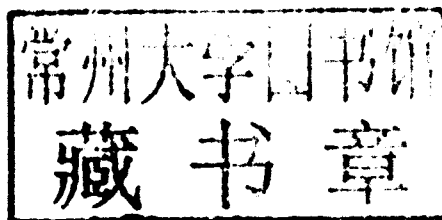

ADVANCED ELASTOMERS

**TECHNOLOGY, PROPERTIES AND
APPLICATIONS**

Edited by **Anna Boczkowska**

ADVANCED ELASTOMERS – TECHNOLOGY, PROPERTIES AND APPLICATIONS

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Advanced Elastomers – Technology, Properties and Applications

<http://dx.doi.org/10.5772/2784>

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Published by InTech

Janeza Trdine 9, 51000 Rijeka, Croatia

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Publishing Process Manager Ivona Lovric

Typesetting InTech Prepress, Novi Sad

Cover InTech Design Team

First published September, 2012

Printed in Croatia

A free online edition of this book is available at www.intechopen.com
Additional hard copies can be obtained from orders@intechopen.com

Advanced Elastomers – Technology, Properties and Applications,

Edited by Anna Boczkowska

p. cm.

ISBN 978-953-51-0739-2

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Preface

Elastomers due to their versatile properties have been widely explored in academic and industry since they were invented in the nineteen century. By definition, elastomer is a polymer with viscoelasticity, having low Young's modulus and high yield strain in comparison to other materials. The term "elastomer" is derived from "elastic polymer". The elasticity is caused by the ability of the long chains (macromolecules) to reconfigure themselves to distribute an applied stress. Elastomers are amorphous polymers used above their glass transition temperature, so that significant motion of macromolecule's segments is possible. The chemical (covalent) or physical (hydrogen) cross-linkages ensure that the elastomer returns to its original shape after deformation, when the stress is removed. As a result of this extreme flexibility, elastomers can reversibly extend from a few up to one hundred percent or even more, dependently on their chemical and physical structure. Elastomers can be thermoplastics or thermosets if the segments of macromolecules are cross-linked during curing. They are commonly used in a large range of industrial and household applications, however, the progress in development of new methods of synthesis and characterisation, new materials, especially "smart" and nanocomposites based on elastomer matrix is still up-to-date.

This book provides an extensive overview of current trends in the area of elastomers and their composites from the chapters contributed by internationally recognized specialists. The book deals with novel synthesis, modelling and experimental methods in elastomers and provides a unique opportunity to discover the latest research on elastomer advances from laboratories around the world. Numerous references are given at the end of each chapter to enable the reader to explore the topics covered in greater detail.

The book has a broad scope and brings together 7 Chapters that deal with the various aspects of processing, as well as experimental and analytical approaches to elastomer characterisation. Each section demonstrates how enhancements in materials, processes and characterization techniques can improve performance in the field of engineering.

Chapter 1 presents new approach to crosslinking of elastomers by means of electron beam. Usually elastomers show low thermal conductivity, and therefore, require complex and high cost heating methods; thus, the ionizing (accelerated electrons)

method shows high interest for the grafting and cross-linking processes. In addition to the lack of environmental impact, reliability, flexibility and low costs render the radiation technologies especially attractive.

Chapter 2 contains the overview on liquid crystal elastomers, which combines the properties of liquid crystals order characteristic with the elasticity typical for the conventional rubbers, as they are today being considered as promising candidates for media that can easily be driven by external stimuli (heat, electromagnetic or optic field) for applications such as data storage, image processing, or optical modulators.

Chapter 3 discusses structure, rheological and vulcanization properties of elastomer based nanocomposites and presents the remarkable improvements of mechanical and barrier properties of a rubber matrix, obtained due to addition of clays or hybrid pigments, that led to their application in tire and sport goods. Mechanical properties, spectrophotometric and DSC data of the vulcanizates produced with the composite pigment fillers are also reported before and after being aged under UV radiation.

Chapter 4 provides information about smart materials such as magnetorheological elastomers and thermo-shrinkable-elastomers. The overview on magnetorheological elastomers is given, as well as basic knowledge about rheology and magnetorheology. The effect of the amount, size and orientation of the particles on their microstructure and properties is discussed. Also in this chapter the crosslink density, the static and dynamic mechanical properties and the thermo-shrinkable behaviour of vulcanisates is described. The effect of the ionic crosslink content on the ability of the vulcanisates to recover their primary shape upon thermal treatment is discussed.

