Ceramics Manufacturing Processes

2nd. Shaping and Forming

There is a special vocabulary for shaping ceramics because it is an ancient art. Once the constituent powders have been prepared in the desired purity and the particle size, most ceramic products must be fabricated into useful shapes.

Many shaping methods are used for ceramic products and these routes can be mainly grouped into three basic categories, which are not necessarily independent.

❖ Powder Compaction

Powder compaction (such as dry pressing, hot pressing, cold isostatic pressing, etc.) is simply the pressing of a free flowing powder. The powder may be dry pressed (i.e. without the addition of a binder) or pressed with the addition of a small amount of a suitable binder. The pressure is applied either uniaxially or isostatically.

The choice of the pressing method depends on the shape of the final product. We make simple shapes by applying pressure uniaxially; more complex shapes require isostatic pressing. Also, the process can be done by using pressure and temperature to produce high density parts with fine grained microstructures.

❖ Casting

Casting ceramics (using a mold with ceramic as, or containing, a liquid or slurry) is carried out at room temperature and generally requires ceramic powder particles to be suspended in a liquid to form a slurry. The slurry is then poured into a porous mold that removes the liquid (it diffuses out through the mold) and leaves a particulate compact in the mold.

This process is known as slip casting. The process has been used to form many traditional ceramic products (e.g., sanitary ware) and more recently has been used in forming advanced ceramic products (e.g., rotor blades for gas turbines). The other main casting process for ceramics products is tape casting, which is used to make thick films or sheets.

❖ Plastic Forming

Plastic forming (like extrusion, injection molding, etc. using pressure to shape the green ceramic) consists of mixing the ceramic powder with a large volume fraction of a liquid to produce a mass that is deformable (plastic) under pressure. Such processes were developed and used originally for the clay and have since been adapted to the polymeric materials.

For traditional clay-based ceramics, the liquid is mainly water. For ceramic systems that are not based on clay, an organic may be used in place of, or in addition to, water. In forming processes, there are special terms must be known in order to understand the procedure of forming processes:

- Binder: is a component that is added to hold the powder together while we shape the body.
- Slurry: is a suspension of ceramic particles in a liquid.
- Plasticizer: is a component that is added to keep the powder soft or pliable, it improves the rheological properties.
- Green: is a ceramic before it is fired. Brown, white, or gray potter's clays are well known green ceramics.
- Slip: is a suspension of particles in a liquid or organic medium.

Some of shaping methods produce a ceramic compact that is strong enough to be handled and machined; however, it is not fully dense and the bonds between grains are not strong. This is called "green" state, which represents a transition state between the loose powder and high-density sintered product.

Binders and Plasticizers

It is often necessary to add a binder to ceramic powder. The binder has two functions. In some shaping methods, such as extrusion, the binder provides the plasticity necessary for the forming. The binder also provides the dry (green) shape with strength sufficient to survive the handling process between shaping and sintering. One of the most important requirements for the binder is that we must be able to eliminate it from the compact during the firing process without any disruptive effect; so that polymers binders are considered as ideal binders for ceramics.

In the pottery, the binder is often water that is present in sufficient quantity to make the clay easily shaped with the shape being retained during firing. The idea is that we then add a plasticizer to optimize the rheology of material. Note that these processes are not exclusive to ceramics but are general to powder processing.

A. Shaping by Compaction

1. Dry Pressing

Dry pressing is ideally suited to the formation of simple solid shapes and consists of three basic steps: filling die, compacting the contents, and ejecting the pressed solid. Fig .1 shows a schematic diagram of the double action dry pressing process. In a double-action press both the top and bottom punches are movable. When the bottom punch is in the low position, a cavity is formed in the die and this cavity is filled with powder.

In dry pressing technique, the powder mixture will contain between (0 - 5 wt%) of a binder. Once the cavity has been filled with powders, the top punch descends and compresses powder to a predetermined volume. During pressing, the powder particles must flow between the closing punches in order to fill spaces uniformly. A particle size distribution between (20-200 μm) is often preferred for dry pressing: the high-volume fraction of small particles causes problems with particle flow and results in sticking of punches.

Pressures used in dry pressing may be as high as 300 MPa, depending upon material and press type, to maximize density of compact. After pressing, both punches are moved and the compact is ejected. As dry-pressing process is so simple and involves low capital equipment costs, it is the most widely used high-volume forming process for ceramics. Production rates depend on the size and shape of the part and on the type of the press used.

