, photocatalytic activity and relatively low prices and without toxic effects on the human body, etc, so it can be used in solar cells, self-cleaning and anti-fog glass, UV screening agents, degradation of contaminant and antibacterial materials. It is considered to be a kind of materials applied widely in industry, and become a hot topic. Although TiO_2 has photocatalytic properties, but currently TiO_2-based photocatalytic technology still has low quantum efficiency and narrow absorption range of light (only absorbed ultraviolet light), and most TiO_2 photocatalysis with powder status is difficult to recycle, which makes it greatly restricted in industrial applications.

In order to improve the light absorption range of TiO_2 and reduce the recombination between $e^-$and h^+, doping can be one of the effective ways. The doped elements such as C, N, Au, Ag, etc. can effectively reduce the band gap of TiO_2 and make the absorption edge move to visible light. Simultaneously, many researchers immobilize TiO_2 on different substrates with coating prepared by magnetron sputtering and sol-gel to solve the problem of recycle and reuse of TiO_2 powder. The films with complex composition can be prepared by magnetron sputtering. However, IF (Intermediate Frequency) and DC Reactive Magnetron Sputtering with higher deposition speed persists response lag effect and problems of instability. Plasma Emission Monitoring(PEM) System have high-speed response, and it can control reactive magnetron sputtering working in the transition zone of the hysteresis curve stability by precise control of the reaction gas flow rate. Thus, different stoichiometry films can be prepared with the help of PEM.

In this paper, carbon-doped Ti-O films were prepared on steel, glasses and silicon by reactive magnetron sputtering using high purity titanium as titanium plasma source, CO_2 and O_2 as the C and O source. PEM was used to monitor glow intensity of target during deposition. A series of Ti-O films with different components and structures were prepared by depending the Set Point (SP) of PEM. Atomic Force Microscope (AFM), X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) were used to analyze surface morphology, structure and composition of as-prepared Ti-O films. The mechanical properties of Ti-O films such as hardness, adhesion strength and friction coefficient and hardwearing were detected via Multi-function Micromechanics Test System. The optical absorption curve was measured by ultraviolet–visible (UV–Vis) spectrophotometer and band gap of the film can be calculated. The results showed that carbon-doped Ti-O films were synthesized using CO_2 as C source and the absorption bands of the films were extend successfully. Mechanical properties of the films increased with the increases of oxygen content in reaction atmosphere, but with oxygen increasing continuously, wear and friction properties of the films and adhesion strength between substrate and films reduced.

Keywords: Ti-O films; Reactive Magnetron Sputtering; PEM; Photocatalysis; Mechanical properties

M05

Current-induced Magnetization Switching in Heavy Metal/Ferromagnet Multilayers Without Magnetic Field

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Spin-orbit torque (SOT) usually originates from a heterostructure with strong spin-orbit coupling (SOC) and structural inversion symmetry under an in-plane electric current, which in turn induces magnetization switching [1-5]. However, the SOT induced magnetization switching usually needs an external magnetic field to act as symmetry-breaking bias. Therefore, it is inconvenience for SOT based spintronic devices in real applications if we always need a field.

In this letter, we report the current-induced switching with the absence of external field in heavy metal (HM)/ferromagnet (FM) based films. Multilayers were deposited on SiO2 substrate using magnetron sputtering system with a stacking structure of Ta (8)/Pt (3)/Co (1)/AlOx (3)/Ta (1) (thickness in nanometer and from bottom to top). Then the films were fabricated into Hall bars as shown in Figure. 1(a). The Hall resistance (RH) as a function of the out-of-plane magnetic field is presented in Fig. 1(b), which is measured at various DC current of 0.01, 0.5 and 1 mA. Fig. 2 shows RH-I curves with different field applied along x-axis. According to Fig. 2(b), SOT can be observed without external magnetic fields. In the multilayer structure, we employed Ta and Pt layers with different spin Hall angle. Both layers have contributions to the spin accumulation at the interface of HM/FM heterostructures and can strengthen the SOC, which is important for getting large spin Hall and Rashba field. It is interesting behavior we observed here at some critical point the switching sequence is not reverse while changing of the magnetic field direction from negative to positive fields, as shown in Fig. 2(c). This character can keep until a critical field value, after that value we observed absence of switching as shown in Fig. 2(d).

