RADIATION PROCESSING OF POLYMER MATERIALS AND ITS INDUSTRIAL APPLICATIONS

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PREFACE

Radiation processing of polymers involves treatment of polymer materials with ionizing radiation to modify their physical and chemical properties to make property improvement and add value. The industrial applications of radiation processing of polymers are an important part of the peaceful use of nuclear energy besides nuclear power generation. Radioisotopes and ionizing radiation–generating accelerators have found a very wide range of applications in medicine, agriculture, industry, transportation, space, and environmental protection. In the United States, the overall sales revenue from nonpower applications of radioisotopes was US$257 billion in 1991 and US$331 billion in 1995, more than three times that from nuclear power generation [1]. In Japan, the net sales revenue from nonpower applications was US$37.3 billion in 2005 [2]. (The big difference between the United States and Japan in the economic scale of nonpower applications comes from the different estimation methods. The U.S. method was based on input–output analysis, and the overall sales revenue included indirect costs. The Japanese estimation was based on the net shipment value.)

Radiation processing of polymers is a part of the nonpower applications and mainly consists of crosslinking, curing, grafting, and degradation. The main products produced by radiation crosslinking in the world include

- Wire and cable insulation materials
- Heat-shrinkable products
- Plastic foams
- Gaskets and seals
- Polyethylene pipes
- Polymeric positive temperature coefficient (PTC) products
- Prevulcanized components of radial tires
- Hydrogel wound dressings
- Molded engineering plastics
- Components of hip and knee joint prostheses

The types of radiation crosslinked products differ greatly from region to region. For example, radiation crosslinked polyethylene pipes are produced mainly in Europe and not produced in Japan and the United States. Table 0.1 shows the shipment values of radiation-processed products in Japan

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TABLE 0.1 Shipment Values of Radiation-Processed Products in Japan in 2005 (¥110.21/US$)

Radiation Process	Product	Shipment Value (millions of US$)
Crosslinking	Radial tire	1,527
	Wire and cable	199
	Plastic foam	160
	Heat shrinkable plastic	150
	Others (SiC, Hydrogel, etc.)	1
Subtotal		*2,037*
Curing	Paper and film (coating and printing)	27
Grafting	Battery separator, adsorbents	23
Degradation	PTFE powder	4
Total		*2,091*

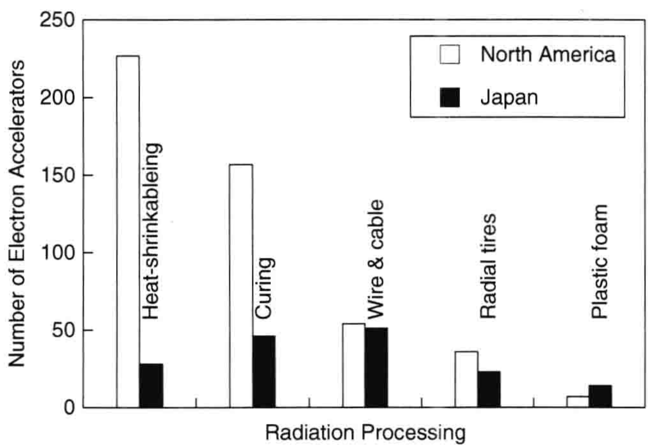

FIGURE 0.1 The number of electron accelerators for radiation processing installed in Japan and North America from 1970 to 1998.

for 2005 [3]. About 93% of the total shipment value is accounted for by crosslinked products. The contribution of radial tires is especially high (73%) in Japan.

Figure 0.1 shows the number of electron accelerators for a few radiation-processing applications installed in Japan and North America (United States, Canada, and Mexico) from 1970 to 1998 (a period of 29 years) [4]. The total number of electron accelerators installed was 308 for Japan and 648 for North America. A large number of accelerators were installed for the applications of heat shrinkable plastics and curing in North America. There is no big difference between Japan and the United States in the number of accelerators for wires and cables and radial tires. More accelerators were installed for plastic foam in Japan than in the United States.