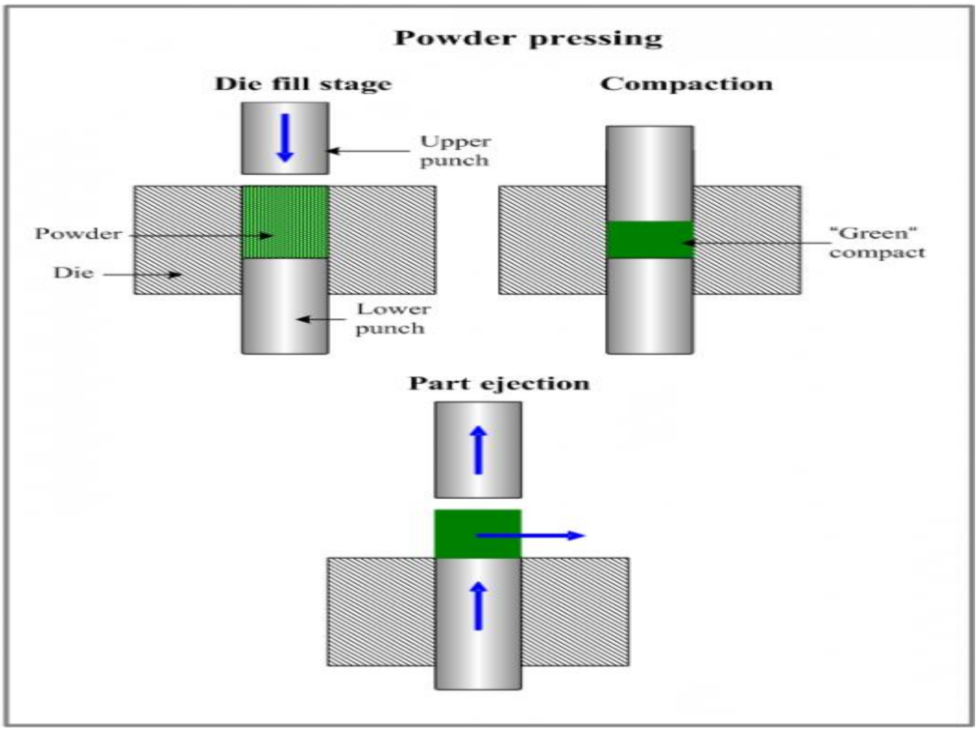

Fig (1): Stages of dry pressing.

2. Hot Pressing

Pressing can also be performed at high temperatures; this process is known as hot pressing. The die assembly used for hot pressing is very similar to for dry pressing.

The main difference is that in the hot pressing, the die assembly is contained within a high temperature furnace as shown in the fig.2. During the hot pressing, the ceramic powders may sinter together to form high-density component. We can summarize the advantages of this process:

- The powder does not have to be of the highest quality.
- Large pores that are caused by non-uniform mixing are easily removed.
- We can densify at temperatures lower than those needed for conventional pressureless sintering.
- The extensive grain growth does not occur when we keep the temperature low during densification.
- We can densify covalently bonded materials such as B_4C, SiC, and Si_3N_4 without additives.

The principal disadvantage is also important. Dies for use at the high temperatures are expensive and do not generally last long. Most metals are of little use as die materials above 1000 °C because they become ductile, and the die bulges. Special alloys, mostly based on Mo, can be used up to 1000 °C at pressures of 80 MPa. Ceramics such as Al_2O_3, SiC, and Si_3N_4 can be used up to about 1400°C at similar pressures.

Graphite is the most widely used die material and can be used at temperatures up to 2200 °C and pressures between 10 and 30 MPa. However, the graphite does have many properties that make it suitable for a die.

- It is easy to machine.
- It is inexpensive.
- Its strength increases with increasing temperature.
- It has good creep resistance.
- It has excellent thermal conductivity.
- It has a relatively low coefficient of thermal expansion.

