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Investigation on Surface Roughness of Polyimide Film from Vapour Deposition Polymerization

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Polyimide film is an important candidate as an ablator material for inertial confinement fusion targets due to its superior mechanical and thermal properties. During the centripetal blast of the inertial confinement fusion ignition experiment, the roughness would have great effect on the hydrodynamic instabilities. Many inertial confinement fusion experiments exploit controlled variations in initial capsule roughness. This paper mainly presented the investigation on the surface roughness of polyimide film from vapour deposition polymerization. Polyimide films of pyromellitic dianhydride-oxydianiline (PMDA-ODA) were fabricated by vapour deposition polymerization method at high vacuum pressure of 10^{-4} Pa. The effects investigated on the surface roughness include: equipment adjustments, substrate temperature, heating pattern and the monomers deposition rate ratio. Surface morphology of the films was characterized by an interferometer microscopy and scanning electron microscopy. Surface roughness was measured by an atomic force microscopy. The results show that consecutive film can be fabricated at the optimal source-substrate distance of 74 mm. Surface roughness for one-step heating and segmented heating process were 291.2 and 61.9 nm, respectively. Splash of solids can be effectively avoided by adopting a fine mesh. A film with surface roughness of 3.3 nm was obtained when pyromellitic dianhydride and oxydianiline deposition rate ratio was set at 0.9:1. Setting the substrate temperature at 30 ºC is in favour of forming a planar film. The authors then concluded that high-quality film satisfying with inertial confinement fusion experiment can be fabricated by vapour deposition polymerization method.

Key Words: Polyimide film, Inertial confinement fusion, Vapour deposition polymerization.

INTRODUCTION

Polyimide (PI) is one of the most valuable polymers; it has extremely good resistance to high temperature, radiation rays and mechanical stress. Therefore, it has been widely used as an insulating material in various fields, such as space applications, microelectronics, liquid crystal alignment in display devices and membrane films. However, polyimide possesses additional properties in contrast to other polymers[1]: appropriate permeability for deuterium and tritium gas in room-temperature or higher temperature, greater opacity at 351 nm, larger elastic moduli and surviving in a cryogenic temperature. These properties will bring polyimide coating to a more advanced utilization level, especially in the inertial confinement fusion (ICF) applications recently. Inertial confinement fusion is an approach to fusion that relies on the inertia of the fuel mass to provide confinement in laboratory condition. In the forthcoming implementary experiment of fusion ignition, the polyimide film with submicron thickness is used as hohlraum sealed film, while polyimide microsphere is also one of the main candidates of ignition capsule[2,3].

Polyimides were conventionally prepared by a two-stage synthesis[4]; solution condensation of an aromatic dianhydride with an aromatic diamine yielded a soluble polyamic acid (PAA) in highly polar solvents such as N-methylpyrrolidone. Polyamic acid solution was spun-cast onto flat substrates and then thermally cured at 300 ºC or chemical dehydration of this polymer yielded an insoluble and intractable polyimide. Unfortunately, there is no straightforward way this technique can be adapted to make inertial confinement fusion targets by established microencapsulation methods. Furthermore, there are technical issues with this technique that may render the material unsuitable as a target shell material: Polyamic acid is hydrolytically unstable, solvents may remain in the film, adverse interaction may occur between the solvent and the substrate (*i.e.*, mandrel) and it is difficult to control the thickness and uniformity of the shell wall due to the intimate association of processing to the rheological properties and the viscosity of the polymer solution.

The vapour deposition polymerization (VDP)[5-10] technique of making polyimide film is a solventless method for depositing conformal coatings. The process involved three steps:

(1) dianhydride and diamine monomers were sublimed under high vacuum, (2) the vapour phase monomers reacted to form polyamic acid on the substrate surface and (3) the polyamic acid was thermally cured at 300 ºC to form polyimide. The outstanding advantages of this technique were solventless effects and thickness uniformity. Only can the polyimide films by vapour deposition polymerization technique (VDP-PI) be more suitable for inertial confinement fusion application[3]. This process was applied to fabricate polyimide capsule connect with the depolymerizable mandrels technique.

