Limitations of Test Methods for PLASTICS

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Foreword

This publication, *Limitations of Test Methods for Plastics,* contains papers presented at the symposium of the same name held in Norfolk, Virginia, on 1 November 1998. The symposium was sponsored by ASTM Committee D20 on Plastics. The symposium chairman was James S. Peraro, consultant, Newark, Delaware.

Overview

Testing is the means by which information (data) is developed on materials or products, and tests have been used for over 2000 years to provide a wide range of technical information describing a material's properties and characteristics. The first published test standard for plastics was written by ASTM Committee D20 in 1937. The early published test standards were simple in form and composition. Test methods were usually generic and written for the limited number of the then-known polymeric materials. They addressed all material types and were used for the determination of traditional properties such as tensile, flex, impact, and flammability. As polymers evolved into a vast array of polymer types, all different in structure and properties, so have test methods. ASTM standards are no longer those simple documents prepared when plastics were the new curious materials, but have continued to evolve as the technology of plastics has evolved. Test methods range from the very simple to very complex, such as those used to generate property data for engineering applications. Every ASTM committee attempts to provide standards that reflect the latest technology in testing of materials to meet the widening need of the global marketplace. The end result is that today's test methods not only generate more meaningful data but are used for a wide range of applications.

What started out to be simple generic test methods have necessarily become more complicated and difficult to comprehend. As test methods have become more sophisticated and complicated in scope and application, more knowledge about materials and their characteristics is needed by those using ASTM test methods to develop test data and by those who analyze and utilize the data. Generally, the result is a lack of understanding of the variables that contribute to and influence test results. It has been long understood by the testing community that every test method ever written, whether written for metals or non-metals, is composed of variables. There are many sources of variables and all have a direct influence on the accuracy of the generated test data. The sum total of all variables defines test limitations.

Test limitations are a compilation of the variables (1) present within a test method; (2) associated with the material under investigation; and (3) those external to but not related to the test method or material. Test and material variables are the primary source of variability. The external variables are primarily those influenced by an individual's knowledge of the characteristics of the material under investigation or the test method(s) to be used in its evaluation, and the ability to properly analyze the generated test results as related to the intended use or application. Misinterpretation, misuse, or misapplication of the test method or the use of the data generated all contribute to test limitations. Unfortunately these limitations are not fully understood, resulting in inappropriate claims or conclusions pertaining to materials or products made from plastics.

ASTM enjoys an excellent reputation as a leading organization in the development of test methods used worldwide. ASTM technical committees have developed over 10,000 test standards. Unfortunately, there is a general belief that the results obtained from these test standards are absolute, which is not the case since each has its limitations. ASTM standards are living documents and are continually being updated and revised to reflect the latest in testing technology. Limitations are not limited to the ASTM test standards. In the United States there are over 400 standards writing organizations, and when you add all the test standards worldwide (ISO, DIN, BSI etc.) there are an enormous number of test standards all with their own set of limitations.

It has been acknowledged for many years that there was a need for a symposium discussing the limitations inherent in all test methods. ASTM has always encouraged the use of symposia or other formal programs to educate those interested in the proper use and application of ASTM standards or the principles by which they were developed. In order to promote and educate the business and technical communities about the limitations of test methods of plastics, ASTM D20 on Plastics decided to schedule a symposium on this very important and timely subject. In November 1998 a symposium entitled Limitations of Testing was held in Norfolk, Virginia.

In this symposium, 21 papers from both Europe and the United States were grouped into four major categories, namely General/Design, Mechanical, Impact/Fracture, and Chemical/Rheology. Some of the papers could have been placed in more than one category. It was a difficult task for the committee to make the final decision on the location of the paper and the order of presentation.

General/Design

In this section papers are presented covering issues facing engineers in the selection of the optimum material candidate and the development of test data for a specific performance criteria. There is a generally accepted protocol that is used by engineers in making a qualified decision based on available facts. The problem is knowing what is required of the product and what is the true functional behavior of the polymer. What is not often completely understood is the correlation of published data and the relevance to design. The various options and concerns are reviewed.