Modification of polymer materials is a significant part of the industrial application of radiation processing. Besides the economic benefits, radiation processing of polymers also has many technical and environmental advantages. The low pollution and low energy consumption of radiation processing of polymers, along with its potential in polymer recycling and other environmental protection areas, makes it a green technology with exceptional attractiveness in the era of sustainability of the 21st century.

For more than a half century, processing of polymer materials by ionizing radiation on commercial scale has been demonstrated to be a very effective means of improving physical properties of various polymers. It is a well-established and economical method of precisely modifying the properties of bulk polymer resins and formed polymer components. The reactions of crosslinking, chain scission, oxidation, grafting, and long chain branching on polymers initiated by radiation have found many useful applications in plastic and rubber materials. Important properties of polymer materials, such as mechanical properties, thermal stability, chemical resistance, melt flow, processability, and surface properties can be significantly improved by radiation processing. To name a few well-known examples, radiation crosslinking of plastics has been employed for decades to improve properties for wires and cables, heat-shrinkable materials, pipes and tubes, self-limiting heating cables, resettable fuses, and other formed parts. Plastic foams and hydrogels have been manufactured by radiation crosslinking, and rubber materials have also been crosslinked (vulcanized) by radiation. Radiation curing of coatings and inks can be solvent free or use significantly reduced volatile organic compounds (VOCs). Electron beam (EB) processing has been employed to cure fiber-reinforced composite materials and adhesives. Manufacturers have taken advantage of radiation degradation to make fine, micronized polytetrafluoroethylene (PTFE) powders. The processability of polymers has been improved by radiation-induced long-chain branching. Radiation grafting has been shown to be a good method for introducing new functions for base polymers and/or to modify surface properties. The list continues to grow as new applications are continuously being developed [5–14].

Radiation chemistry and effects of radiation on polymers has long been the object of academic research and study. In the 1940s, with the creation of many nuclear energy programs all over the world, radioisotope sources and electronic accelerators that could provide high-energy ionizing radiation became available to many scientists. The interaction of ionizing radiation with all kinds of materials started to draw wide attention. In the 1940s and 1950s, the effects of ionizing radiation on various polymers were investigated. For example, scientists started to understand that some polymers would crosslink while others would degrade after being exposed to radiation. Because crosslinking would generally bring about improvement in mechanical, thermal, and other properties of the polymer, radiation crosslinking was the first to find practical application.

In January 1957, Paul Cook founded the company Irradiated Products, Inc., which later became Raychem, whose main products were manufactured by radiation crosslinking. The company's first products included a flame-retardant, polyethylene-insulated hookup wire, a foamed linear polyethylene subminiature coaxial cable, and heat shrinkable, flame-retardant polyethylene tubing. The initial customers for these early products were from the military because the light weight and superior performance of irradiated products were ideal for military applications [15]. After the 1950s, other companies followed Raychem's pioneering work to develop their own businesses based on radiation processing of polymers, mostly around the applications of wires and cables and heat-shrinkable materials. These material applications now account for about 33% of the market use of high-current industrial electron accelerators [16].

At the same time, great volumes of data were generated and academic theories were quickly developed for radiation chemistry of polymers. In the early 1960s, two important books on radiation chemistry of polymers—by Charlesby and Chapiro, respectively—were published [17, 18]. These books formed the basis for academic theory and the understanding of the effects of radiation on various polymers; they are regarded as classic references for scientists and engineers in the field. Because of his vast publications and patents in the field and his great effort to popularize the technology, Charlesby is thought of as the pioneer of radiation chemistry of polymers [19].

In Japan, radiation processing of polymers started to find commercial application in the 1950s. A comprehensive research report by Lawton et al. [20] inspired many Japanese engineers to apply radiation crosslinking for wire and cable and heat-shrinkable tubing. The results of research and development (R&D) on radiation processing of polymers were presented at the Japan Conference on Radioisotopes (1956–1981) and the Japan Conference on Radiation and Radioisotopes (1983–1998). Applications were expanded into new areas of crosslinking of plastic foams and tire rubber components in the 1960s. Radiation processing of polymers in Japan has been developed with the growth of Japanese car industry.

