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Novel polymer nanocomposite with silicon carbide nanoparticles

A.I. Wozniak, V.S. Ivanov, A.S. Yegorov*

Federal State Unitary Enterprise «State Scientific Research Institute of Chemical Reagents and High Purity Chemical Substances» (FSUE «IREA»), 3, Bogorodsky val, Moscow, 107076, Russia

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Polyimides rank among the most heat-resistant polymers and are widely used in high temperature plastics, adhesives, dielectrics, photoresists, nonlinear optical materials, membrane materials for separation, and Langmuir–Blodgett (LB) films, among others. While there is a variety of high temperature polyimides, there is a demand for utilizing these materials at higher and higher temperatures in oxidizing and aggressive environments. Therefore, we sought to use materials which do not oxidize to enhance properties of the polyimide composition maintaining polyimide weights and processing advantages. In this paper we introduced results of utilizing inorganic nanostructured silicon carbide particles to produce an inorganic particle filled polyimide.

Keywords: Particle-reinforced nanocomposite, Silicon carbide, Polyimide.

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1. INTRODUCTION

In order to maintain further rapid development of aircraft industry, rocket construction, astronautics, nuclear power, electronics industry, radio engineering and other fields of industry the polymeric materials with high durability, thermal stability, elasticity and resistance to radiation are needed. As a result of increasing body of research in this direction a new class of polymers with cyclic chain - polyimides was invented [1].

Over the last decade a lot of research has been devoted to combining of polymers with nanoparticles to produce materials with high stiffness, toughness and tribological properties [2]. The growing demand for nanomaterials is caused by the fact that the new chemical and physical properties can be achieved by adding nanoscale fillers in a polymer matrix, even if the same material without the nanofiller does not have such an advantage. This is due to the influence of the unique nature of the nanofiller bulk properties of the polymer-based nanocomposites [3,4].

Aeronautic is being viewed in perspective as one of the most intensive sphere in terms of composite consumption. In aircraft industry metal or ceramic structures are being replaced with fiber reinforced polymeric composites. With the emergence of composite materials aircraft construction has survived a powerful scientific and technical push. Moreover, the process of influence is reciprocal. In many ways, the current high rate of increasing of market demand for composite materials in aircraft construction is predetermined. Functional limitations of metal alloys and aluminum are obvious, and in conjunction with the constantly rising prices, the issue of substitution by more affordable counterparts with high functionality is relevant as ever. From the set of considerable advantages of aircraft parts made of composite materials, light weight can be identified (by 60-80% less than aluminum), saving the weight leads to improved fuel efficiency/operational range for an aircraft, moreover it helps to avoid corrosion issues encountered with metals or to yield complex structures which cannot be easily made from ceramic. However, these polymeric materials must survive extremes of temperature (heat, flame) in places where metals and ceramics were originally been used. Therefore the combination of the most advantageous features of metals and ceramics into a polymeric material and development of a hybrid material is demanded.

Lockheed Martin has issued the F-35 Lightning II will be the first mass-produced aircraft to integrate structural nanocomposites in non-load bearing airframe components. The material being used is identified as "advanced polymers engineered for the extreme - first generation", or APEX. It is described within the display as "best-in-class ultra-lightweight and affordable structural thermoplastic enhanced with nanoparticles that delivers increased mechanical properties, thermal stability, electrical conductivity and processability over currently available projects [5].

Nanoparticles of silicon carbide (SiC) were chosen because of their unique physical properties such as excellent chemical resistance, heat resistance, high electron mobility, excellent thermal conductivity and outstanding mechanical properties. They are used for high-performance composites [6-9] and in electronics [10, 11]. These properties make SiC nanoparticles a suitable material for the production of polymer nanocomposites with reinforced structure [7].

Our initial work is focused on studying the effects of nanostructured SiC (at loading level of 6 wt%) to enhance the thermal durability of the polyimides. In this work we start from the most commonly used polyimide - poly-oxydiphenylene-pyromellitimide (see Fig. 1) which is widely used in civil and military aircraft.

*egorov@irea.org.ru

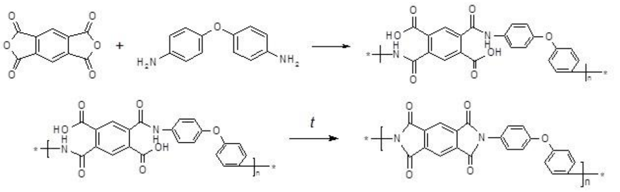

Fig. 1 – Reaction scheme and structure of poly-oxydiphenylene-pyromellitimide

2. EXPERIMENTAL SECTION

2.1 Materials

1-Methyl-2-pyrrolidinone (NMP, Component-Reaktiv) was purified prior to use in poly(amic acid) synthesis by drying over calcium hydride for at least 8 hours, followed by distillation under reduced pressure (using a vacuum pump) (b.p. 202ºC/760mmHg).