In summary, current-induced switching with absence of external field is observed in Ta/Pt/Co/AlOx/Ta multilayer. Both Ta and Pt can enhance either the interfacial SOC strength or the efficiency of generating the damping-like torque for a given interfacial SOC strength.

M06 (Invited)

Synchrotron based High-throughput Characterization

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High-throughput characterization is crucial for materials genomic research. On one hand, physics properties of dense arrayed micro-materials on a materials chip need be quantitatively characterized. On the other hand, characterization speed must quick enough to meet the practical requirement. Among various high-throughput characterization techniques, interacting electromagnetic waves (generated light) of different spectral bands with materials, detecting the secondary effects, and imaging the spatial distribution of physics properties on a materials chip is one of the best choices. Compared with laboratory light sources, synchrotron radiation shows wide spectrum range, high brightness and strong flux. So, developing synchrotron based high-throughput characterization methods is a natural consequence. In this talk, we will discuss, based on the general requirements of high-throughput characterization, the advantages and necessity of synchrotron based high-throughput characterization. We will also give a few case studies of our group.

M07 (Invited)

Establishment and Application of Low-temperature TEM Platform Composed of Electromagnetic Multi-field Coupling

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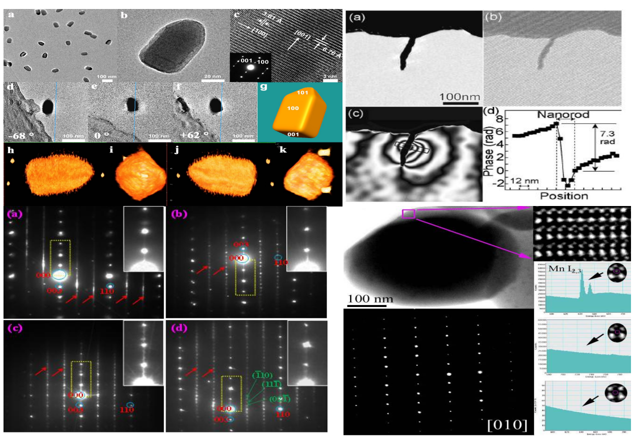
Magnetic nanoparticle is important both scientifically and technologically. However, to deeply understand the physical association between microstructure and magnetic property is still in lack. Herein, via a series of advanced transmission electron microscopy (TEM), such as 3-D electron tomography, off-axis electron holography, and electron energy loss spectroscopy (EELS), the microstructure and magnetic property of manganese selenide (MnSe), L_{10}-FePt, α-Fe/CNTs are comprehensively investigated. The 3-D morphology of the bullet-shaped MnSe nanorods has been demonstrated by the advanced TEM 3D-tomography technology. Examinations with HRTEM and EELS show that planar-defect structures such as stacking fault and twin along [001] direction are formed during the bullet-shapes growth. The differences of structure and composition between the Fe/CNxnanotubes and Fe/C nanotubes were investigated by EELS. At the boundary between Fe and the wall of a CNx nanotube, the additional electrons contributed from the doped 'pyridinic-like' nitrogen might transfer to the empty 3d orbital of the encapsulated iron, therefore leading to an intensity suppression of the iron $L_{2,3}$ edge and an intensity enhancement of the carbon Kedge. The influences of the Li^+/Ni^{2+} replacement modulated by minor Co dopant on cyclic capacity and rate performance of lithium-rich cathode material $Li_{1.2}Ni_{0.2-z/2}Mn_{0.6-z/2}Co_zO_2$ (z=0, 0.02, 0.04, 0.10) were investigated. Co played a vital role in decreasing the Li^+/Ni^{2+} replacement ratio in the hexagonal layered $Li_{1.2}Ni_{0.2-z/2}Mn_{0.6-z/2}Co_zO_2$ ($R\bar{3}m$). An evident cationic ordering in the transition metal layers and stacking sequence vertical to the Li^+ diffusion orientation were observed from $Li_{1.2}Ni_{0.2-z/2}Mn_{0.6-z/2}Co_zO_2$ (z>0) system rather than $Li_{1.2}Ni_{0.2}Mn_{0.6}O_2$ system. Three superstructure vectors modulated by $1/4\vec{\mathbf{q}}$, $2/4\vec{\mathbf{q}}$, $3/4\vec{\mathbf{q}}$ ($\vec{\mathbf{q}}$ =[$0\bar{1}1$]) were simultaneously observed from $Li_{1.2}Ni_{0.18}Mn_{0.58}Co_{0.04}O_2$, indicating a high-degree ordering.



