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Characterization of Plastics by Physical Methods

Experimental Techniques and Practical Application

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Foreword

This survey of physical methods used to characterise the solid structure of plastics is the result of expanding a series of lectures held at the "Institut für Kunststoffverarbeitung" (IKV) at the Technische Hochschule at Aachen, West Germany. In the main, the students were from engineering and physics faculties and specialising in plastics processing. The aim of the course was to provide an overview of those physical methods relevant to polymers whilst at the same time keeping it fairly simple and without recourse to the theoretical principles which were taken for granted; further it was intended to make it more approachable by introducing numerous examples from technical practice. The following questions were addressed primarily:

- which physical methods are commonly employed in the characterisation of high polymers
- what does the engineer/physicist/chemist concerned with the production/processing/application have to know in order to be able to form a clear idea of suitability of the method, the resources and effort required to carry it out and the cost to benefit ratio of these methods
- what is it necessary to know about boundary conditions, sample preparation, instrumentation and personnel costs, the extent and quality of the results.

The following topics are not discussed:

- exotic methods far removed from everyday practice as well as those methods not yet adequately developed for their inclusion in such practice

- methods for the determination of the crystal structure of crystalline polymers as well as methods for the determination of the structure and conformation of single polymer molecules

- the theoretical background of the methods, insofar as this exceeds the basis necessary for the understanding of the method

- the particulars of instrumentation which do not contribute to the basic understanding of the procedure.

The following comprise topics in overview form:

- combination of different physical structural analysis methods to solve complex questions and
- correlations between the structure of plastics and their technological properties; the dependence on the conditions of production and processing.

This survey is intended to provide a completion to the volumes "Polymeranalytik" I and II (Eds. Drs. M. Hoffmann, H. Krömer, and R. Kuhn, published by Thieme Verlag, Stuttgart, West Germany, 1977), since it builds on to various descriptions given there. I would like to extend my thanks to the authors and the publisher Georg Thieme, Stuttgart for their willing permission to reproduce figures from these two books.

It was first Professor Dr. Menges (Director of the Institut für Kunststoffverarbeitung, Technische Hochschule Aachen) who suggested that I give the series of lectures and subsequently expand them into a book and I am especially grateful to him for the continuous support and great interest he has shown throughout.

I am indebted to very many colleagues who have helped both in word and deed, and particularly to Dr. Holm (Chap. 7), Dr. Morbitzer (Chap. 6), Dr. Mueller (Chap. 7), Dr. Schmid (Chaps. 3 and 8), Dr. Wangermann (Chap. 4), Dr. Weber (Chap. 5). Particular thanks are due to Dr. Kroemer for checking the manuscript and offering numerous suggestions as well as to Miss Wegemann for typing the manuscript so accurately. I wish to thank Dir. Dr. Bottenbruch and the management of Baver AG for permitting me to publish this work. Finally I owe my thanks to the Carl Hanser publishers for their cooperative help in organising and presenting the book.

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Introduction

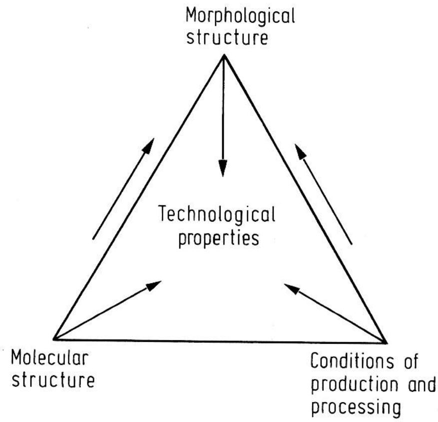
Following the pioneering work of STAUDINGER in the 1920s, plastics chemistry and technology have taken a considerable upward turn with the synthesis of new polymers and with the continuous development of production and processing techniques. Thus the production of plastics in 1979 had already outstripped the production of steel on a volume basis.

