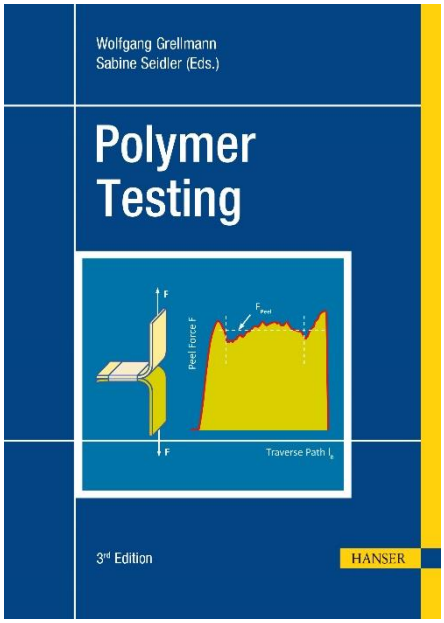
HANSER



Sample Pages

Polymer Testing

Wolfgang Grellmann and Sabine Seidler (Eds.)

ISBN: 978-1-56990-806-8
E-Book ISBN: 978-1-56990-807-5
ePub ISBN: 978-1-56990-808-2

For further information and order see

www.hanserpublications.com (in the Americas)

www.hanser-fachbuch.de (outside the Americas)

Preface to the Third Edition

The present third edition of the textbook *"Polymer Testing"* considers notes and requests for changes given by colleagues and cooperation partners in their numerous, very positive reviews. Furthermore, all chapters were critically reviewed by the editors and authors. The general bibliography of the individual subjects summarized in Chapter 1 was brought up to date with recently published textbooks and monographs. Due to the rapid changes in standardization, more than 400 international and German standards have been updated up to January 2022.

The editors were very pleased to acquire Prof. Dr. Katrin Reincke as a well-known expert in her field of expertise to add a new Chapter 12 in this edition dealing with "Testing of Polymer Films". Polymeric films have extremely wide application fields and most of the polymers produced all over the world are used for packaging, where films comprise one very important group of products. Due to the low thickness, specific requirements to the properties and applications and also certain requirements with respect to the testing of films arise.

Apart from this book, another source of information covering definitions of technical terms and how to perform methods of polymer testing in practice is provided by the encyclopedia "Kunststoffprüfung und Diagnostik" (Plastics Testing and Diagnostics, in German) available online at *https://wiki.polymerservice-merseburg.de* (version 12.0, 2022).

We would like to thank Carl Hanser Verlag, especially and representatively Dr. Mark Smith for his much-appreciated and reliable assistance. Finally, the editors acknowledge Dr. Ralf Lach for his technical and organizational suggestions during revision of both the text and figures of this edition.

January 2022 The Editors

Preface to the Second Edition

The textbook *"Polymer Testing"* is mainly intended for the education of university students and students of universities of applied sciences. This textbook was deemed to be necessary because the testing of polymers has become established as a separate scientific discipline within polymer sciences in recent years. The textbook was first published in German in 2005. An improved English version was published in 2007, and a Russian edition appeared in 2010 with special consideration given to the specific GOST standards.

The positive reviews from our colleagues demonstrate that the concept "Method – Parameters – Examples" meets students' needs and is also accepted in practice.

Although there have been no significant changes to basic testing methods since the first edition appeared, there have been considerable advances in the evaluation of structure–property correlations and standardisation. It has become increasingly necessary to provide material-scientific parameters to quantify the relationship between microstructure and macroscopic properties. Therefore, it seemed necessary to publish a second edition. The previous edition has been comprehensively revised, and the new edition covers all the latest developments in the field, including all amendments to the most important polymer test standards up to May 2013.

Using the same concept and methodical structure in the presentation of polymer test procedures, the parameters obtained by the latter and the selected examples, the new edition provides university students and students of universities of applied sciences with a good and fast source of information. This is why the textbook has been widely adopted by universities and universities of applied sciences for the teaching of *"Polymer Testing"*.

In order to provide support the lecturers, a PowerPoint presentation has been created for all pictures and tables. It can be downloaded from *www.hanserpublications.com*. In this regard, we would like to thank Prof. Dr.-Ing. Christian Bierögel, in particular, for his valuable advice in the preparation of this edition and especially for the new publication of the pictures, which are now in colour, and his extensive work on producing the PowerPoint presentation of all pictures.

A Wiki dictionary, *"Plastics Testing and Diagnostics"*, has been produced on the scientific basis of the book and of publications from the Merseburg scientific school, and it often provides more detail than the book. The dictionary is available at *www.polymerservice-merseburg.de/wiki-lexikon-kunststoffpruefung* and can be used for practical work. An extensive compilation of fracture mechanics test specimens and approximation equations to calculate parameters in fracture mechanics are just two examples of what the dictionary offers.

We would like to thank Carl Hanser Verlag, especially Ms. Dr. N. Warkotsch, Ms. Dr. C. Strohm, Ms. Dipl.-Ing. (FH) U. Wittmann and Mr. S. Jörg, for their much appreciated and reliable assistance.

June 2013 The Editors

Preface to the First Edition

This book is based on the editors' extensive experience in research, development and education in the field of materials science and especially polymer testing, polymer diagnostics and failure analysis. The results of their work were published in several reference books about deformation and fracture behavior of polymers, in numerous single publications in peer-reviewed scientific journals and in proceedings. Given the fact that the field of science undergoes a rapid and dynamic development it seemed prudent to present these results in a textbook for students.

The following factors convinced us that a comprehensive representation of the state of knowledge was needed:

- The ever-increasing importance of this materials group for continued technical progress led to an increasing share of polymers and compounds in various applications.
- The increased safety awareness led to the development of hybrid methods of polymer diagnostics, which enable a complex view of the connection between loading and material behavior under actual loading conditions and ambient influences
- As a result of the development of fiber-reinforced thermoplastic and thermosetting composite materials, new challenges to polymer testing methods emerged.
- The increasing use of polymers and elastomers in medical technology for various applications requires the development of technological testing methods for viability, serviceability, operating safety and/or service life.
- As a consequence of the trend to miniaturization components (microsystems), more suitable testing methods are necessary for the evaluation of various thermomechanical loadings of materials properties, e.g., in highly integrated electronic components.

In addition, a number of new standards and regulatory codes for polymer testing have been introduced over the past years, further emphasizing the need for a redesigned textbook for this discipline of science. The book presents a comprehensive representation of knowledge provided by respected colleagues from universities,

universities of applied sciences and the polymer industry. A list of co-authors as well as acknowledgements for numerous colleagues and co-workers follow on separate pages.

The editors and co-authors tried hard to overcome the limits of classic polymer testing using ASTM and ISO standards in order to make the importance of polymer testing for the development and application of new polymers, composite materials and materials compounds, as well as the introduction of new technologies, more recognizable.

This book is primarily designed for students of bachelor, diploma and master courses of material science, material technology, plastic technology, mechanical engineering, process engineering and chemical engineering. It can be used by students, teachers of universities and colleges for supplementary studies in the disciplines of chemistry and industrial engineering. The methods of polymer testing are also essential to the development and application of biomedical or nanostructured materials.

With the publication of this book we hope that it will not only serve the important task of training of young scientists in physical and material oriented disciplines, but will also make a contribution to further education of professional polymer testers, design engineers, and technologists.

We thank Carl Hanser Publishers for publishing this book, entitled “Polymer Testing”, especially we are grateful to Dr. Christine Strohm who thoroughly revised the complete text for this edition. We also thank Dr. Paul I. Anderson for the translation of several chapters. The main idea of this book was based on the 1992s book by Dr. Heinz Schmiedel “Handbook of Polymer Testing”, written in German language. We kept the physical-methodical approach and also, the comprehensive chapter “Fracture Toughness Measurements in Engineering Plastics” based on our research work in this field for many years. For example it is pointed out on the extensive collection of fracture mechanics specimen and the evaluation equations for determination of fracture mechanics parameters.

We want to thank sincerely all co-workers from the Center of Engineering Science and the Institute of Polymer Materials e. V. of the Martin-Luther-University of Halle-Wittenberg and all collaborators from the Institute of Materials Science and Technology of the Vienna University of Technology who, with their commitment and their willing cooperation, made the publication of this book possible in the first place.

May 2007

Sabine Seidler, Vienna

Wolfgang Grellmann, Halle

The Authors

The Editors

Wolfgang Grellmann

Prof. Wolfgang Grellmann was the holder of the chair in Material Diagnostics/Material Testing at the Center of Engineering at Martin Luther University Halle-Wittenberg, Germany from 1995 to 2014. He is the honorary president of the foundation Akademie Mitteldeutsche Kunststoffinnovationen and chief executive officer of Polymer Service GmbH Merseburg (established as an associated institution of Martin Luther University Halle-Wittenberg, and now an associated institution of Merseburg University of Applied Sciences). From a scientific perspective, he is interessed in deformation and fracture behavior of polymers and composites, development of methods of experimental fracture mechanics, hybrid methods of non-destructive polymer diagnostics, and failure analysis. He is author or co-author of numerous books and book chapters, and reviewer of international scientific journals.

Sabine Seidler

(Source: © Raimund Appel)

Prof. Sabine Seidler is the holder of the chair in Non-metallic Materials of the Institute of Materials Science and Technology at TU Wien (Vienna, Austria) since 1996. She is the rector of the TU Wien, president of Universities Austria, vice president of the Association of the Austrian Universities of Technology “TU Austria”, and supervisory board member of Austria Metall AG as well as of the Helmholtz-Zentren Berlin, and presidium member of the foundation Akademie Mitteldeutsche Kunststoffinnovationen. From a scientific perspective, she is interested in mor-

phology–property relationships of polymers and composites. She is author of numerous papers in international scientific journals (and recognized reviewer of these journals), and author or co-author of various books and book chapters.

The Co-Authors

- Prof. Dr. *Volker Altstädt*
  University of Bayreuth, Germany
  (Chapter 10)
- Prof. Dr. *Monika Bauer*
  Fraunhofer-Einrichtung für Polymermaterialien und Composite PYCO, Teltow, Germany
  Technical University of Brandenburg (BTU), Cottbus, Germany
  (Section 11.2)
- Prof. Dr. *Christian Bierögel* (†)
  Martin Luther University Halle-Wittenberg and Polymer Service GmbH Merseburg, Institute of Martin Luther University Halle-Wittenberg, Germany
  (Chapter 2, Section 4.3 and Chapter 9)
- Prof. Dr. *Gert Busse*
  University of Stuttgart, Germany
  (Chapter 8)
- Prof. Dr. Dr. *Klaus Friedrich* (†)
  Institute for Composite Materials (IVW), Technical University of Kaiserslautern, Germany
  (Section 4.8)
- Dr. *Henrik Höninger*
  IMA Materialforschung und Anwendungstechnik Dresden, Germany (formerly)
  (Sections 4.5, 4.6 and 11.3)
- Dr. *Thomas Lüpke*
  Kunststoff-Zentrum (KUZ) Leipzig, Germany
  (Sections 4.1 and 4.2)
- Prof. Dr. *Bernd Michel*
  FhG Institute for Reliability and Microintegration (IZM) Berlin, Germany
  (Chapter 13)

- Prof. Dr. *Hans-Joachim Radusch*
  Martin Luther University Halle-Wittenberg, Germany
  (Chapter 3)
- Dr. *Falko Ramsteiner*
  BASF Group Ludwigshafen, Germany (formerly)
  (Chapter 7)
- Prof. Dr. *Katrin Reincke*
  Polymer Service GmbH Merseburg, Germany *(www.psm-merseburg.de)*
  (Chapter 12)
- Prof. Dr. *Andreas Schönhals*
  Federal Institute for Materials Research and Testing (BAM) Berlin, Germany
  (Section 6.3)
- Dr. *Jörg Trempler*
  Martin Luther University Halle-Wittenberg, Germany (formerly)
  (Section 6.2)

The chapters and sections not listed above were written by the editors.

