Abstract

Environment, energy, health and transport issues are dominating our modern daily live. The complex interactions of these problems can only be solved by sustainable processing and the development of improved components. Materials science will play an important role in this new approach. One domain with specific relevance are porous ceramics and metals. It are substrates with pores sizes ranging from vacancies at the atomic level to macro pores with sizes of millimeters. There are plenty of emerging applications for porous components in different industrial sectors. Each application will specify the window of properties of the porous material.

This review is limited to inorganic porous materials which can be synthesized by dry and wet powder processing methods. In order to cope with this large application domain and window of properties, several processing and coating routes and related characterization techniques have been developed. The overview of applications for porous inorganic materials is focused on macro porous components with a designed functional coating. Examples of applications include catalytic supports, diesel particulate filters, molten metal filters, biomedical scaffolds for tissue engineering and ceramic membranes for different separation purposes.

Keywords: Porous materials, processing, applications

1. Introduction

Porous ceramics and metals materials find their applications in a very broad field, each demanding a specific window of properties. Within the last decades, several manufacture and processing routes have been developed, enabling the manufacture of materials with a wide variety of porous architectures, pore size distribution (from a few angstroms to several millimeters), interconnectivity, pore gradients or layers with different pores sizes.

This contribution presents an overview of the different manufacturing routes for porous metallic and ceramic materials produced by powder metallurgical techniques. Their production process and optimization are discussed. The complex 3-dimensional architecture of most porous materials requires a multitude of characterization techniques in order to have a full view on its properties.

The practical use of porous materials and their important role to solve the problems of our modern daily life is illustrated by several examples of applications coming from different industrial sectors.

2. Definitions

Some statements about pore sizes are needed because they are connected to definitions used in specific application sectors. According to the International Union of Pure and Applied Chemistry (IUPAC), pore sizes are classified into three categories, namely micro pores, meso pores and macro pores with pore sizes less than 2 nm, between 2 and 50 nm, and larger than 50nm respectively\(^1,2\). This nomenclature is used in the membrane scientific world. However, biomedical sciences uses micro pores for pore with micrometer diameters. Another example is the definition of ceramic foams as a class of materials comprising large voids (cells), with linear dimen-
sion approximately ranging from 10 μm to 5mm. Therefore careful interpretation of the nomenclature of porous materials as function of the application sector is necessary.

3. Overview of Applications

Table 1 classifies the main applications for porous materials as a function of their pore structure.

4. Developing a Porous Component for a Well Determined Application

The development of a porous component starts with the pore structure requirements. These include the pore size distribution, the porosity, the nature and shape of the pores (open or closed; spherical, elongated or random), interconnectivity and so on. In addition to these requirements on porous architecture, other demands have to be fulfilled as well for the porous material to be considered for a specific application: mechanical strength, specific surface area, thermal shock resistance, corrosion resistance and so on. All this questions have to be solved by defining the combination of a material and a processing route. In this contribution, as we mentioned, we limit our self to components which can be produced by powder processing. Therefore the macro porous structure will be obtained by different shaping routes followed by conventional thermal treatments as drying, calcining and sintering.

In the case of a functionalized porous material the material is comprised of two or more pore sizes, two or more materials and a mix of manufacturing routes. The functional layer can be realized by various (non-line of sight) coating techniques. For example, inorganic membranes aim at a high performance (i.e. separation factor in combination with a high gas or liquid flux), combined with mechanical, chemical and thermal stability. As such, they are composed of different layers with a gradient in pore sizes and layer thickness. Depending of the elements which has to be separated, the pore sizes goes from 5 - 10 μm to less than 2nm or even dens. The functional layer, i.e. the separation layer, will be obtained by a sol gel coating technique for e.g. nano filtration membranes.