Creep tests can be conducted in either tensile or flexural modes. The time-dependent viscoelastic deformation of polymers and composites is compared and the differences in material compliance is analyzed. The constitutive relationship for creep compliance that takes into account the effect of dilatational stresses is determined. Estimation of lifetime under non-isothermal conditions is also presented. Not only are the thermal and mechanical loading of great importance to estimation of life expectancy, but also the influence of the chemical medium and immersion time. Two possible methods of obtaining this information are discussed: (1) time-temperature extrapolation of the measured aging process, and (2) a functional estimation of time-temperature collectives, the latter being more precise.

Mechanical

In this section, traditional tests such as tensile, and deflection under flexural load (DTUL) are covered. Papers discuss the development of testing procedures for materials and the influence of variables on the generated data. The implications of conversion from ASTM to ISO standards for material characterization for greater opportunity and to compete more effectively in the global market are reviewed. As global interaction increases, it is important that the concerns raised during conversion can be harmonized between the two sets of standards. Also, the comparison of tensile data generated by ASTM and ISO procedures and the results obtained from round-robin tests are discussed for a variety of polymers. Common errors made by laboratories were examined. Data are also presented showing the common variables that affect test results in both ASTM and ISO tensile tests.

Deflection temperature under load (DTUL) measures the temperature at which a specimen of a certain geometry deflects a fixed amount under a very specific set of conditions. However, it is often used in material selection as a measure of the maximum continuous use temperature for that material. The development of dynamic mechanical analysis (DMA) has shown that traditional DTUL test results often give a false measure of the thermal performance of polymeric materials. By measuring the elastic modulus versus temperature by DMA the thermal profile of any polymer can be obtained and a more realistic assessment of the elevated temperature performance can be obtained. New techniques were also presented for testing adhesive bond strength tests for piping systems. The technique developed utilized lap-shear plaques to predict performance in the pipe joint systems. Results indicate extreme sensitivity to minor variations in preparation.

Impact/Fracture

Papers in this section discuss the variables that have a significant effect on impact resistance. Impact tests measure the response of materials to dynamic loading. Pendulum impact tests such as IZOD and Charpy are used widely to quantify the impact performance of plastic materials. Both tests are used widely to develop impact data and are considered as a primary performance index for impact properties, but cannot be used for design considerations. In these tests there are a large number of variables associated with sample preparation, the test apparatus, and the test procedure. Data are presented comparing instrumented and non-instrumented IZOD and Charpy tests, the effects of the variables, and their influence on the test results. A new approach using fracture mechanics is presented for the determination of the impact fracture resistance G_c, or impact fracture toughness K_{1c}. The fracture mechanics perspective is based on an explanation of impact speed and geometry based on the thermal decohesion model. Analysis leads to a prediction of an apparent impact fracture resistance G_{ca}. Also, a new standardized test procedure to measure K_{Ic} and G_{Ic} for plastics at a moderately high rate of loading, namely 1 m/s, has been proposed. The test procedure is based on previously developed fracture mechanics technology for the determination of K_c and G_c. Round robin test data developed over a period of five years are reviewed and show the consistency in the test data, validating the test protocol.

Chemical/Rheological

Papers on advanced testing techniques primarily in the area of rheological testing were presented. Thermomechanical analysis (TMA) is compared to the coefficient of linear thermal expansion (CLTE) and the measurement of the glass transition temperature (*Tg*). Variables are identified and the effect on temperature measurements is discussed for CLTE and *Tg*. In another presentation, capillary and rotational viscometry is compared. The flow curve of the apparent viscosity versus shear rate emphasizes the dangers of using a single viscosity value such as Melt Flow Index. Both orthodox and unorthodox measurements are discussed for viscosity measurements for controlled stress and controlled rate devices. A more direct volumetric method to measure volume swell ratio has been developed for cross-linked polyethylene and compared to the gravimetric method using the deswelling or solvent evaporation techniques. The results show that the direct volumetric technique is more accurate and not subject to the limitations of the other techniques.

