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RESEARCH

Friction and Adhesion in Porous Biomaterial Structure

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A B S T R A C T

The paper presents short review of different aspects of the introduction of porosity into the bulk biomaterial and effects on different material characteristics, especially related to friction and adhesion. Nowadays, there is a great interest to investigate relations between porosity, different mechanical responses due to controlled topography and cell responses generated accordingly. Examples of current investigations of custom developed scaffolds for tissue engineering related to cell seeding and hip stem component are shown. Friction, adhesion and adhesive forces are briefly defined as related to porous material structures and the relevance of nano- and micro- level surface layers in such structures. Patterning techniques and micro-fabrication techniques for production of controlled and random porous surface layers are given. Influence of porosity on adhesion and friction is presented through several existing experimental results. However, there is still general lack of data related to many aspects of these novel porous materials and structures.

1. INTRODUCTION

Porosity and permeability are two com-monly used parameters to define porous structures. Porosity is a measure of existing voids within a dense material structure. Permeability determines the ease of fluid flow through the porous material, in case of open cell porosity and it is usually defined by Darcy's law.

Porous metallic materials have long been considered as good candidates in many areas of applications, such as vibration and sound absorption, light materials, heat transfer media, sandwich core for different panels, various membranes and during the last years as suitable biomaterial structures for design of medical implants. Depending on the end application, different properties are important and series of studies can be found accordingly, related to the changes of mechanical properties such as strength, strain, modulus, permeability, as well as damage mechanics, fatigue, creep, different reinforcements, coatings, effects of design (e.g. stent struts thickness or shape), effects of pore shape, etc. Some approaches include complex metal - polymer composites with remaining con-trolled porosity, in order to improve certain specific property. Generally, introduction of porosity in otherwise dense material, inevitably leads to decrease of strength but this is not always the case, especially for some novel complex classes of materials. Based on the shape of pores, size and orientation, a general classification of porous structures can be given as: 1) Foams (open cell and closed cell) and 2) Periodic cellular metals (prismatic, shell, and truss) [1].

It is accepted that metal foams have in-creased internal friction compared to the same dense material, as well as that the stress-strain state is developed as a very complex non-homogenous under cyclic loads, but the governing mechanisms are still un-der discussions [2].

2. POROSITY RELATED TO APPROPRIATE TISSUE RESPONSE

Initially, research in tissue engineering was related only to cell seeding and biological responses and mechanisms. Nowadays, it is a broad area comprising different sub-categories of research, where engineering of scaffolds on which the cell cultures are investigated became one promising area of future advancements. Ideally, a scaffold should closely resemble, or duplicate, the natural architecture of extracellular matrix (ECM). So far, the majority of developed scaffolds have been made of polymer materials, but very recently metallic foams have started to be studied extensively. The largest number of human cells needs something to adhere to, in order to survive and function, usually ECM and scaffolds which mimic ECM geometry and structure are of great importance. Generally, cell environment which is important for proper functioning is within a nano to micro scale. For example, collagen fiber is of 0.5 μm length and 50 nm thick approximately (0.5 - 3 μm), while cells themselves can be of different sizes, ranging from 1 μm to 100 μm or biological membranes with pores 1 - 5 μm [3]. Accordingly, 3D scaffold structures with features within a range of these scales are needed.

In recent years some of investigations have been focused on the influence of biophysical signals on properties of cell-substrate interface. Biochemical or biophysical signals comes mostly from extracellular matrix and adjacent cells. On the other hand, topography patterns and mechanical stiffness have major role in describing some cellular processes such as adhesion, migration and differentiation of various types of cells. Mechanical contact is certainly the important physical cue governing the features of cell structures. Different physical signals very often transform into biochemical signals, changing some of the cell's functions. This phenomenon is known as mechanotrunsduction and it is responsible for changing the irritability of some sensory cells. Topography of ECM varies from nanometer to micrometer scale, hence behavior of cells and sub-cells (cell morphology, organization, migration etc.) depends on a range of length scales [5].

