Strengthening of polyimide composites by SiC whiskers

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Abstract. To improve the strength of the polyimide (PI), composites with SiC whiskers doping in the polyimide matrix were prepared. In order to avoid the conglobation of the whiskers, SiC whiskers pre-dispersed in the ethanol. SiC whiskers/PI composites were prepared by high-speed mechanical stirring. The resulting composites were analyzed in terms of dispersion and deposition of whiskers, mechanical performance by means of scanning electron microscope (SEM) and tensile testing respectively.TheSiC whiskers uniformly distributed in the PI matrix. And there was nearly no SiC whiskers deposition.TheSiC whiskers/PI composites showed better mechanical performance than pure PI.

Introduction

Polyimide (PI) film having a high strength, high toughness, wear resistance, high temperature, corrosion and other special properties, can meet the light, thin, short, small design requirements. It is a high temperature insulation material with competitive advantages. After several decades of development, it has become an important material of electronic, electrical products, ,widely used in the field of electronic soft boards, semiconductor packages, photovoltaic (solar) energy, liquid crystal display and so on[1-5]. However, PI belonging to organic material has some limitations. Organic material is easy to form deformation when undertaking load due to its poor mechanical properties [6]. Most polymers have low thermal conductivity and large coefficient of thermal expansion [7]. In order to overcome the limitations,various fiber reinforcements, such as glass, carbon, natural fiber, liquid crystalline polymer, are frequently added to polyimide[8].In general, the incorporation of discontinuous fibers into polyimide leads to a dramatic increase in stiffness, strength and fracture toughness[9]. Among all the enhancements, Whisker, with large length-radius-ratio, is a kind of short andfiber-shaped single crystal that arranged in a highly ordered atomic structure [10].In order to improve the comprehensive performance of PI, we proposed SiC whiskers reinforced polyimide composites. The improvement of mechanical properties of the composites is attributed to the effective load transfer from the matrix to the enhancement. The dispersion of the SiC whiskers in the matrix is important to the the load transfer. We adopt the pre-dispersion and high-speed mechanical stirring method to improve the dispersion condition of the SiCwhiskers. TheSiC whiskers which were disorderly distributed in the polyimide matrix can enhance the mechanical properties of the PI.

Experimental

2.1. Materials

The polyimide, a kind of polymer (ZKPI-30511G, Beijing, China), was chosen as the matrix of the composites.The polyimide has a good corrosion resistance, dielectric property, stable chemical property and good insulation property. Among all the polymers, polyimide (PI) has a respectively lower coefficient and a higher degree of thermal stability.Compared with the other polymer such as epoxy resin, the coefficient of polyimide is much smaller. Because its coefficient of thermal expansion (CTE) is close to copper over a fairly wide temperature range, the mechanical stresses induced in copper during thermal excursions are minimized[11].Whiskers have the following advantages: large length-radius-ratio,good mechanical properties and stable chemical properties. Compared with other whiskers, SiC have higher strength, stiffness and higher temperature stability.

SiC whiskers(β-SiC， Changsha Sinet Advanced Materials Co. Ltd.） was chosen as the reinforcements of the composites.

2.2 Preparation of SiC whiskers/PI composite films

The composites of SiC whiskers and polyimide were prepared by high-speed mechanical stirring. Firstly, adding a certain quality of SiC whiskers to absolute ethanol. Then the SiC whiskers can realize a good dispersion in the ethanol through high speed mechanical stirring (500-1000r/min), which usually takes about1-2 hours. Under the condition of high-speed mechanical stirring, slowly pouring polyimide in the above SiC whiskers/ethanol. The high-speed string need to continue until most ethanol have volatilized. It is better that the viscosity of the polyimide can be enhanced. In this way, the SiC whiskers can be fixed in the polyimide matrix, which can improve the dispersion of the whiskers and avoid the deposition. Through this method, we can get the composites with good whiskers dispersion without damaging morphology of the whiskers. The above composites were spin coated on a glass wafer which will be dried through programmed heating, and the highest temperature is 250 ℃. Then SiC whiskers/PI composite films with thickness 30-50um were obtained.

2.3 Characterization

2.3.1 The choice of dispensing agents

The surface profile of the SiC whiskers was observed by scanning electronic microscope (SEM). Firstly, the SiC whiskers dispersed in the ethanol/ acetone by high-speed mechanical stirring method. Then the mixture with SiC whiskers uniformly dispersed in the ethanol was coated on the glass wafer, dry at room temperature in the fume hood. A layer of gold was sprayed over the surface before observation. The dispersion conditions of SiC whiskers in different dispersing agents (ethanol /acetone) were shown inFig. 1. The SiC whiskers dispersed in the ethanol uniformly as shown in Fig. 1(a), while the SiC whiskers dispersed in the acetone had obvious aggregation as shown in Fig. 1(b). The aggregation of the whiskers is not good for the properties improvement of the composites. So we chose the ethanol as the dispersing agent.