Chapter 5 reviews the recent research advances in the emerging field of elastomeric electronics. Various strategies in implementing electronic devices and systems that can be flexed, twisted, and compressed are addressed, with special emphasis on stretchable microfluidic electronics. The recently demonstrated elastomeric electronic devices, e.g. foldable and stretchable antennas, radiation sensors, as well as wireless strain sensors, using the microfluidic approach, are presented and analyzed in detail. Examples of elastomers used in microelectronics and microfluidics are shown, as well as elastomer based composites with conductive particles as a filler. Future perspectives and outlook for elastomeric electronics are discussed.

Chapter 6 aims at expounding properties, application and performance of natural rubber latex (NRL) as a modifier in cement concrete and mortar. Properties of NRL influencing performance in mechanical and durability functions are discussed. Also influence of high temperature on concrete strength and durability is shown. Recent trends in research activities and challenges facing applications of elastomers are provided.

In Chapter 7 the development of experimental testing and various analytical, as well as FEM models for characterising of elastomer microstructure and properties are presented.

This book addresses to industrial and academic researchers in the fields of physical, chemical, biological sciences and engineering. It is intended to be useful not only for to engineers, academics, researchers and designers in the field of materials sciences, but also to postgraduate research students and manufacturers of elastomer's products.

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New Approach to Elastomer Crosslinking

Aspects Regarding Radiation Crosslinking of Elastomers

Elena Manaila, Maria Daniela Stelescu and Gabriela Craciun

Additional information is available at the end of the chapter

<http://dx.doi.org/10.5772/47747>

1. Introduction

Elastomers or rubbers are materials used in a large range of industrial and household applications. A common physical-chemical treatment is curing (crosslinking), imparting the rubber mechanical and thermal stability. Elastomers show low thermal conductivity values, and therefore, require complex and high cost heating methods; thus, the ionizing (accelerated electrons) method shows high interest for the grafting and crosslinking processes. In addition to the lack of environmental impact, reliability, flexibility and low costs render the radiation technologies especially attractive.

Vulcanisation is the process by which rubber is changed from essentially a plastic material to either an elastic or a hard material (Stelescu et al., 2010). In this process, an elastomer is transformed from a 'plastic', 'formable' material into an 'elastic' material by the formation of a three-dimensional network with different types of junctions. The word vulcanisation derives from *Vulcan*, the Roman God of fire. Not accidentally, it also means volcano, a hot place where quite some sulphur species can be found. The term vulcanisation was therefore originally exclusively applied to the crosslinking reaction achieved by sulphur at high temperatures.

Nowadays this term is also applied to refer to other crosslinking processes, such as peroxide cure. There are several possibilities for the crosslinking of rubber. The already mentioned sulphur vulcanisation was the first to be discovered and still is today's most common cure system. The sulphur vulcanisation process requires the presence of carbon-carbon unsaturation in the polymer and it leads to a three-dimensional rubber network in which the polymer chains are linked to each other by sulphur bridges. As a result, sulphur cured articles have good tensile and tear strength, good dynamic properties, but poor high temperature properties like ageing, for instance (Alvarez Grima, 2007; Dluzneski, 2001).

Other vulcanisation systems, i.e. peroxides, ultraviolet light, electron beam, microwave, resins, etc. were later discovered and gained more importance with the progressive development of synthetic rubbers.

The use of organic peroxides as crosslinking agents for rubber was first reported by Ostromislenski in 1915 (Alvarez Grima, 2007) and at present it is the only vulcanization method that can compete with accelerated sulphur cure, with respect to vulcanization rate. Interest in the industrial use of peroxides as curing agents increased with the introduction of a number of fully saturated elastomers, such as ethylene-propylene rubber (EPM), fluoro elastomers (FKM), etc., which cannot be cured by sulphur vulcanization (Alvarez Grima, 2007). Peroxide vulcanisation leads to a rubber network in which the polymer chains are linked to each other by very stable covalent carbon-carbon bonds. Peroxide cured vulcanisates have therefore good high temperature properties, like heat ageing and compression set, compared to sulphur cured articles (Alvarez Grima, 2007; Dluzneski, 2001)

