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Electrophoretic deposition: From traditional ceramics to nanotechnology

Ilaria Corni, Mary P. Ryan, Aldo R. Boccaccini *

Department of Materials, Imperial College London, Prince Consort Road, London SW7 2BP, UK

Abstract

Electrophoretic deposition (EPD) is attracting increasing interest as a materials processing technique for a wide range of technical applications. This technique enables the production of unique microstructures and nanostructures as well as novel and complex material combinations in a variety of macroscopic shapes, dimensions and arrangements starting from micron-sized or nanosized particles. This review presents a comprehensive summary of relevant recent work on EPD describing the application of the technique in the processing of several traditional and advanced materials (functional and structural ceramic coatings, composite and porous materials, laminated ceramics, functionally graded materials, thin films and nanostructured materials), with the intention to highlight how EPD evolved from being a technique restricted only to traditional ceramics to become an important tool in advanced materials processing and nanotechnology. Moreover the fundamental EPD mechanisms and novel theories proposed to clarify the processes involved are explained.

Keywords: Electrophoretic deposition; Films; Composites; Suspension; Fuel cells

1. Introduction

Electrophoretic deposition (EPD) is an electrochemical method attracting increasing interest as a material processing technique.[1–3] EPD is usually carried out in a two electrode cell, as schematically shown in Fig. 1. The mechanism of electrophoretic deposition involves two steps. In the first step an electric field is applied between two electrodes and charged particles suspended in a suitable liquid move toward the oppositely charged electrode (electrophoresis). In the second step the particles accumulate at the deposition electrode and create a relatively compact and homogeneous film (deposition). In order to effectively apply this technique to process materials, it is essential to produce a stable suspension containing charged particles free to move when an electric field is applied. Therefore EPD can be applied to any solid that is available as a fine powder (e.g. $<\sim 30\ \mu m$ particle size) or as a colloidal suspension, including metals, polymers, ceramics and glasses. After the deposition, a heat-treatment step is normally needed to further densify the deposit and to eliminate porosity.[1–5]

EPD was discovered in 1808 by the Russian scientist Ruess and it was first used in a practical application in 1933 to deposit thoria particles on a platinum cathode as an emitter for electron tube applications.[1] In the following years, e.g. until the early 1990s, EPD was mainly used for the processing of traditional ceramics, including enamels and porcelain, and very limited work was carried on the EPD of engineering ceramics.[6] However, in the last 15 years the interest in electrophoretic deposition as a technique to produce advanced materials has widely increased, both in academia and in the industrial sector, and since then a wide range of new applications of EPD for processing a variety of bulk materials and coatings has been reported.[1–6] EPD has demonstrated the possibility to realize unique microstructures and nanostructures as well as novel and complex materials combinations in a variety of macroscopic shapes, dimensions and arrangements. Fig. 2 shows the extraordinary increase of the number of published scientific papers, identified searching the tool Web of Science® by the keyword “electrophoretic deposition”, from only a few papers per year in the 1970s to just under two hundred papers published in 2006.

Recently, EPD has been employed for the processing of functional and composite ceramics, layered and functionally graded materials, thin films, high performance ceramic and composite coatings and biomaterials and also for the deposition of nanoparticles and carbon nanotubes to produce advanced nanostructured

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* Corresponding author. Tel.: +44 2075946731; fax: +44 2075946757.
E-mail address: a.boccaccini@ic.ac.uk (A.R. Boccaccini).

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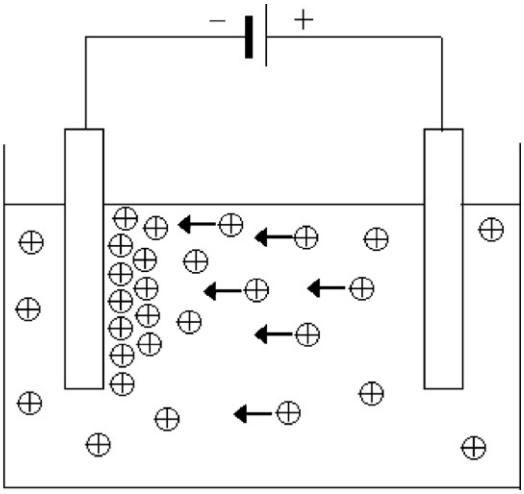

Fig. 1. Two electrodes cell for electrophoretic deposition showing positively charged particles in suspension migrating towards the negative electrode.

materials.[7–9] The increasing significance of this electrochemical technique in materials processing follows from its high versatility for application with different materials and combinations of materials, its cost-effectiveness, simplicity, the requirement of only basic equipment and the ability to be scaled-up to large product volumes and sizes.[1–5] Moreover, compared with other processing methods based on the packing of particles, EPD is able to produce uniform deposits with high microstructural homogeneity, to provide adequate control of deposit thickness and to deposit coatings on a wide range of shapes and 3D complex and porous structures.[1–3,5,7,8]

The success of EPD as processing method for advanced materials and the increasing opportunities being explored for its application in a wide range of materials have been confirmed by the establishment of an international conference series dedicated exclusively to EPD, the proceedings of the first two conferences (held in 2002 and 2005) have been published.[10,11]

Despite the numerous improvements of the EPD technique and the large range of applications achieved, there is need for further theoretical and modelling work to gain a full and quantitative understanding of the mechanisms of EPD. In fact many experimental studies are presently carried out using unsatisfactory and time-consuming trial-and-error approaches, due to the lack of available relationships linking the parameters of the EPD process to the final deposit properties.[10,11]

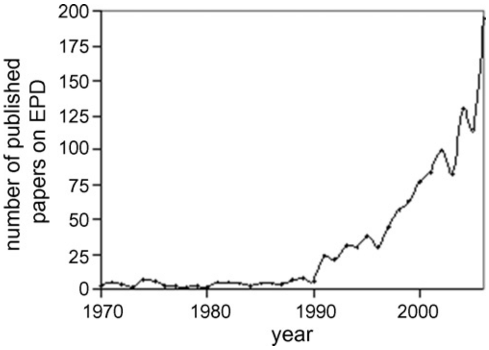

Fig. 2. Increasing number of publications featuring as the keyword “electrophoretic deposition” in the open literature (Web-of-Science® research-Jul-2007), from 1960 until 2006.

The intention of this review is to present a comprehensive summary of relevant previous work on EPD describing the application of the technique in the processing of a range of traditional and advanced materials, with the intention to highlight how EPD evolved from being a technique restricted only to traditional ceramics to become an important tool in advanced materials processing, including nanomaterials. The review is divided into two main parts. One is dedicated to briefly revise the mechanisms proposed to explain the phenomena involved in EPD (Section 2) and the other section presents an overview of EPD applications, dividing them into traditional ceramics, advanced materials and nanotechnology (Section 3). Due to the availability of previous comprehensive review articles covering different aspects of the theory and applications of EPD,[1–8] the focus of the present article is to highlight the most recent published research in this rapidly expanding field.