For applications as separation and biomedical porous scaffold for tissue engineering, the interconnectivity of the pores is very important but this is not the case for materials which are used, as isolation, impact material, or kill furniture. The most common diesel particle filter (DPF) material is a honeycomb structure with openings in the range of mm, porous walls with pore sizes ranging from a few microns to ~20 micron, onto which a meso porous wash coat is deposited for the catalytic combustion of the collected soot. Other catalytic supports are zeolite coatings (Fig. 1) produced by hydrothermal treatment. With these examples in mind it is clear that the synthesis of functional porous materials can be divided into manufacturing routes to obtain (macro) porous components and in techniques to coat these substrates

| Pore structure                  | Application                                      |
|--------------------------------|--------------------------------------------------|
| Micro-and meso porous materials| Desiccant materials                              |
|                                 | Sensors and actuators                             |
|                                 | Thermal isolation                                |
|                                 | Catalyst (support)                               |
|                                 | Drug delivery systems, coatings, carriers        |
| Foam- and honeycomb structures  | Exhaust gas filter                               |
|                                 | Diesel particle filters                          |
|                                 | Filters for molten metal                         |
|                                 | Porous Electrodes of fuel cells                  |
|                                 | Porous Burners                                   |
|                                 | Catalytic substrates                             |
|                                 | Biomedical porous scaffolds for tissue engineering|
|                                 | Impact and acoustic materials                    |
|                                 | Kiln furniture                                   |
|                                 | Lightweight sandwich structures                  |
| Multilayer materials            | Ultra filtration membranes                       |
|                                 | Nanofiltration membranes                         |
|                                 | Gas separation membranes                         |
|                                 | Zeolite membranes                                |
|                                 | Pervaporation membranes                          |
|                                 | Dens membranes (oxygen or proton conductors)     |

Table 1 Classification of applications by the type of pore structure
with a porous layer.

5. Synthesis Routes

Table 2 gives such overview of manufacturing routes to produce the macro porous structures and the coating techniques to functionalize their surface.

Table 3 shows a summary of techniques which are conventionally used to determine the pore size distribution, the interconnectivity and the specific surface area of porous materials. Although useful as such, no single technique is able to cover the wide range of characteristics needed to describe the porous material. The complex 3 dimensional architecture of this class of materials necessitate a range of analysis tools to get a view on the structure of the porous network.

These analysis techniques can be classified based on
- the type of pores that are measured (open, closed, or blind pores)
- the pore size range (from 1 nm – millimeters),
- the dimensional determination (2D versus 3D),
- the (non) destructive nature of the measurement, or
- the amount of pores typically taken into account.

Apart from the information on the pore size distribution, other information on the porous architecture can be obtained by the combination of different techniques (shape of pores, pore volume, surface area or strut thickness).

Also some specific application-oriented testing

**Table 2** Synthesis techniques for macro substrates and coatings for the functionalization

| Manufacture of macroporous support | Coating techniques |
|-----------------------------------|-------------------|
| Replication of                    | Sol gel           |
| - Polymer (e.g. PU foam)          | - Colloidal       |
| - Natural materials (e.g. wood)   | - Polymeric       |
| Direct foaming                    | Wash coat         |
| - Preceramic polymers             |                   |
| - Gel casting                     |                   |
| Use of pore formers               | Vapor Deposition  |
| Freeze casting                    | - Chemical        |
| Extrusion (e.g. honeycombs)       | - Physical        |
| Gas introduction                  | Precipitation     |
| Hollow building blocks            | - Hydrothermal    |
| additive manufacturing            | - Biomimetic      |
| - Selective Laser Melting         |                  |
| - Selective Laser Sintering       |                  |
| - E-beam melting                  |                  |
| - Stereolithography               |                  |
| - Fused Deposition Modelling      |                  |
| - 3D Printing                     |                  |
| - 3D FD                           |                  |
| Partial sintering                 | Plasma coatings   |
| - Partial                         | - Vacuum          |
| - Large grains                    | - Atmospheric     |
| Connected fiber, rod, other shapes|                  |
methods are frequently performed (e.g. cut-off measurements for membrane development). A full description of these characterization techniques is outside the scope of this publication. For more details, reference is made to some review publications.

In addition to the analysis of their pore structure other properties related to their application have also to be tested: mechanical properties, thermal conductivity, dielectric constant, specific surface area, chemical resistance, hardness/wear, tortuosity of the flow path and so on.

