1.11.1.6 Microwave Sintering

Microwave heating is effective for heating more complex ceramic forms very rapidly. Heating rates in excess of 1000°C/min can be achieved. In this method, the ceramic body, usually contained in non-absorbing or weakly absorbing insulation such as loose, non-conducting powder, is placed within a microwave cavity. A schematic of the method is shown in Figure 1.24. The method is quite straightforward. It is even possible to use a simple consumer microwave oven to achieve densification of ceramic powder compacts when properly insulated. The shape of the ceramic body affects the local heating rates significantly and achieving uniform heating can be difficult. The microwave frequency plays a significant role in the temperature gradients that can develop within the ceramic body. High frequencies tend to heat the exterior of the sample more than the interior, and a combination of frequencies ranging between about 2.5 and 85 GHz has been proposed as providing more uniform heating.

A variant of microwave sintering is one where the microwaves ignite a plasma that surrounds the ceramic part. Very high sintering rates can be achieved this way. The schematic of the method is shown in Figure 1.25.

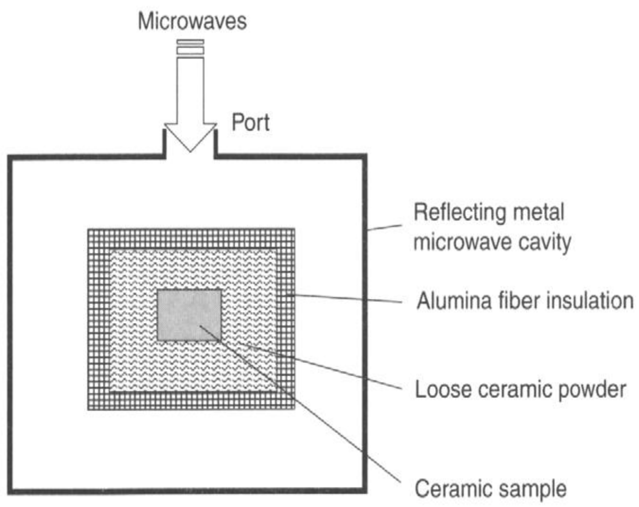


FIGURE 1.24 Schematic microwave sintering setup.

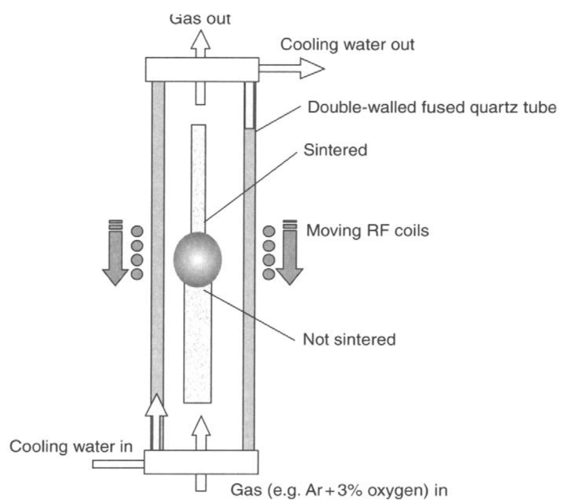


FIGURE 1.25 Schematic of plasma Sintering setup.

1.11.1.7 Plasma-Assisted Sintering

Several attempts have been made at increasing the heating rates, causing a need for various superlatives such as "superfast" or "ultrafast" sintering, by passing a DC current pulse through a powder compact contained in a graphite die, under an applied pressure of 30-50 MPa. This method has been called spark plasma sintering (SPS). A schematic of the method is shown in Figure 1.26. Specimen temperatures are difficult to assess in this method, and are usually measured by optical pyrometer on the graphite die wall. The current pulse heats both the die and the specimen. Heating rates of 600 K/min and more have been reported. The high heating rates are thought to be caused, in part, by spark discharges generated in the voids between the particles. Remarkably high densification rates may be achieved under such conditions, with minimal grain growth. This approach is particularly useful in producing dense ceramic bodies from nanosized powders. The specimen shapes that can be prepared by SPS are limited to simple slabs that can be contained in the compression die.

