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Chapter 15
Characterization and Spectroscopic Applications of Metal Foams From New Lightweight Materials

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ABSTRACT

Lightweight materials such as metallic foams possess good mechanical, chemical, and physical properties, which make them suitable for a wide range of functional and structural applications. Metal foams have recently gained substantial interest in both industry and academia due to their low cost, thermal conductivity, high working temperature, vibration damping, specific mechanical properties, energy absorption, and heat resistance. The use of metal foams on a large scale and successful applications depend on a detailed understanding of their characteristic properties. Metallic foams are characterized by the morphology of the porous cells (size and shape, open or closed, macro and micro), pore topology, relative density, properties of the pore wall, and the degree of anisotropy. This contribution focuses on x-ray diffraction, Fourier transform infrared (FT-IR), and Raman spectroscopic applications used for the characterization of metal foam, and also a brief of the most important applications, including a significant number of examples given.

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GENERAL INTRODUCTION TO METAL FOAMS

Metal foams were first produced in the 1950s, and with the developments in recent years, they have attracted attention among the light-weight materials. Metallic foams have the advantages of a unique combination of mechanical properties like lower weight, high energy absorbing capacity, high stiffness, and high damping capacity. For these reasons, they are becoming the most promising class of materials in the industrial and scientific areas. Metallic foams have a role in many areas of our lives day by day. Metal foams are being used in many industrial applications such as light weight structural applications, biomedical applications, filters, electrodes, heat exchangers, home appliances etc. In addition, metallic foams are also used at high temperatures. They are used in areas such as the transpiration cooled rocket nozzles, a cooling system in the combustion chamber of gas and steam turbines, and as heat shielding for aircraft exhaust. (Ashby, 2001; Bauer et al., 2013; Qin et al., 2016; Liu and Chen, 2014; Banhart et al., 2019; Sutygina et al., 2020). Metal foams are a structure consisting of air and metal, and they can be obtained by many production methods with different pore ratio. The advancement of technology and materials science has enabled the discovery of many metallic foam production methods and the production of metallic foams with different properties. Photograph of metal foams with different porosity produced in the laboratory is given in Figure 1. This figure is taken from the PhD thesis of Nuray Beköz Üllen, one of the authors of the chapter (Beköz, 2011).

Figure 1. Steel foam specimens having different porosities (Beköz, 2011)



In fact, it is desired that the material does not contain pores in many production methods such as casting and welding, but the advantages of the pore structure are used in these materials (Degischer and Kristz, 2010). Porous structure provides thermal and sound insulation as well as impact absorption in addition to light-weight construction (Banhart, 2003). Usage areas and properties of porous metals depend on the pore type. Foam structures can be defined as several types which includes pore type: open foams (pores connected to each other so that matter can pass through them), closed foams (isolated closed pores filled with any gas), and partially open foams (Beköz, 2011; Patel et al., 2018). Metal foams with closed pores are used in light structures thanks to their high hardness and low-density properties. Due to their deformation capacity when undergoing constant stress, they come to the fore as ideal energy absorbers in packaging applications and especially in the automobile industry. They can also be used in acoustic damping applications. On the other hand, open cell foams offer a wide variety of applications, e.g. as a

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transmission and filtering applications, heat exchanger etc. Open cell foams have a high percentage of interconnected pores and high surface area. These features make it charming for use as heat absorbers in electronic appliances, and in transpiration cooling (Aly, 2004; Dukhan, 2013). The interconnected porosity is specific point for parts like filters, bearings, diaphragms, membranes. In addition, they also have importance in biomedical applications (Aly; 2004; Ashby et al., 2001). The properties of metallic foams vary according to the porosity, shape, size and the connection types of the pores with each other and create much different potential for usage areas (Bekoz and Oktay, 2013; Tunçer, 2006; Hakamada et al., 2007; Alhusseny and Nasser, 2018).

All aforesaid types of foams have different applications. In lightweight metals foam category Ti, Al, Ni, Fe, Zn, Cu and Mg are widely used. Metallic foams, due to having extremely low densities in comparison to solid structures, possess high specific bending strength and stiffness, they easy recycling, having non-toxic outstanding properties for sound absorption, vibration, and impact energy (Rajak et al., 2014). In this respect, characterization studies are important in terms of examining the pore properties and determining their effects on metallic foam. Therefore, this chapter includes the methods used in the characterization of metallic foams from past to present.

Characterization Methods of Metal Foams

As a result of the advancement of technology and studies, characterization methods are developed day by day. The successful application of foams depends not only upon inventing new techniques to produce foams that are cheap and of improved quality, but also upon the development of a detailed understanding of all their properties. The physical and chemical properties of metal foams can be characterized in many ways. To characterize foam structures

and to obtain useful information about their microstructure, morphology, mechanical and other properties, various techniques are used. The various methods for the characterization of metal foams will be briefly reviewed in this section.

DETERMINATION OF PORE STRUCTURE

It is known that foam properties strongly depend on the pore structure. Pore structure is a general term. What is meant by this term is pore size, shape, connections with each other and their environment, distributions of the pores and pore ratio. The structural features of foam influence its specific surface area, fluid flow resistance, and electromagnetic transmission or absorption. Various equations were suggested for the characterization and modeling of the relationship between the structure and the various properties of the foam. (Korner and Singer, 2000; Ashby et al., 2001). Metallic foams, having a wide dispersion of cell size and cell shape, are highly disordered. The terms used to characterize porous structure are given in Table 1. The characteristics of a foam material given in Table 1 are important and determine the wide variety of material properties that can be obtained from that foam. (Andure et. al., 2012).

