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Original Article

Temperature environmental adaptability on electrical and stress properties of Ge film in space

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ABSTRACT

The Ge-film-based photon-counting imaging detector is a key component of optical payload in space weather observation. It's important to study the space temperature adaptability of Ge film since the performances of Ge film are extremely sensitive to the temperature. Herein, the in-situ tests are proposed to measure the resistance and stress of the Ge film at the operating temperature. It is demonstrated that the resistance decreases from 520 MΩ/□ to 124 MΩ/□ linearly between −20 °C and 80 °C, which can meet the requirement of the imaging system well. And the stress gradually releases in the thermal cycle. The ex-situ test is adopted to investigate the limited temperature adaptability and the intrinsic mechanism of performance variations. When the temperature increased to 600 °C, Ge film failed and cracked because of resistance reducing to 30 KΩ/□ and stress increasing to 504 Mpa. Meanwhile, methods were proposed to enhance the temperature adaptability of Ge film by analyzing crystal phase and optical band gap. Thus, the resistance and stress changes of Ge film in space have little effect on the performances of imaging system. The Ge film with high temperature adaptability and low stress we prepared is promising in the space observation.

1. Introduction

Space weather phenomena such as the plasmasphere, aurora and corona have a great impact on the production, survival and space activities of human being. The space weather monitoring which used in scientific researches and applications has been established gradually in China. The extreme ultraviolet camera (EUVC) [1] of Chinese Chang'E−3 (CE-3) and Wide Angle Aurora Imager(WAAI) [2,3] of Fengyun-3 have observed the Earth's plasmasphere and aurora successfully, and the corona will be imaged by Lyman-alpha Solar Telescope (LST) [4,5]of the Advanced Space-based Solar Observatory (ASO-S) in 2022.

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Photon counting imaging detector [6,7], with the advantages of low noise, high sensitivity, high dynamic range and strong anti-radiation ability, can meet the imaging requirements of observation mission of space weather. It has been adopted in the space weather detection by the EUVC, WAAI, and LST mentioned above. It is consisted of a microchannel plate (MCP), a position sensitive anode [8,9] and a position readout circuit. The Ge film, as a charge induced layer of the position sensitive anode, can effectively reduce the image distortion and improve the imaging signal-to-noise ratio. During the operation of the detector, the resistance of the Ge film must be stabilized within a certain high resistance range, otherwise, the image quality will be reduced seriously [10]. Moreover, large stress may induce film cracking and deformation of the position sensitive anode, leading to the failure of the imaging system. However, the resistance and stress of Ge film are extremely sensitive to the ambient temperature. The space environment temperature of spacecraft is general between −180 °C and 150 °C. Thus, it's necessary to study the resistance and stress performance of the Ge film in the space temperature environment.

The influence of thermal annealing on the photoelectric properties of Ge film has been studied by different researchers. Tsou et al. [11] deposited the Ge film through e-beam evaporation, then compared the optoelectronic properties difference between the microwave annealing and rapid thermal annealing. Khan et al. [12] deposited the Ge thin films by e-beam evaporation either, and investigated the optical and electrical properties of Ge films annealed in the temperature range from 100 to 500 °C in air. Peng et al. [13] studied preparation method of polycrystalline Ge film through the Al-induced annealing for thin-film transistors and solar cells. Liao et al. [14] prepared the metal-induced crystallization of ultrathin Ge films by rapid thermal annealing for CMOS production. Witvrouw et al. [15] and Fahnline et al. [16] demonstrated that the stress of Ge films released after annealing process. Ponraj et al. [17] studied the residual stress of Ge film which grown as an epilayer at different temperature. Zhao et al. [18] studied the influence of Ge films annealing at different temperatures on the imaging quality of photon-counting imaging detector. What should be noticed is that, all the thermal studies of Ge film mentioned above were the ex-situ measurements after annealing instead of in-situ measurements. So the in-situ and real-time performances of Ge film in space temperature environment have not been studied yet. Moreover, little work has been devoted to the limited temperature adaptability of Ge film. Furthermore, the principle of the photoelectric performances of Ge films affected by temperature has not been fully investigated.