2. Mechanisms of EPD

2.1. Traditional approaches

The fundamental mechanisms of EPD have been largely described in the literature mainly in the framework of the Derjaguin–Landau–Verwey–Overbeek (DLVO) theory and in relation to the distortion of the particle double layer under the application of a DC electric field, as discussed by Sarkar and Nicholson in their key reference in EPD.[1] However, numerous other theories (flocculation by particle accumulation, particle charge neutralization, electrochemical particle coagulation, electrical double layer (EDL) distortion and thinning mechanism) have been proposed to explain the particle interactions and the kinetics of deposition[1–3,5,12]. Additional theoretical and modelling studies are being carried out in order to clarify the mechanisms of deposition and the role of electrochemical parameters on the complex interactions between solvent, particles and electric field.

2.1.1. Flocculation by particle accumulation

Hamaker and Verwey[13,14] observed similarities between formation of deposits by electrophoresis and gravitation. In fact, in both processes, the pressure exerted by the arriving particles enables the particles close to the deposit to prevail over the inter-particle repulsion. Therefore the primary function of the applied electric field in EPD is to move the particles towards the electrode to accumulate. This mechanism can also explain the deposition of coatings onto membranes that are not serving as electrodes.

2.1.2. Particle charge neutralization mechanism

Grillon et al.[15] suggested that the charged particles are neutralized when they touch the electrode. This mechanism explains the deposition of single particles and monolayers and the deposition of powders charged by the addition of salts to the suspension (e.g. the experiments described by Brown and Salt[16]). However this mechanism cannot clarify depositions carried out for longer times, or for processes in which the particle-electrode contact is not permitted, for example when the deposition occurs on a semi-permeable membrane placed *between* the electrodes.

2.1.3. Electrochemical particle coagulation mechanism

This mechanism implies the reduction of the repulsive forces between the particles in suspension. Koelmans[17] calculated the rise of the ionic strength close to the electrode when a difference of potential was applied. This behaviour was due to an increase of the electrolyte concentration around the particles. He discovered that the value of ionic strength was similar to that required to flocculate a suspension. Therefore, Koelmans[17] proposed a mechanism based on the fact that an increase of the electrolyte concentration produces a decrease of the repulsion between the particles close to the electrode (lower ζ-potential) and consequently the particles coagulate. Considering that a finite time is needed for the increase of the electrolyte concentration next to the electrode, it can be concluded that a certain time has to pass in order to have deposition. This time is inversely proportional to the square of the applied voltage ($t \propto 1/E^2$), i.e. the higher the applied potential the shorter the time required for deposition. This mechanism is plausible when the electrode reactions generate OH^- ions, e.g., suspensions containing water, but it is invalid when there is no increase of electrolyte concentration near the electrode.

2.1.4. Electrical double layer (EDL) distortion and thinning mechanism

Sarkar and Nicholson[1] proposed a model mainly based on the distortion of the particle double layer to explain the invalidation of the electrochemical coagulation mechanism when there is no increase of electrolyte concentration near the electrode. They noted that when a positive particle and its shell are moving towards the cathode, the double layer is distorted (thinner ahead and wider behind), as shown in Fig. 3, due to fluid dynamics and to the effect of the applied electric field. As a result the counter ions (negative) in the extended tail experience a smaller coulombic attraction to the positively charged particle and can more easily react with other cations moving towards the cathode. This process reduces the thickness of the double layer and therefore, when another particle with a thin double layer is approaching, the two particles come close enough to interact through London Van der Waal attractive forces and coagulate. This mechanism is plausible considering a high concentration of particles close to the electrode (or high collision frequency). This mechanism works also for incoming particles with thin double layer heads, coagulating with particles already in the deposit.

Subsequently Nicholson et al.[12] showed that the model previously proposed by Sarkar and Nicholson[1] was not complete and proposed a new theory based on a decrease of the concentration of H^+ at the cathode due to particle discharge or other chemical reactions. Therefore the local pH increases towards the isoelectric point (iep), ζ-potential decreases and the particles coagulate. This mechanism is general for all suspensions containing hydrogen ions.

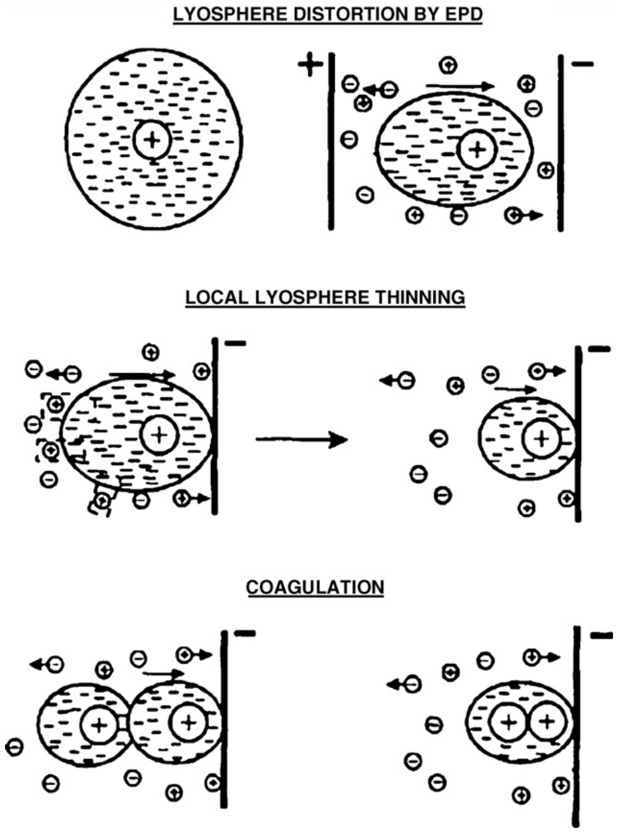

Fig. 3. Schematic representation of the deposition mechanism due to electrical double layer distortion and thinning.[1] (Reproduced with permission of Blackwell Publishing.)

2.2. Novel theories and models

Studies of electrodynamic particle aggregation during EPD have been carried out under steady[18] and alternating electric fields.[19] These models produced equations for the time evolution of the probability of separation between deposited particles in different conditions. These equations are able to explain the experimentally observed clustering of colloidal particles deposited near an electrode in a DC electric field by considering convection by electro-osmotic flow about the particles.[20] Numerical simulations have been also employed to a limited extent to model the accumulation of charged particles on an electrode during EPD.[21,22] These studies are of fundamental and practical interest to describe the local variations of particle interaction during deposition, which can be used to optimize the EPD technique.

Regarding the growth of colloidal films during EPD, Sarkar et al.[23] provided another fundamental study observing the deposition of silica particles on silicon wafers as a function of deposition time. They compared the nucleation and growth of the silica particle layer with that of atomic film growth via molecular-beam epitaxy and noticed a prominent similarity between the two processes. From this observation a new direction for further research could follow in order to optimize the microstructure of EPD films. Theoretical work was also carried out by Van der Biest et al.[24–27] who produced a model to predict the yield of the electrophoretic deposition process taking into account the changes of the electric field over the suspension due to the potential drop over the growing deposit. This model was validated for Al_2O_3 suspensions in ethanol with different concentrations and with addition of HNO_3.

