

Investigation on Fabrication of Aluminium based porous materials

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in partial fulfillment of the requirements
for the degree of*

Master of Engineering

in

Production Engineering

by

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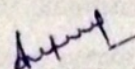


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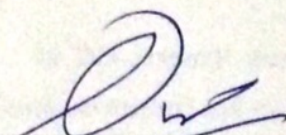
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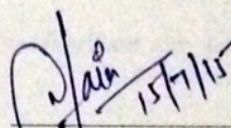
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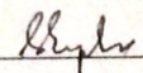

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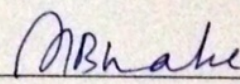
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In this research work an attempt was made to fabricate the porous material by conventional method and traditional method.

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Abstract

An experimental study has been conducted to investigation on fabrication of aluminium based porous materials. In this research work an attempt was made to fabricate the porous material by conventional method and traditional method. The porous sample was prepared using conventional method and porous foam sample was prepared using gas releasing blowing agents. For each, porous and porous foam sample investigated the porosity, compressive behaviour and metallurgical test are compared. It was shown that although aluminium porous foam has constant plateau stress which resulted higher energy absorption than the porous aluminium fabricated by conventional method, but the uniformity of pores maintained in case of porous material fabricated by conventional method. The line mapping results have further shown that adding elements were still present on the walls of pore cells.

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Nomenclature

| | | |
|-----------------------------------|---|------------------------------|
| 3-D | = | Three Dimensional |
| Al | = | Aluminium |
| CAD | = | Computer Aided Drawing |
| CNC | = | Computer Numeric Control |
| FDM | = | Fused Deposition Machine |
| HGM | = | Hollow Glass Microsphere |
| HSS | = | High Speed Steel |
| MgCO ₃ | = | Magnesium Carbonate |
| Na(CO ₃) ₂ | = | Sodium Bicarbonate |
| NaCl | = | Sodium Chloride |
| RF | = | Radio Frequency |
| RMPM | = | Red Mud Porous Material |
| RP | = | Rapid Prototype |
| SEM | = | Scanning Electron Microscope |
| SiC | = | Silica Carbide |
| STL | = | Stereo Lithography |
| TCP | = | Tricalcium Phosphate |
| TiH ₂ | = | Titanium Hydroxide |
| UTM | = | Universal Testing Machine |
| V _p | = | Volume of Pores |
| XRD | = | X-Ray Diffraction |

Chapter 1

Introduction

1.1 Porous material

Porous means the material contains pores (small or tiny holes) while non-porous means the material which does not contain pores. If the material will pass liquid or gas, it is porous material. Examples of porous materials: dry wall, wood, concrete, rubber, metal foams. Examples of nonporous materials: hard fired brick, some ceramic tiles, some types of marble, granite, solid pure materials.

In another words porous materials are organic materials, polymeric forms. A huge amount of inorganic porous materials have been developed.



Figure 1.1: Porous material

1.1.1 Porosity

It is the ratio of total pore volume V_p to the apparent volume V of the material or powder. Weight or mass is also depend on porosity of the material, if the porosity increases the weight of material decreases.

$$Porosity = \frac{Volume\ of\ Pores\ (V_p)}{Apparent\ Volume\ (V)}$$

1.1.2 Measuring porosity

The measured value is dependent on the method used. Some detects just open pores, e.g. adsorption of molecules. (Open pore and accessibility is then defined by the probe molecule) Other may probe also closed pores, e.g. spectroscopy, diffraction, scattering.

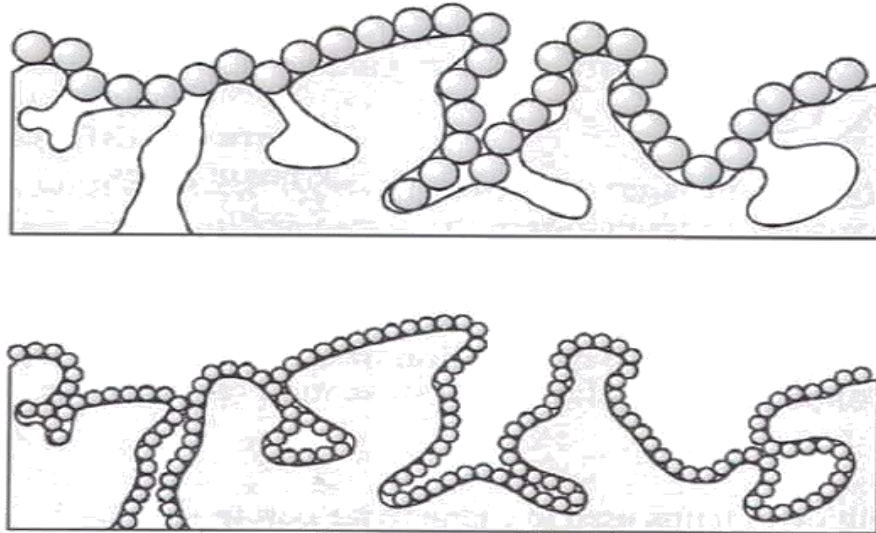


Figure 1.2: Different form of porosity

1.1.3 Pore size

Pore size is important parameter. IUPAC, three pore size regimes, associated with the transport mechanism. The classification of pore size in case of fluid flow are- Micro porous, Meso porous and Macro porous.

- **Micro porous:** (Smaller than 2 nm) in this the pore size comparable to the molecules.
- **Meso porous:** (between 2 and 50 nm) same order or smaller than the mean free path length
- **Macro porous:** (larger than 50 nm) larger than typical mean free path length of typical fluid. Bulk diffusion and viscous flow.

1.1.4 Different types of pores:

1. **Accessibility** as shown in Fig. 1.3

a: closed pores

b, c, d, e, f: open pores

b, f: blind pores (dead-end)

e: through pores

2. Shape:

- c: Cylindrical open
- f: Cylindrical blind
- b: ink-bottle-shaped
- d: funnel shaped
- g: roughness

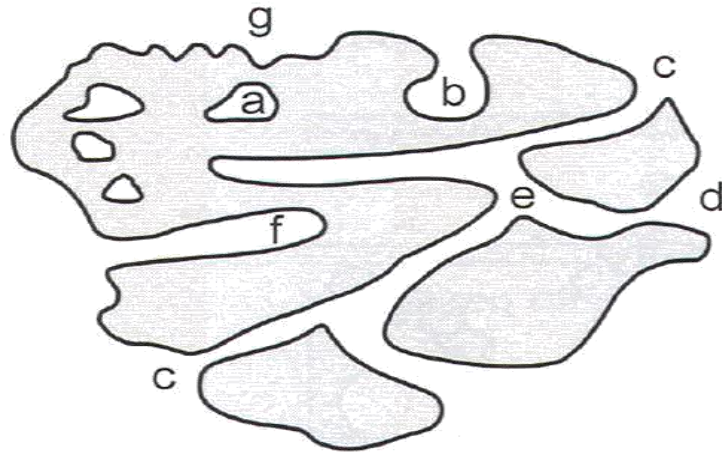


Figure 1.3: Types of pores

1.1.5 Metallic form and Porous metals

Composite materials are consisting of a solid and a gaseous phase. It is a cellular structure consisting of a solid metal.

- **High bulk density-** independent, distributed voids Metallic foams:
- **Low bulk density-** interconnected voids.
- **Porosity:** 30-98% of the volume

Porous metals: are used due to- Impact energy adsorption, air and water permeability, acoustical properties, low thermal conductivity, Energy absorbing systems, porous electrodes, sound absorbers, filters, insulating materials, heat exchangers, construction materials, electromagnetic shielding, membrane and membrane support.



Figure 1.4: Porous material in metallic foam

1.1.6 Application of porous materials-

- Filtration product in aerospace industries Orthopaedics in medical
- In transport system to carry drugs Aqua pressure pads
- Home appliances
- Turbine blade nuts
- Fuel cells and solar cells Lead acid batteries
- Sensor materials
- Nuclear waste disposal Super capacitor
- Optical sensor
- Metallurgical materials Foam material
- ANG (absorbed natural gas tank)

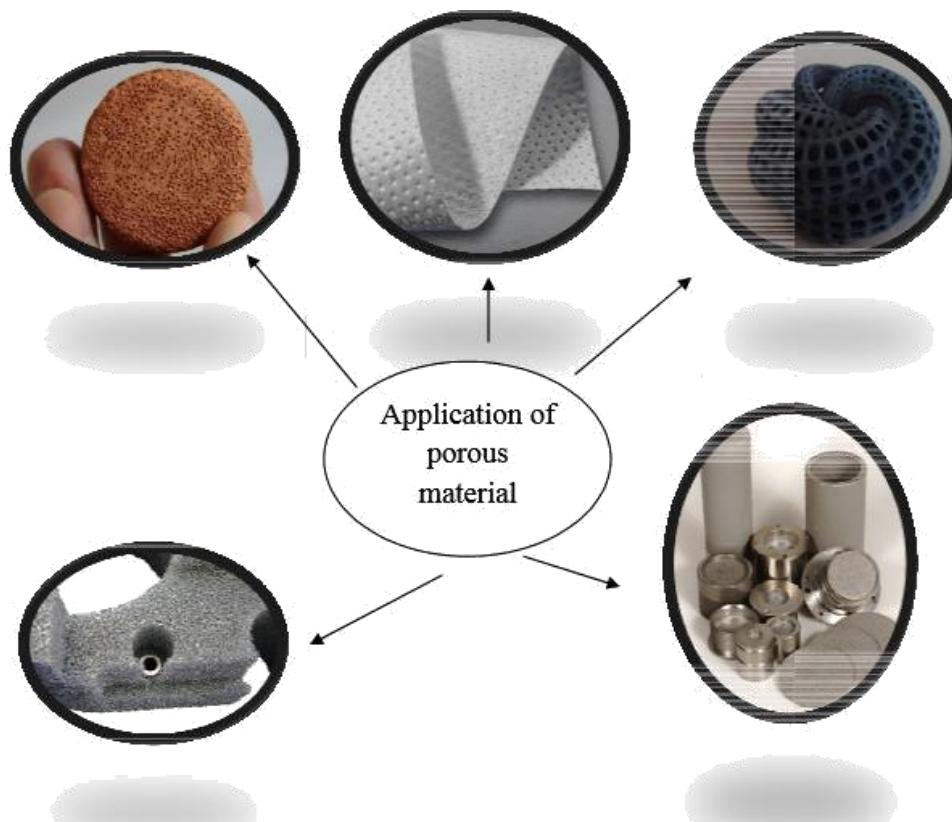


Figure 1.5: Different applications of porous material

1.2 Different method for fabrication of porous materials

1.2.1 3D fiber deposition method

Porous Ti6Al4V scaffold (by RP), is fabricated using three-dimensional (3D) rapid prototyping technology known as fibre deposition (3DF). The main aspect of this technology is to manufacture a scaffold, at room temperature, by 3D computer-controlled fibre depositing of Ti6Al4V slurry, which consists of layers of directionally aligned Ti6Al4V fibres. In this 3D fibre depositing process is fabricate porous Ti6Al4V by layer by layer deposition.

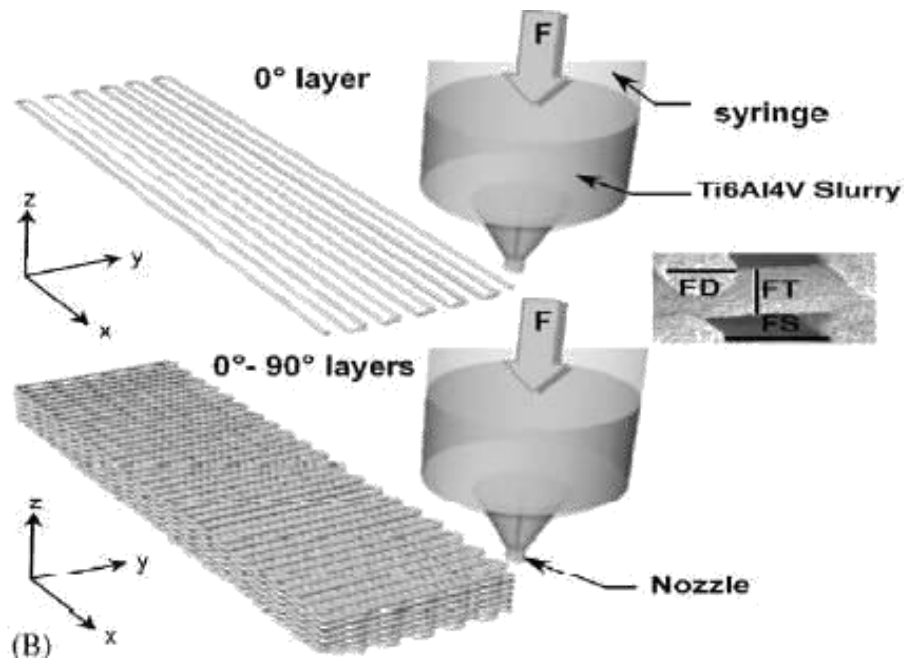


Figure 1.6: 3D fiber deposition process. [Garrett, 2008]

1.2.2 Selective laser melting

Selective laser melting is a technique to fabricate the porous material by melting the material. It then converts the material into 3-D fine filter element. The diagram shows the filter porous material fabricated by selective laser sintering as below. It is an additive fabrication process that uses 3-D data as signal information. As the process start the material spread in the form of layer by layer, each layer deposited to the other layer according to 2-D design of each layer. The laser beam is focussed in the X and Y directions with two great frequency scanning mirrors. The laser energy is penetrating enough to permit full melting of the particles to form solid metal.

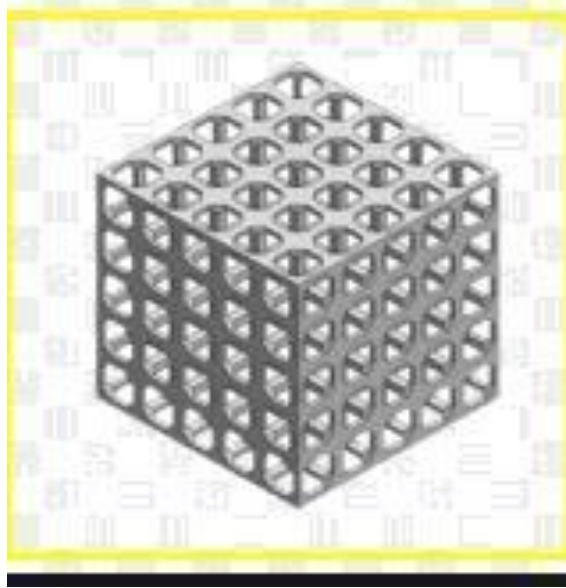


Figure 1.7: Porous material fabricated by SLM. [Yadroitsev et al., 2009]

1.2.3 Hydrofluoric acid

In this method hollow glass microspheres are fabricated. In this method HGM (hollow glass microsphere) are placed with hydrofluoric acid in reciprocating shaker. After this the resultant HGM are washed with distilled water and they float on the layer of distilled water from which they can collect in the foam like surface with spherical structure.

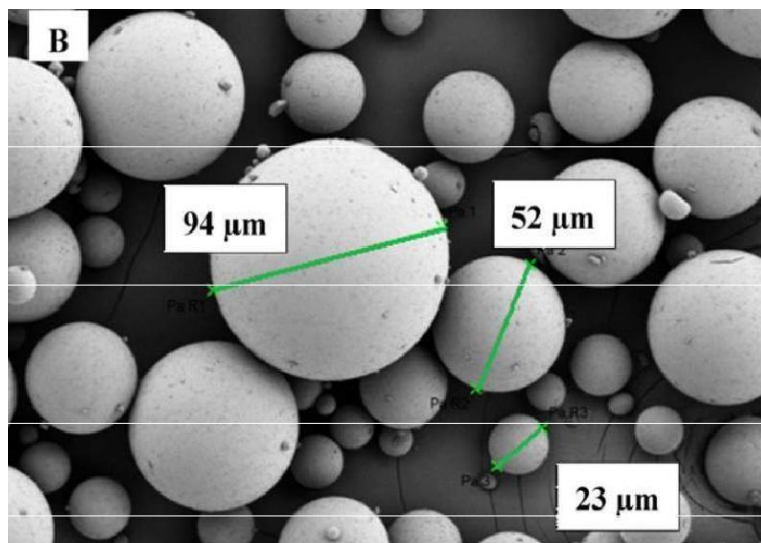


Figure1.8: Hollow glass microsphere. [Yuqun et al., 2011]

1.2.4 By radio frequency (RF) magnetron sputtering

First step, a polystyrene template under a direct current electric field is prepared. A kind of uniform porous material is fabricated on a template of polystyrene by RF magnetron sputtering. After adding ethanol and ammonia solution (pH=8) to polystyrene templates, The polystyrene template was placed on the sample holder in the sputtering chamber of RF magnetron sputtering system. It is seems that alumina porous materials are composed of uniform pores. Two pieces of alumina ceramic disks with a diameter of 5 cm were placed on the target holders as the target materials.

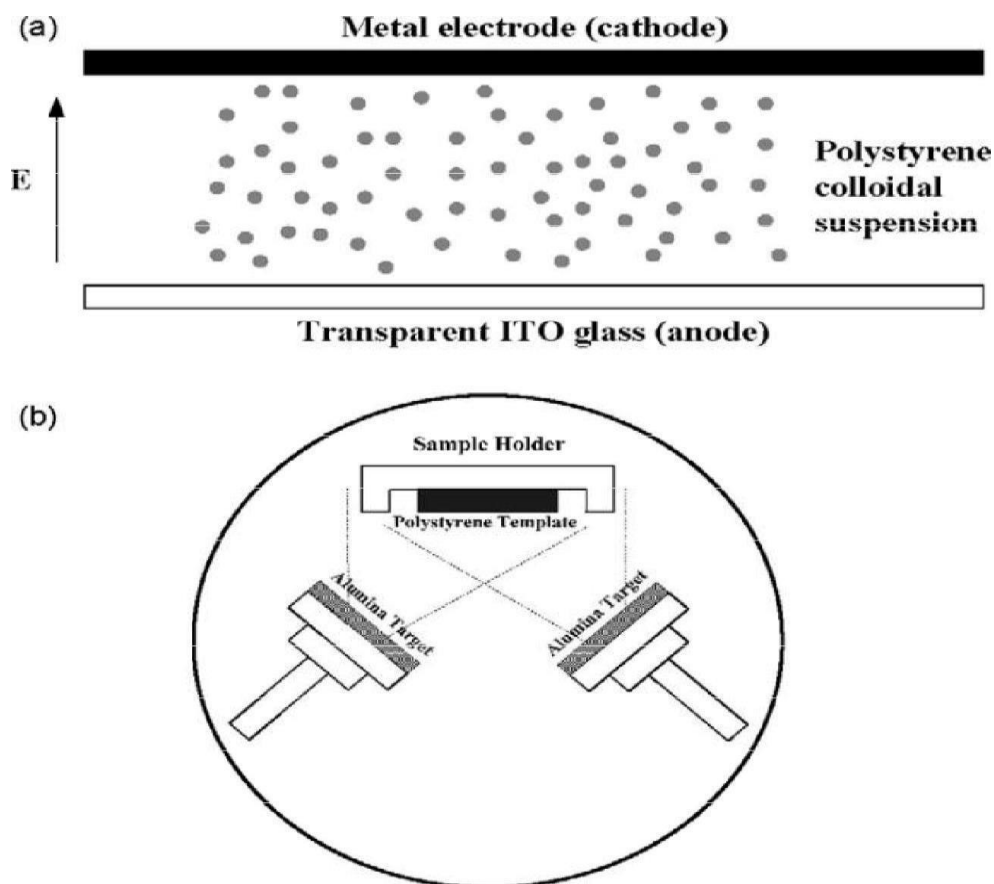


Figure 1.9: (a). A scheme of preparation of a polystyrene template is under direct current electric field. (b) A scheme of the dual-RF. [Tang et al., 2008]

1.2.5 Injection moulding

The tricalcium phosphate (TCP) scaffold of this study was designed by this method. It is a manufacturing process in which material is injected to mould. The part material is fed into a heated barrel, mixed, and forced into a mould cavity, where it cools and hardens to the configuration of the cavity to have an ordered architecture with macro porosity. Similar to cancellous bone, which presents porosity and pore diameters in the range of 200-500 μ m.

