Fabrication of green and sintered Al: Al_2O_3 composites using gel-casting and powder metallurgy technique

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

Master of Technology

In

Ceramic Engineering

By

Denny K. Philip

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National Institute of Technology Rourkela

ODISHA-769 008, INDIA

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Under the guidance of

Prof. Bibhuti B. Nayak and Prof. Sumit K. Pal

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National Institute of Technology

Rourkela

CERTIFICATE

This is to certify that this thesis entitled, "Fabrication of green and sintered Al: Al_2O_3 composites using gel-casting and powder metallurgy technique" submitted by Denny K. Philip in partial fulfillments for the requirements for the award of Master of Technology Degree in Ceramic Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under our guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

Prof. Sumit K. Pal
Assistant Professor
Department of Ceramic Engineering
National Institute of Technology
Rourkela- 769 008

Prof. Bibhuti B. Nayak
Associate Professor
Department of Ceramic Engineering
National Institute of Technology
Rourkela- 769 008

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Denny K Philip
Roll no: 211CR1042
denkp1@yahoo.co.in

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CONTENTS

Abstract

An effort had been made to fabricate composite of (1-x) wt% Al: x wt % Al_2O_3 (where x = 0, 10, 20 30, 40, and 50) using both gel casting and powder metallurgy techniques To the best of our knowledge, there was no such literature available on Al-Al_2O_3 composites prepared through gel-casting technique. Prior to gel-casting zeta-potential, concentration of deflocculating agent, and viscosity were optimized. A mould was designed and fabricated and a new processing route was employed to produce a homogeneous green body which was successful. Microstructure implies that the sintered body was highly porous which could not provide enough mechanical strength. However, the green product was easily machinable. XRD analysis shows no sign of oxidation of aluminium. It has to be further studied before using it for the production.

In powder metallurgy, to attain proper mixing aluminium and alumina in their powdered form are milled for 1h in a planetary milling machine with isopropyl alcohol as the wetting agent. Powders were pressed to samples of different shape and were sintered under vacuum. Normally such composites are sintered at nitrogen or argon atmosphere. Some attachments for were designed and manufactured that could be used in the tubular furnace to obtain vacuum. The design was successful and was able to produce a vacuum pressure of 5.6E-6 mbars inside the tube. It had been found that by reinforcing with 20% alumina the hardness of aluminium was increased to 35 HV. Bending test gives the indication that the plasticity is reduced slowly by the addition of alumina into aluminium matrix. It was also found that the tribological properties can be improved by adding 20 weight % of alumina into the metal. Microstructure indicates that the alumina was well dispersed in the aluminium matrix. The composite is easily machinable with HSS tool with minimum tool wear. Therefore this composite can be used to replace conventional materials in various automotive, aerospace, and automobile industries.

Keywords: Gel-casting, powder metallurgy, Al-Al_2O_3, composites, metal-ceramic, vacuum sintering

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Chapter-1

General Introduction

1.1 Introduction

Composites consisting of metal-ceramic such as Aluminum (Al): Alumina (Al_2O_3) are most important materials for advanced technologies applied in high temperature applications due to its high strength/stiffness to weight ratio, light weight, environmental resistance and adequate mechanical properties. The melting point of Al is high enough to satisfy many application requirements, yet low enough that it can be processed with convenience. Al_2O_3 is chosen as the reinforcement since it is chemically inert with Al and can also be used at higher temperatures compared with the un-reinforced Al and also having good benefits on the mechanical properties. In technology point of view, Al-Al_2O_3 composite material has more advantageous and more popular than the lightweight monolithic titanium, magnesium, and aluminum alloys. Aluminum alloy with discontinuous ceramic reinforced MMC was rapidly replacing conventional materials in various automotive, aerospace, and automobile industries. These Metal ceramic composites are the best choice in applications that involve high temperatures, such as thermal barrier coatings, turbine engines, and piston rod, due to their good resistance to elevated temperatures.

Lots of researchers have prepared this composite material through commonly used powder metallurgy process due to better homogeneity in the distribution of metal and ceramics in the composites. Execution of this process is limited to some simple shape as it is difficult to attain uniform density throughout for complex shapes. In this research work, we have followed gel-casting process for preparing Al-Al_2O_3 composites, as this process is found to be developing any complex shape materials. Gel-casting is a novel method for manufacturing of advanced structural ceramic components, based on concepts derived from traditional ceramics and polymer chemistry. Also for comparison purpose, we have prepared Al: Al_2O_3 composites using powder metallurgy process.

In this project work, we have also design special mould used for gel-casting process and also design vacuum chamber and special attachments for heating the samples under vacuum. An effort was being made to produce Al: Al_2O_3 composite at different compositions using both gel casting and powder metallurgy techniques.