In order to provide cell support, adhesion, proliferation and suitable mechanical properties of the scaffold, various fabrication processes has been used, such as different chemical approaches, textile technologies, particulate-leaching techniques, phase separation, electrospinning, freeze drying, and other, but all of these produce random cell sizes or interconnections [4]. Topography at micro level and with various geometries can be generated using different fabrication methods [28]. These methods mostly depend on the type of designed structure or end applications. Randomly arranged topographies can also be achieved using processes such as polishing, grinding, abrasion, plasma spraying, sandblasting, grit-blasting, etc.

For surfaces with ordered structures micro-fabrication is recognised as the most suitable technology today [3]. Micro-fabrication technologies can provide better control over cell's functions on micro and nano-meter scale and can produce controlled structures such as pits, grooves, pillars, wires and many others. In a very recent time researchers have intensively studied methods for control of cell's behavior. Ventre et al. [6] demonstrated possibility of controlling adhesion and migration of osteoblast MC3T3 cells by adjusting micro-topographic features and chemical condition of the surface. They also presented cell's trajectories and described migration of cells on untreated and oxygen treated substrates in the case of 2 μm and 5 μm size of patterns.

Two examples of custom developed scaffolding for tissue engineering are given in Figs. 1 and 2, showing structures produced by two different new additive manufacturing technologies which can be used for controlled porosity generation, but with a recognised problem to obtain fine roughness and high level of precision of the pore sizes.

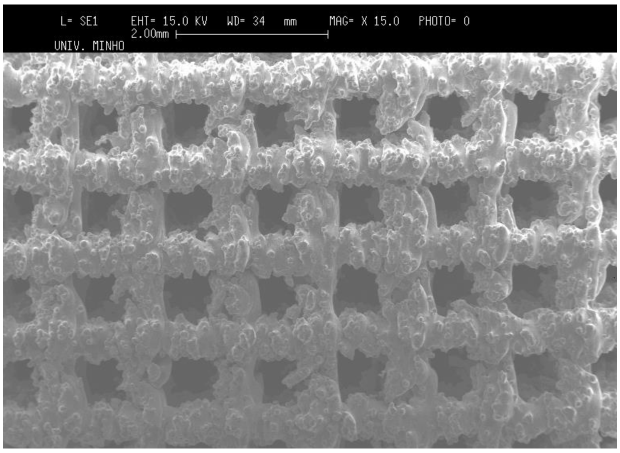

Fig. 1. SEM image of the surface of porous Ti6Al4V sample, fabricated by additive manufacturing technology, Electron beam melting (EBM), aimed for the artificial hip stem (AIMME Institute Spain).

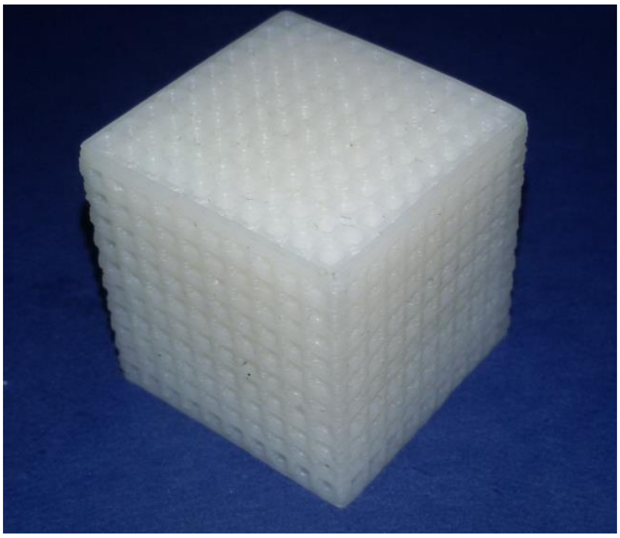

Fig. 2. Porous polymer scaffold aimed for tissue engineering - seeding of 3D cell culture, fabricated by additive manufacturing technology, Fused deposition modeling (FDM) (Faculty of Engineering Kragujevac, Serbia).

