



Maryann C. Wythers
EDITOR

ADVANCES IN

Materials Science Research

VOLUME

55

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Additional color graphics may be available in the e-book version of this book.

Library of Congress Cataloging-in-Publication Data

ISBN: 979-8-88697-213-9

ISSN: 2159-1997

Published by Nova Science Publishers, Inc. † New York

Preface

This book consists of eight chapters on the latest advances in materials science research. Chapter 1 considers the production of nano-layered materials for various functional applications. Chapter 2 presents the results of a research project aimed at increasing the mechanical properties of a rolled sheet of S355 steels, microalloyed with a complex of V-Nb-Al, by means of optimization of its chemical composition and parameters of normalizing heat treatment. Chapter 3 deals with the selection criteria of brake pad material for both dry and wet environmental conditions. The review provides an insight knowledge in the development of dry and wet friction brake pad materials. Chapter 4 explores a strategy for preparing potentially useful iron oxide nanoparticles utilizing ferrocene and its derivatives as source materials through the solid state thermal decomposition technique. Chapter 5 presents a detailed review on the recent research directed towards developing epoxy-based materials for radiation shielding applications. Chapter 6 focuses on the evolution of microcrystalline cellulose (MCC) in co-processed excipient technology. In Chapter 7, the drilling and machining behavior such as speed, feed, drill bit type, drill bit diameter, feed rate and their influence of natural fiber reinforced epoxy composites are discussed. In Chapter 8, the physical properties of alkali borate glasses are reviewed, as well as their relation to the changes of microscopic structure such as structural units, bridging and nonbridging oxygens. The mixed alkali effect of borate glass is also discussed.

Chapter 1 - This chapter considers the production of nano-layered materials for various functional applications, including a core (dispersed silica or alumina) and a titanium oxide shell with nanometer thickness.

The chemical assembling by Molecular Layering (ML) method using $TiCl_4$ and H_2O vapors as low-molecular-weight reagents was performed for synthesis of titanium-oxide nanostructures on the surface of dispersed matrices. The technological features of the ML process, which allow targeted control of the chemical composition and thickness of a nanoscale coating formed on the surface of an inert carrier, will be considered in detail.

It is shown that varying of temperature at different ML stages in the range of 200-700°C, and synthesis cycles number, the chemical and phase composition of the formed titanium-oxide nanolayers can be controlled over a wide range. The formation of a coating with both anatase and rutile structure, as well as the joint presence of both TiO₂ crystalline phases in the composition of the surface titanium-oxide layer is possible. The presence of a chemical bond between the matrix and the synthesized titanium-oxide layer was established using IR spectroscopy. The local structure of titanium-oxide polyhedra and the phase composition of coatings were determined using Electronic Spectroscopy of Diffuse Reflectance (ESDR) and XRD.

The calculation of the thickness of applied coating, which ensures the formation of shell titanium-oxide pigments, was carried out.

The course of polymorphic transformations of the synthesized nanolayer oxide systems in the temperature range 200-1200°C was considered.

Chapter 2 - The chapter presents the results of a research project aimed at increasing the mechanical properties (YTS, UTS, Elongation, KCVL, KCVT) of a rolled sheet of S355 steel (EN 10025), microalloyed with a complex of V-Nb-Al, by means of optimization of its chemical composition and parameters of normalizing heat treatment. The microstructure of steel was characterized using LOM, SEM, and TEM techniques. The optimal normalization parameters for 10-140 mm thick rolled sheets were found as heating temperature – 880-900°C, and specific heating time – 1.5 min/mm. The optimum Nb content in S355 steel was defined as $\square 0.035$ wt.%, providing 2.5 times grain refinement and an increasing yield strength and low-temperature (minus 20-60°C) impact toughness as compared with the as-rolled state. Adding V of about 0.050% was found to be appropriate only for Nb-free steel. In Nb-containing steel, vanadium did not provide additional grain refinement and caused a reduction in impact toughness. Regression models of the influence of chemical elements, sheet thickness, and test temperature on the mechanical properties of normalized S355 steel were developed and verified in manufacturing conditions.

Chapter 3 - The material characterization and its applications of any part are based on domain and its usage. The braking system is one of the basic unit which controls the running system of any mechanism. The selection of brake friction materials needs experimentation rather than trial and error method for clear understanding. The materials considered for the development of brake pad in this study are semi-metallic, composites, steel, ceramics, carbon composition and fiber reinforced. This chapter deals with the selection criteria of the brake pad material for both dry and wet environmental conditions. This

review provides an insight knowledge in the development of dry and wet friction brake pad materials. The fibres, binders, fillers and additives used to fabricate dry friction material are addressed in this chapter. The different types of wet friction materials such as paper, sintered and fabric used for the production of brake pad materials are discussed.