We owe particular gratitude for their assistance with the development and compilation of the manuscript to:

- Dipl.-Ing. *Yvonne Chowdhury,* InnoMat GmbH, Teltow, Germany (Section 11.2),
- Dipl.-Ing. *Ivonne Pegel,* ESW GmbH, Wedel, Germany (Chapter 10) and
- Dr. *Hans Walter,* FhG Institute for Reliability and Microintegration (IZM) Berlin, Germany (Chapter 13).

In particular we would like to thank co-author Prof. Dr. *Christian Bierögel* (†) not only for his contributions to the book, but moreover for his comprehensive assistance and critical advice during the composition of the manuscript.

For the critical revision of single chapters we thank our longtime co-workers Ao. Prof. Dr. mont. *Vasiliki-Maria Archodoulaki,* Dr. *Thomas Koch*, Prof. Dr. *Ines Kotter* and Dr. *Ralf Lach*.

Finally, we thank Dr. *Mark Smith* from Carl Hanser Verlag for his much-appreciated and reliable assistance during the revision of the 3rd edition of the textbook.

Contents

1 Introduction

1.1 The Genesis of Polymer Testing as a Science

The development of polymer testing is intimately involved with the economic rise of the polymer industry. The spectacular progress that has taken place in macromolecular chemistry since the 1920s owes much to the efforts of the polymer chemists *Hermann Staudinger* und *Karl Ziegler*. The awareness of how to use macromolecules as materials is based on research into methods of synthesizing both to produce new monomers and polymers, as well as to introduce new catalyst systems. This in turn necessitated systematic basic research to uncover the fundamental principles affecting polymer synthesis and structure, on the one hand, and microscopic structure and macroscopic properties, on the other. Uncovering the interrelationships between microstructure and macroscopic properties, especially the mechanical and thermal properties, is of course one of the fundamental tasks of *polymer testing*.

The worldwide boom in the plastic industry began in the 1950s when the industry reduced costs and raised profitability by shifting to petroleum for its raw material base. Today, plastics are finding applications in almost all areas of human activity. Despite the considerable problems involved in disposing and recycling of plastics, the area of applications for this group of materials continues to expand. The worldwide increase in the production of plastics and the range of monomers being utilized has altered the economic significance of these materials to such a degree that historians are beginning to speak of a dawning “Age of Polymers”. The image of plastics, at first considered as a “substitute material”, an alternative for the “real thing”, is now that of an innovative material for economically indispensable structural and functional applications. Without the wide range of modern polymer materials and their composites, the progress made in microelectronics, microsystem technology and even nanotechnology would be quite unthinkable.

According to a 2004 European study entitled “Plastics – Pathmaker of Progress”, worldwide production of plastics surpassed the production of crude steel in terms of volume for the first time at the end of the 1980s due to the exceptionally dynamic growth rate of plastic materials.

By 2017, worldwide production of crude steel amounted to 217×10^9 dm^3, far less than the 348×10^9 dm^3 achieved by plastics. Since 8 kg of crude steel and 1 kg of plastics both correspond to 1 dm^3 of each, one can clarify the relationship between these quantities by a simple conversion. The result is approx. $1{,}691 \times 10^6$ t of crude steel produced and 348×10^6 t of plastics.

The worldwide consumption for all “commodities”, such as PE-HD, PE-LD/LLD, PP, PVC and PS shows annual growth rates continuously increasing. For several “engineering plastics”, such as PC and especially PET, average growth rates are above the ordinary. Some years ago, PP took over the role of market leader for bulk plastics. At the same time, the wide variety of application areas, especially for bottles in the food and pharmaceutical industries, will lead to a high increase in PET consumption. The steep production increase of PET will transform this material from a “technical” to a “standard” or “commodity” polymer. In this age of misleading use of technical terms, society’s growing acceptance of this class of materials is evidenced, for example, by the largely proper use of the designation “PET bottle”.

The main application area for innovative materials lies in the automotive industry. According to the results of a market research study of various automotive manufacturers, the trend in materials’ use in automotive manufacturing in the last years will show the following changes:

- Steel: 10 % reduction
- Aluminum: 100 % increase
- Plastics: 5 % increase
- Glass: constant at 2 %

The mass share of plastics will then be as high as 20 %. The trends in auto manufacture continue toward the so-called hybrid applications, i. e., metal parts encapsulated by plastics, metal-plastics sandwich structures, components with steel or plastic cores in doors and hoods, new modules of component integration and material combinations (multi-component injection molding).

Despite the undoubted advantages of polymer materials, this group of materials has been the subject of public discussion in recent years due to ecological aspects, especially in packaging applications. Worldwide, 5 to 13 million tonnes of plastics land in the sea every year – that is 1.5 % to 4 % of the world’s plastics production. As in other areas, the market for plastics applications is primarily determined by economic competition, which often hinders the further development of processes. Country-specific, region-specific and even non-existent collection systems have a

negative impact. Sorting is not always in line with the complexity of the products, and their technical possibilities are often not fully exploited. The recyclate is often a product of inferior quality; high-quality regranulate is almost as expensive as virgin material, i. e. economic incentives are often lacking. Nevertheless, a view solely based on the recyclability of materials is one-sided, the complete material cycle must be considered. Since a holistic ecological assessment also takes production into account, comparisons between different products may well lead to constellations where a higher ecological benefit is associated with a lower recyclability. This is the case, for example, if significantly fewer resources are used in the production of a product and the poorer recyclability is accepted in return. The last aspect to be mentioned at this point is the participation fees. These are levied on a mass basis, i. e. there is already a strong incentive to reduce weight. This remains dominant, even if recyclability becomes a victim. For plastics applications, as for all other material applications, the responsible use of resources must be the top priority.

Beginning in the 1950s, parallel to the enormous worldwide growth in production, the clamor grew ever louder for scientific parameter capable of quantifying the relationship between microstructure and macroscopic properties. The efficient use of materials requires the complete utilization of material properties which, in turn, necessitates the development of adequate, meaningful measuring and testing procedures. This required improvements in the informative content of the methods which only continuously advancing electronic technology could provide. The classic testing procedure, e. g., for determining hardness and toughness of plastics, developed into the instrumented hardness test methods and the instrumented toughness test methods, e. g., the instrumented *Charpy* impact test. All instrumented methods have one aim in common: to electronically acquire the constituents of deformation – force and elongation and/or deflection – with the highest possible degree of precision, and to utilize the improved information content for a differentiated evaluation of material behavior. These experimental methods for providing structurally sensitive material parameters have only been widely developed within the last twenty years. In many cases, it is not possible to adopt standard processes for the testing of metals, since the measurement ranges for directly measured characteristics may differ by ranges of magnitude, i. e., the demands placed on the required measurement techniques are correspondingly different.

For some time, there was no generally accepted term for this specific discipline in the literature, even though the subject matter is defined by its content. In its early years, presentations began with detailed descriptions of the structure of plastics and polymer processing, before these two developed into individual scientific disciplines. Today, the concept of *polymer testing* has found general acceptance, just as the testing of plastics and plastic components has gained great significance in the plastics industry. In the last 35 years, a plethora of empirically acquired facts and

experience has been collected that are being viewed, as much as possible, from a uniform perspective based on the insights of material science. Theoretical assumptions are no longer made before they have been confirmed by experimental results.

Like all other technical scientific disciplines, *polymer testing* has a decidedly interdisciplinary character (see Figure 1.1). Figure 1.1 clearly shows how polymer testing functions to provide a link between the *synthesis* and *processing* of polymers, on the one hand, and between the *characterization/analytics* of polymers and their *morphology/micromechanics,* on the other. Although the terms plastics and polymers are often used synonymously. Figure 1.1. follows corresponding usage.

Methods for measuring fracture behavior are necessary to satisfy growing demands for reliability, safety and service life of machines, plants and components, as well as to eliminate breakage as one of the most common material-related causes for plastics failure. This involves using the methods of *engineering fracture mechanics*. The current level of research on this is provided, for example, in Chapter 5 for plastics and in Chapter 10 for composite materials. Within the polymer sciences, the independent areas of *polymer material science* and *plastics engineering* have found secure niches, as can be ascertained from the course schedules at institutions offering degrees in polymer technology, as well as from the literature references at the end of this chapter. Designing with polymers is the subject matter of *plastics engineering*, whereby the designer of polymer-based products is increasingly faced with the task of selecting dimensions and shape according to data derived from research in material science.

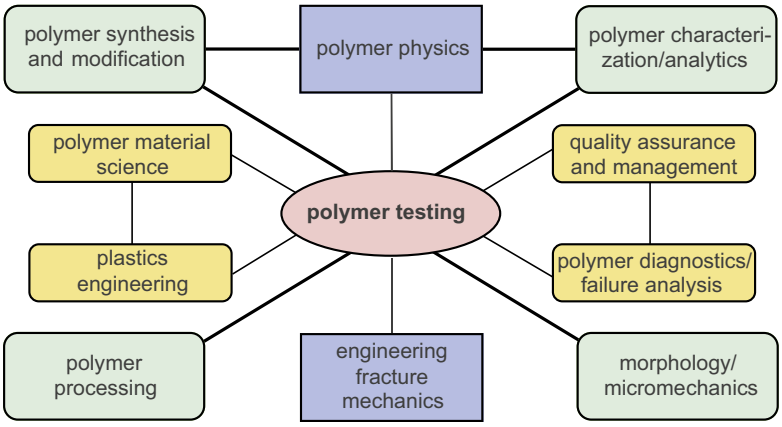


Figure 1.1 The interdisciplinary character of polymer testing

Also increasing in importance are the disciplines of *quality assurance* and *quality management,* whereby the totality of quality-relevant activities falls under quality management. One essential element is the quality test which itself can take on

many forms. One important, but technically hard to achieve step consists in integrating the polymer testing procedures in-line into each particular production process with the goal of assuring, as much as possible, that quality requirements are being fulfilled by the product and the process. *Polymer diagnostics/failure analysis* involves combining the various methods for investigating material composition (*analytics*), structural make-up, mechanical, thermal, electrical and optical properties, as well as environmental influences. Chapter 9 presents the areas of emphasis for hybrid methods of polymer diagnostics, by which one can understand the in-situ combination of mechanical and fracture-mechanical experiments with non-destructive testing methods, such as ultrasonic testing analysis, thermography or laser extensometry. The goal remains to increase the informational value of classic testing methods and to derive methods for quantifying states of damage and their thresholds.

