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Cristina Prisacariu

# Polyurethane Elastomers

From Morphology to Mechanical Aspects

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*This book is dedicated to Professor Adrian A. Caraculacu, the “father” of dibenzyl based polyurethanes, and not only.*



# Foreword

The World Wide Web offers wonderful resources. This is a comfortable medium for today, especially for those who have grown up in a virtual world of information. But printed books remain unique vehicles of information and ideas. They are convenient and organized. They can be thought provoking. Many current textbooks are excellent encyclopedias of information. But too many are overbearing and stately, often mannered in presentation. So they go unread or they have a small audience. And that is a shame. I therefore try here with a writing style that I hope will people to read, to pause and consider, and then to read further.

I also think a scientific book needs to be part of a scientific conversation with the readers. A book must not look like a “*cemetery of dead ideas*”, (Miguel de Unamuno, 1913). It has to be filled with ideas and evidence that go beyond what is found in the journal articles. A scientific book has to act as an argument, as a commentary, not merely a summary of other people ideas and works. It needs to allow the author to enter into dialogue with the readers and must be able to generate discussion with audiences. A scientific book has to give the readers a relevant description of the topic as well as its overall perspective, argument, or purpose. Authors should always immerse themselves in their books, writing a book they would like to read.

Not doing so, there is the risk that “*the books have the same enemies as people: fire, humidity. . . weather, and their own content.*” (Paul Valery, 1921)

\* \* \*

This book is designed as a review of the state of the art but also brings new contributions in highly specialized subjects, in a still rapidly moving field: the fundamentals of polyurethane elastomers: their chemistry and properties and materials choice for formulation development.

Why choosing to write a book on the fundamental science of polyurethanes? These materials have played a pivotal role in achieving the improvement of the human condition. They have long become an inherent part of our everyday lives. In fact polyurethane products are almost everywhere we look. Just think of a Parisian boulevard or elsewhere, sitting outside a café, think—people watching. Watch them wherever you happen to be—at a bus stop, walking down the high street, sitting in

a meeting... Think of what they are wearing, how they are walking (their clothes, their shoes...), what their job might be. Or chose an object in the room. You will find polyurethanes everywhere we look.

Things are moving. If only thinking of the footwear industry—how much it has changed in a century in so many significant ways: machinery, footwear testing, location of manufacturing plants, and especially alternatives to leather where polyurethanes play a leading role. Inexpensively made, polyurethanes do have consistent properties.

Year 1894. The T. & A. Bata Shoe Company in Zlín, The Czech Republic by the siblings Tomáš, Anna and Antonín Bat'a – the eighth generation of shoe-making Batas. (<http://www.batova-vila.cz/EN/Thomas-Bata-Foundation-History.html>)



Or think of the developments brought by polyurethanes in the clothing industry... Here are example pictures of the benefits of progress by doing polyurethane clothes... isn't it remarkable? You can judge yourselves ☺...



Year 2010 new design — 100% PU jacket.

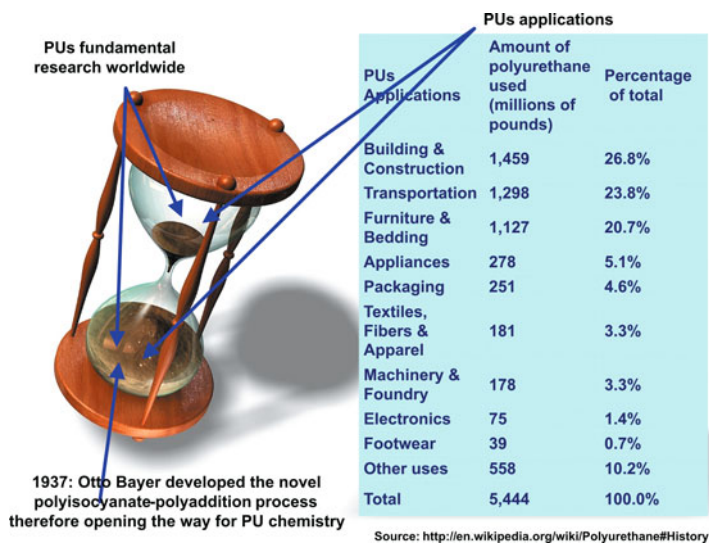


Year 1910. Dress designed by Paul Poiret. (<http://upload.wikimedia.org/wikipedia/commons/4/4b/Poiretdress.jpg>)

Polyurethanes are unique materials where the elasticity of rubber is combined with the toughness and durability of metals which allows the engineer to replace rubber, plastic and metal with the ultimate in abrasion resistance and physical



properties. Polyurethanes have a large variety of applications due to their unique properties, such as high strength, high hardness, high modulus, and high elongation at break. Therefore there are so many problems these materials can solve: from automotive parts to building and construction, from medicine to electronics, from textiles to furniture. And even so, this is not all.



