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Review Article

Research Progress of Optical Fabrication and Surface-Microstructure Modification of SiC

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SiC has become the best candidate material for space mirror and optical devices due to a series of favorable physical and chemical properties. Fine surface optical quality with the surface roughness (RMS) less than 1 nm is necessary for fine optical application. However, various defects are present in SiC ceramics, and it is very difficult to polish SiC ceramic matrix with the 1 nm RMS. Surface modification of SiC ceramics must be done on the SiC substrate. Four kinds of surface-modification routes including the hot pressed glass, the C/SiC clapping, SiC clapping, and Si clapping on SiC surface have been reported and reviewed here. The methods of surface modification, the mechanism of preparation, and the disadvantages and advantages are focused on in this paper. In our view, PVD Si is the best choice for surface modification of SiC mirror.

1. Introduction

At present, mirror systems as the most important device are applied commonly in the optical system with high precision. Until now, three generations of reflector materials have been developed. The first generation is glass-ceramic; the second one is mainly made of metal, such as Beryllium metal and its alloys; the third generation of the reflector material is based on silicon carbide. SiC may be the best material available for mirror optics because of its outstanding combination of thermal and mechanical properties. It has remarkable dimensional stability even under the disturbances of temperature, humidity, and chemicals. Its specific stiffness and elastic modulus are higher than that of beryllium, which has toxicity. The density of SiC is slightly higher than aluminum and its fracture toughness is higher than glass. The remarkable properties of SiC in terms of hardness, stiffness, and thermal stability combined with a reasonable density are indeed of primary importance for all space applications. This combination of material advantages makes SiC an excellent material candidate for space optical instruments [1, 2].

Based on microstructure and processing routes, four kinds of SiC ceramics including hot-pressed SiC (HP-SiC), reaction-bonded SiC (RB-SiC), sintered SiC (S-SiC), and chemical vapor deposition SiC (CVD-SiC) were developed. The properties of different SiC materials and the brief description of various SiC component manufacturing techniques are summarized in Table 1 [2, 17]. Whatever the preparation process, it is difficult to obtain high-quality optical surface due to polishing the bare SiC very difficultly. Moreover, microstructure defects, like pores, steps at different phases, and grain boundary damages, are unavoidable under certain surfacing condition and present further difficulty in polishing this material. The AFM topography images of surfaces polished by 1 um abrasive grains of different SiC ceramics are shown in Figure 1. The steps still exist at the interfaces between two phases both in RB-SiC and S-SiC, and it cannot meet the optical requirements (<1 nm RMS). The rms surface roughness values of SiC ceramics

Table 1: Different preparation methods and properties of silicon carbide.

Materials	Density (g/cm^3)	Free state of Si	Isotropic	Cost	Preparation cycle	Surface modification
HP-SiC	3.20	No	Bad	High	Short term	Requirement
RB-SiC	3.04	Yes	Good	Low	Short term	Requirement
S-SiC	3.10	No	Worse	Lower	Long term	Requirement
CVD-SiC	3.21	No	Best	High	Long term	Nonrequirement

are 6.464 nm and 3.017 nm [18]. The high hardness and complex crystal-phase structure of the SiC material indicate that the fabrication of high precision optical components is costly and slow. The key to solving this problem is to select the appropriate preparation process for surface modification of SiC matrix material, the dense SiC mirror optics plated coating, and the coating polishing. Efficiency of surface finishing for large optical components can be greatly improved as well. Moreover, microstructure defects on polished surface of SiC ceramic mentioned above can be covered up by the coating process. This paper presents the optical surface processes and the recent developments of SiC substrate by hot pressed glass, C/SiC clapping, CVD- and PVD-coated SiC and Si coating method in detail [19–21].

2. Optical Fabrication of Silicon Carbide

However, RB-SiC is typically a difficult material to machine. SiC is harder than most other materials except diamond, cubic boron nitride (cBN), and boron carbide (B_4C), and hence available cutting tool materials for machining RB-SiC are very limited. Recent efforts have focused on the precision machining of RB-SiC by grinding, lapping, polishing, and combinations of these [24, 25]. Main features and classification of optical fabrication methods of SiC are shown in Table 2 [26–28].

