

Abstracts from

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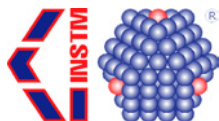
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SELECTIVE LASER MELTING OF ALUMINIUM MATRIX NANOCOMPOSITES

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Introduction: Selective Laser Melting (SLM) is a powder based layer additive manufacturing process that can produce fully dense metal parts without the need of post processing steps. In the past years a wide range of materials such as steel, titanium, nickel and aluminum alloy were produced by this technology. This near net shape layer-by-layer process could be particularly suitable for the production of metal matrix composites (MMCs) because of the possibility to homogeneously disperse the second phase within the matrix. In this work an AlSi10Mg+Al₂O₃ composite samples were produced by SLM taking advantage of the reaction that arises between aluminum and silicon oxide.

Materials and methods: An EOS M270 Xtended system was used to build all the samples. This system works in argon atmosphere and uses and ytterbium fiber laser as energy source to selectively melt thin layers of metal powders. An AlSi10Mg spherical powder was used as a matrix and dry mixed with 0.5% wt SiO₂ nanoparticles in order to obtain an AlSi10Mg-Al₂O₃ composite. Cubic samples were built by varying the main building parameters in order select the ones that allow the obtainment of fully dense samples. The Volumetric Energy Density (VED) parameter was used to understand the overall effect of the building parameters on the consolidation phenomena. The VED value is calculated by the following equation:

$$VED = \frac{P}{vhd} \quad [1]$$

where P is the laser power v the scan speed h the hatching distance and d the layer thickness. Samples for the mechanical test were built with the selected parameters and compared with the AlSi10Mg ones.

Results: The analyses of the porosity values revealed that the introduction of a small quantity of SiO₂ nanoparticles strongly influences the consolidation phenomena. The highest densities of the AlSi10Mg+Al₂O₃ samples were achieved by using low energy densities. On the contrary, in most of the cases, the higher the VED the lower the porosity. The mechanical tests revealed that the composites samples produced with the optimized parameters are characterized by lower yield strength.

Discussion: It is possible to produce fully dense aluminum based MMCs by SLM by using the optimized parameters. It was noticed that the nanoparticles alters the consolidation phenomena. The optical micrographs of the porous samples revealed that most of the pores have a spherical shape indicating that they are due to trap gas rather than to a consolidation issue. The mechanical tests revealed that the mechanical properties of the composites are strongly affected by the building parameters it is necessary to use.

MULTISCALE FABRICATION OF BIOCOMPATIBLE AND BIODEGRADABLE SCAFFOLDS FOR REGENERATIVE MEDICINE

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Introduction: Regenerative medicine attempts to repair or improve the biological functions of tissues that have been damaged or have ceased to

perform their role through three main components: a biocompatible scaffold, cellular component and bioactive molecules. Nanotechnology, using advanced manufacturing techniques such as conventional and unconventional lithography, allows fabricating supports with geometries, sizes and displaying physical chemical properties tuneable over different length scales. In this work we report the fabrication of scaffolds made of fibrin gel and squid pen chitin, for the regeneration of tissues.

Materials and methods: Fibrin gel films are used to observe the growth of cells on random networks. Films of different thickness have been fabricated with special attention to the realization of a micro-frame that allows a simple manipulation of the structure. Such a standing fibrin scaffold prevents the artefacts arising from the interaction of the films with other surfaces. The fibrin gel clots have then been stretched to observe if the cells growth and migration is influenced by the orientation of the fibers. Fabricating a scaffold via bottom-up techniques to mimic tissues is not simple. We have chosen to adopt a top-down strategy using a scaffold manufactured by nature and composed of extremely ordered chitin fibers: squid pen. The squid's pen is a support consisting of chitin fibers transparent, biocompatible, low cost and displaying high mechanical resistance. We have changed the chemical and physical properties of the scaffold to promote cell adhesion. Both supports were functionalized with retinoic acid to obtain a support able to induce cell differentiation. We characterized the scaffolds by scanning electron microscopy and immunofluorescence technique.

Results: We have fabricated two types of scaffolds and we have observed cell growth on both scaffolds. We fabricated active scaffolds able to differentiate the stem cells into neuronal cells. The ultrastructure characterization of the scaffolds is crucial for evaluating their morphology, porosity and biological efficacy. Power spectrum analyses of the images have been performed to provide the characteristic lengths of the supports.