In this paper, we proposed the in-situ and real-time resistance and stress tests of Ge film at the typical operating temperature between −20 °C and 80 °C controlled by the thermal control system in spacecraft. To investigate the limited temperature adaptability of Ge film, ex-situ resistance and stress tests were studied from −190 °C to 600 °C, which covers different kinds of environment temperatures in the space. Meanwhile, by analyzing the crystal phase and optical band gap of the microstructure in the Ge film at different temperatures, the inclusive mechanism of the changing photoelectric properties were studied. Based on this research, we presents the preparation method of high-adaptability Ge film that is promising in aerospace applications.

2. Experimental details

2.1. Ge film synthesis and characterization

Ge films were deposited by magnetron sputtering coating machine on the silica substrates with a diameter of 33 mm, a thickness of 1.5 mm, and a surface roughness (RMS) of 0.6 nm. The purity of the Ge target was 99.999%. The resistance was measured by the sheet resistance meter (Jandel RM3-AR). The X-ray diffraction (XRD) was performed by the Rigaku DMAX2000 X-ray diffractometer which uses the Cu K-α line (λ = 0.154 nm) with an angle resolution of 0.005°. The surface morphology was measured by the optical microscope and the surface profilometer (New View 6000STP, zygo). The surface shape of each substrate was measured by zygo GPI XP/D interferometer.

2.2. In-situ and ex-situ electrical test

The in-situ temperature control device is shown in Fig. 1 (a). Ge film sample was placed in an aluminum temperature control fixture, that inner side of the fixture was smeared with heat-conducting silica gel, and the back of the sample was fully contacted with the heat-conducting silica gel, so that heat could be effectively conduct. Temperature sensors were attached on the front and back of the fixture to monitor the

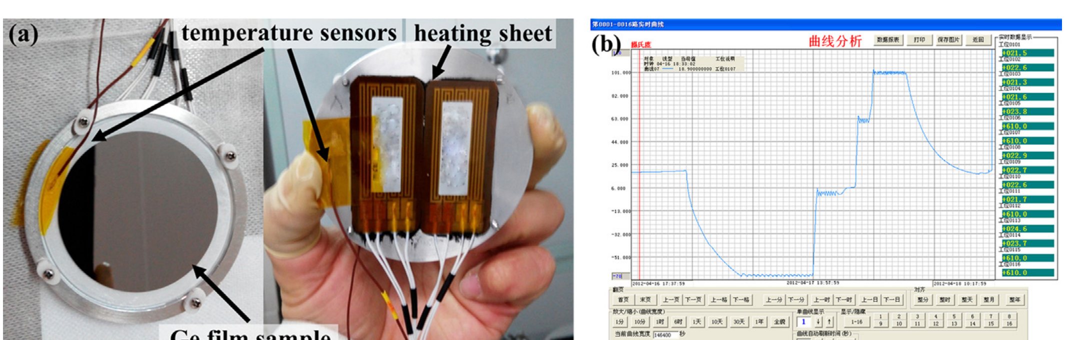

Fig. 1 – Aluminum temperature control fixture (a), temperature real-time controlled system (b).

