New composite of polyimide and a single crystal whisker of potassium titanate

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Abstract

Polyimide/potassium titanate whisker composite has been prepared by two methods of dry mixing and wet mixing. The properties of composites prepared by two methods have been compared. The composites of polyimide and whisker exhibit high strength, high heat resistance and good wear resistance, comparable to that of polyimide. The flexural modulus of composite is two times more than that of polyimide, reached 2GPa. The friction coefficient is 0.25, lower than that of polyimide. The surface of composite was observed by scanning electron micrography (SEM). It may be applied in advanced technical fields.

Key Words: Polyimide/Whisker/Potassium Titanate/Composite

Introduction

With development of science and technology, the synthetic materials have been widely applied in various industrial departments. Especially, it is required that the synthetic material exhibits high strength, superior wear resistance and high heat resistance in advanced industry. We have known that polyimide is super engineering plastic, exhibits good mechanical properties and high heat resistance. In order to increase strength and heat resistance, the polyimide is always reinforced with glass fiber and carbon fiber, but its wear resistance and processability decrease. The new single crystal whisker is used to improve these defects. It is different from glass fiber and carbon fiber. Its characteristics are not only to increase strength but also to improve dimensional accuracy, surface flatness and superior wear resistance, of those properties the greatest weakness of glass fiber and carbon fiber. The single crystal whisker of potassium titanate was selected, because it exhibits high strength, high modulus and a large aspect ratio. It has been combined with nylon, POM, PBT, PPO, PPS, PSF, PESF, PEI, and other types of thermoplastics.[1] We combined their characteristics of polyimide molding plastic and crystal whisker to prepare the composite of polyimide and crystal whisker of potassium titanate in order to increase strength, heat resistance and other physical properties. The results are reported here.

Experimental

Chemicals and Equipment

Polyimide powder was made by Shanghai Research Institute of Synthetic Resins. It is called YS-20 polyimide molding compound.[2] It was condensed with ODPA and ODA. The single crystal whisker of potassium titanate called TISMO was obtained from Japan Otsuka Chemical Co., Ltd.. Dynamic machanical analysis (DMA) experiments (the method of three points bending) were performed with a PE DMA7 instrument. Scanning electron micrographs (SEM) were taken with a Shimadzu EPMA-8705 QH2 instrument. Shimadzu Autograph AG-50KNE was used for testing mechanical properties by GB standard. The friction ceofficient was measured by Wear Test Machine MM200 made in Xuan Hua Material Test Machine Factory.

Preparation of Polyimide/Potassium Titanate Whisker Composite

There were two methods in mixing polyimide and potassium titanate whisker. They were dry and wet methods. The dry method was to mix polyimide powder and potassium titanate whisker directly by a mixer. The wet method was to add whisker to polyamidic acid solution and mix them at high speed stirring, then proceed chemical imidization by adding dehydrating agent. The moding compound was obtained by filtration, washing and dry.

The composite materials were obtained by compression molding in condition of high temperature and pressure.

400~420℃

添加量 30%

Result and Discussion

With development of advanced technology, the heat resistance synthetic materials have been required to use in aircraft and aerospace industries especially. We have known that polyimide molding plastics had always been used in those fields, because they exhibit high heat resistance, good mechanical and other physical properties. In general, the highest using temperature of polyimide molding plastics is about 250°C in long term. More Recently, the using temperature was required higher than 250°C in advanced technical fields, so polyimide composites reinforced with glass fiber and carbon fiber have been applied. The temperature of their heat deformation was increased, but their processability and wear resistance became bad. In order to improve these defects, we selected single crystal whisker of potassium titanate for reinforcement. The single crystal whisker of potassium titanate, whose chemical composition is K_2O-nTiO_2(n=6), has five features. They are microscopic reinforcement, superior wear resistance, outstanding dimensional stability, maximum surface smoothness and easy processing, because diameter of this whisker is about 0.5μm and it exhibits high strength, high modulus and a large aspect ratio. We knew the characteristics of TISMO whisker from table1 and scanning electron microscopic photograph of TISMO whisker was shown in Fig.1.[1]

