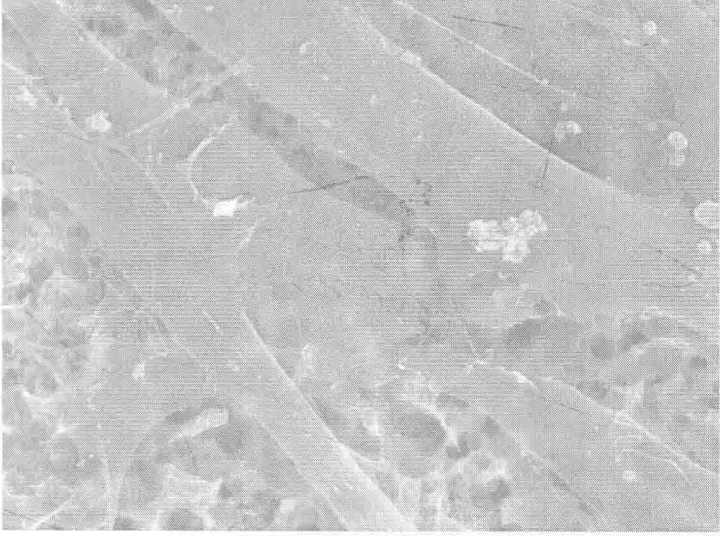
Edited by
Mieczyslaw Jurczyk

Bionanomaterials for Dental Applications



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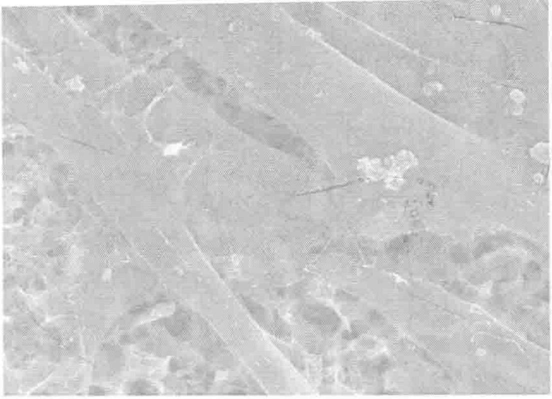

Bionanomaterials for Dental Applications

Preface

Nanotechnology involves the precise manipulation and control of atoms, the building elements of all matter, to create new materials. It is widely accepted that this technology is developing into a major driver for commercial success in the 21st century. Over the last decade, the use of nanostructured metallic and ceramic materials has already changed the approach to materials design in many applications, by seeking structural control at the atomic level and by the tailoring of the mechanical engineering, physico-chemical, and biological properties. Today, it is possible to prepare metal and ceramic nanocrystals with nearly monodispersive size distribution. Nanomaterials demonstrate novel properties compared with conventional (microcrystalline) materials owing to their nanoscale features.

Recently, the mechanical alloying method and the powder metallurgy process for the fabrication of metal/alloy–ceramic nanocomposites with a unique microstructure have been developed. The processes permit the control of microstructural properties such as the size of pore openings, surface properties, and the nature of the base metal/alloy. The availability of large amounts of specifically tailored nanostructured metal/alloy-based powders is crucial for the successful development of new dental implants.

One of the potential applications of nanostructured materials is dentistry. Although Ti is widely used for clinical purposes, some unresolved issues still remain. The clinical failure rate for implant materials occurs in the range from a few to over 10%.

The lack of sufficient bonding of synthetic implants to surrounding body tissues has led to the investigations of novel material formulations. Nanomaterials can be used to synthesize implants with surface roughness similar to that of natural tissues. The mechanical properties are improved, and what is more, the book highlights the enhanced cytocompatibility of the nanomaterials, leading to increased tissue regeneration.

The present research aims to fabricate porous scaffolds to promote bone or tissue ingrowth into pores and provide biological anchorage. Several factors have shown their influence on bone ingrowth into porous implants, such as porous structure (pore size, pore shape, porosity, and interconnecting pore size) of the implant, duration of implantation, biocompatibility, implant stiffness, and micromotion between the implant and the adjacent bone. The architecture of a porous implant has been suggested to have a great effect on implant integration by newly grown bone.