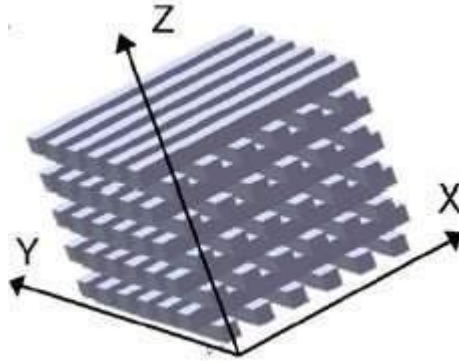


Figure 1.10: Porous material by inject moulding. [Vivanco et al., 2012]

1.3 Metal Foam

In the last few years, due to the weight of metallic foam researcher show more interest in metallic foam.

Metal foam is also called cellular structure. Porous metal foam is made from a solid metal, often aluminium. Porous metal foam have large volume fraction of pores. That pores are filled with gas. The pores can be type of closed cell or open cell. Closed cell is a sealed type and open cell is an interconnected network. Generally metal foam has void spaces, which are 70-95% the volume of the solid metal. Due to the void space the metal foam becomes ultra-light materials. The strength of the metal foam is related with the relative density of metal foam i.e. 20% dense material is double strong than the metal foam with 10% dense. A advantage of this metal foam is, it retain some physical properties of their parent metal. It is a cellular structure.

By the wide research technologies, many issues related to metal foam have been solved. Types of issues are solidification, shrinkage, low foam ability and different size and shape of cellular structure. In past due to relatively high cost, many attempts have been made to fabricate metallic foam structures, due to high cost, they were not successful.

Now, a day with the extensive research metal foam can reproduce to change the size or shape of cells, structure. It consists of solid metal, as well as large volume fraction of pores (voids).

The pores are of two types:

1. Closed pores
2. Open pores

In metal foam porosity is typically 70-95% of the volume occupied by pores or void space. This porosity makes the material light weight. Metal foams retain some physical properties of their parent metals.

Note: The coefficient of thermal expansion will remain same as parent metal but thermal conductivity is 20 times less than the parent metal.

There are many foam materials which are present in the market besides from these metallic foams, aluminium foam offers a great potential for application in Automobile industries, Engineering field, House hold appliances and specially crash protection material for cars.

The main features of foam are:-

1. Foams are light weight
2. Energy absorbing
3. Incombustible and sound absorbing properties

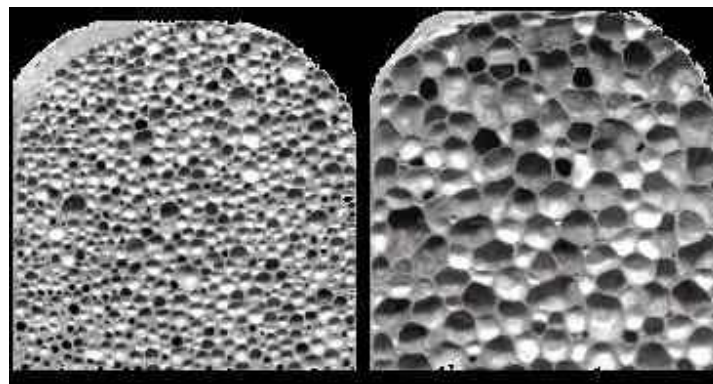


Figure 1.11: Metal foam structure. [Banhart, 2001]

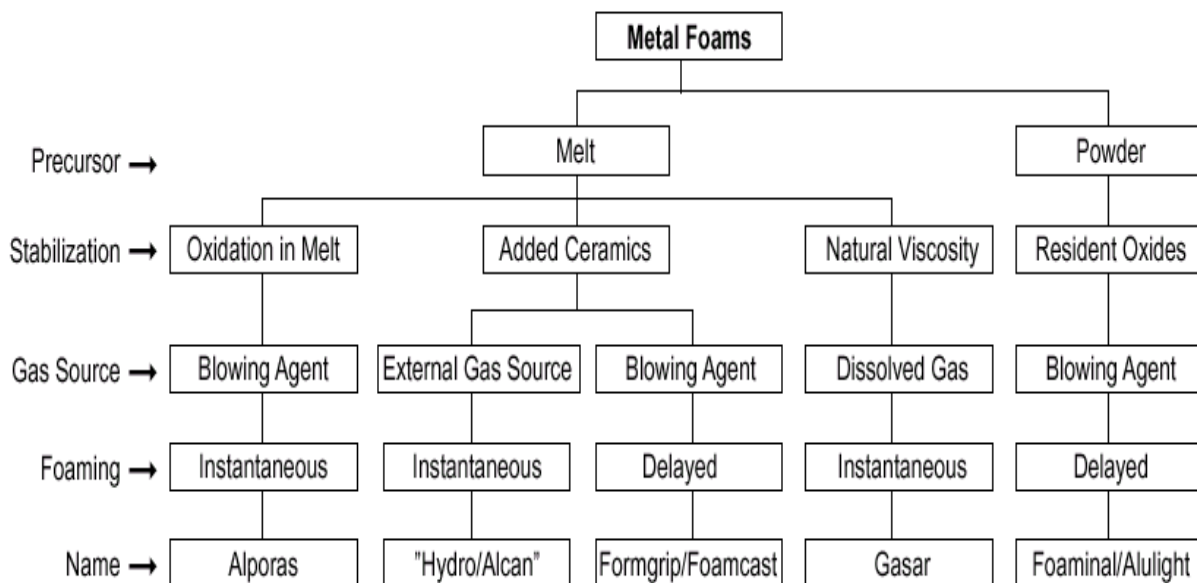


Figure 1.12: Flow chart of fabrication method of Metal foams

1.4 Fabrication methods for metal foam are:

Many technologies had been developed by many researchers so far for the fabrication of porous metal foam but few of them are not suited for the production in an industrial level. The methods for the fabrication of metal foam are divided on the basis of liquid metal route or metal powder route. The methods for the fabrication of metal foam are:

1. Metal melts by gas injection.
2. Metal melts with blowing agent.
3. Metal foam from powder compact
4. Foaming liquid metals

1.4.1 Foaming liquid metals

Metal foam can be fabricated by adding alloying elements or adding fine ceramic powder to the melt. During the fabrication assumption that the developing foam is equally stable for the foaming metallic melts there are three known methods.

1. Gas injecting into the metal melt.
2. Addition of blowing agent into the molten metal.
3. Melted gas precipitation.

1.4.2 Metal melts by gas injection

The first fabrication process of aluminium foam is established on gas injection into metal melts as shown in Fig. 1.13. To produce aluminium foam in this process, hydro aluminium manufactures are applied. SiC, aluminium oxides or many other particles are used in this process to increase the viscosity of the liquid metal and also helping to modify its foaming properties because liquid metals cannot simply be foamed by creating the bubbles through the molten melt. Stability of bubbles is also depends on the percentage of particles added to the melt. The further step with the help of rotating impellers or nozzles the various gases like air, argon or nitrogen is injected into molten aluminium.

Aluminium alloy is usually used as a base metal. The volume fraction of particles fluctuates from 8% to 20% and the mean size of the particles varies from 5 μm . This process permits the fabrication of closed cell foams with dimensions 1 m wide to 0.2 m thick slabs and diameters varies from 5 mm to 20 mm. The relative density of foam fabricated by this method lies between 0.03-0.1.

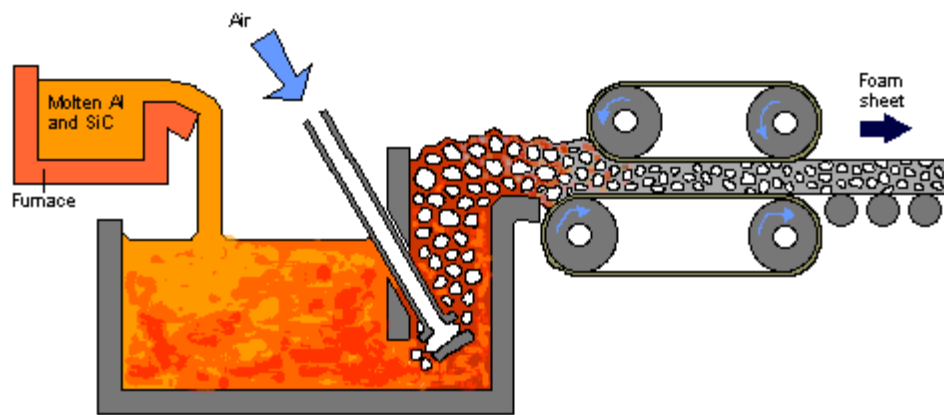


Figure 1.13: Schematic diagram of manufacturing of aluminium foam by melt gas Injection method. [Banhart, 1999]

Due to the presence of ceramic particles in the molten metal, the foam is relatively stable. The molten metal and viscous combination of bubbles drifts up to the surface of the liquid where liquid metal drains out and it converts into dry liquid foam. After this the dry liquid foam is left for cooling and solidification. Many non-metallic assistances counter with molten metals including silicon carbide, boron nitride, alumina but mostly silicon carbide is used in preparation. Gas injection process is the inexpensive one between all others. It is the only continuous production process. At the rates up to 900 kg/hr foam panels can be fabricated by this process. By varying processing parameters like rotating shaft speed and gas injection rate, average cell size, wall thickness and relative density can be adjusted.

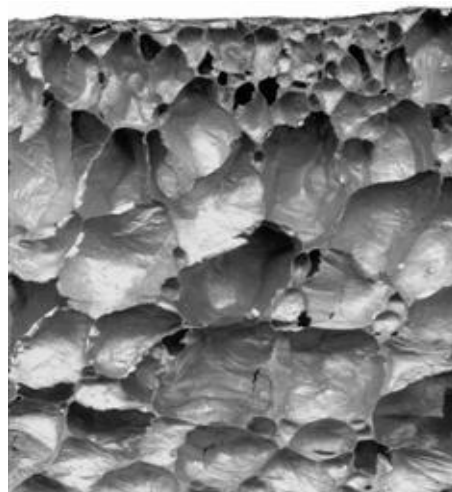


Figure 1.14: Al foam produced by a continuous process of gas injection to a viscous melt, showing the density gradient through the thickness.

Sometimes large cell size foam with irregularities is produced by this method. This is the main disadvantage of this process is the poor quality of foam. The foamed material is moreover used with a closed outer surface or sometimes cut into the desired shape. Machining of this foam can be problematic due to their high content of ceramic particles. The large volume of foam continuously produced with low densities is the main advantage of this process.

1.4.3 Metal melts with blowing agent

Metal foam is fabricated by mixing of blowing agent or foaming agent with the metal alloys. These foaming agents when heated above their melting temperature, release gases. Many foaming agents are there like NaCl, $\text{Na}(\text{CO}_3)_2$, TiH_2 and MgCO_3 , these foaming agent has been used in this process to fabricate the metal foam. Blowing agent when heated, it decomposes into gaseous. By adding blowing agent particles to aluminium melt and gases in large volume are instantly produced. Due to the gas, formation of bubbles on the surface that can lead to closed cell foam. After this stabilize the melt temperature inside the furnace.

To raised its viscosity or in other word to increase foam drainage rate, 5-10 % silica carbide and 2-3% calcium carbonate is added. In this process a graphite stirrer is used for mixing the metal melt with alloys. After withdrawn the stirring system, foam is allowed to form above the melt. The melt is then cooled to solidify the foam before the gases escape and the bubbles of gas collapse. Relative density can be determines by adding the volume fraction of blowing agent and alloys to the melt.

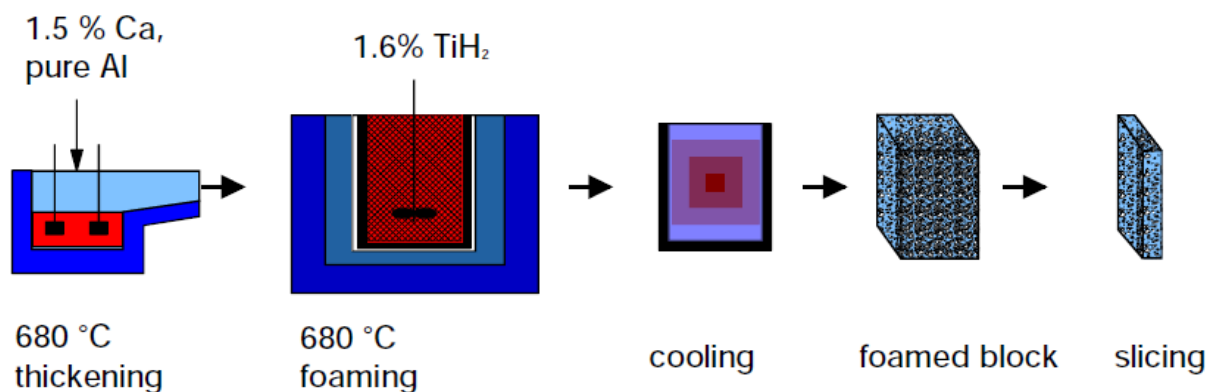
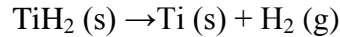


Figure 1.15: The process steps of aluminium foam fabricated by gas blowing agent [Catriing Kammer, 1999]

By changing the cooling and foaming conditions cell size can be varied from 0.5 to 5mm by changing the foaming agent content. Sometimes to attainment a best viscosity of the melt, titanium hydride is added in an amount typically 1.6 weight %, which acts as a blowing agent according to the following reaction:



A subsequent rolling treatment can be used to fracture many of the cell walls in order to increase their acoustic damping. Today, only aluminium alloys are made in this way because carbon dioxide embrittles many metals and because the decomposition of MgCO_3 occurs too quickly in higher melting point alloys.

The melt starts to increase gradually and it slowly fills the foaming vessel.

1.4.4 Powder Compact method:

Powder compact method: this process starts with elementary metal powder mixed with alloy powders and blowing agent powder and then compacted to produce a thick and semi-finished product as shown in Fig. 1.16 besides metal hydrides (e.g. TiH_2 , ZrH), Carbonates (e.g. CaCO_3 , MgCO_3 and $\text{Na}(\text{CO}_3)_2$ or elements that dissolve fast can be used as foaming agent. Powder rolling, rod extrusion uniformly or uni-axial compression techniques are including in this process. To produce a bar, extrusion can be used and also helps to break the oxide films at the faces of the metal powders. During the heating process decomposition of the blowing agent and material enlarged by the released gas forces. By this heating process (350°C - 450°C) a highly porous structure is formed. Cold compact mixture of metal powder blowing agent and powders when extruded to solid metal blowing agent is dispersed on the entire solid metal. The foaming agent decomposes to release gas, when heated above to its melting temperature. The metal foam is produced as a result of heating. Main problem in this process is the cooling system after the foam fabricated. The heat source could be turn off rapidly. But the metal would still be hot. Due to this reason metal foam is disposed to collapsing back into molten metal before it freezes. To avoid this problem water cooling is used. For the fabrication of foam by this method cooling problem may become a significant challenge. This foam has a closed cell structure with the pore diameters varies from 1 mm to 5 mm.

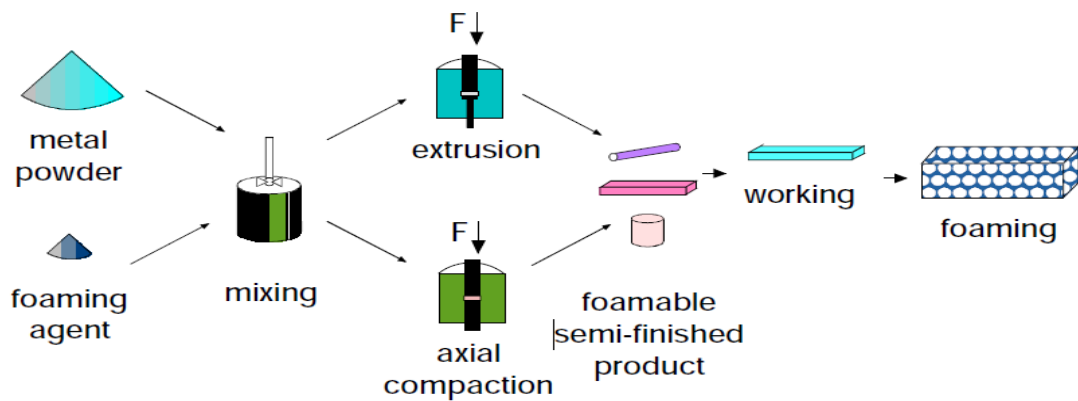


Figure 1.16: Metal Foaming from powder compact process. [Catring Kammer, 1999]

1.5 Properties of metal foams

Metal foams are metallic porous materials. These materials are having some unusual properties due to which metal foam is mainly suited for some applications. The metal foams can easily float in the water due to their low densities but this happens only in case of closed pores. The strength of metal foam is lower than the dense material and decreases with decreasing density. Foams are unchanging up to the melting point temperature. They are flame proof and risk free. In most foam fabrication technologies the properties can be diverse over an extensive range by regulating the fabrication parameters. [Catring Kammer, 1999]

1.5.1 Mechanical properties

When foams are compared to conventional metal behave differently in testing due to their cellular structure. Conventional testing like tensile testing cannot be used because foam shows different behaviour from conventional metal. The compression test has most meaningful results in case of metal foam which distribute the stress strain diagram with a partition into three parts as shown in Fig. 1.17 for the foams behaviour with both open and closed cell walls was found to be distinctive. It displays a linear increase of stress with strain (I) at the start of deformation a plateau constant stress is found (II) then the II region is followed by a sharp increase in flow at the end (III). The following process in Fig. 1.17 is showing the reasons for this distinctive behaviour.

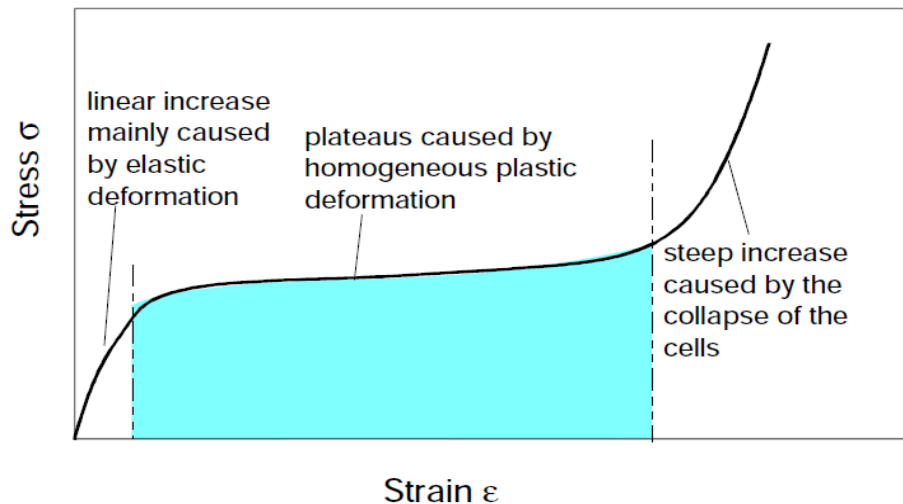
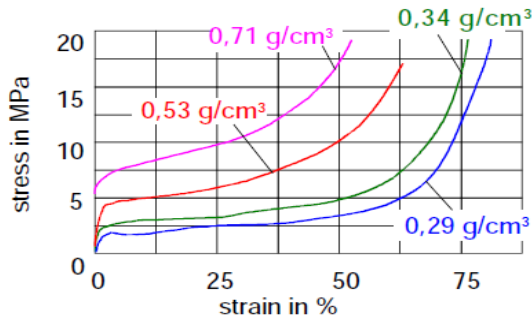


Figure 1.17: Stress-strain diagram for aluminium foam. [Catring Kammer, 1999]

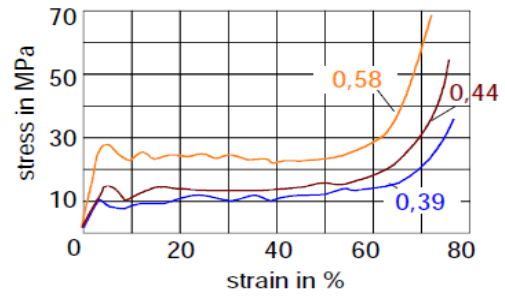
In case of solid metals-

- First stage is affected by an elastic deformation but in foams plastic deformation can happen at low stresses.
- Due to the homogeneous plastic deformation the plateau is produced.
- The sharp increase (3) is produced by breakdown of the cells; opposing cell walls start to trace each other.