While the pore wall cross-sectional shape and actual size are controlled by relative density, the number and nominal size of foam pore walls are controlled by pore size. The features of the base metal determine all the physical properties of the produced foam, such as melting temperature, thermal expansion coefficient, specific heat, strut hardness, chemical reactivity and oxidation limits. The metal selected to be foamed together with relative density affects the mechanical properties of the foam such

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Table 1. Definition of terms used to characterize porous structure (Dukhan, 2013)

Density	**True density:** Density of a porous material except pores and inter-particle cavities, just dense part	**Apparent density:** Density of a porous material with closed and inaccessible pores	**Bulk density:** Density of a porous material including pores and inter-particle cavities
Pore Volume	Volume of the pores of (Vp)		
Pore Size	The length between two opposite walls of the pore		
Porosity	The apparent volume (V) of the particle or powder per the ratio of the total pore volume (Vp)		
Surface Area	The ratio of accessible area of solid surface to the unit mass of material		

as elastic modulus, electrical and thermal conductivity, crush strength (Aly, 2004). The mechanisms of linear elasticity depend on whether metallic foam with open (interconnected) or closed (connected) pore structure. Cell edge bending is an effective deformation mechanism in open porous metal foams. In closed-cell metal foams, deformation begins with plastic bending of the cell walls and stretching of the cell surfaces when a compression load greater than the collapsing strength of the foam is applied (Aly, 2004; Rajak and Gupta, 2020).

It is important to characterize the densities of the utilised foams, since density is the factor that significantly affects the mechanical properties of metallic foams (Aly, 2004; Degischer and Kriszt, 2010). The term relative density is used instead of density of the metallic foam. Relative density is the ratio of the densities of the metallic foam and the metal from which the metallic foam is made. Therefore, also this property has been examined in this section as it depends on the amount of pore. As mentioned before, the pore structure is the most important factor determining the usage area and properties of metallic foam. The nominal pore wall length, pore diameter and cross-sectional size are directly affected by the pore size of the metallic foam. These microstructural properties of the foam also affect different properties such as spesific surface area, electromagnetic transmission or absorption, fluid flow properties. It is required to optimize the pore size. Although small porous structures are better in terms of strength, their permeability is poor. For example, pores smaller than 200 microns and larger than 600 microns are not desired in biomaterial applications (Xue et al., 2007). The pore shape varies according to the production method and can be in various forms. It is important because it affects the mechanical properties and causes anisotropy (Harders et al., 2005; Borovinšek et al., 2016).

Density measurements of a porous metal can basically be made using Archimedes' Principle. The buoyancy in a liquid of known density is determined. If the structure does not have a closed surface, it is recommended to cover it with a polymer surface in order not to fill the pores (Banhart, 2001; Esen and Bor, 2011). In addition to this method, the Eddy current method is also used for relative density measurement. A magnetic field is created around a coil by passing alternating current, and this magnetic field changes over time. It creates a second magnetic field on the electrically conductive sample. The interaction between the two magnetic fields changes, causing a change in impedance on the coil. By measuring this impedance change, the properties of the material can be revealed (Banhart, 2001; Bor et al., 2007; Ma and Peyton, 2006).

Determination of pore size distributions or shape analysis of the pores can perform using commercial image analysis programs. Individual pores in the preparation plane can be identified with these programs. Preparation of materials should be carried out very carefully. In order to distinguish between cell membranes and inside cells, they must appear in different brightness. One of the ways to prepare is by

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embedding the porous material in a black resin and then polishing the surface. Manual correction may be required in order for the program to define the individual pores and calculate pore size distributions.

Many methods can be preferred for determination of other pore properties. Non-destructive methods such as porosimetry and permeametry measurement, as well as X-Ray radiography/ tomography, neutron small angle scattering and ultrasound imaging methods can be used (Banhart, 2001; Patel et al., 2018). The use of X-ray measurements explained in section 1.9. In addition, eddy current method is also used for pore size determination. Porosimetry and permeametry measurement methods are used to measure the specific surface areas, distribution of pore size and channel dimensions of metallic foams with open cell structure. Measurements are performed using methods as mercury porosimetry, gas absorption techniques, permeametry and bubble point method (Banhart, 2001).

Mercury Porosimetry: This method is based on the penetration of mercury under the effect of pressure into the pore volume and extrusion. The pressure data and penetrated volume are related to the size of the pores. It is difficult to detect pores larger than a few hundred microns with this method (Banhart, 2001; Dukhan, 2013).

Gas Absorption Techniques (BET): With this method, the total internal surface of a porous material can be determined with very high accuracy. Even very tiny pores of atomic size can be detected (Banhart, 2001; Ashby; 2001).

Permeametry: The basis of this method is to examine the flow of a gas or liquid (m^3/s) per unit time through a unit volume of a porous medium. A linear relationship is found for very slow flow by measuring the pressure drop at a foam column of height as a function of the average fluid velocity. There is a quadratic dependence for higher flow rates when the energy losses due to the inertia of the flowing fluid increase to significant degrees. If the viscosity of the fluid is known, the permeability can be derived as a coefficient of friction. Considering that the internal surface area is related to the friction of the pore volume, an estimate for the specific surface area can be obtained (Banhart, 2001; Smorygo et al., 2011).

Bubble Point Method: In this method, first the pore volume is filled with a liquid and then an increased external pressure is applied. When a certain pressure value is reached, the capillary force of the liquid is exceeded. The liquid is blown out of the sample giving rise to a resistance drop. Finally, the smaller pores are emptied until the sample is dry. Some information about the largest open pore of the specimen and the pore size distribution can be determined by comparing the flow rate as a function of pressure drop to the corresponding curve of a dry specimen (Banhart, 2001; Park, 2016).

