Soluble Polyimide Impregnating Varnish

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Abstract

A selected dianhydride was used to react with 4,4′ —diaminodiphenyl ether to produce an amide acid. Then the amide acid was imidized by dehydrating in bolling solvent, which produced a kind of soluble polyimide impregnating varnish. The product has good thermal endurance and good acro-oil resistance.

Introduction

Since polyimide has very good thermal endurance, radiation resistance, good mechanical and electrical properties at high temperature, it has been widely used in industry nowday. But its application is still limited, when use as impregnating varnish because of its insoluble feature.

Take the pyromellitic dianhydride — type polyimide as an example. When it used as an impregnating varnish, only 8 to 12 percent of polyamide acid solution can be obtained, because of the demands of its viscosity and coating ability for impregnating varnish. This kind of impregnating varnish has following Shortcomings:

a) The thickness of coating film must be generally less than 0.10mm, otherwise the coating film will crack, since the water emitted from the imidization reaction cannat be let out of the coating film immediately. The properties of coating film become deteriorated.

b) Since the solid content of the impregnating varnish is too low, we have to impregnate 4 to 6 times to get a satisfied thickness of the coating film. The working efficiency is not satisfactory.

c) As an amide acid in B-stage, it is easy to be hydrolyzed, so that the molecular weight and viscosity of the varnish drop down rappdly during storage. The potlife is too short, generally only 1 to 3 month.

It is desired to make an impregnating varnish from selected soluble polyimide to solve the problems above-mentioned:

a) Because the imidization reaction has been basically finished before impregnating, water will not be relieved during baking the soluble polyimide impregnating varnish. Geater than 1mm of thickness of the coating film may be obtained.

b) By using selected solvent, the solid content of the soluble polyimide impregneating varnish can exceed 25%.

c) Since the imidized soluble polyimide is hard to be hydrolyzed, its potlife will be greatly longer than the promellitic dianhydride-type polyimide.

In this paper, we use 4,4′ —diaminodiphenyl ether to react with the following dianhydride, and certain amount of appropriate mono-anhydride as an end-sealer of the macromolecule.

The amide acidic reaction is carried out in DMAc and certain ammount of xylene, and the imidezation reaction is finished by refluxing untill no water is seperated from the reflux condenser:

