HIGH MODULUS POLYPROPYLENE FILAMENTS: PREPARATION, STRUCTURE AND PROPERTIES

by

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CERTIFICATE

This is to certify that the thesis entitled "**HIGH MODULUS POLYPROPYLENE FILAMENTS: PREPARATION, STRUCTURE AND PROPERTIES**" being submitted by **Mr. Prabir Kar,** to the India Institute of Technology, Delhi for the award of the degree of **Doctor of Philosophy** in the department of Textile Technology, is a record of bonafied research work carried out by him Mr. Kar has worked under my guidance and supervision and fulfilled the requirements for the submission of the thesis.

The results contained in this thesis have not been submitted, in part or full, to any other University or Institute for the award pf any degree or diploma

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ABSTRACT

An innovative method is developed to draw polypropylene (PP) filaments to very high draw ratio (~21x). The PP filaments are produced by melt spinning and drawing in two stages in sequence in typical monofilament production process. These filaments are used as feed materials for ultra drawing by draw heat setting (DHS). DHS is a batch process and is carried out under constant tension in a silicon oil bath. Superior stress transfer and reduced molecular relaxation is achieved by this. Extensive analysis of the drawing behavior and straining rate has been carried out with range of polymer MFI, stress and temperature. The process is optimized for high sonic modulus as well as for process speed. Optimized temperature and nominal stress taken for experimental set up are 160°C and 40 MPa respectively. Sonic modulus achieved is 25 GPa and process speed is ~2 mt/min. Drawability is found to be sensitive to draw temperature, polymer MFI and stress applied.

DHS process leads to very high crystallinity (76%) and molecular orientation (f_c~0.98, f_a~0.86). Both crystallinity and overall orientation increase with draw ratio. Super heating phenomenon suggests presence of extended chain crystals. WAXS and DSC studies indicate existence of high degree of crystal perfection with narrow crystal size distribution. Near absence of SAXS meridional intensity and very high dynamic modulus (~30 GPa) at low temperature suggest existence of crystalline continuity in terms of crystalline bridges. Absence of β transition and shifting of α transition beyond 140°C supports the above proposition. Void size and fraction increase with draw ratio

upto 13x then it reaches near saturation when drawn at 160°C, but continue to increase for lower draw temperature. These fibres are highly fibrillar, with microfibril dimensions in the range of 150 nm. A structural model for the DHS sample is also proposed.

Very high modulus (18 GPa) and tenacity (700 MPa) are obtained for DHS filaments. Lower MFI materials result in slightly higher tenacity with relatively lower modulus, whereas higher MFI materials give higher modulus with slightly lower tenacity. Modulus increases with draw ratio almost linearly. Tenacity increases with draw ratio upto ~13x, beyond which it remains almost unchanged at draw temperature of 160°C, but decreases when drawn at lower draw temperature of 140°C. Tenacity is highly influenced by void contents. At lower temperature of 140°C, void generation is high, which leads to decrease of tenacity at high draw ratio. These samples show excellent thermal stability. A low value of 8% shrinkage is observed when free shrinkage is allowed at 160°C..

Creep compliance of DHS samples is very low even at 50% of breaking stress and recovery from creep extension is high at lower stress. These samples exhibit considerable relaxation from stress, though less compared to drawn samples. Fatigue in cyclic loading at low strain is low, but at high strain, samples show considerable fatigue. These behavior is related to inter microfibrillar movement and partial failure of crystalline bridges.

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