Chapter -2
Literature Review

2.1 Literature Review

In order to fabricate a composite having a well distributed Al_2O_3 particles uniformly in Al matrix, the most appropriate method that can be employed is the powder metallurgy technique [1]. Its main advantage is that it is a near net shape process and can be used for fabricating almost all shapes. The main challenge to attain homogeneity in distribution of ceramic in the metal matrix is to eliminate the agglomeration of the reinforcement particles [2]. It becomes more complicated when the size of the reinforcement is small and its volume fraction. Rational usage of the processing technique could eliminate this problem. One such method is high-energy ball milling (also known as mechanical alloying). It involves repeated cold welding, fracturing, and rewelding of powder particles at the atomic level. The particles undergo severe plastic deformation during collision between balls and with the wall as the particles are trapped between. [2-4]. Continuous process of fracture and welding events, which leads to a uniform distribution of the reinforcement particles in the metallic matrix. The additions of alumina particles accelerate the milling process, leading to faster work hardening rate and fracture of the aluminum matrix [2].

Another technique called infiltration is used for producing composite with higher volume percentage of alumina. This technique is employed for manufacturing components of structural application which require high specific modulus, low thermal expansion coefficient and wear resistance [5]. In this molten metal is infiltrated into ceramic preform. A preform is the sintered body made of ceramic particles which has large pores in it. These pores are filled with the molten metal during infiltration. MMCs can also be manufactured using melting process in which the ceramic powder is added to the molten metal at high temperature. The basic limitation of this method is the poor wettability of ceramic particles with liquid Al alloys. Wettability can be defined as the ability of a liquid to spread on a solid surface, and it represents the extent of intimate contact between a liquid and a solid, and this enhances the tendency of reinforcement agglomeration [6].

Aluminum is taken for study because of its specific properties like low density, relatively low price, and corrosion resistance in many applications as well as availability in a large quantity. A number of reinforcing materials such as Alumina, silicon carbide, boron nitride graphite, Zirconia etc have been used to reinforce Al using various techniques [6]. Al_2O_3 is taken as the reinforcing material, since it is chemically inert with Al and can also be used at higher temperatures, comparative with the un-reinforced aluminum, also having good benefits on the mechanical properties, especially on the creep resistance [2]. The mechanical properties of the composite can be improved by dispersing the fine reinforcements and a fine grain size of the matrix. Further, the mechanical properties of the composite also get improved with increasing volume fraction and decreasing particle size of the reinforcements [2, 3]. This improvement in mechanical properties is achieved because MMCs have high dislocation densities due to dislocation generation as a result of difference in coefficient of thermal expansion [7]. The Young's modulus of the composite was measured as 170 GPa and was nearly thrice the un-reinforced alloy (65 GPa) [8]. Even if the strengths were nominally the same in both tension and compression, greater ultimate strengths and strain-to-failure were observed in compression than in tension. The composites also show a nearly constant fracture toughness for all compositions, with values approaching 20MPam½ compared to a value of 29MPam½ for the base alloy [8].

The compacting pressure has a significant importance in the compaction process as the elastic moduli of the composites depend not only on the alumina volume content, but also on the porosity of the samples [1]. If the high pressure is given that would lead to high stresses in the aluminum constituent which could lead to plastic deformation, and high stresses in the alumina constituent could cause cracking. In addition, debonding can also occur at higher temperature also resulting in large stress discontinuities between the interfaces of aluminum and alumina. All these factors could significantly reduce the elastic moduli of the composites [1, 2]. Porosity for composite systems increases significantly with an increase in the alumina volume contents. For composites with higher contents of alumina particles, the alumina particles are close to each other and some of them are possibly in contact, preventing the aluminum particles to fill the gaps in between them during cold pressing. Using Aluminum with smaller particle size are desired as it helps the aluminum particles to fill the empty gaps between the alumina by reducing the porosity [1]. Composites with relative density ranging from 96.5 to 99% were manufactured using finer particles and sintering temperature of 600 °C.

Sintering atmosphere has a greater effect in the processing of metal matrix composites. Certain works suggests that nitrogen is the best atmosphere for sintering aluminum to produce samples with high relative density, followed by argon [9]. However, the use of nitrogen forms aluminum nitride (AlN) as an additional phase, therefore argon gas is mostly used.

Increasing the compacting pressures up to 502 MPa increases the relative density of the composite [1]. It was also stated that the cold compaction pressure, together with particle size of initial powders are the main parameters that affect the final density of the sintered composites. It has been stated on the basis of work in which density of the composites increases for less than 2% after sintering even at temperatures that are close to the melting point of aluminum [1].