Picture of a polyurethane clepsydra counting history and present of PUs development in the areas of fundamental research and applications

Polyurethanes can be manufactured in an extremely wide range of grades, in densities from 6 to 1 220 kg/m<sup>3</sup> and from flexible elastomers to rigid, hard plastics.

Polyurethane materials exist in a variety of forms including flexible or rigid foams, chemical resistant coatings, specialty adhesives and sealants, and elastomers. Most polyurethanes are *thermoset materials*; they cannot be melted and reshaped as thermoplastic materials. Once the reactions have ceased the thermoset polyurethanes are cured and cannot be heat shaped without degradation. The thermal stability results from the crosslinking degree of polymer chains (the crosslink density) and from the nature and frequency of repeating units within the polymer chains.

*Thermoplastic polyurethane elastomers* are polymers that bridge the gap between rubbers and plastics. They can be used in a wide range of properties, from hard rubbers to soft engineering thermoplastics as they are elastic and melt-processable. They can be processed on extrusion as well as injection, blow and compression molding equipment. They can be vacuum-formed or solution-coated and are suited for a wide variety of fabrication methodologies. They provide a considerable number of physical property combinations: high resilience, good compression set, resistance to abrasions, tears, impacts, weather, and even hydrocarbons. Such materials

offer flexibility without the use of plasticizers as well as a broad range of hardness and high elasticity.

The world's consumption of polyurethanes has been growing continuously because as materials they offer so many different properties and unique ecological and economic advantages. In fact, its consumption rate is increasing by up to 5% a year. (source: [www.inntecsr.eu/cms](http://www.inntecsr.eu/cms)). And there seems to be no end to this trend, quite the opposite actually: polyurethane systems are finding new fields of application and market segments: in Europe or the U.S., in Central or South America, in Asia, Africa or Australia.

Over 2 million people are employed in industries involving polyurethanes, of which more than 800 000 workers are in the EU. The market value of the polyurethanes industry in the EU today is over €150 billion (source: [www.polyurethane.org](http://www.polyurethane.org)).

*Cristina Prisacariu*  
*April 2011*

# Preface

*Everything connects to everything else.* (Leonardo da Vinci)

Things are always changing, while everything connects together to everything else, to form a balance of our chances, experiences and things, so to maintain *a structure*. From letters that build the word to language, from the relationship between our actions to their consequences, everything is connected. From social networking to digit communication, or from neuron networks in the brain to chemical neuromediators generating thoughts and viewpoints of people, everything is connected.

They all work together, while each relationship is a discrete thing in itself.

The overwhelming majority of research studies rely on the opinions of individuals, but all of them are linked.

The connections go beyond human relationship. Various systems of interacting things act together to perform a common goal. The details make the product. In this picture, polyurethanes architecture is a small world where everything is linked to everything too by a two — phase microstructure consisting of hard and soft domains. The overall properties of polyurethanes depend on the intrinsic properties of each of the phases, which in turn depend on details of molecular packing of the constituents within the phases, including the density of hydrogen bonds.

There are no hard and fast rules for obtaining the optimum polyurethane elastomers end products, success depends on good formulation selection with well chosen and appropriate processing parameters. Polyurethane elastomers are polymers with remarkable versatility. Through suitable choice of the diisocyanates and diols combined in their synthesis, a huge range of physical properties are achievable. For example, these may be varied from those typical of soft elastomers to those of hard plastics, simply by varying the dominant diol from a flexible long-chain molecule to a small molecule such as ethylene glycol.

This versatility is combined with the processing advantages of a thermoplastic. It is therefore understandable that the polyurethane elastomers are employed in an exceptionally wide range of manufactured products.

An important feature of polyurethane elastomers is that their elastomeric behaviour is highly sensitive to chemical and physical structure of the material, that are potentially under the control of the synthesist. For this advantage to be exploited more effectively, however, there is a need for deeper understanding of the mechanisms by which structure determines the properties of importance.

The present book aims to advance this understanding, by means of a systematic study of the effects of varying chemical composition of model polyurethane elastomers on: (a) their physical structure at the important nanometre length-scale, and (b) the resulting mechanical properties of interest.