This book is our contribution to this innovative area of bionanomaterials and bionanocomposites for dental applications. Wherever possible, we used our own results to illustrate the discussed subject. The content of this book is classified into 13 chapters. The first chapter emphasizes the motivation for the transformation to the bionanomaterials and synthesis of nanomaterials, aiming at describing the principles and approaches of the synthesis techniques. We provide a comprehensive history of the development of biomaterials, including the existing fabrication methods, with special emphasis on ball milling in high-energy mills. The second chapter focuses on the stomatognathic system. In Chapters 3 and 4, we review the properties of selected biomaterials and the application of nanotechnology in dental materials. Chapter 5 presents a thorough review of the corrosion of metallic biomaterials and implants. The book also describes Ni-free austenitic stainless steel–hydroxyapatite nanocomposites (Chapter 6), Ti-based ceramic nanocomposites (Chapter 7), and shape memory Ni–Ti materials (Chapter 8). Chapters 9 and 10 provide information on the surface treatment of Ti-based nanocrystalline biomaterials and carbon materials. The present state of knowledge related to nanomaterials in preventive dentistry and osteoblast behavior on nanostructured metal implants are presented in Chapters 11 and 12. Chapter 13, the last chapter, focuses on the application of bulk nanostructured materials in dentistry. The objective is to show their unique properties.

Our goal is to provide comprehensive and complete knowledge about bionanomaterials for dental applications to graduate students and researchers, whose background can be in chemistry, physics, chemical engineering, materials science, biomedical science, or even dental science.

I express my appreciation to all of the authors for their contributions.

Mieczyslaw Jurczyk

Contents

Chapter 1

Introduction

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1.1 Motivation

In 1959 Richard Feynman, a Physics Nobel laureate, presented his famous idea of nanostructure materials production [17]. He stated: "The principles of physics, as far as I can see, do not speak against the possibility of maneuvering things atom by atom." Feynman proceeded to describe building with atomic precision, and outlined a pathway involving a series of increasingly smaller machines. Today, it is possible to prepare metal, ceramic, and alloy nanocrystals with nearly monodispersive size distribution. Nanostructures represent key building blocks for nanoscale science and technology.

Nanotechnology is a technology that owes its name to the prefix *nano*, a Greek word for *dwarf*, as applied to objects that exhibit billionth (10^{-9}) meter dimensions. Recently, nanotechnology has led to a remarkable convergence of disparate fields including biology,

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applied physics, chemistry, materials science, and computational modeling [3]. Broadly speaking, nanotechnology is the development and use of techniques to construct structures in the physical size range of 1–100 nanometers (nm), as well as the incorporation of these structures into applications. Now, nanotechnology is entering many industry sectors including energy, electronics, aerospace as well as medicine.

Nanoscience and nanotechnologies are not new. Size-dependent properties have been exploited for centuries. For example, Au and Ag nanoparticles have been used as colored pigments in stained glass and ceramics since the 10^{th} century AD. Many chemicals and chemical processes have nanoscale features and, for example, chemists have been making polymers (large molecules made up of nanoscalar subunits) for many decades. But now, due to imaging techniques like the scanning tunneling microscope and the atomic force microscope, the understanding of the nanoworld has improved considerably [14, 16].

During the past few years, interest in the study of bionanostructure materials has been increasing at an accelerating rate, stimulated by recent advances in materials synthesis and characterization techniques and the realization that these materials exhibit many interesting and unexpected properties with a number of potential technological applications [6, 18, 40, 52]. Nanotechnology provides the tools and technology platforms for the investigation and transformation of biological systems, and biology offers inspiration models and bio-assembled components to nanotechnology [21]. For example, the London Centre for Nanotechnology has a wide range of bionanotechnology and health care research programs: bionanoparticles, bionanosensors, biocompatible nanomaterials, advanced medical imaging, technologies for diagnosis, self-assembled biostructures, degenerative disease studies, molecular simulation, lab on a chip and screening, drug screening technologies, and molecular simulation.

Application of new materials such as biomaterials and implants increases steadily. However not all replacement systems have provided trouble-free service. In dental implants the rate of success is 96–98%, which, by millions of implants, gives a significant number of patients in trouble [2]. Therefore, in a failure-free replacement system, no particulate or corrosion debris would be generated and no loosing of the implant components should occur. The source of debris particles is the wear process and grit blast, which includes Al_2O_3, ZrO_2, or SiO_2 particles on the surfaces of especially treated implants.

An appropriate surface modification would prevent their transfer into nearby tissues. The absence of debris particle generation is crucial for the prevention of implant malfunction. The determination of the mechanisms of debris generation and appreciate modification of implant surface bulk structure and properties is one of the main aims of current research projects.

The main purpose of current research is to prevent the failures caused by infection by changing the biomaterial's properties and making them highly friendly for surrounding tissues.

Ti and Ti-based alloys are preferred materials in the production of implants in both medical and dental applications. These biomaterials have relatively poor tribological properties because of their low hardness. One of the methods that allow the change of biological properties of Ti alloys is the modification of its chemical composition. The other way is to produce a composite that will exhibit the favorable mechanical properties of titanium and excellent biocompatibility and bioactivity of ceramic. The most commonly used ceramics employed in medicine are hydroxyapatite (HA), silica, and bioglass. HA shows good biocompatibility because of its similar chemical and crystallographic structure to the apatite of living bone. The ceramic coating on the titanium improves the surface bioactivity but often flakes off as a result of poor ceramic/metal interface bonding, which may cause the surgery to fail. For this reason, composite materials containing titanium and ceramic as a reinforced phase are expected to have broad practical applications.

