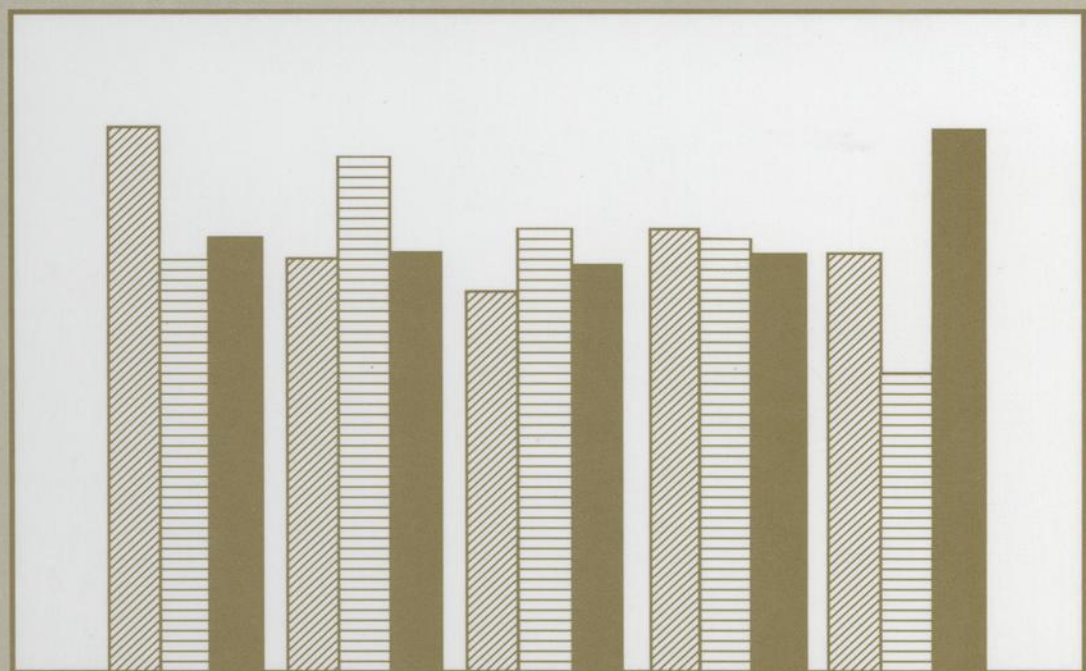


# MATERIALS CHARACTERIZATION BY THERMOMECHANICAL ANALYSIS



Riga/Neag, editors



STP 1136



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# ***Materials Characterization by Thermomechanical Analysis***

*Alan T. Riga and C. Michael Neag, Editors*

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### **Peer Review Policy**

Each paper published in this volume was evaluated by three peer reviewers. The authors addressed all of the reviewers' comments to the satisfaction of both the technical editor(s) and the ASTM Committee on Publications.

The quality of the papers in this publication reflects not only the obvious efforts of the authors and the technical editor(s), but also the work of these peer reviewers. The ASTM Committee on Publications acknowledges with appreciation their dedication and contribution of time and effort on behalf of ASTM.

# Foreword

Materials Characterization by Thermomechanical Analysis was presented at ASTM Headquarters, Philadelphia, PA, on 19–20 March 1990. The symposium was sponsored by ASTM Committee E-37 on Thermal Measurements. Alan T. Riga, Lubrizol Corporation, and C. Michael Neag, Glidden Company, served as symposium chairmen and are editors of the resulting publication.

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# Overview

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The papers included in this Special Technical Publication (STP) evolved from an international symposium on Material Characterization by Thermomechanical Analysis (TMA) held in Philadelphia in March of 1990. This symposium examined the uses of thermomechanical analysis and thermodilatometry in materials science and included presentations involving instrumentation, techniques, and applications of TMA. The program featured the 25 original presentations by individuals from five countries; fifteen of these original papers were included in this STP. In all, the symposium attracted about 70 people representing a variety of academic, industrial, and governmental interests.

The symposium marked the third in a continuing series on the use of thermal analysis in materials science sponsored by ASTM Subcommittee E37.04 on Technical Programs. This activity represents just one of the many functions supported by Committee E-37 on Thermal Measurements. The TMA symposium was organized Alan T. Riga (Lubrizol, then E37.04 chairman) and C. Michael Neag (Glidden) with the support of committee members Rick Seyler (Eastmond Kodak) and Charlie Earnest (Berry College).