The plateau stress in case of metal foam is the main property because tensile strength of foams is nearly the same as the stress, where plateau stress occurs. Due to the large middle region of stress strain curve of metal foam or in other words the long plateau large amount of energy absorbed by metallic foam at low stresses. Density and choice of matrix alloy affecting the behaviour of foams in the compression test.as shown in the fig 1.18. With increase in density plateau stress decreases but it is also to be seen that lower density having lower plateau stress. That means the strength increases with increasing density.



Al99,5-foam (MEPURA; TU Wien)



AlSi6Cu-foam (IFAM, Germany)

Figure 1.18: Stress-strain-diagrams for aluminium foams with closed cell walls of several densities and different alloys. [Catriing Kammer, 1999]

Mechanical properties are also depends on the content of alloying elements. As shown in fig 1.18, pure aluminium foam having lower values than the distinct alloy foam. For open or closed cells the plateau stress is approximately same, if the compression is done between the same alloy foam. Mechanical properties are also affected by the surface of the foam. Dense casting surface has higher stress as compared to foam without dense casting surface. Dense casting surface is also called casting skin. The reason for this is that the casting surface acts as a stiffening component equivalent to a sandwich structure. The same cooling rate is not touched in the complete foam due to this reason quenching is difficult. Water cannot be used as cooling agent, because it would terminate the cells compressed air is used as quenching agent, which leads to lower cooling rates

1.5.2 Strength–Absorption of energy: Dense casting surface gives a higher strength to the foam material. Stiffening effect is the main reason for the higher strength. By using

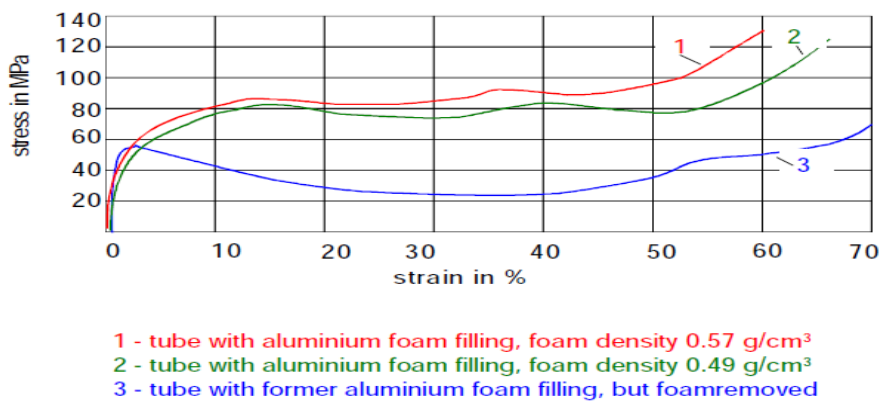


Figure 1.19: Stress-strain-diagrams for tubes with or without aluminium foam. [Catriing Kammer, 1999]

solid aluminium sheet or aluminium plates that are occupied with aluminium foam can also rise this stiffness. When the tubes are filled by aluminium foam they can achieved a higher stiffness and inelasticity. The comparison of compression test results on many tubes with or without foam filling as shown in Fig. 1.20. Plateau stress in case of filled tubes is more than in the hollow tubes. Also plateau stress is longer in case of filled tubes. The energy absorption capacity depends on plateau stress as discussed in mechanical properties. Therefore energy absorption capacity of filled tube is more than the without foam filled tubes. For this characteristic filled tubes are used as crash absorbers for cars. Also filled tubes required less space because filled tube with smaller diameter can absorb same energy as the hollow larger tubes absorb. This is the great advantage for the automotive industry.



Figure 1.20: Several types of tubes and profiles - filled with aluminium foam. [Catriing Kammer, 1999]

1.5.3 Modulus of elasticity

The specific modulus of solid aluminium is much higher than the aluminium foam. Solid aluminium having specific modulus is about 25Gpa/cm^3 for a density of 2.7 g/cm^3 while that of specific modulus of aluminium foam is about $0.6\text{ Gpa/cm}^3/\text{g}$, for a density of 0.4 g/cm^3 .

For some special application the modulus can be adapted by regulating the density of the foam.

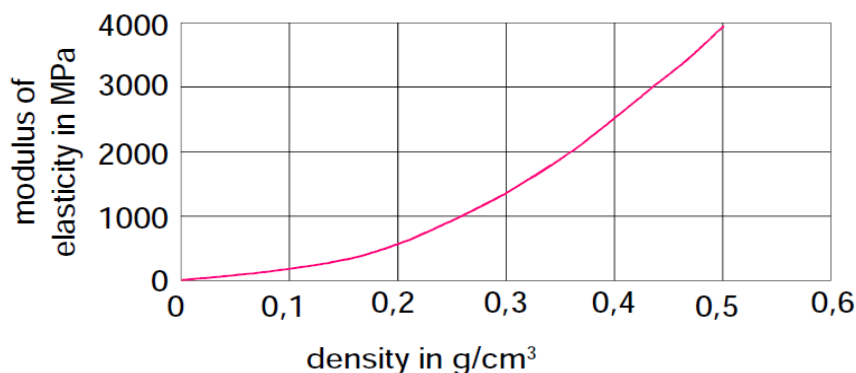


Figure 1.21: Modulus of elasticity of aluminium foams. [Catriing Kammer, 1999]

1.5.4 Physical properties

From the solid metals physical and chemical properties of metal foam are different. But these differences can give benefits for many applications.

a) Conductivity

The conductivity of metal foams is mostly lower than the conductivity of solid metal. In metal foam metallic cell walls share the lowest foam volume as compared to gas filled pores. This is the main reason for lower the conductivity of metal foams. As the density of metal foam increases conductivity also increases. Thermal conductivity of metal foam fabricated by metal casting is nearby one- tenth that of solid metal of same alloy. But thermal expansion coefficient for the solid metal and metal foam unchanged; due to this metal foams will not drop their structure up to high temperatures. Metal foam also acts as thermal insulating material because of their low thermal conductivity.

b) Sound-proofing properties

In metal foam uneven porous structure is present. The sound waves are reflected by uneven porous structure. Due to this it act as sound proofing from the solid metal dissipation factor is much lower. The heat energy produced from vibrational energy which cause minimal collapse of the pore walls. Therefore intensity of returned sound decreases. When the uniformity of pores and very fine pores are there, sound absorbing degree is increased. Open cell walls leads to improved sound absorbing than closed cell walls. Extensive investigations of the sound absorbing coefficient of aluminium porous metal showed that it has a sound absorbing coefficient equal to glass wool.

1.5.5 Chemical properties

Aluminium foam is fireproof. Due to heat, poisonous gases do not discharge foams. The corrosion behaviour is comparable to that of solid aluminium alloys, but further surveys will deliver more information.

1.6 Applications

Metal foam is a combination of mechanical and physicochemical properties. The combinations of such properties are not presently enclosed by other materials. Metal foams can be possibly be used in many structural applications. Structural applications are including the energy absorption and explosions impacts. Metal foam having light weight, due to this reason metal foam has wide applications. Now, a day's aluminium foam can become exciting materials for applications in the automobile industry and in aerospace. Synthetic materials are also light weight but if metal foam is compared with synthetic materials then metal foams

have special advantages. Because foams are comparatively costly, their use is interested by an interesting sum of their specific properties.

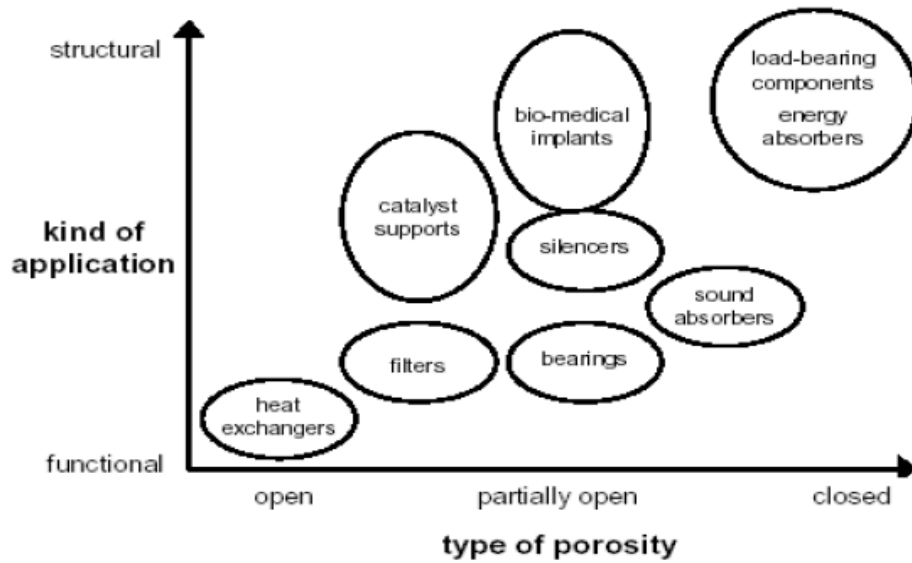


Figure 1.22: Application areas of metal foam, according to the type of porosity [Banhart, 1999]

But it is to be expected that the price of foams will decrease in the next years, as the volume production increases. Therefore, one may classify metallic foams as the materials of multi functionality. They are also recyclable and nontoxic, which make them more attractive materials. In Fig. 1.22 some potential applications of the metal foams as function of open, partially open and closed pores are shown. While open cell foams are mostly preferred for functional applications, closed cell foams are suitable for structural applications.

1.6.1 Automotive applications

In automobile industries main focus is to advance the safety of cars, which is mostly affected by the selection of materials and design of the car. Also essential are all features of material reprocessing. Metal foam can become more useful due to its three main applications under the automobile sector- energy absorption, light weight structure and insulation.

1. First application under this section is showed in the case of crash absorbers against side and frontal impact. However, deformable energy absorbing elements are already includes in the vehicle structure. These elements have to absorb the collision energy. These energy absorbing elements can be filled with aluminium metal foams. This is essential especially in the case of compact city cars, in which the energy absorption to such level is difficult task because of the less space available.

2. Lightweight aluminium foam is used in automobile vehicle body sheets are design parts. Due to the relation between weight and stiffness foams are used in the large area. There is no elastic deformation in these parts caused by the air pressure.

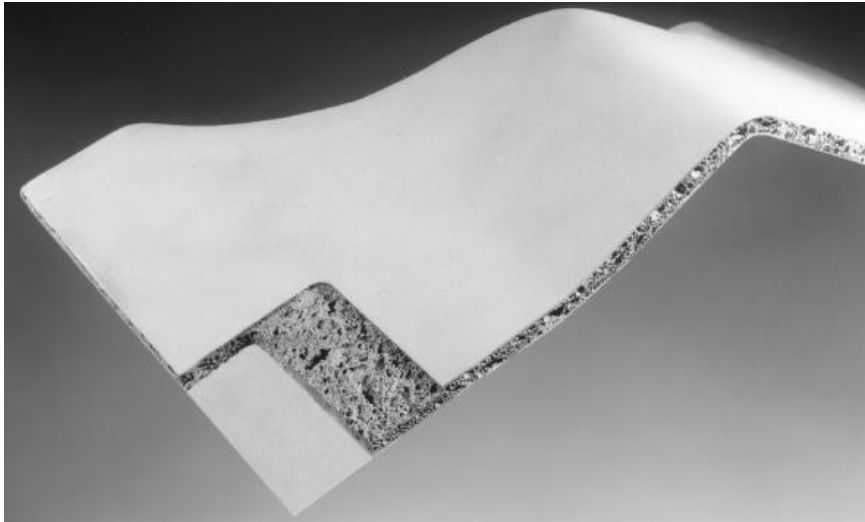


Figure 1.23: Aluminium Foam Sandwich. [Catring Kammer, 1999]

3. Sound absorbing is belonging to the third group of application in automobiles industries. The sound absorbing properties make foams valuable for a noise insulating wrapping of the engine section of cars. The main objective in this case, is to freeze the noise or in other words prevent from noise transfer between environments to the passenger compartment.

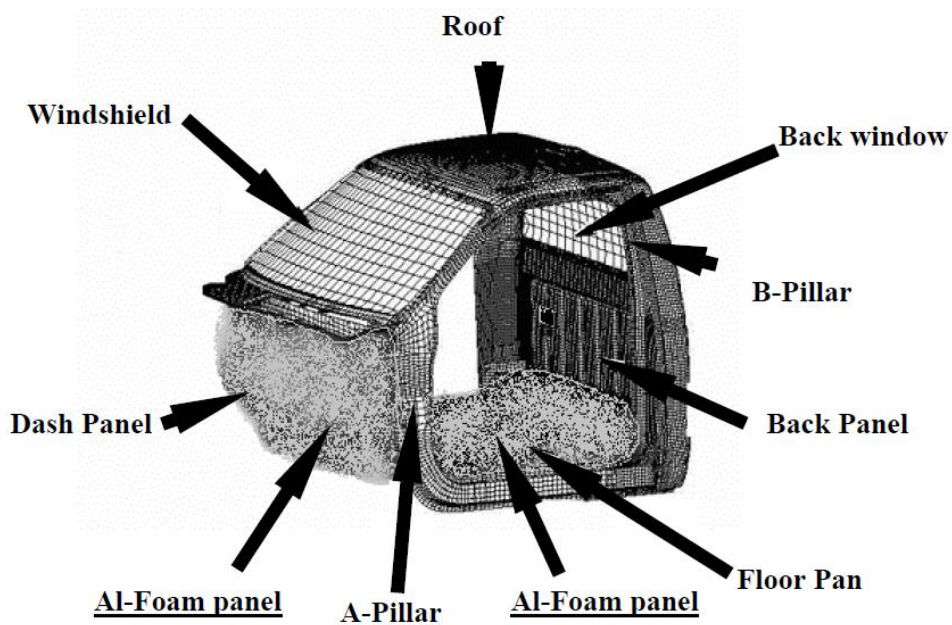


Figure 1.24: Application parts of aluminium foams in a car cab.

1.6.2 Metal foams in aerospace applications

Aluminium foams are mostly used in aerospace due to its light weight. Due to this reason heavy honeycomb structure has been replaced in many applications. The main advantage of using Al foam is that it reduces cost of the part. Besides this, isotropy of this foam is better and there is no adhesive bonding in this. This property is useful when aerospace gets fired. But there is an issue of fatigue in panels which has been described in this study.

1.6.3 Metal foams in ships

Light weight material is also important in ships construction. If ship properties are compared with car, it was found that a high flexible material processing part is required because ships are not made in large amount and not with standardized parts. So Al foams can be used in this case. Previous studies have been done on Al foams in salt water and corrosion properties were checked. It was found that for closed cell NaCl solution comes in the upper layer of the foam and does not harm the foam body.



Figure 1.25: Ship's wall Al foam panel coatings for noise attenuation

1.6.4 Metal foams in building and construction

There are many advantages of using Al foams in building and construction because it has good thermal insulation properties and it can avoid fire penetration. These foams can be used in both inside and outside region of the building. In both cases, these foams act as an energy saver due to its good thermal insulation properties.



Figure 1.26: Sound absorbing structure on the under-side of an elevated via duct
[Catring Kammer, 1999]

Excellent acoustic insulation is another reason for using Al foams in building .Al foams have been used in rail door because it has a good sound absorbing capacity so it absorb the sound of tunnel. Alporas foams under the bridge which absorbs the noise of surroundings as shown in fig 1.26. A sound absorber has also put over noise reflecting surface, due to this outside noise can be avoided in the building. Al foams made light weight elements in the structure which can be used in mobile bridges. These foams reduce the energy consumption of elevate due to its light weight. It is very easy to handle and does not require any mechanical instrument to lift it. It can be used in ceiling, walls and roofs.

1.6.5 Metal foams in the household and furniture industry

Due to its easy handling, it can be used for different house hold parts like table, lamp, house article and different accessories of house. It provides the better combination with the wood and makes the room more watchable. Due to its light weight characteristics, it is often used in trade fair.

1.6.6 Flame Arresters

For stopping flame propagation in combustible gases, cellular metals with high thermal conductivities have been used. The open cells of these foams can stop the propagation of flame when flame velocity is around 550 m/s. Generally, pipes carrying combustible gases are protected from the possible sources of ignition so that, if ignition does occur, the flame cannot accelerate to high velocities. They can also be used in blast protection applications.

1.6.7 Silencers

In industry of powder metallurgy, different dampening components are used which reduce the pressure pulse and mechanical vibration. Material containing porosity can be used to avoid these vibrations for a certain frequency level due to change in pressure. After that level, they pass the vibration. So these foams are useful in compressor and pneumatic devices. In modern period, invest cast foams are used due to its cost and effectiveness.

1.6.8 Heat Exchangers and Cooling Machines

Open cell foam has been used in typical and conventional heat exchanger and sinks. In that heat exchanger, fluid is contacted with both open cell and material which required to be cooled. This surface is connected with some electronic power source. There are many advantages of using Al foams like high surface area, low flow resistivity and good thermal conductivity which provide better results in heat sinks. Aluminium foams are sealed or nearly so on all surfaces which interface with the surface of the mould. There is a good advantage of using al foams that it reduces the pressure drop. The examples for Al foams are compact heat sinks for cooling of microelectronic devices with a high power dissipation density such as computer chips or power electronics. Another application field for open cellular materials is transpiration cooling.

Chapter 2

Literature Review

2.1 Literature Review

Banhart and Brinkers [1999] showed that what difficulties arising in measurement of compression strength. In this study aluminium silicon foams were characterised under uniaxial static and cyclic loading. For the compressive strength static compression test was used to obtain the values. Fatigue test on the foam sample was then performed at the maximum stress level. Various difficulties like the static compression strength varies even when the density of foam is same.

Duarte and Banhart [2000] showed that aluminium foam was produced by mixing metal powders and powdered gas releasing blow agents. After mixing the powder pressing them to a foam able precursor material this method is known as powder compact melting method. In this paper author also showed the variation of temperature and volume of metal foam with the means function of time. Conclusion of experimentation is maximum expansion occur between 620 and 700 seconds after the start of acquisition up to height of 5mm.

Banhart [2001] showed that various possibilities for manufacturing metal foams. According to the state of matter like solid, gas and liquid, there are many fabrication methods of metal foam. In various methods like metal gas injection, melting of powder compacts (containing blowing agent), indirect method including investment casting, destructive and non-destructive methods are outlined. Metal foams are fabricated by different methods divided into various group of application and treated according to their purpose for the industrial scale.

Nakazawa et al. [2005] had conducted an experiment on hollow tube without foam filled or with foam filled. A compression test had been conducted on both tubes on universal testing machine and result founded that the tube with foam filled had more compressive strength than the tube without foam filled. Hence the author has proposed the new design shape of crash box to protect the high value of energy absorption.

Ping et al. [2006] purposed that Porous Titanium alloy (Ti6Al4V) scaffold is fabricated with Rapid Prototype technique 3D fibre deposition. 3D computer controlled fibre depositing of Ti6Al4V is a very special feature of this technology; it is used at room temperature to produce a scaffold. The scaffold consisting the layer of Ti6Al4V fibres. These fibres are directionally aligned. In this study, parameters of 3D fibre depositing were optimized. To developed the 3D fibre of Ti6Al4V slurry.

Garrett [2008] showed that Porous titanium scaffolds material was used to fabricate the porous material with the help of Powder metallurgy and Rapid prototyping technique. The characterisations of porous scaffolds were examined by sacrificial wax template. For filling the gap around the wax template powder metallurgy process were employed to generate the titanium scaffold. This gap was filled with titanium slurry. In this process two parameters compaction pressure and sintering temperature were investigated. To optimize the variation in slurry concentration, powder metallurgy technique, these parameters were investigated. The results of this study demonstrate the potential of such scaffolds for use in orthopaedic tissue engineering applications.