Mechanical Testing

Mechanical testing of metallic foams to be used for any structural application is a prerequisite. Mechanical data and databases should be created to evaluate specific applications or to model porous metals or containing components in computer-aided modeling. In principle, the characterization methods are the similar as for dense materials. However, it is necessary to make adjustments to some test procedures, taking into account the different structure of porous materials. This difference is related to statistics. For example, when metallic foam made of a particular alloy is tested, the mechanical properties obtained as result of examinining several samples with the same foam density will show a much greater scatter than the test results of the dense state. The reason for this is the difference in some properties such as the mass distribution, the distribution of pores, the heterogeneous microstructure, etc. which are “hidden parameters” that make difference in the porous material samples having the same total density.For this reason, more samples may be required to obtain more accurate results in testing porous structures

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compared to the test standards required for non-porous materials. Also, the length scale of macroscopic heterogeneities in porous materials is sometimes in the millimeter range. This may require an enlargement of specimen dimensions in mechanical tests to current standards. Various mechanical tests can be labelled with one of the following properties (Banhart, 2001; Yang et al., 2017):

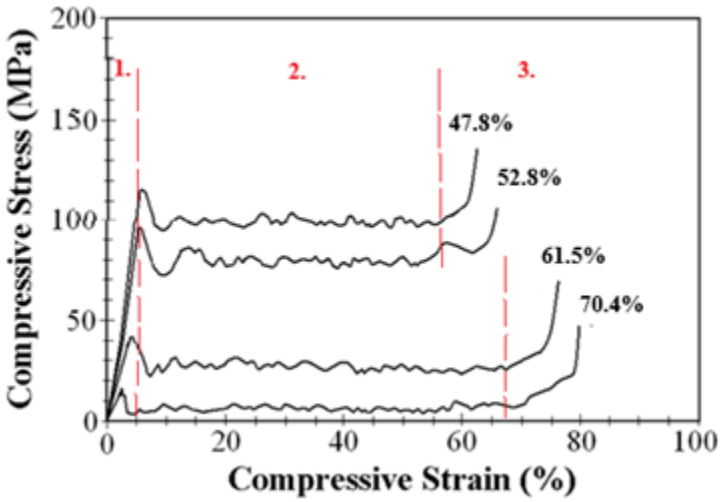
Mode of Loading: tension, compression, shear, torsion and bending

Type of Applied Stress: uniaxial, biaxial, multiaxial, quasi-static and hydrostatic

Time Dependence of Load: slowly increasing dynamic, cyclic and constant

Quasi-static and uniaxial compression tests are frequently used for characterization of porous metals. These measurements enable the mechanical stability of the porous metal to be defined. In the publication of one of the authors of this chapter (Bekoz and Oktay 2013), the effect of porosity and pore size parameters on the mechanical properties of steel foam samples was investigated. The strain-stress curve of the compression test, that applied to the steel foam samples reported in this study was given in Figure 2. Along with these tests, fracture toughness measurements on notched specimens and indentation tests with various indentor shapes are also applied porous samples. There are also tests for foam sandwich panels, such as the drum peel test (ASTM D1781), which describe the bond between the core and the face sheets. Mechanical deformation can be monitored by recording acoustic emissions with the breaking cell walls, which is a technical measurement in addition to ordinary tests (Banhart, 2001; Yang et al., 2017).

Figure 2. Compressive stress-strain curves of the steel foams having different porosities (Bekoz and Oktay, 2013)



As shown in Figure 2, the stress-strain curve consists of three regions. These are the linear elastic zone (1), plateau zone (2) and densification zone (3). Linear elastic zone ends at maximum compressive strength value, after which a long plateau stress is observed where deformation occurs at almost constant stress (Aly 2004; Beköz, 2011; Dukhan, 2013). This long plateau is formed by the collapse of the cell walls and the struts in the cellular structure. Deformed pores form deformation bands in a direction perpendicular to the deformation direction (Aly, 2004). In the case of brittle materials, compression strength is defined as the first maximum stress value in the compressive stress-strain curve. However, in

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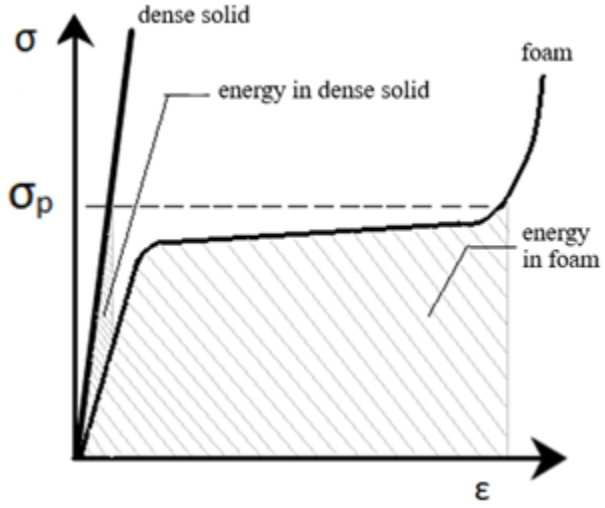
the case of ductile materials, the compression strength of the material is lies at the point where the slope of stress-strain curve equals zero for the first time. However, there are other ductile materials that pass directly into the plateau region without showing any maximum point (Ashby et al., 2001; Aly, 2004). Florek et al. (2010), observed these differences in brittle and ductile materials with stress-strain curves obtained as a result of the uniaxial compression test of metallic foams made of different aluminum alloys. When this happens, the intersection point of the tangent drawn to the linear elastic regime and drawn to the plateau is defined as the compressive strength of the material (Ashby et al., 2001). The stress-strain curve ends with a zone that is the stress increases quickly. This zone is known as the densification regime (Aly, 2004). The areas with large pores begin to deform at the beginning of the compression test. By increasing the compression, other pores in the deformed areas are deformed. When the compression deformation ends, it is observed that deformation bands with a thickness equal to the pore diameter are formed within the fractured areas (Aly, 2004; Haag et. al., 2003).