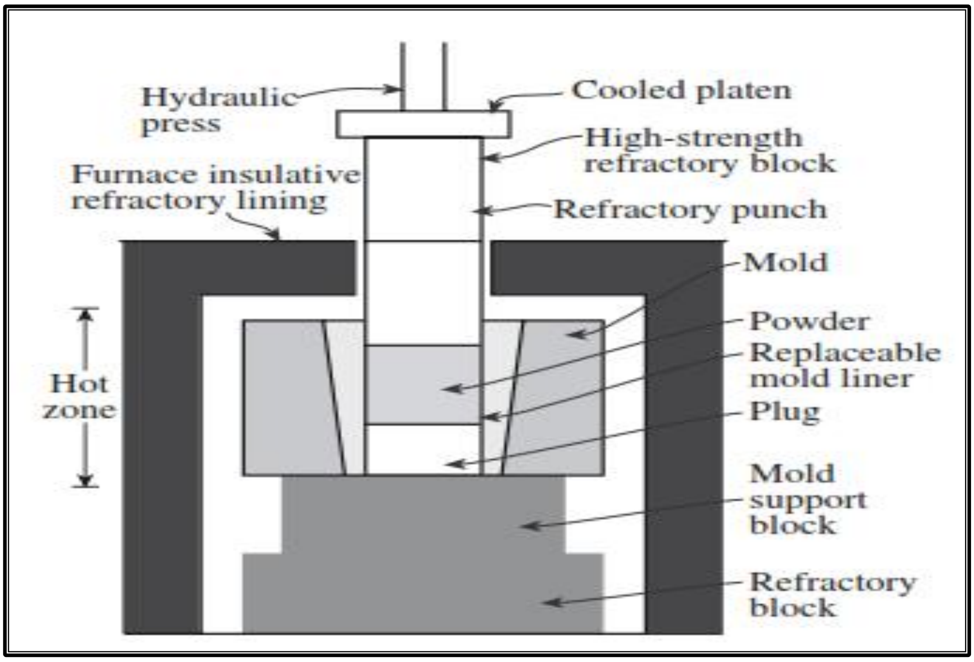

Fig (2): Schematic of hot pressing.

Hot pressing, like dry pressing, is limited to simple solid shapes, such as flat plates, blocks, and the cylinders. More complex or large shapes are difficult and often impossible to produce by hot pressing. Hot pressing is widely used in the research laboratory for processing very dense, high-purity ceramic components.

Although it is extensively used in the university and the government laboratories, the technique is limited as a production tool because of its high cost and the low productivity.

3. Cold Isostatic Pressing

Fig.3. illustrates the wet-bag CIP process. The ceramic powder is weighed into a sealed rubber bag. The sealed bag is placed inside a high-pressure chamber that is filled with a fluid and is hydrostatically pressed to the final shape.

The pressures used can vary from 20 MPa up to 1 GPa, depending upon press and the application. Once the pressing is complete, the pressure is released slowly, the mold is removed from the pressure chamber, and the pressed component is removed from the mold. The advantages of the wet-bag process are:

- Wide range of shapes and sizes can be produced.
- Uniform density of the pressed product.
- Low tooling costs.

The disadvantages of process are poor shape and dimension control.