1.2 Factors Influencing Data Acquisition

Processing has a substantial effect on the structural formation in plastics and the properties resulting from it. This pronounced sensitivity to processing is a fundamental reason for the limited value of available parameter on polymers. Therefore, the internal structure of polymeric solids and the properties describing them are not solely dependent on their chemical composition.

The problems involved in acquiring characteristic values arise from the fact that we do not ascertain the properties of the material (molding compound) to be tested, but the properties of specimens made from that material under conditions determined by the processing method. Thus, there is no assurance that values acquired from specimens or components with specified geometries can be applied to components with different geometries due to various internal conditions. The influencing factors are listed extensively in Chapter 2, The Production of Specimens. Besides the molding compounds themselves, the most important of these include factors involved in producing specimens, in specimen geometry and testing conditions. The most significant factors for plastics are testing temperature, test rate and environmental factors, the simplest being humidity. Due to the large number of factors influencing the test results, values on polymers are reproducible only if they are acquired on the basis of comparable chemical and physical structure, similar geometric conditions and the same testing methods. Thus, values must always be acquired on a structural basis. The uniqueness lies in the fact that these influencing factors do not work individually, but one has to assume a complex functionality among the parameters listed. Consequently, both the quantitative measurement of

all individual marginal conditions as well as their complex interactions are significant for the overall evaluation of properties. Evaluation makes use of values based on standardized test methods that guarantee comparability and thus ensure that products are suitably classified. Knowledge of fundamental scientific relations in the formation of properties and their description in terms of values is always tied to a hierarchical perspective on molecular structure and morphogenesis during processing.

Innovative testing methods based on scientific discoveries with theoretical backgrounds are often better suited for evaluating and optimizing polymers than methods used in industrial testing practice, i.e., based on empirical experience. One such example is the replacement of the conventional *Charpy* V-notch impact test (see Section 4.4) by the instrumented impact test that provides geometry-independent fracture-mechanical values (cf. Section 5.4.2).

In summary, it can be stated that the fundamental task of polymer testing lies in clarifying the relations between microstructure and the physical-technical properties of polymers while taking their physical and chemical description into consideration.

1.3 Classification of Polymer Testing Methods

As in general material testing, several content-oriented perspectives can be given for classifying the experimental methods of polymer testing. In polymer testing, the distinction between destructive and non-destructive is again the primary criterion of classification. Potential categories for classifying destructive polymer testing include:

- Rate of test procedure
  - Static, quasi-static and dynamic load
- Type of load
  - Tensile, compression, bending, torsion and shear load
  - Uni- and biaxial or multiaxial load
- Type of material to be investigated
  - Polymers and fiber composite materials
- Type of physical property
  - Thermal, optical, electrical and dielectrical properties.

In addition, individual mechanical material testing methods have distinctive features that simplify the characterization of the various methods in use. For example in hardness testing, the methods of acquiring the indentation process or magnitude of indentation and/or testing force and indentation depth serve as criteria by which we can distinguish between conventional and instrumented hardness testing, and between macro-, micro- and nanohardness.

In the area of *mechanical material testing*, the rate of the test procedure is used as a classifying criterion. In static testing procedures, it is not assumed that various testing rates affect the test results, whereas in quasi-static testing, a slowly increasing testing force is a rate-related influence. Compared to quasi-static loading, results of dynamic testing are expected to be influenced considerably by the test rate. Therefore, we distinguish between the following test methods:

- Static test methods,
- Quasi-static test methods, and
- Dynamic test methods (shock and impact type, fatigue).

Within the test rate ranges, tests can be divided according to the type of load involved:

- Tensile test,
- Compression test,
- Bending test.

Besides these load types commonly applied in quasi-static loading, torsion and shear tests are also performed.

Independently of the particular type of load, the differences in testing methods between polymers and composite materials, as well the depth of knowledge to be presented in this book, require that the testing of composite materials be presented separately (see Chapter 10).

The special discussion of environmental stress cracking resistance in Chapter 7 and the evaluation of toughness of polymers using fracture-mechanical methods in Chapter 5 can be classified as mechanical material testing.

An essential additional criterion in the testing of fiber composite materials is the orientation of fibers relative to the main direction of load. The anisotropy of these materials requires special mechanical testing methods that are often defined according to the specimens developed for them. Some examples of these are the:

- Boeing compression test method,
- Celanese test method,
- IITRI test method,
- Two- and three-rail shear test,

- *Iosipescu* thrust test, and the
- Plate-twist thrust test.

The peculiarities in composite structure and complex loads are reflected in the fracture-mechanical testing of composites that require evaluation of the crack growth behavior with regard to their interlaminar fracture modes (mode I, mode II and mixed mode). To do so, special specimens are defined as criteria while they also provide a foundation for a special method whose goal is to provide geometry-independent fracture-mechanical material parameters. Such parameters have proven to be of assistance for optimizing the toughness of fiber composite materials and are a prerequisite for dimensioning products made from fiber composite materials.

1.4 Standards and Regulatory Codes for Polymer Testing

Globalization and expansion of markets into new economic zones, demands for shorter development times, generally shorter life-service cycles of products and requirements resulting from the increasing technological convergence are all having their effect on the trends in setting national and international standards:

- Standards and standardization become market- and need-oriented to achieve strategic and economic advantages in international competition.
- Standards and standardization are strategic instruments for supporting the success of the economy and society.
- Standards and standardization reduce the need for governmental regulation.
- Standards and standardization, as well as standards committees, support technological convergence.

In order to satisfy these demands, the Standards Committee on Material Testing (NMP) within the DIN organization (German Institute of Industrial Standards) is working on a new strategy for formulating standards. In order to ensure repeatability and reproducibility of procedures used in material testing, and thus in polymer testing, standards for the performing of tests and requirements for test equipment and specimens were set. In fulfillment of the fundamental principles for establishing standards laid down in DIN 820-1 to 4, the standards to be established shall support efficiency and quality assurance in business, technology, science and administration. Thus, standards serve the safety of people, equipment, technologies and processes, on the one hand, while they provide a means for targeting improvements in quality in all areas of life and business. Such standards have by no means

the force of law, but rather provide all users with "accepted rules of technology". Their use makes it easier to compare product properties or production methods. The basis for comparison, however, is the ability of all members of the standards community to meet the technical and scientific demands of the standards. The results of the work on standards in the DIN are the national standards published under the association logo DIN. In consequence of the harmonization of international (ISO) and European (EN) standards, the DIN EN, DIN ISO and DIN EN ISO standards also enjoy the status of national standards. In addition to these standards, various manufacturers and user organizations publish guidelines or process recommendations for plastics producers which amount to defined, but not standard supplements. In this connection, considerable importance is attached to automotive manufacturers' quality requirements for original equipment manufacturers (e.g., GME: General Motors Specification, DBL: Daimler Benz Specification, BMW N: Bavarian Motor Works Specification) that amount to binding instructions for their suppliers.

The ASTM (American Society of Testing and Materials) standards, which include standard test methods and procedures for testing polymers and composites (see Chapter 10) are widely spread in the American speech area. ASTM International is a non-profit organization founded in 1898. It provides a global forum for the development and publication of standards and test methods. Its membership is comprised of producers, users, consumers, and representatives of government and academia. ASTM International provides standards that are accepted and used in research and development, product testing, and quality systems. Within ASTM, the primary responsibility for plastics lies with a committee designated for this purpose. This committee, called D-20 on Plastics, is responsible for more than 500 standard test methods, recommended practices, and guides. One of the key components of D-20 is the continuous review and updating of existing documents and the authoring of new protocols that are necessary. It has to be pointed out that there are a number of fundamental differences between ASTM and ISO standards especially referring to specimen geometry and dimensions in addition to test condition requirements. Due to the large number of factors influencing the test results of polymers determined with different standards are not comparable.

Test laboratories can gain what amounts to formal recognition of their competency and admission to perform clearly defined tests on the basis of standards or verified test specifications if they obtain accreditation by their national accreditation organization (e.g., the American National Standards Institute (ANSI) in the US).

ISO/IEC standard 17025 states the criteria for judging organizational structure, employment of test personnel and technical facilities, preparation of test reports, as well as work procedures for testing and calibrating laboratories. Fulfillment of these standards requirements constitutes recognition for a quality management system according to ISO 9001 or 9002, whereby accreditation according to ISO/IEC

17025 is not the equivalent of certification according to ISO 9001 or 9002. Together with the introduction of this standard, the determination of measuring uncertainty is advanced as a main criterion for the application of test results in quality assurance and design.

Testing laboratories whose competency has been certified in one domain according to ISO/IEC 17025 and that have their own test standards based on wide experience, can have these judged by their national agencies within the framework of accreditation. Such special testing procedures as the MPK procedures for the instrumented notched impact test (MPK-ICIT) and the instrumented free-falling dart test (MPK-IFDT) are used to illustrate data acquisition in Chapters 4 and 5.

The basic activities in the areas of material or polymer testing can best be described by the concepts of measurement and testing. Measurement is an experimental procedure based on one or more physically effective principles from which a specific value (data bit) is acquired as the multiple of a unity or of a defined reference value, supplemented by its own measurement uncertainty. To test means to determine whether the acquired value including their measurement uncertainty meet one or more specified requirements (tolerances or error limits). Since measurable characteristics are acquired as value by most modern processes of material and quality testing and compared with corresponding requirements, “measuring testing” is defined as the opposite of “counting testing”.

Important measures for ensuring reproducibility of testing methods include adjustment, calibration and gauging. Round-robin tests among several test laboratories using suitable reference specimens can serve as an additional measure. Adjustment is the balancing of test equipment that must not be done by the operator of the equipment and which guarantees that measurement discrepancies will be minimal and/or that error limits will be maintained. Calibration means testing under comparable conditions and the ability to return the result to international reference values, in order to determine the true or correct measurement value while taking systematic variations into account. Gauging is a procedure in which a competent gauging office confirms that a testing or measuring device satisfies the stated requirements or regulations according to law as regards its character and measurement-technical characteristics (e.g., class of equipment). Calibration and gauging have to be repeated by the gauging office or device operator at regular intervals in order to ensure that error limits are maintained.

An essential legal basis for material testing is provided by national laws pertaining to liability for defective products. According to ISO 9000, a defect is defined as a non-conformity, i.e., the non-fulfillment of set demands. The following necessary measures for material and polymer testing derive from the product liability law:

- Use-related relevant and informative test procedures and methods must be selected,

- Design and assembly must be suited for testing and/or readily testable,
- Testing must be done by agreed upon methods with meaningful results,
- Test results must be evaluated with regard to proper intended use,
- Product and process observation, anticipatory error prevention and, if required, failure analysis.