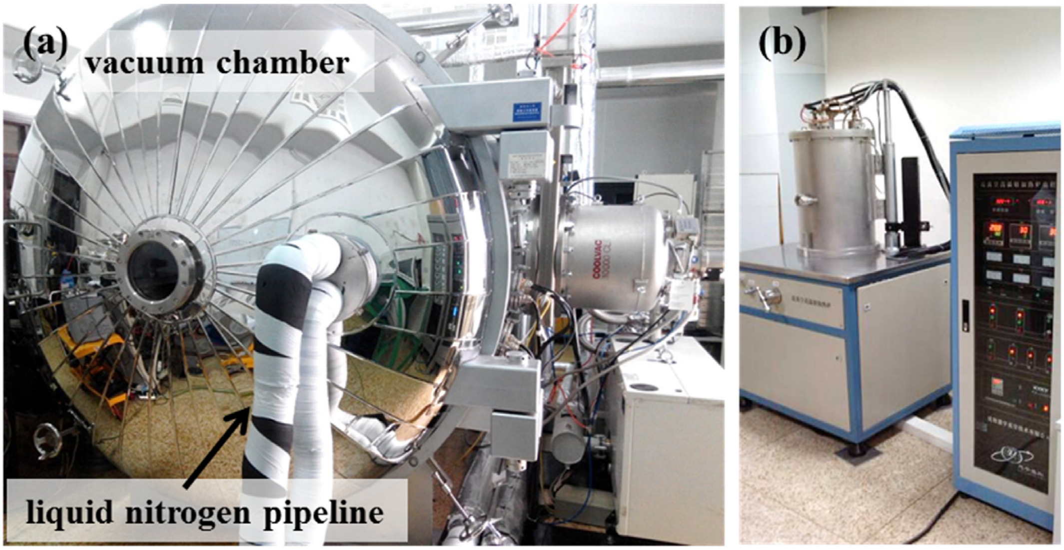

Fig. 2 – Low temperature environment vacuum chamber (a), High temperature molybdenum annealing vacuum furnace (b).

temperature respectively, and the high-temperature heating sheets were attached to the back of the fixture. The temperature control system controlled the heating power in real time through the sample temperature fed back by the temperature sensor, so as to realize the precise control of the sample temperature [19], as shown in Fig. 1(b). In the in-situ experiment, the sample that in the fixture was put on the resistance test stage, then put into the low temperature environment vacuum chamber as shown in Fig. 2(a), and the leadwires were led out to the resistance meter and temperature control system outside. The resistance of samples was measured for real-time in the temperature range from −20 °C to 80 °C, which was the operating temperature of photon-counting detector controlled by temperature controlled system of payload in space.

The ex-situ electrical tests mean that the resistance of samples was measured at room temperature (RT, 22 °C) after the samples being treated at −190 °C, RT and 100 °C–600 °C with the step of 100 °C for an hour, respectively. As shown in Fig. 2 (a), the inner wall pipeline of low temperature environment vacuum chamber can be filled with liquid nitrogen to reduce the chamber temperature, and the lowest temperature can reach −190 °C. As shown in Fig. 2 (b), the high temperature molybdenum annealing vacuum furnace can be heated up to maximum 1000 °C. Both of the chambers are vacuumed above 1×10^{-4} Pa when testing.

2.3. In-situ and ex-situ stress test

The surface shape of each substrate was measured by zygo GPI XP/D interferometer before and after coating. The sample's curvature was calculated by the PV value. Then, the film stress can be calculated by Stoney equation [20]:

$$\delta = \frac{E_s}{6(1-\upsilon_s)} \frac{t_s^2}{t_f} \left(\frac{1}{R} - \frac{1}{R_0} \right) \tag{1}$$

where δ is the stress of the Ge film, $E_s = 71.7$ Gpa is the Young's modulus, $\upsilon_s = 0.17$ is the Poisson's ratio, t_s is the thickness of the substrate, t_f is the thickness of film, and R_0 and R are the curvatures of the substrate before and after film deposition respectively.

In the in-situ stress tests, we also used the temperature controlled system mentioned in section 2.2. As is shown in Fig. 3, the zygo GPI XP/D interferometer can be only operating in air, so the experimental equipment can just keep the sample in the temperature range from 22 °C to 130 °C without temperature under 0 °C. The stress was tested at different temperature by zygo GPI XP/D interferometer. The condition of ex-situ stress tests was the same with the ex-situ electrical tests as mentioned in section 2.2. The samples were treated at −190 °C, RT and 100 °C–600 °C with the step of 100 °C for an hour, respectively. And the stress was calculated after the thermal treatment.

3. Electrical test results and discussion

3.1. In-situ electrical test

Ge film is the charge induced layer of the photon-counting imaging detector, and its electrical properties have a great influence on the optical imagers in Space misson [1,2,4]. In our earlier studies [10], the resistance and thickness of Ge films have dramatic influence on the resolution of imaging system. The imaging performance is excellent and stable as the resistance of Ge film is within the range from 100 MΩ/□ to 1G/Ω. The image quality becomes worse when the resistance is out of this range more. For instance, if the resistance of Ge film is 20KΩ/□ or 2G/Ω, the resolution will be greatly reduced. In the in-situ resistance tests of our study, as shown in Fig. 4, the resistance of Ge film decreases linearly from 520 to 124 MΩ/□ when the temperature ranges from −20 to 80 °C. This range of resistance variation is within the optimum imaging range of resistance. Therefore, the in-situ resistance studies demonstrate that the resolution of imaging system can't be affected by the