4,4'-Oxydianiline (4,4'-ODA, Vekton) was sublimed under vacuum (~5 torr) at ~155ºC.

Pyromellitic dianhydride (PMDA) was synthesized according to the literature [12].

Silicon carbide (SiC) nanoparticles were purchased from InKhimSintez. The properties of the SiC-nanopowder are listed in Table 1.

Table 1 – Characteristic properties of the SiC nanoparticles

Purity	97 %
Average particle diameter	70-80 nm
Surface area	30 – 35 m^2/g
Colour	Black
Morphology	Nearly spherical
Bulk density	<0.5 g/cm^3

2.2 Preparation of the nanocomposite

0.6153 g of nanostructured SiC and NMP (50 mL) were sonicated into a three-necked round-bottom flask equipped with a condenser and an argon inlet for 15 min. Then 5.0461 g (25.2 mmol) of ODA was added and sonication continued for another 15 min, afterwards 5.5000 g (25.2 mmol) of PMDA was added portionwise. The mixture was sonicated for more 30 min. The resulting highly viscous poly(amic acid) solution was cast onto clean and dry glass plates by a scalpel; the films were dried in an vacuum oven at 80°C for 6 h and then at 150, 200, 250, and 300 °C for 1 h at each temperature as well as 350 °C for 15min.

2.3 Analytical techniques

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) analysis was collected with a TA Instruments SDT Q600, under atmosphere of argon and air at a heating rate of 10ºC/min from room temperature to 800ºC.

For scanning electron microscopy (SEM), a Hitachi SU-1510 VP-SEM was used under an accelerating voltage of 7 kV and a working distance of 5.5 mm. IR-spectra were recorded with a VERTEX 70 FT-IR, Bruker Optics.

3. RESULTS AND DISCUSSION

3.1 Synthesis of nanocomposite

The nano-SiC was mixed with ODA and PMDA under sonication to give the corresponding poly(amic acid) with uniformly distributed particles with viscosity of 4929,89 Pa·s. Thermal imidization, the most common approach, was chosen to achieve imidization at the final stage of the reaction. The reaction mixture was cast on clean glass plate, and the film was heated through various stages up to 350°C in vacuum to remove solvent and water formed by the imidization. Black opaque solid film was obtained.

3.2 Nanocomposite Properties

The formation of the polyimide nanocomposite was confirmed by Fourier transform infrared (FTIR) spectroscopy. FTIR spectra of the polyimide films prepared by the thermal imidization method showed absorption bands at about 1775 cm-1 (C=O asymmetric stretching), 1715 cm-1 (C=O symmetric stretching), 1368 cm-1 (C-N stretching), and 721 cm-1 (C=O bending), which are characteristic for imide rings. The absence of absorption bands at 1660 cm-1 corresponding to C=O amide stretching in the FTIR spectra of polyimides indicates complete imidization.

The thermal properties of the polyimide nanocomposite were evaluated by thermogravimetric analysis (TGA). The 5% weight loss temperature of this material is 593°C in air. The Tg of the nanocomposite was obtained from DSC measurements and found to be 460ºC.

Solid film sample was examined by Scanning Electron Microscopy (SEM) to determine how the particles were dispersed in the polyimide (see Fig. 2).

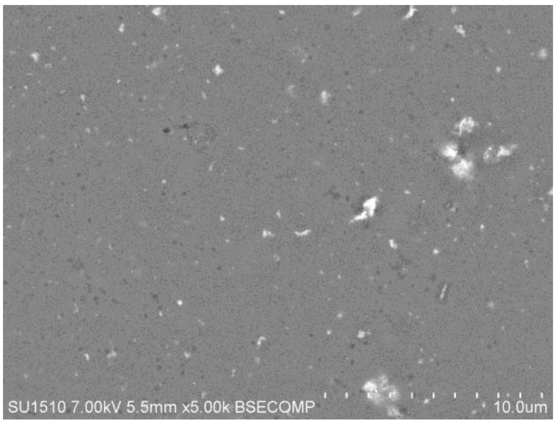

Fig. 2 – SEM image of the composite film

4. CONCLUSION

A new material based on polyimide and nanostructured SiC has been synthesized successfully, it exhibited good thermal stability. Based upon the results collected to date, the use of loadings of a silicon carbide presents some interesting materials science phenomena and effects. At short periods of time, the use of these particles improves thermal stability of the base polymer. The results are complex and still not fully understood at this time and therefore the outcome of our measurements will require more study before it is definitive and even then. The next step of our research group is enlarging the variety of polyimides and loadings of nano-SiC used.

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