In addition, peroxide cure allows vulcanisation of both unsaturated and saturated polymers, which is not possible with sulphur vulcanisation. On the other hand, some mechanical properties of peroxide cured articles are inferior to those achieved by sulphur cure, i.e. tensile strength, dynamic properties, and therefore entail a limitation on the use of this vulcanization system. Another serious problem with peroxides is the lack of sufficient scorch time. Scorch time: the time elapsed until vulcanisation starts, is of major importance in order to control the vulcanisation reaction. The term scorch safety refers to a certain scorch time which is enough to provide good processing of the material before it starts vulcanising; this is of great importance especially in industry. Upon reaching the typical dissociation temperature of a peroxide, the crosslinking reaction immediately proceeds at full speed, leaving the processor little or no time for shaping of the rubber article. The addition of certain scorch-retarders is sometimes of help, but this usually goes at the expense of crosslink density obtained: a waste of money (Alvarez Grima, 2007).

2. Electron beam and microwaves vulcanization

Radiation curing has historically been used as an alternative to peroxides in applications where the curatives themselves or sideproducts of vulcanization are viewed as impurities in the final product. Peroxide cure progresses through a series of radical intermediates, each of which can undergo side reactions which may not necessarily contribute to crosslink density. Radiation cure, on the other hand, has been promoted as a cleaner and more homogeneous cure process. Electron beam irradiation has been used in the wire and cable industry for longer than 30 years and applied to a wide range of commodity and specialty elastomers. A survey of the types of elastomers susceptible to radiation curing is available, as are review articles describing the electron-beam curing of commercially significant grades (Bhowmick & Vijayabaskar, 2006; Henning, 2008). Variables such as radiation dosage and the effect of polymer microstructure and chemical additives on the efficiency of electron beam cure have been studied. Some researchers (Zaharescu et al., 2000; Chowdhury & Banerji, 2005) studied radiation-induced crosslinking in thermoplastic elastomers based on ethylene-propylene rubber (EPDM) and polyethylene (PE) or polypropylene (PP) plastics.

Elastomer crosslinking by means of electron beam (EB) is done without heating and in the absence of vulcanization agents. The reaction mechanism is similar to crosslinking with peroxides, but in this case, reaction initiation is due to the action of EB and in the presence of the polyfunctional monomers. Ionizing radiation produces an excitation of polymer molecules. The energies associated with the excitation are dependent on the irradiation dosage of electrons. The interaction results in formation of free radicals formed by dissociation of molecules in the excited state or by interaction of molecular ions. The free radicals or molecular ions can react by connecting the polymer chains directly or initiating grafting reactions.

EB vulcanization has demonstrated extremely positive results compared to the conventional curing system such as: no polymer degradation due to high temperature as EB crosslinking occurs at room temperature, no oxidative degeneration in polymers as observed in classical crosslinking, direct crosslinking by C-C linkage by EB, extremely strong bonds, high degree of crosslinking, extremely short curing cycles, zero blooming effects; extremely high tensile strength; extremely high resistance to compression set; extremely high resistance to oils, grease, lubricants; highly improved accelerated ageing properties, very high productivity, perfect for thin products, lower material waste (MGM Rubber Company-Research and Development, 2007). However, the radiation crosslinking of rubbers was not used in larger technical applications because of the high cost of irradiation to bring about vulcanization, but could become an industrial process when the radiation dose decreased with the use of some sensitizers.

Modification of thermoplastic and rubbery materials by EB and microwave (MW) radiations is a potential method for the development of new materials like polymers and composites. Interaction of each of the two physical systems, EB and MW, with a substance has revealed their ability for changing physical and chemical properties of the treated substance.

Therefore, the physical and chemical effects of the EB and MW radiation have drawn high interest, resulting in a large industrial application range of the new materials with improved characteristics. A comparative study of the effects of separate interaction of EB and MW with the substance, effects on which the modern processes for the production of new materials with special characteristics have been developed, has revealed the following: (a) production of materials by EB (ionizing) radiation is based on the coulomb interaction of accelerated electrons with the atoms of the substances they are penetrating. From this interaction, secondary electrons, excited molecules, free ions and radicals result, which are precursors of some chemical reactions resulting in new materials; (b) production of materials by MW (non-ionizing) radiation is based on thermal effects (dielectric heating) of the interaction of the electromagnetic waves with the substance. MW energy absorbed within the substance is dependent on the molecule characteristics, that is on the permittivity and magnetic permeability resulting in a high selectivity level for the chemical reactions induced by MW interaction with the constituents of the material they are penetrating; (c) by separate/ combined EB and MW irradiation, materials with single characteristics are obtained with high yields, inaccessible by the traditional curing methods.

