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Study on properties of Al film on CFRP after cryogenic-thermal cycling

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Abstract

Al film on CFRP has been tested by cryogenic-thermal cycling according to the especial condition of space. Properties of aluminum film have been characterized by electron pull apparatus, XRD and SEM. The result shows that the adhesion of Al film increases slowly at early stage of cryogenic-thermal cycling. When the times of cryogenic-thermal cycling exceed 50, the adhesion of Al film becomes stability, and then the adhesion of Al film decrease slowly when cycling times from 100 to 600. After 600 times, the adhesion of Al film becomes stability again. The microcrack appears on the surface of Al film after 50 times, and the amounts of microcrack increase and microcrack is coarsening versus times of cryogenic-thermal cycling. The structure of Al film is changing slowly during cryogenic-thermal cycling.

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1. Introduction

The CFRP (Carbon fiber reinforce polymer) is widely applied in space for their relatively high specific strength and stiffness, and the reduction in weight leads to significant cost savings in launching. CFRP will be used widely more and more ^[1, 2] in future because of requirement of "Gossamer Spacecraft" and improvement in the technology of CFRP. However, Total mass loss (TML) of CFRP would make the spacecraft structure hurt, and the surface of sensitive spacecraft devices would be polluted by collected volatile condensed material (CVCM) of CFRP when CFRP exposing in space^[3], on the other side, most satellite antenna is made of CFRP^[4], but electric performance of the material is not enough for the need of high frequency and high power to comsat in future. Metallization on CFRP can improve protection and electronic performance for CFRP^[5, 6].

Al film has excellent property, such as low density, low resistivity and rapid passivation, as aluminum^[7]. It has been used for metallization on CFRP widely. In this work, the properties of Al film on CFRP are studied after cryogenic-thermal cycling test.

2. Specimens

Specimens consist of Al film and sheet made of CRPF. Carbon fiber is M40 and epoxy resin is E-51. The purity of Al target exceeds 99.9%. Al film is deposited on these sheets by vacuum arc technology. Thickness of Al film is 1µm.

The vacuum arc technology has advantage including high ionization ^[8], high energy of particle and high deposition rate ,etc. the properties of Al film deposited by vacuum arc technology are same as bulk aluminum.

3. experiment

The systems of cryogenic-thermal cycling include a heat preservation container and a self-regulation temperature oven. The specimen, is packed with 0.2 μ m aluminum foil in order to protecting from liquid nitrogen infiltrating, is cryogenic cooling in liquid nitrogen which is filled in the heat preservation container. The temperature of the oven is 400°C, it can make the temperature of specimen to go up quickly. Fig.1 shows the process of a single cryogenic-thermal cycling test. Adhesion of Al film is tested per 20 times in former 100 times, and per 100 times after 100 times, and the surface SEM image was taken when the adhesion testing.



Fig.1. the process of a single cryogenic- thermal cycling

4. Results and discussion

4.1 analysis and test before experiment

Cryogenic- thermal cycling is a way to test aircraft life on ground. Al film is analyzed with D/max 240OX XRD apparatus made by Japan. Fig 2 shows XRD patterns of Al film before testing, the range of incident angle is between 30° and 100°. The target is Cu Ka, the tube voltage is 40kV and the electric current is 60 mA.



Fig.2. XRD patterns of Al film before cryogenic- thermal cycling test

Fig 3 and fig 4 show Al film surface and profile image before cryogenic- thermal cycling test. The type of SEM (scanning electron microscope) is JSM-5600LV made by Japan in The Electron Optics Company.



Fig.3.SEM image of specimen surface

Fig.4.SEM image of specimen profile

There are lots of micro-particles on Al film surface for no filter system when the film deposition process, and the microcracks were not observed, as show in Fig. 3. The Fig. 4 shows that the Al film is column structure and the Al film is well-crystallized as Al crystal.

4.2 Adhesion analysis

The film adhesion of Al films had been tested per 20 times in former 100 times of cryogenic-thermal cycling, and the adhesion versus times of cryogenic-thermal cycling is showed in Fig. 5.