6. Designing Porous Materials

Recently a new trend towards the design of porous materials with a specific periodic architecture and a highly reproducible porous architecture has emerged. Additive manufacturing routes, already many years used in the shaping of polymeric materials, have been developed to shape metallic and ceramics materials. The class of additive manufacturing routes varies widely with regard to the nature of the starting material (liquid, solid or powder based) and the solidification process (laser induced melting or sintering, e-beam melting). Related to the rapid manufacturing process, also material properties, speed of building, cost, volume, surface finish and complexity of the part varies widely.

The progress of rapid manufacturing techniques for ceramic materials has somewhat been lacking behind. One possibility to overcome this problem is to combine additive manufacturing for the shaping of the ceramic powder with the conventional thermal treatments. One technologies which uses such an approach is 3 Dimensional Fiber Deposition (3DFD). This manufacturing technique enables the production of periodic porous ceramics and metals.

3D Fiber Deposition (also called direct-writing or robocasting) comprises the extrusion of a highly viscous paste loaded with metallic or ceramic particles through a thin nozzle. By computer controlled movement in x, y and z-direction, the porous architecture is built layer-by-layer. After sintering, a highly reproducible and periodic porous structure is obtained. The process variables include a.o. the nozzle opening (−fiber thickness), the type of nozzle (−fiber shape), the interfiber distance (−pore size) and the stacking of the layers (−porous architecture, see Fig. 2). As such, flow path for either gas or liquids can carefully be controlled and optimized with regard to pressure drop or contact time with the active coating. Moreover, by a patent-pending technology, the microporosity and surface roughness of the fibers can be controlled.

The custom-built equipment for 3DFD consists of a paste reservoir with nozzle, mounted on a CNC machine or on an x, y, z-table. The input for the equipment is a CAD design. This design is sliced (by a customized software) in order to generate the beginning and end positions of each fiber. These positions serve as coordinates for the x,y,z-table.

Typical dimensions of parts are 40 mm by 40 mm and a height of approximately 35 mm, with fiber thickness from $300 \mu m$ to $900 \mu m$. Multiple nozzles can be mounted onto the equipment in order to speed the production of similar pieces.

The keys for this type of technology are the composition of the paste that is extruded and the production parameters: a careful balance between a high viscosity to be able to bridge the underlying gaps, the drying behavior, the speed of deposition and the extrusion force. Both ceramic and metallic parts have
been manufactured (see examples in Fig. 4).

7. Some Applications in Detail

7.1. Multilayer ceramic membranes

Due to their inherent cost, ceramic membranes will only be used in situations where polymeric cannot meet the requirements: in the case where a high chemical, thermal or mechanical resistance is needed. The optimization of a membrane focuses in the first place on the combination of a high flux and a high separation factor. This can only be obtained by a multilayer component built up by layers with a gradient in pore sizes and layer thickness: in the sense that how smaller the pores how smaller will be the layer (Fig. 5).

Tubes, hollow fibers and plates are the most common support shapes. The macro porous substrates with pores of 5 to 10 μm and a porosity of 30 to 50% offer the mechanical strength of the membrane. Mostly, the tubes are produced by extrusion, the hollow fibers by a spinning technique and the plates by pressing or tape casting. By slip casting with powders suspensions with different particles sizes different layer were built up on the macro porous layers till a pore size of about 0.1 to 0.2 μm. A layer with such a pores size can support the thin separation layers which are mostly produced by a sol gel process. A first sol layer(s) is prepared following the colloidal route. After hydrolyses and condensation reactions of the precursor, metal salt or metal-organic precursor with water, a colloidal sol is obtained. After coating, calcining and sintering, a mesoporous layer is obtained. For the polymeric route, the precursors are dissolved in organic solvent -with or without a limit amount of water-. This results in a sol with fractals, and finally with a micro porous (<2nm) top layer. The top layer can also be produced by a hydrothermal treatment as is the case of zeolite membranes. Further surface modifications e. g. by grafting can be performed in function of the specific application e.g. separation of solvents. In a membrane reactor, the catalytic reaction can be enhanced by the separation function of the membrane.