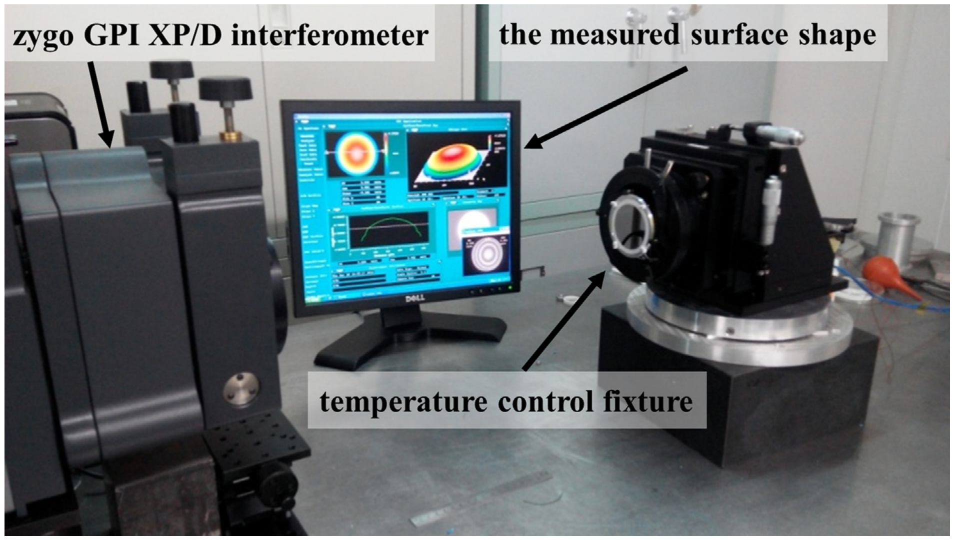

Fig. 3 – Thin film residual stress testing device.

resistance variation when the photon-counting detector of payload operating in the space temperature environment.

In addition, through the slope of the linear portion of the resistance–temperature curve in Fig. 4, the TCR (temperature coefficient of resistance) can be calculated from Eq. (2):

$$TCR = \frac{1}{R}\frac{dR}{dT} \tag{2}$$

The TCR of Ge films is determined to be −9480 ppm/°C.

Generally, the detector temperature can be controlled between −20 and 80 °C with the temperature control system of the payload. However, once the temperature control system is broken or out of control, the Ge film will be exposed to the extreme space temperature environment ranging from −180 °C to 150 °C. Therefore, it's necessary to investigate the limited temperature that the Ge film can adapt to, and the intrinsic mechanism of performances variation of the Ge film. However, the temperature control range of the in-situ experimental system is limited, so we perform the ex-situ electrical tests, which temperature range can reach from −190 °C to 600 °C.

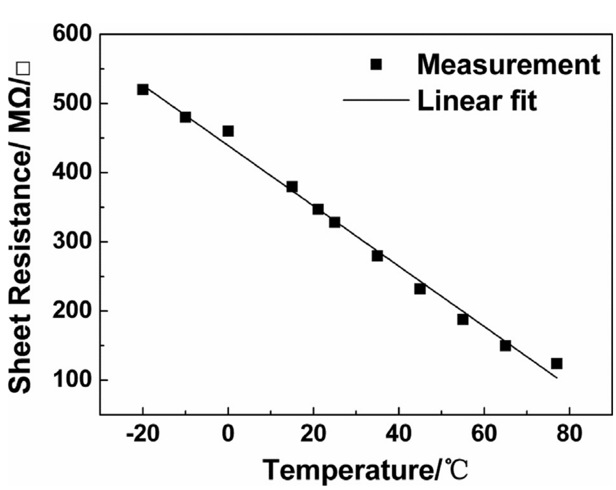

Fig. 4 – The in-situ sheet resistance of the Ge films as a function of temperature.