3. Surface Modification

Surface modification of SiC material is to add a thick film coating to the substrate of SiC for obtaining better surface quality and easier polishing. After fine polishing, the surface shows an extremely smooth surface and it can meet the optical requirement. The earliest study method is hot pressing glass, the recent developments of SiC surface modification is CVD- and PVD-coated SiC and Si, and C/SiC clapping is also applied to space optical devices.

3.1. Hot Pressed Glass. The hot pressed glass process was developed by the German IABG Company and the thin preshaped borosilicate glass plate is thoroughly pressed to a preground C/SiC substrate at elevated temperature near the glass softening temperature. After cooling down, the glass plate is bonded to the substrate without the use of any adhesive. The resulted layer of approximate 1 mm thickness is well and economically polishable. But there are some drawbacks for usage of the glass cladding on large surfaces. For example the application of the high pressure led to the CTE mismatching and residual internal stresses. Now, this process has rarely been used in the size of a large mirror [29].

3.2. Si/SiC Clapping. The Si/SiC claddings are processed by an economic deposition of SiC-containing slurry onto the preground surface. Subsequent ceramization of the deposited cladding by thermal treatment leads to the conversion of the slurry cladding into a dense and homogeneous two-phase ceramic surface layer consisting of SiC and silicon. The Si/SiC layer exhibits a strong chemical bonding on the C/SiC base material due to its related chemical nature. By optimizing the ratio of Si to SiC in the layer, its CTE can be tuned to match with that of the C/SiC bulk material over a very broad temperature range. This is important for a thermally stable mirror. Although the described two-phase Si/SiC cladding might not be suitable for superpolished surfaces below 1 nm RMS microroughness, the advantages of the Si/SiC cladding technology for conventional optical components can be summarized as that surface roughness with <2 nm RMS can be obtained with standard optical polishing techniques. A series of studies for this technology [29–31] have been reported by IABG and DSS company.

3.3. SiC Clapping. SiC clapping has the same mechanical properties and thermal physical properties as the SiC matrix, so that SiC clapping as a surface modification coating of silicon carbide mirrors attracts extensive attention of researchers. SiC coatings exhibit many outstanding properties, like good isotropic, high hardness, high thermal conductivity, and excellent optical performance characteristics. The amorphous SiC coating prepared by PVD technique with the low temperature and short cycle has also been reported.

3.3.1. CVD SiC Clapping. Chemical vapor deposition has been widely applied in thick-film preparation from the 1960s, and its principle is that the reaction materials resulted from the decomposition of Si-containing precursors are deposited on the substrate surface to form a thin film. The film is of good uniformity and repeatability. Such a method is applied to the preparation of SiC coating. Polished CVD-SiC (crystalline cubic α-SiC) holds also good physical properties for making mirrors such as low density, high melting point, and low expansion coefficient.

The main precursors for CVD SiC is CH_3SiCl_3, and the reaction equations are as follows: $CH_3SiCl_3(g) \rightarrow SiC(s) + 3HCl(g)$. The dense SiC coating with excellent optical properties (surface roughness <0.3 nm RMS) can be obtained. It can meet the application requirements of the mirror surface