Another category are the dense membranes, the oxygen and proton conductors (Fig. 6). Vacancies are here the main transport mechanism of O- or H-ions in elevated temperature ranges. Nevertheless, a membrane with modified surface has a better performance because the total mechanism of transport exists of three steps: the adsorption of O₂ or H₂ at one side of the membrane, the hopping of these
atoms through the dense structure with the help of vacancies and the desorption at the other side of the membrane. Surface modification can be obtained by another structure, by roughness, with a certain micro porosity or by a layer with a catalyst to enhance this adsorption- or desorption step.

7.2 Porous scaffolds for tissue engineering

Although the large regenerative capacity of bone is generally recognized, large bone defects due to trauma or tumors are still a big challenge for surgeons, as no spontaneous healing of the bone occurs. Bone tissue engineering is a new approach to these
kind of problems, whereby a porous scaffold is used, onto which stem cells can be seeded, and coated with proteins and other biologically active species.

As the exact biological response of cells in a porous scaffold remains unclear until now, the synthesis of the porous scaffolds is maybe the easiest step in this complex procedure. However, the criteria posed onto a candidate scaffold material is long. First of all, a wide range of biocompatible materials is available. Calcium phosphates (CaP) and bioglasses will be absorbed by the body after some time but they are too brittle to be used as a load bearing material. Metallic materials, mostly Ti and Ti-alloys, offer sufficient mechanical properties, are ductile and the small layer of TiO₂ onto the surface impedes a fast dissolution of metal ions. Mg and its alloys are reported to combine load bearing properties and bio absorbability. Untill now, its high reactivity during synthesis, high corrosion rate and biological interactions need further study before clinical implementation.

For a good integration in the bone structure, the properties of the scaffold (E-modulus, compression strength, porosity, pore sizes, ductility) are to be tuned to the properties of the bone (Table 4). The optimal pore size is considered to be in the range of 100 to 500 μm, to allow cell seeding, to stimulate the vascularisation of the scaffold, and the transport of nutrients to the cells and the removal of waste products. Depending on the material and the porosity, the E-modulus and mechanical strength can tuned to avoid stress shielding, a phenomenon which is known to cause implant loosening due to a mismatch in mechanical properties between surrounding bone and implant.

In the case of Ti and Ti alloys, ductility is function of the quantity of the interstitial impurities (O, N, H, and C) of the metal. These impurities can already be present in the powder or are introduced during the processing or the thermal treatments of the component.

Apart from structural and mechanical requirements posed onto the scaffold, its surface should enhance the bioactivity. Cell attachment and the adhesion of a bioactive coating are improved by these microporosity or surface roughness. Examples of porous structures used as biomedical scaffold are given in Fig. 7 a and b, structures respectively produced by gel casting and by the 3DFD technology.

A further development in scaffold materials are the bioactive coatings. Fig. 8 shows a CaP coating prepared on a Ti scaffold by a precipitation technique. Such porous CaP structures can be used as drug

| Property                        | Range            |
|---------------------------------|------------------|
| porosity                        | >65%             |
| Interconnectivity of the pores   | >90%             |
| Pore size d                     | 50<d<500 μm      |
| Modulus                         | <3 GPa           |
| Compression strength            | >40 MPa          |
| Ductility                       | >10%             |

Table 4  Some macro porous properties of porous Ti - en Ti- alloys scaffolds

Fig. 7  Porous architecture of Ti6Al4V scaffold produced by (a) gel casting and (b) 3DFD.
delivery system to overcome bone infections. **Fig. 9** shows the antibiotic release curve of a drug delivery system existing of porous CaP fibers coated with a barrier of poly lactic acid (PLA) coatings with different layer thickness.

### 7.3 Removal of intermetallic particles for the purification of Aluminum alloys \(^{45-48}\)

In the framework of a collaborative program on recycling (Growth program of the EC, GIRD-CT-2002-00728), the purification of molten aluminum has been studied. The approach was to capture elements like Fe, Mn, and Si in intermetallic particles, which subsequently have to be removed. One of the different techniques which were used here was filtration. The filtration process is well known for the removal of inclusions from the melt, but due to the formation of rather long intermetallic fiber-like precipitates (**Fig. 10**), clogging of the filter occurred.