The book shares the same organizational structure as the symposium: the STP includes three sections encompassing instrumentation, techniques, and the application of thermomechanical methods in characterizing inorganic and polymeric materials. The first section opens with a thorough literature review of instrumentation, basic thermomechanical methods and applications by Michael Neag. This paper represents the first and only comprehensive review on thermomechanical analysis and thermodilatometry and includes some 160 references on topics ranging from instrument design to the characterization of fundamental material properties to industrial applications of TMA. This section of the STP also includes an important paper by Seyler and Earnest that was also sponsored by E-37. These papers describe the development and testing of a method for TMA temperature calibration. They report on the experimental results of round robin that included seven labs. Although interlaboratory results varied by several degrees, the bias (accuracy) in these measurements was less than 0.1°C when corrections for temperature linearity in each instrument was included in the analysis. These are fine papers from two standpoints: (1) they provide excellent insight into the development of new test methods and (2) they act as outstanding primers for developing precision and bias tests. Statistics are accentuated again in a paper by Matsumori et. al. (Seiko) on the accuracy of coefficient of thermal expansion (CTE) measurements. The error analysis embodied in this paper is a rarity in the open literature; it is an important contribution because, more often than not, the authors give little attention or even acknowledge the precision, accuracy, or validity of their results. The first section of the book closes with an article describing a new dilatometer for metallic ribbons by Harmelin et al. (French National Center for Scientific Research).

The second section of the book emphasizes the general utility of TMA for characterizing materials and explores techniques. Riga and Collins (Lubrizol) open the section with a sweeping discussion of materials characterization by TMA, describing techniques and standard methods for a variety applications. Other authors emphasize more specific techniques: Weidemann (Mettler) describes a method for the visco-elastic characterization of polymers; Moscato (Eastman Kodak) discusses an alternative method for examining heat distortion temper-

atures, and Cassel and Twombly (Perkin-Elmer) provide an interesting examination of the glass transition temperature by TMA and DMA. The section concludes with a paper by Gill et al. (TA Instruments) describing a new TMA and its utility for characterizing high polymers.

The book's final segment emphasizes the application of TMA in characterizing materials. This section stresses detailed characterization of both material properties and product properties. McGhie (Pennsylvania University) describes electrolyte characterization by simultaneous TMA and dielectric techniques. His work offers a view of the delicate correlation between a material's mechanical and electrical properties. Schoff and Kamarchik (PPG) describe the benefits and pitfalls of applying thermal mechanical analysis to organic coatings. The influence of film thickness, substrates, and the relative degree of cure are underscored in their thorough overview. Thermomechanical methods form the cornerstone of Robert Adam's (Lord Corp) paper examining the relationship between formulation and performance in sheet molding compounds. This work furnishes an excellent example of the efficacy of statistical design and underscores recent trends away from traditional one-at-time experiments and towards comprehensive statistical designs. When compared with more traditional experimentation, designs like the one used by Adams can provide more information with substantially reduced experimental effort.

The book concludes with the application of TMA for product characterization. Ibrahim (Martin-Marietta) describes the use of CTE measurements for characterizing the dimensional stability of high performance circuit boards; Glen Gaddy (John Hopkins, NIST) describes the utility of TMA for characterizing aging effects in EPDM roofing materials and Lee et al. (Iowa State University) discuss the use of TMA to characterize asphalts used in road construction. The latter paper may be an archetype for future work in this area because it furnishes a detailed statistical analysis of a variety of data related to both intrinsic properties, formulation, and end-use performance.

As with any successful technical effort, in this instance the conference and the subsequent book, many people contribute. The tireless efforts and perseverance of the ASTM staff, Jamie Kerr, Therse Pravitz, Barbara Stafford, Dorothy Savini, Kathy Greene, and Susan Gebremedhin—deserve special mention. These folks fashioned an administrative “infrastructure” that made the conference and STP a reality. Without their tireless efforts, endless phone calls, and many faxes, the editors might still have papers in review. Many thanks also go to the numerous reviewers who volunteered both time and talent to work with the ASTM staff. Their prompt and constructive criticisms are reflected in the timeliness and quality of this STP. Finally, the authors themselves merit great thanks for their diligence in preparing each paper and their willingness to improve their original manuscripts when asked. Although nearly all of the papers required painstaking revision, the fine quality of the papers in this book made the extra effort worthwhile. Thanks to all for making this such a successful endeavor, AR and CMN, June 1991.

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