Discussion: An important goal of this work was the fabrication of 3D scaffolds with a well-defined morphology to be used as a starting point for the regeneration of portions of tissue. The realization of a fibrin scaffold, of different thickness and easy to handle fabricated under controlled spatial confinement has been demonstrated. Characterization of the fibrin network has helped us to understand how the morphology of fibrin network might affect the cell growth. We propose the squid pen as a scaffold because it is biocompatible, transparent, it withstands surgical sutures and, moreover, it is a waste material of the industrial chain. The purpose is to exploit as much as possible the original remarkable properties provided by the structure of squid pen chitin to obtain a scaffold transparent and able to support all the mechanical stress. Furthermore, in a period characterized by the necessity of producing goods by a sustainable chemistry and a green economy, finding the way for using an available natural biomass has become a must for all scientists and industrial manufacturers.

INFLUENCE OF CLIMATE CONDITIONS IN THE SETTING TIMES OF HYBRID MATRIX SYSTEMS USED FOR RECOVERY OF DAMAGED BUILDINGS

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Introduction: Nowadays, composite materials -fibers inside a polymeric resin- are successfully applied in the field of civil engineering. A promising variant of this technology involves the rehabilitation of damaged structures using hybrid matrices (inorganic component plus a polymeric resin). However, their use in construction yards with variable climate conditions influences the setting time (ST) of the mixture. This variability results in practical limitations that compromise the success of the application. In this research, a study of the influence of the climate conditions in the ST of three different hybrid matrices composed of gypsum and a polymeric resin was performed.

Materials and methods: Three commercial gypsums (A, B and C) and one aqueous polymeric resin were used. The composition of the gypsums was determined by XRD and DTA-TG. The measurements of the ST's were carried out inside a glove bag at controlled relative humidity (RH) and temperature (T). Their values were chosen based in their average maximum and minimum values in Europe: T = 5, 20 and 35°C and RH = 45, 67 and 90%. The tests were performed by combinations of each value of T and RH.

treatments on fibres. SEM has also shown a good dispersion of paper fibers and wood particles and the interaction between two different morphologies reinforcements: the simultaneous presence of fibres and particles allowed reducing the microporosity. The same result was confirmed from the water absorption. In fact, water absorption's test showed a higher mass variation for samples with 30 wt% of wood than 30 wt% of wood and 10 wt% of fibres: the fibres' presence improved the consistence of the composite during the mixing process, reducing microporosity.

Discussion: The results obtained support papers' introduction as filler in WPC. Nevertheless paper micronization treatment is needed: this step influence paper morphology before its introduction in the matrix. In future works particles will be tested to analyse soundproofing or damping. Turbomixing production wasn't the only option: an industrial widespread alternative is the twin-screw extruder, even if it results more expensive and complex to manage. In order to reduce the amount of polymer, a future improvement will be blending with starch derived polymers. Other tests will be performed to complete the composite characterization like impact tests, thermal analysis, and fire and UV resistance.

PAPER-BASED SMART INTERFACES: CONTROLLING INFORMATION STORAGE BY CHEMICALLY ACTIVATED OSCILLATIONS

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Introduction: Research on paper-based devices is currently blooming in a large variety of applications, in particular advanced diagnostics and flexible electronics. The capability to store information with controlled lifetime is expected to have major impact on cellulose-based materials, with interesting applications in security, anti-counterfeiting, device self-diagnostics, labeling, packaging and quality control. The idea of this work is to extend the advantages of adaptive and non-static materials to commercial cellulose, in order to store self-expiring graphical information on paper-based supports with precise temporal and spatial control. This goal is pursued by activating chemical oscillations from non-oscillating Belousov-Zhabotinsky (BZ) solutions loaded in cellulose supports.

Materials and methods: In the present work, we use the BZ reaction as a pacemaker to store information with spatial and temporal control in paper-based devices. In order to do this, we prepared the Belousov-Zhabotinsky reaction medium in an excitable nonoscillating state, by mixing the appropriate amount of KBrO_3 , H_2SO_4 , KBr , citric acid and $\text{Fe}(\text{phen})_3\text{SO}_4$. Then, different amounts were dropped onto commercial cellulose filters. Writing/self-erasing was activated by means of iron tips or bullets. Analogous tests were carried out using small pieces of zinc, aluminum, magnesium, glass and polyethylene terephthalate. Further experiments of chemical activation were carried out by using aqueous solutions of different salts (FeCl_2 , FeCl_3 , KBr , KNO_3 , NaCl , KCl and AgNO_3).