Because of the poor wear resistance of aluminium alloy, their applications as structural materials and machine parts are often limited to a greater extend. Fortunately the wear resistance can be improved to a higher extend by incorporating alumina into the metal or some alloys [7]. Amro et al. [10] claims that by higher load and higher concentration of Al2O3 particles lead to higher wear rates. For 10 and 20% Al_2O_3 concentrations, the wear rate decreases with increasing sliding speed, while it increases for 30% Al_2O_3. At lower sliding speeds abrasion is dominant, while at higher sliding speeds delamination and adhesion increases. Results also indicate that the friction coefficient between the composite and the mating steel surface decreases with increasing sliding speed to a steady state. Heat treatment can increase the abrasive wear resistance of Aluminium matrix composite [11].

Gel-casting is a method used for moulding ceramic powder based on concepts derived from traditional ceramics and polymer chemistry. It is a promising technology for manufacturing of advanced structural ceramic components [12].This forming process was developed to overcome some of the limitations of other complex-shape forming techniques such as injection molding and slip casting. It is a new near net shaping technique [12]. Gelcasting has established itself as an attractive ceramic forming process for making high-quality, complex-shaped ceramic parts. Figure 2.1 shows the schematic representation of the steps involved in the process of gelcasting.

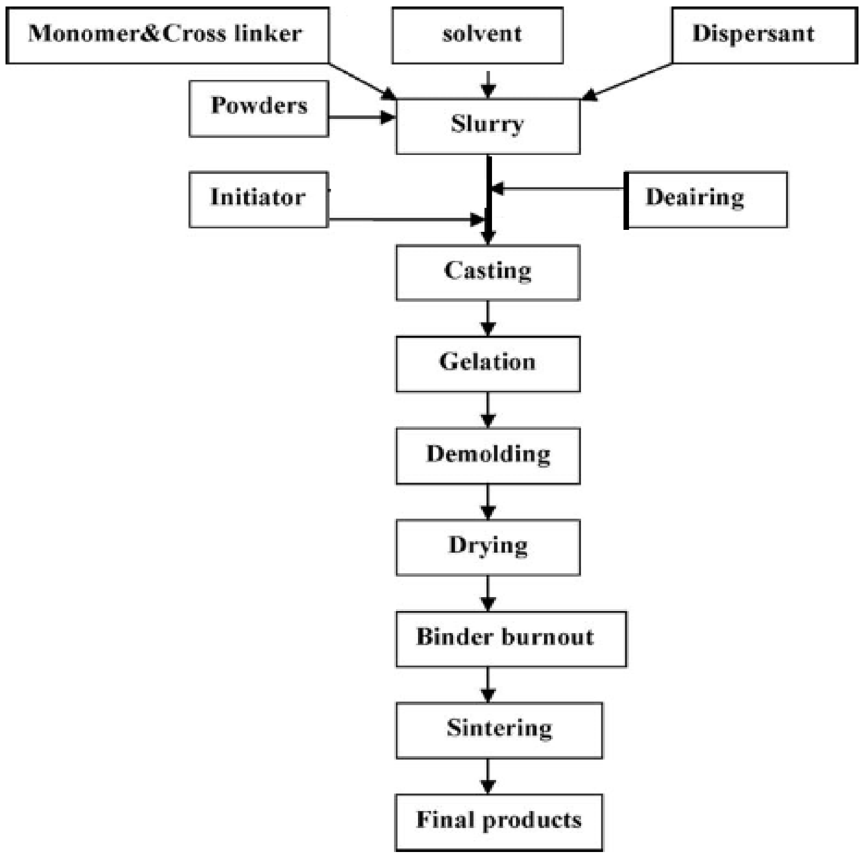


Fig 2.1 Steps involved in gelcasting

Colloidal solution is been prepared for the powder to be casted in the solvent. Deflocculating agent has to be added in order to obtain a stable solution. pH has a significant role in stabilizing the colloidal solution as it directly affects the zeta potential. In this process a monomer and cross linker is added to the slurry with ceramic particles suspended in it. When it is dissolved the initiator and cross linker is added which starts a polymerization reaction [12]. The monomer and cross linker form a three dimensional network of polymers which holds the ceramic particles together. The green strength of the components made of gel casting is higher than any other process. Degassing helps in reducing the porosity to a greater extend.