Banhart [2008] proposed the various industrial applications of metal foams. Porous material and metal foams are presently the very active research area. Apart from manufacturing processes and development techniques author showed the various application such as light weight structure, biomedical, implants, filters, catalysts and heat exchanger.

Tang et al. [2008] studied the experimental process to fabricate the porous materials by Radio frequency (RF) magnetron sputtering. First step, a polystyrene template under a direct current electric field had been prepared. A kind of uniform porous material was fabricated on a template of polystyrene by RF magnetron sputtering. After adding ethanol and ammonia solution (pH=8) to polystyrene templates, It is seems that alumina porous materials were composed of uniform pores.

Said and Tan [2009] studied the crushing characteristics and directional dependency by the uniaxial compression loading at the rate of 5mm/min. in this study eight specimen of uniaxial compression were used. The load response were examined and related with biaxial compression

loading. Biaxial compression directed in custom check ability. Five specimens in biaxial compression were used. Result showed that higher energy absorption was seen in biaxial compression in assessment by uniaxial compression loading.

Yadroitsev [2009] studied that the Fine structured 3D porous filter element is fabricated with the help of Selective Laser melting technique. In this paper relation between laser operation parameters, composition and microstructure of the obtained fine porous structure are discussed. The influence of the major parameter such as speed, laser power was studied. The obtained result could be successfully implemented in chemical and biomedical applications.

Klinter et al. [2009] had conducted an experiment using replication casting process for fabrication of open pore aluminium foam, such as foam shape with porosity 60%. In this process blowing agent NaCl is used and poorly soaked by melted aluminium, which lead to development of air collapse. The equation for minimum pore radius of aluminium foam compared with the replicated aluminium foam resulting on the basis of the bottleneck model, which shows that experiment can be functional effectively to fabricate porous casting for noise reduction and filtering.

Jianlue et al. [2010] had conducted an experiment on measuring the value of compressive strength of aluminium porous foam at different strain rates. It has been well established that aluminium porous foam is a strain rate sensitive material. The compressive test conducted over the universal testing machine varying the strain rate from .001 to 220 per second, with each test conducted at constant strain rate. Results showed that decreases of densification strain with strain rate.

Kevorkijan et al. [2010] investigated viability of foaming agent CaCO_3 powder and TiH_2 . Metal foams were prepared by CaCO_3 powder as blowing agent. For this comparison there were two methods- Powder metallurgy and the melt route. Precursor obtained by the melt routes were machined and additionally cold isostatically pressed. The quality of foam able precursor was examined by their initial density and foaming efficiency. In addition quality of foam obtained was characterised by their density, microstructure. **Results-** the experiment finding confirmed

that aluminium foam synthesized with CaCO_3 powder as blowing agent can be prepared by both metallurgy and the melt route.

Dawood and Nazirudeen [2010] suggested that metal foams exhibit different characteristics when compared to their solid material counterpart. In the past history when a large dense material any kind of pores, it was considered defects. In this paper author had conducted an experiment and developed a porous grey iron casting using casting techniques. Density, porosity percentage, microstructure, SEM analysis, compression all are studied. The developments of porous grey iron casting fabricated by sand ball method.

Florek et al. [2010] studied that how non uniformities in foam structure, structural affect the stress strain curve in compression. Metal foam compressive strength test under universal testing machine had been conducted and found out that stress strain curve not smooth and expected plateau stress is missing. Moreover curve always exhibits lot of peaks with locally dropping stress. This research is therefore to explain this effect by conducted experiments on 100 samples. It has been shown that structural and no uniformities in foam structure affects the stress strain curve.

Wang and Zhou [2011] showed that aluminium foam is fabricated by direct foaming techniques in which TiH_2 powder as an additive to the molten metal for foaming. In this foam cell size 2mm and relative density of 0.09. A sample size $100 \times 100 \times 15 \text{ mm}^3$ prepared for universal testing machine indentation testing. A 50KN instron 5569 universal test is used to static indentation test. In this experimental study diameter of indent is 12mm (2a) that is 6 times cell diameter. Relatively small radius chosen because it was desirable that the contact between indenter and specimen considered as point load. to minimize the effect indenter lubricated with PTFE spray. Indentation was good because indentation strength of aluminium foam is larger than the uniaxial compression strength because the work done in tearing cell walls around the peripheral of indentation and because of friction.

Yuqun [2011] studied that Fabrication of porous glass microspheres by using Hydrofluoric acid and sodium hydroxide. In this hollow glass microsphere is placed in shaker with hydrofluoric acid of 1ml for approximate 20 min. after that the resultant hollow glass microsphere are washed

in distilled water and then filter the washed HGM . Then porous hollow glass microspheres were then dried in oven at 100°c. In this method we formed that the regular formation of pores are present smaller than 500nm. The wall structure changes from solid to spongy one.

Aboraia et al. [2011] showed that aluminium foam were fabricated with different densities using different percentage of calcium carbonate as a foaming agent. The foam materials were tested on universal testing machine. The parameters Plateau stress, strain densification and energy absorption capacity U, were calculated. By these results author concluded that collapse strength and absorbed energy foam increases with increasing the density.

Khodai et al. [2011] studied the effect of casting volume, foam rate degradation and melting point temperature on mold filling structure and purposed that these parameters have significant effect on mold filling structure. Two parameters gas gap length and gas generation are considered as important. These two parameters are investigated by photography technique. Results of these parameters showed that by increasing coating thickness and density of foam gas gap length and mold filling time increased. The gas gap length measured in aluminium was approximate 4-5 mm and gas generation was about 50 cc/g.

Vivanco et al. [2012] purposed a new method to fabricate the porous material. In this method tricalcium phosphate (TCP) which is bioactive material is used in injection moulding. Mainly focus on the two parameters material and architectural design. These two parameters play critical role in strength of bone scaffold. The cost effective fabrication process is sintering temperature on architectural and mechanical properties, which controlled the macro porous bio ceramic bone scaffold. Enviourmental testing conditions are also under the observation and had a significant effect on compressive strength, but only at lower sintered temperatures.

Tirta et al. [2012] suggested the method of fabrication the porous material for micro component application. The method was Direct X-Ray lithography and sintering. In this surfactant with composition 50% mixed with steel powder with 3mm diameter at a speed of 30 rpm for 1 hour. After mixing the slurry it was casted onto silicon wafer and adjusted to 500µm. X-ray lithography varies with 25GeV acceleration voltage during the process. After this it can be seen

100 μm in width and 550 μm in thickness gives this achievement can be promising in some application.

Guocheng [2013] found out that the Red Mud Sintered porous material is manufactured by casting technique. For removal of fluoride a porous material (Red Mud Sintered Porous Material) was prepared. Zirconium hydroxide can be better adhered to three dimensional space of the RMPM. RMPM is prepared to search the quantity of Zirconium hydroxide. The kinetic of adsorption of fluoride is slow down by pore diffusion.

Xia et al. [2013] fabricated the AZ31 magnesium alloy metal foam by metal foaming method using CaCO_3 and Ca as blowing agent and thickening respectively. In this paper author investigated the influences of porosity and pore size on the quasi static compressive properties of the foams. The results showed that the energy absorption capacity, energy absorption efficiency and yield strength were decreased with the increase in porosity. However the specimens from where the results had been taken with the porosity 60%, 65% and 70% were possessed similar total energy absorption capacity and absorption efficiency. Results showed that mean plateau stress was increased first then decreased with increase in pore size.

Yavari [2014] found out that porous Titanium biomaterial Ti6Al4V is manufactured with selective laser melting technique. Porous titanium is used in orthopaedics application to mimic the properties of bone. Anodizing is done to improve the bioactivity of porous titanium structure for bone grafting application.

Shutian [2014] studied that Fibrous Material (optimal sound absorption under set frequency) is prepared with the help of mathematical model. A mathematical model was constructed to obtain the microstructure design of porous fibrous material, radius and gap is process parameter. Fibre gap: with increase the fibre gap the sound absorption increases till its peak value then it decreases gradually. Fibre radius effect: with less value of radius (10 μm) the sound absorption is good from this for a given thickness of porous fibrous material layer, the analytical relationship between fibre radius and optimal porosity under set frequency band is constructed.

Wang et al. [2014] purposed the fabrication of porous CuO nanoplates –film by oxidation assisted dealloying method. In this XRD, EDX, UV-Vis testing is carried out for a detailed characterization. It is observed that as fabricated CuO nanoplate films present a porous structure with high porosity with large surface area. In this pure target of aluminium and copper were mounted on radio frequency and direct current sources respectively. Before deposition a pre sputtering of both targets for 30 min was performed to remove the oxide layers. After this a rough surface is generated. Average thickness and diameter of nanoplates are 20 nm and 300 nm.

Khan [2014] purposed that SDP (sintering and dissolution process) is used to synthesize aluminium based open cell foam. After the synthesis of foam, foam has been studied by SEM (scanning electron microscope). In this paper two parameters were studied (compressive strength and absorption capacity). Results show energy absorption capacity increases with increase in strain rate and compressive strength increases with adding of alloying agent.

Tatsuya et al. [2014] had conducted an experiment to fabricate the porous material by anodizing in squaric acid. A high pure aluminium foil was anodizing in a 0.1M squaric acid solution at 293K and a 100-150 constant potential applied. Author was found that an oxides layer build up on aluminium foil at applied potential but it burnt out at high potential. Result had been found out that cell size of anodic oxide increased with increase in anodizing time.

Negi et al. [2015] showed that aluminium foam was prepared by the method of melt route that means melting the material by addition of nickel particle additives. Different compositions are used to find out the best conditions of producing better quality of aluminium metal foam. Different samples foamed by different composition of nickel testing under universal testing machine having load capacity 400KN. The result shows that different percentage of nickel particles affect the compressive strength of the material foam.

Rajak [2015] showed that shape of sample is important factor. Comparison of the energy absorption rate per unit volume according to the shape of samples; means the experiment were performed on universal testing machine the results showed the round cross section had more energy absorption than the rectangular and square cross section separately.

Chapter 3

Problem Define

3.1 Problem formulation

From the extensive literature survey, it has been found that porous material fabricated by traditional method like fused deposition method, Selective laser sintering, Metal casting, Radio frequency magnetron, Hydrofluoric acid and many more, but not much research has been done by conventional method like in which tool actual touches the specimen during the fabrication process. So the new problem is defined fabrication of porous materials by conventional drilling. By this process open pores will be fabricated.

For the metal foam, the extensive literature survey has been found out that closed pore metal foam has wide application either in industries, home appliances or engineering field also. Metal foam has been fabricated by melt gas injection, gas releasing particles, hollow sphere structure, gas metal eutectic solidification, but strength related to densities is the main focus area during all the process. So the new problem formulation in this case is comparison of energy absorption with compressive strength of aluminium foam.

3.2 Objectives:

- To maximize the porosity of metal foam
- To maintain the uniform porosity for some application
- To decrease the process cost
- To increase the energy absorption capacity
- Define the relation between energy absorption and stress strain curve.

Chapter 4

Experimentation

4.1 Introduction

Porous materials and metal foams are now a days the focus of very active research and development activities. There are presently near about 150 institutes working on porous metal foam worldwide, some of them focusing on characterisation of metal foam, some on properties, some on applications and some on fabrication methods. According to their applications there are many methods to fabricate the porous materials and metal foams, like laser beam machining, Fuse deposition machining, Metal casting, Red mud sintering, Conventional drilling, Powder compaction and many more. In this chapter porous materials are fabricated by conventional drilling and metal melt casting techniques. Conventional method is selected because of some of its application which requires uniformity of pores in the material. Metal melt casting method is selected because of its properties like light weight and energy absorption. The following steps are followed for fabrication the materials are shown in table below:

4.2 Conventional drilling Method

Conventional Drilling is a type of cutting process in which drill or cut a hole of desired cross section, with the help of drill bit. Drill bit has multi cutting point and it is a rotary cutting tool. The drill bit rotated at rates of thousands of rpm (revolution per minutes). When drill bit touches the work piece it is pressed against the work piece. Work is done by cutting edge forces against the work piece, which results a hole are produced.

For the purpose of drilling of minimum diameter of hole as such as possible CNC (Computer Numerically Control) milling machine has been selected, in which adapter BT40 and Collet, which hold the drill bit diameter from 1mm to 3mm has been used, and a program has been prepared with the help of computer numerical control codes (G & M codes) for drill a hole through the sample.

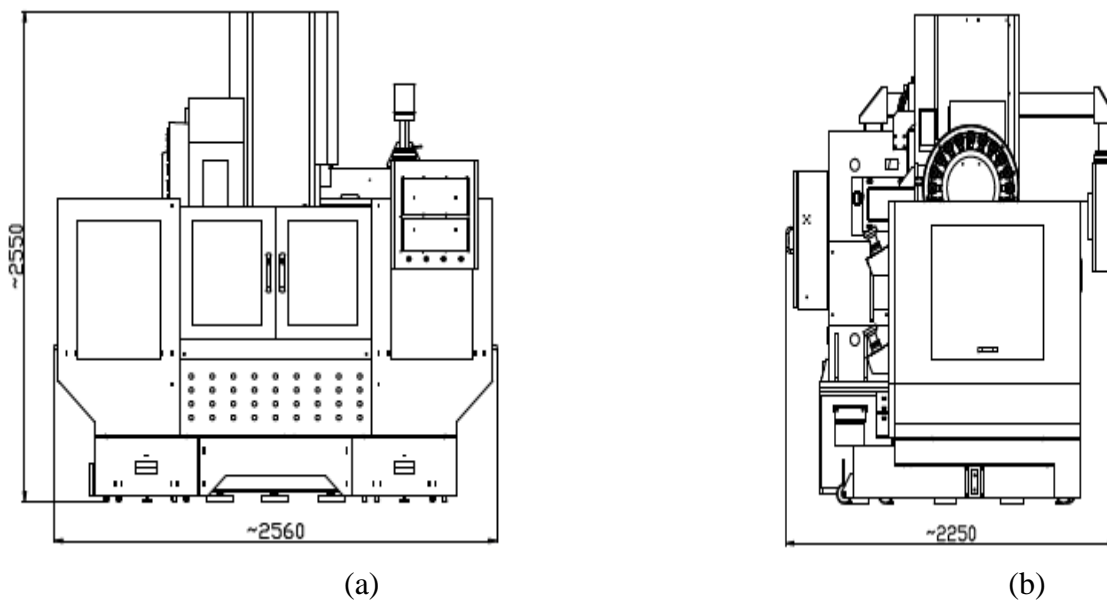
4.2.1 CNC Vertical Milling machine:

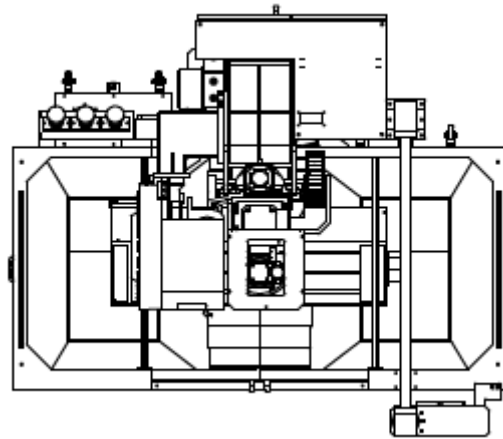
(a) CNC Machine Overview:



Figure 4.1: CNC vertical milling machine (BFW Chandra+) [W.1]

(b) Design of machine





(c)

Figure 4.2: (a) front view (b) side view and (c) side view of CNC milling machine.

(c) Technical specifications

Table 4.1: Detailed specification of CNC milling machine

| Parameter(Units) | Specification |
|---|---------------|
| Spindle Speed(rpm) | 60-6000 |
| Axis feed rate(mm/min) | 1-5000 |
| Vertical Movement: Z-axis (mm) | 380 |
| Transverse Movement: Y-axis (mm) | 350 |
| Table top-spindle face distance(mm) | 75-455 |
| Tool changing time: tool to tool (s) | 2.5 |
| Table Clamping area(mm x mm) | 315 x 1060 |
| Traverse Longitudinal Movement: X-axis (mm) | 800 |

(d) Features

- Coolant tank with chip tray
- Pneumatic tool clamping and unclamping
- Air gun
- Job counter display
- Linear and circular interpolation
- Absolute encoders on all three axis
- Automatic centralised lubrication system

4.2.2 Fabrication of porous material by convention drilling

The following steps have been followed during the fabrication of porous material:

A physical prototype of porous materials has been prepared with the help of Fused Deposition Machine (FDM). In the first step, a 3-D solid model is prepared by many CAD package. Due to the common format, all CAD software easily export and reduces the part to the basic components. ProE CAD package has been selected for the 3-D design of prototype. With the help of STL (stereo lithography) format the part had been exported to the FDM quick slice software. STL format reduces the part into set of small triangles. After the STL file exported to quick slice, thin horizontally sliced layer by layer has started. These sliced layers represent two dimensional part that will generated by the FDM. After implemented the FDM process physical model is fabricated. Acrylonitrile butadiene styrene (ABS) had been used in this process to fabricate the prototype.

1. First Step

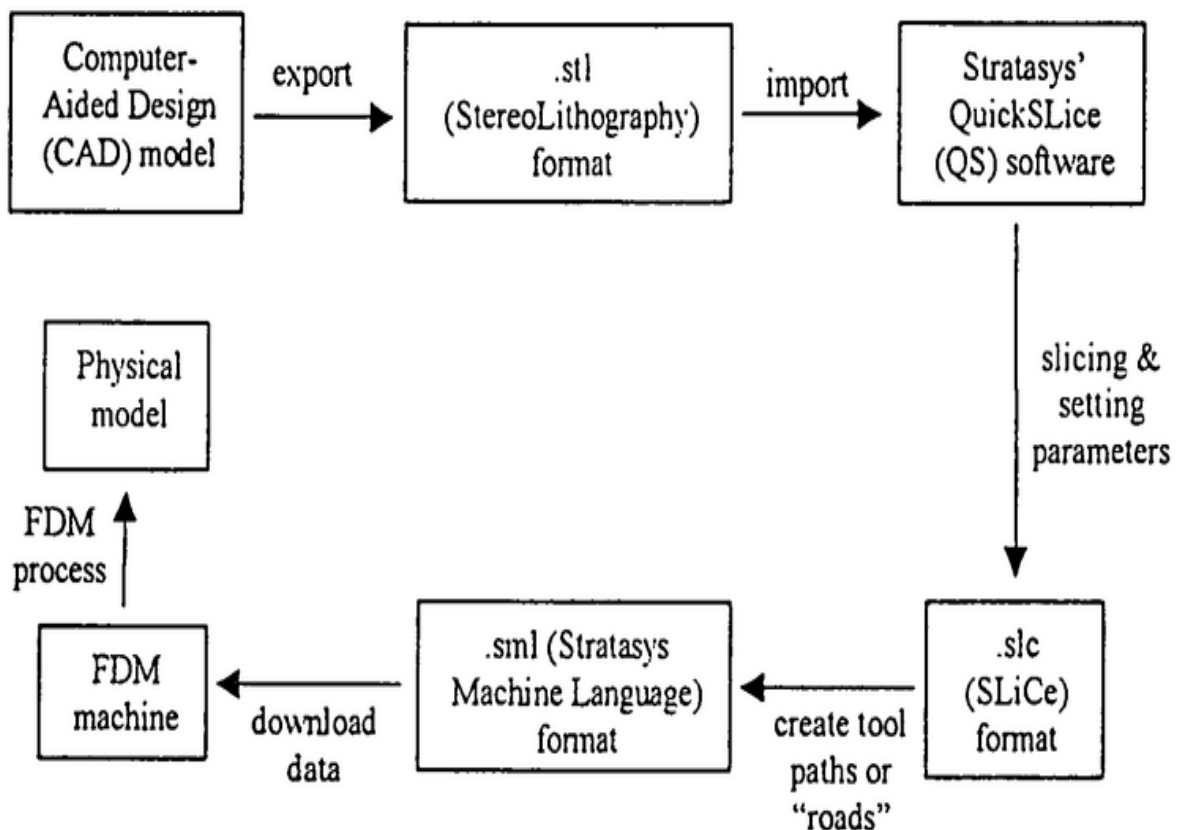


Figure 4.3: Flow chart of FDM process

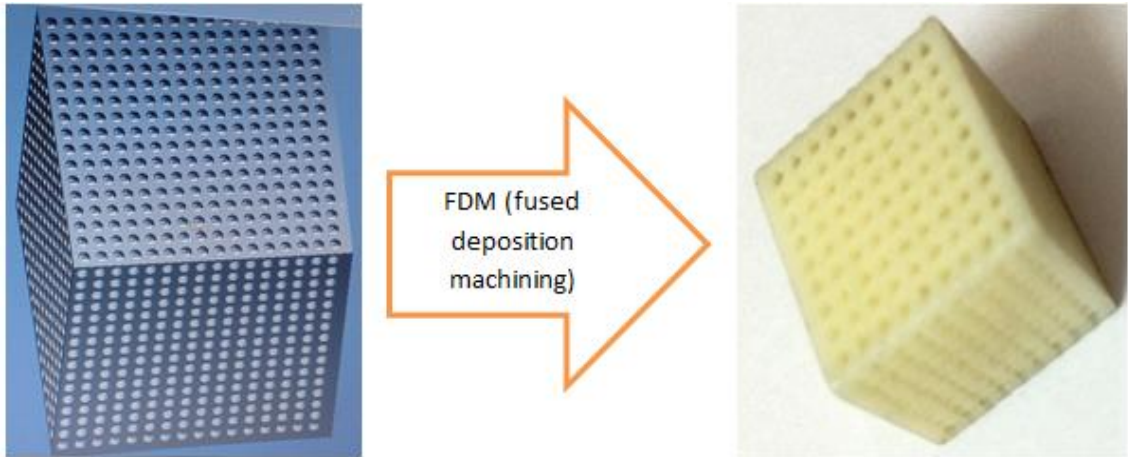


Figure 4.4: CAD model to physical prototype by FDM process.