1.5 Compilation of Standards

DIN 820 (2014–2022)	Standardization ■ Part 1 (2022): Principles (Draft) ■ Part 2 (2022): Presentation of Documents (Draft) ■ Part 3 (2021): Terms and Definitions ■ Part 4 (2021): Working Procedure ■ Part 11 (2020): Presentation of Standards Concerning Safety Regulations which are VDE-Specifications or VDE-Guidelines ■ Part 12 (2014): Guidelines for the Inclusion of Safety Aspects in Standards (ISO/IEC Guide 51: 2014)
ISO 9000 (2015)	Quality Management Systems – Fundamentals and Vocabulary
ISO 9001 (2015)	Quality Management Systems – Requirements
ISO 9004 (2018)	Quality Management – Quality of an Organization – Guidance to Achieve Sustained Success
ISO/IEC 17025 (2017)	General Requirements for the Competence of Testing and Calibration Laboratories

References by Area of Specialization

Polymer Synthesis and Modification

[1.1] Braun, D.; Cherdron, H.; Rehahn, M.; Ritter, H.; Voit, B.: *Polymer Synthesis: Theory and Practice. Fundamentals, Methods, Experiments*. Springer, Berlin (2013)

[1.2] Elias, H. G.: *Macromolecules. Volume 1, Chemical Structures and Synthesis*. Wiley VCH, Weinheim (2005)

[1.3] Elias, H. G.: *An Introduction to Plastics*. Wiley VCH, Weinheim (2003)

[1.4] Rempp, P.; Merill, E. W.: *Polymer Synthesis*. Wiley VCH, Weinheim (1991)

[1.5] Odian, G.: *Principles of Polymerization*. Wiley VCH, Weinheim (2004)

Polymer Characterization/Analytics

[1.6] Arndt, K.-F.; Müller, G.; Schröder, E.: *Polymer Characterization*. Carl Hanser, Munich (1989)

[1.7] Wagner, M.: *Thermal Analysis in Practice – Fundamental Aspects*. Carl Hanser, Munich (2017)

[1.8] Hamid, S. H. (Ed.): *Handbook of Polymer Degradation*. Marcel Dekker, New York Basel (2000)

[1.9] Braun, D.: *Simple Methods for Identification of Plastics*. Carl Hanser, Munich (1999)

[1.10] Turi, E. (Ed.): *Thermal Characterization of Polymeric Material*. Academic Press, San Diego London (1997)

Polymer Processing

[1.11] Osswald, T.; Hernandez-Ortiz, J. P.: *Polymer Processing*. Carl Hanser, Munich Cincinnati (2006)

[1.12] Michaeli, W.; Greif, H.; Kretzschmar, G.; Ehrig, F.: *Training in Injection Molding*. Carl Hanser, Munich (2001)

[1.13] Chung, C. I.: *Extrusion of Polymers. Theory and Practice*. 3rd Ed., Carl Hanser, Munich (2019)

Morphology/ Micromechanics

[1.14] Michler, G. H.: *Atlas of Polymer Structures. Morphology, Deformation and Fracture Structures*. Carl Hanser, Munich (2016)

[1.15] Stellbrink, K. K.U.: *Micromechanics of Composites – Composite Properties of Fibre and Matrix Constituents*. Carl Hanser, Munich (1996)

[1.16] Woodward, A. E.: *Understanding Polymer Morphology*. Carl Hanser, Munich (1994)

[1.17] Michler, G. H.; Balta-Calleja, F. J.: *Nano- and Micromechanics of Polymers: Structure Modification and Improvement of Properties*. Carl Hanser, Munich (2012)

Polymer Material Science

[1.18] Menges, G.; Osswald, T. A.: *Materials Science of Polymers for Engineers*. Carl Hanser, Munich (2003)

[1.19] Domininghaus, H.: *Plastics for Engineers: Materials – Properties – Applications*. Carl Hanser, Munich (1992)

[1.20] Birley, A. W.; Batchelor, J.; Haworth, B.: *Physics of Plastics: Processing, Properties and Materials Engineering*. Carl Hanser, Munich (1992)

[1.21] Ehrenstein, G. W.: *Polymeric Materials: Structure – Properties – Applications*. Carl Hanser, Munich (2001)

Plastics Engineering

[1.22] Erhard, G.: *Designing with Plastics*. Carl Hanser, Munich (2006)

[1.23] Grewell, D.; Benatar, A.; Park, J. B.: *Plastics and Composites Welding Handbook*. Carl Hanser, Munich (2003)

[1.24] Rosato, D. V.: *Designing with Reinforced Composites*. Carl Hanser, Munich (1997)

[1.25] Rao, N. S.; Schumacher, G.: *Design Formulas for Plastic Engineers*. Carl Hanser, Munich (2004)

[1.26] Rotheiser, J.: *Joining of Plastics – Handbook for Designers and Engineers*. Carl Hanser, Munich (2004)

[1.27] Belofsky, H.: *Plastics: Product Design and Process Engineering*. Carl Hanser, Munich (1995)

Quality Assurance and Management

[1.28] Masing, W.: *Handbuch der Qualitätssicherung*. Carl Hanser, Munich (1990)

[1.29] Keating, M.: *How to Assure Quality in Plastics*. Carl Hanser, Munich (1995)

[1.30] Dealy, J. M.; Saucier, P. C.: *Rheology in Plastics Quality Control*. Carl Hanser, Munich (2000)

[1.31] Gordon, M. J.: *Total Quality Process Control for Injection Molding*. Carl Hanser, Munich (1992)

[1.32] Kerkstra, R.; Brammer, S.: *Injection Molding Advanced Troubleshooting Guide. The 4M Approach*. 2nd. Ed., Carl Hanser, Munich (2021)

[1.33] Pfeifer, T.: *Quality Management – Strategies, Methods, Techniques*. Carl Hanser, Munich (2002)

Polymer Diagnostics/Failure Analysis

[1.34] Ezrin, M.: *Plastics Failure Guide – Cause and Prevention*. 2nd Ed., Carl Hanser, Munich (2013)

[1.35] Moalli, J. (Ed.): *Plastics Failure – Analysis and Prevention*. Society of Plastics Engineers, Plastics Design Library, Norwich, NY (2001)

[1.36] Ehrenstein, G. W.; Engel, L.; Klingele, H.; Schaper, H.: *Scanning Electron Microscopy of Plastics Failure. Rasterelektronenmikroskopie von Kunststoffschäden*. Carl Hanser, Munich (2011)

Polymer Physics

[1.37] Strobl, G.: *The Physics of Polymers: Concepts for Understanding their Structures and Behavior*. Springer, Berlin (1997)

[1.38] Argon, A. S.: *The Physics of Deformation and Fracture of Polymers*. Cambridge University Press (2013)

[1.39] Bower, D.: *An Introduction to Polymer Physics*. Cambridge University Press (2002)

Engineering Fracture Mechanics/Materials Testing

[1.40] Blumenauer, H. (Ed.): *Werkstoffprüfung*. Wiley VCH, Weinheim (1994)

[1.41] Antolovich, S.: *Fundamentals of Fracture Mechanics*. Elsevier, Amsterdam (2006)

[1.42] Anderson, T. L.: *Fracture Mechanics. Fundamentals and Applications*. 3rd Ed., CRC Press, Boca Raton (2005)

Polymer Testing

[1.43] Brown, R. (Ed.): *Handbook of Polymer Testing: Physical Methods*. Marcel Dekker, New York Basel (1999)

[1.44] Grellmann, W.; Bierögel, C.; Reincke, K. (Eds.): Wiki "Lexikon Kunststoffprüfung und Diagnostik", Version 12.0 (2022), *wiki.polymerservice-merseburg.de*

[1.45] Ehrenstein, G. W.; Trawiel, P.; Riedel, G.: *Thermal Analysis of Plastics – Theory and Practice*. Carl Hanser, Munich (2004)

[1.46] Hylton, D. C.: *Understanding Plastics Testing*. Carl Hanser, Munich (2004)

[1.47] Shah, V.: *Handbook of Plastics Testing Technology*. Wiley VCH, Weinheim (1998)

[1.48] Swallowe, G. M.: *Mechanical Properties and Testing of Polymers – An A–Z Reference*. Kluwer Academic Publishers, Dordrecht Boston London (1999)

Handbooks/Data Banks

[1.49] Baur, E.; Osswald, T. A.; Rudolph, N. (Eds.): *Plastics Handbook – The Resource for Plastics Engineers*. Carl Hanser, Munich (2019)

[1.50] Carlowitz, B.: *Tabellarische Übersicht über die Prüfung von Kunststoffen*. Giesel Verlag für Publizität, Isernhagen (1992)

[1.51] Grellmann, W.: Seidler, S. (Eds.): *Mechanical and Thermomechanical Properties of Polymers*. Landolt-Börnstein. Volume VIII/6A3, Springer, Heidelberg (2014)

[1.52] Rosato, D. V.: *Rosato's Plastics Encyclopedia and Dictionary*. Carl Hanser, Munich (1992)

Computer-Aided Polymer Data Banks

[1.53] Data sheets in Campus® plastics data base 5.2, *www.campusplastics.com*

[1.54] Mbase-Material Data Center in *www.m-base.de* or *www.materialdatacenter.com*

[1.55] Polymers: A Property Database: *https://poly.chemnetbase.com*

[1.56] Data sheets in MatWeb-Material property data, *www.matweb.com*

[1.57] Neue Materialien – Der Marktplatz der Werkstofftechnik: *http://www.neuematerialien.de*

[1.58] Material Data Base FORMAT (data base not online available)

[1.59] Mechanical and Thermomechanical Properties of Polymers. Springer Materials, The Landoldt-Börnstein Database: *https://materials.springer.com*

2 Preparation of Specimens

2.1 Introduction

The main tasks of polymer testing consist of the investigation, evaluation and characterization of various materials and the provision of data with their corresponding measurement uncertainty. Polymers can be tested in powder or granulate form, or as specimens, semi-finished or finished products, or component parts.

By molding material we mean un- or pre-shaped materials that are processed and shaped into semi-finished or finished products by means of mechanical loading and raised temperatures. Plastic moldings are products that can be manufactured from molding materials by compression molding, transfer molding or injection molding with subsequent cooling in completely enclosed tools [1.52, 2.1].

Characteristic chemical, physical and mechanical material values are required to identify and classify polymers for use in quality assurance, comparison and selection of materials, as well as for predicting the properties of molded parts. From this perspective, the data to be acquired create a link, however limited, between material properties and conditions of load. For a component part to be able to fulfill its function during the service life expected of it, the property profile of the material in the part must be in equilibrium with the requirement profile containing the sum of all loads acting on the part. As a rule, the component requirement profile, such as its range of loading, dimensional stability, or thermal and medial load, can be established rather precisely. By contrast, the property profile of the polymer in a particular component part depends on a variety of influencing factors. The most important of these factors are:

- Structural parameters
  - Molecular structure of the polymer (constitution, conformation, configuration)
  - Molecular weight and molecular weight distribution
  - Morphology
  - Orientation of the polymer and its fillers or reinforcing materials

- Residual stresses and their distribution
- Additives (e. g., stabilizers, coupling agents) and fillers (e. g., talcum, chalk)
- Reinforcing materials (e. g., glass, carbon, mineral and natural fibers)
- Long period and tie-molecule density
- Degree of crystallinity

- Geometric parameters
  - Shape and dimensions
  - Notches and sink marks
  - Flow and weld lines
  - Inhomogeneities (e. g., cavities, inclusions, agglomerations)
- Load parameters
  - Type of load (tension, compression, bending, multi-axial loading)
  - Duration and rate of load (creep and impact behavior)
  - Load frequency
  - Temperature and thermal spalling load
  - Environmental influences (humidity, UV radiation, etc.)