Gel casting can be aqueous or non aqueous depending upon the solvent used. Different monomer cross linker systems should also be used depending upon the solvent and the ceramic particles to be casted [13]. Methacrylamide (MAM) and methylene-bisacrylamide (MBAM) are the monomer and crosslinker which are extensively used. Poly ethylene Glycol (PEG) is added to prevent the reaction of the chemicals with the atmosphere. PEG forms a small layer separating the slurry with the atmosphere. Ammonium persulphate (APS) is used as the initiator. Tetramethylethylenediamine (TEMED) is used as catalyst. Bera et al. [14] described the crosslinking copolymerization of acrylamide and bisacrylamide monomers in three different steps: (i) pregel reaction, (ii) gelation, and (iii) postgel reactions. In the first step (pregel reaction), soluble microparticles are formed that are richer in bisacrylamide molecules due to their higher reactivity compared to the acrylamide molecules. In the second step the pregel particles are linked by polymerized acrylamide chains that are poorer in bisacrylamide molecules than microgels. The third step corresponds to the formation of dangling acrylamide chains as most of the bisacrylamide molecules have previously been consumed.

The following parameters can be taken for the gelcasting as it has been optimized in certain work. MAM and MBAM together forming 15 % of solid in 3:1 ratio respectively gives a good green strength when polymerized. APS which is 1 % of MAM+MBAM is sufficient enough to start the reaction TEMED concentration can be 3 % of APS.

2.2 Objective

An effort had been made to make gel-casting used for the making of Auminium - Alumina composite. Gel casting composites in the conventional way would create settling problem which could lead to layer formation. To overcome this limitation a special mould has to be designed to produce a homogeneously distributed green body. Samples made in powder metallurgy technique and sintered under vacuum with simple arrangements instead of sintering in inert atmosphere. Different properties especially mechanical properties of the final product were characterized and finally the green and sintered products were test for its machinability.

Chapter – 3

Experimental Work

Composite of (1-x) wt% Al: x wt % Al_2O_3 (where x = 0, 10, 20 30, 40, and 50) were prepared using both gel-casting and powder metallurgy technique. The detail experimental procedure of both the processes is given below.

3.1 Gel casting

In order to optimize the pH and concentration and concentration of deflocculating agent, slurry was made at different pH (3, 5, 7, 9, 11) by varying the deflocculant concentration (0, 0.25, 0.5, 0.75 % of solid). Zeta potential was analyzed using Zeta-Sizer.

Viscosity at different strain rate (0, 100, 200, 300, 400, 500, 600, 700, 800, 900 and 1000) were measured for slurry having 40, 45, 50, 55, 60, 65, 70 and 75% of solid loading with 0.5 % deflocculating agent with Al_20_3 and Al. Visocity was measured using Rheometer.

Slurry was made with 70 % solid loading having 50 % Al: 50 % Al_2O_3 and 0.5% deflocculating agent. The slurry was stirred thoroughly for 15 minutes and degassing was done using a vacuum desicator. To ensure perfect degassing, slurry was taken out and stirred periodically and kept again in vacuum desicator till no air bubbles will come out of the slurry. Monomer and cross linker was added to the slurry and stirred till it is dissolved completely. Mean while a small amount of white petroleum jelly was applied on the inner portion of the mould for the easy removal of the cast after the gelation. A small layer of fevicol was applied at the joint and the lid was closed air tight using the screw and nut. The fevicol was allowed to dry to ensure perfect sealing. Calculated amount of initiator and catalyst was added to the slurry and it was suddenly transferred to the mould (mould design was fabricated and discussed in design section) through the runner. The conical shape of the lid ensures complete filling inside the mould. The runner was closed with the bolt and the mould was attached to the lathe jaw followed by continued rotation at 160 rpm, the rotation was continued for about 10 minutes till complete gelation occurs. As the gelation was completed the lid was opened and the cast was removed. Enough time was given so that the cast shrinks and could be removed easily. The cast was allowed to dry completely. Same experiment was repeated at 240, 400 and 580 rpm and the cast was observed for cracks after drying. The optimum speed was found to be 240 rpm and this value was affirmed in 70% Al: 30% Al_20_3 composites too by following the entire procedure mentioned above, this speed was taken for further casting of the composite for entire composition. After drying samples of different sizes are cut from the cast for further experiment.

A diamond tipped saw cutter was used for cutting purpose. The gelcasted body was further calcined in the furnace at 620 °C for 2h under vacuum which was design (discussed in design section) and made from mild steel. XRD and SEM was performed for both green and sintered body for phase analysis and microstructural features, respectively.

3.2 Powder metallurgy

Powdered of Al and Al_2O_3 at different compositions were taken (10 , 20, 30 , 40, 50 % of Al_2O_3 and remaining Al) and mixed in the planetary ball mill for one hour in wet conditions using silicon nitride balls. Iso-propyl alcohol was used as the wetting media for the milling. The mixture was taken out of the ball mill and dried at 50 °C. This dry mixture was used for further processing. Dry powder mixture of different compositions are weighed and pressed in a die punch using a uniaxial press. No binders are added as the Al content in the mixture holds the particles together and give enough green strength. Different loads are applied and the load was optimised to 6 tonnes for all composition so that a strong green body should obtained. Acetone was used for cleaning the die punch and after each compression stearic acid was applied on the walls which act as a lubricant.