(a) 2nd Step

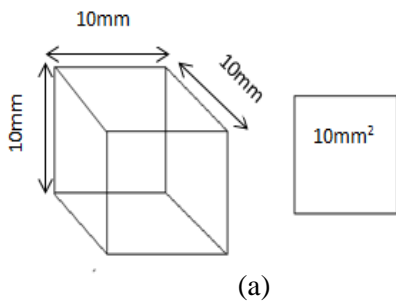
For making a real model of this prototype aluminium and stainless steel material has been selected for their different application, cut the material as per prototype shape of cuboid having dimensions 1mm x 1mm x 1mm and the cross section of square in shape, for both the materials.



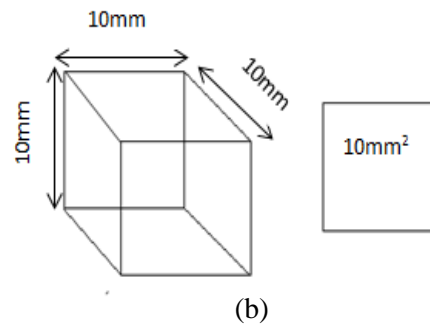
(Aluminium)



(Stainless steel)



(a)



(b)

Figure 4.5: (a) Design of aluminium sample (b) Design of stainless steel

(b) Tool Selection: A tool having more strength than the sample material, HSS (high speed steel) have been selected.

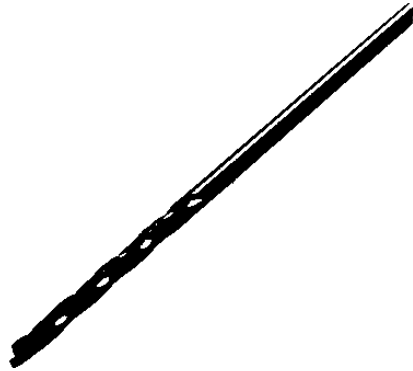


Figure 4.6: HSS tool drill bit of fluid length 10mm

HSS (high speed steel) drills are designed in such a way, that the drills have little bit drift and chatter to start work easily. With this bit drift and chatter, they can provide more accuracy. These drill bits have high cutting speed.

After the selection of material and tool, sample has been hold on vice of vertical milling machine and a program had loaded to drill a hole. Tools have been clamped to vertical drive with the help of adopter and collect by pneumatic clamp.

(c) Program for drill a hole of 1mm throughout the surface

```
(  
  O8899  
  G21  
  G90;  
  M03 S3000;  
  G00 X0 Y0 Z0;  
  G01 Z-10 F10;  
  G01 Z3;  
  M30;  
)
```

The position has been marked on the sample before drill. The process has been going on as one hole drill after the other till the last whole program was run. At last the specimen was ready.

Table 4.2: Parameters that have been consider for stainless steel material.

| S no. | FEED RATE | SPINDLE SPEED | CENTRE TO CENTRE DISTANCE BETWEEN ADJACENT HOLES |
|-------|-----------|---------------|--|
| 1. | 10mm/min | 2500 rpm | 1.5mm |
| 2. | 8mm/min | 2500 rpm | 1.5mm |
| 3. | 3mm/min | 3000 rpm | 1.5mm |

Table 4.3: Parameters that have been consider for Aluminium material.

| S no. | FEED RATE | SPINDLE SPEED | CENTRE TO CENTRE DISTANCE BETWEEN ADJACENT HOLES |
|-------|-----------|---------------|--|
| 1. | 10mm/min | 1200 rpm | 1.8mm |
| 2. | 8mm/min | 1500 rpm | 1.8mm |
| 3. | 5mm/min | 1800 rpm | 1.8mm |



(a)



(b)

Figure 4.7 (a) Pores in Aluminium (b) Pores in Stainless steel

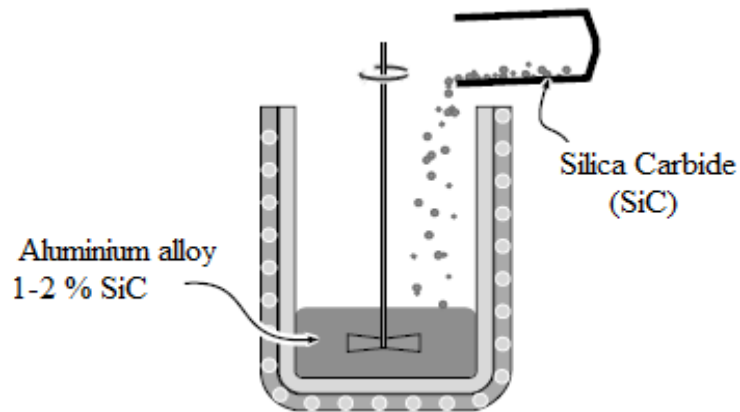
4.3 Fabrication of Aluminium foam by Gas-releasing particle decomposition in the melt:

Metal foam is fabricated by mixing of blowing agent or foaming agent with the metal alloys. These foaming agents when heated above their melting temperature, release gases. Many foaming agents are there like NaCl, $\text{Na}(\text{CO}_3)_2$, TiH_2 and MgCO_3 , but MgCO_3 foaming agent has been used in this process to fabricate the metal foam. MgCO_3 when heated above 465°C , it decomposes into Mg and CO_2 gaseous. By adding magnesium carbonate particles to aluminium melt, carbon dioxide gases in large volume are instantly produced. Due to the gas, formation of bubbles on the surface that can lead to closed cell foam. The process begins, first ceramic crucible having 500 gm aluminium by weight in solid state has placed inside the stir casting furnace. Stabilized the melt temperature inside the furnace is between the range 670°C and 690°C . To raised its viscosity or in other word to increase foam drainage rate, 5-10 % silica carbide and 2-3% calcium carbonate is added. This rapidly oxidizes and forms CaO and SiAl_2O_4 particles. With the help of graphite stirrer the melt is then continuously stirred and 1-2% of MgCO_3 is added uniformly in the melt foam, in powder form. The stirring system is withdrawn from the furnaces, as these are disseminate. After withdrawn the stirring system, foam is allowed to form above the melt. To, totally go off the magnesium carbonate it takes, nearly 8-10 minutes. The melt is then cooled to solidify the foam before the carbon dioxide escapes and the bubbles of gas collapse. Relative density can be determines by adding the volume fraction of silica and magnesium carbonate to the melt. By changing the cooling and foaming conditions cell size can be varied from 0.5 to 5mm by changing the foaming agent content. A subsequent rolling treatment can be used to fracture many of the cell walls in order to increase their acoustic damping. Today, only aluminium alloys are made in this way because carbon dioxide embrittles many metals and because the decomposition of MgCO_3 occurs too quickly in higher melting point alloys. Relative density from 0.2 to as low as 0.07 can be manufactured by this process. While only small volume fractions of silica and magnesium are used, however this process is costlier than gas injection process. A design procedure of the above procedure step by step in shown below:

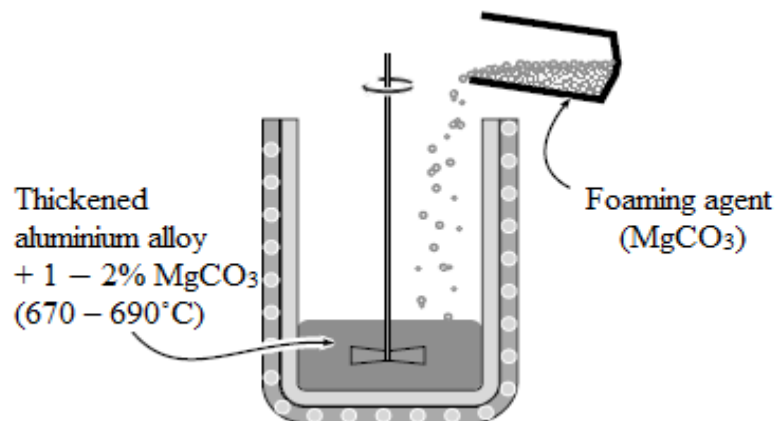
4.3.1 Metal Foams: A Design Procedure

PARTICLE DECOMPOSITION IN LIQUID

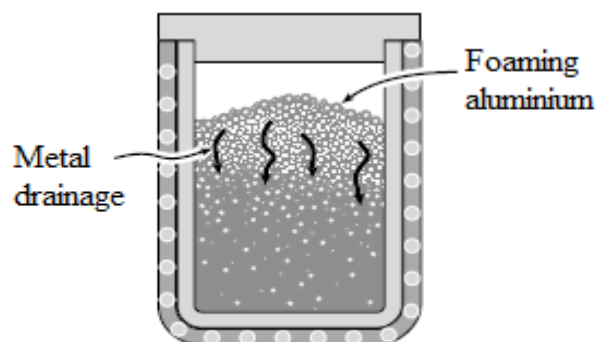
a) Viscosity modification



b) Foaming agent addition



c) Isothermal foaming



d) Cooling of foamed aluminium

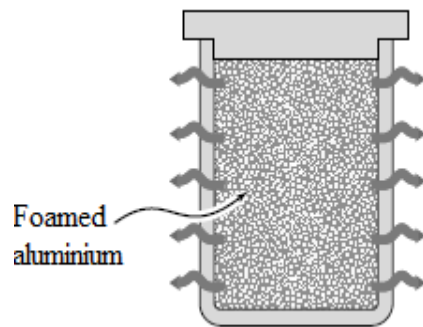


Figure 4.8: The process steps used in the manufacture of aluminium foams by gas-releasing particle decomposition in the melt

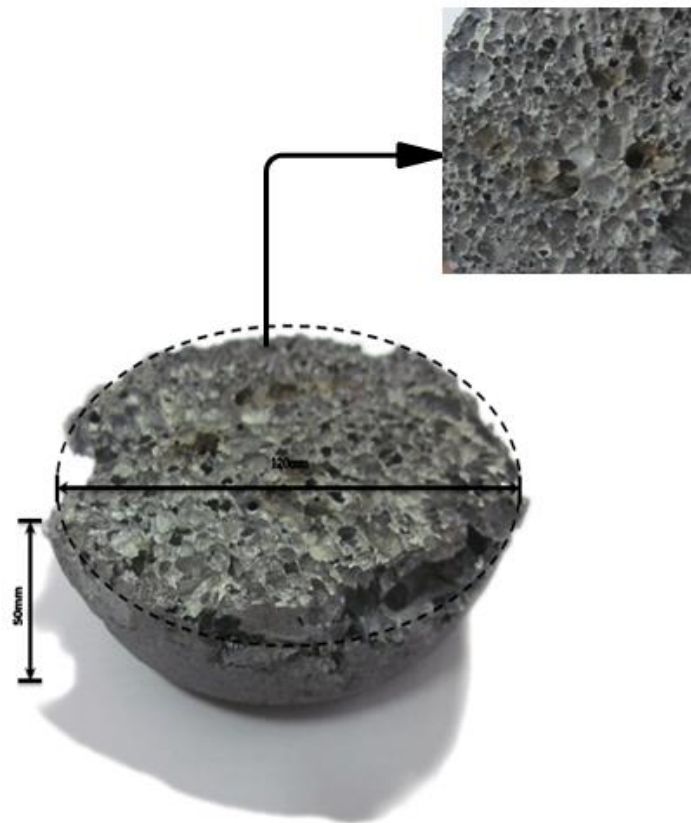


Figure 4.9: Metals foam fabricated by gas injection method and surface of metal foam

4.4 Experimental equipment:

1. UTM (Universal Testing Machine)
2. Scanning Electron Microscope (SEM)
3. Cylindrical Measuring glass (Archimedes principal)

4. XRD (X- Ray Diffraction)

4.4.1 UTM (Universal Testing Machine)

1. Constructions

- (a) Loading Frame
- (b) Hydraulic system
- (c) Electrical system



Figure 4.10: Computerised Universal Testing Machine

2. Computerised Control Panel

(a) Load measuring system

In the cylinder the oil pressure also converted to an electronic pressure transducer which gives a relative signal to the Data Processing System. Push buttons controls the hydraulic and cross head motion of motor and self-locked to avoid the skidding of the Rotary Encoder. Encoder signal is send to the Data Processing System to obtained displacement in mm.

(b) Features of data acquisition unit

- Auto detection of over load, over travel & specimen break. On detection of any of the above conditions, the machine is automatically switched off.
- Tare load & reset elongation facilities available.
- Elongation is indicated with a resolution 0.01mm.
- Storage capacity to save data of one off-line experiment.

(c) Features of Window software

- Variable sample break detection
- The Window UTM software retrieve data from previous test & prepare test report and can run a variety of mechanical tests.
- The software is human friendly.
- Experimental Graphs and data can easily retrieved by the printer or USB port.
- Storage & retrieval of test parameters.
- Built in facility for printing the test results are test graph from Computer. A printer copy the collectively experiment results conducted on a specific date can also be obtained. Graphs of Load Vs Crosshead travel, Load Vs Time, Crosshead travel Vs Time, Load Vs Extension, and Stress Vs Strain etc. are available.

(d) Load rate control

Servo based automatic programmable load rate control which will load the sample at a defined rate up to specified load, Keep it constant up to set time and unload at a present rate. Operating Range: 5 to 100 % of full scale load per minute.

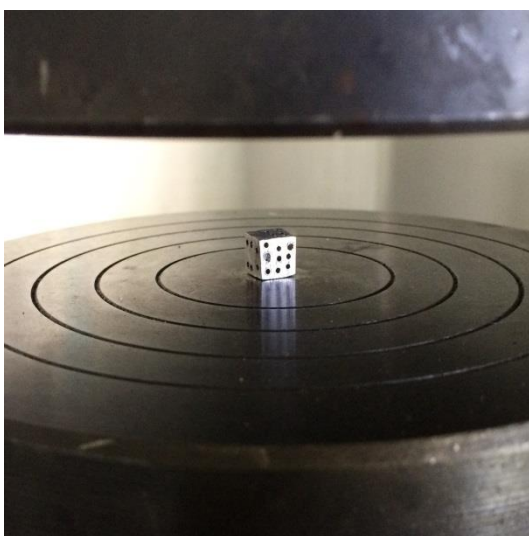
3. Technical specification

Table 4.4: Specification of Computerised UTM machine

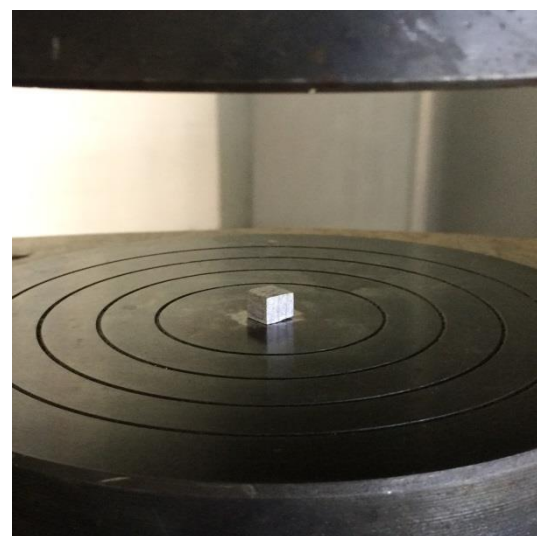
| MODEL | TUE-C-1000 |
|---|---------------|
| Measuring Capacity | 1000 |
| Measuring Range | 0-1000 |
| Resolution of Piston Movement (mm) | 0.01 |
| Over all dimension approx. (mm) | 2350x800x2700 |
| Load Range in KN with accuracy of measurement $\pm 1\%$ | 20-1000 |

4. Process for compressive strength

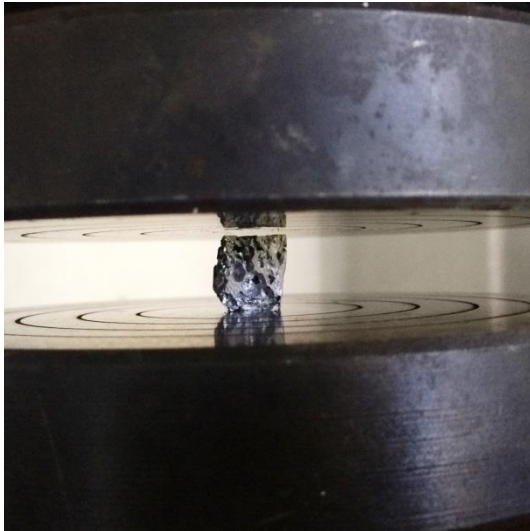
The size of aluminium cubes and stainless steel cube 8.36 mm x 8.36 mm x 8.36 mm and 9.7 mm x 9.7 mm x 9.7 mm had been taken. Similarly to determine the compressive strength of aluminium metal foam fabricated by casting of size 13.3 mm x 13.3 mm x 13.3 mm had been tested. All compression tests have been conducted using 1000 kN compression testing machine. A total number of 4 samples have been tested. The head has been provided at each end of sample. The load has been applied gradually in a controlled manner in increment of 1-1.5 kN by hand pumping of manually operated hydraulic jack. The maximum load at the end of crush material has been taken.



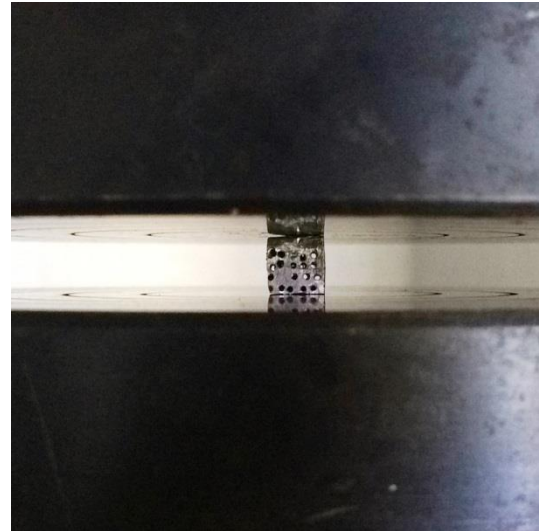
(a)



(b)



(c)



(d)

Figure 4.11: (a) Aluminium with pores (b) Pure aluminium (c) aluminium metal foam (d) Stainless steel with pores.

Compressive strength (N/mm^2) = Ultimate compression load/Area of cross section

4.4.2 Scanning Electron Microscope (SEM)

SEM is a type of electron microscope that generates an image of sample by scanning it and with the help of focused beam of electron. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. With the help of SEM cell wall thickness, break or crack on surface and cell uniformity have been studied, whose results are discussed in next chapter



Figure 4.12: Scanning Electron Microscope [W.2]

Aluminium metal foam has been scanned by SEM. It provided surface structure, internal structure of pores, porosity and material formation point to point.