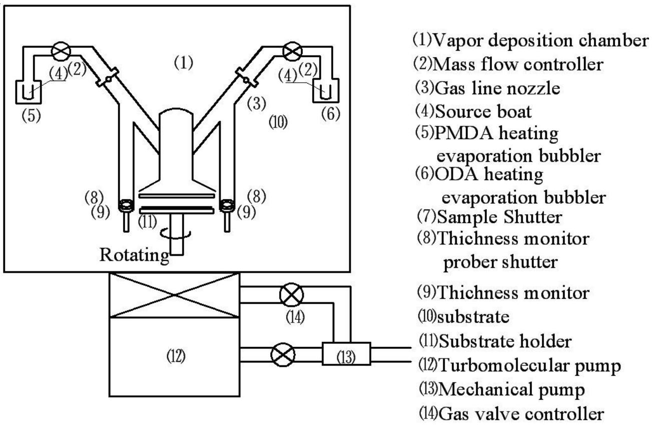


Fig. 1. Experimental equipment of vapour deposition

During the centripetal blast of the inertial confinement fusion ignition experiment, the roughness would enhance greatly the hydrodynamic instabilities. Many inertial confinement fusion experiments exploit controlled variations in initial capsule roughness. Ignition target shells and hohlraum sealed film required the films with 10-30 nm surface roughness and uniform thickness in the inertial confinement fusion ignition experiment. To make polyimide films applied into inertial confinement fusion possible, it was needed to develop the investigation on surface roughness of thin coating. Although polyimide materials had become hot in microelectronic domain increasingly, it was not reported specially about surface roughness of vapour deposition polymerization polyimide films applied into inertial confinement fusion field. In the present work, we report on the preparation of polyamic acid and polyimide films by covaporation of pyromellitic dianhydride (PMDA) and oxydianiline (ODA) monomers on the original vacuum deposition equipment at high vacuum pressure of 10^{-4} Pa and we mainly develop the investigation on the surface roughness of film and influential factors.

EXPERIMENTAL

Polyimides are typically synthesized from the reaction of diamines and dianhydrides. Pyromellitic dianhydride (PMDA; 1,2,4,5-benzenetetracarboxylic dianhydride; 97 % purity) and 4,4'-oxydianiline(ODA; 99+% purity; zone refined) were purchased from the Company of Shanghai Synthesis Resin Academe. Both pyromellitic dianhydride (PMDA) and *bis*(4-aninophenyl)ether (ODA) were purified by preliminary sublimation on the sublimation purification equipment. The substrate was glass slice (4 cm × 4 cm × 0.6 cm) and its root mean square roughness (RMS = Rq) is only 0.8 nm. The glass slides were sufficiently washed in nitric acid (50 %) and acetone and subjected to ultrasound cleaning.

The dual-source vapour deposition polymerization equipment is presented in Fig. 1. The polymer deposition system consists of the vacuum chamber, oil-free mechanical pump, turbo molecular pump, computer collection system and circulating cooling system. Vacuum chamber comprised two source ovens surrounded by ceramic, two thickness monitors, two vapour-flow pipes, two mass flow meters, substrate shutter and substrate holder. The source ovens are mounted on the top flange. The cylindrical boats for containing materials, made of boron nitride material, are placed in the source ovens. The heating element of each source oven is a tungsten wire that surrounded the crucible. Two INFICON quartz crystal oscillator rate monitors with water-cooled are positioned symmetrically at the bilateral area of substrate. A partition is placed between the sources, so that each of the two monitors that measured the molecular beam flux from each source is exposed to only one of the beams. Each rate thickness monitor is connected to an INFICON model XTM-2 deposition process controller. Each of two vapour-flow pipes is also surrounded by a tungsten wire which is used for heating pipes to prevent condensation of either source material. Due to that vapour-flow pipes could steer the monomer vapour flux from top to bottom, this deposition system is used for fabricating film or capsule. In a typical deposition cycle, a substrate is mounted on the substrate holder which is revolving.

The polyimide films were usually prepared by a two-stage synthesis[11]. Two monomers of the polymer were pyromellitic dianhydride (PMDA) and diamine, 4,4'-oxydianiline (ODA). These two monomers, 2.5 g each, were sublimed from two sources. Then vapour molecules collided with each other to form polyamic acid on the substrate. Oxydianiline was sublimed at 110 ºC and pyromellitic dianhydride was sublimed at 125 ºC under a pressure below 10^{-4} Pa. When the deposition rates of two monomers attained stoichiometric ratio, deposition experiment started to initiate by opening the substrate shutter. Typical deposition pressure was 1×10^{-4}-8×10^{-4} Pa. Typical deposition rates were 1-2 nm/s, although higher rates also could be achieved. Films thickness ranging from a few nanometers to a few microns could be easily obtained. As-deposited films were annealed for imidization to take place. Annealing was performed by a segmented cure schedule (3600 s at 170 ºC, 3600 s at 350 ºC) in an oven purged with nitrogen. After curing treatment, residual monomers were desorbed from the polyamic acid film and polyamic acid was dehydrated to form polyimide.