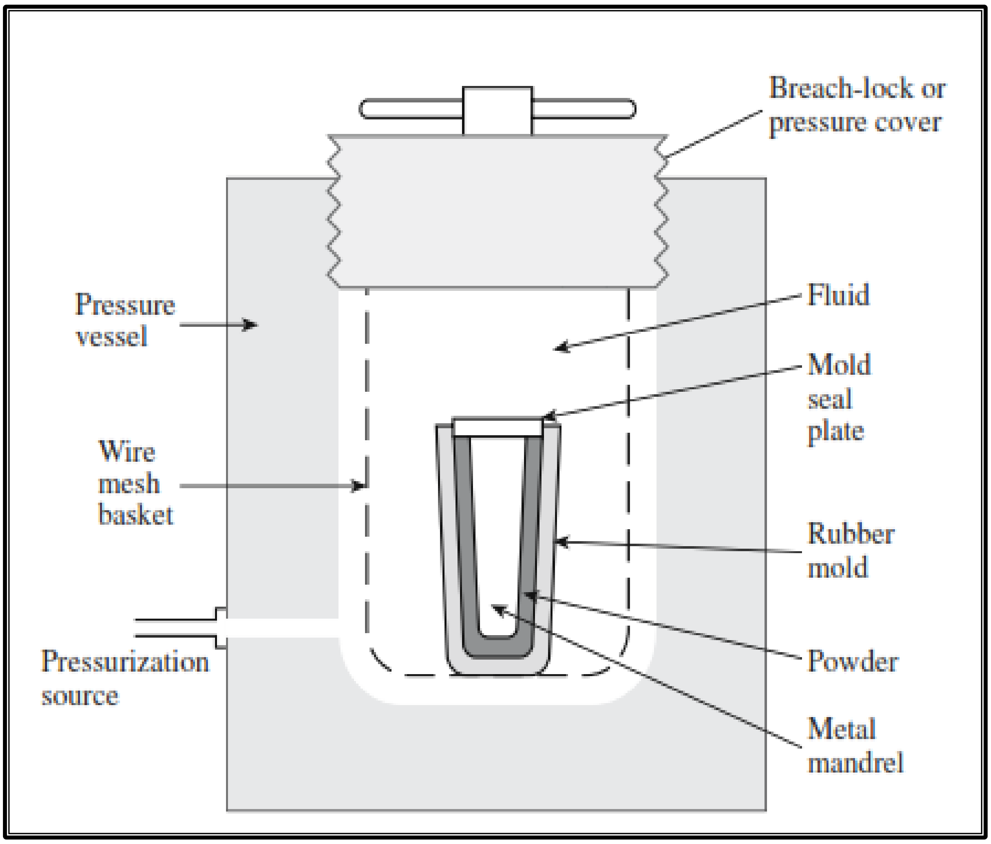

Fig (3): Schematic of cold isostatic pressing.

4. Hot Isostatic Pressing

Hot isostatic press (HIP) uses the simultaneous application of heat and pressure. We refer to this process as (HIPing) technique. A furnace is constructed within a high-pressure vessel and the objects to be pressed are placed inside the vessel. Fig.4. shows a typical HIP arrangement.

Temperatures can be up to (2000 °C) and pressures are typically in the range of (30–100) MPa. A gas is used as a pressure medium, unlike the (CIP) in which a liquid is often used. Argon is the most common gas that is used for HIPing.

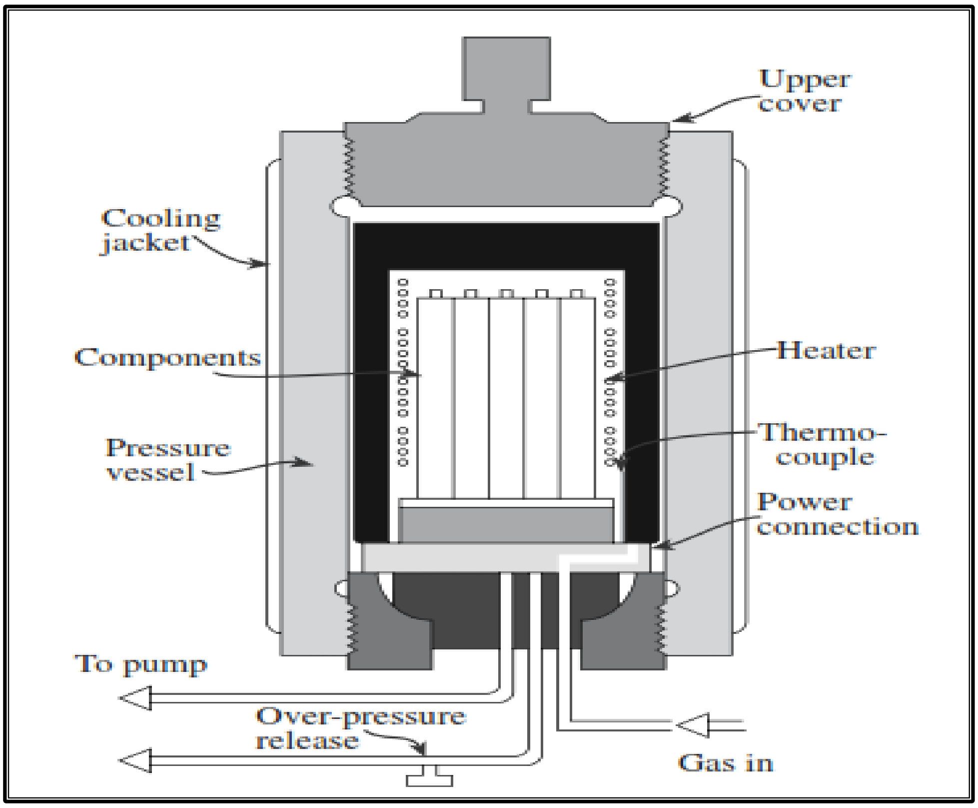

Fig (4): Schematic of hot isostatic pressing.

There are two variants of HIPing process: Encapsulated by using a deformable container. Not encapsulated when it is shaped and sintered first, then HIPed.