The variety and number of potential parameters underlines the necessity for precise survey of all boundary conditions. On the other hand, it also shows that only a multiparametric description of polymer properties provides a sensible approach to designing component parts and evaluating their durability. However, it also means that, instead of simple values, characteristic functions dependent on various parameters should be applied (see CAMPUS data bank [1.53] for polymers). The main reason for the limited informational value of the applied data and their limited transferability to component behavior lies in the pronounced processing sensitivity of these materials. Consequently, the material properties in the component, and the specimen as well, depend not only on the chemical composition, but are decisively influenced by prehistory, i. e., by the type and method of transition from molten to solid state during the molding process.

For testing practice, this means that values can be measured reproducibly only if they are acquired on the basis of comparable chemical and physical structure, identical geometric conditions, as well as identical testing method and even testing technology. In other words, this means that the presented values do not represent the molding material properties of the investigated material, but rather the properties of a specimen produced from this material under conditions determined by the molding procedure that is not identical with the technological properties of any molded component.

Moreover, this statement implicitly demands structure-related acquisition of material values, as well as a clear distinction between the properties of the molding material and those of the molded component.

2.2 Testing Molding Materials

Molding materials properties are determined essentially by their chemical structure and the process used for producing them, and are thus almost completely free of influencing by geometry and prehistory. However, this is only the case when their manufacturing is not followed by extrusion with subsequent granulation involving processing additives or by the inclusion of reinforcing or filler materials.

This type of testing corresponds to an analytic task that provides both information as to chemical and physical structure of the polymer as well as characteristic rheological and processing values. These physical testing methods are not just used for analytical characterization. They also represent the determinative basis for establishing correlations between the structure of macromolecular materials, their manufacturing and processing conditions, as well as their technological properties. Among the typical industrial methods that are used in receiving inspection and provide indicative data on the structure and/or classification of polymers are: density measurement, determination of melting and glass transition temperature, incineration as well as burn and/or pyrolysis tests that may be supplemented by spectroscopic test methods such as infrared spectroscopy.

The evaluation of the processability of polymers can be performed with simple technological or more advanced rheological testing methods, depending on the specific task and the type of material. Especially significant from the standpoint of industry are analysis of particle size and measurement of viscosity or melt flow index. Due to the well-known correlation between molecular mass, molecular mass distribution, macromolecular structure and engineering properties such as strength, ductility, toughness and density, conclusions can be drawn from the characteristic values acquired as they relate to the influence of machining and molding processes on chemical degradation. More detailed information on individual testing methods and their applicability and meaningfulness for various polymers is provided in [1.6–1.9, 1.19, 1.21, 1.45, 2.1–2.3] and in Chapter 3.

Sampling procedure is of decisive importance for characterizing molding material properties, since the statistical lot removed from the specimen – generally a small amount of material – is supposed to represent the universe of properties. The precision of property characterization depends mainly on the type and method of sampling, in addition to the measurement technique used. If no suitable sample

splitter or divider is available, the total amount to be characterized has to be mixed thoroughly, after which samples have to be removed at three points sufficiently far away from the surface. Sampling at different points ought to compensate for transport and storage changes in particle size distribution, in moisture and segregation effects. If thorough mixing is not possible, such as is the case for silo storage, specimens should be taken similarly at several depth levels. Moisture measurement can then be performed online, for example using moisture sensors. By using trace moisture analyzers, the moisture of bulk materials can be controlled. A common technique for sampling granulates and powders is the so-called quartering [1.43, 2.3]. In order to expedite allocation and tracing, all characterizing features (type of polymer, sack number, filling date, type and condition of packaging, batch number, etc.) have to be documented when preparing the test report. Analogous to materials processing, pre-treatment suited to the material has to precede the test, for example, to remove any water condensation or foreign particles from storage and to obtain a defined reference condition.

2.3 Specimen Preparation

2.3.1 General Remarks

Precisely specified specimens are an operational requirement for characterizing the properties of polymer molding materials by means of mechanical, thermal or electrical parameters. They must correspond to the relevant standards and meet specifications regarding the dimensions and condition of such specimens. Such specimens can be produced separately or together with a component or plastic part, or be taken from one, e.g., to investigate the property profile in the molded part or for failure analysis [1.33]. The following list provides the direct and indirect processes commonly used in forming technology:

- Direct shaping processes
  - Injection molding
  - Injection stamping
  - Compression molding
  - Casting
- Indirect shaping processes
  - Extrusion
  - Calendering

- Stamping
- Cutting

Industrial progress has also created new, combined production methods (e. g., pultrusion) that will not be presented individually. Additional processes which, however, cannot be clearly classified, include laminating, film blowing or subsequent thermal treatment (tempering).

Regardless of the type of shaping process, energy-elastic, entropy-elastic and viscous deformations peculiar to the material occur during production. These deformations are caused by shearing, e. g., during the injection and flow process, stretching and orienting of macromolecules, as well as by cooling and curing sequences in the tool. They also have a decisive effect on the subsequent internal state of the component part or specimen. Energy-elastic deformation is due to reversible changes in the conditions of oscillation and rotation of atoms and parts of the macromolecules, and is consequently time-dependent. Entropy-elastic deformations correspond to changes in entropy, i. e., the conditions of internal order, whereby translatory movements take place in chain segments at increased temperatures. These processes are reversible, albeit time and temperature dependent. Irreversible viscous deformations are caused by plastic shear in macromolecules due to shearing and/or stretching during the production process (see Chapter 4).

During the transition from the molten to the solid state, the material undergoes a specific volume shrinkage, also called process shrinkage. It has to be compensated by a corresponding oversizing of the mold design [2.4]. Shrinkage affects dimensional stability and tolerance; it is typically less pronounced in filled or reinforced materials than in the matrix materials.

Depending on the complexity of the plastic part, these various processes generally determine the uneven distribution of internal stresses (residual stress) and matrix/filler orientations in the resultant shape, as well as morphological material parameters of the polymer. Therefore, material values acquired from specimens as a rule do not reflect molding materials properties, but rather characterize the properties of a specimen that happens to be in a state determined by the circumstances of its production. Data acquisition suited to such materials thus demands fundamental information as to the state of the specimen and the test conditions selected for it.

2.3.2 Specimen Preparation by Direct Shaping

2.3.2.1 Production of Specimens from Thermoplastic Molding Materials

From the preceding presentation, we see that properties can be repeatably measured only if both the measuring methods applied and the state of the specimen are reproducible. Method-oriented test standards only define specimen geometry and dimensions in addition to test condition requirements. Since the standards for producing specimens from a particular material (product standards) consider only the basic expectations of the particular materials group, but cannot satisfy the enormous variety and range of engineering polymers, they also refer to manufacturer's guidelines. This means that the optimum parameters for materials processing are passed on to polymer processors as the know-how of molding materials producers, e.g., in the form of processing guidelines or company standards. Thus, there is no single comparable materials state, but only states determined by the influencing factors in the particular process used.

As opposed to metal materials, there is therefore no reference specimen (e.g., hardness reference plate) for polymers. Moreover, retained samples exhibit aging together with changes in materials properties. Problems occur especially when calibration is required for the precise determination of measurement uncertainty of the test result, since the internal state of the specimen is itself a component of the total compliance of the test system. The influence of residual stress on test procedure has been observed, for example, in tensile testing specifically for the E modulus. Due to the cooling process in closed tool, residual compression stress forms at the edge and residual tensile stress in the center of the specimen (Figure 2.1a). Thereby, the residual stress profile $\sigma_r = f(y)$ spreads for example over the width of the specimen. If the stress components do not balance each other out, cracks and cavities arise in the center. If this specimen is subjected to tensile load, the resulting force generates tensile stress σ uniformly distributed across the width in relation to the cross-section of the specimen (cf. Section 4.3.2.1 and Eq. 4.76). In consequence thereof, particularly at very small stress values in the starting range of the stress–strain diagram, overlapping occurs between load-induced and residual stress that leads to the resulting stress distribution σ_{res}. Since the modulus of elasticity E is determined in the starting range of the stress–strain curve (Fig. 4.27 and Eq. 4.81), the absolute value of this materials parameter is subject to influence. In actual tests, this is illustrated by comparing the E modulus of tempered and untempered specimens. Tempering leads to a reduction of residual stress, which results in a reduced E modulus.

Orientations within the specimen affect both the E modulus as well as the characteristic values of strength and deformation, whereby significant differences can result as to tensile strength and tensile strain at break (Figure 2.1b). Comparison

of stress–strain diagrams of short glass-fiber reinforced polyamide materials produced directly by injection molding (specimen 1 in Figure 2.1b) or by injection molding of plates and subsequent shaping (specimens 2 and 3 in Figure 2.1b) shows that the tensile strength corresponding to the peak of the stress–strain curve is greatest in directly injected specimens. The cause for this is the high state of anisotropy with a high content of fibers oriented in the direction of injection molding. Orientation on the plates is less due to production conditions; however, there is a striking difference between values acquired in transverse and injection molding direction. Tensile strength and tensile strain at break exhibit inverse tendency, i. e., the specimen with the greatest tensile strength exhibits the least tensile strain at break.

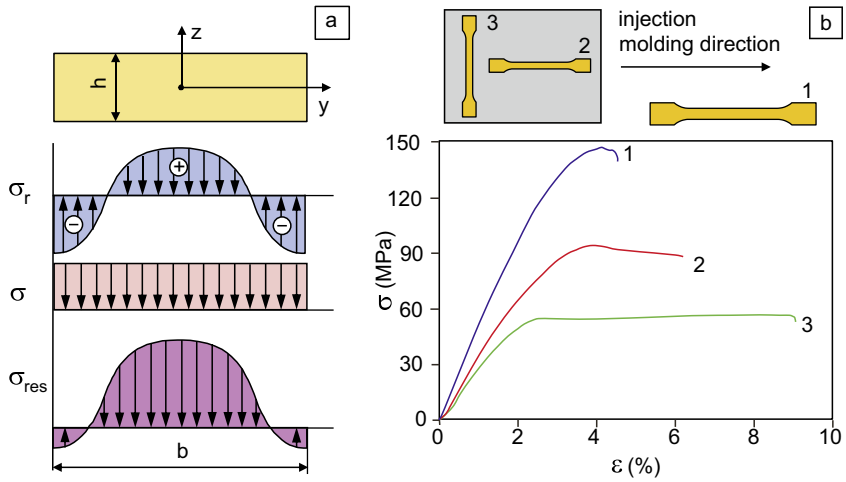


Figure 2.1 Overlapping of residual stresses σ_r with loading stress σ in the tensile test for a defined load state (a) and stress–strain diagrams of PA 6 with 30 wt.-% GF in the tensile test for injection molded specimens (1), specimens milled-out in injection molding direction (2), and specimens milled transverse to injection molding direction (3) (b)

Regardless of the type of direct shaping process (mostly injection molding), type 1A multipurpose specimen according to ISO 3167 is the specimen of preference for thermoplastic molding materials. Besides the tensile test, the plane-parallel center of this specimen can be used for very different mechanical, electrical or thermal tests (Figure 2.2). These specimens are preferred since they provide a uniform reference standard regarding orientation and residual stress (internal state), as well as identical thickness and width (external state). When specimens have to be taken from molded parts or components, it is generally impossible to remove 170 mm long specimens. In this case, proportionally reduced specimens can be prepared, whereby care must taken to scale down the test speed and strain measuring technique (cf. Section 4.3.2).