Throughout the rest of the 20th^{h} century, research on the radiation chemistry of polymers continued to flourish, and more and more new applications were discovered for modification of polymers for property improvement. Since the 1970s, because of the availability of more reliable industrial electron accelerators (e.g., the Dynamitron), the lowering of the operation cost of electron beam accelerators and the optimization of γ-irradiator design and safety, radiation processing of polymers has developed into an industry of its own, with new in-house irradiators and irradiation service providers established all over the world. New commercial applications, such as radiation degradation of PTFE for making ultra-fine powders, emerged. The R&D in the field of radiation chemistry and radiation processing of polymers was active in the United States, Europe, Russia, and Japan in the last quarter of the 20th century. Since the 1970s, R&D in the field of radiation chemistry and radiation processing of polymers has been active in Asia, especially in China. A notable Chinese achievement is the discovery of radiation crosslinking of PTFE [21].

At the same time, the International Atomic Energy Agency (IAEA) began to have more influence on the development of industrial applications of radiation processing of polymers. Through the Regional Cooperative Agreement (RCA) for Research, Development and Training Related to Nuclear Science and Technology for Asia and the Pacific that started in 1972, many developing countries such as China and others in Southeast Asia received scientific and financial help for R&D in the field of radiation chemistry and radiation processing of polymers. Indigenous radiation-processing applications, such as radiation vulcanization of natural rubber latex [22] and radiation degradation of polysaccharide [23] were developed.

The development of the radiation-processing industry in China was about 20 years behind that in the United States and 10 years behind that in Japan. The industry started to emerge in 1980s in the form of "irradiation centers," first within academic research institutions and universities, then in some of the industrial companies. Although the industry had very fast growth after the 1990s, the products in polymer materials were limited to a narrow range, mainly including crosslinked wires and cables and heat-shrinkable materials. The revenue the industry has created is still at a much lower proportion to the whole economy compared to that in the United States and in Japan. Nonetheless, there has been very significant development of the industry from basically nonexistence to a real industry in the past 20 years. By the end of 2008 China already had 140 γ-irradiators with $>300{,}000$ Ci in capacity and over 140 industrial EB accelerators with a total power of $>6{,}000$ kW. The annual revenue from the radiation-processing industry for 2006 was $7.7 billion, including $3.3 billion from ion implantation–related applications. That was 2.7 times the revenue of 2000 and a 20% increase over that of 2005 [24].

Since the 1990s, with a more mature global radiation-processing industry (which includes radiation sterilization, food processing, and processing of nonpolymer materials), several books have been published in English on radiation chemistry and radiation processing of polymers, summarizing new developments as well as previous data [5–9]. These books had different focuses and targeted audiences. The handbook compiled by Tabata et al. [5] was more on the academic side and focused on radiation chemistry of polymers created by Japanese, European, and U.S. researchers. Ivanov's book [6] was mainly meant to be a textbook for graduate students of chemistry and provided examples heavily oriented toward R&D achievements in Russia. The book edited by Singh and Silverman [7] was mainly a collection of academic papers. Wood and Pikaev's book [8] placed emphasis on the chemical changes and principles of various applications and provided good references. However, only a small portion of the book deals with the industrial application of polymers. Most chapters focused on more fundamental and scientific principles and nonpolymer applications such as food processing and radiation sterilization. The book by Drobny [9] focused on ultraviolet (UV) and EB irradiation and stressed radiation technologies more than industrial applications, especially for electron beams. A few books on the same subject were also published in other languages, including Japanese and Chinese [25–29].

A number of technical journals are dedicated to the subject of radiation processing of polymers or regularly publish papers on the subject. Many papers in two of these international journals—namely *Radiation Physics and Chemistry* and *Nuclear Instrument and Methods in Physics Research Session B: Beam Interactions with Materials and Atoms*—are cited herein. Reflecting the recent remarkable economic development of China, many related papers have been published in Chinese journals, mostly the *Journal of Radiation Research and Radiation Processing*, a bimonthly journal published since 1983 by the China Academy of Science. The majority of references herein are technical papers from these journals and industrial patents.