Results: When the BZ medium is absorbed onto the cellulose substrates, it acquires a blue color, typical of the reduced state of the catalyst present in the reaction medium. It has been noticed that when a contact between the BZ-functionalized cellulose and an external object is introduced, a change of color from blue to red, that is representative of a change of oxidation state of the catalyst, occurs. For example, small pieces of iron, like bullets, tips or wires, are able to activate the change of color and can be used as pens for writing graphical tracks on the paper substrate. Thanks to the oscillatory nature of the involved chemical reaction, the original color is restored over the time, as a consequence of a spontaneous self-erasing process. This offers the opportunity to program the storage of optical information within a specific temporal interval.

Discussion: Different experiments were carried out to gain more insight into the mechanism at the base of the writing process, demonstrated the chemical nature of activation. What is relevant is the capability of the material utilized as an "activator" to generate a local gradient of concentration of the species involved in the BZ reaction. The specific contribution of the main parameters affecting writing and self-erasing processes (temperature, reaction medium volume) was evaluated in a series of systematic experiments. This concept can be extended to other types of oscillating reactions, which offers a rich palette of applications for sensing, diagnostics, energy conversion and paper-based electronics.

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SHAPE MEMORY POLYCAPROLACTONE NON-WOVEN MAT BY COMBINING ELECTROSPINNING AND SOL-GEL REACTION

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Introduction: Shape memory polymers (SMPs) are a class of smart materials able to interconvert between a 'temporary' shape obtained after a programming step and a previously set 'permanent' shape, upon the application of an external stimulus. Electrospinning technology has been exploited to fabricate micro/nano fibrous matrices with shape memory (SM) properties that in some cases displayed an enhanced SM behavior than the corresponding bulk films. In order to obtain electrospun mats with good SM properties and to overcome the limitations of complex approaches, in this work we propose a novel approach by combining electrospinning process and sol-gel reaction that enabled to achieve high crosslinking degrees without using any template polymer.

Materials and methods: A partially crosslinked polycaprolactone (PCL), synthesized through sol-gel chemistry starting from a α,ω -triethoxysilane-terminated PCL was used to gain bead-free fibers. After electrospinning, the crosslinking degree has been subsequently increased and tuned by applying a controlled thermal treatment. Electrospun samples with different crosslinking degrees have been prepared and the effect of the different crosslinking extent was correlated to the SM property of the material by applying an ad hoc thermomechanical cycle. Furthermore, gel content, degree of swelling and morphology of the samples have been studied.

Results: Partially crosslinked α,ω -triethoxysilane-terminated PCL solutions were successfully electrospun, allowing to obtain bead-free fibrous non-woven mats. The viscosity of the electro-spinnable solution was optimized by controlling the hydrolysis/condensation reaction time activated by water and HCl, while the crosslinking degree of PCL fibers was controlled by varying the exposure time of non-woven mats to acidic vapors. The mechanical properties (storage modulus and tensile modulus) in the rubbery plateau region were consequently governed by the crosslinking degree of PCL.

Discussion: The evolution of fiber architecture within the non-woven mat with the applied strain was observed: the fibers underwent rotational/flexural motions, reorienting as the strain increases and, for strain values higher than 50% the fibers became highly stretched and aligned along the direction of strain. All the prepared electrospun non-woven mats exhibited very good one-way SM properties (high fixation and recovery capabilities), almost independently on the applied strain and the crosslinking degree of PCL fibers.

EFFECTS OF WAVINESS FACTOR ON DISTRIBUTION LENGTH AND IFSS OF NATURAL FIBERS COMPOSITES

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Introduction: During extrusion and injection moulding of composites, natural fibres suffer of entanglement phenomena in the polymeric matrix. Consequently the fibre waviness lead to lower mechanical properties of the composites than those expected on the basis of predictive models application. In fact the most widely used models for the estimation of the interfacial shear strength (IFSS) do not consider this phenomenon. This work introduces a method for IFSS calculation taking into account the waviness factor, which affects the weight distribution of the fibre length.

the best film configuration, in terms of thicknesses of the different layers and positions, mathematical modeling can be recognized as an effective virtualization tool to limit the experimental trials before film production. In this work, a mathematical model was developed to predict the barrier properties and the scavenging performances of multilayer polyester films with oxygen scavenging activity. Model results were validated by experimental characterization with particular regard to oxygen permeability and transmission rate.