3.2. *Ex-situ electrical test*

We performed the ex-situ electrical test to research into limited temperature adaptability of Ge film. Figure 5 (a) shows the change of the sheet resistance of Ge films in room temperature after different temperature annealing. The curve in the figure shows that low temperature treatment (−190 °C) has no effect on the resistance, and the resistance increases continuously from 9.8 MΩ/□ to 502 MΩ/□ in the temperature range from 100 °C to 500 °C. The increase of resistance is due to the changed optical band gap after annealing [21–23]. The optical band gap is determined by the disorder degree and the density of defects. During the annealing, the unsaturated defects bonds of the materials are gradually annealed out, and a large number of saturated bonds generate. Therefore, as shown in Fig. 5 (b), the optical band gap of Ge film increases from 0.82 to 0.96 eV in the temperature range from RT to 500 °C, and the resistance increases along with it. After this increase, the resistance of the sample drops significantly by 3 orders to a minimum value of 30 KΩ/□ at 600 °C, and the optical band gap also rapidly decrease to 0.56 eV at this time. The Ge film fails due to the resistance reducing to 30 KΩ/□. So it is concluded that the limited temperature of Ge film is 500 °C. The sharp decrease of resistance is attributed to the transformation from amorphous to crystalline of Ge film, which is demonstrated by the XRD patterns in Fig. 6.

As can be seen from Fig. 6, the wide band peak around 27° is the non-crystalline diffraction peak of Ge film. The absence of sharp structural peaks of XRD spectra between 400 °C and −190 °C indicates the film is amorphous before 400 °C, and low temperature (−190 °C) can't change the structure of Ge films. When the film is annealed at 600 °C, the non-crystalline peak diminishes and the sharp peaks of Ge [012], Ge [220], Ge [310] at 31°, 39° and 46° appear, indicating the Ge film is completely crystalline. The experimental results obtained are in

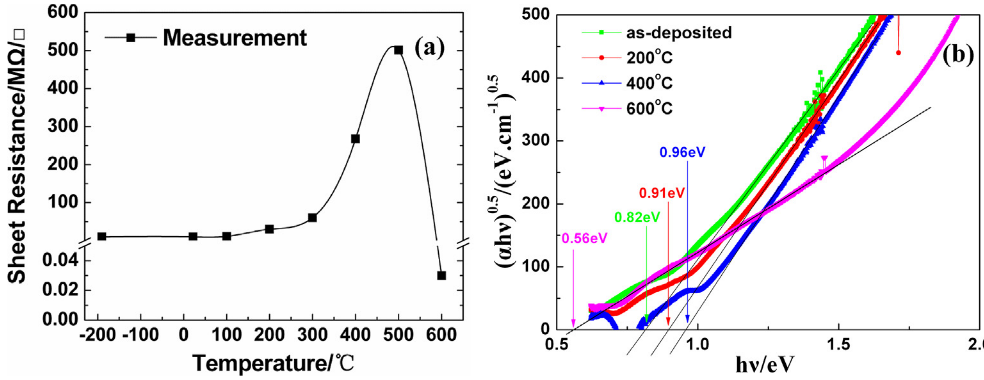

Fig. 5 – The sheet resistance of the Ge films (a) and the optical band gap of Ge films (b) against the annealing temperature.

accordance with the decrease of sheet resistance at 600 °C mentioned above. It also shows that the crystalline temperature of DC magnetron sputtering Ge film is 600 °C.

3.3. *Adaptability enhancement of Ge film*

According to the analysis of the mechanism of performances change of Ge film with temperature in section 3.2, it can be seen that the Ge film resistance changes only when the annealing temperature increases the optical band gap of the film. Therefore, the resistance of annealed Ge film doesn't change in the next annealing with a lower annealing temperature because the lower temperature can't provide enough active energy to enhance the optical band gap. To enhance the adaptability of Ge film, the Ge films used in image payload of spacecraft have been annealed at a high temperature above 150 °C. The temperature of space such as lunar surface ranges from −180 °C to 150 °C. Therefore, the temperature environment of space environment can't change the resistance of the Ge films we prepared. The sheet resistance of the Ge films only varies with the different operating temperature, but the changes we demonstrated in section 3.2 has no effect on the imaging performance. The high-adaptability Ge film we prepared is promising in the optical payload of spacecraft.

