Research on structure and performance of polyurethane elastomer/superfine talcum powder composite

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Abstract. The structure and performance of polyurethane elastomer/superfine talcum powder composites were investigated by X ray diffraction (XRD), FTIR spectra and thermogravimetric analysis (TG - DTA, DSC). The results showed that talcum powder and polyurethane elastomer formed com pound structure of heterogeneous nucleation when the content of superfine talcum powder was right(such as 15\%) , which caused the soft segments, hard segments and talcum powder of polyurethane elastomer to form interactional congeries system and the integrated mechanical properties and thermostability of polyurethane elastomer were improved.

1. Introduction
Polyurethane (PUR) resin is made of isocyanate and polyol reaction a with urethane segment repetitive structure unit of polymer material, the modulus of elasticity between rubber and plastic, both of the flexibility of the rubber, and plastics rigidity, with good use performance, processing performance and excellent abrasion resistance, impact resistance, good low temperature resistance, ozone resistance, insulation and other characteristics, is more widely used a new kind of polymer material. For PUR elastomer modified to make it become a high performance engineering materials, is an important content of polymer materials science and engineering applications [1].

In the organic polymer filler made from inorganic mineral composite material, improve the comprehensive performance of polymer materials, the effective method to reduce the cost of materials [2]. Ultrafine talcum powder is a new type of inorganic filler, its chemical composition is mainly silicon, aluminum, magnesium and other composite salt. Because of iron and other impurities in the talcum powder, white degree is high, and has a relatively low refractive index and the fine and uniform particle size, and adding suitable amount of talcum powder of high polymer material can still present a good translucency color and appearance. The structure of pure talc by a layer of brucite (MgO·H2O) sandwiched between two layers of silica, overlay each other between the layers, adjacent layers of talc by weak vander Waals force. When imposed shearing action, it is easy to occur between sliding between the layers. Tale powder in most chemical reagent rendered inert, not contact with acid decomposition, is a poor conductor of electricity, low thermal conductivity and thermal shock, heated to a temperature. The high temperature of 900\degree C still don't break down. The excellent properties of talcum powder to make it become a kind of very good filling agent [3], can be used in the filling of polymeric materials.
To PUR elastomer modified with talcum powder, is to improve the comprehensive mechanical properties and thermal properties of PUR, explore the talcum powder modified polymer material is a beneficial attempt in the field of application.

The author use talcum powder treated by silane coupling agent and PUR elastomer produced PUR elastomer/talc powder materials, and study the structure of the composite material and the effect of talcum powder on thermal and mechanical properties of PUR.

2. Experimental

2.1. Material
Polyether polyols: N-204, Jiangsu Zhongshan chemical co., LTD. 1,4-butyl glycol (BDO): analysis of pure Chinese medicine; Shanghai chemical reagent company Toluene diisocyanate (TDI). 2,4,2,6-isomer (quality Than 80/20), industrial products, Japan's mitsuitakeda corporation. Ultrafine talcum powder: 10 Lm, Liaoning Haicheng talc plant. Silane coupling agent KH-550 (C-ammonia propyl triethoxy silane), Nanjing Xiang Fei chemical research institute.

2.2. Instrument
Automatic X-ray diffraction (XRD) instrument: D/M ax2400 type, Rigaku Japanese company. Fourier transform infrared spectroscopy (FTIR) instrument: Avatar370 type, The United States Nicolet company. Thermogravimetric (TG) analyzer: STA409 type, Netzsch male in Germany Division. New type electronic universal testing machine: CM5104, Shenzhen think twice Group.

2.3. Samples preparation
(1) The talcum powder surface treatment
KH - 550 according to 1% of the mass of talcum powder mixture of 95% ethanol solution, add to the talcum powder, again into the low-frequency ultrasonic cavitation field generator, adjust the ultrasonic time working conditions for the 60s, 20s, intermittent time work 45 times, around 600 w power, for the amplitude f15 mm; Work cycle twice, total working time is 2 h. After dry, add the oven dry 1 h 120oC, and set aside.

(2) sample preparation
The preparation of PUR elastomer/talc composites process is shown in Figure 1.

According to process flow shown in figure 1, will join the 500 ml polyether polyol in 4 mouth flask equipped with a thermometer and mixer, vacuum dewatering under 100 ~ 110 oC 1 h, then cool to 80oC join the talcum powder, stirring after 30 m in add quantitative TD I solution, 80 oC thermal reaction of 1 h, then cool to 55oC after joining chain extender BDO, fast Stir speed, controlling the reaction temperature not to exceed 90oC, after 10 m in the reactant in a temperature of 80~100 oC with remover mold, curing 6 h under 130 oC, demoulding is made after standard sample.
2.4. Testing and characterization

(1) XRD analysis: the experimental conditions for target, Cu Kα radiation (K = 0.154mm), after the monochromatic pipe, tube 40 kV voltage, tube current 100 ma, scan rate of 2 (°)/m, in sample 10 mm ×10 mm the smooth small pieces of 1 mm.

