SOME INTER-RELATIONSHIPS BETWEEN STRUCTURE, PHYSICAL CHARACTERISTICS AND PROPERTIES IN POLY (ETHYLENE TEREPHTHALATE) FIBRES AND FILMS

By

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THESIS SUBMITTED
IN FULFILMENT OF THE REQUIREMENT
OF THE DEGREE OF

DOCTOR OF PHILOSOPHY

TO THE

INDIAN INSTITUTE OF TECHNOLOGY, DELHI

APRIL, 1993

to my beloved parents

CERTIFICATE

*This is to certify that the thesis entitled "**SOME INTER-RELATIONSHIPS BETWEEN STRUCTURE, PHYSICAL CHARACTERISTICS AND PROPERTIES IN POLY (ETHYLENE TEREPHTHALATE) FIBRES AND FILMS**" being submitted by Mr. J. Radhakrishnan, 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. J. Radhakrishnan 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 of diploma.

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ACKNOWLEDGEMENTS

I express my deep sense of gratitude to Prof. V.B.Gupta for his excellent guidance throughout the course of this research work. In these formative years of my research career, I have immensely benefited from his vast experience and knowledge, and it was indeed a great privilege and pleasure for me to have been associated with him. And, the importance of his role in moulding my career can not be expressed in words.

During the course of my research work, I have drawn help from a number of people; all of them deserve my sincere gratitude.

I wish to thank Dr.P.K.Chidambareswaran of CIRCOT Bombay, and Dr. C.Ramesh of Physics Department, University of Bristol, UK, for the assistance in X-ray diffraction work and for some useful discussions.

I also wish to thank Dr. T.Manabe and Dr. R.Jalan of M/s Modipon Ltd., Dr. S.G.Naik of M/s Garware Polyester film Division, and Dr. R.Huisman of Enka, Holland for providing the necessary samples.

I am thankful to Dr. A.Venkataraman of BTRA, Bombay, Dr. A.K.Jain of JTRL, Calcutta, Dr. D.Rama Rao of NAARM, Hyderabad, and Dr. C.Brahatheeswaran of CIPET, Madras for their help and co-operation.

I wish to express my gratitude to Prof. M.L.Gulrajani, Prof. Bhaskar Datta, Prof. P.K.Hari, Prof. P.Bajaj, Prof. B.L.Deopura, Prof. V.K.Kothari, Prof. A.K.Mukherjee, Prof. R.B.Chavan, Dr. Kushal Sen, and Dr. S.M.Ishtiaque for their help and encouragement.

I wish to thank Mr. V.K.Kala, Mr. R.Khattar, Mr. S.K.Diwedi, Mr. Ashok, Mr. Shivcharan, Mr. Patilal, Mr. Amarjeet, Mr. Khan, Mr. Jaipal of Fibre science and Fibre production lab and Mr. Rajesh Arora for their help. Mr. D.K.Sharma of SEM lab deserves my special thanks for the excellent work in SEM and for the preparation of photographs.

I am extremely thankful to Ms. Meenakshi Goyal for her help and companionship during this research work.

Mr. S.J.Mahajan and Mr. Rakesh Koul deserve my special gratitude. My thanks are due to all my past and present colleagues; Dr. Anand Kumar, Dr. Kashinath Bhowmick, Mr. G. Nalankilli, Mr. D.K.Paliwal, Dr. M.Patnaik, Mr. S.K.Rana, Mr.

A.K.Roopanwal, Mr. H. Bahrami, Mr. Sanjay Mehta,Mr. Praveen Arora and Mr. Y.C.Bhuvanesh.

I am thankful to Mr. Balachandran for word processing and Mr. B.B.Arora and Mr. C.Saraswat for figure tracings.

I offer my thanks to Mrs. and Mr. N.V.Nair, Vani and Vishnu for their love and affection during my stay in IIT.

Mention must be made of my brothers who have always been a source of encouragement to me.