This symposium reflects the current work being undertaken within the ASTM D20 subcommittees to insure that all test methods are written in such a way as to be understood and used properly.

Acknowledgments

The symposium committee gratefully acknowledges the efforts of the authors, the ASTM D20 technical committee that helped put this symposium in motion, and the 44 individuals who reviewed the presented papers prior to publication of this STP. Finally, for any symposium to be successful, the majority of the individuals involved work behind the scenes, especially Dorothy Fitzpatrick and her staff at ASTM who provided the administrative services. Without their effort this symposium would not have been so successful.

James S. Peraro;
Symposium Chairman and Editor

Contents

General/Design

Stephen Burke Driscoll[1] and Christopher M. Shaffer[2]

What Does a Property Data Sheet Really Tell You?

Reference: Driscoll, S.B., and Shaffer, C.M., "**What Does a Property Data Sheet Really Tell You?**," *Limitations of Test Methods for Plastics, ASTM STP 1369,* J. S. Peraro, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: Today's engineer, whether a student in training or a practicing professional, is faced with a myriad of design considerations when selecting the optimum material candidate for a functional product. The normal protocol followed includes prioritizing performance credentials, selecting candidate polymeric material , reviewing the properties and processing characteristics of each, and then making a qualified decision based on the available facts.

The problems, however, are both knowing fully what will be required of the product, in a wide range of use/abuse environments, and understanding as thoroughly as possible the true functional behavior of the polymer. What often is not completely understood is the correlation between published data sheets and the relevance to design considerations. How meaningful is the information found in a typical data sheet? How easily, if at all, can the design engineer integrate this information in a series of design iterations, leading to an improved product?

How useful is the information most routinely published? Certainly a great deal of information is readily available in a number of formats, including proprietary databanks maintained and freely distributed by the various material suppliers. This information is equally available via the Internet, and much follows the CAMPUS template containing both single- and multi-point data (International Standards Organization-ISO 10350 and 11403 respectively).

Regardless of the quantity and ready availability of this information, how can published Izod impact behavior be successfully used to make a better, more durable product? How can the Distortion Temperature Under Load (DTUL) of a material translate into practical continuous use temperature? What published data are really useful? And what are meaningless?

Finally, what really is needed - and is usually missing - to help the design engineer predictably produce a safe, reliable, and durable product?

Keywords: Plastics, physical properties, impact and flow behavior.

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Introduction

The design engineer is faced with a myriad of problems when selecting material candidates for a product. As we become more sophisticated in our use of information resources, we expect our resin suppliers to equal or better our demands for performance data. Our history of being a "creature of habit", routinely using materials that have worked well for us in past product development programs, is no longer valid in contemporary design strategies.

Considerable work in our industry has led to the global recognition of ASTM D4000, Classification System for Specifying Plastics Materials. This protocol provides the necessary guidelines for developing line call-outs for polymeric materials, and reduces to consensus-balloted formats a range of tables/cells for specific properties. By prioritizing the requirements, and assigning minimum performance values, it is possible to generate a listing of material candidates which can be used for initial screening purposes.

A problem, woven throughout the fabric of this protocol, is the quality and utility of data generated and incorporated into these tables/cells. As stated in the abstract, how meaningful is the information found in a data sheet or product bulletin? It is important to recognize that the data sheet has very specific purposes, often sales and marketing related, and the technical content is not always intended for design and processing engineers. In fact, most date sheet do contain a caveat, warning the reader that the information supplied is general in nature, only approximate values, and intended to serve only as a guideline, and is not part of a material specification.

What Properties Should Be Reported?

Granted, there is a dichotomy existing with the data sheet. What physical properties should be published, as requested by the sales and marketing groups, versus what information is really useful to the materials, process, and design engineers?