In the original HIPing technique, the ceramic powder was filled into a deformable container and then subjected to the heat and the pressure.

The second variant does not involve encapsulation. The ceramic powder is first compacted using another shaping method such as dry pressing or injection molding followed by sintering process at relatively high temperatures in a furnace to close all the surface pores, which prevents the entry of the gas during subsequent HIPing procedure. The steps in this process, which is sometimes referred to as sinter-plus HIP.

Now HIPing is used for a wide variety of ceramic components, such as alumina-based tool and silicon nitride nozzles used in flue-gas desulfurization plants by utility industry. The advantages of HIPing process are becoming more important as interest in the structural ceramics such as the Si_3N_4 grows.

HIPing is used for a wide variety of ceramic components, non-oxide ceramics can be HIPed to full density while keeping the grain size small and not using additives. Very high densities combined with small grain sizes lead to formation products with special mechanical properties.

In addition, HIPing produces dense materials without growing the grains. HIPing has also been applied to formation of advanced ceramic like piezoelectric ceramics. Disadvantage of this process is the high cost.

5. Roller Compressing

In this technique, the granulated powder can be continuously compressed between rollers for the production of the sheets with thickness of about (1) mm.

B. Shaping by Casting

1. Slip Casting

Slip casting is the most conventional technique of producing very varied pieces that can have complex forms (sanitary, refractory and technical ceramics).

This method consists of casting a suspension (slip) in a porous mold, generally made of plaster. The fineness of powder (in the slip) and the consequent high surface area ensure that electrostatic forces dominate gravity so that settling does not occur. Electrochemistry of the slip is quite complex: Na silicate (or soda ash) is added to the slip to deflocculate the particles.

Since the deflocculation is defined as the process by which floccules present in liquid break up into fine particles producing a dispersion, a deflocculant is an additive that causes this process. In other words, deflocculation is the opposite of coagulation.

Water from slip is absorbed into porous mold, leaving behind a solid layer on mold wall, the thickness of which depends on the time. This process may be continued until the entire mold cavity becomes solid. (See Fig.5). The main advantages of slip casting are: (i) complexity of forms that can be produced, (ii) its low cost and (iii) dense and homogenous green ceramic microstructures are obtained. The main disadvantage is the low production capacity.

Aqueous suspensions are the most common, but materials like (MgO, CaO, La_2O_3, SiC, Si_3N_4) are cast in an organic environment such as alcohol and ketone.

The external form of the ceramic piece is defined by the mold, and when the thickness of the consolidated wall is sufficient, suspension is emptied. The green part, after drying, must have enough cohesion to be demolded. The low shrinkage and variable walls thicknesses are also produced in this technique.

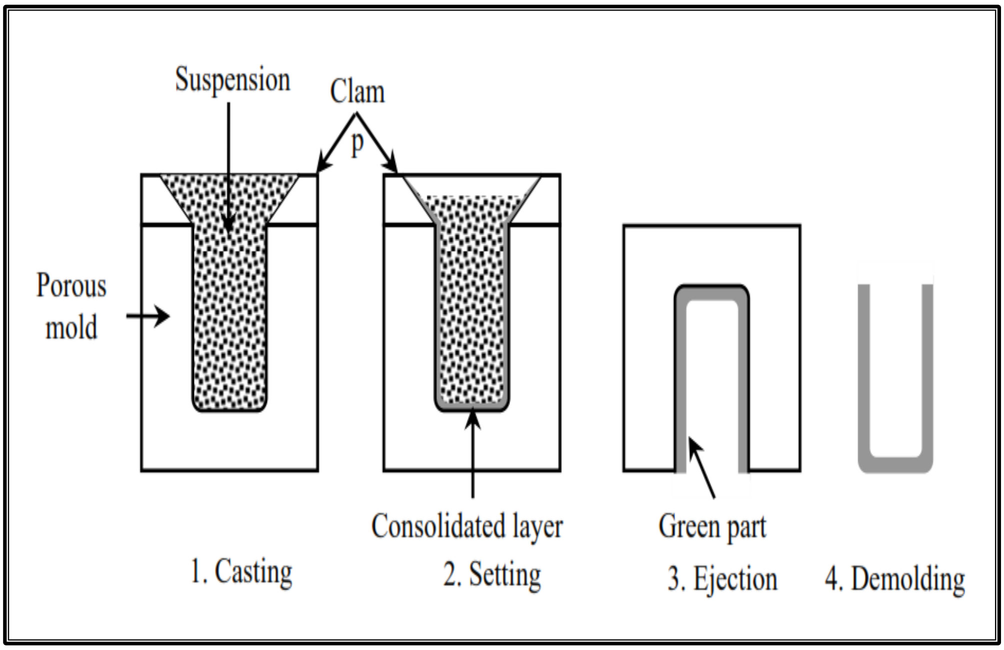

Fig (5): Stages of slip casting.