The macroscopical surface topology of thin film was observed by interferometer microscopy (1.2 mm × 0.9 mm scanning size, Leitz Company, German) and electronic microscopy (VHC-600, Japan). The surface roughness of thin film quantitively was surveyed by atomic force microscopy (AFM) (5 µm × 5 µm scanning size, Digital Instrument Company, D300, America).The microscopical surface status was scanned by scanning electronic microscopy (S440, Leica Camberidge Company, UK).

RESULTS AND DISCUSSION

Source-substrate distance: On the smooth glass-slice substrate (RMS = 0.8 nm), three polyamic acid films (a, b, c) corresponding different distances (64, 74 and 84 mm, respectively) were prepared by the deposition device (Fig. 1). Pyromellitic dianhydride monomer and oxydianiline monomer were sublimed at 125 and 110 ºC, respectively, under a pressure of 5×10^{-4} Pa. Deposition took place during 4 h.

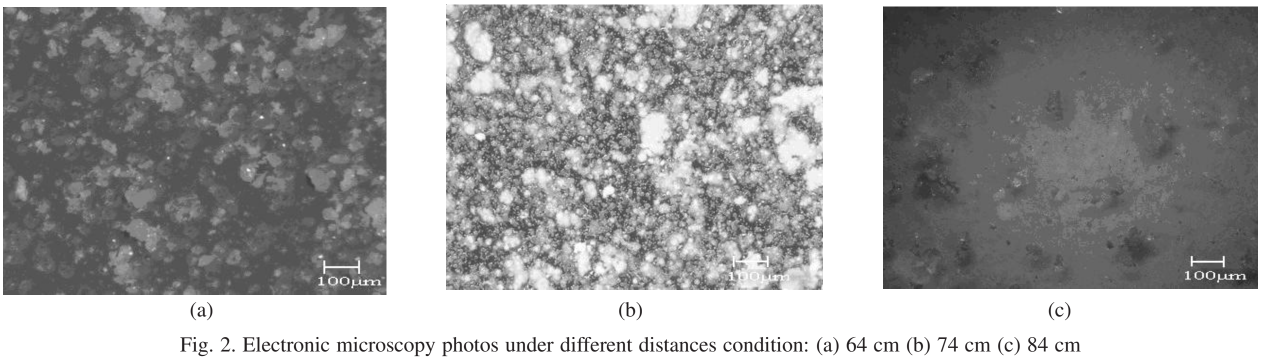
Fig. 2 shows the electronic microscopy photos of three films at different distances. Loose agglomerate islands disperse on substrate surface, as shown in Fig. 2(a), indicating that consecutive film do not shape. Owing to that too short source-substrate distance lead to swift diffuse rate of vapour molecules, vapour molecules polymerized directly on the substrate without undergoing the cycle of migratory adjustment. This phenomenon can be understood by island growing pattern of film propagation. Fig. 2(b) shows the surface of polyamic acid film according to the 74 mm source-substrate distance. Agglomerate islands decrease abundantly and particles become smaller and films structure is emerging. This phenomenon can be explained by layer by layer growing pattern. It takes enough time for vapour molecules at a suitable diffuse rate to migrate on the substrate surface. There is almost no deposit observed in the Fig. 2(c).

The film-forming process involved following steps[12]: (1) atom absorption, (2) atom migration, (3) atom agglomeration and nucleation, (4) the critical nucleus start to forming, (5) small islands bring into existence, (6) channel film begin to engender and (7) continuous film finally shape. Surface roughness depends on atom migration velocity in the course of nucleus-forming, granularity, particle distribution, mass transmission and surface energy distribution. These results can be explained in terms of molecular absorption rate, reaction rate and migration rate. Too short source-substrate distance will lead to a small angle of incidence atom, so too swift absorption rate of atoms or molecules may result into a slow migration rate and there is no enough time for molecules to adjust. Because the probability of pyromellitic dianhydride molecules colliding with oxydianiline molecules is decreasing, monomers reaction rate becomes small. Therefore, absorptive pyromellitic dianhydride or oxydianiline in the form of monomer solid will remains on the substrate, greatly increasing the surface roughness of film. On the contrary, if the distance is far away, absorption rate decreases greatly and migration rate increases abundantly, leading to fewer materials deposition on the substrate. Thus, it is favorable for gaining a consecutive film with reduced surface roughness from vapour deposition polymerization method to adopt an appropriate source-substrate distance.

