The relationship of heat treatment and heat deflection temperature (HDT) of polyimide

Liu Shaohua Tang Jinwen Wu Jianhua Cai xianqin

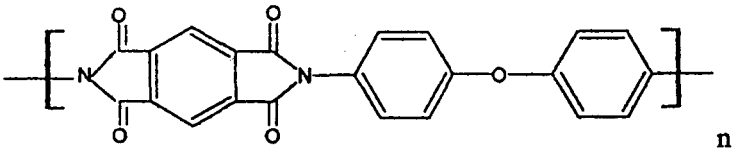
Aromatic Polyimides (PI) has high heat-resistant and mechanical properties. Because of the high molecular rigidity, such as the resin from Pyromellitic Diannydride (PMDA) and 4,4'-oxydianiline (ODA), theoretical melting point is higher than decomposed temperature. That can't be pressed by conventional techniques. From 80s, a lot of thermoplastic PI has been found, such as polyetherimides of GE company (USA) and meltable PI from 4,4'-oxydipethalic anhydride (ODPA) and ODA. Those can be molded by heat plasticity , but all the HDT of this kind of PI is lower than 240℃ and the use temperature is low.

From 90s, people imagine to mould meltable PI at low temperature and crosslink by heating in air at high temperature. Rising HDT, let PI can be used in high temperature.

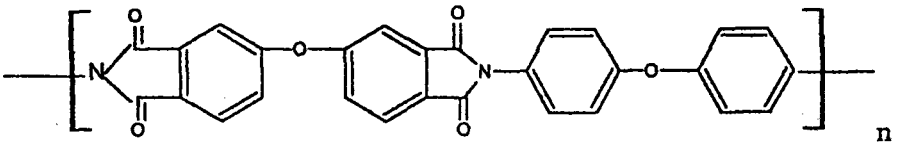
In this paper, researched crosslinked PI plastic by heat treatment and observed the change of HDT of the different PI structure under different treatment condition. The result is as the follows:

The PI plastic used:

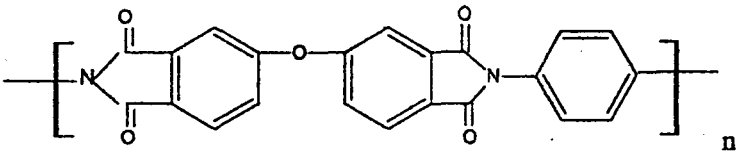
1. PMDA-ODA



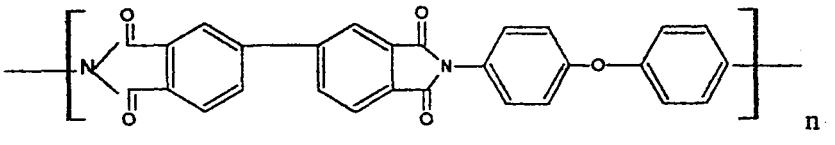
2. ODPA-ODA



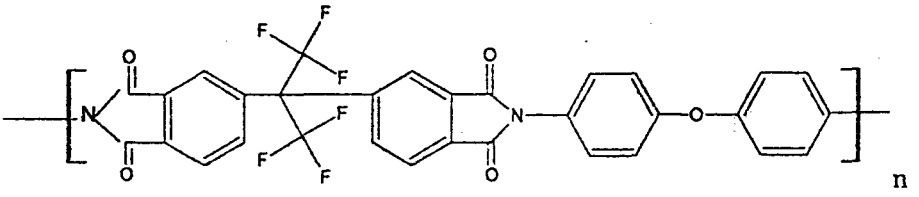
3. ODPA-PDA*



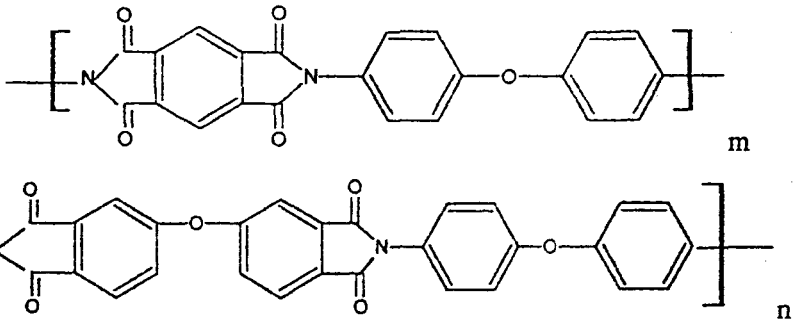
4. BPDA*-ODA



5.6FDA*-ODA



6. PMDA-ODA-ODPA



*PDA: 1,4-dianiline

*BPDA: Biphenyl tetracarboxylic dianhydride

*6FDA: Hexafluorodianhydride

Equipment

Heat treatment oven: hot air oven with program control

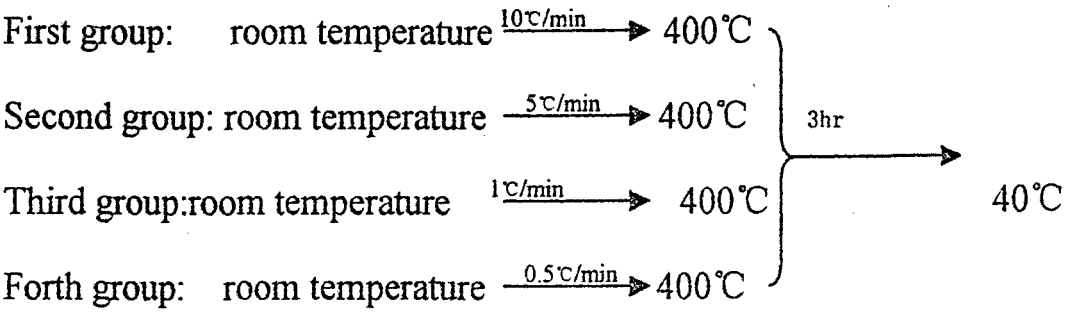
Heat deflection temperature appliance: air medium

Make sample:

First synthesized PI powder

Second moulded the test samples under high temperature (size: 10*15*120mm)

Heat treatment conditions:



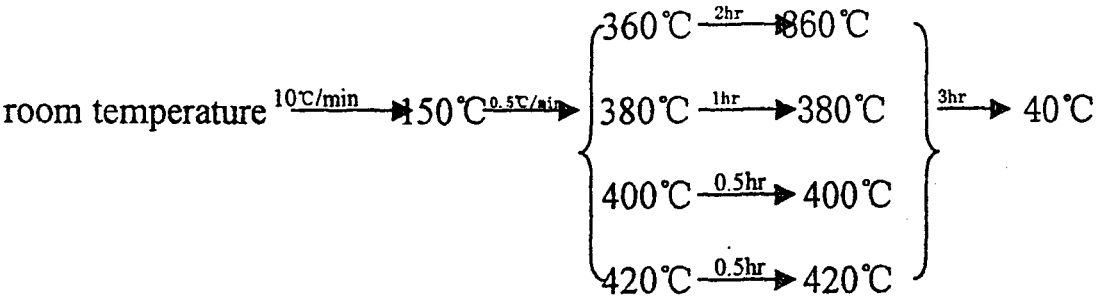
Six types PI samples were selected by four groups heat treatment conditions. We can draw a conclusion that the forth group changed most and most useful. Then we chose to use the forth condition to heat-treat all the PI plastic.

The result is as the follows:

Structure	Appearance	Change of HDT
1. PMDA-ODA	no change	increase 10℃
2. ODPA-ODA	warp, foam	can't test
3. ODPA-PDA	no change	>450℃
4. BPDA-ODA	little warp	no
5. 6FDA-ODA	no change	no
6. PMDA-ODA-ODPA (CPI)*	(no to a little warp)	rise 30-60℃

*in CPI there are different composites, including graphite

CPI test samples test in different treated conditions.



Result

Sample No	Heat treatment temperature	lost weight(%)	HDT(℃)	change of impact strength(%)
1#(original)	/	/	340	100
109#	360℃	0.239	389.5	104
104#	380℃	0.292	404.6	150
100#	400℃	0.410	422.3	114*
17#	420	0.440	445	140*

*size increase in direction forming

As result, the HDT of CPI raised to 400℃ by heat treatment. Increased treatment temperature, the HDT can be increased more but the samples warped. Addition to the impact strength of CPI increased.

The mechanism of heat treatment increased HDT, used to analysis crystallinity by WAXD, found that in the non-direction forming the crystallinity raised obviously. At the 380℃ the crystallinity reaches the highest value, then decreased again. Further information needs deep research.