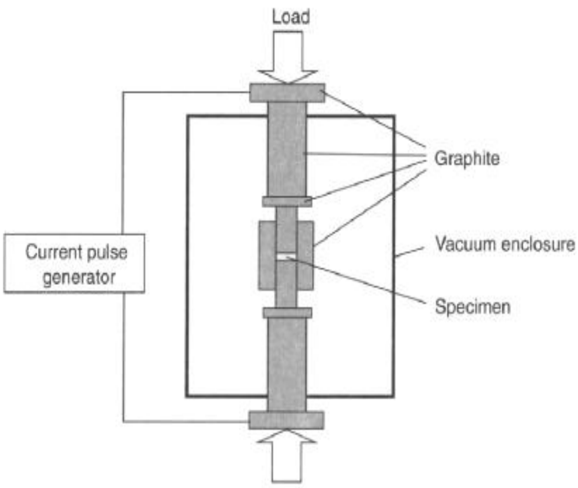


FIGURE 1.26 Schematic of a spark plasma sintering apparatus.

1.11.2 PRESSURE-ASSISTED SINTERING

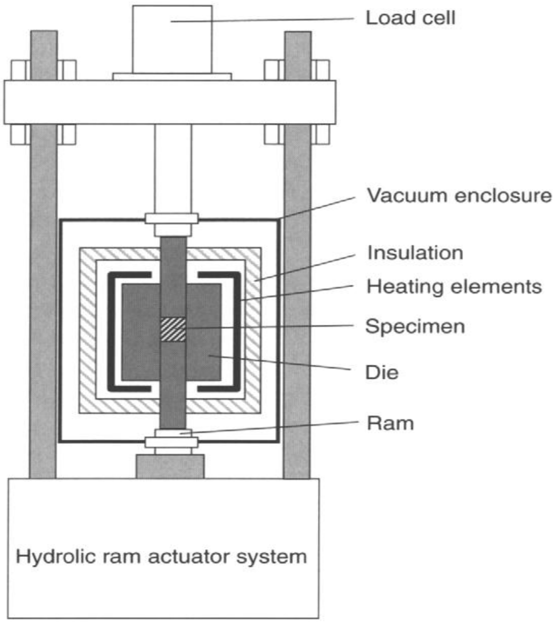
1.11.2.1 Hot pressing

Hot pressing is a convenient laboratory method for preparing dense samples. Heat and pressure are applied to a sample contained typically in a high strength graphite die, at applied pressures around 50 MPa. The die wall thickness is approximately equal to the sample diameter if high-strength graphite is used. Other die material such as aluminum oxide, silicon carbide, or dies confined by an outer metal or fiber-wound mantle are also used in rare cases. In this method, the sample is packed in coarse powder in the hot pressing die, developing a roughly isostatic pressure on the work piece.

A schematic of the standard process is shown in Figure 1.27. Systems that are more complex have also been developed. In a typical hot pressing process, a moderate pressure is applied form the start, 10-20 MPa. Upon reaching the sintering temperature, the pressure is increased to maximum. An applied pressure is usually maintained during the cool-down period as well. A schematic of the thermo-mechanical treatment is shown in Figure 1.28. If no reliable temperature measurement is available, it is possible to follow the ram displacement as the temperature is increased. An appropriate sintering temperature is then reached when the rate of the ram displacement rapidly increases to indicate a projected full densification in 20-30 min. Hot pressing is less effective for very fine powder that is well compacted prior to hot pressing. Benefits may still be derived from the applied pressure when it can assist in the rearrangement process or in the collapsing of large pores.

In case full densification is difficult to achieve, additives can be used similar to the free sintering method. Because of the significant uniaxial strain in hot pressing, texture may develop in the finished part. Trapping of insoluble gases in residual pores may cause swelling of the ceramic at elevated service temperatures. This can be avoided by hot pressing in vacuum.

Reactive hot pressing has also been used successfully. Related to reactive hot pressing is hydrothermal hot pressing. In this method, powders are compacted under hydrothermal conditions. The powder is essentially compacted inside an autoclave at temperatures between 100 and 350°C. The ram design should allow fluid to leave the sample. Materials such as hydroxyapatite and amorphous Titania have been compacted this way.



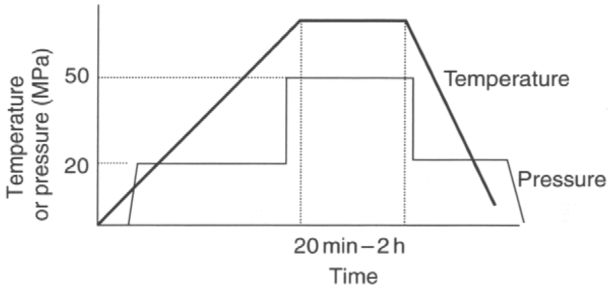


FIGURE 1.28 Schematic temperature/ pressure schedule for hot pressing with a high-strength graphite die.

FIGURE 1.27 Schematic of the hot pressing process.