M08

Observation of Dual Relaxation Processes in Barium Titanate During Paraelectric-Ferroelectric Phase Transition

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Measurement of polarization correlation for polarization clusters during paraelectric-ferroelectric phase transition in barium titanate. Recently, polarization clusters were investigated as a new approach to research the mechanism of this phase transition. The coexistence of displacive and order-disorder processes was considered closely related to the formation of polarization clusters. However, the roles played by these two mechanisms during this phase transition is still unclear and dual relaxation processes for polarization clusters have never been directly observed. Base on the improved He-Ne laser photon correlation spectroscopy (PCS) technique, we actually observe this dual relaxation processes, clarifying the role of the dynamics process of polarization clusters during this phase transition and revealing the origin of the large dielectric response of this prototype ferroelectric.

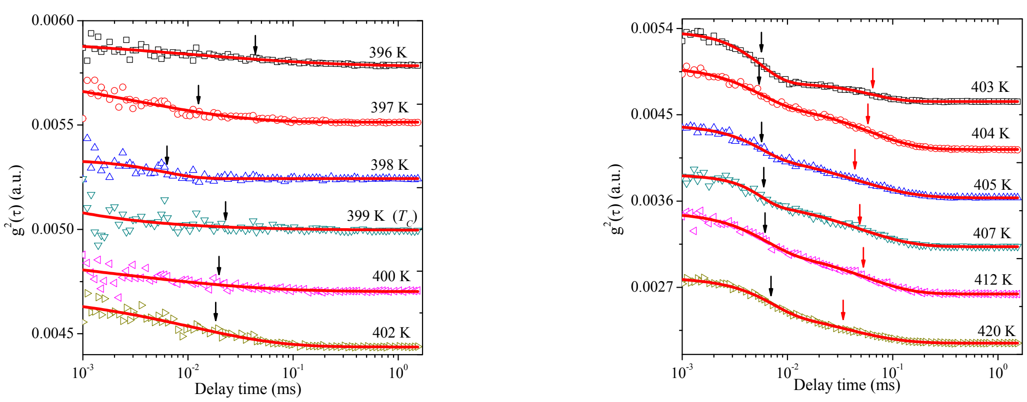

FIG.1. Intensity correlation $g^2(\tau)$ of the polarization clusters in $BaTiO_3$ near T_C from 396 K to 420 K.

M09

Surface Analysis from ThermoFisher Scientific：XPS Technology and Applications in Modern Functional Materials

Albert.Ge

Thermo Fisher Scientific (China)Co.,Ltd,USA

X-ray photoelectron spectroscopy (XPS) is a technique for analyzing the surface chemistry of a material. XPS can measure the elemental composition, empirical formula, chemical state and electronic state of the elements within a material.

As the demand for high performance materials increases, so does the importance of surface engineering. Many of the problems associated with modern materials can be solved only by understanding the physical and chemical interactions that occur at the surface or at the interfaces of a material's layers. The surface chemistry will influence such factors as corrosion rates, catalytic activity, adhesive properties, wettability, contact potential and failure mechanisms.

The material's surface is the point of interaction with the external environment and other materials. Therefore, surface modification can be used in a wide variety of applications to alter or improve the performance and behavior of a material. XPS can be used to analyze the surface chemistry of a material after an applied treatment such as fracturing, cutting or scraping. From non-stick cookware coatings to thin-film electronics and bio-active surfaces, XPS is the standard tool for surface material characterization.

M10

A Novel Benzene Sensor Based on Ag-$LaFeO_3$ Using Molecular Imprinting Technique

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A novel gas sensor for the determination of benzene was developed based on molecular imprinting technique (MIT). MIT was for the first time used to recognize small organic molecule by our group. The molecular imprinting nanoparticles (MINs) with a small dimension which possess extremely high surface-to-volume ratio were synthesized using imprinting polymerization with benzene as template and Ag-$LaFeO_3$ as substrate material. The structure of the MINs is orthogonal perovskite. And then the MINs were printed onto an alumina tube. Subsequently, a high selectivity molecular imprinting gas sensor for detection of benzene was achieved. At 125℃, the response to 1 ppm benzene based on the sensor is 20.5, and the response is lower than 5 for the other test gases. The response time and recovery time are 80 s and 120 s, respectively.