Radiation (EB-ionizing radiation and MWs-non-ionizing radiation) are used successfully instead of the traditional methods and, in addition, provides new possibilities like as

crosslinking in thick polymer articles, in complex sections or production of recycled rubbers of high quality.

The high-molecular compounds (elastomers) are made up of a large number of elementary molecules – monomers. These molecules can be of the same class or of different classes, resulting thus homopolymers and copolymers, respectively. The radiation acts on the high-molecular compounds as follows: (1) at a relatively low radiation dose (radiation dose = the energy absorbed per unit of mass of the irradiated material; it is measured in J/Kg; unit of measure for the absorbed dose is a gray: $1\text{Gy} = 1\text{ J/kg}$), only „pure polymerization” occurs. With the linear increase in the radiation dose, the linear polymer chains increase progressively by addition of a molecule from a blend with low molecular weight to a radical at the end of an increasing chain; (2) with the dose increase during the pure polymerization, a new process named „grafting” takes place. By this process side chains are made in the polymer, resulting in modified surface properties in some materials, like as: biocompatibility, moisture-absorbent and moisture-repellent, and mechanical, chemical and thermal characteristics. This process is very promising for the biomaterial synthesis. (3) with the further increase in radiation dose, the „crosslinking” process takes place, when links are formed between polymer chains resulting in two- and three-dimensional structures. During this process insignificant chemical changes occur but significant structural changes. Simultaneously degradation occurs. Crosslinking and degradation (through chain scission) are two competing processes that always co-exist under radiation. Both processes have many applications in the field of new materials with improved characteristics, like as elastomer crosslinking. (4) the combined effects of the two processes are used in many industry processes, being known as „radiation curing”. Three radiation induced processes are used in „radiation curing”: monomer polymerization, polymer chain crosslinking and chemical bonding between polymer and substrate molecules.

Use of ionizing and non-ionizing radiation to obtain materials with improved characteristics is based on their advantages like as: (I) *electrons beam (EB)*: (I.1.) accelerate the polymerization/vulcanization process, thus reducing the preparation time with tens to hundreds times; (I.2.) increase the conversion of raw material into the finished material up to a 100% yield and, therefore, the percentage of the residual monomer is nearly 0%, which is very important with noxious monomers; (I.3.) reduce the energy required in crosslinking and grafting with 5 –10 times as compared with the traditional method; (II) *microwaves (MW)*: (II.1.) promote a narrow distribution of the molecular masses, as the crosslinking and grafting occurs simultaneously in the bulk due to the rapid energy transfer, volumetric and selective properties of MW dielectric heating, (II.2.) promote fast crosslinking and grafting processes, by tens to hundreds times faster than in classical procedure because the electromagnetic energy is directly transferred from the MW to the atoms or molecules of the irradiated material

3. Polyfunctional monomers (co-agents)

Reported papers suggest that appropriate polyfunctional monomers (co-agents) in polymer matrix (Vijayabaskar & Bhowmick, 2005; Yasin et al., 2005) could be used to obtain desired

rubber physical properties at lower irradiation doses (Hafezi et al., 2006; Stelescu et al., 2011; Stelescu et al., 2012). Co-agents are multi-functional organic molecules which are highly reactive towards free radicals (Alvarez Grima, 2007). They are used as reactive additives to boost the vulcanization efficiency (Endstra, 1990). The most used coagents are molecules with maleimide groups, (meth)acrylate groups, or allylic groups, (Dikland et al., 1993) but polymeric materials with a high vinyl content, i.e. 1,2-polybutadiene, can also act as co-agents.

The co-agents can be divided into two groups: Type I and Type II co-agents.

Type I: Addition and hydrogen abstraction reactions: these co-agents consist of rather polar molecules with a low molecular weight and activated double bonds. Their main characteristic is that they are highly reactive towards radicals, so scorch takes place very fast, which sometimes can be a disadvantage. By using this kind of coagents not only the rate of cure is increased but also the crosslink density or state of cure. A disadvantage that may be present when using this type of co-agents is that, due to polarity, the compatibility of these co-agents with the polymer matrix is limited. Some examples of Type I co-agents are: acrylates, methacrylates, bismaleimides and zinc salts.