Adjustment of cell polarity is closely related with topology of artificial ECMs. For example, with respect to hepatocyte cells, it is generally accepted that three-dimensional artificial ECMs generate enhanced cell's reorganization compared to two-dimensional ones. 3D artificial environment provides similar properties as in vivo cells. According to Ranucci and Moghe [7] PLGA foams with subcellular porosity of 3 μm perform two-dimensional cellular differentiation. In contrast, in foams with improved cell-cell contact, with sub-cellular pores of 67 μm three-dimensional hepatocyte reorganization has been achieved. Better cell-cell interactions promote higher degree of cell spreading throughout the scaffold (approximate 17 - 82 μm size of voids). Travis and Horst [30] presented three-dimensional artificial nano scale fibers by using electrospinning for fabrication. These structures which contain high porosity and interconnectivity resemble the nature collagen fibers, and can be used to improve interaction between scaffolds and cells. Park et al. [8] seeded hepatocytes on glass substrates with micro-channels between each substrate. They demonstrated that these cell's cultures have better characteristics suited for liver replacements, due to the surface modification: 100 μm high micro-channels on glass substrate which provides protection from shear stress.

Different types of cells and their responses to various cues (mechanical, biological, chemical etc.) have been studied, but generally the majority of these investigations were related to their functions within the immune system. For instance, macrophages were studied through their immune defense capability and they have been used in various healing processes [9]. Recently, many researchers have started to investigate design of the scaffold material in order to interact with the living tissue, such as the research related to the characteristics of macrophage response to various topographies [10]. In this way, controlled design of the scaffold geometry and void sizes and shapes should ideally control the tissue response. Chen et al. [10] demonstrated how macrophages seeded on the polymer substrates behave when interspace between parallel gratings changes within the established range (250 nm-2 μm). They showed that larger topography (2 μm) generated lower macrophage activities, especially inflammatory response. Several authors reaffirmed dependence of the macrophage behavior on micro and nanometer topology features [11]. Several studies indicated that topography features smaller than 500 nm do not influence the cells behavior [9]. Different types of cells require different sizes for spreading, but the type of host wanted response is also the major influence. Namely, in certain cases, it is required that scaffold produce no response to implanted device and surface features of 1 - 5 mm sizes are showed to be adequate [9].

3. FRICTION, ADHESION AND ADHESIVE FORCES

Physical phenomenon known as friction represents the resistance to motion of two bodies brought into contact, and is related to the surfaces layers, or the contact zone. When the distance between surfaces is small enough forces between atoms and molecules bond those surfaces, forming junctions. Junctions' formation and growth determine adhesion level. Surface conditions, surface chemistry and degree of shear stresses influence the junction's features. It is important that cohesive forces of the weaker material are larger than interfacial bonding, otherwise material fractures. According to IUPAC definition, adhesion can be obtained if appropriate quantity of energy is delivered to the materials in contact, by using physical or chemical interactions. On the other hand, the term adhesion in biology usually refers to the behavior of cells when they are in contact with the surface.

There are generally five types of adhesions: mechanical, chemical, dispersive, electrostatic and diffusive adhesion. Mechanical adhesion is represented by the mechanical interlocking of two materials by forming bonds through filling of the surface voids of one material with another one. Chemical adhesion binds two materials together by chemical bonds (covalent, ionic or hydrogen). Electrostatic adhesion occurs when surfaces of the substrate material and some adhesive material are brought together into the electrical field which due to the difference in electrical charge holds those two surfaces together, by exchange of electrons. Diffusive adhesion or diffusive bonding is present in materials having mobile and soluble molecules, such as polymer chains which enable two polymers to bond to each other by diffusion of their polymer chains to another one. For example, one of the reasons for cross-linking of the polymers is to lower the freedom of chain motion. Another example is the sintering process that uses this adhesion mechanism - at high pressure and temperature mixed ceramic and metal powder will bond and create new material structure, by diffusion of atoms from one particle to another, to form a new particle.