Since 1996 a research program was initiated at the Institute of Materials Science and Engineering, Poznan University of Technology, in which fine grained, intermetallic compounds were produced by mechanical alloying, high-energy ball milling, hydrogenation-disproportionation-desorption-recombination (HDDR), or mechano-chemical processing (MCP) [22–28, 34–36]. The mechanical synthesis of nanopowders and their subsequent consolidation is an example how this idea can be realized in metals by a so-called bottom-up approach. On the other hand, other methods have been developed, which are based on the concept of the production of nanomaterials from conventional bulk materials via the top-down approach. The investigations by severe plastic deformation (e.g., cyclic extrusion compression method (CEC) or equal channel angular extrusion (ECAE)) [39, 46, 50, 51], show that such a transformation is indeed possible. Currently, at Poznan University of Technology, we facilitate the multidisciplinary interaction of physicists, chemists, materials engineers, biologists, and dentists collaborating on nanoscience, with the goal of integrating nanoscale materials with biological systems. The aim of our research is to develop a new generation of titanium (Ni-free stainless steel)-ceramic bionanocomposites by producing the porous structures with a strictly specified chemical and phase compositions, porosity and surface morphology and, as such, will adhere well to the substrate, show high hardness, high resistance to biological corrosion and good biocompatibility with human tissues.

Nanomaterials can be metals, ceramics, polymers, and composite materials that demonstrate novel properties compared with conventional (microcrystalline) materials due to their nanoscale features. Moreover, researchers have exhibited an increased interest in exploring numerous biomedical applications of nanomaterials and nanocomposites [3, 6, 40]. Till now, it has been shown that implants made from metallic, carbon, or oxide bionanomaterials considerably improved the prosthesis strength and their biocompatibility. These nanocrystalline structures can be produced by non-equilibrium processing techniques such as mechanical alloying [4, 9, 47].

The current projects aim to fabricate Ti-based porous scaffolds to promote bone or tissue ingrowth into pores and provide biological anchorage. Generally, porous metallic scaffolds are fabricated using a variety of processes to provide a high degree of interconnected porosity to allow bone ingrowth. Fabrication technologies include chemical vapor infiltration to deposit tantalum onto vitreous carbon foams, solid freeform fabrication, self-propagating high-temperature synthesis, and powder metallurgy [13, 20, 29, 34, 45, 48]. While these porous metals have been successful at encouraging bone ingrowth both *in vivo* and in clinical trials, the range of materials and microstructures available is still rather limited. It is important to use appropriate surface modification to increase the anti-corrosive and biocompatible properties of Ti implants for long-term clinical applications.

Mechanical alloying, high-energy ball milling, reactive milling, chemical vapor transport, solid–liquid–vapor growth, solvothermal synthesis, solid–gas high-temperature reactions, microwave chemistry, arc furnace techniques, aerosol spray techniques, liquid metals chemistry, and powder metallurgy process for the fabrication of titanium (Ni-free stainless steel)–ceramic nanocomposites with a unique microstructure were developed. Those processes permit the control of microstructural properties such as the size of pore openings, surfaces properties, and the nature of the base metal/alloy. A new type of bulk three-dimensional porous Ti (Ni-free stainless steel)-based nanocomposite biomaterials with desired size of porous and three-dimensional capillary-porous coatings on these nanobiocomposites was developed. Materials with nanoscale grains would offer new structural and functional properties for innovative products in medical/dental applications.

Various methodologies are being used in an effort to improve the interfacial properties between the biological tissues and the existing implants, e.g., Ti and Ti-based alloy. The electrochemical technique, a simpler and faster method, can be used as a potential alternative for producing porous Ti-based metals for medical implants. Good corrosion resistance of the titanium is provided by the passive titanium oxide film on the surface. This layer is important for the good biocompatibility. The native oxide has thickness of a few nanometers. In the case of anodic oxidation, the oxide thickness can be multiplied up to the micrometer range. The structure and thickness of the grown oxide depend on the electrochemical etching conditions, for example: current density, voltage, electrolyte composition. In the electrochemical etching of titanium, electrolytes containing H_3PO_4, CH_3COOH, and H_2SO_4 are used. In Ti anodization, the dissolution is enhanced by HF- or NH_4F-containing electrolytes, which results in pore or nanotube formation. The current density in this case is much higher than in the electrolyte without HF or NH_4F [22]. Fluoride ions form soluble $[TiF_6]^{2-}$ complexes resulting in the dissolution of the titanium oxides. In this way, the dissolution process limits the thickness of the porous layer.