Porous metals have great potential for applications that require absorption of crash energy. The efficiency of the energy absorbing metals in the parts used at the points where the crash energy is desired to be absorbed is significantly affected by the stress-strain behavior under uniaxial compression. The characteristic shape of the compressive stress-strain curves applied to porous metal with an almost constant stress allows large amounts of energy to be absorbed at a relatively low stress level. A comparison of the energy absorption behavior of a fully dense elastic solid with metallic foam was given in Figure 3, taken from the PhD thesis of Nuray Beköz Üllen, one of the authors of the chapter (Beköz, 2011). Metallic foam can absorb more energy than its dense form under a specified peak stress level (Claar et al., 2000; Beköz, 2011).

Figure 3. Comparison of energy absorbtion behavior via compressive deformation for foam and dense solid materials (Beköz, 2011).



An immense range of signals can be obtained with Transmission Electron Microscope that infer diffraction patterns, images and many different types of spectra from a very small specimen area (Patel et al., 2018; Banhart, 2013). There are also different TEM types such as HRTEMs, STEMs, IVEMs, HVEMs,

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and AEMs. In addition, the X-ray diffraction method can be used to determine the crystal structure of all components in the specimen (Raj and Daniel; 2007). Various tests are performed on metallic foams to determine mechanical properties like compressive and tensile strength, flexural strength, hardness etc. (Bekoz and Oktay, 2012; Bekoz and Oktay, 2014; Ashby et al., 2001; Smith et al., 2012; Dukhan, 2013; Degischer and Kriszt, 2010). The tests mentioned include compression, tensile, shear, multi-axial, creep, fatigue, hardness, surface strain mapping tests. In addition to these tests, the non-destructive resonance frequency method has also been proposed in literature for tensile test and damping measurements (Patel et al., 2018).

Corrosion Testing

Metallic foams have less corrosion resistance due to the pores in their structure, as contact with air is also in the interior and have greater surface area. There is no standard corrosion test method used for metallic foams. These tests can be performed depending on the area of use (Banhart, 2001). The corrosion behavior of the material can be characterized by methods such as anodic polarisation measurements (Rossi et al., 2017), loss weight tests and electrochemical tests (Zhang et al., 2009; Ajinola, 2016; Palaniswamy et al., 2020).

Microstructural Characterization

Microstructure characterization is important for achieving the desired properties in metallic foams. As the density of metallic foams increases, their mechanical properties increase due to the increase in the pore walls. However, instead of increasing the density of a product that promises light weight, it is desirable to improve its mechanical properties by another method. Improving the mechanical properties of metallic foams is possible by optimizing the pore properties and the microstructure (Degischer and Kristz, 2010; Bekoz and Oktay, 2012; Taherishargh et al., 2017). Therefore, characterization of the microstructure is required.

Microstructural analysis of metallic foams can be determined by optical image analysis as a destructive method (Banhart, 2001; Patel et al., 2018). In the optical microscopy method, the pore microstructure of the specimen can be observed after the preparation processes. The samples are ground and polished using the normal grinding and polishing techniques. It is very important to preserve the porous structure of the material, during the preparation of the specimens for the microstructural examination. Various methods are applied for this. Resin method is one of the easiest and most frequently applied methods. The samples are infiltrated in vacuum by a low viscosity resin. The resin is pour onto the samples and on admitting the air to the chamber; it is impregnated into the small pores. Then, a sample is taken from the foam and ground and polished (Beköz, 2011). In Figure 4, which was taken from the article of Nuray Beköz Üllen, one of the chapter authors, the optical microscope image of the samples was given in the study of the steel foams produced by space holder-water leaching technique in powder metallurgy (Bekoz and Oktay 2014).

Optical microscope is not sufficient in parts such as analysis of grain structures and surface. Scanning electron microscopy (SEM) or field emission scanning electron microscopy (FESEM) may be preferred because it provides a large depth of field in such analyzes (Duarte and Banhart, 2000; Vendra et al., 2011; Jain et al., 2020; Marx and Rabiei, 2020; Oveisi and Geramipour, 2020). The differences that may occur on the surfaces can be observed with these methods. Thanks to the high depth of focus and

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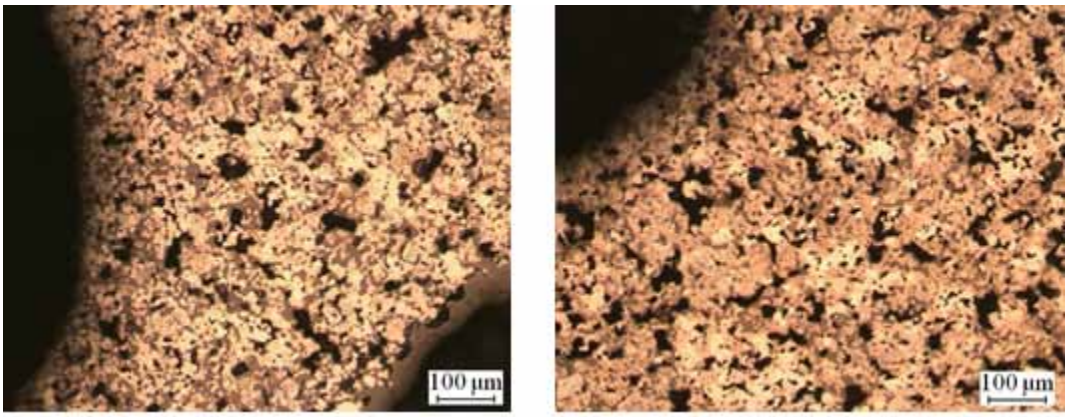
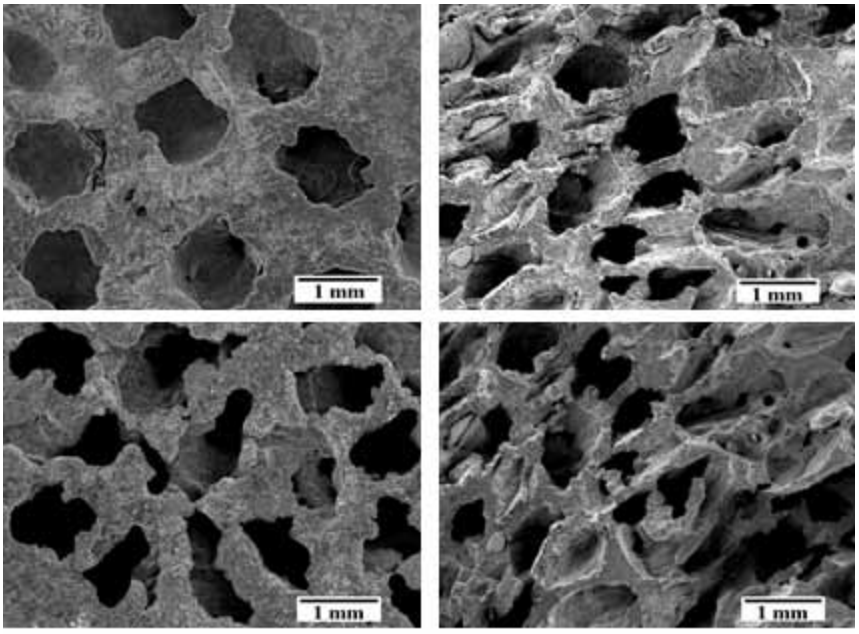
Figure 4. Optical microscope images of the pore walls (Bekoz and Oktay, 2014).