This is not a new problem. We have been attempting to balance these two opposing requests for many years. In fact, you might be surprise to learn that many years ago, back in 1969, there was a special session at the Society of Plastics Engineers (SPE) Annual Technical Conference (ANTEC) devoted to this same question. (1)

In the December 1979 issue of Plastics Engineering an excellent article, "Get the MOST for Your Money from Your Resin Supplier", stated that fewer than half of the reviewed data sheet from international suppliers provided all of the information needed to enable you to make the best choices. (2) The article continued by contrasting the surveyed property data. The tabulation indicated three strong trends. The majority of the published data was aimed at the end product (materials engineer), considerably less information was for the mold design and processing engineers.

Noted below is an abbreviation of these published observations:

Table 1 - *Published Data*

Available data	Where needed End Product	Mold Design	Processing
A) Properties included in more than 80 percent of product data sheets surveyed:			
Specific gravity	Y	Y	Y
Tensile strength	Y	N	N
Elongation	Y	Y	N
Izod impact	Y	N	N

Flexural modulus	Y	Y	N
Hardness	Y	Y	N

B) Properties included in 50 to 80 percent of product data sheets surveyed:

Mold shrinkage	N	Y	Y
Melting point	Y	N	N
Tensile modulus	Y	N	N
Flexural strength	Y	N	N
Coeff. of Linear Thermal Exp.	Y	Y	Y
Moisture absorption	Y	Y	Y
Thermal conductivity	Y	Y	Y

C) Properties included in fewer than 50 percent of product data sheets surveyed:

Flow temperature or normal processing temperature	N	Y	Y
Specific volume	Y	Y	Y
Specific heat	Y	Y	Y
Fatigue endurance	Y	N	N
Poisson's ratio	Y	N	N

The author, Paul E. Sample, the current chair of the D20 Committee on Plastics, derived very nicely a series of six check-points for selecting the appropriate family and grade of material. These are:

1. polymeric family
2. molecular weight
3. melt behavior
4. specific gravity
5. mold shrinkage
6. strength & impact behavior

This primer was complemented ten years later by another impressive article, "Interpreting Supplier Data Sheets".(3) In this encyclopedia chapter, the authors intended their article to delineate the limitations of data for engineering design. ASTM and other testing organizations were cited for their development of standards to ensure reproducibility of data for quality control purposes and purchase specifications, although not always for generating engineering data.

The authors included a caveat that in many of the testing procedures the data should not be used for design purposes unless the application conditions are similar to the test conditions in terms of size, shape, strain rate, and environmental conditions, including temperature and relative humidity. They also noted that the properties of plastics parts are seldom as high as data sheet values. This can be attributed, in part, to the fact that the reported values are obtained using specimens specifically designed and molded at conditions optimized for that test.

At the 1993 SPE ANTEC, a thought-provoking paper, "Heat Deflection Temperature (HDT) and Notched Izod: Two Key Measurements That Have Outlived Their Usefulness" addressed the use of short-term tests to design for rigidity and strength under load at elevated temperatures. (4)The author concluded that such simple tests as DTUL and Izod are meaningless in judging material suitability in applications in which exposure to elevated temperatures is more than momentary.

This same battle cry was voiced five years later, at the 1998 SPE ANTEC in Atlanta.(5) A special session focused on materials properties that are often misused. For example, two papers addressed again the usefulness of the DTUL test and Charpy/Izod impact behavior in relation to material toughness. (During this symposium and printed in this Special Technical Publication (STP) will be additional papers addressing these two topics.)

Consequently, for just shy of three decades, we have continued to discuss these limitations of test methods for plastics. Fortunately, we can report some progress in resolving these issues. Noted below are some abbreviated comments on typical physical properties, commonly published, and what is useful or meaningless about these tests.

Mechanical Properties:

Thanks to the considerable quantity of data published on these important physical properties, this does represent one of the few areas in which the design engineer can use published information to make a better product. Knowing the load bearing characteristics of a material, whether in tension, compression, or flexure, does translate in to very specific geometrical requirements for safely loading a product. Knowing the modulus, which is synonymous with stiffness and rigidity, allows the design engineer to select the appropriate material, minimize wall thickness, and save money.