Heating process: Adopting one-step heating pattern and segmented heating pattern, respectively, two thin films were fabricated, respectively with the vacuum deposition equipment (Fig. 1). Selecting 74 mm as the optimal source-substrate distance, pyromellitic dianhydride was sublimed at 125 ºC, oxydianiline was sublimed at 110 ºC. Heating patterns followed: (a) with one-step heating pattern and (b) with segmented heating pattern to terminal. Deposition took place during 4 h. The average thicknesses (choosing 10 measurement points for each film) were 26.8 and 25.5 µm, respectively.

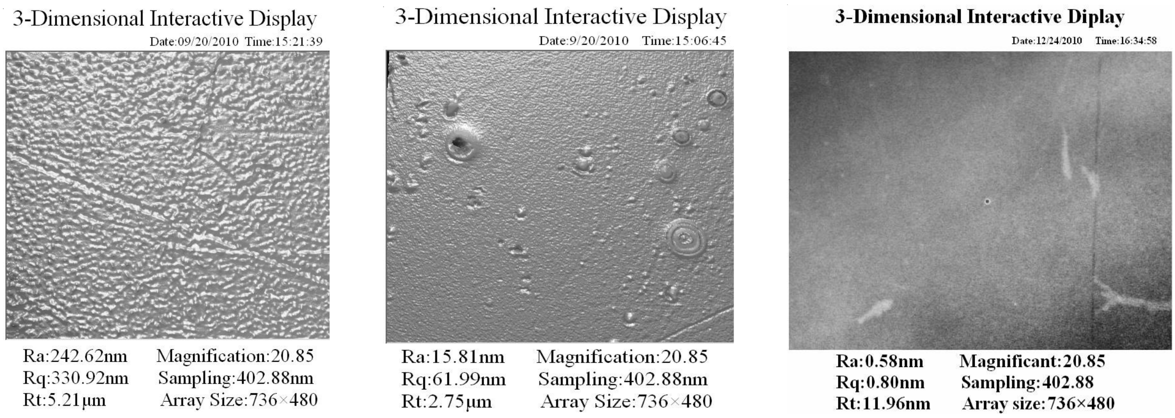
Pyromellitic dianhydride (or oxydianiline) source was heated using the following program: ramp from room temperature (25 ºC) to 87 ºC (72 ºC) in 0.5 h, hold at 87 ºC (72 ºC) for 10 min, ramp to 107 ºC (92 ºC) in 20 min, hold at 107 ºC (92 ºC) for 3 min, ramp to 116 ºC (101 ºC) in 12 min, hold at 116 ºC (101 ºC) for 3 min, ramp to 122 ºC (107 ºC) in 12 min, hold at122 ºC (107 ºC) for 3 min and ramp to 125 ºC (110 ºC) in 12 min, hold at 125 ºC (110 ºC) till deposition over. By observing from above segmented heating pattern, the temperatures increasing rates of pyromellitic dianhydride source (or oxydianiline source) takes on decreasing gradually during every heating interzone, the heating rates are 1, 0.75, 0.5 and 0.25 ºC/min in turns. Thus, this heating process can effectively prevent the evaporation rate of monomer from surge.

Fig. 3(a) shows white-ray interferometer photos of thin film in accordance to one-step heating process pattern. Some big grains lie on the deposition surface, leading to a coarse surface. Quantitively, the Rq is 331 nm. Fig. 3(b) shows white-ray interferometer photos of film in accordance to segmented heating process pattern. The surface barely contains particles, the deposition layer is fine and the Rq decreases to 62 nm, suggesting that the surface quality of film fabricated in the segmented heating pattern is better than the counterpart. During elevating temperature gradually, a large portion of the low molecular weight species would be desorbed from the film, before they reacted with each other to form longer chains polymers. Polymerization and desorption may be competing processes and occur simultaneously. With one-step heating pattern, temperature variation grads are greater. For most of the low molecular weight species, there is no enough time to react with each other to synthesize low polymers and larger oligomers[12]. So small molecules do not polymerize and finally deposit on the film surface in the form of solid particles. Apparently, one-step heating pattern will improve surface roughness of film. With segmented heating pattern, two monomers were sublimed stably. So the fluctuation of vaporization