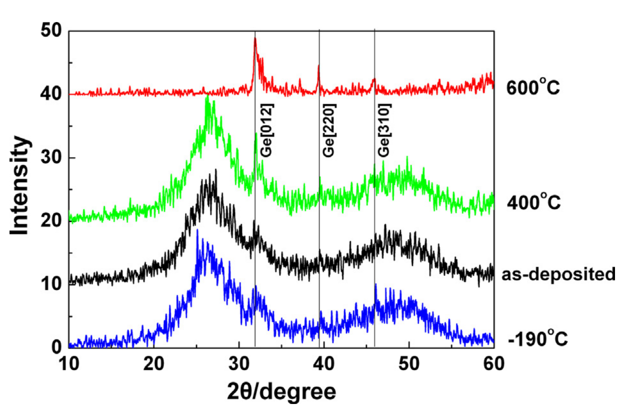

Fig. 6 – XRD patterns of as-deposited and annealed Ge films.

4. Stress test results and discussion

4.1. *Residual stress evolution as measured during in-situ annealing*

Large stress always results in folding and cracking of the Ge film, and it is strongly affected by temperature. In-situ stress measurements of Ge films were performed using the method described in Section 2.3. The stress was measured continuously during the thermal cycle that heating up from RT to 130 °C and then cooling down to RT. As can be seen in Fig. 7, the initial stress of Ge film is −470 Mpa, and it decreases linearly with increasing temperature due to the mismatch of the thermal expansion coefficient between the substrate and the Ge film. When the temperature beyond 60 °C, the stress increases abruptly, and gradually releases to −344 Mpa at 130 °C. The sudden change at 60 °C can be related to the onset of the vapor degassing and defects annihilating [24]. During the cooling process, the stress firstly increases to −257 Mpa, then decreases to −364 Mpa at RT. The stress of Ge films releases 106Mpa in a thermal cycle. This releases of stress are beneficial for the Ge film. And the induced surface shape change of

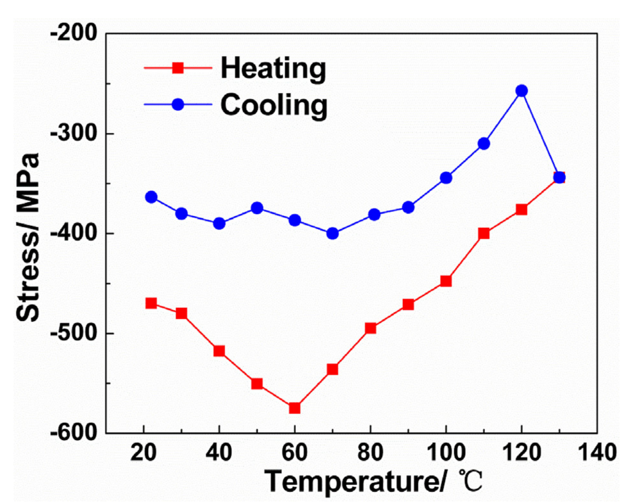

Fig. 7 – Ex-situ stress of Ge films as a function of temperature.