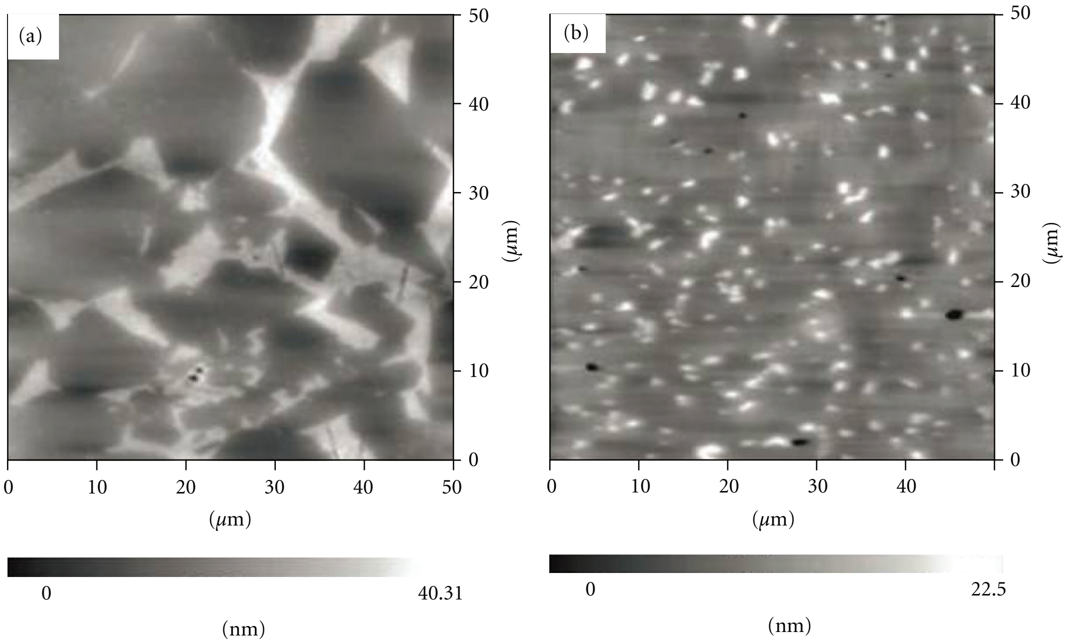

Figure 1: AFM images of SiC surfaces polished by 1 um abrasive grains within 50 um × 50 um area: (a) RB-SiC, (b) S-SiC, reprinted with permission from [18].

Table 2: Main features of optical fabrication methods of SiC.

Optical fabrication of silicon carbide	Principle	Characteristic	Example
Mechanical polishing	Mechanical friction and material removal	Low efficiency	The final surface accuracy of 1 nm RMS was reported by Paquin et al. [3]
Wet polishing	Polished mold is immersed in the slurry, RMS becomes lower by an abrasive	High surface accuracy	The final surface accuracy of 0.75 nm RMS was reported by Xu et al. [4]
Ultraprecision grinding	Ductility grinding	Equipment require high	Bifano et al. [5] has used CVD SiC to obtain the 5.5 nm RMS
Tribochemical polishing	The tribochemical reaction	Low efficiency	In [6], final surface accuracy of 1 nm RMS
Electrolytic in-process dressing (ELID)	The electrolysis process so that the grinding wheel functions	Good surface quality	In [7], final surface accuracy of 1.4 nm RMS
Chemical mechanical Polishing (CMP)	Combination of mechanical grinding and chemical etching	Good surface quality, but is corrosive	In [8], final surface accuracy of 0.5 nm RMS
Magneto rheological Finishing (MRF)	Magnetorheological polishing fluid viscosity increases the shear force generated for material removal in the magnetic field gradient	Low efficiency	Johnson et al. have used this method to fabricate CVD SiC [9]
Laser-induced photochemical polishing	Laser-induced photochemical reactions	High efficiency, but is corrosive	The final surface accuracy of 80 nm RMS was reported by Murahara [10]
Ion beam milling	High-speed ion beam hits the surface of the sample	Good surface quality, expensive equipment	The final surface accuracy of 1 nm RMS was reported by Johnson et al. [11]
Float polishing	The sample is floating on the polishing plate by the high-speed rotating fluid dynamic pressure	Good surface quality	In [12], final surface accuracy of 3 nm RMS

Table 3: Different methods for preparing CVD SiC.

