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Synthesis of a New Polysiloxane Containing Octaphenylcyclodisilazane

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Abstract: A new polysiloxane containing octaphenylcyclodisilazane was synthesized from the anionic polymerization of octamethylcyclotetrasiloxane (D_4) and "seed solution" as initiator, which was prepared by hexamethylcyclotrisiloxane (D_3) and the lithium salt of N,N'-bis (hydroxydi-phenyl)tetraphenylcyclodisilazane.

Keywords: Polysiloxane, octaphenylcyclodisilazane, synthesis.

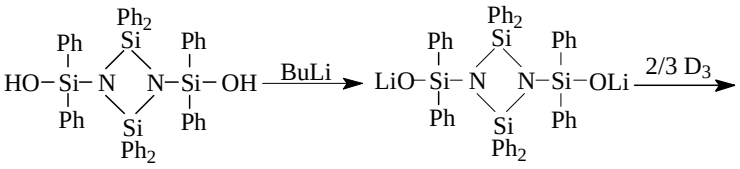
Polysiloxanes have many excellent properties such as high temperature resistance, low temperature resistance, resistance to weather and good electrical property. So they were applied in numerous harsh environments, for example, aviation, aerospace, automobile industry and medical apparatus. In many such applications they were required to resist very high temperature. A special polysiloxane containing cyclodisilazane in the main chain such as tetraphenyltetramethylcyclodisilazane was reported having good resistance to high temperature[1]. But its application was limited by the complicated synthesis of the cyclodisilazane monomer. It was also reported that the more amount of the phenyl groups in cyclodisilazane, the better thermoresistant of the polysiloxanes[1]. In our work, a new kind of polysiloxane containing octaphenylcyclodisilazane was synthesized (**Scheme 1**), which can resist higher temperature. And the synthesis of its monomer was much simpler and more effective than before[2].

First, the compound **1** was prepared from the reaction of N,N'-bis(hydroxy-diphenyl)tetraphenylcyclodisilazane (4.0g, 0.005mol) and butyl-lithium (0.01mol) in THF for 1 hour at -5℃. Stirring was continued for 2 hours at room temperature. The compound **1** was obtained by filtration. (yield: 70%).

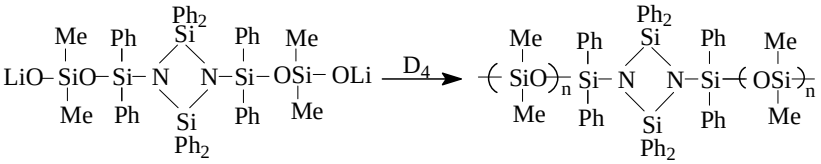
Second, the "seed solution" was prepared by the reaction of hexamethyl-cyclotrisiloxane (D_3) (0.5g, 0.002mol) and **1** (2.48g, 0.003mol) at 110℃ for 12 hours. Polysiloxane **2** was synthesized by anionic polymerization of octamethylcyclo-tetrasiloxane (D_4) with the "seed solution" as initiator and THF as promoter. After stirring at 100℃ for 24 hours the polymerization was terminated (yield 70%). The polymer's structure was determined by ^{29}Si NMR: δ -25.5(N_2SiPh_2), -28.3($OSiMe_2O$), -37.3($NSiPh_2O$). The number-average molecular weight was 32000 measured by GPC. And the viscosity-average molecular weight was 39000 calculated

by $[\eta]=0.03M^{0.63}$, $[\eta]$ was measured in toluene at 30℃. Our experiments showed that the molecular weights of this new polysiloxane were controlled easily by using the "seed solution" as initiator.

Scheme 1



1



seed solution **2**

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