We used two methods in mixing polyimide and whisker. They are dry method and wet method. The properties of polyimide composites made by two methods were compared. In the same time, the polyimide composite was compared with polyimide molding plastic (YS-20). The results were shown in table2. We found that tensile strength, flexural strength and hardness of polyimide composite prepared by wet mixing method were higher than that of polyimide molding plastic. But tensile strength of composite prepared by dry mixing method was lower than that of polyimide molding plastic. The impact strengths of the composites were lower than that of YS-20. It was similar as the general reinforced plastics with the inorganic fillers. This is because TISMO whisker exhibits high strength. It was obvious that the flexural modulus of polyimide composite made by wet mixing method was higher than that of dry mixing method, and they were higher than that of polyimide from dynamic thermomechanical analysis. It was shown in Fig. 2. We think that polyimide was deposited on the surface of whisker during chemical imidization. The interface adhesive force of polyimide and whisker was strengthened, and the mixing of polyimide powder and whisker was uniformed than that of dry method mixed mechanically. The scanning electron microscopic photographs of polyimide composites were shown in Fig.3. It was proved our above conjecture from the SEM. The whisker was separated with polyimide in the powder and molding compound of polyimide composite made by dry mixing method (Fig.3-a, 3-b). But the whisker was covered by polyimide and separation was not observed in the powder and molding compound made by wet mixing method (Fig.3-c.3-d). The reason lead to the difference of mechanical properties in polyimide composites made by dry mixing method and wet mixing method.

We found that there is little difference between the glass transition temperatures of polyimide composites and polyimide by dynamic thermomechanical analysis in Fig.4. But the reason of difference has not been known.

We are interesting that the wear resistance of polyimide composites was lower than that of YS-20 polyimide molding plastic. It is better than that of polyimide composites

reinforced by glass fiber. It may be applied in advanced technical fields required high strength and good wear resistance at high temperature.

Table 1. Characteristics of TISMO

Characteristics	Value
Color, Shape	White, Needle-like crystal
Chemical composition	K2O•6TiO2
Average whisker length	10-20μm
Diameter of whisker	0.2-0.5μm
Specific gravity	3.1-3.3
Mohs'hardness	4.0
Melting point	1300-1350°C
Tensile strength	Approx. 700kgf/mm2
Tensile modulus	Approx. 28000kgf/mm2
Electrical Characteristics	3.3×10^15Ωcm
Dielectric Characteristics	
ε	3.5-3.7
tanδ	0.06-0.09

ODPA/ODA 粉体

Table 2. The physical properties of polyimide composites and polyimide molding plastic

Property	YS-20	YS20WH/Dry	YS20WH/Wet
Density (g/cm3)	1.38	1.63	1.66
Hardness (Mpa)	169	258	264
Tensile Strength (Mpa)	120	97.2	129
Flexural Strength (Mpa)	131	151	167
Impact Strength (KJ/m2)	100	24.0	33.1
Tensile Modulus (Gpa)	1.63	1.23 ⟷	2.77
Friction Coefficient	0.30	0.27	0.25

Note: Tests were done by Chinese Standard (GB) in 23°C.

