# STUDIES ON STRUCTURE AND MORPHOLOGY AND THEIR CORRELATION WITH SOME PROPERTIES OF HEAT-SET POLY (ETHYLENE TEREPHTHALATE) FIBERS

By

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# CERTIFICATE

This is to certify that the thesis entitled "Studies on structure and morphology and their correlation with some properties of heat-set poly(ethylene terephthalate) fibers", being submitted by Mr. A. K. Jain, to the Indian Institute of Technology, Delhi, for the award of the degree of Doctor of Philosophy in the Department of Textile Technology, is a record of bonafide research work carried out by him. Mr. A. K. Jain 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 in full, to any other University or Institute for the award of any degree or diploma.

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# ABSTRACT

A wide range of structure and morphology was developed in commercial Poly(ethylene terephthalate)(PET) continuous filament yarn by heat-setting treatment over a range of temperature (100°C to 258°C) in silicone oil bath for 5 min. under taut-at-constant-length and free-to-relax conditions. Various techniques were used to characterise the morphological structure of these samples. The crystallinity, crystallite size and perfection of crystalline phase were found to improve on heat-setting. The crystallite orientation was found to remain essentially unchanged for taut-annealed (TA) samples whereas for free-annealed (FA) samples a slight decrease was observed. The crystalline unit cell became more and more compact on heat-setting, as was revealed by the measurement of unit cell parameters. The crystallites were also observed under electron microscope and the average crystallite size was found to be in conformity with the X-ray results.

The amorphous phase was characterised by infrared spectroscopy applying Attenuated Total Reflection (ATR) technique. The trans and gauche content, trans orientation and amorphous orientation factors were measured. The results showed that infrared technique of measuring amorphous orientation, which is a direct method, is more representative of amorphous orientation than the indirect sonic velocity method, commonly used in the case of PET fibers.

The dependence of axial modulus on structure and morphology was critically examined in terms of single-phase and two-phase models. The nature of coupling between crystalline and amorphous phases was found to have an important effect on modulus. The non-crystalline phase made a predominant contribution to the axial modulus of the fibers. The deformation mechanisms under axial load were studied by measuring crystallite size using the X-ray diffraction technique. A reduction in crystal size was observed and this was attributed to longitudinal slip process. In case of TA samples the chain unfolding was observed to occur at relatively lower strains and longitudinal slip process appeared to be the main deformation mechanism.

The value of intrinsic crystalline and amorphous birefringences have been evaluated using amorphous orientation factor obtained from infrared method. The higher values of intrinsic crystalline birefringence appeared to be related to the better perfection of crystalline phase in heat-set PET samples. The short-comings in the theoretical calculation of intrinsic crystalline birefringence were critically examined. The analysis suggested that the wide range of values of intrinsic crystalline and amorphous birefringence reported in the literature could arise from their morphology-dependence.

The thermal studies were carried out using differential scanning calorimeter (DSC) with the fiber samples in the unconstrained and constrained states in the DSC cell. The results were analysed in terms of the role played by molecular orientation and the effect of entropy on the melting behaviour of PET fibers.

Computer programs were developed for analysis of crystal perfection, determination of lattice parameters and theoretical calculation of intrinsic crystalline birefringence. A stretching device was designed for carrying out deformation studies using X-ray diffractometer.

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