Modern development in the field of plastics is characterized less by its growth in quantity than by the increasing importance of high-grade plastics, for example engineering plastics. Other than the continuous improvement in the characteristics of high-grade homopolymers (e.g. polyamides, polyesters, polycarbonates, polyurethanes etc.) it has been possible to supply polymer materials possessing characteristics designed to meet particular requirements; this was achieved especially by exploiting the composite structure of products obtained by modification of well-known polymers. Thus the number of known and technically-used monomers (about 50 from around 1000 basic compounds synthesized) has not increased in the past few years; but it has been and still is possible, simply by forming new combinations, to provide a variety of multicomponent polymer systems which possess, in part, surprising new properties.

Among these multicomponent polymer systems the multiphase materials, such as co-, graft and block co-polymers as well as mixtures of (partially) incompatible monomers as well as pigmented, filled and reinforced plastics, are of especial interest. If the concept of multicomponent materials is extended to include also different physical phases of the same basic chemical substance then as long as they form discrete phases, semicrystalline polymers will also fall under this heading.

In the characterization of multicomponent polymer systems, the structural elements at a molecular level have to be distinguished from those at the macromolecular level. The former are solely of a chemical nature, irrespective of the aggregation state of each chain molecule. To this group belong composition, molecular weight distribution, steric arrangement of substituents (tacticity), chain branching and, in the case of copolymers, the proportion and sequence of the structural units. At the macromolecular level, the spatial distribution of these structural elements is described according to their crystallographic, thermodynamic and statistical properties[159]. The morphology is thereby dependent on the nature and proportion of each of the polymer components as well as on the production and processing conditions; it critically influences the technologically-interesting macroscopic characteristics of multicomponent plastics.

The increasingly complex structure of modern chemical materials has required the development and product-based adaptation of methods of physical examination. These physical techniques are not only necessary for the analytical characterization of polymers, and particularly multicomponent plastics but they also represent the experimental basis for the interesting correlations between the structure of macromolecular materials, the conditions of their production and processing and their technological characteristics (see sketch).



This increasing use of physical measurement techniques to characterize plastics and the evident success of this technical discipline are the results of different developments occurring during the past twenty years:

- the introduction of new physical measurement techniques for the examination of plastics as well as improvements in known techniques designed to meet particular problems. Examples: electron beam microprobe EMA (1965), scanning electron microscopy SEM (197), electron spectroscopy ESCA (1969), secondary ion mass spectrometry SIMS (1971), pulse fourier transform nuclear magnetic resonance spectrometry PFT-NMR (1983), laser microprobe mass analysis LAMMA (1985) etc.

- the enormous expansion and improvement of data processing as well as the introduction of microprocessor technology. As a result of the registration and processing of such large quantities of data it has been possible, for example, to introduce the modern Fourier transform technique for nuclear magnetic resonance (FT-NMR) and infrared spectroscopy (FT-IR) and further, to calculate colour formulation directly by means of objective colour measurement and the use of purpose made computers.

- the essential advance in improved understanding of molecular and macromolecular structures as well of the relationships between these structures and the technological properties (for instance relating the energy absorption mechanisms under impact loading to deformation processes at a molecular level like crazing and shear yield). These findings provided the basis for the interpretation of the technical behaviour of polymers and for the specific optimization of engineering plastics.

Correspondingly, with the increased importance of physical methods in the characterization of polymers, there arose the need for a concise survey of the basic apparatus and methodical knowledge in a clear and readily understandable form. The following eight chapters deal with the microscopic methods (light and electron microscopy), diffraction methods (X-ray, electron and neutron diffraction, light scattering), the spectroscopic methods (nuclear magnetic resonance, infrared and Raman spectroscopy), thermal, selected electrical and mechanical measurement techniques, methods of physical trace analysis (with particular reference to surface-specific methods) and objective colour measurement; all are illustrated by typical practical examples.

1 Microscopic Methods

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