Materials and methods: "ABA" type symmetrical multilayer PET films and "B" type monolayer active PET films were considered for this study. The external "A" layers were pure PET, while the middle "B" reactive layer was PET loaded with 10 wt% of oxygen scavenger (Amosorb DFC 4020, by Colormatrix). Films with different layout were produced with a laboratory coextrusion cast film line (Collin Teach Line E20T). Oxygen absorption measurements were performed putting the film in a vial and then measuring the decay of oxygen concentration in the vial itself.

The concentration profiles of oxygen in the film were calculated by making use of a mono-dimensional reaction-diffusion equation in both multilayer and monolayer systems. The equations were solved numerically using a finite element based multiphysics tool to predict the concentration profile.

Results: The scavenging capacity of the multilayer films increases linearly with the increase of the reactive layer thickness, and the oxygen absorption reaction at short times decreases proportionally with the thickness of the external PET layer. Both experimental and theoretical results also revealed that in multilayer PET films, at the exhaustion of the scavenging effect of the active layer, the steady transport properties are slightly different in comparison to pure PET. As a result, the oxygen transfer rates and permeability were also reduced.

Discussion: The oxygen scavenging kinetics depends on the layer thickness and the relative mass ratio of the constituents. At fixed active layer thickness and composition, the flux plateau of the oxygen diffused into the layer is lower in the multilayer films, having the reactive layer in the middle, than in the monolayer active film. As the thickness of the reactive layer increases, diffusion takes longer time and consequently contact time between oxygen and scavenger increases. This slows down the oxygen transport across the layers.

MICROMORPHOLOGY OF QUANTUM DOT SUPRAMOLECULAR SOLIDS

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Introduction: Semiconductor nanoparticles, known as quantum dots (QDs), are inspiring a growing number of applications, mainly due to their peculiar optical properties – such as the very strong and finely tunable absorption and photoluminescence in the visible range. Most of QD-based applications, however, exploit the properties of the individual QDs; however, quite an interesting and untapped potential lies in the collective properties (mainly optical and electrical) that emerge in arrays of interacting, rather than uncoupled, QDs. It is of great interest, therefore, to develop appropriate techniques to assemble QDs in a reliable and controllable way, ensuring on one hand a macroscopic shape appropriate to the selected application, and on the other hand the desired characteristics at the nanoscale – such as for example the degree of electric coupling, or the spatial distribution of the QDs. We have studied the conditions for QD assembly via a supramolecular approach, whereby assembly is mediated by metallic ion-induced bonding between opportunely selected capping molecules. Since the capping can be finely engineered – for example in terms of chemistry, positioning on the QD, electrical conductivity – the supramolecular assembly approach is expected to be a very powerful tool for controlling the morphology and therefore the properties of QD colloidal solids.

Materials and methods: Colloidal quantum dots made of II-VI compound semiconductors were prepared via standard hot injection synthesis in sizes varying between 2.5 and 4 nm, and the native oleic acid capping was then exchanged for selected terpyridines. The terpyridine-QD complexes were mixed with Co(II) ion solutions in concentrations such that the ratio QD/Co(II) would vary between 1:100 to 1:1; aliquots of the mixtures thus obtained were extracted at times varying between 10 minutes and 4 weeks, and examined after solvent evaporation via optical microscopy, scanning electron microscopy, and Raman spectroscopy.

Results: In most cases, a solid powder starts precipitating shortly after the QDs and the Co(II) are mixed; Raman spectroscopy confirms that the precipitate contains QDs. Optical microscopy and SEM show the formation of spheroidal particles of 1-5 μm , loosely aggregated. The general trend is that the diameter of the spheroidal particles grows as the QD/Co(II) ratio decreases.

Discussion: The formation of the precipitates can be explained by the formation of coordinative bonds, mediated by Co(II), between the terpyridine capping of different QDs, thus demonstrating the feasibility of a supramolecular approach for assembling QDs as colloidal solids.