More recently Van Tassel and Randall[28] electrophoretically deposited alumina powder from an acidic suspension obtaining a very uniform, dense alumina layer and observed an anomalous voltage rise across the deposited particulate layer. They showed that these two effects can be explained by the formation of an ion depleted conduction layer in the solvent at the deposition electrode, which presents an extremely high voltage gradient. Therefore the electrophoretic force on the particles in this layer is considerably higher than the force on particles in the rest of the system and this high voltage gradient layer also produces a large self-levelling effect for deposition thickness. Finally, Ristenpart et al.[29,30] have recently studied, both theoretically and experimentally, the flow around a charged spherical colloid next to an electrode in order to understand the nature of long-range particle–particle attraction near the electrodes. From their studies it was clear that the direction of flow of a particle depends on the sign of the dipole coefficient and that the flow consists of two components: the electro-osmotic flow (EOF) and the electrohydrodynamic (EHD) flow. The electro-osmotic flow is proportional to the current density and the particle ζ-potential, while the electrodynamic flow derives from the product of the current density and the applied potential. Comparing these two components, Ristenpart et al.[29,30] found that the attractive EHD flow predominated far from the particle, whereas the attractive EOF predominated over the repulsive EHD flow close to the particle. Moreover they also observed that under certain conditions, the two flows are both directed toward the particle producing aggregation.

The novel theoretical and modelling approaches summarised in this section represent examples of the few efforts available in the literature to investigate basic phenomena occurring during EPD. We highlight here the necessity for further theoretical and modelling work in the field of EPD and the need for establishing reliable correlations between model variables and the experimental processing EPD conditions.

3. Applications of EPD

3.1. Traditional ceramics

Electrophoretic deposition initially found commercial interest and industrial applications for the deposition of uniform coatings made of clay based material, vitreous enamel or alumina, on electrically conductive surfaces from aqueous suspensions.[6] The use of EPD for the production of clay based bodies, e.g. sanitary ware, tiles, table ware, etc., on an industrial scale has been extensively investigated because of the order of magnitude improvement in formation rates achieved compared to slip casting.[4,6] EPD has also been employed to produce vitreous (or porcelain) enamel coatings on metals. After deposition of a layer of glass particles, inorganic coatings were obtained by fusing the powder deposited on the metal surface at temperatures above 425 °C. It was found that EPD coatings presented superior smoothness and uniformity compared to those obtained by conventional dipping or spraying processes.[6] These coatings have found several applications in the industrial production of domestic whiteware in the early 1970s and previous reviews have summarised that earlier work.[1,6]

3.2. Advanced materials

In this section the EPD applications of conventional powders, e.g. μm-sized or submicrometric ($d > 100$ nm), are reviewed, whereas the applications of nanopowders in EPD are considered in Section 3.3.

3.2.1. Coatings and films

First reports on the use of EPD to prepare advanced ceramic coatings were published in the late 1980s. For example hydrated alumina prepared by the sol–gel method was deposited by EPD on aluminium alloy substrates and it was demonstrated that these coatings were thicker, denser and more adherent than those produced by conventional dip-coating techniques.[31] In the last two decades EPD has been increasingly employed to produce advanced ceramic coatings on solid substrates in order to enhance the substrate properties. For example EPD has been utilized to deposit materials with improved wear and oxidation resistance, to deposit bioactive coatings for biomedical implants and to produce functional coatings for electronic, magnetic and related applications, and key early references are given in previous review articles.[1–3,5] In order to improve the wear and abrasion resistance of materials, research has been also focused on the development of metal/ceramic and ceramic/ceramic composite coatings. For the production of metal/ceramic composite coatings EPD is usually employed in combination with electroplating or galvanic deposition of metals.[32–37] Moreover yttria stabilized zirconia (YSZ)/alumina composite coatings have been produced on Fecralloys by EPD[38,39] and densified by a subsequent reaction bonding processes.[38]

It is clear that the most difficult task in the production of ceramic coatings on a metal substrates is related to the limited temperature capability of the metals and the high temperatures required for sintering the ceramic layer. Wang et al.[38] partially resolved this problem by demonstrating that reaction-bonding is an excellent alternative to conventional sintering. Electrophoretic deposition has also been used in ceramic joining applications. Mixtures of SiC or Si_3N_4 and reactive carbon were deposited onto SiC or Si_3N_4 parts to provide intermediate layers for reaction bonding with molten silicon.[40] The results obtained by Lessing et al.[40] are significant because they showed for the first time how the combination of EPD and reaction bonding allows for the fabrication of large complex ceramic structures manufactured from smaller components made of SiC or Si_3N_4. De Riccardis et al.[41] electrophoretically deposited alumina and alumina–zirconia coatings with uniform thickness and homogeneous composition on stainless steel substrates starting from ethanolic suspensions. They extensively studied the suspension properties (conductivity, stability, particle size, transmittance and ζ-potential) to optimize the composition and the amount of

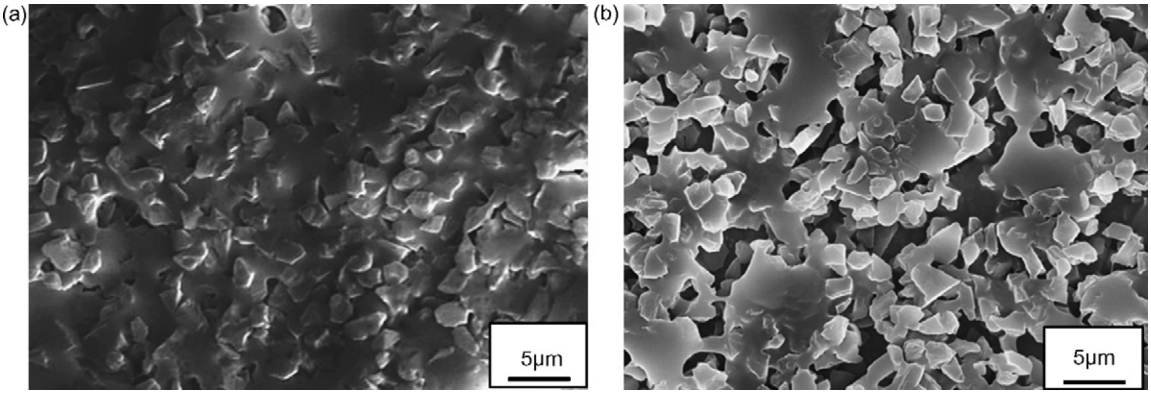

Fig. 4. SEM micrographs of diamond/borosilicate glass composite coatings after sintering. These coatings have been electrophoretically co-deposited on stainless steel substrates from (a) 1.5 g/100 ml diamond and 1.0 g/100 ml borosilicate glass in ethanol and (b) 3.0 g/100 ml diamond and 1.0 g/100 ml borosilicate glass in ethanol.[43] (Published with permission of Elsevier.)

dispersants (citric acid and triethylamine) to be added in order to obtain the required particle dispersion and high suspension stability. Novak et al.[42] produced a firm and pore-free SiC fibre–SiC particle composite by EPD. They studied the effect of suspension composition on the deposition results and they observed that the suspension pH, the solid loading and the particle size (micro or nano) have all a strong influence on the process and on the properties of the fresh SiC deposits. In a recent investigation, Wang et al.[43] deposited smooth, uniform, dense diamond/borosilicate-glass composite coatings onto stainless steel by electrophoretic co-deposition. They also demonstrated that is possible to control the coating microstructure and composition by tailoring the EPD suspension, in fact, for this system, the concentration ratio of the two materials in the coating (borosilicate glass and diamond) was found to be in direct correlation with the diphasic suspension concentration. The coatings were sintered to spread the glass over the diamond particle surface and to protect the diamond particles from oxidization or graphitization. The microstructure of the sintered EPD coatings is shown in Fig. 4.