The book reviews aspects from the up-to-date literature focused on these topics including our research. In addition the book records selected results from an international NATO project collaboration between The Department of Engineering Science, University of Oxford and our Romanian laboratory at the Institute of Macromolecular Chemistry “Petru Poni”, Iasi. This has made possible an unusually comprehensive study of the structure and important physical properties of polyurethane elastomers—a class of polymer of such great industrial importance.

A particular topic approached in this book is new insight into the physical origin of inelastic effects in reinforced elastomers, to assist with the development of physically-based constitutive models.

In the book, a study was made of how aspects of the constitutive responses of polyurethane elastomers vary with composition: the polyaddition procedure, the hard segment, soft segment and chain extender (*mostly diols*) were varied systematically in a large number of systems of model and novel materials. Results were related to: microstructural changes, on the basis of evidence from X-ray scattering, scanning and transmission electron microscopy, atomic force microscopy, and also dynamic mechanical analyses, differential scanning calorimetry and IR dichroic measurements.

The book covers aspects from the morphology to mechanical aspects focused on the elasticity and inelasticity of amorphous to crystalline polyurethane elastomers, in relation to their sensitivity to chemical and physical structure. In such polymers, resilience of the material is an important attribute. In many applications they are in commercial competition with other, relatively soft, elastomeric materials. The choice of material for any given application then hinges on a spectrum of key properties offered by relatively soft polymers—stiffness and strain recovery characterizing their elasticity, but also inelastic effects such as hysteresis and stress relaxation. In these respects the mechanical properties of polyurethane elastomers are similar to those of other elastomers.

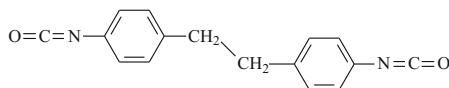
In this book, inelastic effects correlated with systematically varying the chemical structure, were also investigated by including quantitative correlations between the magnitude of the Mullins effect and the fractional energy dissipation by hysteresis under cyclic straining, giving common relations approached by all the materials studied.

*A major structural feature that was explored in this book is the relationship between the nature of the hard segment (crystallizing or not) and that of the soft segments. Crystallinity has been sometimes observed in the commercial polyurethane*

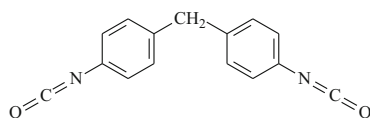
elastomers hard phase but this is usually limited to only a few percent for most hard segment structures when solidified from the melt.

*But there is one particular diisocyanate, 4,4'-dibenzyl diisocyanate (DBDI) that, in the presence of suitable chain extenders (diols or diamines), gives rise to significant degrees of crystallinity and this is included in the present work.*

Schematic of the flexible  
4,4'-dibenzyl diisocyanate  
DBDI crystallizing



Schematic of a conventional  
rigid diisocyanate,  
4,4'-diphenylmethane  
diisocyanate (MDI)  
non-crystallizing



Recent developments in polyurethane materials have been made in our Romanian laboratory, by producing and studying polymers based on the diisocyanate DBDI giving dibenzyl based hard segments (only available from Romania), that allows the variation of hard domain crystallinity as a key structural variable. The conformational mobility of the novel DBDI causes an unusually wide range of mechanical, physical and chemical properties, associated with the possibility of pronounced phase separation into a domain-matrix morphology, and with a high tendency to crystallization and self-association by hydrogen bonding, which is not available with the conventional diisocyanates in traditional melt-cast polyurethanes.

Thus, new polymers were achieved by us, with a controlled ordering of copolymer hard segment blocks on the macromolecular chain.

*This book concerns with the study of a large series of systems of PUs based on the aromatic DBDI diisocyanate as compared to materials derived from conventional diisocyanates, mainly 4,4'-diphenylmethane diisocyanate (MDI), as one of the most representative and commonly used aromatic diisocyanates.*

The present book is organized into 6 chapters. Chapter 1 describes general aspects on the chemistry of polyurethane elastomers: their origins and development, the principles and synthesis mechanisms, as well as general considerations on the main chemical parameters that define such materials, i. e. diisocyanate, macrodiol and chain extender. Selected considerations regarding the reactivity of diisocyanates, the hydrogen bonding and its dynamic and quantum aspects are also discussed in this chapter.