Finally, I express my gratitude towards my parents who always stood by me in my venture and to them I solemnly dedicate this thesis.

(J. Radhakrishnan)

ABSTRACT

The studies reported in this thesis have been made on fibres and films produced from poly(ethylene terephthalate) (PET) and are mainly concerned with inter-relationships between structure and properties. The starting samples used for these studies were filaments spun at speeds ranging from 10 to 5500 m/min and film cast at 10 m/min. Some of these products were uniaxially stretched to various extents at different strain rates and temperatures in air or in water. A wide spectrum of structures and morphologies with different degrees of anisotropy was thus available for studies, which included : (a) characterisation of the molecular network in the spun filaments and flow-drawn non- birefringent films, (b) thermal shrinkage behaviour of a large number of samples at elevated temperatures, (c) generation and decay of shrinkage stress when some of the samples, on which shrinkage studies were made, are heated at constant length, and (d) characterisation of crystal defects and residual stress distribution in some of the highly drawn and heat-set samples. In addition to these investigations on the samples described above, the effect of annealing on crystallisation and morphological structure has been studied on a commercial PET drawn yarn. The emphasis in these studies was on the effects produced on physical properties when the processing parameters used in sample preparation are varied. A summary of the principal features of the investigations made and the main findings is given below.

The molecular networks in PET filaments spun at different speeds and films drawn under different conditions were characterised by studying their load-elongation behaviour and then analysing the data in terms of the statistical theory of rubber elasticity. It was found that the filaments which had been spun from the melt upto take-up speeds of 3000 m/min and flow- drawn, non-birefringent films had rubber-like network with physical entanglements acting as crosslinks; the entanglement density in the flow-drawn films

being 2 to 3 times less than that in the as-spun filaments, apparently as a result of entanglement slippage during flow drawing.

The thermal shrinkage characteristics of undrawn samples and of the samples drawn to low draw ratios were found to be predominantly related to disorientation of the molecules in the amorphous phase which is characteristic of rubber-like network. In samples of high draw ratio, crystallisation could take place concurrently with shrinkage and thus affect shrinkage. This was particularly noticeable in samples produced at very low speeds which showed much lower shrinkage because of rapid crystallisation. These samples have been shown to have highly defective crystals and also tiny nuclei,both of which promote rapid crystallisation which impede shrinkage. The possible reason for the presence of nuclei and imperfections in the crystals have been discussed.

The generation and decay of shrinkage stress in oriented films and fibres, held at constant length and then heated to 100 and 220° C, respectively, have been studied. The stress generation in all samples has its origin in entropic considerations, viz. due to the coiling tendency of the molecules which results in contractile stress. The decay of stress has been attributed to disorientation in samples with very low molecular orientation and low crystallinity and to crystallisation in samples with high molecular orientation and low crystallinity. In samples with high molecular orientation and high crystallinity, the stress did not decay with time. The structural basis for these results has been discussed.

The crystal distortion parameter has been measured for a number of oriented samples produced at different speeds. It is shown that samples produced at low speeds have the lowest crystalline content and the most imperfect crystals. The effect of annealing on crystal perfection has also been studied and it is shown that the crystal defects migrate out of the crystals on annealing. An analysis of the results in terms of their structures has been made.

The residual stress distribution in fibres produced at different speeds has been studied by chemical degradative etching of the fibres. The studies revealed that the samples produced at low speeds and at low stresses had a very different stress cracking pattern compared to the sample produced at high speed. The technique has been shown to be quite sensitive to residual stress distribution and the possible relationship of the cracking patterns with sample morphology has been presented.

The effect of annealing temperature between 100 and 250°C on crystallisation and morphological structure of a commercial, oriented PET yarn has been studied with the help of small-angle and wide-angle X-ray scattering. The studies assist in gaining an insight into how crystallisation occurs in different temperature ranges and how the morphological structure develops in the PET fibres.

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