2. Tape Casting

Tape casting is an important ceramic fabrication technique. In this technique, thin sheets of a flexible tape are produced by means of a casting process. These ceramic sheets are prepared from slips in many respects similar to those employed for slip casting. This type of slip consists of a suspension of ceramic particles in an organic liquid that also contains binders and plasticizers, which incorporated to impart strength and flexibility to the cast tape.

The actual ceramic tape is formed by pouring the slip onto a flat surface (stainless steel or glass); a doctor blade spreads the slip into a thin tape of uniform thickness, as seen in fig.6. In drying process, volatile slip components are removed by the evaporation to produce green product in form of a flexible tape.

Tape thicknesses normally range between 0.1 and 2 mm. The tape casting is widely used in the production of ceramic substrates that are used for the integrated circuits and for multilayered capacitors in electronic devices.

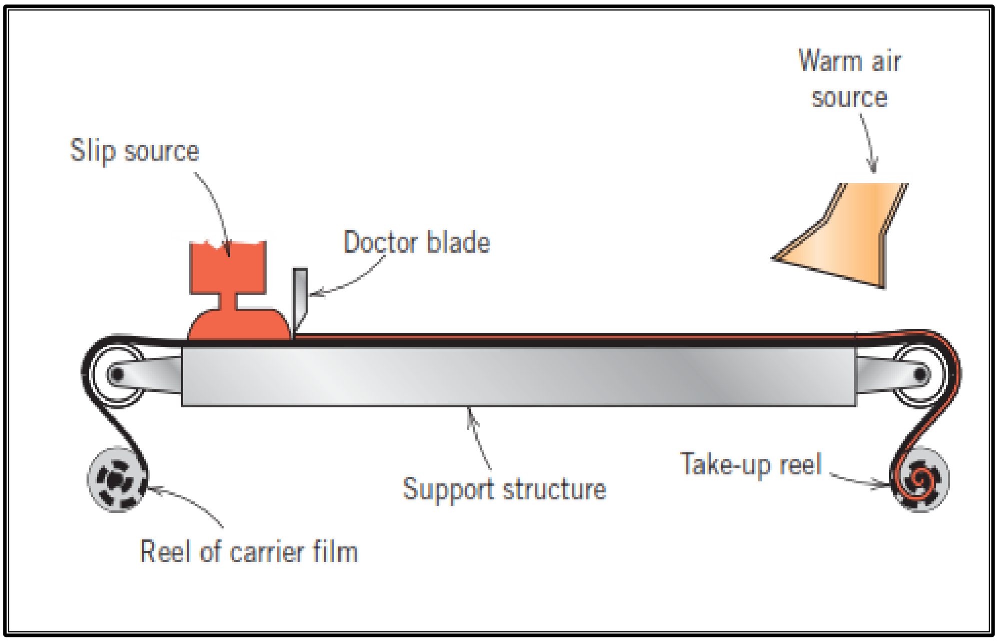

Fig (6): Schematic of tape-casting process.

C. Shaping by Plastic Forming

1. Extrusion

Extrusion technique involves forcing a deformable mass through a die orifice. Extrusion is widely used to produce ceramic components having a uniform cross section and a large length-to-diameter ratio such as ceramic tubes and rods as illustrated in fig.7.

Clay with a suitable rheology for the extrusion process (essentially a paste) can be made by controlling the amount of water. Clay-free starting materials, such as (Al_2O_3), are mixed with a viscous liquid such as polyvinyl alcohol (PVA) and water to produce a plastically deformable mass.