EPD has also found successful applications in the production of bioactive coatings for biomedical implants and devices. For example, the improvement of EPD for deposition of bioactive hydroxyapatite and related calcium phosphate films on biocompatible metallic substrates (e.g. $TiAl_4V$ alloys and Fecralloys)[44–50] and the deposition of zirconia layers on dental crowns and bridges[51] have been recently reported. The deposition of bioactive glass and polyetheretherketone (PEEK)/bioactive glass composite coatings on shape memory alloy substrates has also been successfully achieved.[52–54]

Some other significant recent developments include: the fabrication of $BaTiO_3$ thick films for sensor and actuator applications,[55,56] ZnO thick films for gas sensors,[57] MgO-modified $Ba_{0.6}Sr_{0.4}TiO_3$ thick films for tunable microwave devices,[58] $LiCoO_2$ electrodes for rechargeable lithium batteries,[59,60] $LiNi_{0.5}Mn_{1.5}O_4$ thick-film electrodes for use in high voltage lithium-ion batteries,[61] V_2O_5 microparticles for cathodes for Li-secondary batteries,[62] MgO thick films for electronics,[63] carbon-polytetrafluoroethylene thin films for gas diffusion electrodes,[64] $BaNd_2Ti_5O_{14}$ thick films for microwave communication devices,[65] zeolites for supported membranes for use in gas separations,[66] ordered mesoporous silicate (MPS) for energy-efficient adsorption systems (e.g. desiccant and cooling systems),[67] PZT coatings for embedded components or for optical switches,[68–73] TaO_xN_y on Ti for its catalytic activity in oxygen reduction reactions,[74] yttrium silicate (YSI) coatings on C/C–Si–SiC composites for protection against oxidation at high temperatures,[75] boron films and MgB_2 films obtained by heat treatment of Mg/B coatings for diffusion of the Mg into the boron film,[76,77] Pb–Zr–Ti–Nb–Si–O ferroelectric thick films[78] and aluminium coatings on FeCrAl substrates.[79] Moreover, the electrophoretic deposition of high-temperature superconducting films with controlled thickness on substrates of various shapes and dimensions has gained increasing interest.[80,81]

3.2.2. *Porous materials*

EPD has been increasingly used to coat textile and porous substrates with ceramic particles to produce a range of porous materials that can be applied for filters, porous carriers, bioactive scaffolds, photocatalysis and hollow fibre fabrication. Zhitomirsky and Gal-Or[82] electrophoretically deposited submicron alumina and zirconia powders on carbon fibres and were able to obtain hollow ceramic fibres after burning out the inner carbon core. Moreover Zhitomirsky[83] demonstrated that hydroxyapatite (HA) coated carbon fibres can produce, after burning out the fibrous carbon substrates, hollow HA fibres of various diameters. A similar study was carried out by Wang et al.[84] who performed repeated HA deposition on carbon rod in order to obtain a thick, uniform and crack-free HA film. It was observed that the uniformity of the coatings and the avoidance of cracking were the result of the repeated deposition process which fills up cracks and hinders crack propagation. After burning out the carbon rod a uniform and crack-free HA ceramic tube is produced, as shown in Fig. 5.[84]

EPD has also been applied by Ma et al.[85] to prepare bioactive porous hydroxyapatite (HA) scaffolds. They demonstrated that the pores were interconnected and that pore size was between several microns and hundreds of microns. Moreover these scaffolds exhibited excellent mechanical properties. Hamagami et al.[86,87] studied the fabrication of highly ordered macroporous bioactive ceramic coating onto titanium by EPD followed by a

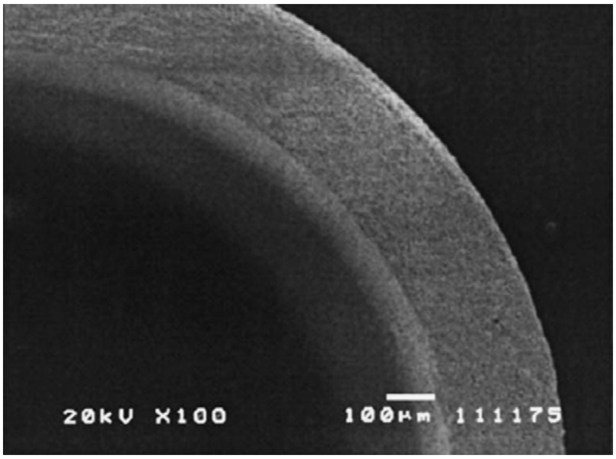

Fig. 5. SEM picture of a uniform and crack-free HA tube produced by EPD after burning out the carbon rod substrate, according to Wang et al.[84] (Published with permission of Elsevier.)

heat treatment. The biocompatibility of these materials was evaluated and demonstrated in vitro using a simulated body fluid (SBF). Yabutsuka et al.[88] deposited wollastonite particles on porous ultrahigh molecular weight polyethylene (UHMWPE) substrates producing a composite. When this composite was soaked in SBF for 14 days an apatite film grew from the wollastonite inside the pores to the top surface of the composite. The apatite presented high adhesive strength to the composite probably due to an interlocking effect. This material might be employed for hard and soft tissue implants for its mechanical properties and high bioactivity. EPD has been also applied to fabricate biodegradable polymer foams coated with bioactive glass particles.[89] Moreover Chen et al.[90] fabricated porous alumina membranes with uniform porous structure and suitable size for application in microfiltration. Huang and Yang[91] produced uniform and dense zeolite membranes on an alumina support and Negishi et al.[92] deposited lanthanum cobaltite on a porous alumina tube by EPD and they were able to obtain dense membranes after a sintering step. Sun et al.[93] deposited by EPD a well-adherent γ-Al_2O_3 washcoat on metallic wire-mesh monoliths for applications in environmental catalysis and other industrial catalysis process (e.g., air pollution abatement devices and automotive emissions controls). In related applications, Yanagida et al.[94] prepared a TiO_2 coating on stainless steel mesh by EPD to examine the synergy effect on photocatalysis of both 1,4-dioxane and ethylene glycol diformate.