(a) (b) (c)

Fig. 2. Electronic microscopy photos under different distances condition: (a) 64 cm (b) 74 cm (c) 84 cm

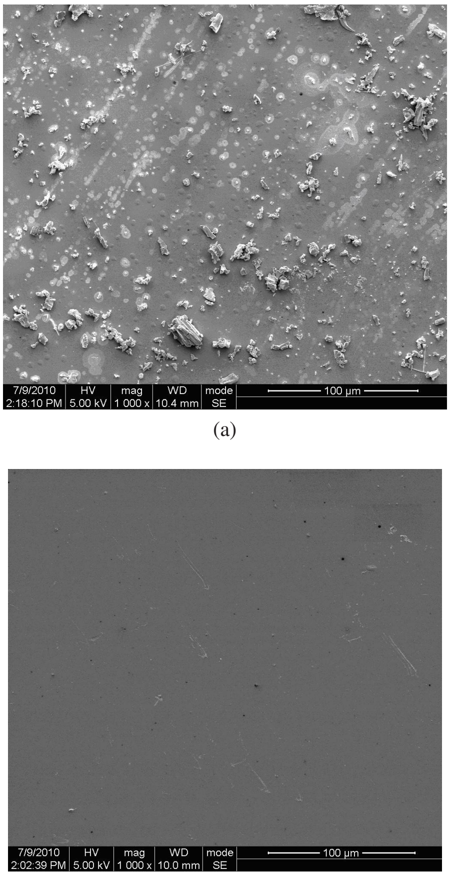


(a) (b) (c)

Fig. 3. White-ray interferometer photos under different heating process (a) one-step heating; (b) multi-step heating; (c) substrate surface

rate and deposition rate were level relatively and polymerization reaction progressed tenderly, resulting into small molecular weight fluctuation of synthetic polymer. From above analysis, to conclude, adopting segmented heating pattern to sublime two monomers can improve apparently the surface roughness of vapour deposition polymerization film. Polymerization and desorption may be competing processes and occur simultaneously. With the fast temperature ramp in the one-step heating process, there was too short a time available for most of the low molecular weight species to react with each other and form larger oligomers[13]. Thus, relative to the case of a slower temperature ramp rate resulting from segmented heating process, most of the low molecular weight species desorbs and unreacted low molecular weight species deposit on substrate again, deteriorating the film surface quality greatly.

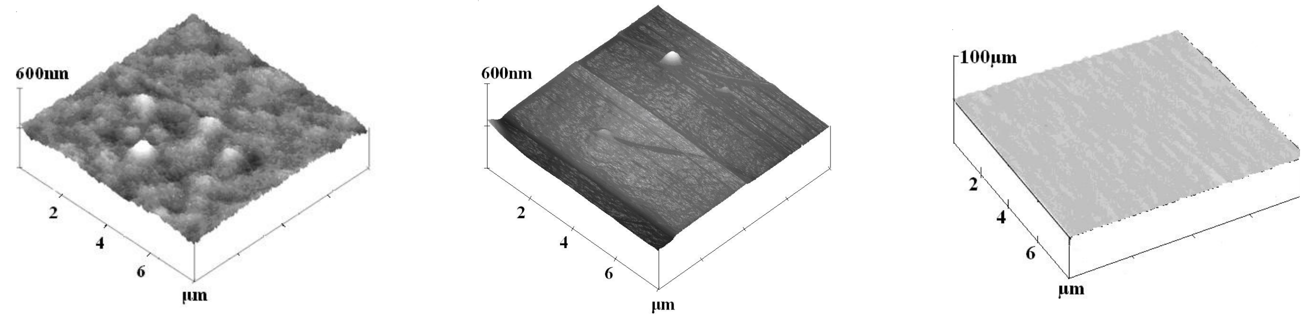
Fine mesh: Two films were fabricated in this section. Source-substrate distance was set the optimal distance (74 mm), pyromellitic dianhydride and oxydianiline were sublimed at 125 and 110 ºC, respectively, with segmented heating pattern heating monomers. The first film was fabricated inserting the mesh between crucible and vapour-flow pipes (each aperture size is 250 μm × 250 μm , 65 apertures per inch ,inserting a fine round mesh into the source gas tube to filtrate the monomers solid and some impurities) and the second film was fabricated without the mesh. Deposition took place during 4 h. The average thicknesses after deposition were 25.7 and 25.4 μm, respectively. Fig. 4 shows the SEM photos of these two films. Some particles and protrudes are observed from the first SEM picture (Fig. 4(a)), while another SEM picture [Fig. 4(b)] presents clean almost, indicating that adding a fine mesh between crucible and vapour-flow pipe can improve the deposition film' surface roughness. Due to adopting the column source boats to contain monomers, materials situating at various layers absorb energy unevenly, which will bring about splash solid of pyromellitic dianhydride and oxydianiline, deteriorating the surface smoothness. It is indicating that adding fine mesh can hinder materials from splash onto the substrate and improve the vapour deposition polymerization-film surface status.