Practical applications for radiation processing of polymer materials have been continuously evolving since the introduction of this technology. For example, in recent years, with increased awareness and concerns about environmental and energy issues, more attention has been paid to natural polymers and biopolymers as well as to more environmentally friendly processes and to the green chemistry of polymers. As a result, there has been more and more R&D on radiation modifications of natural polymers and biopolymers. Examples of other new applications involve adding value to existing and novel polymers. New requirements for polymer materials have been emerging for heat resistance and processability, for example. New technologies, such as radiation crosslinking of engineering plastics for thermal stability enhancement and radiation-induced long-chain branching of polyolefins for processability improvement have found practical and useful industrial applications.

The authors of this book believe that a volume focusing on the practical industrial applications of radiation processing of polymers with detailed discussions about developments that have emerged since about 1990 is needed. This book will benefit many readers, including technical and sales or marketing people in the radiation-processing industry and polymer-related industries as well as scholars and students in the related academic fields. The book has a dedicated focus on radiation processing of polymers. Unlike other books on radiation processing, this book does not include discussions on radiation processing of foods, nonpolymeric materials, or radiation sterilization (except for the issue of radiation stability of polymers during radiation sterilization). It is our hope that this book not only reviews the fundamental principles of radiation chemistry and radiation processing of polymers but also emphasizes how the knowledge and science can be translated into practical applications add value and manufacture marketable products. Special attention is given to the commercial feasibility and economic analysis of the applications. Through case studies of examples, critiques are given to a variety of applications to determine why some have found commercial success while others have floundered. Important factors such as cost vs. added value and competition with chemical processes are analyzed for specific applications. Readers will find summaries of new developments, recent novel applications, and current trends in radiation modification of polymers.

This book is primarily intended to be a reference for scientists, engineers, technicians, sales and marketing professionals in the polymer and radiation-processing industries who are involved in or interested in using ionizing radiation to modify polymer materials for the purpose of improving and adding value to products. Currently there are many radiation-processing service providers around the world. There are also a number of polymer-related industrial companies that own and operate in-house irradiation facilities, mainly with EB accelerators. With the continuous growth of both the polymer industry and the radiation-processing industry globally, there is great need for professionals with sound knowledge and expertise in radiation chemistry and radiation processing, especially in countries new to this field, such as China, India, and countries in Southeast Asia, the Middle East, and South America.

Historically there has been lack of cooperation between the radiation-processing industry and the polymer industry in development of new applications of radiation processing of polymers. The authors of this book hope to help bridge the two industries. In recent years, the authors have given seminars and lectures on the radiation processing of polymers to both the chemical industry and the radiation-processing industry. We believe that many researchers and engineers in both the industries can obtain practical information and useful guidance from this book. It is also the authors' hope that this book can help promote R&D and commercialization of radiation processing in different parts of the world.

Radiation chemistry and the processing of polymers is an interdisciplinary field. Because of this, few universities have specialized programs on the subject. For the past 20 years the community of the radiation processing has had the strong feeling that more college- and graduate-level education on radiation chemistry and processing is seriously needed to promote interest from other industries and cooperation among the radiation-processing industry, the chemical industry, and academia. This book can also be used as a textbook for universities and technical colleges that are involved in education in the field of radiation processing of polymers. It can be used for graduate-level or senior-level undergraduate courses on the subject.

The layout of the book is as the following. After an introduction in Chapter 1, radiation-induced crosslinking is discussed in Chapters 2 to 6. Chapter 2 covers the fundamental aspects of crosslinking and Chapter 3 focuses on ways to enhance radiation crosslinking. Physicochemical properties of crosslinked polymers are reviewed in Chapter 4. Chapter 5 deals with recent progress in the traditional application of radiation crosslinking in the fields of wire and cable insulation, plastic foams, heat-shrinkable plastics, and hot water pipes. Chapter 6 is dedicated to new applications of radiation crosslinking, including positive temperature coefficient polymer products, artificial joints, and engineering plastics.