Methods	Advantages and disadvantages	Example
APCVD	The process is simple and has fast response, but with poor uniformity	Chung and Kim [13] use APCVD growth of single-crystalline 3C-SiC (cubic silicon carbide) thin films. The 3C-SiC film had a very good crystal quality without twins, defects, or dislocations, and a very low residual stress
LPCVD	It is possible to grow thin films with uniform and smooth morphology during the 3C-SiC film growth. LPCVD has disadvantages of low growth rate	Clavaguera-Mora et al. [14] have used Si $(CH_3)_4$ as the source materials to deposit SiC film by hot CVD device. Films grown at 900–980°C are amorphous with nanocrystals of mean grain size 10 nm and have smooth surfaces. Surface profilometry measurements give a root mean square roughness (RMS) of 6 nm
PECVD	Substrate temperature is low and deposition rate is fast. SiC films deposited by PECVD appeared to be in an amorphous state	Novi et al. [15] have used PECVD to prepare amorphous SiC, and there has been a lot of cubic structures on the surface, which indicates the existence of polycrystalline structures

and it is one of the most effective methods for surface modification to prepare SiC-based reflection mirror. But the CVD process with high substrate temperature (>1000°C) would lead to the deformation of SiC-matrix. Another disadvantage of the CVD process is time consumption.

There are different treating methods for CVD SiC process, including atmospheric pressure chemical vapor deposition SiC (APCVD SiC), low-pressure chemical vapor deposition SiC (LPCVD SiC), and plasma enhanced chemical vapor deposition SiC (PECVD SiC) [32–34]. Different methods for preparing CVD SiC are shown in Table 3.

CVD SiC coating has been widely applied to surface modification of SiC matrix. For example, CVD SiC with 0.2 nm RMS on the C/SiC substrates had been reported by Trex Advanced Materials [21]. The CVD SiC on S-SiC substrates in the polished roughness of less than 0.1 nm has also been reported by BOOSTEC. In China, Zhang [35] applied this method to obtain CVD-SiC with the surface roughness of 1.478 nm RMS.

3.3.2. PVD SiC. High temperature (typically 1300°C) in the preparation process of CVD SiC may result in stronger film stress on the SiC mirror. This is due to the unacceptable high residual stress buildup in heavy cross-sections. The cost of producing CVD SiC as a large self-supporting substrate is very high. However, PVD SiC appears to be very attractive due to its relative simplicity, low substrate temperature, and wide accessibility by industry.

Ion-Assisted Deposition SiC (IAD SiC). The ion implantation of semiconductors rapidly became an accessible technology from the 1970s because of its ability to produce superior electronic devices. Ion beam modification of nonsemiconductor materials for enhancing surface sensitive properties has been actively pursued in the international R&D community. The advantages of this technique include high density, superior adhesion, and the ability to produce arbitrarily thick coatings. Perhaps the most important feature of the IBAD technology is the frequently demonstrated ability to control many coatings properties such as morphology, adhesion, and stress, as well as stoichiometry. The amorphous SiC coating with large area can be prepared by this method on the SiC substrate. The α-SiC coating has been prepared on the RB SiC and polished with surface roughness up to 0.2 nm RMS by U.S. HDOS [3, 36] using ion beam deposition method. Hall ion source [37] and high-energy Kaufman ion source [38] can both be used as the ion-beam resources for preparing α-SiC and its system of SiC-modified membrane and the surface roughness can be down to 0.867 nm RMS and 0.743 nm RMS.

Magnetron Sputtering SiC Clapping. Magnetron sputtering is used widely due to its low cost, high deposition rate, low deposition temperature, and good adhesion of the film feature. The SiC coatings were deposited by RF magnetron sputtering from a sintered SiC target onto commercially monopolished RB SiC substrate kept at low temperature. The deposition rate is fast and the desired temperature is low, but the film stability of its system is not sustainable [39]. Magnetron sputtering SiC clapping was prepared with the roughness down to 1.394 nm RMS [40] and 3.184 nm RMS as shown in Figure 2 by Tang et al. [22] and the surface roughness of 2 angstrom by Kortright and Windt [41].

Pulsed Laser Deposition SiC (PLD SiC). PLD is a relatively new technique and attracting great attention for its simplicity, reduced investment cost, and flexibility. In the PLD process [42], a high flux pulsed laser beam is focused on the target material leading to the formation of a plasma plume. The high heating rate of the target surface (≈100 K/s) due to pulsed laser irradiation leads to a congruent evaporation of the target irrespective of the evaporating point of its constituent elements or compounds, so that the target stoichiometry can be retained in the deposited films.