M11 (Invited)

Design of Environmental Microchips for *In-situ* Microscopic and Spectroscopic Characterizations

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Comparing with traditional microscopic or spectroscopic characterization methods, the *in-situ* microscopy or spectroscopy is generally defined as a method to apply some form of stimuli to a sample while it is observed in a microscope or spectroscope. The stimuli include heating, electric current, mechanical force, magnetic field, and non-vacuum environmental molecular interactions (such as those in gas and liquid). Based on well-developed MEMS fabrication technology, we designed and fabricated environmental microchips, especially electrochemical (EC) chips and liquid cells, for *in-situ* microscopic and spectroscopic characterizations of various chemical and biological processes. EC-TEM or EC-SEM microchips are used to study electrochemical reactions with ionic-liquid or organic electrolyte in a standard TEM or SEM. Liquid cells with electron-transparent thin silicon nitride windows are used to observe chemical reactions in liquid or living bacteria/cells in liquid medium with a standard TEM, SEM or an optical spectroscope. These in-situ characterization tools are expected to play an increasingly important role in understanding the true mechanisms behind various physical, chemical, and biological phenomena.

M12

In situ Transmission Electron Microscopy Observation of the

Electrochemical Behavior of Anodes in Li-ion battery

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Transition metal sulfides (oxides) are regarded as a type of high-performance anode materials for lithium ion batteries (LIBs). However, their electrochemical process and lithium-storage mechanism are complicated and remain controversial. The comprehension of fundamental electrochemical behavior and mechanism is urgent for the design of advanced electrode materials for Li-ion batteries. Using advanced in situ transmission electron microscopy, the electrochemical process of individual nanostructure has been directly visualized and studied. The electrochemical processes of MoS_2, Fe_2O_3,Co_3O_4, CuO, ZnO, Co_9S_8/CNT in LIBs have also been studied by us. For examples, the filled CNTs behave differently in LIBs depending on their structures. The CNT with an open end shows a notable axial elongation of the lithiated filler by 76.5% while the closed CNT shows a major radial expansion of 22.8%. The reversible conversion between Co_9S_8 and Co is revealed during the multiple cycles. However, Fe_2O_3,Co_3O_4 and CuO show irreversible phase transformation as anode in LIBs. Our in situ real-time characterization results have provided direct evidence and achieved a profound understanding of the lithium-storage mechanism of these anode materials.

M13

Application of SSRF in materials characterization

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Shanghai Synchrotron Radiation Facility (SSRF) is one of the advanced third generation light sources in the world. It supports and pushes the cutting-edge scientific research and the innovation in China. The facility has been used in many areas of scientific research and industrial development, including biology, physics, material science, chemistry, environmental science, archeology, biomedical applications, medicine and drug development, etc. There have been 14 beamlines commissioned to users now. In this report, we will mainly introduce the diffraction beamline (BL14B1), other beamlines and their application will also been briefly introducted.

X-ray diffraction technique is one of the most widely used techniques to obtain the structure of crystalline materials under various conditions, such as temperature, pressure, electric and magnetic field, etc. BL14B1 is one of the 7 phase I beamlines at SSRF, it is dedicated to x-ray diffraction studies. It focused on materials science, condensed matter physics and other relevant fields. Polycrystalline powder, surface and interface of film / ultra-thin films, and microstructure of nano-materials were the main research objective. Several experimental techniques are available at this beamline, including high-resolution powder diffraction (HRXRD), x-ray reflectivity (XRR), grazing incidence x-ray diffraction (GIXRD), reciprocal space mappings (RSMs), polar map, atomic pair distribution function analysis (PDF), and total reflection x-ray fluorescence spectroscopy (TXRF), etc.

M14

Preparation of fixed abrasive pad and its applications in ultra-precision machining functional materials

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Application of functional materials involve super smooth surface in most cases, which is inseparable from ultra-precision machining technology. The existing problems in loose abrasive lapping and polishing process will be analyzed and a new technology-fixed abrasive lapping and polishing, one of ultra-precision machining methods, will be introduced. The advantages of fixed abrasive technique will be illuminated and the preparation of hydrophilic fixed abrasive pad (FAP) and its evaluation system will be introduced. After that, the processing of ultra-precision machining for functional materials using fixed abrasive technology will be demonstrated, such as K9 glass, quartz, sapphire, LBO crystal, ZnS crystal, CaF_2 crystal and so on.