(a) (b)

Fig. 4. Scan electron microscopy photos (a)without fine mesh; (b)with fine mesh

Deposition rate ratio: So far as sublimation process is concerned, sublimation rate mainly depends on temperature and its variation. The pyromellitic dianhydride and oxydianiline sublimation rates are the function of temperature under some constant pressure, so controlling precisely the heating temperatures can gain an accurate deposition rate. In order to gain pure polyamic acid, it is indispensible to keep the ratio of two monomers deposition rate at stoichiometric ratio (1:1) approximately. Therefore, the heating temperature, deposition rate and thickness need to be monitored on the screen real-time. (According to our present equipment, given the area of two round crystals is identical, the deposition thickness is equals to deposition volume of monomer solid. Based on mathematical knowledge, to keep the stoichiometric ratio must keep same deposition thickness for monomers).



RMS roughness (a) 9.1 nm (b) 3.3 nm (c) 0.8 nm (substrate surface)

Fig. 5 AFM picture of polyimide film

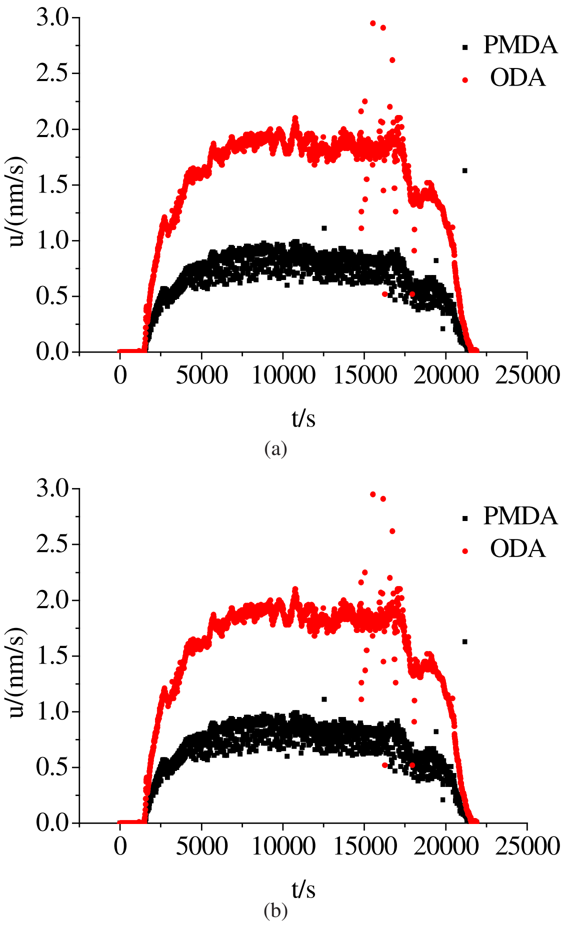


Fig. 6. Curve of pyromellitic dianhydride/oxydianiline deposition rate *vs.* time deposition rate ratio (a) 1:2; (b) 0.9:1

Two thin films were fabricated on glass-slice substrate adopting two sets of terminal subliming temperatures, namely, the first film was fabricated with pyromellitic dianhydride at 125 ºC and oxydianiline at 110 ºC, respectively, the second film was fabricated with pyromellitic dianhydride at 135 ºC and oxydianiline at 105 ºC, respectively. Other process conditions was the same as above section C's parameters: 74 mm source-substrate distance, segmented heating pattern, adding meshes, deposition time took place during 4 h. After deposition, the average thicknesses measured for each film were 25.0-26.4 µm, respectively.

