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STANDARD OPERATING PROCEDURES
(Polymerization of Syndiotactic Polystyrene)

Caution: Some materials used in this experiment are **hazardous**.
Always wear goggles, (face shield), Rubber Gloves, Apron etc**.**

Contents

Materials

○ Styrene (Aldrich) was vacuum distilled over calcium hydride, and activated alumina was used to remove inhibitor from the monomer.

○ *n*-heptane (Fisher Scientific) was used as a diluent, and it was purified by being refluxed over sodium and benzophenone in nitrogen atmosphere.

○ Cyclopentadienyltitanium(IV) trichloride ($CpTiCl_3$, Aldrich, min 97%) and methylaluminoxane solution (MAO, Aldrich, 10 wt.% in toluene) were used as catalyst and co-catalyst, respectively without further purification.

○ Silicon wafer (University Wafer, P-type (100)) was used as a catalyst support.

Surface Treatment of Silicon Wafer

o Organic coating removal: Immerse wafers in Pirhana solution for 5 to 10 minutes. Pirhana removes organic contaminants by oxidizing them, and metals by forming soluble complexes. Pirhana solution is a 5:1 mixture of H_2SO_4 with H_2O_2. The mixture is self-heating and the H_2O_2 has to be added very slowly.

o Rinse with DI water for at least 1 min

o Oxide removal: Dip in DI water. Immerse the wafer in 50:1 diluted HF or 10:1 mixture of DI water and buffered HF for 5 to 15 sec.

Preparation of Silica-Supported Metallocene Catalysts

o For the anchoring of metallocene catalyst onto silicon wafer, MAO was used as binding agent. MAO is bonded with silicon wafer, then, the chloride group of $CpTiCl_3$ is complexed with aluminums in MAO.

o First of all, the surface treated silicon wafer was treated with MAO solution at room temperature for 24 hr, washed with toluene, and dried in vacuum overnight. (× 2 times)

o Then, the MAO treated silicon wafer was soaked in $CpTiCl_3$ solution for another 24 hr, washed with toluene, and finally, dried in vacuum overnight.

Polymerization of Syndiotactic Polystyrene

o Polymerization of syndiotactic polystyrene was carried out using glass tube reactor. 1.5 ml of styrene monomer, 1 ml of n-heptane, 0.15 ml of MAO solution, and silica-supported metallocene catalyst were charged into reactor in an argon filled grove box. The experiment was performed at 70℃ for 1-30min.

o After polymerization, the reactor was quenched using liquid nitrogen to conserve the shape of polystyrene.

o Then, the frozen solution was evaporated in vacuum chamber.