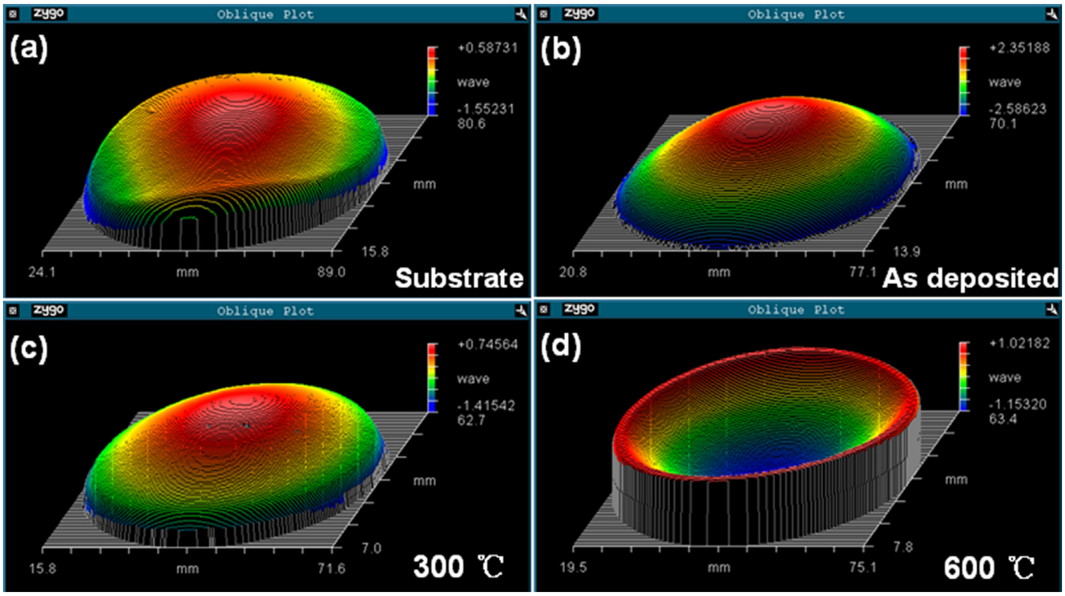

Fig. 8 – Surface shape of Ge film measured by interferometer as a function of temperature (a) substrate, (b) as-deposited, (c) 300 °C, (d) 600 °C.

position sensitive anode is acceptable. Therefore, the in-situ stress studies show that the stress and the morphology of Ge film did not change significantly during operating temperature from 22 °C to 130 °C. We also investigated the ex-situ stress in the temperature range from 190 °C to 600 °C to study the limited adaptability and mechanism of stress variation.

4.2. *Residual stress evolution as measured after ex-situ annealing*

Figure 8 shows the surface shape of the substrate, as deposited, and the Ge film annealed at different temperature. The corresponding stress is given in Fig. 9, the initial stress of the sample is −461 Mpa as shown in Fig. 9 and makes the surface shape like a sphere in Fig. 8 (b). The stress only increase 3 Mpa at −190 °C compared with the initial stress of Ge at RT, indicating that low temperature has no effect on the stress of the Ge films. The stress increases almost linearly when the annealing temperature increases from 100 to 600 °C. Meanwhile, the stress transforms from compressive to tensile and achieves zero at 300 °C during when the surface shape returns to the state of the uncoated substrate as can be seen in Fig. 8 (a) and (c). At 600 °C, the stress achieves 504 Mpa in Fig. 9 and the surface shape concaves downward in Fig. 8(d). Thus, there is no deformation of the position sensitive anode with 300 °C annealing due to absolutely releasing of the stress.

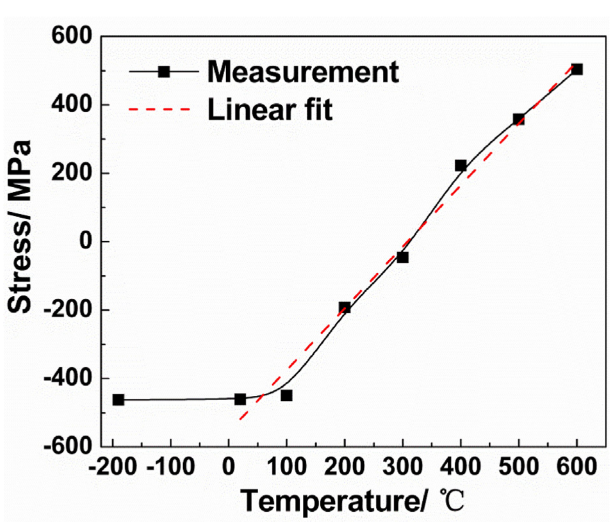

Fig. 9 – Ex-situ stress of Ge films as a function of temperature.

The variation of stress during annealing is resulted from the grain growth and nucleation phenomena. The energy offered by annealing makes the crystallite grains grow and the grain boundary area decrease, which is illustrated by Fig. 6 in section 3.2. The reduction of grain boundary causes film horizontal contraction, which leads to the increase of tensile stress [25]. Moreover, the reduction of defect, voids and water during annealing can also densify the films and favor the increase of tensile stress [26]. Therefore, as can be seen in Fig. 9, the stress of Ge film transforms from compressive to tensile along with the increasing annealing temperature.

The ex-situ stress tests show that the intrinsic stress increase from −464 Mpa to 504 Mpa in the temperature from −190 °C to 600 °C. To investigate the stress at which temperature will results in the folds and cracks in the film, the surface morphology was observed.