Porous implants layer has lower density than respective bulk, and good mechanical strength is provided by bulk substrate. Hence, the latter is attractive with respect to bulk titanium alloys. The porous layer on the Ti substrate is necessary for osseointegration with bones, which is not normally provided by the native oxide.

On the other hand, Ti and its alloys possess favorable properties, such as relatively low modulus, low density, and high strength. Apart from that, these alloys are generally regarded to have good biocompatibility and high corrosion resistance but cannot directly bond to the bone. In addition, metal implants may loosen and even separate from surrounding tissues during implantation. Titanium- and titanium-based alloys have relatively poor tribological properties because of their low hardness. One of the methods that allow the change of biological properties of Ti alloys is to produce a nanocomposite that will exhibit the favorable mechanical properties of titanium and excellent biocompatibility and bioactivity of ceramic. The most commonly used ceramics in medicine are hydroxyapatite, bioglass, and Al_2O_3 [7, 34].

Current research on the synthesis of nanoscale metallic and composite biomaterials, shows that Ti/(Ni-free stainless steel)–HA nanocomposites posses better mechanical and corrosion properties than microcrystalline titanium/Ni-free stainless steel [49]. In the case of Ti–HA nanocomposities, the Vickers hardness also strongly increases for Ti-20 vol% HA nanocomposites (1030 HV0.2) and is four times higher than that of pure microcrystalline Ti metal (250 HV0.2). The corrosion test results indicated that the microcrystalline titanium possesses lower corrosion resistance and thus higher corrosion current density in Ringer's solutions. The result indicated that there was no significant difference in corrosion resistance among Ti-3 vol% HA ($I_C = 9.06 \times 10^{-8}$ A/cm^2, $E_C = -0.34$ V) and Ti-20 vol% HA ($I_C = 8.5 \times 10^{-8}$, A/cm^2, $E_C = -0.55$ V) although there was a significant difference in porosity. For this reason, they are promising biomaterial for use as heavy load-bearing tissue replacement implants.

The availability of large amounts of specifically tailored nanostructure Ti-based powders is crucial for the successful development of new dental implants. The processing of these nanomaterials and their upscaling to enable industrial use has many challenges. Those new approaches are the gateway for traditional industry to nanotechnology and knowledge-based materials, with positive effects on health issues [1, 29, 45, 53].

1.2 Nanomaterials

One of the first scientific reports is the colloidal gold particles synthesized by Michael Faraday as early as 1857 [18]. By the early 1940s, precipitated and fumed silica nanoparticles were being manufactured and sold in the United States and Germany as substitutes for ultrafine carbon black for rubber reinforcements.

In the 1960s and 1970s, metallic nanopowders for magnetic recording tapes were developed. The first nano-size metallic materials were produced in 1960 by the application of rapid quenching process with the cooling rate of 10^6 K s^{-1} by Pol Duvez and coworkers [30]. Using a quench technique capable of cooling metal/alloy melts to ambient temperatures with such extraordinary cooling rates, the process of nucleation and growth was kinetically bypassed to yield a configuration of frozen liquid or amorphous metal. The nanoalloys with nanometric grains were processed by low-temperature annealing of amorphous alloy [12]. Additionally, in 1972 a new rapid quenching process of melt spinning was used to spin the first nonocrystalline ferrous and ferromagnetic metal ribbons ($Fe_{80}B_{20}$) [10]. The outstanding physical and chemical properties of these materials were the direct consequence of the lack of structural crystalline long-range order and the presence of short-range order. It is important to mention that many, if not all of these amorphous alloys, i.e., metallic glasses, when carefully annealed at low temperatures change to nanostructure alloy phases. Those were the first nanomaterials. They were produced in the form of thin ribbons via rapid solidification processing of melt alloys. They allowed controlled exploration of physical, chemical, mechanical, and other properties as arising from nanostructures. At this time, scientists and engineers became refocused from well-ordered crystalline materials to disordered and nanocrystalline phases. Gleiter observed that nanometer-size crystalline materials being polycrystals with very small crystallite sizes of about 2–10 nm in diameter are composed of randomly oriented high-angle grain boundaries [18].

The first such nanocrystalline phases came from Gleiter research group around 1984 by evaporation of the material in a high-purity inert gas atmosphere followed by condensation and compaction in ultrahigh vacuum [5]. The percentage of metal atoms on the surface of grain increases from a few percent in a 100 nm particle to about 90% in a 1 nm crystallite [19, 43]. As above, these materials should be attractive for the development of engineering materials with an outstanding combination of properties or novel properties. In the meantime, among materials that became studied were nanophases produced by mechanical alloying [4, 7, 47].

Nanomaterials continue to attract a great deal of attention because of their potential impact on an incredibly wide range of