3.2.3. Laminated, functionally graded and composite materials

EPD has been employed successfully to fabricate ceramic laminates, fibre reinforced ceramic composites and functionally graded materials (FGMs).[1–3] Laminated materials can be produced via EPD moving the deposition electrode to a second suspension for deposition of a layer of different composition when the desired thickness of the first layer is reached. By changing back and forth, a layered material is readily obtained. Moreover FGM can also be produced using EPD by gradually changing the composition of the suspension from which EPD is carried out. Developments achieved in this field up to year 2000 are reviewed by Van der Biest and Vandeperre[3] and more recent progresses in FGM fabrication have been reported by Put et al.,[95] showing the manufacture of graded WC–Co composites using a suspension of WC powder in acetone with variable Co powder content. Other significant developments achieved by the same group consisted of anodic co-deposition of Al_2O_3 and CeO_2-stabilized zirconia powders to produce cylindrical and tubular-shaped Al_2O_3/zirconia FGM components[96] and the production of functionally graded Si_3N_4–$TiC_{0.5}N_{0.5}$ composites using Si_3N_4 as a matrix and $TiC_{0.5}N_{0.5}$ as the hard phase.[97] Other studies on the EPD production of nickel–alumina,[98] alumina–zirconia,[99–101] Al_2O_3–ZrO_2–Ti(C,N)[102] and hydroxyapatite-bioactive glass[103] FGM coatings have been successfully carried out.

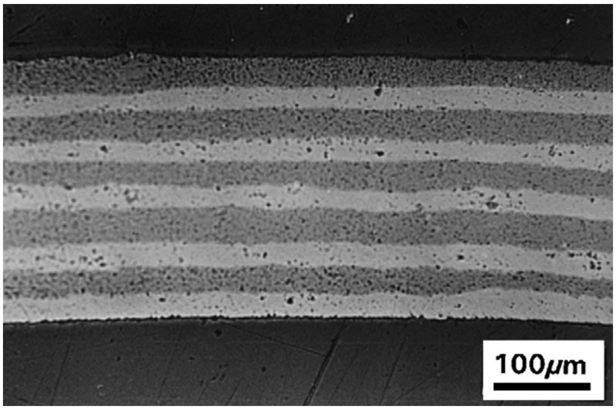

Fig. 6. Microstructure of a laminar material consisting of 10 alternating layers of Al_2O_3 and Y-TZP, prepared by electrophoretic deposition of suspensions of each material by Ferrari et al.[104] (Published with permission of Elsevier.)

Further efforts have been devoted to the development of EPD fabrication approaches for laminated ceramic composites, in particular in the system zirconia/alumina, due to the high fracture resistance of these structures.[104] As an example, a general overview of the microstructure of Al_2O_3/Y-TZP layered composites with 10 layers obtained by Moreno and Ferrari[104] is shown in Fig. 6. The studies of Moreno and Ferrari,[104] Fischer et al.[105] and Uchikoshi et al.[106] have been focused on the optimization of EPD from aqueous suspensions for the production of laminated ceramics. Water should always be preferred over organic solvents due to environmental and economic considerations.[107] In some cases, however, other solvents, e.g. acetone, ethanol, must be used. For example You et al.[108] reported the fabrication of SiC/TiC laminated structures by electrophoretic deposition from acetone-based suspensions. Based on the promising results achieved so far, a significant growth of R&D work related to the electrophoretic deposition of laminated and functionally graded coatings is anticipated.

3.2.4. Ceramic layers for solid oxide fuel cells

Numerous investigations have recently concentrated on solid oxide fuel cells (SOFCs) as new electric power generation systems.[109] The increased interest in SOFCs is due to their high energy conversion efficiency, clean power generation, reliability, modularity, fuel adaptability, the fact that they are noise-free, have excellent long term stability and also due to the versatile nature of the technology for direct conversion of chemical energy to electrical energy.[109–111]

In general, a solid oxide fuel cell device consists of a repeating stack of multiple single fuel cells. In order to render SOFCs economically competitive, however, it is fundamental to significantly reduce the cost of materials and fabrication. For this reason EPD has been progressively considered for the fabrication of cathode- and anode-supported solid oxide fuel cells of both planar and tubular geometry. Several recent papers describe the use of EPD in this field[112–121] and a complete review of the developments achieved so far has been written by Besra and Liu.[5]

From these studies it has emerged that the relative advantages of EPD in the production of SOFCs are the ability to: (a) deposit coatings on substrates of any shape, (b) control the deposition conditions thus being able to prepare porous coating as electrode and dense coating as electrolyte, (c) obtain laminate structures of electrodes and electrolyte and (d) produce Ni-yttria stabilized zirconia (YSZ) cermets (anodes) by electrophoretic co-deposition. Yttria-stabilized zirconia (YSZ) is by far the most popular material used as electrolyte in SOFCs thanks to its exceptional combination of properties such as high chemical and thermal stability and pure ionic conductivity over a wide range of conditions. Also $La_{0.83}Sr_{0.17}Ga_{0.83}Mg_{0.17}O_{2.83}$ (LSGM) has been employed to produce intermediate temperature solid oxide fuel cells (IT-SOFCs).[118] EPD has been also shown to be effective to deposit glass-ceramic layers, which are used as sealant material in some SOFCs designs.[122] Moreover because of the short formation times and the simple equipment needed the use of EPD should simplify the fabrication process of SOFC stacks with complex design architecture achieving further cost reductions. In spite of the progress achieved recently in this area,[5] many problems remain unsolved as described earlier by Zhitomirsky and Petric,[123] who underlined that major difficulties are linked to the selection of adequate solvents and additives, in particular concerning the chemical compatibility of the components of the binder-dispersant-solvent system, the solubility of the binder, the viscosity and the electrical resistivity of the suspensions. Therefore more studies are needed and a better understanding of the process has to be achieved in order to broaden the range of applications of EPD in SOFC technology.

3.3. *Nanotechnology*

3.3.1. *EPD of nanoparticles*

The electrophoretic deposition of ceramic nanoparticles (size <100 nm) is a special colloidal processing method employed to produce a variety of materials, including monolithic ceramics, ceramic coatings and films, functionally graded materials, as well as ceramic laminates and ceramic matrix composites of high microstructural homogeneity. Previous work on EPD of nanoparticles has been reviewed comprehensively elsewhere[8] and only selected recent papers published in the last 2 years are therefore covered in this section.