Chapter 4 - Transition metal oxides are of great interest in numerous scientific fields and applications, both as bulk and nanomaterials. Among those, from the application point of view, the most useful and interesting compound is iron oxide. The magnetic, electronic, and chemical properties of iron oxide nanoparticles greatly depend on the route of its synthesis, and for that reason innovative easy methods of creating required sized magnetic oxide nanoparticles are essential for attaining all the desired properties. Thermal decomposition of iron-bearing organic complexes in solid state is a very popular way to produce iron oxide nanostructures of different phases. Such synthetic technique of magnetic oxides has several advantages, such as a relatively low temperature of formation of magnetic oxides, a short reaction time and a possibility of using inexpensive iron-bearing compounds. The process parameters like the working atmosphere, the temperature, the heating regime, etc. play important roles. Further, to understand any correlation among the thermal decomposition reaction, nature and morphology (hence physical characteristics) of the reaction products, the study of kinetics of thermal decomposition reaction is essential.

Organoiron compound ferrocene $[(C_5H_5)_2Fe]$ has become an important organometallic precursor for preparing iron oxide micro or nano-structures by thermal decomposition because it contains two important elements, C and Fe, which are needed for the synthesis of magnetic micro/nanoparticles, thin films, single walled/ferromagnetic-filled carbon nanotube, nanocomposites, etc. The present article explores a strategy for preparing potentially useful iron oxide nanoparticles utilising ferrocene and its derivatives as source materials through the solid state thermal decomposition technique. It highlights the dependence of the decomposition reaction and the decomposed product on the chemical nature of the precursor, co-precursor and reaction atmosphere used. It provides an understanding on the kinetics and mechanistic aspects of the thermal decomposition of these materials in different reaction environment.

Chapter 5 - Rapidly advancing technologies in the nuclear industry have led to the increased use of X-rays and γ -rays in our day-to-day life. They have emerged to be an integral part of several industries including medical diagnostics and imaging, nuclear medicine, reactor research facilities, industrial gauging, agricultural irradiation, geological exploration and security

purposes. However, considering the adverse effects of prolonged exposure to these radiations on human health, this is also a cause of concern for mankind and radiation shielding and protection have become issues of paramount importance. In the search for alternatives to conventional shielding materials such as lead, metals, glass composites, ceramics and concretes, epoxy-based composites have emerged as promising X-ray and γ -ray shields. Material properties like high mechanical and bonding strength, high temperature resistance, low electrical conductivity and thermal expansion coefficients, dielectric constant with minimal shrinking stress and lightweight structure render epoxy composites to be particularly suitable for structural applications. Epoxy composites incorporated with fillers/additives such as inorganic metal oxides, carbon fibers, clay and carbon nanotubes are an emerging class of high-performance materials. The primary focus of this article is to present a detailed review on the recent research directed towards developing epoxy-based materials for radiation shielding applications. Influence of filler loading, filler size and interfacial adhesion on microstructural, thermo-mechanical and radioprotective efficacy of epoxy composites are discussed. The authors present a general overview and propose new possibilities for further research in this direction.

Chapter 6 - MCC is traditionally used as filler and disintegrant in tablet formulations. Over the years, the excipient technology has been evolved and improved to cater to the fast growing needs of the generic pharmaceutical industry. The pharmaceutical industry is looking for affordable and multifunction excipient that will replace the conventional multi-stages and tedious tablet manufacturing process. As a result, material scientists have proposed co-processed excipient as an alternative to replace the conventional single function excipient. Co-processed excipients are a combination of two or more pharmacopeial or non-pharmacopeial excipients that have been intended to physically change their properties in a way that is not possible with simple physical mixing and no significant chemical change. MCC is a suitable candidate that is oftentimes used as a component in co-processed excipients due to its fast disintegration properties, good compressibility and inert nature. Some marketed co-processed excipients that contain MCC are LustreClear, Vitacel VE-650, Cel-O-Cal, Microcelac, Avicel ce-15 and Prosolv.