Sintering of Al: Al_20_3 composites cannot be done in open atmosphere because as the temperature rises the aluminium begins oxidising and was completely oxidised when sintering temperature was attained. An inert atmosphere has to be maintained throughout the sintering process. So, in order to get rid of the oxygen in the open atmospheres, vacuum was carried out inside the furnace by special design (design was discussed in design section) and a diffusion pump was also attached throughout the sintering process for vacuum the chamber.

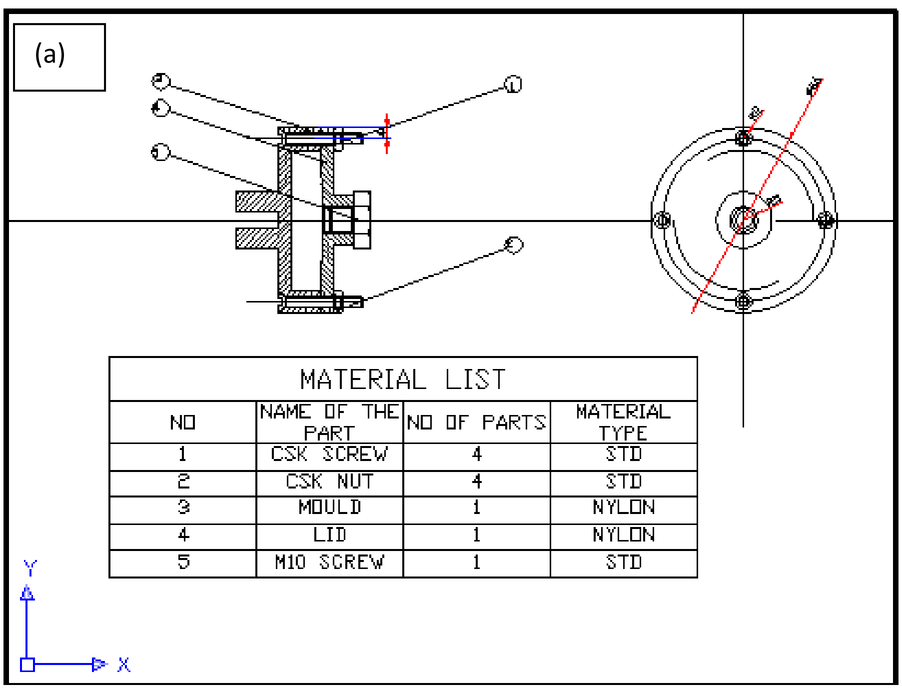
Heating rate was fixed at 3˚C/min till 620 ˚C was obtained, and it was held at that temperature for 2 h, then it was allowed to cool to room temperature inside the furnace normally. Samples which are sintered well are moved on for further testing. Samples of different size and shape are characterized using different apparatus/instruments like apparent porosity/bulk density using Archemedis principle, hardness using Vicker hardness, wear test using pin on disk machine, bending stress using UTM, microstructure using SEM and machinability using lathe and drilling machine.

3.3 Design

1. **Design of mould for gel-casting**

An air tight cylindrical mould has been designed and fabricated in nylon. Several aspects were considered for the design. Proper sealing should be attained in order to attain a sound green body. After the slurry is poured there should not remain any air bubble inside the mould. The mould should be transferred to the lathe jaws as soon as the slurry is poured. Casted body should be easily removable.

By considering all these factors the following design was considered for manufacturing. Figure 3.1a and 3.1b shows the CAD drawing and the photograph of the final mould design. In order to obtain an air tight chamber the joining surface was perfectly machined and finished to smooth surfaces. A thin layer of fevicol is sufficient enough to form a closed chamber even at rotation. The conical shape of lid ensures that no air bubble is trapped inside the mould till it is filled with slurry. When the lid is closed and screwed with four nuts only opening the atmosphere is the runner through which the slurry is been poured. As soon as slurry fills the cavity, it can be suddenly closed with a single bolt at the runner. It takes only second to fill the cavity, close the runner and to attach it to the lathe jaws.