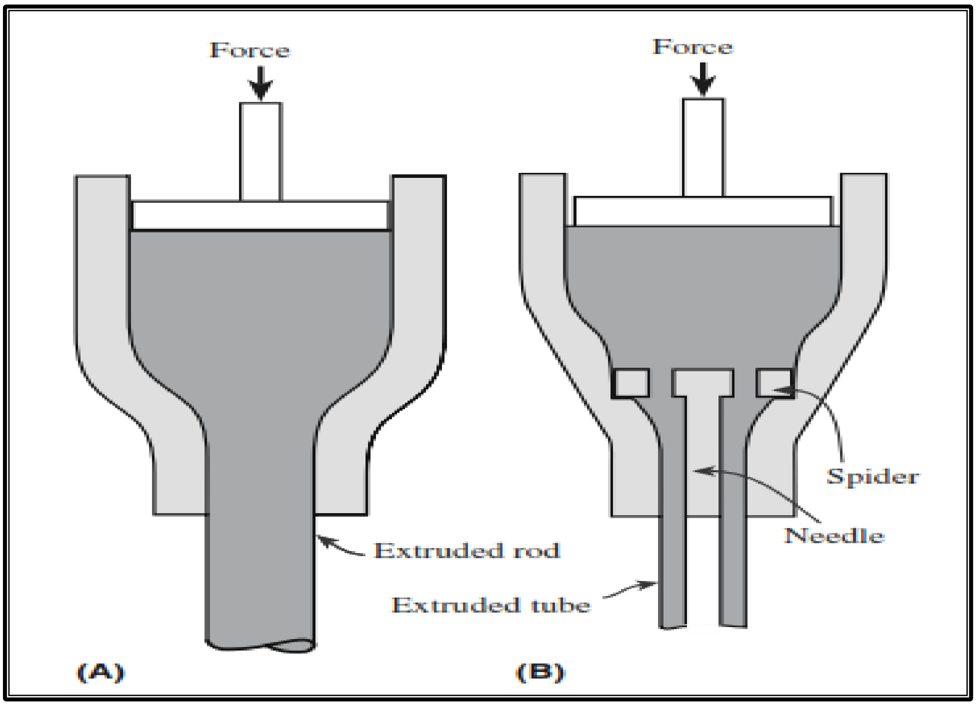

Fig (7): Extrusion process of (a) a rod and (b) a tube.

2. Injection Molding

Injection molding methods can be applied to shaping and forming ceramic components if ceramic powder is added to a thermoplastic polymer. When forming ceramics by injection molding, the polymer is usually referred to as the binder which provide a mass that has the desired rheological properties.

The organic part of the mixture accounts for about (40 vol%). The plastic ceramic mass is first heated, at which point the thermoplastic polymer becomes soft and is then forced into a mold cavity as shown in the fig.8.

The mixture is allowed to cool in the mold, during which time the thermoplastic polymer hardens. Because of the large volume fraction of organic material used in the mixture, there is a high degree of shrinkage of components during sintering. Shrinkage of (15–20%) is typical, so that, the precise control of component dimensions is difficult.

Injection molding is used to fabricate ceramic components with complex shapes; because cycle times can be rapid, injection molding can be a high-volume process. The major limitation is that the initial tooling costs of mold can be quite high. The mold used to fabricate an individual turbine blade can be >$10,000 and a mold for a turbine rotor may be >$100,000, but such molds are reusable since they are never subjected to high temperatures.