Fig.5. the film adhesion versus the times of cryogenic-thermal cycling in former100

The adhesion of Al films increase slowly when the times of cryogenic-thermal cycling increase from 0 to 50, as show in Fig.5, and the adhesion keeps constant approximately as the times increase from 50 to 100. Thompson D F reported that[9] the CFRP would solidify in later stage when it experienced thermal cycling test, and the boundary diffusion is very activity between Al film and substrates in former 50 times of cryogenic-thermal cycling. The boundary diffusion can penetrate and anchor between substrates and Al film, and the adhesion increase.

Topographic image of Al films has been analyzed by JSM-5600LV SEM. We found that the virgin surface and the surface of 50 times cryogenic-thermal cycling was same. The crack appears when the times of cryogenic-thermal cycling increase and the topographic image of after 50 times been show in Fig.6.



Fig.6. SEM image of specimen surface after 50 times

There are 2 conflict factor affect the adhesion of Al films, the harmful effects is the appearance of microcracks, and the other is the CFRP solidify phenomena when it experienced cryogenic-thermal cycling can make the adhesion strengthened. So the adhesion of Al film keeps constant when the times of cryogenic-thermal cycling increased from 50 to 100.

The adhesion versus the times of cryogenic-thermal cycling between 100 and 1000 has been shown in Fig.7. The film adhesion decrease dramatic from 8.2 to 6.2 N/mm² when cryogenic-thermal times increase from 100 to 600. And the adhesion decrease slowly from 6.2 to $6N/mm^2$ when cryogenic-thermal cycle times increase to 1000.



Fig.7 The adhesion versus the times between 100 and 1000

The CFRP solidify phenomena would become unconspicuous when the times of cryogenic-thermal cycling increased from 100 to 600, and the other harmful factor would plays the most part. Therefore, the adhesion decreases when the times of cryogenic-thermal cycling increase.

The topographic image of Al films with different times of cryogenic-thermal cycling have been show in Fig. 8.



Fig.8. different times SEM image of surface of specimens

From SEM images of different cryogenic-thermal cycling times, we found that the results were: (1) the numbers of microcracks were increase when cryogenic-thermal cycling times increase, (2) the volume of microcracks is become larger when the times increase, (3) microcracks grow along the direction of microcrack axes, and the most microcracks run through the surface of specimen, (4) the density of microcracks become uniformity when the times more 600. The adhesion of Al film decrease slowly when cryogenic-thermal cycling times increase from 100 to 600, but it become constant after 600 times for the density of Microcracks become uniformity. *4.3 Microstructure of Al film*

The properties of metal films are related to its structure, the Al film XRD patterns with different cryogenicthermal cycling times have been show in Fig. 9.



Fig.9. XRD patterns of Al film with different cryogenic-thermal cycling times

Fig. 9 shows typical XRD patterns gained from the surface of Al films. In Fig. 9a for virgin surface of Al film, it mainly consistent with standard patterns for fcc Al, as show in Fig. 9c. Fig. 9b is the XRD patterns after 1000 times cryogenic-thermal cycling, all the peaks in Fig. 9b are consistent with Fig. 9c but for half width of (111) peak decrease. This indicated that the grain in film become large after cryogenic-thermal cycling test. The studying of

Thompson G B and Anand T J S ^[10, 11] show that the crystallization strengthens after the specimens been heat treatment. In our case, the high temperature is 125° C- 130° C and holding time is 8 min, it is not high and long enough for crystallization completely during cryogenic-thermal cycling.

5. conclusions

Al film on CFRP made by vacuum arc technology has good properties as bulk aluminium. The structure, adhesion and surface feature of Al film on CFRP has been investigated when the specimens experienced cryogenic-thermal cycling test, and the results included:

(1) The Al films crystallized again when it suffered cryogenic-thermal cycling test.

(2) The microcrack appears on the surface of Al film after 50 times of cryogenic-thermal cycling. And when the times of cryogenic-thermal cycling increased from 100 to 600, the density of microcrack increased, the microcrack grew along the direction of microcrack axes, and the volume of microcrack became larger too. But Microcracks become saturation when the times increased from 600to 1000.

(3) The adhesion of Al films increase slowly when the times of cryogenic-thermal cycling increase from 0 to 50, then it decrease from 8.2 to 6.2 N/mm² when times increase from 100 to 600, and in the end, the film adhesion become constant when times increased from 600 to 1000.

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