MATERIAL LIST			
NO	NAME OF THE PART	NO OF PARTS	MATERIAL TYPE
1	CSK SCREW	4	STD
2	CSK NUT	4	STD
3	MOULD	1	NYLON
4	LID	1	NYLON
5	M10 SCREW	1	STD

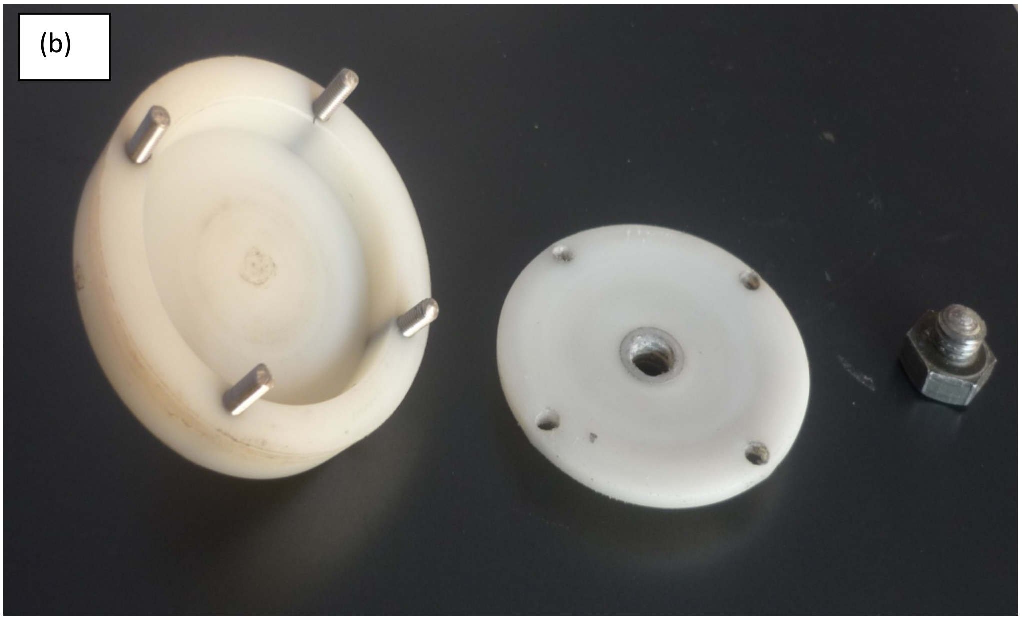


Fig. 3.1: (a) and (b) shows CAD drawing and photograph of the final mould design.

2. Design of vacuum chamber

Figure 3.2 shows the photograph of the attachment made in order to create a vacuum in the chamber, which can be placed inside an ordinary furnace. The chamber is made of mild steel and the tube material is stainless steel. Both are TIG welded air tight so that no gas leakage through the joint. On the other end of the tube a control valve is attached so as to hold the vacuum inside the vacuum chamber. This entire arrangement is connected to the vacuum pump so that a negative pressure can be created inside the chamber. The Junction between the cap and the chamber is perfectly machined and lapped for better surface finish, and is sealed with silicon sealant. A high quality silicon sealant is used for the sealing purpose which can withstand up to 370°C. The design had the advantage that it can be used in any ordinary furnace, provided that a door with a hole large enough for the tube passes should be mane using any thermal insulating material. Insulation silica brick is used which has a good insulation property. The design has the disadvantage that, the joint is inside the furnace, that is the sealant used should be able to give the sealing property at furnace temperature. A temperature of 620°C cannot withstand by the silicon sealant.

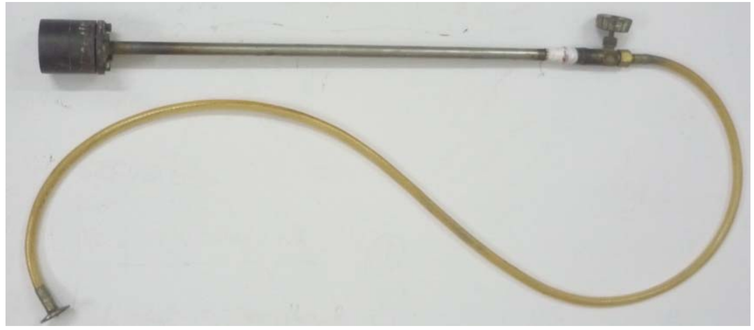


Fig. 3.2: Photograph of vacuum chamber.

3. Design of special attachment in tubular furnace

An ordinary tube furnace is modified using simple attachments to a vacuum furnace using a vacuum diffusion pump is attached to it. The arrangement is used successfully to sinter samples at 620°C at a vacuum pressure of 5.6E-6 mbars. Figure 3.3a and 3.3b shows the CAD drawing and a photograph of the attachments made for attaining vacuum. Two cups are attached to both ends of the alumina tube. One end is closed and a tube is attached to the other end. The tube is also connected to the vacuum diffusion pump. Silicon sealant is used for fixing the cups to the alumina tube. Silicon sealant gives good sealing properties at high temperature as high as 370°C. The tube along with the attachments is inserted into the furnace. The sintering temperature of 620°C will be developed in the central region of the tube and at the ends the temperature will be around 100°C, as it is exposed to the atmosphere.