(2) the FTIR analysis: spectral range 350~400 cm⁻¹, 7 set a resolution of 0.1 cm⁻¹.

(3) the TG analysis: the sample is 4~10 mg, atmosphere of N₂, air flow rate of 80 mL/m in the test range is -70 ~ 100°C, heating rate for 10 in °C/m.

(4) the tensile properties test according to GB/T1042-1042, stretching rate in 50 mm/m, ambient temperature of 25 °C and 30% humidity.

3. Results and discussion

3.1. XRD analysis

Figure 2 is pure PUR elastomer and its filling different content talcum powder XRD pattern of the composite material.

Figure 2. Pure PUR elastomer and filling different content talcum powder XRD pattern.

Talcum powder quality score: a) 0;B) 15%;C) 10%;D) 5%

The composite material can be seen from the Figure 2, the pure PUR appear in 2θ for 21°, with no obvious sharp peaks were observed, that is given priority to with amorphous structure; In composite materials with the addition of talc powder, talcum powder (001) plane diffraction peak obvious sharp move in the direction of small Angle, talcum powder layer of layer spacing for PUR macromolecular join further increase, this is intercalated polymerization macromolecule chain into the talcum powder in the process of performance between two SiO₂ layer; With the increase of the content of talc powder to the small Angle direction of the diffraction peaks increase apparently, absorption features more obvious. Mainly because the talcum powder as the lamellar structure, in-situ polymerization release a large amount of heat can make the part of talcum powder interlayer vander Waals keys are destroyed, which may be due to the layer spacing is too large and interlaminar stripping[4], intercalating polymerization played down the talcum powder particles and the interface between the PUR molecular, achieve mutual fusion between PUR and talcum powder. In addition, you can see from Figure 2 also, join the mass fraction 5% talcum powder PUR elastomer for 10θ in 2 ø, 24 ø, 27 ø no diffraction peak occurred, it may be that the adding quantity is too little, scattered unevenly, cannot reflect the talcum powder itself characteristics. Thus can also infer that fill a small amount of talcum powder on the preparation of the meaning of PUR elastomer/talc composites is not very big.
3.2. FTIR characterization

Figure 3. Is pure PUR elastomer and its filling of talcum powder composites at room temperature FTIR spectrum.

By Figure 3 shows, PUR polyether type has the following characteristic peak, 3369.7 cm⁻¹ broad peak is hard segment N-H stretching vibration. For strong narrow peak at 1736.2 cm⁻¹ C= O stretching vibration, 1524.5 cm⁻¹ peak for short -OH and NCO- reaction to generate amide NHCO absorption peak, 1107.3 cm⁻¹ strong peak as the ether of C-O-C stretching vibration, 2954.4 cm⁻¹ short broad peak are stretching vibration of saturated C-H[5]. By Figure 3 shows, 2943.5 cm⁻¹, 1724.5 cm⁻¹, 1522.5 cm⁻¹ three characteristic peak still exists, and haven't changed much, 3345.6 cm⁻¹ the stretching vibration of hard segment N-H peak deformation is short, this is because some obstacles. Especially should be in 801.0 cm⁻¹ appeared sharp and long strong peak, it is Si-O-Si the characteristic peak[6], that PUR macromolecular chain insert talcum powder SiO₂ interlayer, the polymerization reaction, make originally relative freedom of the molecular chain movement restrictions with talcum powder SiO₂ piece layer in the PUR substrate have the effect of junction, this has to do with 3345.6 cm⁻¹ peak deformation is short, hard segment N-H stretching vibration by some obstacles.

3.3. The PUR elastomer/talc composites TG-DTA analysis

Figure 4 is pure PUR elastomer and its filling TG-DTA curve of talcum powder composites. Can be seen from the Figure 4, pure PUR elastomer heat starting temperature for 268 oC, and PUR elastomer/talc composites thermo-gravimetric starting temperature for 272 oC, showed that the addition of talc powder to improve the heat resistance of materials, makes the thermal stability of the material increased; As can be seen from the figure 4 also, c, d curve, respectively in 342 oC and 339 oC have obvious endothermic peak, this is mainly caused by decomposition of low molecular substances, volatile absorption of heat, c, d curve also indicates that two kinds of materials in exothermic peak 387 oC and 387 oC, can be thought of as carbon chain breaking out larger thermal oxide. Above thermal analysis showed that after adding talc powder in PUR elastomer on the material's thermal properties are improved to a certain extent, but the impact is not very big.

Conclusion

(1) through the test and XRD analysis and DSC, talcum powder is added in the PUR elastomer, PUR occur between layers of talcum powder, and with increase of talcum powder content, characteristics of heterogeneous nucleation is more obvious.

(2) the FTIR analysis proves that after adding talcum powder, PUR elastomer and talcum powder form crosslinking, talcum powder as the core.

(3) the TG analysis showed that adding talcum powder has a tendency to improve the thermal stability of composite material, but the impact is not big.

(4) the suitable amount of filling talcum powder can improve the comprehensive mechanical properties of PUR elastomer.
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