Tabellion and Clasen[107] have discussed previous work on the fabrication of large components, free standing objects, hollow bodies and objects of complex 3D shape using EPD of silica nanoparticles in aqueous suspensions. However, numerous materials can be deposited by EPD starting from water-based or non-aqueous suspensions of nanoparticles. For example nanostructured $BaTiO_3$ and Eu-doped $BaTiO_3$ thin films have been recently reported, which were produced by EPD of nanoparticles from acetylacetone suspensions,[124] aqueous suspensions[125] and ethanol suspensions.[126,127] Zinc oxide nanoparticles have been deposited from aqueous suspensions,[128] isopropyl alcohol suspensions[129] and 2-propanol suspensions.[130] The production of nanostructured zirconia coatings by EPD has been reported both from aqueous suspensions[131,132] to produce dental crowns[132] and from ethanol suspensions to produce thermal barrier coatings.[133]

Moreover a wide range of dense, nanostructured functional films have been produced by electrophoretic deposition of nanoparticles. Mahajan et al.[134] produced nanocrystalline europium oxide (Eu_2O_3) thin films and noticed that the films optical properties varied with deposit morphology: translucent, when there was a uniform size and distribution of the microstructure, and opaque, when there was a marked anisotropy of the size and distribution of the constituents of the microstructure. The deposition of nanostructured titania films by EPD has been carried out by several groups.[135–141] For example Dittrich et al.[135,136] produced TiO_2 coatings with different porosity, systematically changed by pressing, in order to optimize the electron diffusion. These coatings present potential applications in many fields, such as batteries, displays, photocatalysis and solar energy conversion systems. Manríquez and Godínez[140] deposited Ti(III)-doped nanocrystalline TiO_2 films on optically transparent electrodes. Valatka and Kulesius[141] deposited nanosized titania films on stainless steel and then they used the decoloration of methylene blue dye to evaluate the photoelectrocatalytic activity of the coatings. Hydroxyapatite (HA) nanoparticles have been extensively deposited by means of EPD.[142–146] Other nanoparticles were deposited by EPD such as: nanosized lead zirconate titanate (PZT) powder in an ethanol suspension,[69] silicon carbide,[147] ceria,[148] vanadium pentoxide,[149] hydrous ruthenium oxide,[150,151] gamma ferric oxide,[152] nickel,[153,154] iron,[153] aluminium,[153] silver,[155] nickel ferrite,[156] silica,[157,158] titania nanotubes,[159,160] polyimide[161] and yttria-stabilized zirconia.[162]

Recently, research has been carried out on the deposition of polymeric nanocomposite coatings. For example, polythiophene and metal oxide (alumina, titania and silica) nanocomposites have been electrophoretically deposited from an ethanol suspension by Vu et al.[163] They obtained thin films of conducting polymer/metal oxide with a core-shell structure that can be employed in electronic devices. Kim et al.[164] deposited poly(methyl methacrylate)-$BaTiO_3$ nanocomposite coatings from an isopropyl alcohol/acetone solvent mixture on copper foils. The composite films presented a uniform microstructure without particle agglomeration. Furthermore, the deposition of metal–ceramic nanocomposite coatings has also been investigated. For example, nanostructured Ni–WC–Co composite coatings have been produced by means of EPD on nickel-plated stainless steel substrates.[165] These composite coatings

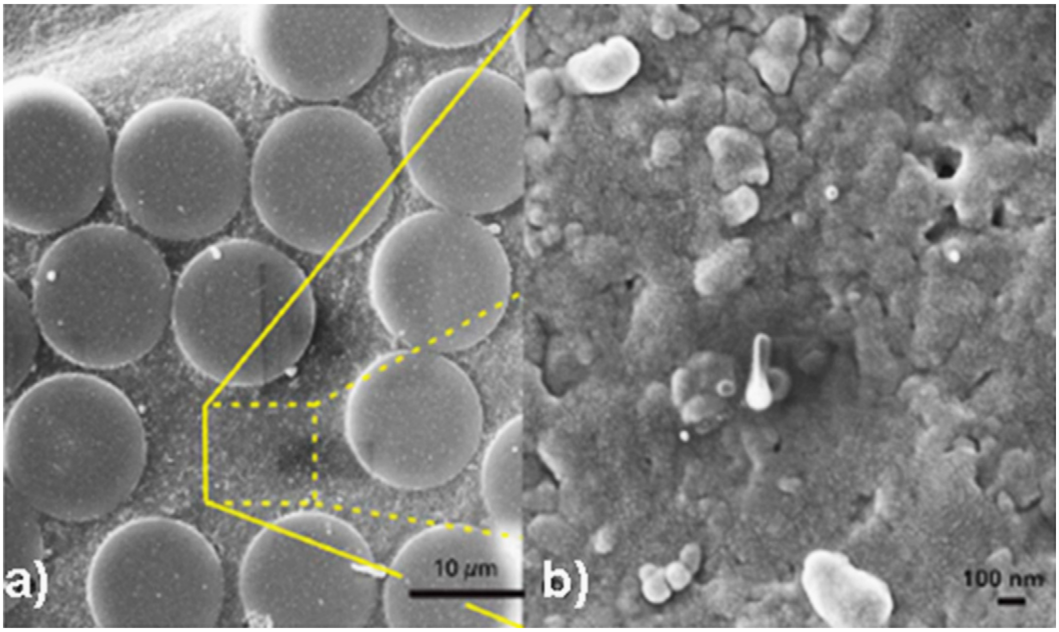

Fig. 7. SEM micrographs of a Nextel™ 720 fibre mat infiltrated with alumina nano particles, (a) shows the complete and homogeneous infiltration of the Nextel™ 720 fibre mat with alumina and (b) shows the high "green" density of the alumina matrix by Stoll et al.[169] (Published with permission of Elsevier.)

presented a significant increase in hardness and wear resistance compared with pure nickel coatings or stainless steel.[165] Wang et al.[146] deposited Co-YSZ/HA nanocomposite coatings on Ti substrate using the combination of electrocodeposition and electrophoretic deposition. They demonstrated that the Co-YSZ/HA composite coatings exhibited better mechanical properties than nano-HA single coatings. Moreover the Co-YSZ interlayer reduced the mismatch of the thermal expansion coefficients between HA and Ti and the adhesive strength of the composite coating and Ti substrate was higher than that of nano-HA single coatings on Ti substrate. Pang and Zhitomirsky[166] deposited by EPD composite hydroxyapatite-chitosan coatings on stainless steel substrates. They observed that the addition of chitosan to the HA suspension promoted the deposition of HA. Moreover, from the analysis of the coatings it was demonstrated that changing the concentration of the two chemicals in the suspension resulted in a variation in the coating composition. These composite coatings were adherent and uniform with increased corrosion protection properties. Louh et al.[167] deposited by EPD nanosized platinum/carbon nanocatalysts and Nafion® solution on carbon-based substrates for applications in proton exchange membrane fuel cells (PEMFCs). They obtained a uniform distribution of catalyst and Nafion® on the electrodes, in fact the Pt/C nanopowders covered the whole carbon fibre surface and also infiltrated into the gaps and voids between them.