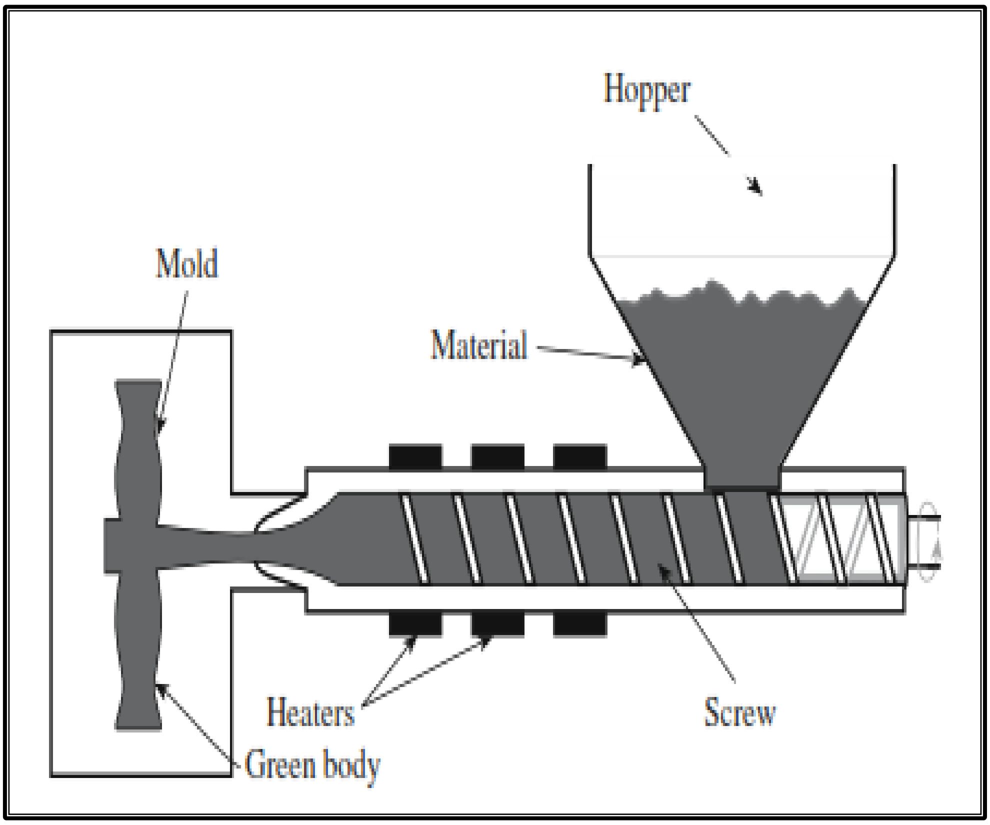

Fig (8): Injection molding process.

Green Machining

To obtain the desired shape of a ceramic product it is often necessary to machine it. Machining can be performed either before or after the product has been sintered. If the machining is done before sintering, while the component is still in the green state, the process is called green machining.

The advantages of the green machining compared to machining the sintered product are that there is a considerable reduction in the machining time and a reduction in cost because of less tool wear and the possibility of using cheaper tools.

Binder Burnout

In pottery, the binder burnout is removal of water from the shaped clay. The rest of the firing process causes structure changes and transformations in silicate itself. Forming methods for engineering ceramics, like injection molding, produce green bodies that can contain (30–50) vol% of organic binder.

The purpose is to remove this binder without cracking or distorting the ceramic compact. Binder burnout is one of the most likely stages to form defects in the processing of a ceramic: macroscopic defects, such as cracks and blisters, can be introduced at this stage, and these will effect on the mechanical strength and the physical properties of the ceramics.

The binder used in the ceramic fabrication often consists of several components. These components have different boiling points and decomposition temperatures. Components with low boiling points (e.g., waxes) may be removed by evaporation process at fairy low temperatures. Oxidation or decomposition at higher temperatures removes high-molecular-weight components.

Final Machining

Ideally, shaping and forming processes that are employed would produce ceramic object in desired shape with specified dimensional tolerances and with an acceptable surface finish. However, in many cases, this is not the situation and some final machining (after firing or sintering) of ceramic is necessary. Generally, final machining is required to:

- Meet dimensional tolerances.
- Improve the surface finish.
- Remove surface flaws.

Machining of fired ceramics are expensive and represent a significant fraction of the total fabrication costs. Ceramic materials are difficult to machine because they are hard and brittle. The tooling costs are high because diamond tools are likely required or if conventional tools are used the tool life is very short.

In addition, the time required to machine ceramics is long because if high tensile loads are applied to the ceramic part it might fracture. The mechanical approaches to machine the ceramics objects include the following:

- Grinding uses tools in which abrasive particles are embedded in a softer matrix such as, rubber, or polymer resin, or a metal.
- Lapping uses loose abrasive particles placed on a soft cloth.
- Sandblasting uses abrasive particles accelerated by compressed air and directed through a nozzle at high velocity.
- Water-jet machining uses a high-pressure water jet to transport the abrasive particles to ceramic surface.

The water-jet method is gaining popularity as a high-speed method for machining hard ceramics. Cutting rates depend on the material being cut and can vary from glass to dense ceramic.

In the water-jet technique, the water is pressurized to about of (~400 MPa) and is forced through a sapphire orifice at a velocity of about 750 m/s.

Porous Ceramics

In many traditional applications, particularly in the structural and electrical applications, the sintered ceramic component is required to have minimum porosity.

The growing number of applications, for example, in the ceramic humidity and gas sensors, the porosity within structure is not just desirable, it is required.

Several different methods can be used to produce porous ceramic structures. Use large particles with a very small size distribution to avoid the dense packing. Underfire the green compact to leave a large amount of fine pores.

Add organic particles (diameter >20 μm) to the powder mixture; when these burn out they will leave behind porosity. Use a binder system that contains a foaming agent and produces a large amount of gas bubbles in the mixture.