M15

Emerging Single Phase State in Manganite Nanodisks

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Electronic phase separation (EPS) which has a great influence on transport and magnetic properties of strongly correlated oxides has been widely studied. But the behavior of EPS in strongly correlated oxides with sample size smaller than length scale of EPS is still unknown.Understanding this behavior is critical for future oxides electronics and spintronics since scaling down of the system is unavoidable for these applications.

In this work, we fabricate micrometer-sized disks from La0.325Pr0.3Ca0.375MnO3(LPCMO) epitaxial thin film which is well-known for its large length scale of EPS and use magnetic force microscopy (MFM) to get magnetic images of the disks.

While EPS state featuring coexistence of ferromagnetic metallic and charge order insulating phases appears to be the low temperature ground state in bulk, thin films and large disks， we find a single ferromagnetic phase state emarge as a new ground state in smaller disks.

The critical size is between 500nm and 800nm, which is smaller than length scale of EPS in LPCMO film. The ability to create a pure ferromagnetic phase in manganite nanodisks is highly desirable for spintronic applications.

Keywords: manganites, electronic phase separation, magnetization, single phase

M16

The Preparation Studies of ZnO Nanoribbon Arrays

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ZnO has various structural forms, which has important applications in light, gas sensors, etc. In this paper, using Sn-Zn alloy anode oxidation in $C_2H_2O_4{\cdot}H_2O$ (oxalic acid) solution, part of Zn was removed, and then high temperature calcination in air, ZnO nanoribbons arrays were obtained. The effects of electrolysis voltage, electrolysis time and the electrolyte

concentration on ZnO nanoribbon arrays were studied. The samples were characterized by field emission scanning electron microscopy (FESEM) and X-ray diffraction (XRD). The results show that ZnO nanoribbon arrays had better growth and the higher sensitivity of the H_2 at room temperature with the condition of 5V, 10~15 min and 0.01mol·L^{-1}.

M17

Growth of Thin Films Through Two Laser Ablation in the Pulsed Laser Deposition System

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The technique of PLD has been used to deposit high quality films of materials for more than a decade. It uses high power laser pulses to melt, evaporate and ionize material from the surface of a target. This ablation event produces a transient, highly luminous plasma plume that expands rapidly away from the target surface and then deposit on the surface placed on the opposite. However, the conventional PLD usually obtained films with small sizes, big particles and poor uniformity because of the avalanche effect during the producing of plume. Here, we demonstrate the growth of thin films through two laser ablation system. One infrared laser has been used for preheating the target materials, and the other ultraviolet pulsed laser as exciting source for ejecting a plume. It was supposed that films with high crystal quality and less big particles can be obtained through modulation and matching of the two lasers.

M18

Preparation and Characterization of PMHS Surface-modification SiO_2 Antireflective Coating

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In this paper, poly(methylthdrogen)siloxane (PMHS) was proposed to modify the surface hydrophoibcity of silica antireflective (AR) coating. The AR coating coated substrates were modified by dipping them into the solution which is a mixture of hexane. PMHS and Kastredt catalyst. Two different kinds of models were built for validating the PMHS modification process of sol-gel silica AR coating. It showed that the transmittance were added up to 99.50% modified by 0.2%PMHS. The transmittance were decreased greatly by modified 1.0% and 1.5%PMHS. The results showed that the hydrophobicity of AR coatings was significantly improved after 1.0% and 1.5% PMHS modification, and the water contact angles were improved to 126º and 129º. And after 0.2%PMHS modification, hydrophobicity of AR coatings was improved, the water contact angles increased to 105º. FTIR results show that SiO_2 powers have two addition absorption bands at 796 cm^{-1} and 1257cm^{-1} corresponding to the -Si-CH_3 stretching and bending vibrations after modification, which indicating that the hydrophobic groups are introduced into the SiO_2 particles successfully. The Young's modulus of AR coating was quantitatively recorded by AFM, and the results showed that the Young's modulus increased from 317 MPa to 482 MPa after 0.2%PMHS surface modification.

Keywords: Sol-gel method; Antireflective coating; Hydrophobicity; Poly (methylhydrogen)siloxane