Rather than publishing single-data point, the prevailing trend, welcomed by many in the global testing community is the generation of multi-point data. For example, the complete stress-strain curve v. line call-outs of data at isolated conditions, such as tensile yield stress or elongation at yield.

For the design engineer, what is even more important, is knowing the mechanical behavior as a function of temperature and other environmental conditions. Until recently, with the market introduction of new, high performance engineering thermoplastics, these data have been rarely published. It is very encouraging that the resin manufacturers are now recognizing that their customers are becoming technically more mature and sophisticated and, consequently, that these data are beginning to be published.

However, most often the data sheet does not indicate which specimen geometry was used, the length of the mandated extensometer, the strain rate, and, most critically, which modulus is being reported. Was it the initial tangent modulus or a chord modulus, now being discussed as the preferred property in some countries. Remember, the data sheet of an imported material might not indicate explicitly the preferred property, such as modulus.

Useful Information:

Analogous to melt index reporting of temperature and weight (e.g. 190/2.16), is it too much to ask the resin manufacturer to report automatically on the data sheet the ASTM D638 Tensile Properties of Plastics specimen type (I - V) and the strain rate along with the extensometer used? Also useful would be including the complete stress v. strain curve, which illustrates overall ductility, rather than a limited series of single data points, such as the elongation and stress at yield or at break.

Even though the D4000-based material specifications should include all of the above-stated information, there might be some missing guidelines in selected documents. Consequently, the various D20 materials Sub-committees are currently reviewing their standards in order to complete this necessary information.

It is very satisfying to report that the D790 Flexural Properties of Plastics protocol has recently undergone complete revision and has passed the consensus balloting process. The three point procedure has been streamlined, in an attempt to harmonize with its ISO

178 counterpart, and the four-point bending procedure is being issued separately under a new ASTM D20 Committee on Plastics jurisdiction number (D6286).

Certainly, this will minimize any confusion when reading future data sheets. No longer will one have to question how the material was tested, which span to depth ratio was used, nor guess the testing speed. However, the question of which modulus should be reported still lingers.

Other mechanical properties-Hardness, which often correlates with modulus, is not commonly reported. Although fixed for Rockwell studies, a major concern for other hardness tests is the time delay between penetrating the plastics material and recording the hardness value. This concern for stress relaxation is legitimate and warrants continued scrutiny.

Creep data are invaluable for predicting the long-term functional behavior of a material or product. However, the current body of data (seldom, if ever, reported on product data sheets) cannot be compared for a series of material candidates. Polymers must be tested exactly the same way (*test mode, initial stress level, time, and temperature) in order to have a valid comparison, without relying on mathematical adjustments. The concept of the use of creep modulus has been widely adopted. However, one must still adhere to the paradigms of linear viscoelasticity to allow valid comparisons of different polymeric materials.

Impact properties-Impact behavior, especially ASTM D256 Pendulum Impact Resistance) is one of the more commonly reported "primary" physical properties. The limited data generated poses serious problems. How was the material molded, which section of the test bar specimen was tested (gate, mid-, or dead end), how was it notched (and at what cutting wheel rpm and sled feed rate), and how many hours after notching was the specimen impacted? When was the last time you saw this information on a data sheet or in a product bulletin?

Unlike instrumented impact testing (D3763), the pendulum test (both D256 and D4812 un-notched specimen) generates a single, fixed data point. Again, knowing the complete, detailed force v. deformation response of the impact event, as generated using D3763, will provide the design engineer with a greater appreciation for the nature of the fracture: punched hole or tear fracture, brittle v. ductile, etc.

Coupling these data with an analysis of impact behavior as a function of temperature, thickness, and impact speed is truly invaluable information, and should lead, obviously, to enhanced product ruggedness and safety.