4.4.3 Cylindrical measuring glass

By the Archimedes principal volume of samples has been measured to find out their porosity. Archimedes' principle indicates that when a body is immersed in a fluid, the upward buoyant force exerted on a body, whether fully or partially submerged the force is equal to the weight of the fluid that the body displaces. For measuring the volume cylindrical measuring glass has been used.

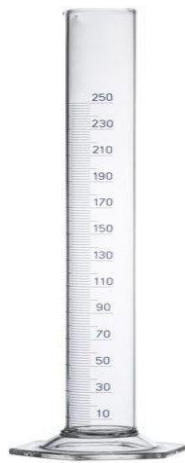


Figure 4.13: Measuring cylindrical glass

To measure the volume of the sample, the cylindrical vessel has been filled up to a fix level after this the sample has been poured to the water fill cylindrical glass. As the sample is poured to glass the water level has been increased by some level that's the volume of the sample.

4.4.4 X-Ray Diffraction

The atomic planes of a crystal cause an incident beam of X-rays to interfere with one another X-Ray Diffraction analysis has been employed for the identification and quantification of the surface. X-RD analysis is a non- destructive technique. X-RD analysis has been carried out to check the presence of an element that might have in AlSi metal foam. Results of this are discussed in next chapter. A diffraction procedure is shown in fig. 4.14.

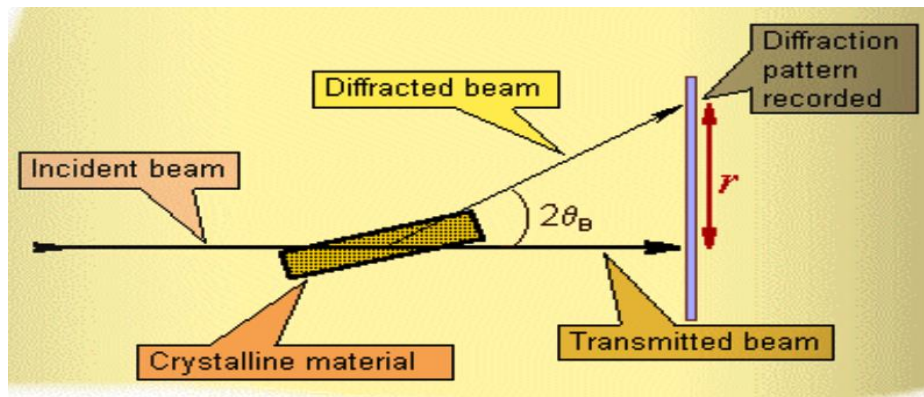


Figure 4.14: X-Ray Diffraction Procedure as they leave the crystal. The phenomenon is called X-ray diffraction. [W.3]

Chapter 5

Results and Discussions

5.1 Results and Discussions

In the following section results about metal foam fabricated by metal casting and porous material fabricated by conventional drilling has been compared and discussed. Two methods has been analysed by these following parameters:

1. Compressive strength
2. Porosity
3. Metallurgical test

5.1.1 Compressive strength

The property by which the capacity of a material and reduce the size by withstand load is called compressive strength. In compressive strength the graph between load force and deformation is plotted. Compressive strength of aluminium metal foam and porous material has been calculated by UTM (Universal Testing Machine).

Porous Aluminium sample, in this case the parameters had been considered in chapter 4 are:

Table 5.1: Parameters that have been consider for pure aluminium material.

| S no. | FEED RATE | SPINDLE SPEED | CENTRE TO CENTRE DISTANCE BETWEEN ADJACENT HOLES |
|-------|-----------|---------------|--|
| 1. | 10mm/min | 1200 rpm | 1.8mm |
| 2. | 8mm/min | 1500 rpm | 1.8mm |
| 3. | 5mm/min | 1800 rpm | 1.8mm |

Experiment no. 1: In this experiment no. feed rate 10mm/min, Spindle speed 1200 rpm, centre to centre distance 1.8mm and the coolant in off mode has been taken. This shows the result, tool is stuck with the sample and no chance of drill a hole.

Experiment no. 2: In this experiments feed rate 8mm/min, Spindle speed 1500 rpm, centre to centre distance 1.8mm and the coolant in off mode has been taken. Which shows tool is again stuck with the sample as shown in the figure no (5.1).

Experiment no. 3: In this experiments feed rate 5mm/min, Spindle speed 1800 rpm, centre to centre distance 1.8mm and the coolant in on mode has been taken. Which shows a hole is drill through sample which has taken cycle time 4 minutes, as shown in the Fig. 5.1.

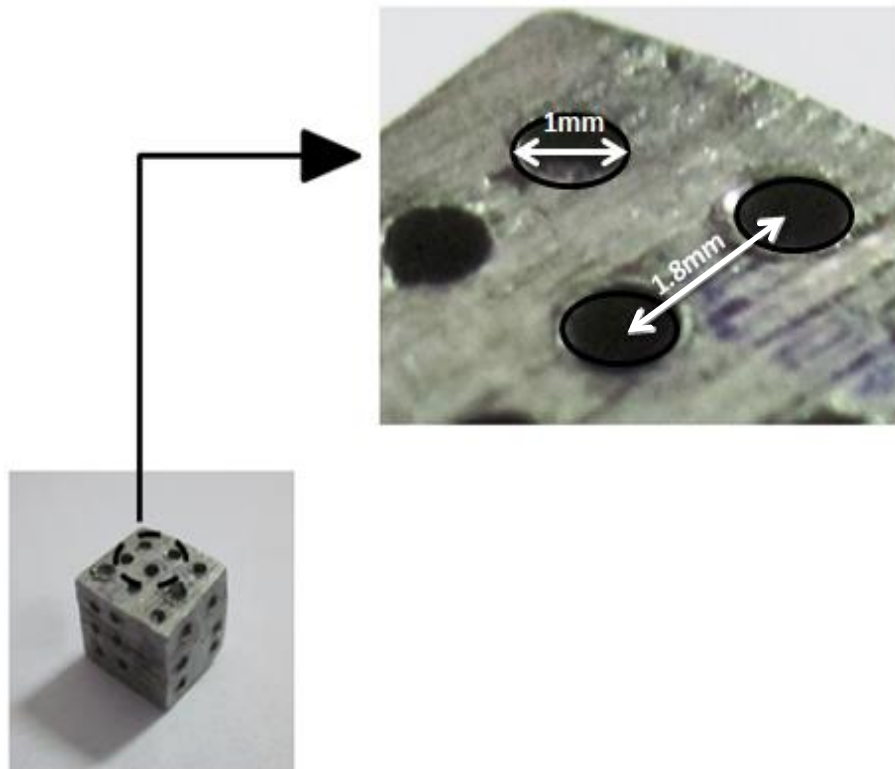


Figure 5.1: Sample of aluminium porous with surface image

In these experiments of method of fabrication of porous aluminium initial off set had also provided to the tool drill bit which was 3 mm above the sample piece in vertical direction, to reduce the cycle time a manual feed control switch was also present on the control panel of the CNC vertical machine. With the help of manual feed control switch, when the drill bit moved in backward stroke, feed rate of backward stroke had controlled by the manual switch, which reduced the cycle time. After the selected parameters as selected in the third experiment, sample of aluminium porous fabricated with pore size of 1 mm and constant centre to centre distance 1.8 mm as shown in Fig. 5.1. The pores generated by drill bit of 1 mm were throughout the surface and on all the six faces of the cubical sample.

1. Compression test on metal foam

Metal foam show a characteristics compressive stress- strain curve composing of three distinct sections; Linear elastic (I), Collapse (II) and densification (III).

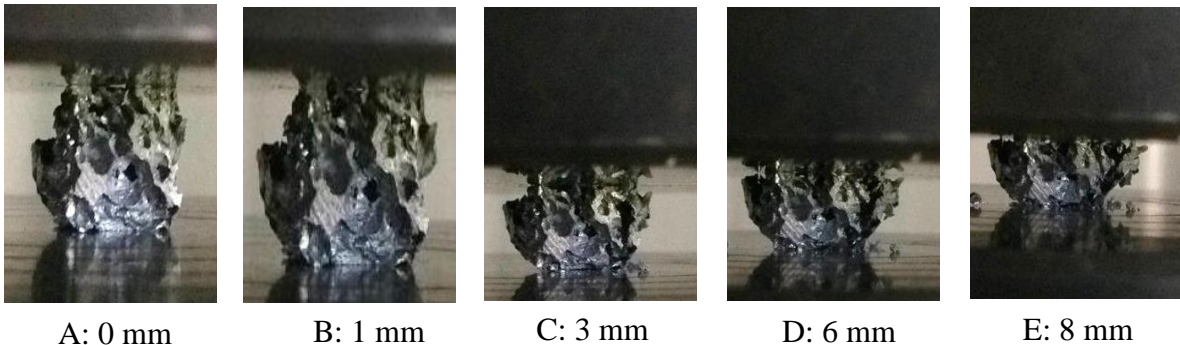
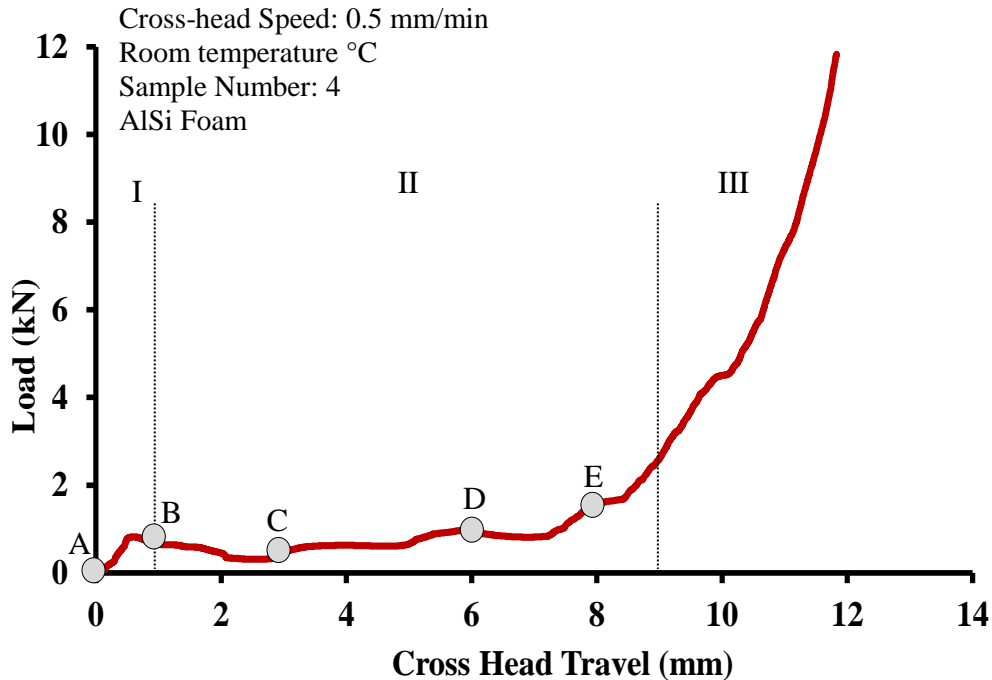


Figure 5.2: Compressive behaviour of aluminium foam and cross head travel over the aluminium foam

First with low strain rate, the foam deforms elastically and deformation is controlled by pores wall bending. Then after this the deformation is followed by a collapse region occupying by many mechanism like elastic buckling and brittle crushing of pores wall. In this region deformation goes from localised to un-deformed region of the sample. Plateau stress characterised the second region which is collapse region and shows constant value with slightly increasing with strain. At the critical strain, sample collapsed totally after which the

third region called densification, as shown in figure. In this figure: (A) shows the cross head is at initial stage, (B) shows cross head travel 1mm occupied the load less than 1 kN, (C) shows cross head travel 3 mm with approximate same load conditions, (D) shows cross head travel 6mm again same load condition with slightly increase in strain rate and (E) shows 70 % deformation at 1.8 kN load after this densification region started. These all deformation with load conditions shows a constant Plateau stress generated through collapse region, which means energy absorption capacity is present in metal foam.

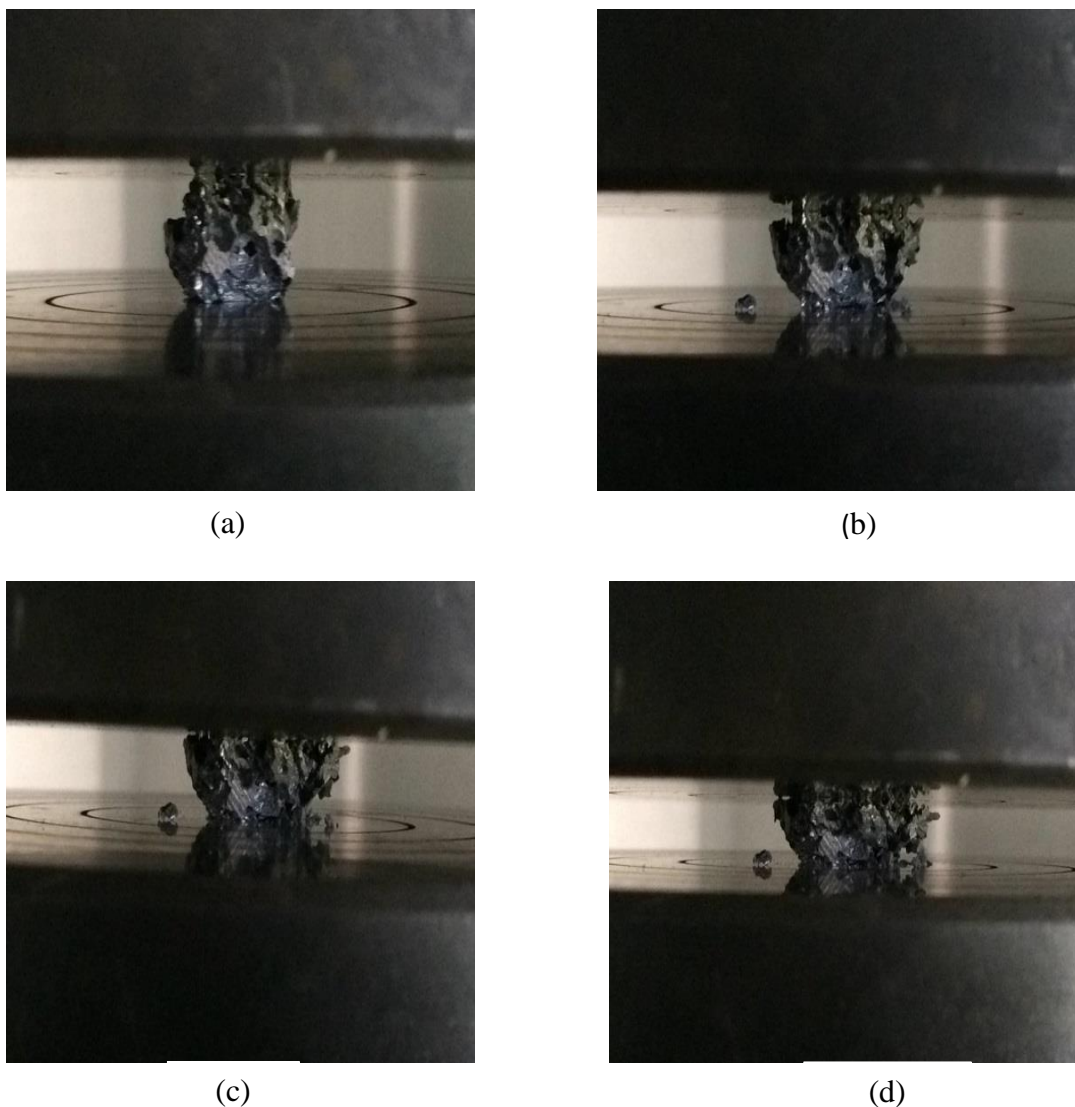


Figure 5.3: Progression of concertina mode of deformation in Al foam at (a) 0%, (b) 20%, (c) 35% and (d) 50% deformation ratios.

2. Compression test on porous aluminium:

Porous aluminium also shows a characteristics compressive stress- strain curve composing of three distinct sections; Linear elastic (I), Collapse (II) and densification (III). In the porous aluminium, Plateau stress of stress strain curve characterised the second region which is collapse region and shows increasing curve with strain. At the critical strain, sample collapsed totally after which the third region called densification, as shown in Fig. 5.4.

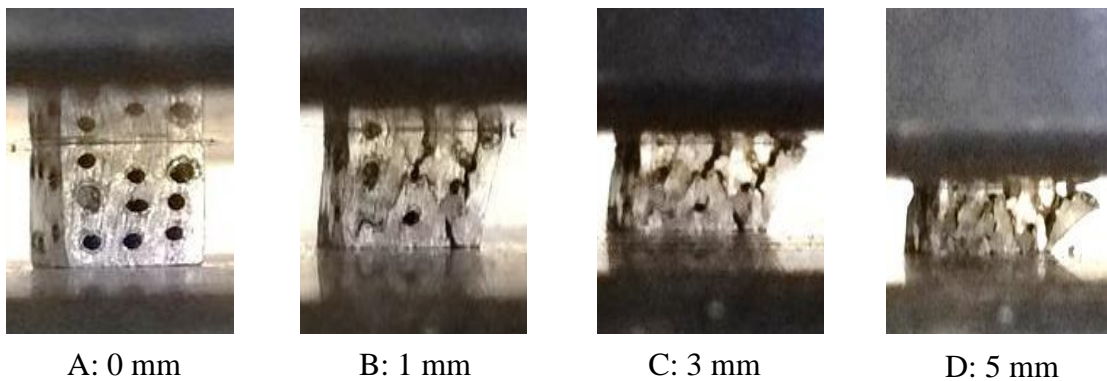
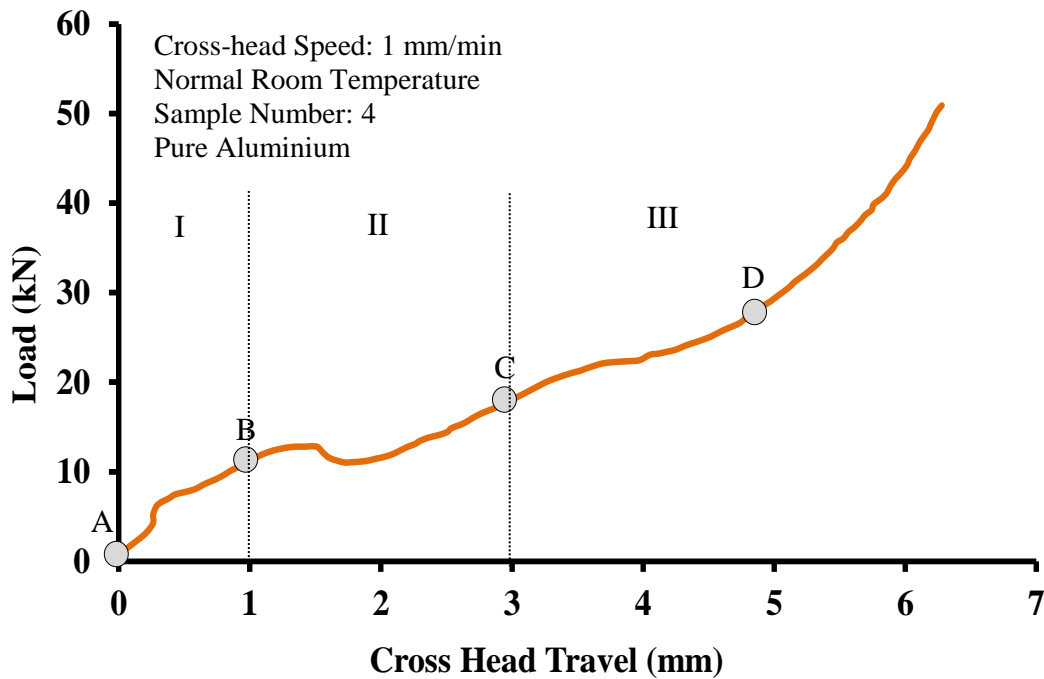