The surface topology of the vapour deposition polymerization-polyimide films was further examined by AFM. The AFM photographs are illustrated in Fig. 5. Comparing with the first photo, the second photo presents few lumps, the tissue is more compact and the roughness characterizing surface roughness of root mean square is one third of counterpart (Rq = 3 nm). To conclude, it is suitable to fabricate high-quality (low surface roughness) film if sublimed temperatures of pyromellitic dianhydride and oxydianiline are set at 135-105 ºC, respectively.

The results can be explained from deposition rate-time curves presented in by Fig. 6. Fig. 6 shows the contact curves (depicted by automatic collected deposition rate data) of pyromellitic dianhydride-oxydianiline deposition rate depending on time during all the deposition process. Fig. 6(a) shows the curve of oxydianiline deposition rate invariably lies above the curve of pyromellitic dianhydride deposition rate. The ratio of depositon rates of pyromellitic dianhydride and oxydianiline is 1:2. Fig. 6(b) shows the pyromellitic dianhydride deposition rates almost keep in accordance with oxydianiline deposition rate simultaneously. Thus, pyromellitic dianhydride and oxydianiline at stoichiometric deposition rate ratio can polymerize adequately to form pure polyamic acid. It is revealed from above results that the pyromellitic dianhydride sublimed at 135 ºC and oxydianiline sumlined at 105 ºC can guarantee two reactants to react at stoichiometric ratio. Wu and Wang[14] concluded that C-N of imines and phenyl groups tended to be oriented parallel to the film surface, while C=O group was oriented perpendicular to the film surface for the thin film. According to Lang's conclusion, as long as the deposition polymer is pure polyamic acid, the content of C-N of imines attains the maximum, so polymer chain tended to be oriented parallel to the plane of substrate. Mixture deposition on substrate will take on disorderly, so that the smooth degree is decreasing. Secondly, if the deposition materials are solid blend including pyromellitic dianhydride or oxydianiline or dimers, or complex, the bonding force of deposited material atom and substrate become smaller, it is difficult to guarantee the different size particles to migrate and diffuse homogeneously and the inner stress of different zones exhibits unbalanced. So the nucleus-forming cycle is always adopting two-dimensional growing pattern in film-forming course, reducing the surface smoothness of film[12]. Thirdly, layer-layer growing pattern is a stable pattern in homogenous extension of film-growing, but in the heterogeneous extension of film-growing layer-layer growing pattern and island growing pattern exist together. So blend deposition tend to grow in heterogeneous way which includes island growing pattern[12], reducing greatly the smoothness of film surface. Above results and three analyses indicate monomer deposition rate ratio is one of important factors and affect the surface roughness of polyimide film. So gaining high-quality vapour deposition polymerization polyimide film must make sure monomer deposition rates ratio stabilize at 1:1.

Substrate temperature: Two films fabrication process condition in this section included: the source-substrate distance is 74 mm, adding mesh between source boats and vapour-flow pipe, controlling the terminal temperatures at 135-105 ºC for pyromellitic dianhydride and oxydianiline, respectively and segmented heating. Difference was following: (a) adding one PTFE shield between heating box of tungsten wire and substrate (substrate temperature is 30.7 ºC) and (b) taking off the PTFE shield (substrate temperature is 60.5 ºC). The deposition took place during 4 h. The average thicknesses for each film measured after depositions were 26.1-26.4 μm, respectively.

Fig. 7 shows white-ray interferometer microscopy of film surface. Seen from them, increasing protrusions emerge in Fig. 7(b) in contrasting with Fig. 7(a) and quantitively the Rq also increases from 4 to 9 nm, suggesting that the surface quality of vapour deposition polymerization film becomes bad, with elevating substrate temperature. The reasons about this diffe-rence are following. Firstly, during vapour deposition course the temperature of substrate can alter molecules tropism[15,16], the molecules tend to be paralleled to the plane of substrate with low temperature, while the molecules are inclined to perpendicular to the plane of substrate with high temperature. Secondly, at the stage of nucleus-forming in early of film-formation. The requisite size of nucleus is bigger and the requisite free energy is higher with the increasing of substrate temperature[12]. So the lower substrate temperature makes for forming a film with fine crystalloid tissue. At the stage of film-growing, the structure of film depends on the relative temperature T_s/T_m and the energy of deposition atom's own[12]. Here, T_s is defined as the temperature of substrate, T_m defines as the melting point of deposition material. According to the SZM principle as mentioned by Wu and Wang[14], if $T_s/T_m < 0.5$, the size of crystal ranged from 5-20 mm, the structure was cylinder crystal configuration with preferential even arrangement. And if $T_s/T_m > 0.5$, the crystal size was exceed 50 mm, the structure was symmetrical axes crystal configuration resulting into uneven arrangement, which increased the surface roughness of film. The results and analysis indicate that controlling the temperature of substrate at low temperature range (30-40 ºC) can improve the surface smoothness of vapour deposition polymerization-polyimide film.