Type II: Addition reactions: these co-agents are, in general, less polar molecules, which form more stable free radicals, so scorch does not take place as fast as with the previous type of co-agents. The use of these co-agents leads to an increase in crosslink density of the vulcanisate but, unlike Type I, they are not capable of increasing the cure rate. Due to their low polarity, these co-agents have a good compatibility with many elastomers. Some examples are: high-vinyl 1,2-polybutadiene, divinylbenzene, allyl esters of cyanurates, isocyanurates and sulphur.

This review gives an overview about our research (Stelescu & Manaila, 2007; Zuga et al., 2007; Zuga, Miu et al., 2007; Manaila et al., 2008; Stelescu et al., 2010; Manaila, Martin, Stelescu et al., 2009; Stelescu et al., 2009; Manaila, Martin, Craciun et al., 2009; Stelescu et al., 2008; Manaila, Stelescu, Ighigeanu et al., 2011; Manaila, Stelescu et al., 2011) on elastomer crosslinking by irradiation with accelerated electrons, a much more ecologic method that does not need to add crosslinking agents into the blend.

In addition, the main advantages that the new technique brings are: (a) almost 100% efficiency in converting raw material into finished materials; (b) reduction from tens to hundreds of times of the production length; (c) achieving unique properties of materials which cannot be obtained by conventional methods; (d) perfect adaptability to the highest demands on the environment because no reaction by-products are released into the atmosphere; (e) adaptability to any degree of automation (allows technological lines with speeds of over 500 m/min) and strict control of technological processes; (f) in many cases, reduction of energy consumption by 20 to 30 times compared to "classic heating" or conventional processes; (g) the process is simple and can be controlled by only one single parameter, i.e. absorbed dose, the quantity that varies with the application; (h) radiation crosslinking is technically and economically feasible as a pure physical process, i.e. without

the addition of sensitizers, there are no residues of alien substance needed for the chemical processes, or of their decomposition products.

At the interaction of ionizing radiation with (co)polymers, breaking of covalent bonds occurs, as well as the emergence of free radicals (transitional chemical species) on the main chain (if the lateral groups break) or in the main chain (if it breaks itself). The final effect is either crosslinking of macromolecular assembly or cutting the main chain of macromolecules and decreasing average molecular weight. In fact, the two effects, crosslinking and degradation, coexist and we need to point out the predominance of one of them.

In our study we used five polyfunctional monomers: TAC (triallylcyanurate) and TAIC (triallylisocyanurate) of type II, and TMPT (trimethylopropane trimethacrylate), EDMA (ethylene glycol dimethacrylate) and ZDA (zinc diacrylate) of type I.

Table 1 presents the chemical structure, type, functionality, and characteristics of polyfunctional monomers (co-agents) PFMs used.

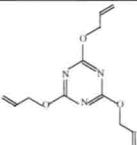
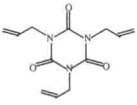
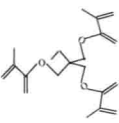
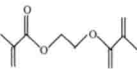
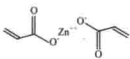
PFMs	Type	Chemical structure	Characteristics
Triallylcyanurate Luvomaxx TAC DL 70 (TAC)	II		Melting point: 26–28 °C; Boiling point: 149–152 °C; Density: 1.34 g/cm³; 26% percentage of ash, 30% active synthetic silica.
Triallylisocyanurate Luvomaxx TAIC DL 70C (TAIC)	II		Melting point: 26–28 °C; Boiling point: 119–120 °C; Density: 1.34 g/cm³; 30% active synthetic silica.
Trimethylolpropanetrimethacrylate Luvomaxx TMPT DL 75 (TMPT)	I		Melting point: -25 °C; Boiling point: >200 °C; Density: 1.36 g/cm³; 22% percentage of ash, 75 ± 3% active ingredient.
Ethylene glycol dimethacrylate Luvomaxx EDMA DL 75 (EDMA)	I		Melting point: -40 °C; Boiling point: 85 °C; Density: 1.25 g/cm³; 23% percentage of ash, 75 ± 3% active ingredient.
Zinc-diacrylate ZDA GR 75 (ZDA)	I		Melting point: 240–244 °C; Boiling point: 141 °C; Density: 1.23 g/cm³; 75 ± 3% active ingredient.

Table 1. Characteristics of polyfunctional monomers (co-agents) PFMs used