Dispersive adhesion is related to the bonding mechanism between surfaces which involves van der Waals forces meaning that surfaces are in a very close proximity to each other. Van der Waals forces exist between any two molecules. These are very weak bonds and separation of surfaces to the distance larger then nanometer will likely break the adhesive forces. Throughout the literature, the term adhesion in surface science usually refers to this type of adhesion. Widely accepted method to characterize dispersive adhesion is measuring the contact angle. The lower the contact angle, the higher adhesive forces are present in the observed contact zone. It is based on the widely accepted hypothesis that low contact angles correspond to a higher surface energy. Surface energy can be defined in several ways, and one of the common approaches is to relate it to the energy needed for appearance of the surface fracture, or complete breakage of the bulk sample. An important property of material that is closely related to adhesion is surface wettability. Wettability and corrosion are very commonly brought into correlation to each other, usually to determine anticorrosion properties by observing the material wettability property. Pronounced hydrophobic properties usually indicate material which is more resistant to corrosion in aqueous environments [12]. Wettability can be evaluated using sessile drop technique in which contact angle between droplet of liquid and proper solid surface is measured.

Determining dispersive adhesion by using measuring instruments is one of the most challenging tasks, due to the reason that intermolecular forces are small and act in a very short proximity. Small change of one of the parameters (force or distance) causes a drastic change of another one. Nowadays, atomic force microscope (AFM) and scanning tunneling microscope (STM) are perhaps the most suitable instruments for these measurements [13]. Dispersive adhesion is a very broad area of research, important for many real end cases applications. Different types of removable stickers that do not use chemical adhesives are common example. However, due to the easy cleavage of surfaces when even slightly separated, still much work is needed in research in order to gain some higher degree of practical applications, but it is the area of high interest for tissue engineering field.

3.1 Micro-fabrication and surface roughness in porous materials

In general, several basic properties are referred to when describing a surface roughness, such as: waviness or micro- and nano-roughness profiles. Very basically, roughness can be defined by two parameters: height and length of surface asperities. For the surfaces with roughness in the range of 1 mm - 1 μm stylus and optical profilometers are appropriate for the surface characterization. Scanning a surface topography using focused beam of electron is a principle used in the scanning electron microscope (SEM). It has been used for higher resolution of surface deviations than optical microscopy. Atomic force microscope (AFM) reveals presence of surface asperities and valleys with space of slightly larger than nanometer and smallest height of approximately 0.1 nm. Scanning tunneling microscope (STM) is used for the most accurate measurements when surface roughness reaches resolution less than 0.1 nm in horizontal and 0.01 nm in vertical direction [14].

In order to enhance certain properties of biomaterials such as corrosion and wear resistance, biocompatibility, or surface energy, it is often necessary to modify the surface by using various methods and introduction of controlled porosity is one of them. When the surface of a load-carrying implant is attached to the bone, there is a need to achieve osseointegration, hence such surface modifications are often implemented in orthopedic and dental applications [15]. In general, surface modification methods can be classified into two basic directions, depending on the approach. First one is the top-down approach in which material layers are removed in a controlled manner, by using certain chemical or electrochemical processes. Second group refers to the bottom-up techniques or deposition of coatings, such as CVD, PVD, plasma laser deposition etc. Surface films and thick or thin coatings can significantly influence the durability of artificial adhesive joint of implant. And various surface modification techniques can be used to obtain porous surfaces, but methods of controlling the exact size and distribution of pores are at the moment state-of-the-art area of research.

Surface of the biomaterials used for medical devices are usually investigated by taking into account the following: 1) it is the first contact with the living environment, 2) chemical composition and morphology of the biomaterial surface is different compared to the bulk, precisely due to its contact with environment or another materials, 3) surface characteristics determine the tissue host response, 4) mechanical stability of the adjoining implant and the tissue in contact are essentially influenced by the surface topography, such in case of the artificial hip stem - bone cement - bone system. Accordingly, different depths of surface layers and processes occurring within those zones are investigated. Depending on the end application governed by the dominant type of interaction, several relevant thicknesses of surface layers and phenomena developing within these ranges of layers depth are observed, as shown in Table 1 [15]. Proper selection of specific materials related to the end application is also very important pre-condition for surface structure patterning, if not the major one. For example, metallic glasses can be efficiently utilized for creation of ultra-fine surface structures, due to their attractive properties based on the absence of grain boundaries [26].