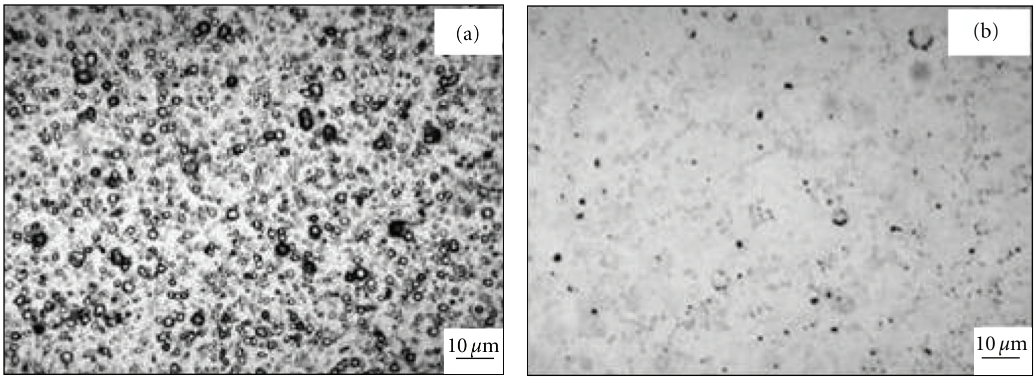

Figure 2: Photomicrograph of (a) unpolished, (b) polished PVD SiC coating on RB SiC, reprinted with permission from [22].

Table 4: Thermal performance of RB-SiC and Si [16].

Materials	α/(ppm/K)	K/(W/mK)	D	α/K	α/D
RB-SiC	2.4	170	80	0.014	0.03
Si	2.6	156	89	0.017	0.029

Furthermore, because of the high heating rate of the ablated materials, PLD of crystalline films demands a much lower substrate temperature than other film growing techniques. The major drawback of this technique is that the high energy involved in the process also leads to the formation of microparticulate on the thin film surface. Vendan et al. [43] has prepared α-SiC thin films by fs-PLD and ns-PLD techniques and found that the surface roughness of SiC films by the ns-PLD technique was bigger than that by fs-PLD. Magida et al. [32] has prepared SiC thin films which exhibit high reflectivity in the ultraviolet band (40.7 nm–121.6 nm) and the surface roughness of 1 nm RMS.

Ion Beam Sputtering SiC (IBS SiC). The ion beam engine concept was firstly developed in the United States. By the IBS technique, the SiC films being flat and smooth, a large area of dense internal stress and low defect density can be obtained. For example, the IBS SiC coatings have been prepared and polished to be less than 2 A RMS by Johnson [36] for the optical system requiring ultralow scatter performance.

3.4. Si Clapping. The thermal properties of Si coating match well with that of SiC as shown in Table 4, then Si clapping can be used for the surface modification of SiC. It can be seen that their thermal performance is well matched, and Si can be used as a good reflector material. CVD and PVD are the main preparation methods.

3.4.1. CVD Si Clapping. The Si clapping is easier to be polished well with better surface quality and low-cost SiC-clapping. Polycrystalline Si produced by a scalable CVD process has exhibited a surface finish of 0.2 nm RMS. Polycrystalline Si was fabricated by reacting trichlorosilane ($SiHCl_3$) with excess H_2 in a hot-wall CVD reactor according to the reaction: $SiHCl_3 + 2H_2 \rightarrow Si(s) + 4HCl(g)$. However, it is not widely applied [14, 44] because columnar structure is often present in CVD Si.

Polycrystalline Si [19] was also used to clad on several advanced ceramic materials such as SiC, sapphire, pyrolytic BN, and Si by a chemical vapor deposition (CVD) process. The thickness of Si cladding ranged from 0.025 to 3.0 mm. CVD Si adhered quite well to all the above materials, where the Si cladding was highly stressed and cracked. The surface roughness can reach 0.2 nm RMS after polishing. Amorphous silicon thin films were formed by chemical vapor deposition by Choi et al. [45]. The amorphous silicon films without reflector bias voltage exhibit 0.119 nm RMS, but down to 0.171 nm RMS with reflector bias voltage of −120 V, respectively.