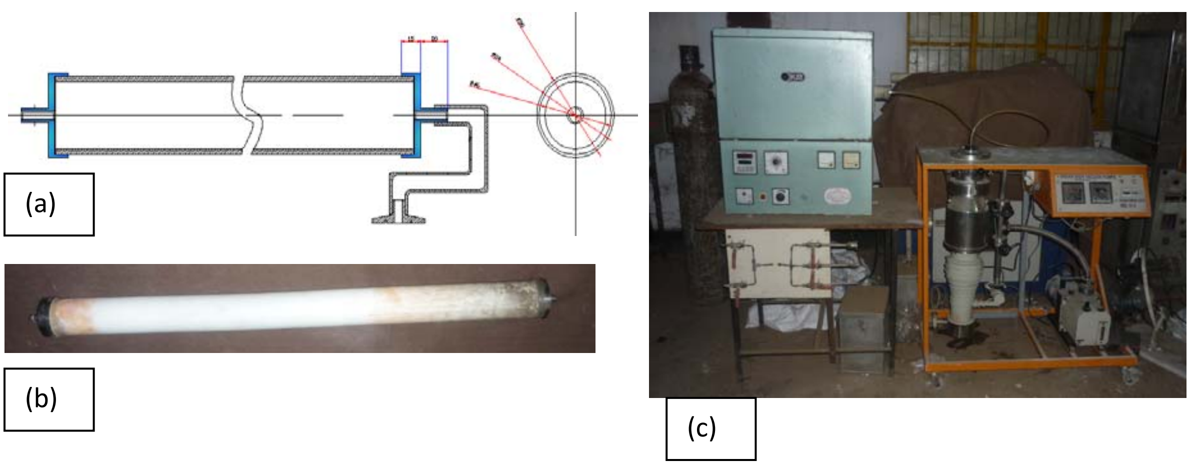


Fig. 3.3: CAD drawing (a) and photograph (b) of the attachments made for attaining vacuum in tubular furnace (c).

Chapter – 4

Results & Discussion

4.1 Fabrication of $Al\text{-}Al_2O_3$ composites using gel-casting technique

Figure 4.1 shows the variation of zeta potential at different pH ranging from 1 to 11 with different concentrations of deflocculating agent varying from 0 to 0.75 weight percentage of solid for preparing colloidal solution made of aluminium powder dispersed in water. From the curves, it was found that the optimum zeta potential was nearly at pH 9 with 0.5 weight percentage of deflocculating agent. These values are adopted for the preparation of slurry for gel casting.

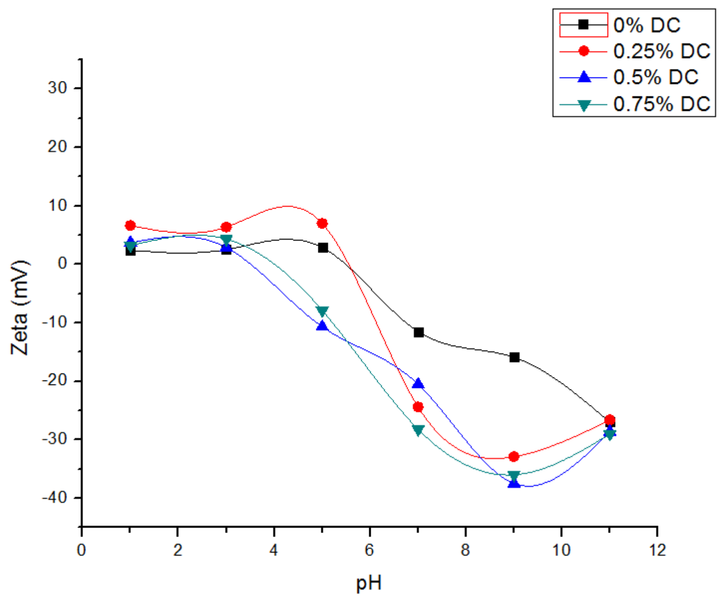


Fig.4.1: Zeta potential versus pH curves at different concentrations of deflocculating agent for preparation of aluminium slurry for gel casting.

Figure 4.2 shows the variation kinematic viscosity and shear stress with respect to strain rate, varying from 0 to 1000 s^{-1} for the colloidal solution having aluminium powder dispersed in water with varying solid loading ranging from 40% to 70%. It was observed that the shear stress was increased exponentially with shear rate at higher solid loading 65 % and 70 %, which result in constant viscosity at higher shear rate for the solid loading of 65 % and 70%. The increase in the viscosity was higher at lower shear rate and varies gradually at higher shear rate.