Table 1. Relevant thickness of the coatings depending on the interactions [15].

Dominant physical/chemical mechanism	Relevant layer thickness
Interatomic reactions (e.g. wetting, adhesion)	100 nm
Mechanical interaction (e.g. surface hardening)	0.1-10 μm
Mass transfer/corrosion	1-100 μm

Nanoimprinting is emerging as one of the very promising techniques for low-cost fabrication of different micro- and nano-devices, such as sensors, biosensors, semiconductors, or plain moulds for creation of small devices and patterning on different materials apart from those used for moulds (e.g. metallic glass based moulds used as moulds for polymer structures). Material selection determines the applied production techniques and technologies, but even with one material several fabrication methods always exist and their selection is governed by different imposed criteria. Micro-fabrication technologies have been developed and intensively studied during the last years as promising to address recognised challenges in tissue engineering and scaffolds design. They promise to provide precise and controlled fabrication of substrates, scaffolds and topography according to pre-defined properties of the end-structure, in order to satisfy complex demands of close mimicking of living tissues. One of the main tasks in utilization of thin film materials is their fabrication or pattering into suitable micro and nano-scale range.

In order to achieve micro- and nano- features of materials, various patterning techniques have been used, some of which are further listed [16, 17]. Direct-write pattern method is based on writing patterning elements directly, after comprehensive scanning of the surface features. This scanning is a very slow process, making the technology not suitable for patterning large surfaces. It is usually used for achieving high resolution and accurate spatial distance. Depending on the type of pen used for printing patterns, there are a few subcategories of this technique: writing with a stylus, inkjets printing, writing with a quills and pins. Dip-Pen Nanolithography (DPN) is controlled by AFM probes which generate pattern directly on substrates by using inks. This method crates patterns within a few hundreds micrometer scale, as limited by the AFM resolution. Imprinting/engraving is very similar to DPN process, and uses a very hard stylus to scrape or engrave the surface. Direct-Write Photolithography changes the substrate material by using photon beam which is focused into small size spot.

Dunn [18] reported that resolution of patterned substrates is limited by optical diffraction which is approximately half of the way of wavelength of light. In order to create nano-scale features of substrates near-field scanning optical microscope (NSOM) is applied. Electron Beam Lithography (EBL) unlike Direct-Write Photolitography uses focused beam of electrons for creating pattern, achieving much higher spatial resolution. Feature size of patterns goes from 10 nm to 100 nm. Patterning with masks method is based on using the mask over the surface, by which some surface parts are exposed to the beam, or provides physical shield and protection of selected surface regions. This process is usually performed with light and generally known as photolithography. The light can affect only the areas which are not covered by mask, thus forming pre-defined pattern.

4. INFLUENCE OF POROSITY ON ADHESION AND FRICTION

The properties of the surfaces in contact with a living tissue, such as topography, density, porosity, wettability, and many other physico-chemical properties, governs the related process of adhesion and friction. The first mentioning of the importance of physical cues, such as mechanical forces on cell properties was in 1892, and was related to bone tissue response to the imposed loads. However, it was largely ignored until recently when it has started to be observed again as the novel promising molecular biology technique to influence biochemical response of cells [3]. Mechanical signals generated in such way to act through some form of shear stress, stiffness, pressure, roughness changes, controlled porosity properties or other, further induce cell response. Micro-fabrication techniques will enable greater variety of these mechanical signal generators by enabling fabrication of precisely controlled scaffolds and their further investigations in different environments.