Chapter 7 - A new generation of reinforcements and integrators for polymer-based products, natural fibre is a form of renewable resource. Natural fibres are increasingly used as reinforcement in composite materials due to their benefits over synthetic fibres. Natural fiber-reinforced composites require specific secondary activities during assembly in order to meet specific

design requirements. In this chapter, drilling behaviour such as speed, feed, drill bit type, drill bit diameter, feed rate and their influence are discussed. Abrasive water jet machining behaviour such as water pressure, traverse rate, standoff distance and abrasive mass flow rate and their influence are also reported. Types of natural fiber and their utilization are presented. Delamination, thrust force, surface roughness and torque during drilling are also reported. Further, influence of drill bit during drilling, chip formation and joining of composites in drilled hole are also reported. From the results, it is identified that, minimum delamination can be achieved by high speed and low feed.

Chapter 8 - Borate glass (B_2O_3) is one of the typical covalent network glasses. The network structure of the borate glass is formed by randomly connected planar BO_3 triangles and most of them form planar boroxol B_3O_6 rings. Alkali metals remarkably modify the network structure of a borate glass, and the variation in the microscopic structure and related physical properties as a function of the alkali content are reviewed. The alkali ions in a borate glass induce the change of the coordination number of a boron atom from three to four and non-bridging oxygens are created as the alkali content increases. The modification by alkali ions has been used to control the various physical properties of glasses. The fundamental physical properties such as density, elastic moduli, thermal and vibrational properties are described in relation with the changes of microscopic structure such as structural units, bridging, and nonbridging oxygens. The mixed alkali effect of a borate glass is also discussed.

Contents

Preface	vii
Chapter 1	Synthesis and Study of Nano-Layered Materials “Disperse Core (SiO₂, Al₂O₃) – Titanium Oxide Shell”	1
	Anatoly A. Malkov, Eugene A. Sosnov and Anatoly A. Malygin	
Chapter 2	Effect of Chemical Composition and Normalization Parameters on Microstructure and Mechanical Properties of Microalloyed Sheet Steel for Structural Purposes	97
	V. E. Stavrovskaya, V. G. Efremenko, V. I. Zurnadzhy, D. S. Zotov, R. I. Sagirov, Yu. G. Chabak, M. N. Brykov and B. V. Efremenko	
Chapter 3	Characteristics of Brake Pad Materials and Their Selection Criteria - A Quantitative Survey	153
	M. Sunil Kumar Hemanth and J. Edwin Raja Dhas	
Chapter 4	Ferrocene – Precursors for the Synthesis of Potentially Useful Iron Oxide Nanoparticles through Solid State Thermal Decomposition	183
	Ashis Bhattacharjee	
Chapter 5	Recent Advances in Lightweight Epoxy-Based Composites for X-Ray and γ-Ray Shielding Applications	227
	Srilakshmi Prabhu, S. G. Bubbly and S. B. Gudennavar	

Chapter 6	Evolution of Microcrystalline Cellulose (MCC) in Co-Processed Excipient Technology	253
	K. B. Liew, M. H. Ee, S. Poonguzhali and Ashok Kumar Janakiraman	
Chapter 7	Machining Behaviour of Natural Fiber Reinforced Epoxy Composites	277
	R. Karthick and V. Anbumalar	
Chapter 8	Physical Properties of Alkali Borate Glasses	295
	S. Kojima	
	Contents of Earlier Volumes	315
Index	321

Chapter 6

Evolution of Microcrystalline Cellulose (MCC) in Co-Processed Excipient Technology

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Abstract

MCC is traditionally used as filler and disintegrant in tablet formulations. Over the years, the excipient technology has been evolved and improved to cater to the fast growing needs of the generic pharmaceutical industry. The pharmaceutical industry is looking for affordable and multifunction excipient that will replace the conventional multi-stages and tedious tablet manufacturing process. As a result, material scientists have proposed co-processed excipient as an alternative to replace the conventional single function excipient. Co-processed excipients are a combination of two or more pharmacopeial or non-pharmacopeial excipients that have been intended to physically change their properties in a way that is not possible with simple physical mixing and no significant chemical change. MCC is a suitable candidate that is oftentimes used as a component in co-processed excipients due to its fast disintegration properties, good compressibility and inert nature. Some

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marketed co-processed excipients that contain MCC are LustreClear, Vitacel VE-650, Cel-O-Cal, Microcelac, Avicel ce-15 and Prosolv.