1.11.2.2 Sinter Forging

Sinter forging is similar to hot pressing, but without confining the sample in a die, as shown in Figure 1.29. Uniaxial strains are, as a result, significantly larger than in die hot pressing. For fine powders, use can be made of possible superplastic deformation modes during sinter forging. During the process, the strain rate has to be limited to avoid damaging the sample.

1.11.2.3 Hot Isostatic Pressing (HIPing)

A schematic of the equipment is shown in Figure 1.30. In this method, the pre-consolidated powder is tightly enclosed in a glass or metal container, sealed under vacuum and positioned in the pressure vessel. Alternatively, the sample can be pre-densified to closed porosity by traditional sintering, after which a canister is not needed in subsequent HIPing.

A compressor introduces inert gas pressure, and the sample is heated to the sintering temperature, which may be up to 2000 ° C. During this time, the gas pressure rises to as much as 30 000 psi, and the container collapses around the sample, transmitting the isostatic pressure to the sample. Commercial hot isostatic presses may have internal chamber diameters approaching (1m). Heating elements are typically graphite, molybdenum, tungsten, or tantalum. The quality of the ceramics produced by hot isostatic pressing is perhaps the highest obtainable by any other pressure method, since externally heated dies cannot withstand the pressure that can be applied by the HIP. HIPing is also used to recondition parts in which internal damage, such as resulting from fatigue or creep, may have accumulated during service.

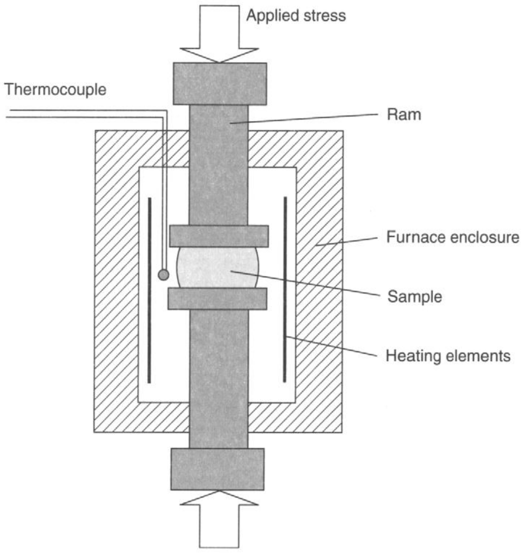


FIGURE 1.29 Schematic setup for sinter forging.

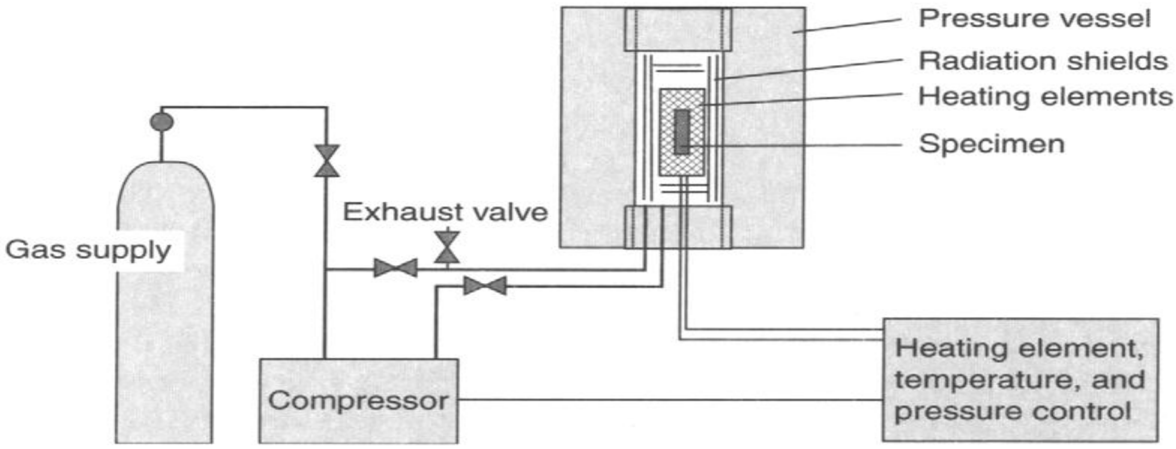


FIGURE 1.30 Schematic of hot isostatic pressing equipment

While the sintering behavior of real powders is considerably more complex than that assumed in the models, the sintering theory clearly indicates the key parameters that must be controlled to optimize sintering. The key factors are the particle size of the powder and the particle packing in the green body but other characteristics, such as size distribution, shape and structure of the particles, can also exert a significant influence.