wide working distance of SEM, three-dimensional micrographs can be obtained from rough samples at all magnifications. The SEM images of the steel foams were shown in Figure 5, which was taken from another study, conducted by one of the chapter authors (Bekoz and Oktay 2012). Electron Backscatter Diffraction (EBSD) may use to determine the structure, phase, and crystal orientation of the material in SEM. No damage occurs during the analysis, but as the material requires cutting, ground or polishing in sample preparation stages, effectively a destructive technique.

Figure 5. SEM images of the steel foams (Bekoz and Oktay, 2012).



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Electrical and Thermal Conductivity Measurement

The thermal conductivity of metallic foams is less than their dense form due to the pores in their structure (Dyga and Płaczek, 2015). Foam structures and pore morphology are property determining factors. Open-cell foams are ideal for use as heat exchanging materials due to their corrosion resistance combined with a large surface area and a cell wall material with high thermal conductivity properties. Metallic foams are used in heat exchangers, resistance heaters, thermal energy stores, volumetric solar receivers, and cooling machines due to their high specific surface. Mostly used closed-cell foams have a much lower thermal conductivity than open-cell foams, which makes them good for thermal shielding (Liu and Chen, 2014; Kuruneru et al., 2020; Iasiello et al., 2021). As a result of this, electrochemical activity and catalysis etc. properties of foam are better than dense form (Qin et al., 2016). For example, it is common to use lead foams in battery electrodes and nickel foams used as electrodes in electrochemical reactors. In addition, metallic foams such as Fe22Cr/Ni20Cr are used as electrode support for the fuel cell (Liu and Chen, 2014; Banhart, 2001). Methods such as low frequency eddy current measuring (Babcsán et al., 2003) and four-point technique (Goodall et al., 2006; Dharmasena and Wadley, 2002) are used for electrical conductivity measurements. Many properties of metallic foams can be measured with the eddy current method. The principles of the method mentioned in the 1.1 section. Thermal conductivity measurements can be made using general conduction tests such as the laser impulse method with diffusivity measurement (Dyga and Płaczek, 2015). This method can also be used to measure local densities and other pore parameters in the sample. Due to the low thermal resistance of metallic foam materials, a small cross-section is require. However, finite cell size affects a minimum cross-section. The contact between the the foam and the leads should be good. Senior (2017) carried out measurements by this method using samples with a cross section of 20x20 mm, following a modified standard procedure in this study on the thermal conductivity of aluminum foams.

Acoustic Measurement

Although polymer foams are generally preferred for sound insulation applications, metallic foams create great potential for these applications. In addition to sound insulation, metallic foams can insulate thermal and impact. For this reason, they are used in automobiles or machines. The unique structure of metal foams allows them to attenuate acoustic energy and sound waves by transforming them into heat energy. For this reason, open-cell metallic foams can act as acoustic absorbers. For sound absorption, cellular metals absorb sound waves once they enter the pore cavities (Sulong et al, 2014). Samples with closed pore structure show higher isolation than interconnected pores. Since the sound absorption property of metal foams can provide fast and precise measurement, it is generally measured in an impedance tube (Banhart, 2001). However, only the vertical incidence of the sound wave on a specimen can be characterized. Therefore, more tests are required for a newly developed part. In the measurement with impedance tube, the sample is prepared as disc-shaped. The tube consists of a sample near one end with changeable distance to the rear wall and a sound emitter and detector on its opposite side (Banhart, 2001). In tests on the sample of interconnected porous foam, the absorption curves are shifted to lower frequencies when the gap is expanded. However, dense specimens or porous specimens with closed pores have the same absorption because sound cannot penetrate the specimen and absorption occurs at the surface. Especially for small tube diameters, it is necessary to measure on more than one specimen. It is useful to make two measurements for one possible orientation of each side for each specimen. The absorption of closed pore

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foams is clearly different for the two orientations as it occurs at the surface, whereas the absorption of open pore foams spreads to the volume, so it remains unchanged when the specimen is turned (Xu et al., 2017). Lu et al., (2009) investigated the effect of different processing methods on sound absorption properties of open and closed metal cell foams. Closed-cell foams were reported poor sound absorbers due to preventing air particles to penetrate inside the material.