ENHANCED PHOTOCATALYTIC ACTIVITY OF TITANIA NANOTUBES BY LIGAND-FREE AU NANOPARTICLE LOADING

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Introduction: In the last years titania nanotubes arrays (TNTAs) have attracted increasing attention in photocatalysis due to their unique properties, not least the possibility of a direct production of supported TNTAs by anodic oxidation of metallic titanium foils. Besides, loading of TiO₂-based photocatalysts with Au nanoparticles (NPs) has been extensively investigated so as to improve the photocatalytic efficiency. In order to combine the advantages of TNTAs with Au NPs decoration and to overcome the problems derived from current deposition methods this work proposes a simple and scalable synthetic protocol able to load size-controlled (<10 nm) and ligand-free Au NPs onto vertically aligned anatase TNTAs.

Materials and methods: Metal-supported TNTAs were produced by titanium foils anodization in a solution of 0.5 wt.% NaF/1M Na₂SO₄. The samples were then thermally annealed in air at 400°C for 2 h in order to achieve the crystallization of the obtained amorphous TiO₂. Ligand-free Au NPs were produced by the Metal Vapour Synthesis technique. Au vapours generated at 10⁻⁴ mBar by resistive heating of an alumina crucible filled with gold pellets were co-condensed at liquid nitrogen temperature (-196°C) with acetone. The Au NPs were loaded onto the TNTAs surface by simply dipping the samples into the resulting acetone NPs suspension. The photocatalytic activity was assessed by measuring the toluene degradation in air using a continuous-flow stirred photoreactor operated at constant toluene concentration.

Results: The obtained Au loadings were strictly related to the sample dipping time into the ligand-free Au NPs suspension. The morphological features of Au/TNTAs composites revealed a highly homogeneous Au NPs dispersion without the particle coalescence often reported using colloidal deposition methodologies. The photocatalytic activity of the pristine batch of bare TNTAs samples was quite homogeneous and a marked increase of activity after Au NPs deposition was observed for the 3.3 and 7.4 $\mu\text{g cm}^{-2}$ Au loading samples. At lower Au loading (<2 $\mu\text{g cm}^{-2}$) the reaction rate was comparable to the untreated sample whereas at higher loading (>10 $\mu\text{g cm}^{-2}$) a pronounced deactivation was observed.

Discussion: Using the described method the Au loading was finely tuned ranging from 1.5 to 11.6 $\mu\text{g cm}^{-2}$ by controlling the support/Au dipping time without significant change of Au NPs mean size. No further thermal or chemical post-treatments were necessary. The Au loadings strongly influenced the photocatalytic activities and the best performance results in a three times enhancement of the bare TNTAs activity obtained with a sample containing 3.3 $\mu\text{g cm}^{-2}$ of Au. The proposed protocol demonstrated a remarkable enhancement of the photocatalytic activity of metal-supported TNTAs at room temperature without any post-deposition process.

PHOTOCATALYTIC TOLUENE DEGRADATION IN AIR AT AMBIENT CONCENTRATION BY TITANIA NANOTUBE ARRAY: INFLUENCE OF SYNTHESIS PROCESS PARAMETERS

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Introduction: Titania nanotube arrays (TNTAs) are very interesting photocatalysts. Particularly, TNTAs obtained by anodic oxidation of metallic titanium foils allow the direct synthesis of high efficiency supported photocatalytic layers without the drawbacks related to the high temperature sintering of anatase nanopowders. The synthesis of TNTAs is however a critical process

both the particle size and their morphology. The structural and morphological features have been investigated by TEM and FE-SEM coupled to EDS, N_2 adsorption-desorption, XPS as well as their ability to form hydroxyapatite in vitro. The release profiles of the therapeutic ions have been measured by inductively coupled plasma-atomic emission spectrometry. FESEM image of spray-dried mesoporous glasses shows micro-sized spherical morphology, with a size ranging between 500 nm and 5 μ m without the formation of aggregates. The parti-

cles prepared by ultra-sonication showed spheroidal nanoparticles with size of ca 100 nm, with a slight tendency to aggregate. The EDS quantitative analysis revealed element ratios very close to the theoretical ones.

Discussion: The proposed approach will allow to codify how biomaterial chemistry and topography at the macro-, micro- and nano-scale influence the multifaceted coupling process of bone resorption and formation, with special emphasis on the cell cross-talk between osteoclasts and osteoblasts.

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