Figure 5.4: Compressive behaviour of porous aluminium and cross head travel over the aluminium porous

In this figure: (A) shows the cross head is at initial stage, (B) shows cross head travel 1 mm occupied the load less than 10 kN, (C) shows cross head travel 3 mm with different load conditions and (D) shows cross head travel 5 mm increase load condition with increase in

strain rate, after this densification region started. These all deformation with load conditions shows an increasing curve of Plateau stress generated through collapse region, which means less energy absorption capacity is present as compared to metal foam

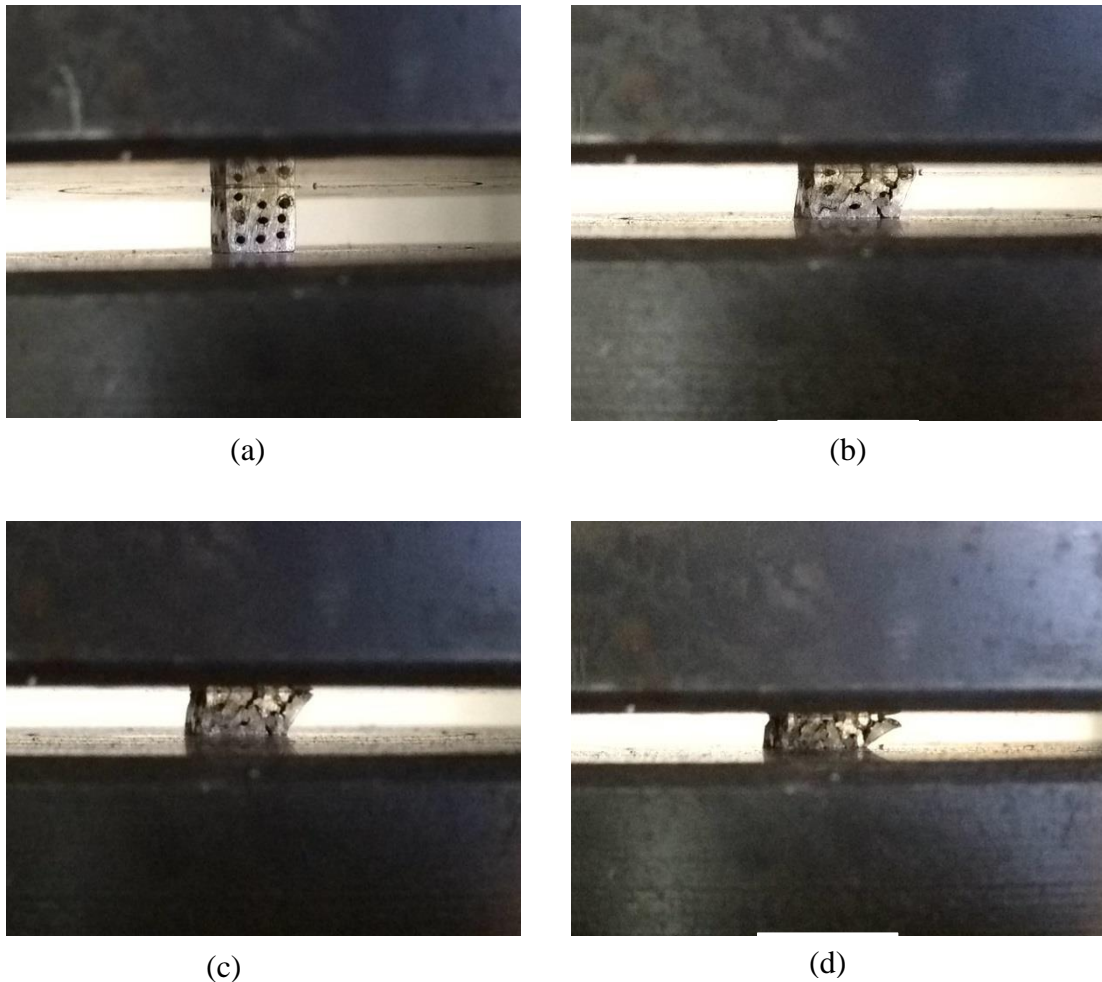


Figure 5.5: Progression of concertina mode of deformation in porous Al 10 mm edge cube at (a) 0%, (b) 20%, (c) 35% and (d) 50% deformation ratios.

5.1.2 Porosity

For measurement the porosity of the samples, Archimedes principal phenomena have been taken as discussed in chapter 4.

1. Metal aluminium foam

Apparent volume of sample calculated by mathematical formula edge x edge x edge mm^3 . The measurement of edge has been measured by digital Vernier calliper, the results show the dimension of edge is 16.6 mm and the volume is 4.574 cm^3 . After the measuring of apparent

volume, volume of sample measured by Archimedes principal, the result gives the volume 1.32 ml.

Volume of sample = 1.32 ml

1 ml = 1cm³

Therefore the volume of sample in cm³ = 1.32 cm³

Apparent volume = 4.574 cm³

Volume of voids = Apparent volume – Volume of sample
= 4.574 – 1.32 cm³
= 3.254 cm³

Porosity = (volume of voids)/ (apparent volume)
= 3.254/4.57
= 0.71

Porosity in % = 0.71 X 100 = **71 %**

2. Porous aluminium fabricated by conventional drilling

Apparent volume of sample calculated by mathematical formula edge x edge x edge mm³. The measurement of edge has been measured by digital Vernier calliper, the results show the dimension of edge is 8.36 mm and the volume is 0.585 cm³. After the measuring of apparent volume, volume of sample measured by Archimedes principal, the result gives the volume 0.216 ml.

Volume of sample = 0.216 ml

1 ml = 1cm³

Therefore the volume of sample in cm³ = 0.216 cm³

Apparent volume = 0.585 cm³

Volume of voids = Apparent volume – Volume of sample
= 0.585 – 0.216 cm³
= 0.368 cm³

Porosity = (volume of voids) / (apparent volume)
= 0.368/0.585
= 0.629

Porosity in % = 0.629 X 100 = **62.9 %**

5.1.3 Metallurgical test on metal foam

1. SEM (Scanning Electron Microscope)
2. EDS (Energy Dispersive X-Ray Spectroscopy)
3. XRD (X-Ray Diffraction)

1. **SEM (Scanning Electron Microscope)** the non-uniformities of pores at different magnification are shown in Structure image capture by SEM. SEM is used for inspecting surface shape and features of specimen at very high magnification. To analysis of cracks and fracture surfaces, porosity, defects on surfaces and cell bond failure SEM inspection is used. For this research acceleration voltage 10kv in order to provide details of surface and porosity as shown in Fig. 5.6 & 5.7.

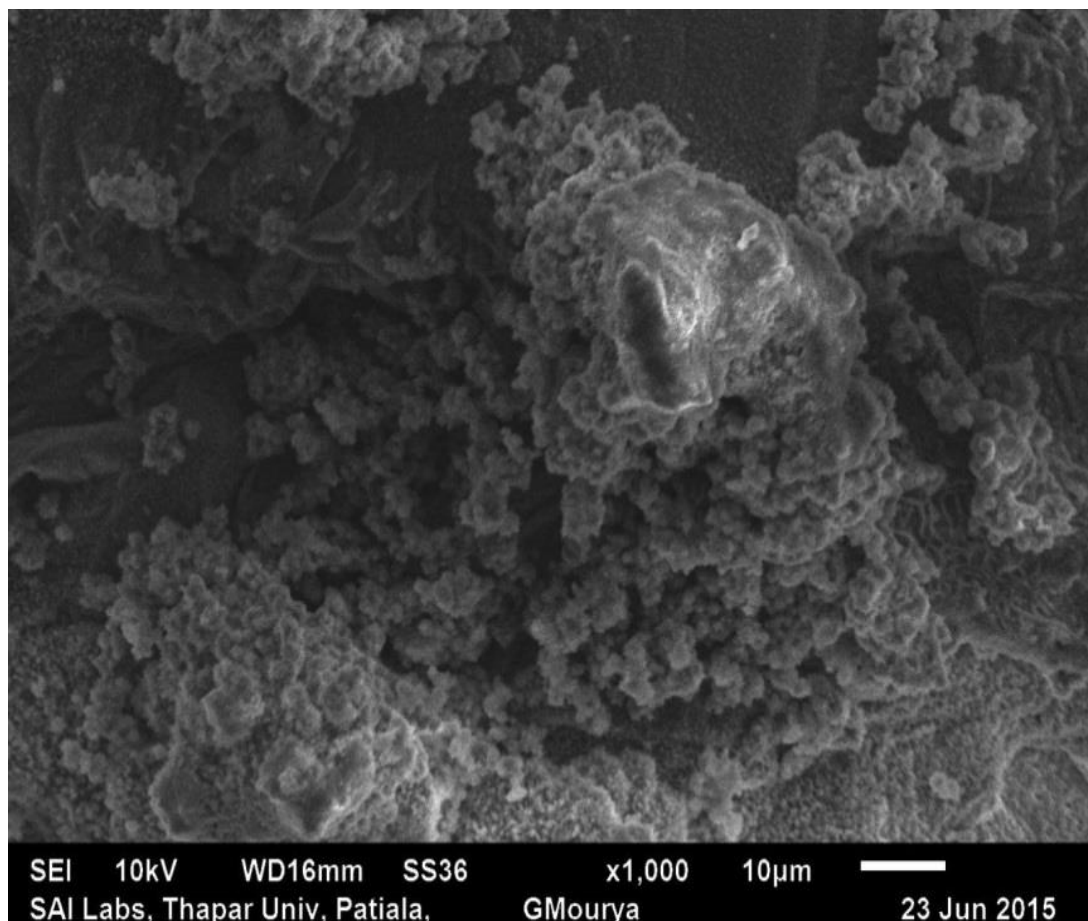


Figure 5.6: Surface structure image of metal foam at magnification 1000X

SEM of surface of foam sample at 1000 X is shown in Fig. 5.6. SEM of porous foam sample at 2500X is shown in Fig. 5.7. SEM can provide high magnification and high resolution image up to magnification 50,000 X.

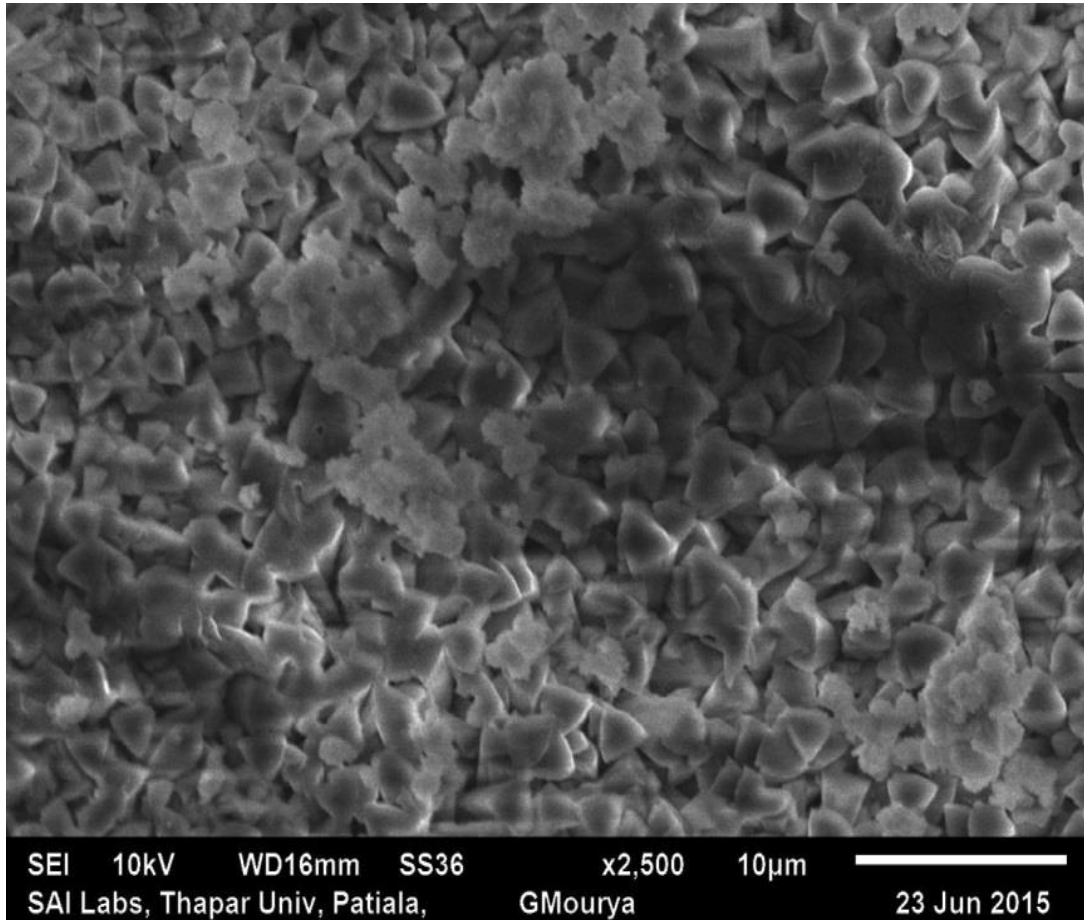


Figure 5.7: Structure image of porosity of metal foam at magnification 2500X

2. EDS (Energy Diffraction X-Ray Spectroscopy)

In the gas melt injection method due to the mixing of blowing agent and alloy, the metal oxides filaments, or the solid component of particular alloy remains in the cell walls and edges. These oxides particles may greatly reduce the thermal conductivity of the foam. Microscopic analyses of cell wall of foam sample also confirm this fact. The boundaries between the actual particles, and matrix generated between the walls is clearly shown in Fig. 5.7. To analyse the different element formed on the surface or inside the pores a line mapping has been done by Energy Diffraction X-Ray. A selected surface for line mapping is as shown in Fig. 5.8. Aluminium and Oxygen has been found in maximum surface area shown in Fig. 5.9(c), this is due to aluminium had the parent metal and solidification of melt aluminium had done in open atmosphere where oxygen mixed with melt aluminium.

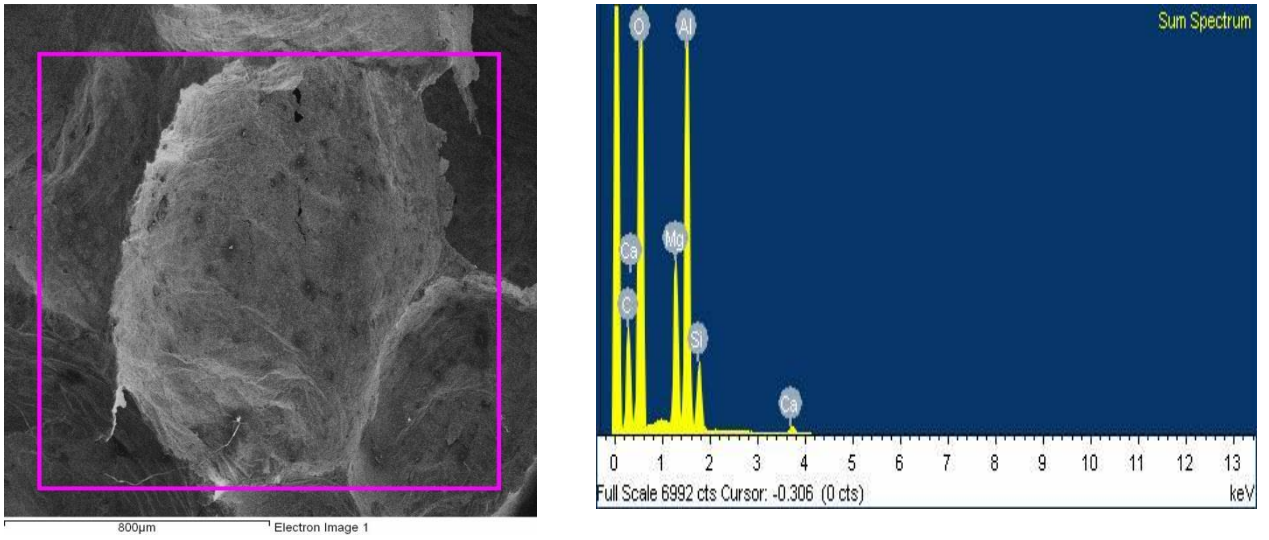
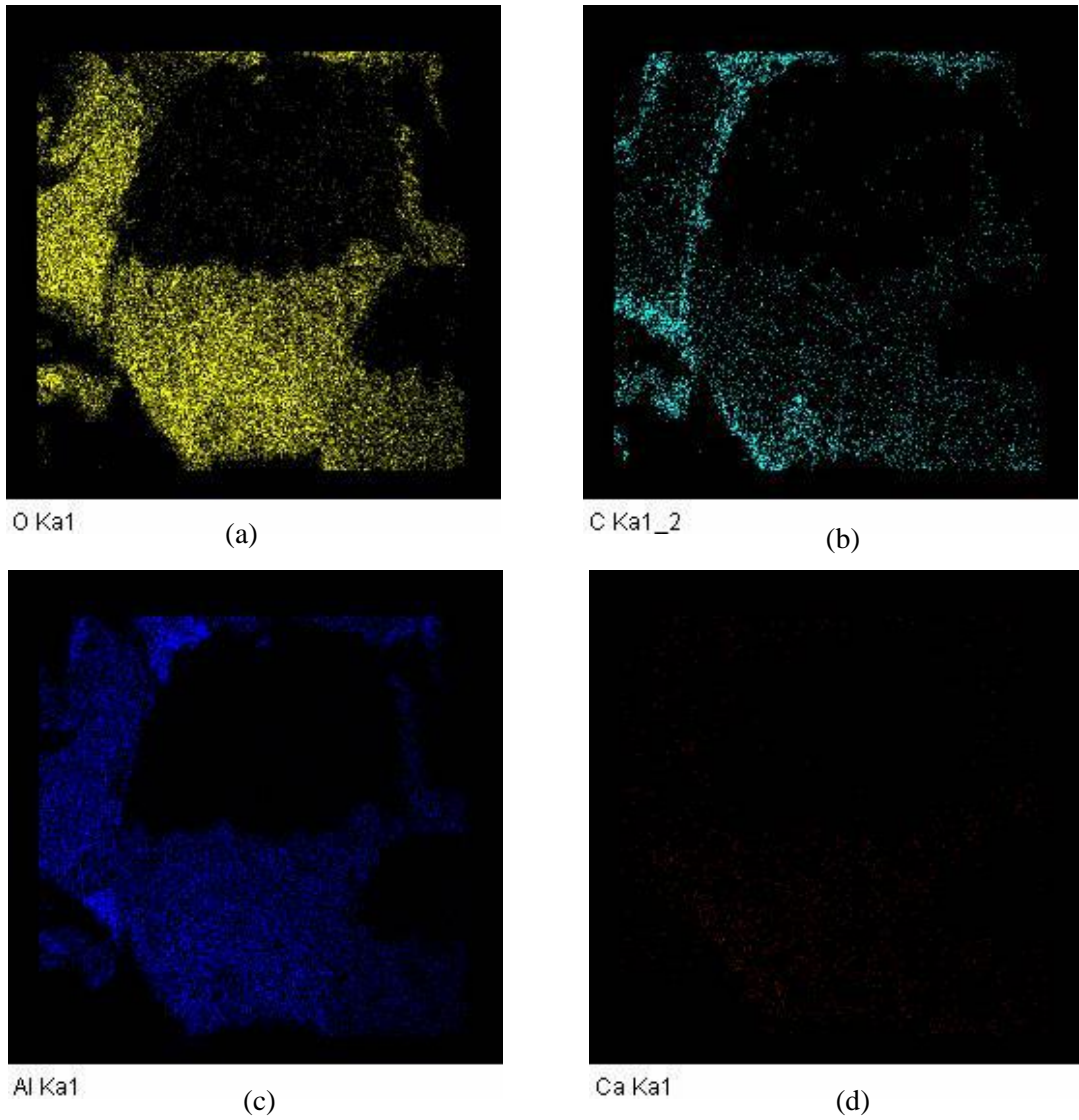
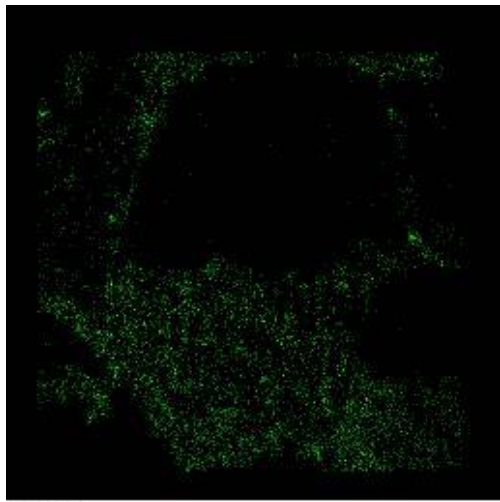