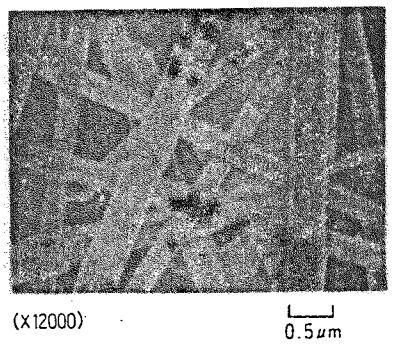


Fig. 1 Scanning electron microscopic photograph of TISMO

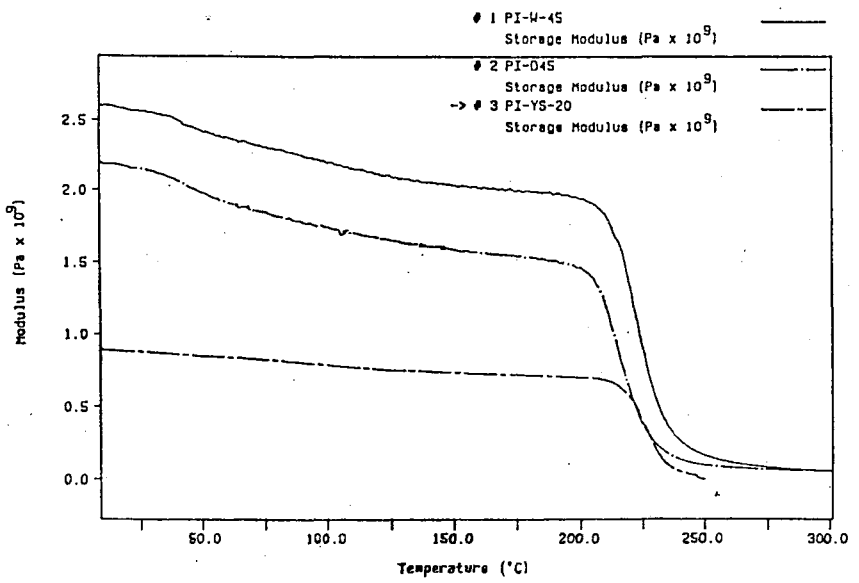
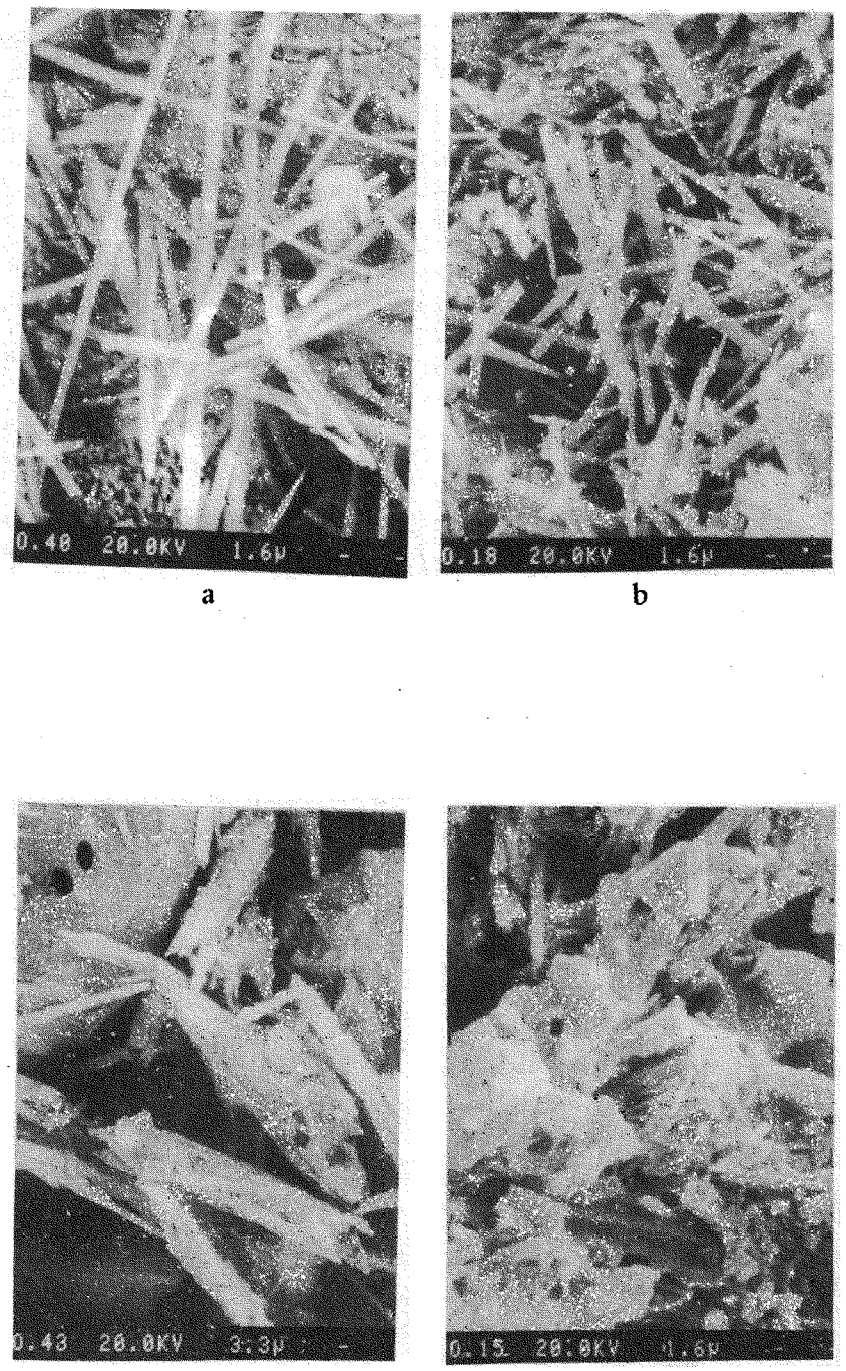