Chapter 7 discusses radiation-induced chain scission (degradation) and oxidation and includes a section on stability of polymers against radiation sterilization. Chapter 8 reviews radiation curing of composites and adhesives.

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Chapter 9 is unique and contains discussions about crosslinking and degradation of aqueous solutions of polymers, such as hydrogels, bioactive polysaccharides, and natural rubber latex. Chapter 10 covers radiation induced long-chain branching and its applications, a new subject that many other books on radiation and polymers have little or no information on. Another key technology, graft polymerization, is dealt with in Chapter 11. Finally, Chapter 12 discusses some of the newly developed applications and the prospect for the future of the radiation processing of polymers.

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ABBREVIATIONS

14G	polypropylene glycol (#600) dimethacrylate
2G	diethyleneglycol dimethacrylate
3G	triethyleneglycol dimethacrylate
4G	polyethyleneglycol (#200) dimethacrylate
9G	polyethylene glycol (#400) dimethacrylate
9PG	polypropyleneglycol (#400) dimethacrylate
A-4G	polyethyleneglycol (#200) diacrylate
AAc	acrylic acid
AAm	acrylamide
A-BG	1,3-butyleneglycol diacrylate; butanediol diacrylate
ABS	acrylonitrile–butadiene–styrene
ADCA	azodicarbonamide
AECL	Atomic Energy of Canada, Ltd.
AMA	allyl methacrylate
AN	acrylonitrile
A-NPG	neopentylglycol diacrylate
AO	antioxidant
ARL	Army Research Laboratory
ASTM	American Society for Testing and Materials
ATH	aluminum trihydroxide
A-TMMT	tetramethylolmethane tetraacrylate
A-TMPT; TMPTA	trimethylolpropane triacrylate
BDDA	1,4-butanediol diacrylate
BG	1,4-butyleneglycol dimethacrylate; butanediol dimethacrylate
BHT	butyl hydroxytoluene
BIIR	brominated IIR; bromo-isobutylene-isoprene rubber
BMA	butyl methacrylate
BR	butadiene rubber; polybutadiene
CB	carbon black
CF	carbon fiber
CIIR	chlorinated IIR; chloro-isobutylene-isoprene rubber
CMC	carboxymethyl cellulose; critical micelle concentration
CMS	chloromethylstyrene
CR	chloroprene rubber
CRADA	Cooperative Research and Development Agreement

CSPE	chlorosulfonated polyethylene
DBLP	di-basic lead phthalate
DCHP	dicyclohexyl phthalate
DCP	dicumyl peroxide
DEHP	di(2-ethylhexyl) phthalate
DGEBA	digycidyl ether of bis-phenol A
DIBP	diisobutyl phthalate
DM	diene monomer
DMF	*n,n*-dimethylformamide
DOP	dioctyl phthalate
DOTP	di(2-ethylhexyl terephthalate)
DSC	differential scanning calorimeter
DTDP	ditridecyl phthalate
DTMPTA	di(trimethylol propane) tetraacrylate
DVB	divinylbenzene
EA	ethyl acrylate
EB	electron beam
ECTFE	poly(ethylene-co-chlorotrifluoroethylene)
EEA	poly(ethylene-co-ethyl acrylate)
EHEC	ethyl hydroxyethyl cellulose
EHPC	di-2-ethylhexyl peroxy dicarbonate
EN	5-ethylidene-2-norbornene
EPDM	ethylene-propylene-diene
EPM	ethylene-propylene copolymer; ethylene-propylene rubber
ESR	electron spin resonance
ETFE	poly(ethylene-co-tetrafluoroethylene)
EVA	poly(ethylene-co-vinyl acetate)
EVOH	poly(ethylene-co-vinyl alcohol)
FEP	poly(tetrafluoroethylene-co-hexafluoropropylene)
FMQ	SR having fluoro, methyl, and phenyl substituent
FTIR	Fourier transform infrared
FVMQ	SR having fluoro, methyl, and vinyl substituent
G(S)	chain scission G value, number of chain scissions occurred per 100eV of absorbed energy
G(X)	cross-linking G value, number of cross-links formed per 100eV of absorbed energy
GC/MS	gas chromatography/mass spectroscopy
GMA	glycidyl methacrylate
HA	hyaluronic acid
HALS	hindered-amine light stabilizers
HD	1,6-hexanediol dimethacrylate
HDDA	1,6-hexanediol diacrylate
HDEs	homolytic dissociation energies