Electrical properties-Interestingly, much of the electrical data is obtained by varying the testing frequency, analogous to instrumented variable rate impact testing. Strangely, what is standard operating procedure for one grouping of physical properties is not automatically adopted when measuring other equally important properties.

Flow Behavior and Thermal Characteristics:

Time and space allow only abbreviated discussion of two other commonly reported data sheet line items: melt flow rate - melt index (flow behavior) and DTUL (functional performance of a solid). Similar to Izod impact, these are also single data point tests. While providing some information for quality control purposes, neither one is eligible for consideration as design-caliber data.

The shear regime encountered during an extrusion plastometer trial (Melt Index/Melt Flow Rate - MI/MFR) does not fairly represent actual processing conditions. This single data point, tested at a fixed temperature, certainly does not reflect the actual processability of a polymer, most often at quite different melt temperatures. The advent of capillary rheometry enhances the value of viscosity v. shear rate. When performed at several temperatures, these data provide more meaningful information to the processing engineer. When, however, did you read capillary data in a typical data sheet?

An extension of this upgraded strategy was the important introduction of dynamic mechanical techniques for measuring the complex melt viscosity (both elastic and viscous components) as a function of temperature, shear rate, and time...all within the linear viscoelastic region. While ASTM D4440 Complex Melt Viscosity using Dynamic Mechanical Rheological Techniques (DMRT) has been globally adopted since the early 1980s, and most routinely used by resin manufacturers in their own R&D efforts, these data still are a rarity on a product bulletin. Why?

The ASTM D648 DTUL "...covers the determination of the temperature at which an arbitrary deformation occurs when specimens are subjected to an arbitrary set of testing conditions." As stated in the Significance and Use section, "...this test is particularly suited to control and development work...and data obtained (by this test method) may not be used to predict the behavior of plastics materials at elevated temperatures...unless under similar conditions..."

If we truly practice what we preach, working smart and not hard, we really should question why the DTUL is routinely reported. It does not have any immediate value to either the processing or the design engineer. Knowing that poly "X" has a DTUL of 80 C tells them nothing about the behavior of the material 5- or 10 degrees either side of the single data point. How often will the design engineer encounter a product environment that mimics the 264 psi outer fiber stress level cited in D648? How can a student engineer in training, use successfully one material boasting a DTUL 80 C to replace a second polymer exhibiting a DTUL of 90 C, or only 60 C?

Again, it is truly gratifying to state, as a teacher, that the new generation of product brochures, bulletins, and data sheets now include multi-point thermal characteristics. Publishing the tensile strength v. temperature and the more commonly used modulus v. temperature (Clash-Berg, ASTM D1043) and more recently, the fully instrumented ASTM D4065 series dynamic mechanical properties in tension (D5026) compression (D5024), and three point bending (D5023) as well as in torsion (D5279) and dual cantilever beam geometries (D5418) has significantly expanded our body of knowledge.

Knowing the change in stiffness v. temperature, especially when contrasting an amorphous v. semi-crystalline polymer, is critical to the design engineer when selecting the appropriate material candidate(s) for a product which will be used a wide range of environmental conditions. The decreasing slope of modulus with increasing temperature does provide the necessary information about the maximum continuous use temperature over a wide range of stiffness values. Work published by Michael Takemori of GE Corporate Research, in the late 1970s, has illustrated very convincingly this approach. (6)

Conclusion:

In conclusion, as we review our usual collection of data sheets, we should question the quality and relevance of these published data. With an eye towards learning more about how a polymer can be processed (melting temperature, Tm, viscosity v. shear rate, shrinkage characteristics, thermal conductivity, as well as thermal stability and degradation and the ability to be recycled, etc.), we must question the disadvantage of not having these data.

And certainly, we must recognize that the design engineer needs to know more meaningful information about how the polymer will perform under intended conventional use and abnormal abuse conditions (modulus v. temperature, creep behavior, impact toughness, and fatigue endurance, etc.).

Do we continue our reluctance , either through omission or commission, to upgrade our data sheets with information that is more useful to the materials, design, and