In principle, there exist two possibilities for minimizing the effects of processing conditions or creating a defined reference standard:

- Preparation of specimens in the initial state and
- Preparation of specimens with a reference state dependent on processing conditions.

Initial state specimens should consequently be homogeneous with respect to the distribution of morphological texture, be macroscopically isotropic (without preferred orientation) and without residual stresses. This state, which can be achieved by compression molding, does not occur in actual component or molded parts.

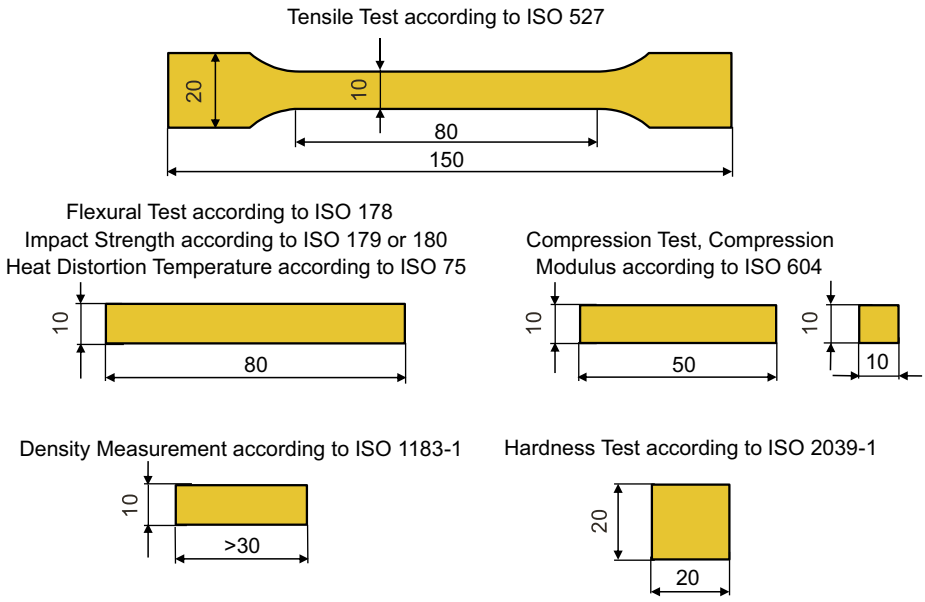


Figure 2.2 Multipurpose specimen according to ISO 3167 and specimens producible from it for conducting various other tests

Specimens can be produced either directly in a compression mold, or the required specimens can be cut from plates. Thanks to preheating, minimal shear effects during compression and controlled slow cooling rates, the formation of residual stresses and orientations can be almost entirely eliminated. Pressure and temperature have to be selected accordingly. In semicrystalline polymers, the cooling rate achieved also controls the degree of crystallinity, as well as the crystallite and/or spheroidal structure.

Practical experience shows that the holding temperature required should lie approx. 100 °C above the *Vicat* softening temperature (VST) of the amorphous or semicrystalline molding material involved, in order to obtain sufficient homogeni-

zation. Table 2.1 provides exemplary guidelines for compression molding specimens or plates from PS molding materials.

The plates or specimens produced under these conditions can be considered to be homogeneous as well as free from stress and orientation if, after a rest period of 30 min at approx. 150 °C, they exhibit neither shrinkage nor shape distortion including surface textures, and if the basic mechanical characterization of the tempered and untempered specimens is identical within the degree of scatter.

Table 2.1 Guidelines for Pressure Molding Specimens and Plates from PS Molding Materials

Molding material	Molding press temperature (°C)	Molding pressure (MPa)	Preheat time without pressure (s)	Holding time with pressure (s)	Cooling time under pressure (s)
PS	190 … 210	4.0	300	300	300
SAN	200 … 210	4.0	300	300	300
ABS	240 … 250	4.0	300	300	300

To produce specimens (usually multipurpose specimens according to ISO 3167) in reference state, the preferred method is injection molding using the optimized parameters of the molding materials manufacturer. The internal state arising thereby has to be characterized using measurements at this point, since the state of the specimen depends on the selected processing and mold parameters (pressure, time and temperature), as well as the type of machine used and the layout of the mold (e. g., flow distance).

Experience shows that dimensional stability and accuracy of plastic products are largely determined by cooling contraction in the mold and shrinkage of the component at increased temperatures. Contraction is a consequence of the volume contraction determined by the processing technology during transition from molten to solid state and is compensated by appropriate mold design and draft angle [2.4]. Shrinkage of components or specimens is observed as a result of orientation-related relaxation of the molecules during heating. Its result is a change in macroscopic dimension and length depending on the processing conditions selected [2.5] that cause an entropically unfavorable state compared to the initial state. The following effects are responsible for this change of state:

- Degree of plastic deformation, e. g., during deep-drawing or extrusion,
- Thickness differences that lead to, e. g., residual stresses and flow lines from heating and cooling during the injection molding process,
- Orientations that arise during, e. g., extrusion or injection molding, and lead to anisotropy of properties,
- Surface textures and rough spots, as well as
- Notch stresses and static weld lines.

Due to the altered physical structure, i. e., molecular and/or filler or reinforcement orientations, as well as residual stresses in mechanically shaped, stretched or injection molded parts or specimens, shrinkage S is observed when temperatures are increased and recovery is not prevented. When the external geometry is maintained, deformation is restrained expressed as shrinkage stress σ_s.

The causes of both these effects are thermally induced reorientation processes due to increased molecular movement, also called memory effects, that cause a change in the entropy state. The process of free shrinking is often described as a special form of retardation (recreep), while inhibited shrinkage is also described as relaxation (residual stress relaxation). These processes can follow very different chronological sequences under identical conditions (temperature, pressure). During both thermal stress analysis and thermal strain analysis, an overlapping heat strain is observed that has to be considered in the interpretation of experimental results [2.6].

The measure for reversible frozen deformation is the entropy-elastic strain ε_e measured in the shrinkage test:

$$\varepsilon_e = \frac{\Delta L}{L_0} = \frac{L - L_0}{L_0} \tag{2.1}$$

whereby L is the actual length at temperature T, and L_0 corresponds to the length of the reoriented state. Shrinkage S that tries to achieve the unoriented state beginning at the initial length L_a, is determined by Equation 2.2:

$$S = \frac{\Delta L}{L_a} = \frac{L_a - L}{L_a} \tag{2.2}$$

Between the entropy-elastic initial strain ε_{ea} determined by the shaping process and total shrinkage S_e in the unoriented state there exists the relation

$$\varepsilon_{ea} = \frac{S_e}{1 - S_e} \tag{2.3}$$

Under the condition of hindered shrinkage, the measured shrinking force F_s can be expressed as shrinkage stress σ_s:

$$\sigma_s = \frac{F_s}{A_0} \tag{2.4}$$

$$\sigma_s = \sigma_s\left(T_g\right)\frac{T}{T_g} \tag{2.5}$$

whereby A_0 is the initial cross-section area of the specimen, T the actual temperature, T_g the glass transition temperature and $\sigma_s(T_g)$ the shrinkage stress frozen at glass transition temperature. Given these assumptions and comparable specimens that may be taken from, e. g., multipurpose specimens, specimen shrinkage for the particular reference state can be determined in a heating cabinet or temperature

chamber, with postheat treatment temperature depending on the molding material. For amorphous thermoplastic polymers, the temperature ought to lie approx. 20 °C above VST and act for approx. 120 min. If in these tests, an approximately constant shrinkage S or entropy-elastic strain ε_e result, it can be assumed that a comparable and reproducible reference state has been reached.

Figure 2.3 illustrates the result of a shrinkage test on biaxial stretched PP film with a thickness of 30 µm for continuous warming at 2 °C min^{-1}. It can be seen that shrinkage on the film mechanically loaded in the main orientation direction begins at decidedly higher temperatures. This behavior is caused by stronger stretching of the molecules in the main orientation direction compared to the cross-sectional direction with its accompanying higher proportion of secondary valence bonding. A cast film that is produced practically without any orientation shows continuous lengthening with increasing temperature, exhibiting linear thermal expansion behavior.

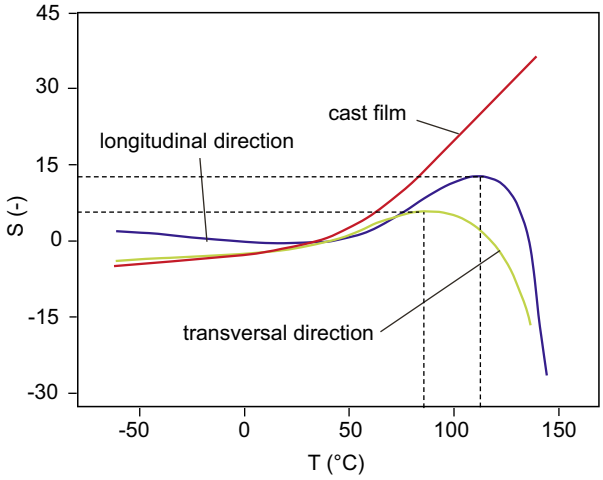


Figure 2.3 Shrinkage of a biaxial stretched PP film in the direction of orientation and transverse to orientation compared with an unoriented PP cast film

Shrinkage–temperature diagrams for the films investigated are presented in Figure 2.4. The varying anisotropy (orientation) of the specimens generated by their different processing conditions can be seen in the curve progression and in the temperature levels at transition. With increasing temperature, the measured force decreases dependent on the degree of orientation. This decrease is caused by a reduction of the E modulus at increasing test temperature under *Hooke's* law. Transverse to orientation, shrinkage starts at approx. 90 °C and in the direction of orientation it begins at approx. 120 °C. In contrast to biaxial stretched films, cast films do not exhibit shrinkage phenomena.

The shrinkage test is sensible to the deformation kinetics of the production process and illustrates the changes in shape stability and shape distortion tendency caused byincreased temperatures. Both tests permit conclusions to be drawn as to the state of molecular network, transformation phenomena and technically relevant limiting temperatures. For the interpretation of the measurement results, the influence of specimen thickness, thermal expansion coefficient (thermal expansion) and heat conductivity always have to be considered.