Vibrational Analysis

In porous structures, during the propagation the sound is attenuated due to vibration and friction losses as gas flows between cells, and thus repeated reflections within the porous structure are required for full absorption to be possible, resulting in long paths. Metal foams are used due to their sound absorption ability for the minimization of noise (Amjad, 2001). Vibration properties of materials used in automotive applications are very important. The automobile industry is move towards using more aluminum to reduce weight. However, this becomes critical because aluminum has a lower vibration damping behavior compared to cast iron and steel. However, in the tests studied collaboration with the "Karmann GmbH" and Dresden Technical University on the damping properties of aluminum sheet, steel sheet and aluminum foam sandwich (AFS), AFS demonstrated significantly better damping behavior compared to other sheets (Claar et. al.,2000).

The loss factor of metal foam and Young's modulus can be determined by performing vibrational analysis. In short, the sample (long bar of rectangular/round cross section or a thin quadratic plate) is forced to vibration. Once fixed, vibrations can be investigated by excitation (longitudinal, transverse or torsional), excited with a magnetic field or using an electromagnetic shaker without applying compression. However, applying vibration analysis to determine these properties is not entirely correct. Because foams are not homogeneous and Young modulus depends on the mass distribution (Banhart, 2001; Fenjan et al., 2019).

Biological Compatibility Tests

Metallic foams have attracted attention because many metals can be produced in porous form and the pore structure increases osseointegration in bio implant applications. Besides the porosity, the porous forms are more compatible with the mechanical properties of the bone than of the dense forms of the metals. The pore morphology and the mechanical and the corrosion properties of the metallic foam should be known for a suitable design (Wen et al., 2001; Matassi et al., 2013). Determination of pore properties is explained in Section 1.1 and measurement of mechanical properties of porous samples is explained in Section 1.2. Corrosion tests of metallic foam to be used in biological applications are different from normal corrosion tests. Corrosion behaviors are observed in-vitro and in-vivo environment (Lewis, 2013). In-vitro tests are carried out in a solution similar to body fluid prepared in a laboratory environment by examining the corrosion behavior for different times. In-vitro immersion tests are performed in different solutions such as Hank's solution (Franciska et al., 2017) and saline solution (Oriňáková et al., 2013). In addition to corrosion tests, in-vitro studies are performed in tests such as tissue/cell growth (Xue et al., 2007; Cheung et al., 2007; Ryan et al., 2008), cytotoxicity (Glorius et al., 2011) and antibacterial properties (Tong et al., 2020). In vivo tests are carried out with animal experiments (Li et al., 2007; Wegener et al., 2020) and clinical studies (Levine et al., 2006; Matsushita et al., 2017).

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X-Ray Diffraction Applications on Them

After Wilhelm Conrad Röntgen discovered X-rays in 1895, there were important advances in the field of crystallography (Saravanan et al., 2011). Discovery of X-ray diffraction in crystals by Max von Laue in 1912; In 1915 W.H. Bragg and W. L. Bragg's X-ray diffraction analysis of single-crystal studies; Analysis of Polycrystalline materials proposed by Debay and P. Scherrer in 1916 and the explanation of a new chemical analysis method by Hull through X-ray diffraction (Hull, 1919; Jenkin, 2001; Borisov et al., 2012) are some of these important studies. X-ray diffraction is a highly useful method used to gain atomic scale knowledge from crystalline and amorphous materials in the field of material characterization (Suryanarayana et al., 1998). First of all, presume an isolated atom in order to illustrate the principle of this methodology. X-ray scattering is known as the process of absorption and re-emission of electromagnetic radiation (X-ray) by the atom. In the case of elastic scattering, the atom absorbs the X-ray photon, releasing another photon, with the same energy as the incident radiation (Suryanarayana et al., 1998). The constructive interference between monochromatic X-rays and crystalline samples is the basis of X-ray diffraction. When conditions obey Bragg's Law, the interaction of the incident rays with the sample generates constructive interference and as a result a diffraction pattern. In a crystalline sample, Bragg's law depends on the wavelength of electromagnetic radiation, the angle of diffraction, and the lattice spacing. The conversion of the diffraction peaks to d-spacings helps the mineral to be defined since each mineral has a specific set of d-spacings. This is usually done by comparing d-spacings to recommended samples (Dutrow et al., 2020).

The X-ray diffraction technique, which has become increasingly important in recent years, is among the techniques that are widely used in qualitative and quantitative analyzes, and in the illumination of the properties and structures of polymers.The technique provides a huge amount of details from a small amount of sample, and does not cause any damage on the sample. The X-ray diffraction technique can also be modified for quantitative purposes, in addition to being used as a qualitative analysis tool, since there is proportionality between the intensity of the diffraction peaks of a given substance in a mixture and the fraction of the material (Ryland, 1958).

In addition to ensuring deep knowledge about the crystallographic structures, chemical compositions, and physical properties of materials, XRD may also be used to classify powdered metals and mineral compounds, quantify crystal phases, quantify desired orientation, and calculate stress in metals and ceramics. (Di Gianfrancesco, 2017; Sima et al., 2016).

Since the structures of metals and alloys are interrelated with their properties, their structures must be known to improve the overall properties of materials. X-ray techniques, which include a much clearer, simpler, and more objective testing tool, have also made a major contribution to metallurgy study. If the crystal structures of metals and alloys had not been discovered by XRD, the knowledge about the shapes, defects, and properties of metals and alloys would not be available. XRD is a non-destructive tool for examining a wide variety of materials such as crystals, metals, stones, polymers, thin film coatings, and ceramics (Saravanan et al., 2011).

The following are studies of X-ray diffraction applications in this area:

In a study performed by Huang and his team in 2017, The XRD analysis of the chemical and crystalloid structure of Myristyl alcohol (MA) / metal foam composites was performed and verified that MA was physically absorbed, without chemical interaction into metal foam composites (Huang et al., 2017).

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A research carried out by Michailidis et al. in 2013, the possibility of producing an intermetallic aluminum-nickel coating on Ni foam, that did not impact the original structural properties of the foam, was explored and several slurries were tested for this process. By X-ray diffraction analysis (XRD), the phases formed by the aluminized slurry phase were determined (Michailidis et al., 2013).