Fig. 2 The relationship of modulus and temperature in dynamic thermomechanical analysis of polyimide and composites
1, Composite made by wet method; 2, Composite made by dry method;
3, Polyimide YS-20; 10.0°C/min. in N2.



a b

c d

Fig. 3 Scanning electron micrographs of surface of polyimide composites
a, Powder made by dry method; b, Molding plastic made by dry method
c, Powder made by wet method; d, Molding plastic made by wet method

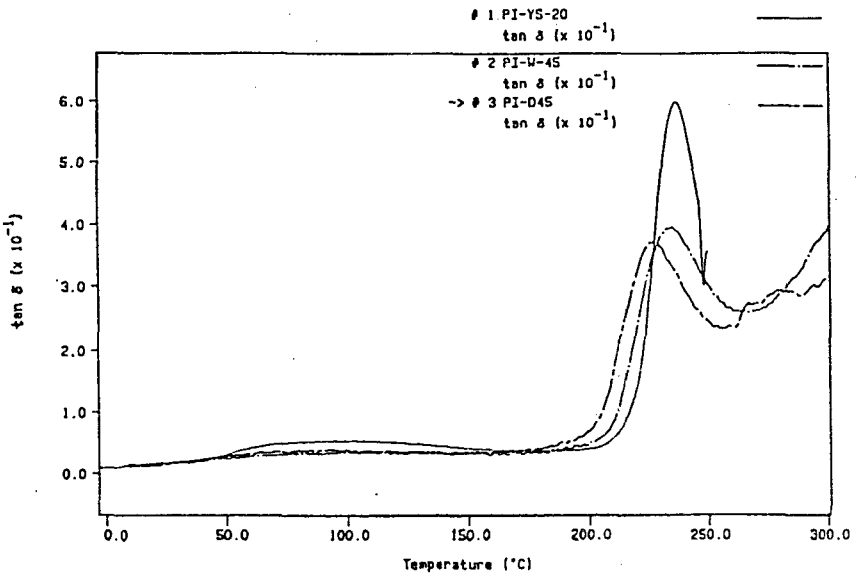


Fig. 4 The relationship of tanδ and temperature in dynamic thermomechanical analysis of polyimide and composites
1, Polyimide YS-20; 2, Composite made by wet method;
3, Composite made by dry method; 10.0°C/min. in N2.

Reference

1. Catalogue of TISMO POTICON from Otsuka Chemical Co., Ltd.
2. Report of polyimide YS-20 synthesis and properties from Shanghai Research Institute of Synthetic Resins.