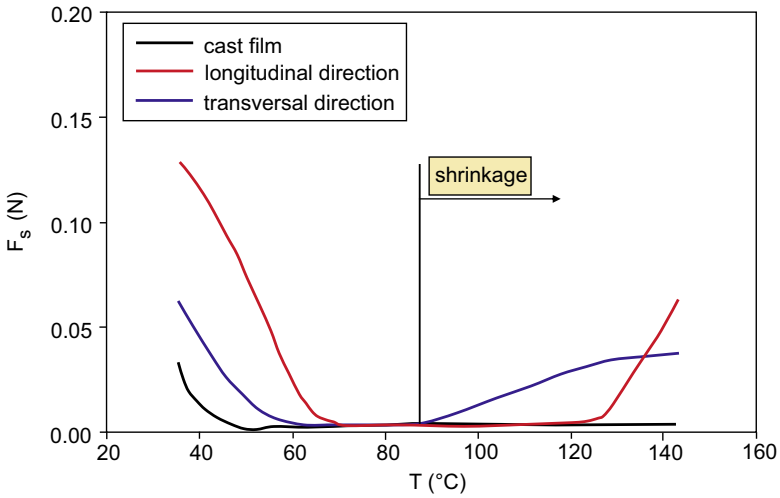


Figure 2.4 Shrink force F_s dependent on temperature at constant 0.1 % strain on a biaxial stretched PP film and a PP cast film

2.3.2.2 Production of Specimens from Thermosetting Molding Materials

Thermosetting specimens can be produced by compression molding (e.g., melamine-formaldehyde resin, aminoplastics and phenoplastics) or by casting (polyester and epoxy resins).

In compression molding, the molding material is generally cast directly into the die without prior conditioning, pre-drying or pre-heating, and shaped into specimens or semi-finished products under the effect of compression and the required temperature. In order to secure isotropic properties, the material specific pressure and the die temperature have to remain constant throughout curing time. During mold filling, the charge must be precisely metered, taking shrinkage effects into consideration, in order to fill the mold completely. Mold lubricants can only be used to expedite demolding if they have no influence on component or molded part properties. When parts with complex geometrical shapes are produced, it should be noted that especially the bottom sides are subject to increased thermal load due to the filling, compacting and heating sequence. To ensure identification of the

orientation of the specimen in the die, the die should be marked on the inside. To avoid problems or destruction of the compression molded part, the finished part should be removed from the tool no later than 30 s after opening. If shape distortion or warping has taken place, e.g., due to shrinkage, storage under plane load can be undertaken until cooling is complete. To avoid excessive cooling rates, the loading weights should have low heat conductivity. If the molds do not close precisely, flash can occur on the part's surface, which can be subsequently carefully removed, as long as visual scratches do not result. Prior to the particular test, the specimens produced have to be stored in standard climate according to valid product standards for a sufficient duration of time, or at least 16 h.

For the preparation of specimens from casting resins, there are two principally different methods: direct casting of specimens, or cutting specimens to shape from cast plate. Essential criteria to be observed for casting resins include the technology prescribed by the manufacturer as it relates to the mix ratio of resin and curing agent, or curing agent and accelerators, as well as pot life (time until gel). Molds that open on one side and that can be made from any number of materials, depending on the length of use, are utilized to produce specimens. If only a few specimens are required, a silicon or Teflon mold can be used; otherwise steel or brass molds should be used. For optimum demolding, these molds should be sealed with silicon coating; prior to casting, additional spraying with silicon oil film (epoxy resins) is recommended. If unsaturated polyester resins (UP resin) is involved, a 1 % solution of hard paraffin in carbon tetrachloride should be used instead as releasing agent. Sufficient practical experience is required to cast specimens of this type in order to produce them without gas bubbles or flash, and at the same time with good surface quality. Care must be taken with the mixing method, since tiny gas bubbles can easily become mixed in and thereby generate porosity which is not easily eliminated. If resin system pot life permits it, vacuum storage can at least reduce such porosity. However, if the resins are mixed with fillers and then stored in this manner, separation and segregation effects are to be expected and can only be eliminated by further mixing. For the characterization of pure resins, usually tensile and bending test, shrinkage determination and heat distorsion stability are used besides their chemical properties. For this reason, the required number of specimen geometries is small. Shrinkage or defective surface quality requires that the specimens be machined by sawing and/or milling, whereby the cutting surface should be heated up as little as possible.

For reinforced or filled specimens (laminates, glop top) produced by prepreg processes, pultrusion or lay-up molding, special instructions have to be followed, since these composites react very sensitively to scratches or thickness variations. Depending on the standard, these specimens should be equipped with cap strips or fixing holes in the shoulder range in order to facilitate fracture in the plane parallel part of the specimen. Further information on preparation and test procedure for these materials is presented in Chapter 10.

2.3.2.3 Production of Specimens from Elastomeric Materials

In order to acquire physical-mechanical values for vulcanized elastomers, specimens can be produced by cutting or punching them from rubber or rubber-fabric plates, as well as from finished parts. Low-temperature cutting is an alternative to these two production processes, but can only be recommended for very small specimen geometries. There are standardized preferred thicknesses for macroscopic specimens depending on the type of test to be performed:

0.5 ± 0.05 mm, 1.0 ± 0.2 mm, 2.0 ± 0.2 mm (e.g., for tensile specimens);
4.0 ± 0.2 mm, 6.3 ± 0.3 mm, 12.5 ± 0.5 mm (e.g., compression set).

Independent of how specimens are produced (cutting, punching), it is not permitted to produce specimens from plate stacks, since plate deformation occurs with increasing cutting depth. If the processing direction (rolling or calendering) of the plates is known, specimens should be preferably cut in this direction. If data on anisotropy are desired, additional specimens can be prepared and tested transverse to the processing direction. The quality of the specimens produced depends mainly on the condition of the cutting edge or punch, especially its sharpness. Damaged knives or punches must be disposed of, since the specimens produced by them probably exhibit notches, flash or serrations that can decisively influence the quality of the material values measured. Precise specimen preparation requires that the cutting machine (slicer) and/or punch template (punch press) be precisely positioned in the direction of cutting or punching. To avoid mechanical damage to the cutting equipment, suitable cardboard or PVC backing, but never rubber, should be used.

When plates are taken from finished parts with non-standard component thickness, it is permissible in principle to grind them to the desired shape or surface finish. However, the specimens should not be taken from the plate until after such machining. If they are to be ground, care must be taken that specimens are not heated to more than 60 °C; this can be achieved by using low grinding speeds (10 to 30 m s^{-1}) and grinding media with medium grain size. Tests using these specimens should be performed generally not sooner than 16 h subsequent to preparation and not later than 30 days after vulcanization. However, these requirements may vary depending on the particular elastomer and its conditions of use.

The same requirements apply for the preparation of specimens from plates of plasticized PVC (PVC-P) as for specimens from elastomeric materials.

2.3.3 Specimen Preparation by Indirect Shaping

By indirect shaping we mean the obtaining of specimens by cutting them from finished larger injection molded, extruded or compression molded plates or component parts. The most important cutting methods for doing so include sawing, milling, turning, grinding, boring and planing. However, the following aspects also need to be considered:

- Standardized specimens (multipurpose specimens) usually can only be produced from flat semi-finished products, whereby it is necessary to clearly mark the preferred direction of reference.
- Geometrically complex parts only rarely allow the preparation of specimens.
- Subsequent to removal and machining, the internal state of the specimen no longer stands in clear relation to the internal state of the component part (exposure or reduction of residual stress).
- Cutting shaping and the thermal load induced can additionally affect the test result.

To secure a low level of measurement uncertainty and to avoid unacceptably high measurement dispersion, fundamental aspects have to be observed in the indirect production of specimens. In case the semi-finished parts exhibit greater thickness than the required standardized specimens, these should be used without additional machining. Only in special cases is the removal of thickness to standardized values permissible; however, a minimum thickness of 1.5 mm must be maintained. When the direction of injection, rolling or flow is known, specimens for determining anisotropy of properties have to be taken length- and crosswise. When the main orientation direction is unknown, process-related anisotropy can be qualitatively determined with the help of the shrinkage test.

To ensure efficient shaping, suitable hardware should be provided (band and circular saws, as well as milling machines) while complying with the guidelines for saw blades and milling tools suitable for machining thermoplastics or thermosets. Generally speaking, all of the above mechanical machining methods can be applied to these various materials (see Table 2.2). However, with filled or, e. g., glass-fiber reinforced materials, increased tool wear is to be expected. Blunt tools always have to be replaced, since sawing or milling with such tools results in increased thermal load and/or makes it impossible to maintain specified geometries (notch tip radius). If machining is done in several steps (sawing of strips to be subsequently milled), e. g., when making dumbbell specimens, the influence of milling tool diameter on the quality of lateral edges has to be considered. Regardless of the type of milling, e. g., with a milling template or CNC (computerized numerical control) mill, cutting quality rises with the diameter of the milling head. From flexible and relatively soft polymers, specimens can also be taken by stamping. However, prac-

tical experience shows that the worst results with high measurement variance have been recorded with this method.

Table 2.2 Indirect Shaping Methods and Selected Production Conditions (v – Cutting Rate, s – Feed Rate)

Process	Cutting tool	Thermosets		Thermoplastics	
		v (m min^{-1})	s (mm)	v (m min^{-1})	s (mm)
Turning	High-speed steel	80–100	0.3–0.5	600–800	0.2–0.4
	Hard metal	100–200	0.1–0.3		
Milling	High-speed steel	40–50	0.5–0.8	30–45	0.3–0.8
	Hard metal	200–1,000		200–400	0.2…0.5
Boring	High-speed steel	70–90	0.2–0.4	30–40	0.2–0.4
	Hard metal	90–120		40–70	
Sawing	Band saw	1,500–2,000	manually	1,000	manually
	Circular saw	2,500–3,000		3,000–4,000	
Grinding	Corundum wheel	1,800–2,000	–	500–1,500	–

A relatively new, cost-intensive method is water-jet cutting capable of producing nearly any shape specimen in excellent quality [2.7].

It can be fundamentally stated that the occurrence of microcracks and notches directly correlates to the condition of mechanical tools and cutting surfaces, so that these can decidedly affect the level of properties and dispersion of measurement results. For optimum results, the following aspects have to be attended to:

- Chips formed during the machining process should be “cold” and smooth.
- Polymers with low heat conductivity have to be machined at low cutting rates and with additional cooling (compressed air or water).
- Superficial softening at the cutting surfaces due to frictional heat from blunt tools can cause stress during cooling.
- High cutting rates usually provide the best surface quality at low feed rates.
- Subsequent machining by grinding or polishing should always be done parallel to the longitudinal axis of the specimen.

Additional information regarding the machining of polymers can be found in ISO 2818 as well as in [1.13] and [1.34].