4.3. *Surface morphology*

The surface morphology was observed by optical microscope and surface profilometer as shown in Fig. 10. In the temperature range from 190 °C to 500 °C, the surface morphology

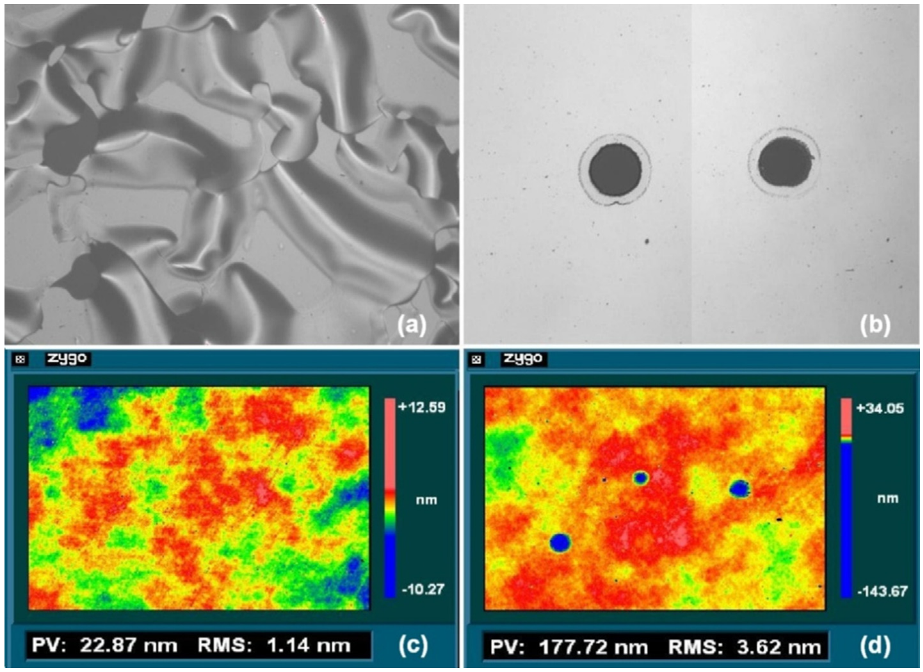

Fig. 10 – (a), (b) Surface morphology image of Ge film after 600 °C annealing measured by 10 × optical microscope. (c) Surface roughness measuring pattern of as-deposited Ge film. (d) Surface roughness measuring pattern of Ge film after 600 °C annealing.

didn't change significantly. At 600 °C, the stress increases to 504 Mpa, and such high stress gives rise to the partial folding and cracking on the edge of the Ge film samples as shown in Fig. 10 (a). Moreover, a large number of 30–150 ηm voids are found after the 600 °C annealing, as shown in Fig. 10 (b) and (d). Simultaneously, the root mean square (RMS) of roughness increases from 1.14 nm to 3.62 nm after 600 °C annealing as shown in Fig. 10 (c) and (d). The Ge film is cracked because of the over large stress after 600 °C annealing. Therefore, the limited temperature is 500 °C, which is far away from the high temperature in space.

Based on the ex-situ and in-situ stress test shown in section 4.1, 4.2 and 4.3. It is concluded that the low temperature of −190 °C in space has little effect on the stress of Ge film and the high temperature of 300 °C is helpful to the absolutely releasing of the Ge film stress.

5. Summary

We have studied the resistance, structure, and stress of Ge film at low and high temperature environments both in-situ and ex-situ.

The in-situ electrical tests show that the resistance decreases linearly from 520 to 124 MΩ/□ with temperature from −20 to 80 °C, and the changing resistance in this range has no influence on the resolution of imaging system. In the in-situ stress test, the sample's initial stress is −470 Mpa, and it decreases linearly to the maximum of −574 MPa when heating up to 60 °C, then gradually releases to −363 Mpa after thermal cycling between RT and 130 °C. It shows that the stress releases after each thermal cycle. And the induced surface shape deformation of position sensitive anode is acceptable. The in-situ measurements demonstrate that the Ge film is able to work effectively at different temperatures of optical payload in space.

In the ex-situ test, we investigated the limited adaptability and mechanism of performances variation of Ge film in temperature range of −190 °C–600 °C. When the Ge film was treated by annealing temperature of 600 °C, the resistance dropped abruptly to 30 KΩ/□ due to the crystallization. Meanwhile, the stress increasing to 504Mpa resulted to the crack of Ge film at 600 °C. Therefore, it can be concluded that the limited temperature that the Ge film can adapt to is 500 °C. The Ge film with high environmental adaptability and low stress we prepared is promising in the space observation, and will play a significant role in aerospace application.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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