Keywords: microcrystalline cellulose, co-processed excipient, formulation, direct compression

Introduction

MCC is natural polymers obtained from refined wood pulp. MCC is partially depolymerized cellulose synthesized from alpha precursor (Hindi 2017). It is abundantly found in the whole plant or certain part of the plant, including roselle, pomelo peel, olive fibre, cotton linters, oil palm biomass residue, tea waste and many more. MCC is extracted through various methods such as hydrolysis treatment, acid hydrolysis treatment, alkaline hydrolysis, bleaching, reactive extrusion and ultrasonication.

MCC is prepared by treating alpha cellulose with the mineral acids, type Ib. One of the main sources of MCC is conifer wood pulp. Cotton is another main source of MCC. The quality of pulp differs within the conifer wood, and it varies based on the chemical composition that includes the proportion of cellulose, hemicellulose, and lignin. Based on the structural organisation, it can be crystalline or amorphous. Amorphous regions are prone to acid hydrolysis and result in more crystalline fragments. Wooden and non-wooden sources with their grades are tabulated in Table 1.

Table 1. Sources of microcrystalline cellulose and its grades

	Wooden source	Non wooden source
Source and description	Soft woods and hard woods. In these, cellulose is packed together in layers and held together by strong hydrogen bonds from the lignin, which is a cross linking polymer.	Lignocellulosic materials developed from cotton linters, cotton wool, soybean hulls, rice husk, corn cob, coconut shell, biomass of palm oil residue, sugarcane, etc.
Grades	Pharmaceutical grades are obtained.	Pharmaceutical and general grades are obtained.

The characteristics of MCC lead to its widespread usage in various fields that mainly includes pharmaceuticals, cosmetics, and food industries. MCC is non-toxic, economically valuable, biodegradable, has high surface area, provides high mechanical properties and biocompatibility. Depending on the

solubility, different types of MCC are available. They are alpha, beta and gamma cellulose.

Table 2. General functions of MCC in pharmaceuticals

Function	Description	References
Fillers in Direct compression	MCC 102 has a median particle size of 100µm with acceptable flow properties for high speed tableting. Flowability can be improved with the coarser grades, as in MCC 200, particle size 200µm. The larger the particle grades, the better the flow, while moisture grades are lower.	Shlicourt (2002) Shi et al. (2011)
Fillers in wet granulation	MCC is water insoluble, having swelling tendencies and water imbibing action. This action promotes rapid wetting of the mixture. Reduces the sensitivity of the wet mass to over wetting and increases the speed of drying. <ul style="list-style-type: none"> • More uniform granules can be obtained. • Better drug content uniformity. • Tablet hardness can be increased at the same compression force with less friability. 	Gamble et al. (2011)
Binders	It's a self-disintegrating binder that has low lubricant requirements due to its dry binding properties. Combinations of MCC and super disintegrants is an alternative to the traditional drug layering on pellets. Extrusion-spheronization aid excipient. MCC acts as an excellent Extrusion-spheronization excipient.	Chacrunisaa et al. (2019)
Sustained release applications	MCC is used to formulate the multiparticulate and matrix tablet dosage forms for sustained release systems. Hydrophilic polymers in matrix tablet formulation are included as a barrier to drug release.	Chacrunisaa et al. (2019)
Thickening and stabilizing agents	These polymers can increase the viscosity of non-aqueous pharmaceutical solutions such as organic-based coating solutions. The viscosity enhancing of drug solutions poses many advantages, such as improving consuming controllability and increasing residence time of drugs in topical and mucosal solutions, which lead to improved bioavailability of topical, nasal or ocular preparations.	Grove et al. (1990)
Disintegrating agent	It will break up the dosage forms into smaller fragments. Increase the available surface area and promote a more rapid release of drug substances from dosage forms	Shokri (2013)
Bioadhesive	Synthetic polymers such as acrylic derivatives, carbopol and polycarbophil, natural polymers such as carageenan, pectin, acacia and alginates and semi-synthetic polymers like chitosan and cellulose derivatives are used in bioadhesive formulations.	Deshpande et al. (2009); Grabovac et al. (2005)

MCC can be used as a primary excipient as well as a secondary excipient. These are considered as dry binders as they can improve the compatibility and tableability of the compression mix. (Reier, 2013). It is used in direct compression and wet granulation processes in the formulation of both oral

VOLUME

55

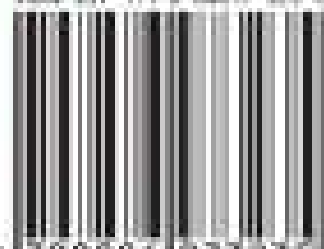
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