In chapter 2, there are investigated aspects regarding the morphology and phase separation on two large categories of polyurethane elastomers: materials based on *single* diisocyanates, and materials derived from *mixtures* of diisocyanates. Results are discussed in terms of the structural studies: wide and small angle X-ray scattering (WAXS) and (SAXS), and transmission and scanning electron microscopy

(TEM and SEM) revealing particularities on the polyurethane elastomers morphology. Conventional materials derived from rigid diisocyanates (*mainly MDI*) are compared with the relatively recent polyurethane elastomers obtained by us, that contain crystallizing hard segments generated by DBDI which displays a variable geometry. *Such materials have higher flow stress in the hard phase caused by stronger phase segregation.*

Thermal techniques such as differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) used to describe the thermal behaviour of the materials are also detailed in this book. A comparison is made in chapter 3, between the thermal behaviour of conventional materials and those derived from dibenzyl structures, from the perspective of the *materials with single diisocyanates as compared to those based on mixtures of diisocyanates*. The thermal behaviour of a series of novel polyurethane blends obtained with the isocyanate DBDI has also been described in chapter 3.

The mechanical performance of polyurethane elastomers is strongly affected by the higher-ordered structure of hard segments on the macromolecular chain, and this is also discussed to a large extent in the present book. Inelastic features in the polyurethane elastomers constitutive response are investigated in chapter 4. Their constitutive responses are sensitive to microstructural detail on length scales of order nm and the hard-domain crystallinity exerts a strong effect on inelasticity of the elastomers and this is largely discussed in chapter 4. Tensile properties and the cyclic tensile responses are investigated and the correlation between the Mullins response and hysteresis is made for a large number of polyurethane elastomers with varying systematically the chemical structure: diisocyanates, macrodiols and chain extenders. A comparison has been made between the mechanical response of elastomers achieved without and with excess of isocyanate NCO groups. For the latter, a study of the kinetics and postcure reaction mechanisms has been devoted.

Special consideration was given to the investigation of polyurethane elastomers deformation induced morphological developments and crystallization. In chapter 5, a brief review has been made of selected research in this area, by including materials with hard segments crystallizing or non-crystallizing, based on diisocyanates of variable geometries.

Chapter 6 is dedicated to a detailed description of a new family of crosslinked polyurethanes, with regard to their thermorheological characterization as shape memory materials and the prediction of their shape-memory performance.

*Recent progress has been done by us, in the field of PUs chain extended with diols. For this reason, in the present book I have mainly focused on this category of materials.*

Understanding the reaction pathways involved, in resolving the subtle morphological evolution at the nanometre level, and capturing mathematically the complex, large-deformation nonlinear viscoelastic mechanical behaviour are assumed to bring new important insights in the world basic research in polyurethanes and towards applied industrial research in this area, and this is the overall target of this book.

Which is why, this book is addressed not only to chemists from both academia and industry, but to all those who are interested in the latest developments in the science of elastomeric materials.

I started from simple to complex approaches so that hopefully this book is also useful and of interest for graduate students and in general for younger audience.

However, while doing so, undoubtedly I am aware that the road to success is always “under construction” ☺.





# Acknowledgements

*Gratitude is the memory of the heart.* (Jean Baptiste Massieu)

Being thankful means to share, and this is what I desire to do, by bringing here my deep appreciation and thankful thoughts to all those whom I have felt close to me while writing this book.

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*Cristina Prisacariu*  
April, 2011

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# Abbreviations and acronyms

PU	polyurethane
SS	soft segment
HS	hard segment
MD	macrodiol
CE	chain extender
DI	diisocyanate
I	isocyanate index
2,4-TDI and 2,6-TDI	toluene-2,4- and toluene-2,6-diisocyanate
MDI	4,4'-methylenebis(phenyl isocyanate)
DBDI	4,4'-dibenzyl diisocyanate
HDI	1,6-diisocyanatohexane
HMDI	hydrogenated MDI
IPDI	isophorone diisocyanate
PEG or PEO	poly(oxyethylene) glycol
PEA	poly(ethylene adipate)diol
PBA	poly(butane adipate) diol
PTMO or PTHF	poly(oxytetramethylene) diol or polytetrahydrofuran diol
PBU	poly(butadiene)diol
PCL or PCD	polycaprolactone diol
BD, BG or BDO	1,4-butanediol
EG	ethylene glycol
DEG	diethylene glycol
$M_n$	number-average molecular weight of polymer [g/mol]
GPC	gel permeation (size exclusion) chromatography
SEM	scanning electron microscopy
DSC	differential scanning calorimetry
DMA	dynamic mechanical analysis
TG	thermogravimetric analysis
SAXS	small angle X-ray scattering
WAXS	wide angle X-ray scattering
ASTM	American Society for Testing and Materials

$T_G$	glass transition temperature
$T_m$	melting point
$E^*$	complex modulus
$E'$	loss modulus
$E''$	loss factor $\tan \delta$
$E_c$	input energy
$E_r$	recoverable energy