3.4.2. PVD Si Clapping. CVD Si is generally prepared with high temperature (>600°C), and the PVD Si film is widely used. The application of Si coating can reduce surface wearing resistance of SiC ceramic without changing mechanical properties of the bulk materials. The efficiency of surface finishing for large optical components can be greatly improved as well [23]. Therefore, PVD Si appears to be very attractive because of its relative simplicity, low substrate temperature, and wide accessibility by industry. Si clapping is easier to be polished well because it is single-phase material without the different heterogeneous phase in the polishing process. PVD Si coating is now becoming a preferred method of the SiC surface modification.

Vacuum Evaporation Si Clapping. Vacuum evaporation deposition of Si film is processed in a high vacuum by heating the Si gasification or sublimation condensing into Si film and thus being deposited on SiC substrate surface. The method is relatively simple and has high deposition rate. But the columnar structure is present in Si film and the physical property of Si film is not stabile. Zhao [46] has obtained amorphous Si films by thermal evaporation. In recent years, this method has been improved. Fang et al. [47] deposited Si film on steel and alumina substrates by electron beam evaporation of solid silicon.

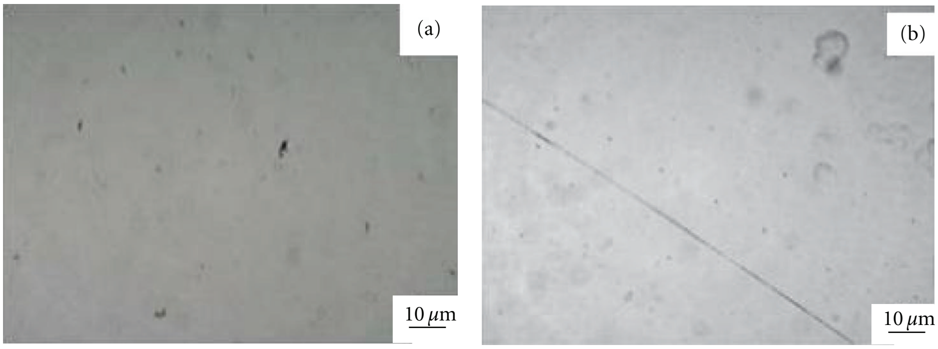

Figure 3: Photomicrograph of (a) unpolished, (b) polished PVD Si coating on RB SiC, reprinted with permission from [22].

Magnetron Sputtering Si Clapping. Magnetron sputtering (belongs to PVD) appears to be very attractive due to its relative simplicity and low substrate temperature. Many researchers have studied this method, Aoucher et al. [48] deposited amorphous silicon by DC magnetron sputtering on a quartz substrate at a rate around 1.5 nm/s. Tang et al. [22] has used RF magnetron sputtering method to prepare Si film with the surface roughness from 17.992 nm RMS to 1.183 nm RMS as shown in Figure 3.

Ion-Beam-Assisted Si Clapping. Hydrogenated amorphous silicon (a-Si:H) films are generally prepared by glow-discharge decomposition of silane or by sputtering of silicon in an argon-hydrogen mixture. The reaction temperature is low (200°C) and the preparation parameters can be controlled. So it was used to prepare amorphous silicon films.

Photoconductive hydrogenated amorphous silicon films were deposited by ion-beam-assisted evaporation using hydrogen-argon plasma. Surface modification for the RB-SiC substrate [49] is carried out using e-beam evaporation with plasma ion assisted. The surface roughness of the RB-SiC substrate is reduced to 0.0632 nm, the scattering coefficient is reduced to 2.81%, and the average reflectance from 500 nm to 1000 nm is raised to 97.05%; these data indicate that good optical quality similar to that of the fine polished glass ceramics can be obtained by the modification process.