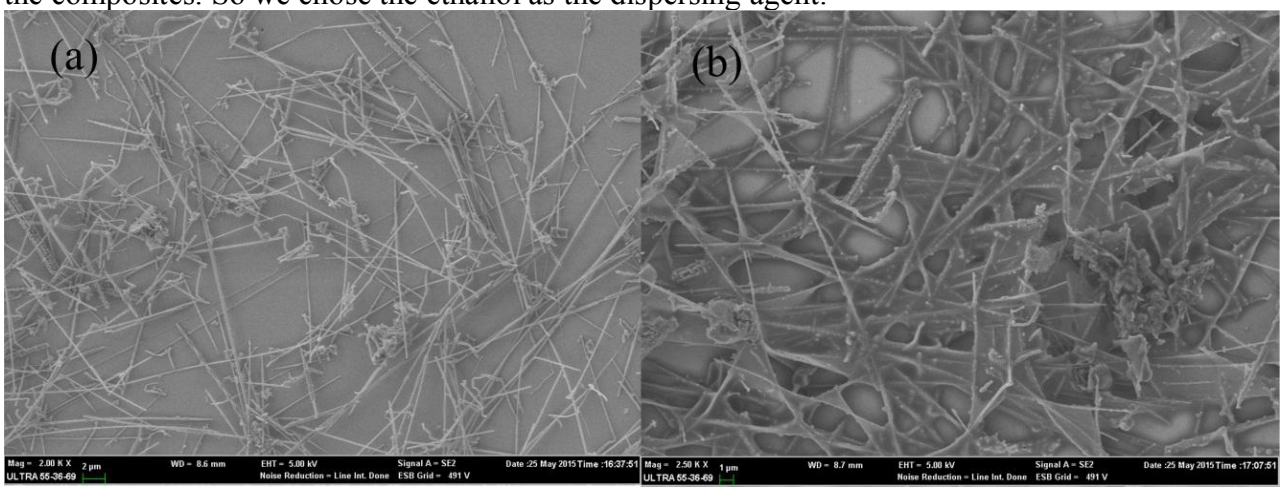

Fig. 1. The dispersion conditions of SiC whiskers in ethanol(a) and acetone(b)

2.3.2 Dispersion of SiCwhiskers

The enhancement of mechanical properties of SiCwhiskers/PI composites is attributed to the effective load transfer and the good dispersion of SiC whiskers. So the good dispersion of SiC whiskers is important. The dispersion of whiskers in the polyimide matrix can be observed by 3D Super Digital Microscope as shown inFig. 2. It revealed that the SiC whiskers uniformly dispersed in the matrix and formed a network structure. These network structures,which can realize the effective load transfer from the PI matrix to the SiC whiskers enhancement, greatly enhance the strength of the composites. The SEM micrographs of the composites' fracture morphology wereshown inFig. 3.The SEM micrographs of dispersion SiC whiskers in the PI matrix. From the fracture morphology, we can see that the SiC whiskers embedded in the matrix and there were no obvious aggregation and deposition. The uniformly distributed SiC whiskers can realize the

effective load transfer and reduce the stress concentration. Good dispersion conditions of the SiC whiskers can effectively improve the mechanical properties of the composites.