EPD has been also employed for the production of fibre reinforced ceramic and glass matrix composites with a variety of ceramic matrices and fibres.[8,168] This method is particularly attractive for the production of CMCs with complex fibre structures (e.g. 2D fibre fabrics) as reinforcement because it allows adequate infiltration of ceramic (nano) particles into the inter-fibre spaces, which would be difficult to be achieved by other routes.[168] Additionally, it should be emphasised that the fibres employed can be both electrically conductive (e.g. SiC Nicalon®, carbon) and non-conductive (e.g. alumina or mullite). When the fibres are non-conductive, the fibre weave is placed in front of the deposition electrode, the ceramic deposit develops on the electrode and grows around and through the fibre mat.[8,168] Stoll et al.[169] fabricated alumina–alumina CMCs by means of EPD from a suspension of Al_2O_3 nanoparticles using Nextel®-type (alumina) fibres. The same composite (alumina particles and Nextel®-type fibres) was investigated recently by Bao and Nicholson.[170] In this particular system it was shown that in order to be able to infiltrate fibre mats by EPD it is fundamental that both the suspended particles and the fibres have the same polarity.[169] In these conditions, when the particles approach the fibre mat repulsion forces take place between them, the magnitude of these forces depends on the relative distance between particles and fibres. Due to the applied external electrical field, each charged particle is attracted to the fibre mat, which is fixed to the electrode, but, at the same time, the particles are repelled before they can reach the fibre surfaces (coagulation point) due to the charge on the fibres. It has been hypothesised that under the effect of the repulsive forces due to the surrounding fibres, the particles follow the path with the fewest possible obstacles until reaching the next interstice between adjacent fibres. Therefore, when the particles reach the electrode or the surface of previously deposited particles, they cannot move further and consequently the electrophoretic ceramic deposit grows with a high particle packing density. Fig. 7(a) and (b) shows the uniformity of the infiltration of alumina particles in the Nextel®-type (alumina) fibre mat and the high level of packing achieved by the electrophoretically deposited alumina particles.[169] Conversely, if the fibre and particles exhibit opposite surface charge, coagulation is expected on the first layer of fibres encountered by the travelling particles. In this case a deposit on the outer fibre layer will block the movement of the particles towards the interior

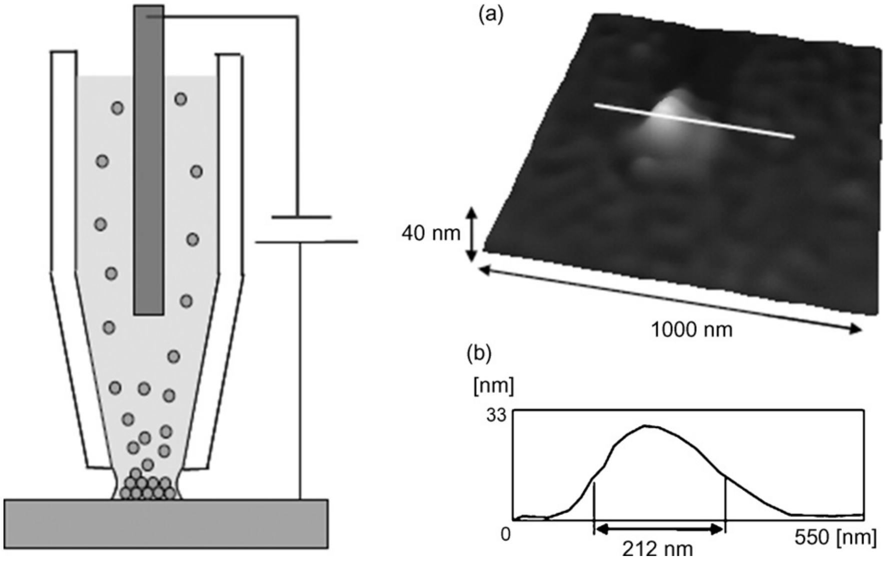

Fig. 8. Schematic of the EPD nanocell employed by Iwata et al.[173] to deposit Au dots: (a) topographical (AFM) image of Au dots deposited by EPD and (b) cross-section of the Au dot shown in image (a). (Reproduced with permission of IOP Publishing Ltd., UK.)

of the fibre mat, resulting in poor infiltration and low quality microstructure.

A further development of EPD is reactive electrophoretic deposition, that has been described by Clasen et al.[171,172] to produce doped functional glasses. These glasses are normally created by melting silica at 2100 °C, but at this high temperature most of the suitable dopants evaporate. An alternative process for the fabrication of doped glasses is the reactive EPD technique that consists of adding soluble salts into a silica suspension, these salts dissociate and the ions are adsorbed on the surface of the particles, which are then deposited by EPD producing a homogeneously doped green body. Shaping of a green body and doping are achieved in a single step by means of reactive EPD, whereas more than one step and very high temperatures are needed by the conventional route.

Iwata et al.[173] demonstrated that EPD can also be employed to fabricate nanostructures, such as nanomachines and components for nanoelectronics. This group utilized local EPD to deposit gold nanoparticles (dots) on Si surfaces from a nanopipette probe filled with the deposition suspension. The nanopipette probe was the EPD cell and the two electrodes were a thin metal wire positioned inside the nanopipette and a conductive surface that was practically in contact with the edge of the nanopipette (Fig. 8). When a difference of potential was applied between them, the colloidal particles migrated toward the edge of the probe and were deposited on the surface. They also demonstrated that it was possible to modify the size of the Au dots by changing the deposition time and the voltage.

3.3.2. EPD of carbon nanotubes (CNTs)

In the last few years the interest of the scientific community in carbon nanotubes (CNTs), both single-walled (SWCNTs) and multi-walled (MWCNTs), has increased dramatically as reflected by the huge number of papers published and patents filed related to CNTs.[7,8,174,175] Many of the properties of CNTs are now well-known and their use in a wide range of applications is spreading, with current research and development efforts focused on expanding the application potential of CNTs.[176–178] However, it is recognized that to produce particular arrangements of CNTs, individually or collectively, for a given application and to combine CNTs with other materials to form composite materials and devices, it is fundamental to disperse the CNTs homogeneously in the appropriate polymer, ceramic or metallic matrix. It is now well known that EPD is a very convenient technique to manipulate CNT in order to form ordered, oriented nanotubes arrays.[7]

To prepare a stable CNT suspension for EPD, several solvents have been employed, including distilled water, mixtures of acetone and ethanol, and pure organic solvents such as ethanol, isopropyl alcohol, *n*-pentanol, ethyl alcohol, tetrahydrofuran, dimethylformamide and deionised water with pyrrole.[7] The preparation of a stable suspension of CNTs, in which CNTs have a high ζ-potential and the suspension has a low ionic conductivity, is necessary for successful EPD. The stability of CNT suspensions, determined by ζ-potential measurements, has been studied mainly in aqueous and ethanol-based suspensions. Furthermore, it has also been shown that the presence of charger salts can play an important role in improving the adhesion of CNTs to substrates and in increasing the deposition rate. The salts can also contribute to the stability of the suspensions by associating a charge to the CNT surface. This charge determines the migration direction of CNTs in suspension during EPD and therefore the deposition electrode. Using different types of salts: quaternary ammonium salts, benzalkonium chloride, $NiCl_2$, $Mg(NO_3)_2$, $MgCl_2$ and NaOH a finer control can be achieved. Additionally, it is worth noting that the high aspect ratio and surface charge of acid-treated CNTs makes them suitable scaffolds or templates for deposition of other nanoparticles, such as metallic and oxide nanoparticles, via adsorption or nucleation at the acidic sites.