Forced filtration (or inverse filtration) was demonstrated as a solution to overcome the clogging on lab scale. After the precipitation of the intermetallics by cooling down the melt to about 600°C, a 40 ppi foam was pressed down through the vessel, compacting the solid intermetallics to the bottom. By repeating this procedure at lower temperatures, by staying at a temperature where liquid and solid phases are present, more intermetallics can be concentrated as a kind of residue at the under part of the crucible. For capturing smaller intermetallic particles an active filter can be a solution. Such filter consists for instance of a foam filter coated with a salt (e.g. NaBr in **Fig. 11 a, b**), which becomes viscous and sticky at the melting temperature of the Al metal.

### 7.4. Diesel particulate filter (DPF) \(^{49-59}\)

Another example of a functionalized porous material is DPF. This kind of filters have to remove the carcinogenic soot particles from the outlet of diesel engines. Apart from a high filtration efficiency, other important requirements include a low pressure drop over the filter material (due to the fuel penalty in-
To design a good filter, a low pressure drop is a prerequisite. Therefore, large pores, high interconnectivity and a high filter surface are important. To combine this high flux with a sufficient separation factor, a thin layer with smaller pores will be needed. Finally, to burn off the soot particles on an economic way, in this case at a relative low temperature, a mesoporous catalyst has to be foreseen.

Several porous architectures have been suggested for use as DPF: ceramic or metallic foams, connected fibers or honeycombs (Fig. 12). The most of the conventional DPF for cars are based on a honeycomb filter which is obtained by an extrusion process, due to its large filtration capacity and efficiency. The channels with openings in the order of millimeters create the large filtration surface. The thin walls of the honeycomb with pores around 15 μm, the tortuosity of the pore structure and the soot cake formation give the filter an efficiency of more than 99%.

Regeneration of the filter is most commonly performed by the catalytic combustion of the collected soot. Therefore, the honeycomb walls are coated with a wash coat existing of a mesoporous structure with a high specific surface as (e.g. Y-AL₂O₃), containing a few percent of precious metals as Pt and/or Pd.

Of course this filter has to fulfill other requirements as mechanical strength, thermo shock resistance, thermal conductivity, thermal capacity, sufficient high melting point, corrosion resistance, and reasonable price. Because all these requirements could not be fulfilled by one material, different variations are on the market: cordierite, SiC and Al₂TiO₅. A recent variation is the honeycomb structure with an arcicular mullite (developed by Dow Co.) based on a process with a fluor exchange. The development
focuses on the mixing of this arcicular mullite structure with common cordierite to improve the other application requirements of a DPF.

8. Summary

This contribution aims to give an overview on the processing and characterization techniques which play a role in the design of inorganic materials for many applications. To illustrate this issue different examples of applications with porous components are discussed in detail. It is clear that porous structures can play a role in solving environment, energy, health and transport issues by their unique combination of porous architecture and materials properties.

Acknowledgement

The authors want to express their appreciation to all the people of the Material Technology group of VITO who has helped in one or another way to realize this research.

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Fig. 12 Honeycomb wall filter (a) and ceramic foam (b) both used as substrates for a soot filter for diesel engines.
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Jan Luyten (born 1951) received his M.S. in metallurgical engineering in 1974 and his Ph.D. in applied sciences in 1979 from the Catholic University of Leuven (KUL, Belgium). After a 1-year post-doc, he worked at Union Miniere (1980), a multinational specialized in mining and refraction ore. From 1981 till 1988, he was a lecturer at the Technical School for Industrial Engineers Groep T in Leuven. In 1988 he joined the Nuclear Research Centre (SCK) in Mol (Belgium), part of which organization was split of in 1991 as the Flemish Institute of Technological Research (VITO). Both at the SCK and now at VITO, he has been working in the field of processing of ceramics, covering areas of chemical sensors and their construction, solid state (mixed) conductors, ceramic membranes, ceramic foams and structural ceramics in general. He is a member of the Belgian, European and American Ceramic Society, and published over 80 articles in the field of ceramic materials and their application. He contributes to conferences, proceedings, and scientific journals both as an author and reviewer. He has participated in several EC-programs.

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