Plasma-Ion-Assisted of Deposition Si Clapping. For the production of thin films of high quality standard, thermal evaporation techniques are applied with the assistance of ion sources which provide additional energy and momentum to influence the growth process. Larger ion current densities on increased substrate areas can be generated by employing plasma sources, and the process is plasma-ion-assisted deposition (PIAD) [50]. In PIAD, a growing thin film is bombarded by energetic ions from a plasma ion source and the columnar microstructure of the film is disrupted, resulting in the improved optical and mechanical properties of thin films [51]. Liu et al. [23] has prepared Si thin film by this method and obtained continuous, homogenous, well-bonded amorphous Si coatings on SiC ceramics. It means that the SiC substrate can be fully covered up and the effect of substrate surface defects on the surface morphology of the Si coating can be overlooked as shown in Figure 4.

Recently, the PVD Si coatings on SiC substrates have been investigated. SSG has applied PVD Si in optical systems of the GEO telescope [52], designed and constructed for an SBIR program funded by NASA's Marshall Space Flight Center (MSFC). The SiC telescope and "GOES-like" scan mirror are designed to "generic" GEO specifications, and the surface roughness decreased to 0.4 nm RMS after polishing. In China, the PVD Si coatings deposited on the surface of polished RB-SiC and S-SiC were demonstrated to improve the optical surface quality after being polished by Tang et al. [22]. Both the surfaces of PVD Si coating on RB-SiC and S-SiC are smoother than that of bare SiC. The RMS can reach to the angstrom grade, and the reflectance improves significantly.

4. Conclusion

Silicon carbide as the third generation of space mirror material has attracted more and more attention and is widely applied. Silicon carbides prepared by different preparation methods have their advantages, but still cannot meet the optical requirements (<1 nm RMS) after the current optical processing. The surface roughness and reflection rate of the SiC material can be well improved after surface modification. Especially in the CVD SiC coating and PVD-Si coating on the SiC ceramic, surface roughness of Angstrom level can be reached. All kinds of surface-modification methods have been developed and every method has its disadvantages. The hot press glass has some drawbacks for usage of the glass cladding on large surfaces. The C/SiC coating may not be suitable for low surface roughness (<1 nm RMS). The CVD process with high temperature (>1000°C) would lead to the deformation of SiC matrix. PVD method to prepare SiC-film is slower and the modified film is very difficult to be polished. In our view, PIAD Si has low reaction temperature (<300°C) and is very easy to be polished. The preparation process is relatively simple, and reproducible preparation of silicon-modified layer gives a dense structure, combined with the base firmly. Therefore, PVD Si is the best choice for surface modification of SiC mirror because of its relative simplicity, low substrate temperature, and wide accessibility by industry, especial PIAD Si.

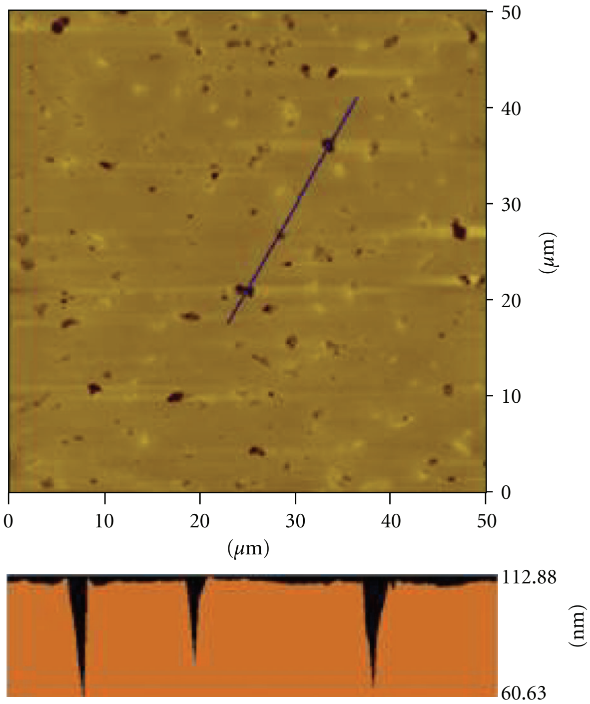

FIGURE 4: Surface topography of Si coating tested by AFM, reprinted with permission from [23].

Acknowledgments

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