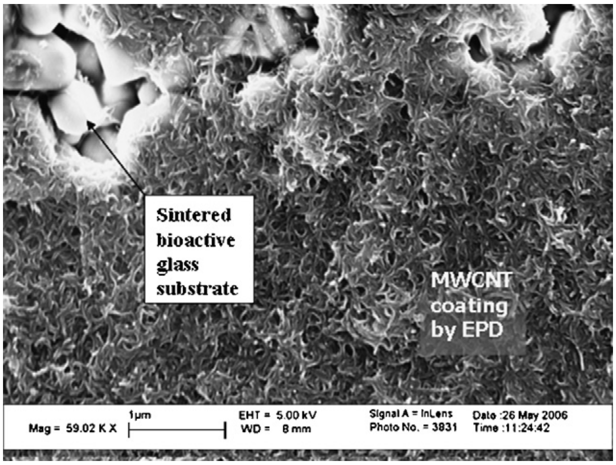

Fig. 9. SEM image of the electrophoretically deposited MWCNT coating on the surface of a bioactive glass foam, which was placed in front of the anode during EPD (MWCNT were negatively charged).[186]

The most effective method for dispersion of carbon nanotubes in a liquid medium is to add a surfactant and to apply ultrasonication.[7] A different method based on the application of an electric field to the suspension has been recently developed by Zhang et al.[179,180] They applied an AC electric field to suspensions of carbon nanotubes and compared the results with those obtained without applying an electric field; they noticed that the dispersion status of carbon nanotubes in liquid media was considerably improved by applying an electric field. Moreover they observed that the dispersion results were strongly dependent on the magnitude and frequency of the applied electric field. Du and Pan[181,182] deposited MWCNT thin films by EPD to produce supercapacitors with high specific power density and a very small equivalent series resistance. These thin films could be applied as coating layers over ordinary current collectors to considerably enhance the electrode performance.

Jung et al.[183,184] deposited a thin film of horizontally aligned SWCNTs from an aqueous mixture of CNT and detergent. The coating was naturally dried and then pressed on the surface to make it smooth. The films strongly adhered to the substrate and showed good field emission properties. Recently, Hasobe et al.[185] deposited SWCNTs and protonated porphyrin on nanostructured SnO_2 electrodes for applications in photochemical solar cells.

As mentioned above, beyond the fabrication of uniform, planar, CNT-based coatings, EPD can be applied to deposit CNTs onto complicated structures, including microwires, porous substrates and fibrous bodies or textiles.[7] Boccaccini et al.[186] have deposited MWCNTs onto highly porous bioactive glass scaffolds with the intention of imparting a monotopography to the pore wall surfaces. Fig. 9 shows the surface of MWCNT deposition on the 3D pore network of the Bioglass® foam, which was placed in front of the anode (MWCNT were negatively charged). There has been also recent development on the fabrication by EPD of CNT reinforced HA coatings for biomedical applications.[187] The fabrication of more complex patterns of CNT deposits can be realized by using masks or by designing combinations of conductive and non-conductive surfaces. Moreover Wang et al.[188] have deposited uniformly CNT coatings on glass plates with a silver or a ITO film layer and they observed that the emission properties of these CNT films were as good as those of the coatings produced by other methods such as screen printing. EPD seems thus to be a low cost and favourable method to produce CNT films for field emitters. Moreover composites consisting of ceramic nanoparticles and MWCNT have been produced recently by sequential EPD and by electrophoretic co-deposition.[189,190]

The results presented in the literature demonstrate that EPD is a very versatile method to manipulate CNTs, thus the technique is likely to be a focus of research efforts in the near future. EPD is a potentially powerful method to produce CNT-based devices, particularly because few alternative techniques exist to deposit and align CNTs on the required surfaces. CNT films may be suitable for a wide range of applications; suggestions to date include field emission devices,[183,184] biomedical scaffolds,[186] catalyst supports, structural composites and coatings, large surface area electrodes for fuel cells, photochemical solar cells,[185] electrodes for electronic devices (supercapacitors and battery manufacturing),[181,182] capacitors and gas sensors. Similarly, EPD of CNTs can be seen as a very effective process to create CNT membranes and nanofilters which are commonly made by slow and tedious filtration of CNT suspensions.

3.3.3. Nanorods, nanowires, nanotubes and nanosheets

There has been an increasing interest in the use of electrophoretic deposition techniques for the fabrication of a variety of nano-architectures, including nanorods, nanowires, nanotubes and nanosheets. This section presents the most recent research carried out in this field.

Zinc oxide nanorods, nanowires and nanotubes have been synthesized and electrophoretically deposited by Rout et al.[191] to produce sensors for hydrogen and ethanol. The sensing characteristics of these nanostructures were examined before and after impregnating them with 1% Pt. The nanowires exhibited excellent hydrogen sensing characteristics at temperatures below 150 °C especially when impregnated with Pt. The nanorods showed a satisfactory dependence of sensitivity on hydrogen concentration. Moreover nanorods and nanowires impregnated with Pt demonstrated high sensitivity for ethanol at temperatures below 150 °C, with short recovery and response times. Kim et al.[192] converted commercial titania nanoparticles to nanotubes using an hydrothermal method and then deposited a titanate nanotube film on a Si substrate by EPD.

Porous nanosheet-stacked $NiCo_2O_4$/Ni composite electrodes have been produced by a novel electrophoretic deposition calcination method. These electrode films present a crystalline structure and they are composed of regular hexagonal $NiCo_2O_4$ nanosheets with an average diameter of 200 nm. Moreover these films exhibited good electrocatalytic properties for water electrolysis.[193,194] Sugimoto et al.[195] prepared stable colloids of ruthenic acid nanosheets in acetonitrile or *N*,*N*-dimethylformamide. The nanosheets were subsequently deposited by EPD on gold, indium-tin oxide coated glass and indium-tin oxide coated poly(ethylene terephthalate) electrodes which presented high energy density at room temperature.

4. Conclusions

Electrophoretic deposition is a very versatile and cost-effective material processing technique, offering control over nano and microstructure, stoichiometry, microscopic and macroscopic dimensions and properties. In the last 10 years there has been a considerable increase of the areas of application of EPD, such as production of ceramic layers for solid oxide fuel cells and fabrication of ceramic matrix composites, functionally graded materials, laminated ceramics and advanced coatings for tribological and functional applications as well as manipulation of nanoparticles, nanotubes and other nanostructures. Moreover EPD demonstrates substantial technical advantages compared with other fabrication routes due to the fact that it operates at low temperatures, can be easily scaled up using inexpensive equipment and, consequently, this method has the potential to lead to commercial success and large-scale production. Specific areas where EPD is expected to expand are: fabrication of nanostructured and hybrid composite materials, functionally graded materials, laminated nanoceramics as well as nanostructured films and coatings for functional applications, in the electric, superconducting, semiconducting, optical, magnetic and biomedical fields.

It is also recognized that further research efforts in the areas of analytical and numerical modelling of the mechanisms of electrophoretic deposition are necessary, in order to change the empirical, often non-satisfactory and time consuming trial-and-error approaches which have dominated the experimental work and technological developments in the area so far. A holistic understanding of the mechanisms involved and development of processing/structure/property relationships will allow better design of EPD systems for given applications.

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