Even with the importance of mechanical responses of porous surfaces contacts, there is a limited number of investigations related to it. Pawlak et al. [19] investigated different tribological aspects of contacts between real surfaces of bone articular cartilage and metal bearings. For instance, friction coefficient decreases with increase of wettability in case of cartilage-cartilage contact surfaces [19]. The porosity they studied were around 75 % for cartilage and 15–28 wt% for metal porous bearings made of hexagonal boron nitride (h-BN). The porosity in this case was of critical influence. The articular cartilage is porous and deformable with micro- and nano-scale thin film of phospholipid bilayers and should produce frictionless lubrication when subjected to load. The governing factor for bearing operation is the fact that they usually function under boundary lubrication. Friction coefficient of human joints is evaluated as 0.005 under natural lubrication regime [19]. Hexagonal boron nitride (h-BN) is considered to be a solid lubricant, used for frictionless contacts and self-lubricating porous sliding bearings have been used for a long time in other application fields (e.g. in industry with oil inside porosity). The volume of cartilage is decreased under pressure, while the intrinsic pressure is increased within the structure connections.

Within this study [19], the friction coefficient decreased rapidly with load increase, from 0.12 to 0.05 (range of loads 0.4 - 0.75 MPa) in case of porous Fe-Cu bearings and impregnation with h-BN micro- particles lowered friction coefficient significantly, by double and even small quantities of h-BN has great influence [19]. It also had a good influence on increasing the load carrying capacity. This study showed the dependence of the friction coefficient and stiffness on material porosity, in case of human joints and artificially made ones with porous surface. The stiffness increased with porosity increase, while the friction coefficient abruptly decreased with porosity increase, with a very sharp slope in case of artificial materials and varying of porosity over a range of 15–28.7 %) [19].

There are also several studies on modeling and prediction of cracks development within voids of such porous materials. Blum and Ovaert [20] developed 3D model of poro-viscoelastic PVA hydrogel in normal contact and sliding and showed that the maximum tensile stress is at the trailing edge of the contact and maximum compressive stress is at the leading edge of the contact, in the direction of sliding. This is important in order to find the way to lower these stresses and thus lowering the wear of the material used for articular cartilage.

There is a very limited literature on the friction and adhesion characteristics of porous structures, especially for novel materials (e.g. metal-polymer composites used as biomaterial end application) or structures fabricated by brand new micro-fabrication technologies, some listed in previous chapter. Also, there is a small number of existing theoretically developed models for micro and nano contacts of porous structures, usually related to a very specific cases and boundary conditions, such as Hertzian model or different micro- and nano-indentation models for prediction of mechanical properties. Also, there is a need to evaluate existing models, in order to improve them and there is a great need for completely new models.

A few studies investigated adhesion and friction in porous structures, but for completely other area of application apart biomedical field. Introduction of porosity into the bulk material or variation of already existing porosity greatly influence the size of the contact surface, or the surface-to-volume ratio and for small devices such as Bio-MEMS such changes of the contact surface can essentially change material properties (mechanical, tribological, optical, magnetic, electronic and other fundamental properties). Additionally, in case of small devices, surface forces, including friction and adhesion govern the surface interactions and still belong to the greater challenges in overcoming various problems of their development (e.g. seizure, uncontrolled stick-slip, corrosion, etc.). Determination of basic dependences related to material properties on the multi-scale levels represent the great challenges today, in order to obtain cost-efficient and reliable mass fabrication of small devices, from aspects of design, as well as operational functioning. There is still a need for comprehensive investigation of different material properties of already developed porous material structures on every scale, but especially on micro and nano levels, in order to determine their tribological behaviour during lifetime. And there are still many recognised issues related to the experimental tribological testing of porous structures. Conventional tribological methods in testing of standard tribological pairs and contacts are usually not valid when micro- and nano-scale ranges of surface layers are observed. As previously stated, AFM can serve as a valuable tool to make a database of different tribology relevant properties [27].