In another study conducted by Banciu et al. in 2020, it was reported that the graphene grown by chemical vapor deposition on metal foam substrates leads to obtaining good graphene structures suitable for different applications. In the same study graphenes grown on metal foams were examined by XRD (Banciu et al., 2020).

In the 2019 study performed by Sutygina, open-cell aluminum foams with a total porosity of ~90% were produced with a sponge copying technique. The effects of atmospheric and temperature conditions on the cell structure, porosity of the phase composition, thermal conductivity and compressive strength of these foams have been studied in this study. It has also been reported that the thermal processing of aluminum foams at temperatures up to 800°C in Ar atmosphere results in aluminum foams with lower strut porosity, lower aluminum oxide content, higher thermal conductivity, and higher compressive strength compared to foams treated thermally in the air. The lower amount of aluminum oxide in foams after thermal treatment explains these results. By performing XRD phase analysis, it was observed that aluminum oxides (α-, γ-Al_2O_3) were formed after thermal processing of Al foams (Sutygina et al., 2019).

In a 2011 study, copper reinforced aluminum foam was developed and the produced foams were characterized. The densities of open cell foams produced using mud mixtures were determined and XRD analysis was conducted; it was observed that CuO and Cu_2O structures are formed as a result of the sintering of copper-reinforced aluminum foams under normal atmospheric conditions, and these copper oxide structures are reduced by sintering in the argon atmosphere (Zaman,2011)

By vapor aluminizing, three separate Ni-Al foams were synthesized. To determine the oxidation resistance and compression properties of foams, oxidation tests and compression tests were applied below 1100 ° C. To analyze the phase composition and microstructure of Ni-Al foams before and after oxidation, an X-ray diffraction (XRD) test was conducted. The findings demonstrate that the vapor aluminization process does not affect the macro- and microstructure of the foam, and as the aluminization period increases, the phase composition changes from β / γ, phase to single β phase (Zeng et al., 2017).

In the analysis carried out by Rosip et al., in 2013, 316L stainless steel was used by the slurry process to produce metal foams. They combined the raw materials and then immersed the polyurethane foams for 24 hours in the dried mixture. The obtained samples were sintered at 1300°C in the furnace. To characterize these samples, various tests, including x-ray diffraction (XRD), were carried out, and the XRD results revealed that after sintering, the samples were oxidized (Rosip et al., 2013).

Cavuslu et al. in 2019, used the plasma electrolytic oxidation (PEO) treatment to improve the mechanical properties of the open-cell aluminum foams. The microstructure and composition of samples were revealed by scanning electron microscope (SEM) and X-ray diffraction (XRD) respectively. It was reported that the coating thickness of the struts was increased with the increasing treatment time. (Cavuslu et al., 2019).

FTIR and Raman Spectroscopic Applications on Them

In the late 1880s, when Albert A. Michelson invented the interferometer known as the Michelson interferometer, on which he and Morley conducted the well-known experiment to calculate the speed of light, the science of Fourier transform interferometry was started. Michelson was unable to take advantage

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of the Fourier transform spectroscopy (FTS) field because computers and electronics enstruments were not adequately usable at the time.

Practical FTS eventually began to appear in the late 1940s. Interferometers were initially used to measure light from celestial bodies, and the first Fourier transform spectrum was developed by scientists in 1949. (Jaggi et al., 2006). In the infrared part of the spectrum, molecular absorption of electromagnetic radiation accommodates the changes between the vibrational or vibrational-rotational energy levels of the ground electronic energy state of the molecule (Ismail et al., 1997). In this technique, the sample is irradiated with infrared radiation (IR) from an infrared source (containing all IR frequencies) and usually the intensity of transmitted radiation is monitored. The radiation would be absorbed at some resonant frequencies that are characteristic of the sample molecules, resulting in a sequence of peaks in the spectrum that can then be used to classify the sample. Such variations in vibrational motion contribute to bands in the vibration spectrum; their frequency and amplitude define each spectral band. (Gomez et al., 2003; . Rodriguez, 2000; Sacksteder et al., 2001; Stuart 2004; Jaggi et al., 2006). In order to classify and compare sample compositions and to evaluate the concentration of different components of a mixture, Fourier Transform Infrared (FTIR) spectroscopy can be used quantitatively. The advantage of this technique is the use of very small amounts of substance (e.g. 1 mg) and the analysis of all substances, whether crystalline or amorphous, organic or inorganic. FTIR is extremely valuable for the study of a wide variety of materials, such as determining the composition of materials such as fired clay, bone and tooth enamel, wood ash, fibers and dyes, and resins (Margaris, 2014).

The process of inelastic scattering was theoretically predicted by Brillouin (1922) and Smekal (1923), and was experimentally observed in a molecular system in 1928 by C.V.Raman and K.S.Krishnan (Jayasooriya &Jenkins, 2002). This phenomenon has since been referred to as the Raman scattering. The incident photons may be absorbed, scattered, and not interfere with the molecules of the substance. If the photon's energy correlates to the energy difference between the molecule's ground state and the excited state, the photon may interact with the molecule, can be absorbed and the molecule can be promoted to the excited state of higher energy (Smith and Dent 2019). In absorption spectroscopy, this transition is evaluated by the detection of the loss of radiation energy. However, if the energy of the photon does not match the difference between the two energy levels of the molecule, there will be no absorption, but the photon can interact with the molecule and scatter from it. In this case, the scattered photon carries information about the molecule (Smith and Dent 2019). By accumulating them at an angle to the incident light beam, the scattered photons may be detected. (Smith and Dent, 2019). Raman spectroscopy investigates the inelastic scattering of light by molecules. The technique provides information about the vibrational energy levels of the molecular systems of the environment, as a result, it offers qualitative and quantitative analysis in various media. The areas of application of Raman spectroscopy are the field of forensic science (explosives, narcotics, inorganic fillers for analysis), in the field of biology (biochemical knowledge on toxic substances, drug reactions, death or post-disease cells), as a diagnostic tool for the analysis of different biomedical materials, and it has in different fields of application, from superconductors to archaeological materials. In addition, the characterization of drug formulations, the description of the kinetic processes in drug delivery, in pharmaceutical and biopharmaceuticals, and in nanotechnology (due to the research and characterization of nanomaterials such as nanosensors, nanotubes and nanowires) are it's different application areas (Das et al., 2011).