HDPE	high-density polyethylene
HDXLPE	cross-linked HDPE
HEC	hydroxyethyl cellulose
HEMA	hydroxyethyl methacrylate
HF	hydrogen fluoride
HLB	hydrophilic–lipophilic balance
HMS PP	high-melt-strength polypropylene
HMS	high melt strength
HNBR	hydrogenated acrylonitrile–butadiene rubber
HPMA	*n*-(2-hydroxypropyl) methacrylamide
HPMC	hydroxypropymethyl cellulose
IAEA	International Atomic Energy Agency
IIR	butyl rubber; isobutylene-isoprene rubber
ILSS	interlaminar shear strength
IPAAm	*n*-isopropylacrylamide
IPN	interpenetrating polymer network
IR	*cis*-1,4-polyisoprene; isoprene rubber; polyisoprene; synthetic isoprene rubber
ISO	International Standard Organization
KFM	poly(vinylidene fluoride-co-hexafluoropropylene)
LALLS	low-angle laser light scattering
LCB	long-chain branching
LCP	liquid crystal polymer
LCST	lower critical solution temperature
LDPE	low-density polyethylene
LLDPE	linear low-density PE; low-density polyethylene
LVL	laminated veneer lumber
MA	maleic anhydride
MALLS	multiangle laser light scattering
MC	methyl cellulose; methylcellulose
MCF	microcellular foams
MEHEC	methyl ethyl hydroxyethyl cellulose
MFA	multifunctional acrylate
MFI	melt flow index
MFR	melt flow rate
miPP	metallocene-catalyzed isotactic polypropylene
mLLDPE	metallocene linear low-density PE
MMA	methyl methacrylate
MQ	poly(dimethylsiloxane); polydimethylsiloxane
MW	molecular weight
MWD	molecular weight distribution (dispersity)
MWNT	multiwalled carbon nanotube
n-BA	*n*-butyl acrylate
NBR	acrylonitrile-butadiene rubber

NDDA	1,9-nonanediol diacrylate
NMAM	*n*-methylolacrylamide
NMR	nuclear magnetic resonance
NPG	neopentylglycol dimethacrylate
NR	natural rubber
NTC	negative temperature coefficient
OPEFBF	oil palm empty fruit bunch fiber
OSB	oriented-strand board
PA 6	poly(ε-caprolactam)
PA 66	poly(hexamethylene adipamide)
PA	polyamide
PAAc	poly(acrylic acid)
PAAm	polyacrylamide
PAI	polyamide-imide
PALF	pineapple leaf fiber
PaM	Pont-a-Mousson
PAMS	poly(α-methyl styrene)
PAN	poly(acrylonitrile)
PAR	polyacrylate
PB	polybutylene
PBA	poly(butylene adipate)
PBS	poly(butylene succinate)
PBT	poly(butylene terephthalate)
PC	polycarbonate
PCL	poly(ε-caprolactone)
PCS	polycarbosilane
PCTFE	polychlorotrifluoroethylene
PE	polyethylene
PEA	phenoxy ethyl acrylate
PE-*b*-PEO	poly(ethylene-block-ethylene oxide)
PEEK	poly(ether ether ketone)
PEG	poly(ethylene glycol)
PEI	poly(ether imide)
PEO	poly(ethylene oxide); polyethylene oxide
PES	poly(ether sulfone)
PET	poly(ethylene terephthalate); polyethylene terephthalate
PFA	poly[tetrafluoroethylene-co-perfluoro(propyl vinyl ether)]; polyfunctional acrylate
PFM	polyfunctional monomer
PHB	poly(hydroxy buthylate)
PIB	poly(isobutylene)
PLA	poly(lactic acid), poly(L-lactic acid)
PMA	poly(methyl acrylate)
PMMA	poly(methyl methacrylate)

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