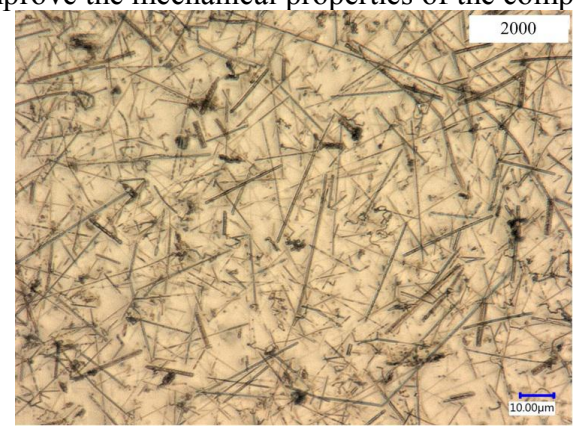

Fig. 2. The dispersion of SiC whiskers in the PI matrix

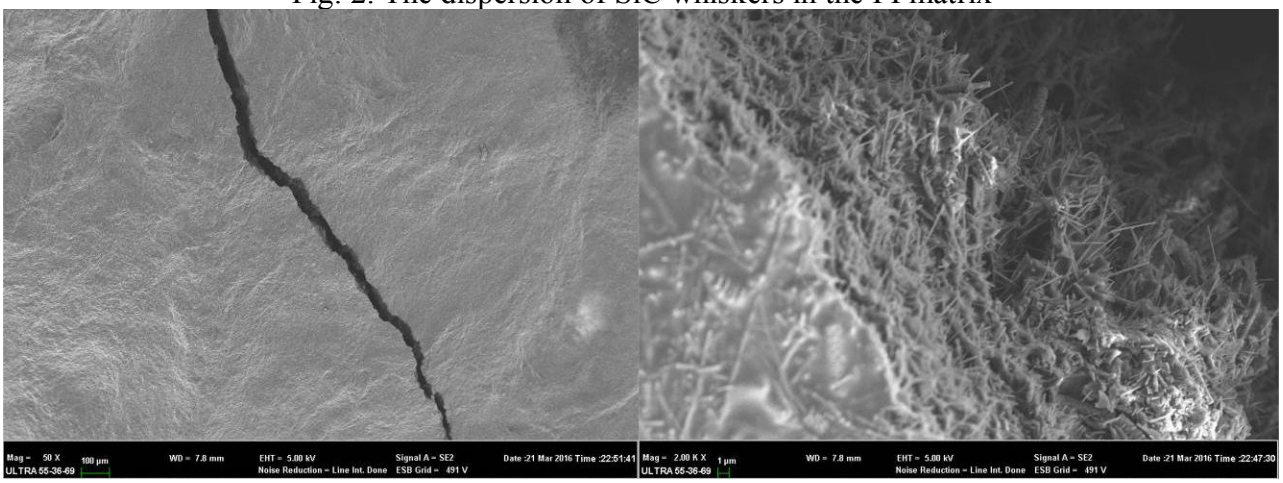

Fig. 3. The SEM micrographs of dispersion SiC whiskers in the PI matrix

2.3.3 Mechanical performance

The mechanical performance of SiCwhiskers/PI composites had been analyzed by Dynamic Mechanical Analyzer (DMA-Q800， TA Instruments Inc, USA). The samples were tested at room temperature. The force applied on the sample was from 0N to 18N, and the rate of force increase was 0.5N/min. The length, width and thickness of sample is 25mm, 2mm, 0.03mm respectively. We chose pure PI,2%, 4%wt and 5%SiC whiskers/PI composites as the samples and analyzed the mechanical properties respectively. The test results were shown inFig. 4.The test results revealed that the introduction of the SiC whiskers improved the strength of the composites.

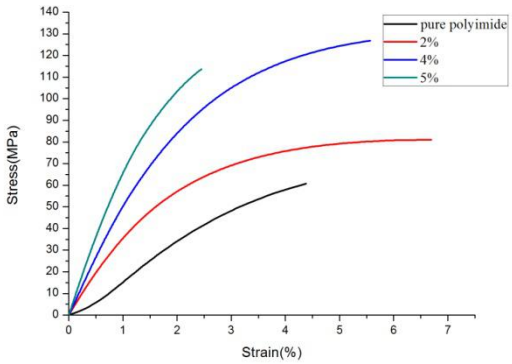

Fig. 4.Stress-Strain curves of SiC whiskers/PI composites

Conclusions

In this paper, SiC whiskers/PI composites were prepared by high-speed mechanical stirring. The dispersion of the SiC whiskers in the PI matrix was analyzed by SEM and 3D Super Digital Microscope. The composites prepared through the above method had good dispersion conditions. DMA was adopted. The introduction of the SiC whiskers can improve the mechanical performance due to the effective load transfer from the matrix to the uniformly dispersed whiskers. The improvement of mechanical performance can expand the application range of the composites.

Acknowledgments

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References

[1]Lee W J, Kim Y B. Adhesion and interfacial characteristics of metal/PI composite film modified by O 2 ion beam[J]. Thin Solid Films, 2008, 517(3): 1191-1194.

[2]Wang Z, Lu J J, Li Y, et al. Studies on thermal and mechanical properties of PI/SiO 2 nanocomposite films at low temperature[J]. Composites Part A: Applied Science and Manufacturing, 2006, 37(1): 74-79.

[3]Yano K, Usuki A, Okada A, et al. Synthesis and properties of polyimide–clay hybrid[J]. Journal of Polymer Science Part A: Polymer Chemistry, 1993, 31(10): 2493-2498.

[4]Ektessabi A M, Hakamata S. XPS study of ion beam modified polyimide films[J]. Thin Solid Films, 2000, 377: 621-625.

[5]Yamaguchi H, Aoki F. Metal film/aromatic polyimide film laminate: U.S. Patent 6,605,366[P]. 2003-8-12.

[6]Govindaraju A, Chakraborty A, Luo C. Reinforcement of PDMS masters using SU-8 truss structures[J]. Journal of Micromechanics and Microengineering, 2005, 15(6): 1303.

[7]Lee G W, Park M, Kim J, et al. Enhanced thermal conductivity of polymer composites filled with hybrid filler[J]. Composites Part A: Applied Science and Manufacturing, 2006, 37(5): 727-734.

[8]Tjong S C, Meng Y Z. Performance of potassium titanate whisker reinforced polyamide-6 composites[J]. Polymer, 1998, 39(22): 5461-5466.

[9]Hao X, Gai G, Lu F, et al. Dynamic mechanical properties of whisker/PA66 composites at high strain rates[J]. Polymer, 2005, 46(10): 3528-3534.

[10] Wen W, Luo B, Qin X, et al. Strengthening and toughening of poly (L-lactide) composites by surface modified MgOwhiskers[J]. Applied Surface Science, 2015, 332: 215-223.

[11] Bergstresser TR, Sallo JS. Adhesiveless copper on polyimide for flexible circuit, hdi and microvia applications. In: Proceedings of the IPC 5th annual flexible circuits national conference. Denver, CO; 1999.