Choi et al. [21] investigated the influence of porosity variation in alumina films, by using AFM. They showed that adhesive forces very rapidly decrease with even slight porosity introduction, independently of the AFM tip radius (ranging from 380 nm to 2280 nm tip). Transition from flat surface to 0.1 of introduced porosity produced around 30 times decrease of the adhesive forces. Their results also demonstrated that after this initial decrease of adhesive forces with initial porosity introduction, further porosity increase showed almost no influence (within 0.1 - 0.5 range of porosity). On the other hand, porosity generally showed almost no influence on the adhesion. Friction coefficient abruptly increased from 0.2 (flat surface) to double value or even to 1.2 for porosity of 0.1 and tip radius of 2280 nm. Namely, this sudden increase of the friction coefficient with introduction of porosity also exhibited pronounced dependence on the applied AFM tip radius, with the largest radius producing the highest value of the friction coefficient. As for the adhesive forces, further increase of porosity produced some variations of friction coefficient value but it was non-linear and rather inconclusive and attributed to intrinsic morphological properties [21].

Mondal et al. [22] observed friction and wear behavior of aluminum syntactic foam as a porous material. They reported that micro-pores reduced nominal contact area and served as collection sites for coarser wear debris where they were partly compacted in case of higher loads, due to frictional heating. The surface thus becomes smoother and friction coefficient is lowered. In case of low loads, and especially low speeds (low frictional heating), pores acted like additional influence on wear process since they could not collect wear debris particles, thus increasing the surface roughness (by cracks originating from pores boundaries) and resulting in higher friction coefficient. Higher loads produced higher adhesion and delamination, mainly focused on pores as cracks initiation sites and weak bonding between collected wear debris and the pore inside the surface, producing sharp transitions in wear rates [22].

Porosity of biomaterials, or tissue scaffolds, and adhesion properties can be investigated with an approach to these materials as novel composites (e.g. hybrid composites), considering that open cell matrix is made of hard material and voids are filled with some polymer (soft) material, leading to complex mechanical behavior and tribological responses. Stempflé et al. [23] investigated nanotribological properties of nacre - hybrid biocomposite, consisting of two types of organic matrices and nanograin structured calcium carbonate. This is a multiscale structure with porosity ranging from 35 % to 59 %. They studied different directions of sliding and obtained various frictional dissipation and wear mechanisms. In one case they even obtaine no wear contact, due to large deformations of this double composite structure, explained precisely as a porosity influence, making a space for material to dislocate, rather than wearing [23].

In the literature, influence of porosity on the friction and wear process has rather limited data, especially in case of metal porous materials, and is often observed as the influence of the porosity originating from microstructural defects, as in the study by Gui et al. [24], who concluded that the main influence of pores is related to the cracks that may originate from pores as initiation sites producing the higher wear rates and decrease of the transition load. However, very recent papers reported strange experimental behavior of porous ceramic. Namely, porous $ZrO_2(Y_2O_3)$ ceramic fabricated by using nanocrystalline powders exhibited significant decrease of strength and effective elastic modulus, after the filling of pores with polymer gel developed for oil recovery, while in case of coarse-ceramic particles used for fabrication, the effect was opposite, as expected [25]. It is evident that tribological behavior of new material classes with controlled and pre-defined porosity needs further investigations, from many aspects.

5. CONCLUSIONS

Porous materials structures have become one of the important research topics due to many recognised benefits they can offer in the development of tissue engineering scaffolds. Further investigations are still needed to comprehensively characterize constantly emerging novel porous structures, especially related to their contact with other surfaces, as well as their influences on different phenomenon, such as cell adhesion, tissue reactions, possible degradation over time and many others. This is especially important for the number of new material classes appearing frequently during the last years. New micro- and nano-fabrication techniques are developing and have already enabled great variety of new structures at nano- and micro-level ranges of surface topography, promising advancements in many end-application areas. These techniques will continue to grow in application and level of details sophistication, also aiming for the lower cost of such processes. Comprehensive tribological and mechanical investigations, from both aspects of experimental testing and theoretical models should provide necessary foundation for further material and technology development.

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