Below are the researches of FT-IR and Raman spectroscopic applications in the subject area:

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The microstructure, crystal structure and phase analysis of the composite foam of aluminium alloy (LM30), prepared by reinforcing the foam with 5 wt% of SiC particles, through melt-stir processing route, were performed by FE-SEM, EDX, XRD and FTIR analyses (Rajack et al.2018). In this study, the FTIR spectrum the foam was given and OH stretching, XH stretching, C=O stretching, -C=C- stretching, =CH bending vibration modes were determined at 3420cm^{-1}, 2910 cm^{-1}, 1720 cm^{-1}, 1605 cm^{-1} and 1100 cm^{-1}, respectively (Rajak et al., 2018).

In another study conducted by Zhang, in 2018. Microalgae were inserted into the microbial fuel cell as a cathode, for the Cd(II) extraction using nickel foam/graphene as electrodes. Graphene on nickel foam was compared with graphene oxide using FTIR spectroscopy. Since the nickel foam graphene retained the oxygen-containing functional group on the graphene oxide, the intensity of this band was significantly weakened and this was interpreted as partially decreasing the amount of graphene on the nickel foam (Zhang et al., 2018).

The synthesis of nanostructured $Ni(OH)_2$/ reduced graphene oxide (rGO) on Ni foam has been investigated using a simple and cost-effective chemical deposition method in a study conducted by Lokhande in 2019. Moreover, $Ni(OH)_2$/rGO composite's FTIR spectrum has been reported, and its vibrational bands and wavenumbers have also been determined (Lokhande et al., 2019).

Graphene was synthesized using nickel and copper foams as substrates in the chemical vapor deposition (CVD) phase with methane in a study performed by Banciu et al in 2020. The graphene grew on metal foams was analyzed using Raman spectroscopy. From the Raman intensity ratio, It was concluded that the number of graphene layers increased as time grows. The loss of the D band revealed low defect content in the case of graphene structures synthesized on Ni foam and no defects were observed in the graphene layer. (Banciu et al., 2020).

Graphene was grown on nickel foam using the chemical vapor deposition process in the study conducted by Lee et al in 2017. Raman spectroscopy technique was used to characterize the morphology and crystallization of graphene films. This research has shown that multilayer graphene grows on nickel foam (Lee et al., 2017).

In another research, Kaur et al grown carbon nanotubes on copper and nickel foams by chemical vapor deposition technique, and it was seen that the Raman spectra of all graphitic materials have G band (1580 cm^{-1} band, due to first-order Raman scattering), D band (1350 cm^{-1} band, due to one phonon second-order Raman scattering), and 2D or G' band (band at 2700 cm^{-1} due to two phonon second-order Raman scattering process) (Kaur et al., 2020).

In Mukanova's research, chemical vapor deposition technology was used and multilayer graphene (GF) thin-film synthesis was performed on Ni foam in 2018. The Raman spectroscopic investigation confirmed that the samples were GF of several layers of high consistency and purity (Mukanova et al., 2018). The MnO_2-3B graphene foam composites were developed in the 2018 by Kasap, and the Raman spectroscopy technique was also used for structural and morphological analysis. The findings showed that MnO_2 nanoparticles were effectively integrated with the foamy three-dimensional graphene structures (Kasap, 2018).

Raman spectroscopy was also used to characterize the structure of metal oxides on the surface of Field's metal foams in the 2019 study (Allioux et al., 2019).

In a study carried out by Peng et al, a series of CuO-CeO_2 / Ni metal foam composite catalysts with hierarchical structures were prepared using copper-cerium hydrosols and macroporous nickel foams. Raman spectroscopic analysis indicated that with the addition of CuO to the prepared catalysts, the band intensity observed at 465 cm^{-1} decreased significantly (Peng et al., 2014).

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CONCLUSION

The characterization of metal foams from new lightweight materials is explored in this chapter. The main conclusions of this chapter can be summarized as follows:

- Metal foams are a class of materials known for their low densities and excellent strength-to-weight ratio, and good physical, chemical, and mechanical properties, which make them suitable for a wide range of industrial applications. As mentioned before; in the last years, many studies in the literature were made for metal foams, but the knowledge in the field of the characterization of metal foams is still very poor. With the development and understanding of the characterization methods of metal foams, this situation will be further clarified. As it is generally discussed in this chapter; foam metals demonstrate a wide variety of specific mechanical, microstructural, and physical properties that make them different from traditional dense metals.
- The properties of metal foams can be characterized in various ways. They are characterized by the morphology of the porous cells (cell geometry, open or closed), cell topology, relative density, pore size, pore shape, pore size distribution, pore wall defects, and degree of anisotropy. The mechanical, corrosion, and biological tests, electrical and thermal conductivity measurement, acoustic and vibrational analysis are very important in characterizing the properties of metal foams. A good understanding of all these features is necessary for a successful implementation in industrial applications.
- Foam structure and pore morphology are property determining factors. The mechanical and physical behaviors of metal foams depend on their structures. The structure of foams can be characterized in terms of geometrical and material structures.
- The details of the properties should be examined in order to use metal foams in a large scale. This chapter adds depth to the subject by considering different analysis techniques such as X-ray diffraction applications, FTIR, and Raman spectroscopic techniques and applications of metal foams.

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