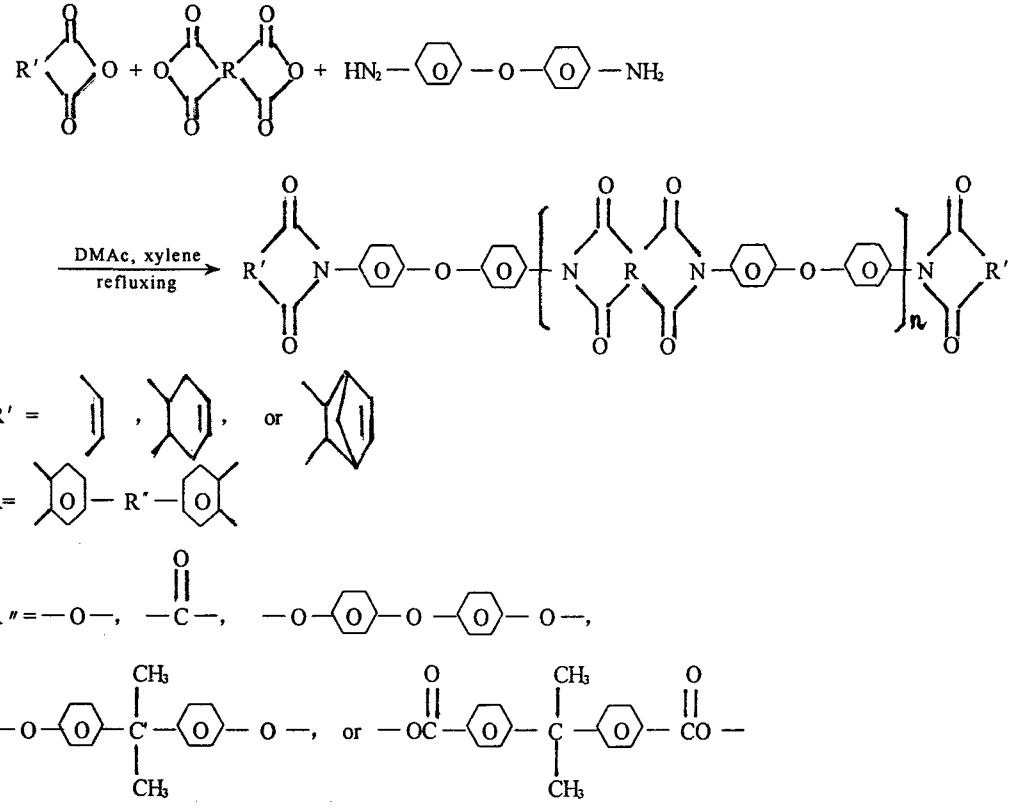

Fig.1

Experimental

Amide Acidic Reaction

1.0 mol 4,4′ —diaminodiphenyl ether was solved in the mixed solvent of DMAc and certain ammount of xylene, then add 0.96 to 0.98 mol selected dianhydride into the solution at 30 to 35℃. After stir 1h at room temperatare, add 0.05～0.09 mol mono-anhydride in it. Keep stirring 3 to 5h at 30 to 35℃.

Imidization Reaction

Dehydrate in the boiling solvent untill no water can be seperated and acid value of the varnish is less than 5mgKOH/g.

Results and Discassion

The principal properties of the soluble polyimide impregnating varnish by this method are shown in Table1.

Table 1 The Principal Properties of the Soluble Polyimide Impregnating Varnish

Order	Properties		Unit	Test Results	Test Methods
1	appearance		—	red-brown transparent liquid, no foreign matter	JB1981
2	solid content (180℃, 2h)		%	25～26	IEC 60464-2
3	viscosity (23±2℃, 4#flow cup)		s	100～120	IEC 60464-2
4	acid value		mgKOH/g	4～5	JB 1981
5	ability to cure in considerable thickness (150℃/5h+180℃/2h, thickness≥1.2mm)		—	S1U1I2.1 uniform	IEC 60462-2
6	stability of the varnish in an enclosed vessel (60±2℃/96h)		multiple of viscosity increment	0.2	IEC 60462-2
7	dielectric strength	original	kV/mm	95～115	IEC 60464-2
		at 250±2℃		90～100	
		after immersed in water for 24h		80～90	
8	volume resistance	original	Ω • m	10^{13}～10^{14}	IEC 60464-2 ((JB 1981)
		250±2℃		10^{11}～10^{12}	
		after immersed in water for 24h		10^{12}～10^{13}	
9	bond strergth (twisted coil test)	orginal	N	220	IEC 61033
		after immersion in aero-oil at 300℃ for 24h		235	
		after exposed to air at 300℃ for 24h		195	

From Table 1, we can see the product has following characteristics:

a) When we make the impregnating varnish have 25% of solid content, its viscosity is still low;

b) From acid value of the varnish, the imidization reaction degree is estimated to be greater than 95%;

c) The thickness of coating film may achieve 1.2mm or more, and still with good properties;

d) The potlife of the varnish at room temperature can be expected to be 1 year or longer;

e) The properties of the coating film at high temperature are at the same level with pyromellitic dianhydride- type polyimide;

f) The coating film has very good aero-ail resistance.

Application

This soluble polyimide impregnating varnish has been used to impregnate the winding of a special oiled generator for a spaceflight . Thermal endurance test of the electrical insulation of the generator has been past: When the temperature of cooling oil at inlet and outlet is respectively 250℃ and 270℃, endured 30h, the reliability of the electrical insulation of the generator exceeds 0.998.

Reference

[1] IEC pub. 60464-2, 1974, 1st edition