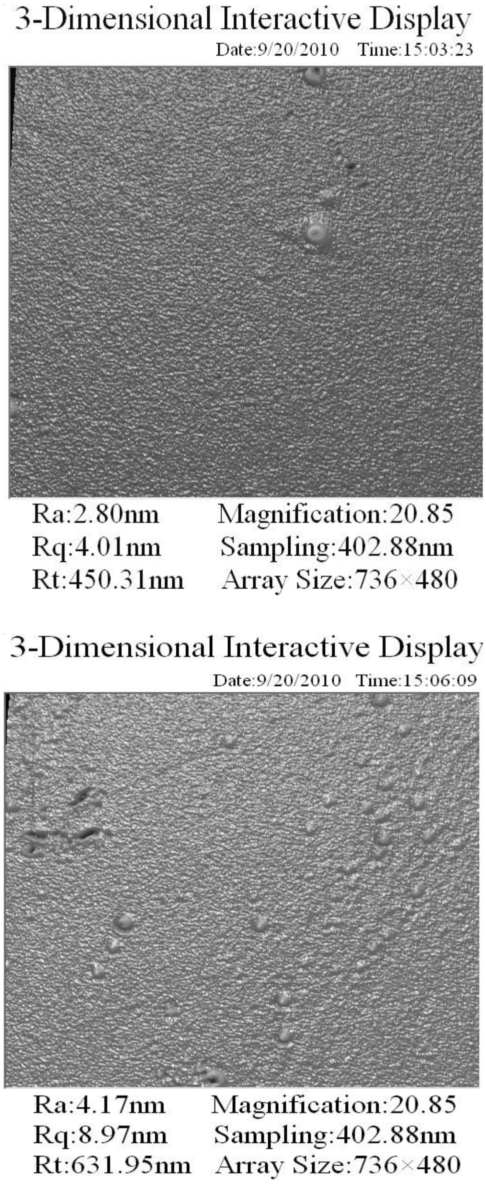


Fig. 7. White-ray interferometer photos under different temperature of substrate (a) 30.7 ºC; (b) 60.5 ºC

Conclusion

From above analysis of the utilized equipment and deposition method, the causes of the surface roughness increase are impurities on the substrate and the island growing pattern at the stage of monomer absorption, migration, polymerization and desorption. It is concluded that effects on vapour deposition polymerization-polyimide film mainly include: materials purification, the source-substrate distance, substrate temperature, monomer vapour filtration, elevating temperature pattern, sublimation temperature, molar ratio of monomers deposition rate, curing temperature and curing time in the course of imidizatioin. To gain the optimal fabrication process parameters, following adjustments must be done: consecutive film can be fabricated at the source-substrate distance of 74 mm, subliming monomers by segmented heating (the heating rates are 1, 0.75, 0.5 and 0.25 ºC/min in turns) pattern can effectively prevent the evaporation rate of monomer from surge, monomers particles and some impurities can be filtrated by a fine round mesh inserted into the source gas tube and it is critical to maintain the stoichiometric ratio (1:1) of monomers when depositing polyamic acid because non-stoichiometric deposition deteriorates greatly the surface finish, the evaporator temperatures for equimolar monomer deposition were pyromellitic dianhydride = 135 ºC and oxydianiline = 115 ºC, controlling the substrate temperature at 30 ºC is in favour of attaining a planar film.

The established vapour deposition polymerization method allows the preparation of polyimide films with a defect-free surface, uniform thickness and desired composition. Therefore, pursuing excellent fabrication parameters based on these factors can produce the polyimide film and capsule up to the surface roughness of inertial confinement fusion experimental demands. To make polyimide film and capsules become the alternative materials of inertial confinement fusion experiment, complementary studies on permeation, thermal property and mechanical performances also must be launched on in future work.

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