2.3.4 Characterization of Specimen State

In principle, any physical-mechanical methodology developed for testing polymers and their composites is technically suited for determining and evaluating the state

of specimens. Microscopic and electron-microscopic methods including image analysis are traditionally of special importance, since they provide a visual image of structural parameters. On the other hand, spectroscopic and non-destructive test methods, e.g., measurement of residual stress [2.8, 2.9] or the description of molecule and fiber orientation and anisotropy [2.10–2.12] are gaining in practical relevance and acceptance, since they function free of contact and can be utilized for components (see Chapter 8). Regardless of the type of measurement technology and the working principle applied, the test method selected has to have sufficient sensitivity to record the relevant structural or morphological parameters.

By structure, we mean a collective concept for the chemical and physical regularities in the make-up of polymers involving not only aspects of each single molecule, but also the formation of molecule aggregations in amorphous and semicrystalline state (morphology), as well as changes due to processing. For thermoplastic polymers, it can be assumed that the chemical properties specific to the material alter only minimally due to processing, whereas the property level of thermosets and elastomers is strongly influenced by chemical crosslinking reaction. However, in all polymers the type and method of shaping decisively affects the shaped physical structure, which can be described by morphology, orientation and residual stress.

By morphology, we mean the totality of supermolecular structures reaching from the smallest details in the nm-range up to several hundred μm. Size, shape and arrangement, as well as quantity ratio depend on the particular interactions and represent characteristic values for the specific polymer. Corresponding to the spatial extension of such structural elements and their stability with regard to mechanical and thermal loads, we can distinguish between micro and macro morphology. For this reason the respective defect mechanisms are subdivided into micro- and macrodamage. Beyond that, the definition of morphology has to be extended, since not all polymers are used as pure matrix materials, but rather as blends, i.e., filled or reinforced materials systems for strengthening, stiffening and toughening designer applications. This extension includes, among other things, the spatial distribution and density of fillers or reinforcements, the arrangement of additional phases, e.g., in the form of co-continuous phase distributions and core–shell structures, as well as nanoparticle filled and nanostructured polymers.

Due to thermal shaping with accompanying shearing and laminar flow in the mold, anisotropies with defined preferred directions arise that are also called orientations. By orientation, we therefore mean the alignment of structural elements with molecular, supermolecular or colloidal dimensions relative to the main axis of a specimen or component.

During the processing of plastics parts in molds that are closed on all sides, internal stresses arise from the volume contraction occurring during cooling; these are called residual stress. The energy-elastic deformation of molecule bonds responsible for this are irreversible without thermal and/or mechanical load, because the

laws of thermodynamics do not allow them to restore themselves by relaxing. However, these inherent stresses can be released by subsequent thermal treatment once the corresponding energy threshold has been exceeded. The resulting internal forces and moments are in equilibrium, so that the material appears to be free of residual stress. Therefore, residual stresses are not genuine structural parameters, but rather the consequence of changes in real polymer structure due to processing technique. On the other hand, it is of great practical relevance when designing and dimensioning molded components to know the internal, frozen state of polymer stress. This is especially the case with regard to the generation of stress cracking and plastic deformation due to local exceeding of the yield point with resulting impairment of mechanical strength. Due to the various accessibility for different measurement technologies (X-ray residual stress analysis or shrinkage measurement), it makes sense to distinguish between micro- and macroscopic residual stress.

Table 2.3 summarizes various parameters for describing and methods of characterizing specimen state without claiming to include all possibilities.

Table 2.3 Overview of Processes and Methods for Determining Specimen State (○ Suitability Limited; ● Suitable)

Morphology/ Structural parameter / Test method	Orientation	Degree of crystallinity	Spherulite distribution	Fiber/Filler distribution	Fiber/Filler orientation	Residual stress
Light microscopy				●	●	
Polarization microscopy	●		●			○
Electron microscopy	○			○	○	
X-ray methods	●	●	○	○	○	●
Radiography				●	●	
Differential scanning calorimetry		●				
Density measurement	○	●		○		
Ultrasonic test method	●			○	○	○
Measurement of anisotropy	●		○	○		
Shrinkage measurement	○					○
Shrinkage force measurement	●					○
Dissection method						●
Stress crack testing	○					○
Laser holography						●
Microwave technique	○			●	●	
Thermography				○	○	

The testing methods conforming to the definitions listed above can be divided into the following groups:

- Methods relating change in physical properties to the internal state of the specimen (e.g., birefringence, density, heat conductivity)
- Methods correlating change of a property affected by application technology with internal state (e.g., directional dependence of *Knoop* hardness, yield stress)
- Testing methods based on degradation effects on internal state (e.g., shrinkage, relaxation by dissection)

Non-destructive methods based on physical working principle and wave length (e.g., X-ray refractometry, ultrasonic testing) that generate measurable interactions at internal interfaces.

In actual testing practice, problems with measurement technology often arise due to the complexity of internal state and overlapping interaction. They can complicate any precise analysis of complex components considerably, or sometimes even render it impossible.

2.4 Specimen Preparation and Conditioning

In order to ensure the reproducibility of test results, not only specified production of specimens and sufficient uniformity of test climate (temperature and humidity) have to be guaranteed, but also the moisture content of the specimen. The reason for this is that for polymers the characteristic value levels change at even small variations in loading rate and other test conditions, such as environmental temperature or humidity [2.13]. Therefore, so-called standard atmospheres that satisfy average climatic conditions, thus simulating conditions in actual practice, have been defined as testing conditions. Standard atmosphere according to ISO 291 with an air temperature of 23 °C and relative humidity of 50 % (designation: 23/50 rh) has to be used for normalizing specimens and performing tests to characterize materials properties at room temperature (RT). Two different classes of standard atmospheres are stated in this standard, corresponding to different ranges of deviation. Class 1 limits temperature deviation to ±1 °C and relative atmospheric humidity to ±5 %; class 2 limits temperature deviation to ±2 °C and relative humidity deviation to ±10 %.

The simplest, but also most expensive method for guaranteeing constant testing climate is to climatize the entire space including the testing equipment by a suitable system. Other technical preconditions for climatizing include the absence of additional heat sources, such as drying cabinets, tempering equipment, etc., and that appropriate measures are taken to neutralize the effect of sunshine.

Climatized testing facilities are an indispensable prerequisite for long-term static (e. g., creep test) or dynamic investigations (e. g., determination of fatigue strength) under standard atmosphere.

For the acquisition of characteristic data on polymers in short-time tests, it is often sufficient to adjust the specimen to the corresponding test climate. For this purpose, the specimens are conditioned to put them in equilibrium with a standard atmosphere. During conditioning, specimens assume the temperature of the surrounding air, while test duration depends on initial temperature and geometrical dimensions, especially thickness. Depending on the diffusion coefficient of the polymer, a state of equilibrium is reached between the specimen moisture content and that of the surrounding air. Duration of storage is determined mainly by the type of polymer to be tested and can vary widely for the same relative humidity.

Specimens are to be stored in normalizing atmosphere in such a way that as much of their surface as possible is subject to atmospheric influence. It is generally no problem to maintain constant storage temperature, but maintaining desired humidity levels is not as simple. The use of dessicators or environmental cabinets is a prerequisite. Table 2.4 lists achievable relative humidities at various temperatures and various saturated solutions.

In case atmospheric conditions in the test facility deviate considerably from those of normalization, testing must be performed immediately after removal of the specimen from the conditioning chamber. Taking humidity into consideration, normalization duration is approx. 88 h for standardized multipurpose specimens; if only temperature is adjusted, approx. 4 h suffice.

Special conditions are specified for specimens made from polyamide (PA), since these materials absorb more than 2 % humidity depending on the type of PA and its reinforcement or filler materials. For normalizing dry-as-molded specimens, accelerated conditioning according to ISO 1110 at 70 °C and 62 % humidity can be performed while monitoring weight gain; storage duration is graduated according to specimen thickness.

16 h is the generally assumed duration for normalizing thermosets; at least 1 h for most elastomers. In addition to the other test conditions, type and duration of normalizing and test atmosphere have to be recorded in the test protocol.

If polymers are to be characterized at temperatures deviating from standard temperature, the testing facility must have an adjoining temperature chamber, or the test equipment has to be completely accommodated in a tempering apparatus. The specimens to be investigated have to be pre-heated at each test temperature in order to obtain sufficiently constant cross-sectional temperature. To obtain sufficient air circulation, specimens have to be stored in such a way that direct surface contact is avoided. Practice shows that approx. 30 min is sufficient for multipurpose specimens with a thickness of 4 mm. If, in addition to increased or decreased

temperatures, a specified humidity level has to be maintained, an environmental chamber has to be used.

Table 2.4 Relative Humidity Over Saturated Salt Solutions at Various Temperatures

Salt	Relative humidity in % at									
	5 °C	10 °C	15 °C	20 °C	25 °C	30 °C	35 °C	40 °C	50 °C	60 °C
Potassium hydroxide	14	13	10	9	8	7	6	6	6	–
Lithium chloride	14	14	13	12	12	12	12	11	11	10
Potassium acetate	–	21	21	22	22	22	21	20	–	–
Magnesium chloride	35	34	34	33	33	33	32	32	31	30
Potash	–	47	44	44	43	43	43	42	–	36
Magnesium nitrate	58	57	56	55	53	52	50	49	46	49
Sodium bichromate	59	58	56	55	54	52	51	50	47	–
Ammonium nitrate	–	73	69	65	62	59	55	53	47	42
Sodium nitrite	–	–	–	66	65	63	62	62	59	59
Sodium chloride	76	76	76	76	75	75	75	75	76	76
Ammonium sulfate	82	82	81	81	80	80	80	79	79	–
Potassium chloride	88	88	87	86	85	85	84	82	81	80
Potassium nitrate	96	95	94	93	92	91	89	88	85	82
Potassium sulfate	98	98	97	97	97	96	96	96	96	96

When polymers are to be used in the automobile and aerospace industries, as well as for household appliances, the changes in their characteristic values such as strength, stiffness and toughness under exposure to any number of media need to be considered. In order to determine environmental-thermal resistance, specimens are exposed to media (oil, water, detergent solution, etc.) at various temperatures, for specimens to be investigated have to be pre-heated at each test temperature in order to obtain sufficiently constant cross-sectional temperature. To obtain sufficient air circulation, specimens have to be stored in such a way that direct surface contact is avoided. Practice shows that approx. 30 min are sufficient for multipurpose specimens with a thickness of 4 mm. If, in addition to increased or decreased temperatures, a specified humidity level has to be maintained, an environmental chamber has to be used.

When polymers are to be used in the automobile and aerospace industries, as well as for household appliances, the changes in their characteristic values such as strength, stiffness and toughness under exposure to any number of media need to be considered. In order to determine environmental-thermal resistance, specimens are exposed to media (oil, water, detergent solution, etc.) at various temperatures, for example, to determine media resistance of polymers in detergent containers up to 2000 h; subsequently the value levels are compared with the initial state. Strict

Index

C

D

E

F

G

H

I

J

K

L

M

N

T

U

V

W

X

Y