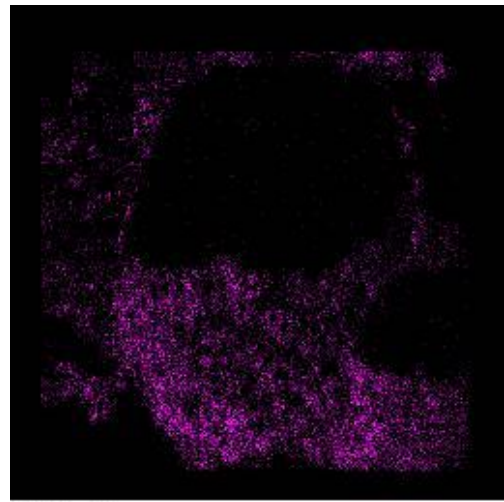
Figure 5.8: Selected surface for line mapping of different components





Si Ka1

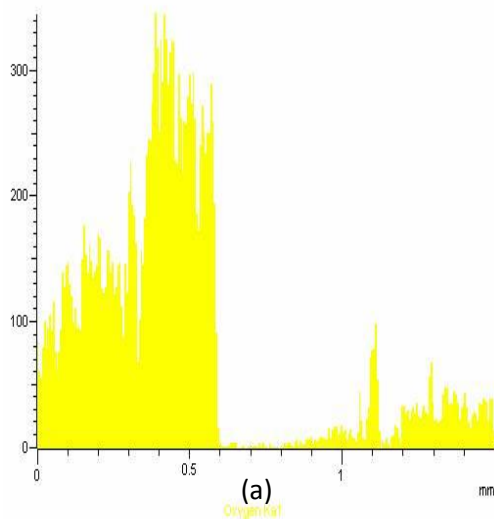
(e)



Mg Ka1_2

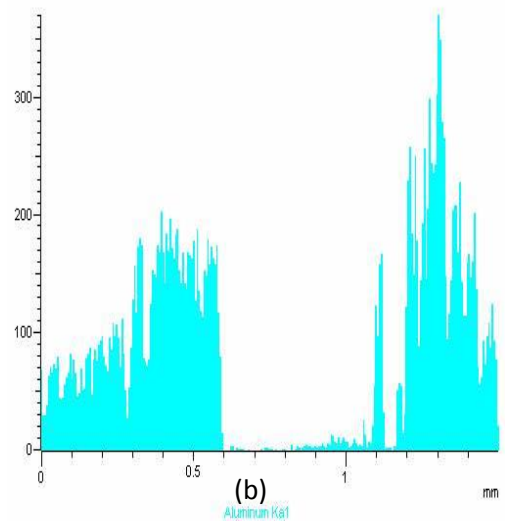
(f)

Figure 5.9: EDS images of different element shows distribution of (a) Oxygen, (b) Carbon, (c) Aluminium, (d) Calcium, (e) Silica and (f) Magnesium over the selected surface



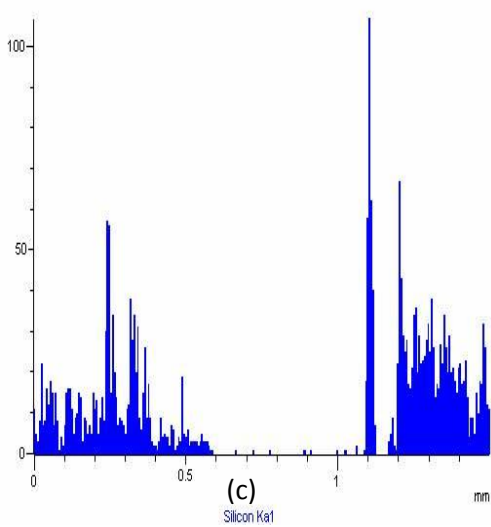
(a)

Oxygen Ka1



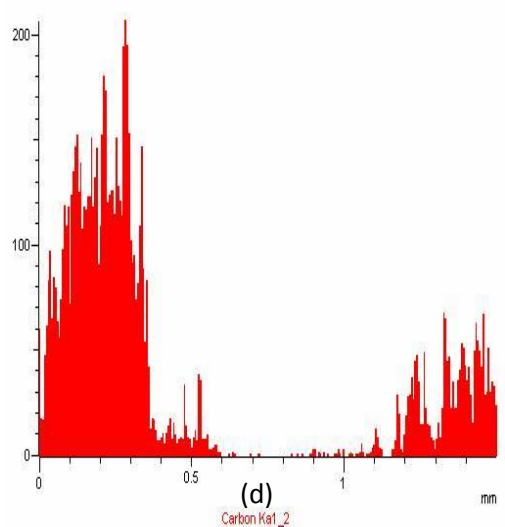
(b)

Aluminum Ka1



(c)

Silicon Ka1



(d)

Carbon Ka1_2

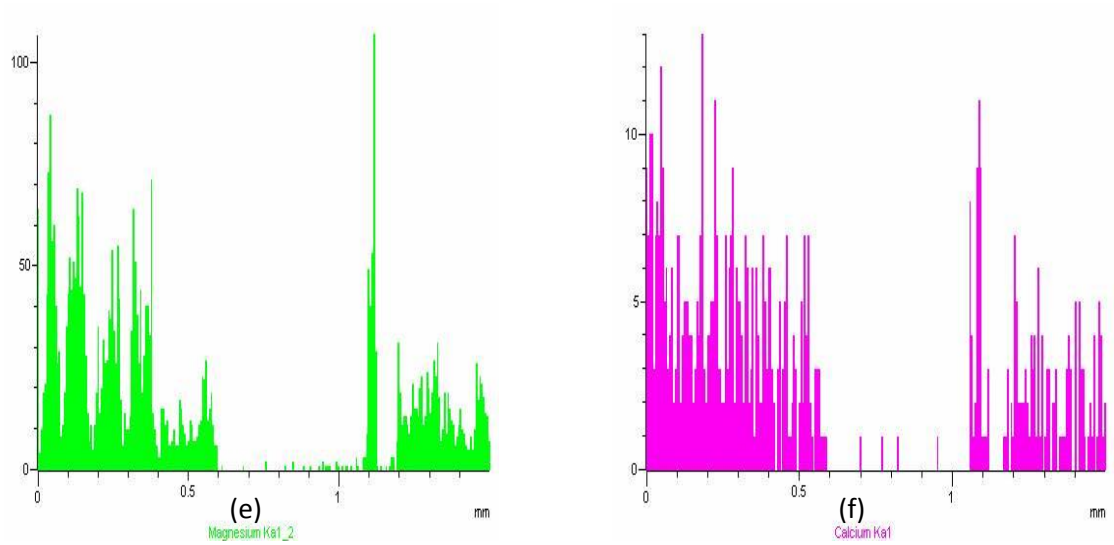


Figure 5.10: Variation of components with distance along the line mapping (a) Oxygen, (b) Aluminium, (c) Silicon, (d) Carbon, (e) Magnesium and (f) Calcium

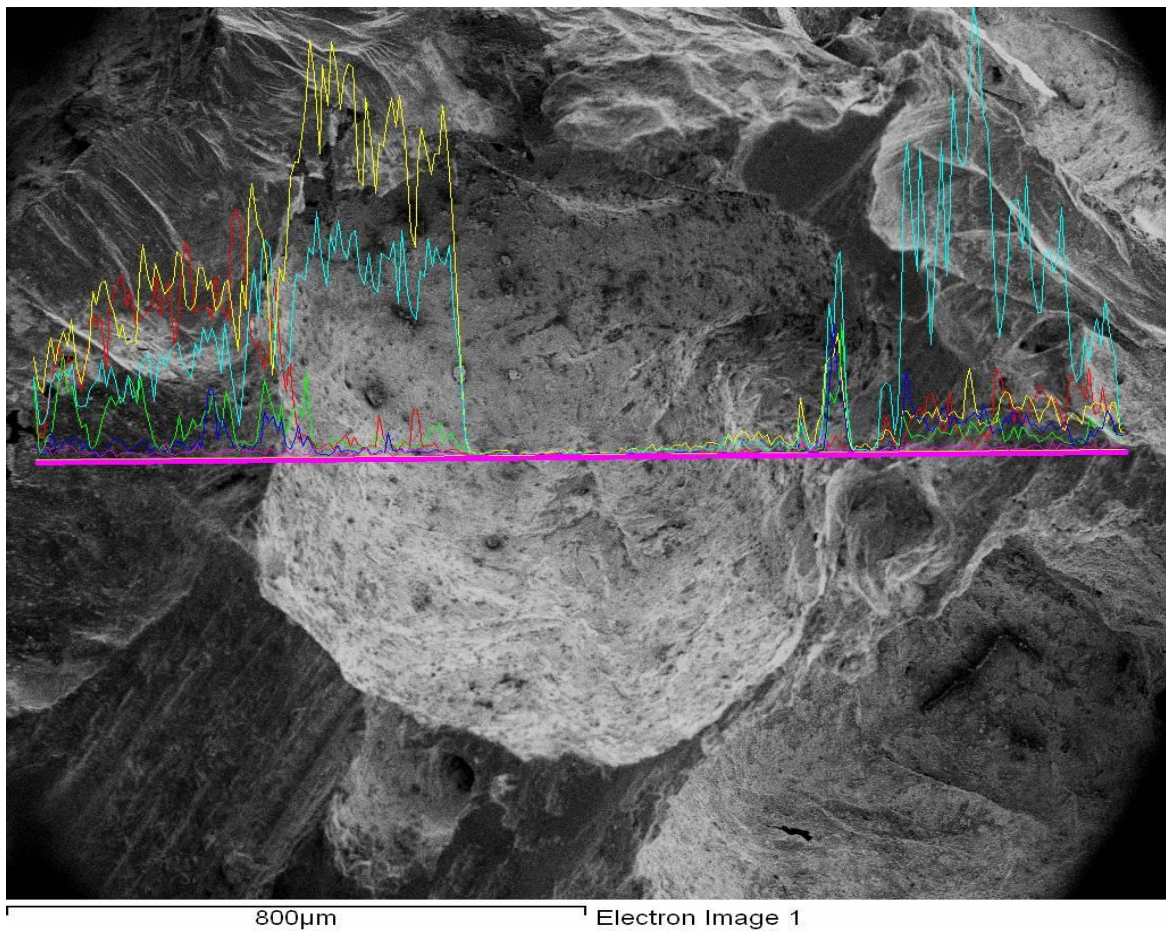


Figure 5.11: Line mapping of variation of the entire element on the selected surface of aluminium metal foam.

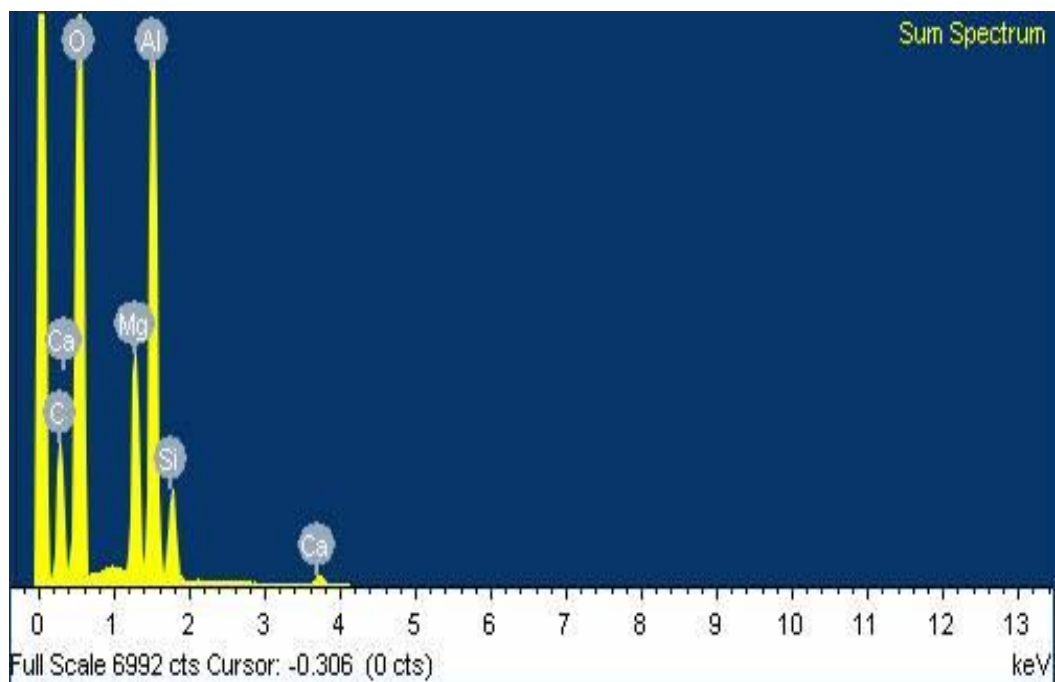


Figure 5.12: Sum Spectrum of the entire elements present in metal foam.

Table 5.2: EDS results for entire elements in weight% and atomic %.

| Element | Weight% | Atomic% |
|---------|---------|---------|
| C | 15.94 | 22.86 |
| O | 41.77 | 55.69 |
| Mg | 4.80 | 3.15 |
| Al | 35.30 | 16.10 |
| Si | 1.92 | 1.09 |
| Ca | 0.28 | 0.11 |
| Totals | 100 | |

3. X-Ray Diffraction

In the fabrication process of aluminium metal foam phase play an important role. X-Ray Diffraction analysis has been employed for the identification and quantification of the surface. X-RD analysis is a non- destructive technique. X-RD analysis has been carried out to check the presence of an element that might have in AlSi metal foam. As shown in Fig. 5.13, X-RD results after sintering highest intensity value for 2 theta at 39.056 based on ref.code

and the highest peak has been found for aluminium. That was absolutely true because aluminium was the parent metal.

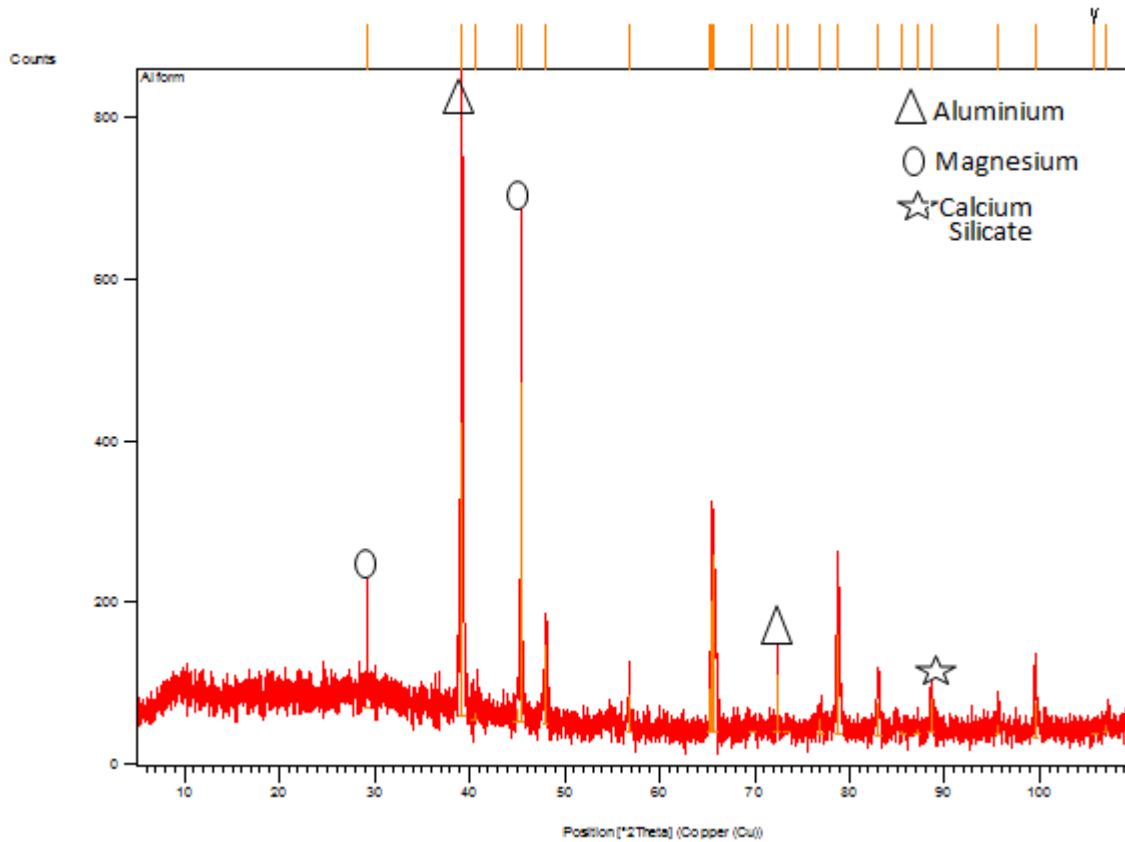


Figure 5.13: The results of the analysis of aluminium metal foam by X-RD.

Table 5.2: X-RD results of aluminium alloy foam

| Visible | Ref. Code | Score | Compound Name | Displacement [°2Th.] | Scale Factor | Chemical Formula | Semi Quant [%] |
|---------|-------------|-------|-----------------------|----------------------|--------------|---------------------------------------|----------------|
| 1. | 01-074-5237 | 49 | Aluminium Magnesium | 0.695 | 0.366 | Al _{0.95} Mg _{0.05} | 31 |
| 2. | 01-070-5680 | 34 | Silicon | 0.579 | 0.074 | Si | 6 |
| 3. | 01-074-0147 | 17 | calcium silicide - II | -0.088 | 0.528 | Ca Si | 64 |

Based on this table, presence of Aluminium and Magnesium sum can be seen has the highest total in metal foam and followed by the presence of silicon and calcium silicide.

5.2 Comparison

1. Number of pores in case of aluminium foam cannot be maintained. But in case of conventional porous this can be possible.
2. Pore size in metal foam cannot predict as required size, but in conventional it can be possible.
3. Weight and density ratio in metal foam is very less as compared to conventional porous, so foam can be used in many application.
4. In metal foam blowing agent is added during the fabrication process, but in conventional, there is no need of this type of agent.
5. In metal foam case uniformity of pores cannot be maintained, but in conventional it can be.

Chapter 6

Conclusion and Future Scope

6.1 Conclusion

In this study the summary of compression deformation behaviour of aluminium foam and aluminium porous at multiple strain rate at room temperature and calculated values as-

1. Aluminium metal foam has a constant plateau stress during compression test rather than in case of aluminium porous, due to this constant plateau stress of aluminium foam consumed more energy during compression. So, metal foam can be used in the application where more energy converted or consumed into other form. Applications in which metal foam can be used are- design of vehicle crash box which absorb collision energy during collision, sound absorbing application.
2. The compressive strength of aluminium foam very less than parent metal, but in case of aluminium porous compressive strength is approximate same as the parent metal, due to this strength aluminium porous can be used where more compressive strength and less weight than parent material is required. So, conventional porous can be used in application of heat transfer, self-lubrication system like self-lubricating bearing.
3. Metallurgical test SEM, EDX shows the different components rather than our additive are present on walls of cells and inside the cells, which reduces the thermal conductivity of the metal foam.

6.2 Future Scope

1. Increases the strength of metal foam by optimizes the value of blowing agent.
2. Reduce the relative density by optimize the quantity of blowing agent, which results increase in strength.
3. Increase in porosity by increase the formation time with increase in viscosity agent weight.
4. With the help of Gang drill all the hole on surface of conventional porous sample can be drilled at once.

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