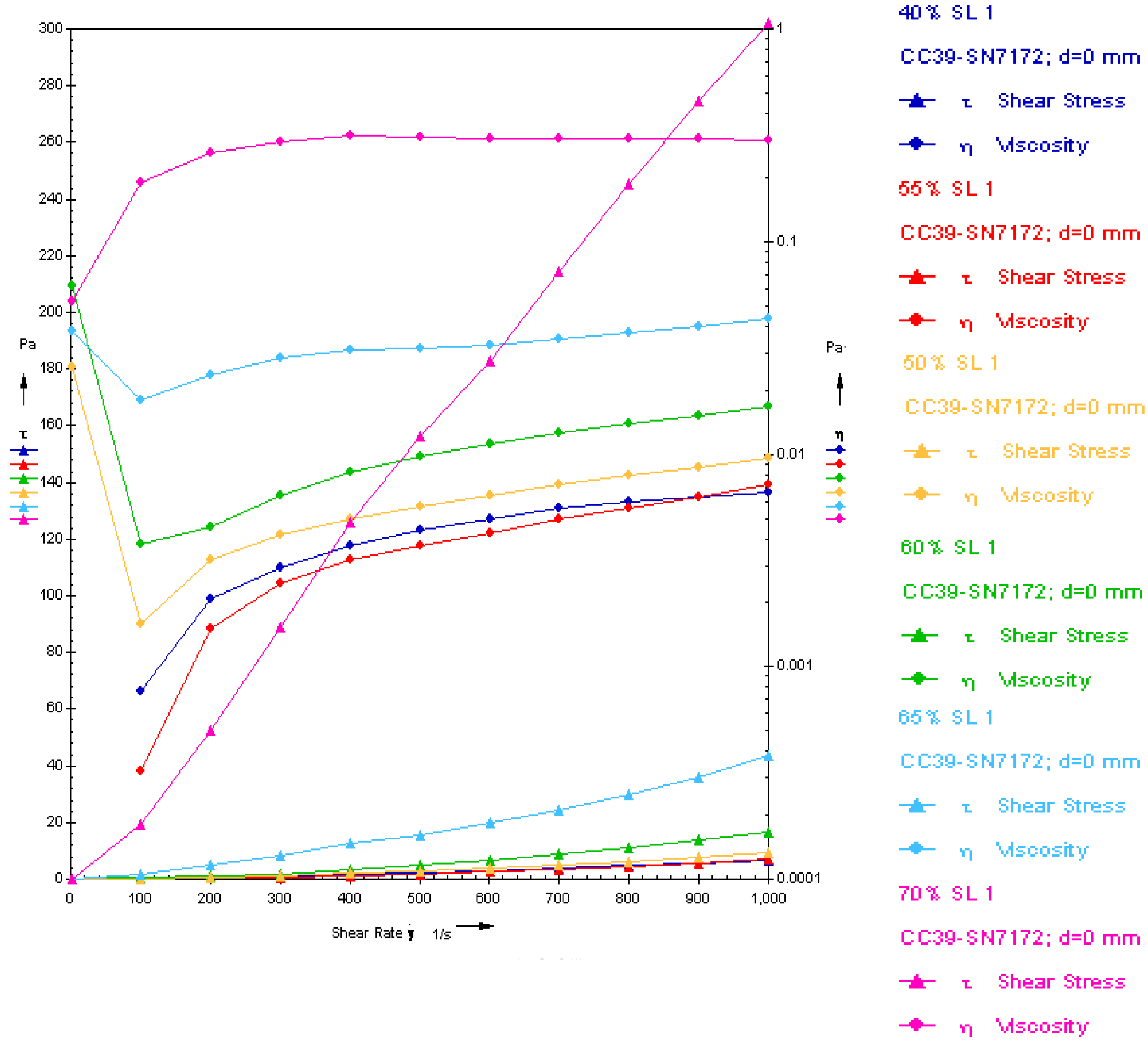


Fig. 4.2: Variation kinematic viscosity and shear stress with respect to strain rate of colloidal solution having aluminium powder with different solid loading.

Figure 4.3 shows the variation kinematic viscosity and shear stress with respect to strain rate, varying from 0 to 1000 s^{-1} for the colloidal solution having alumina powder dispersed in water with varying solid loading ranging from 40% to 75%. It was observed that the shear stress was increased exponentially with shear rate at higher solid loading 70 % and 75 %, which result in constant viscosity at higher shear rate for the solid loading of 70 and 75%. The increase in the viscosity was higher at lower shear rate and varies gradually at higher shear rate.

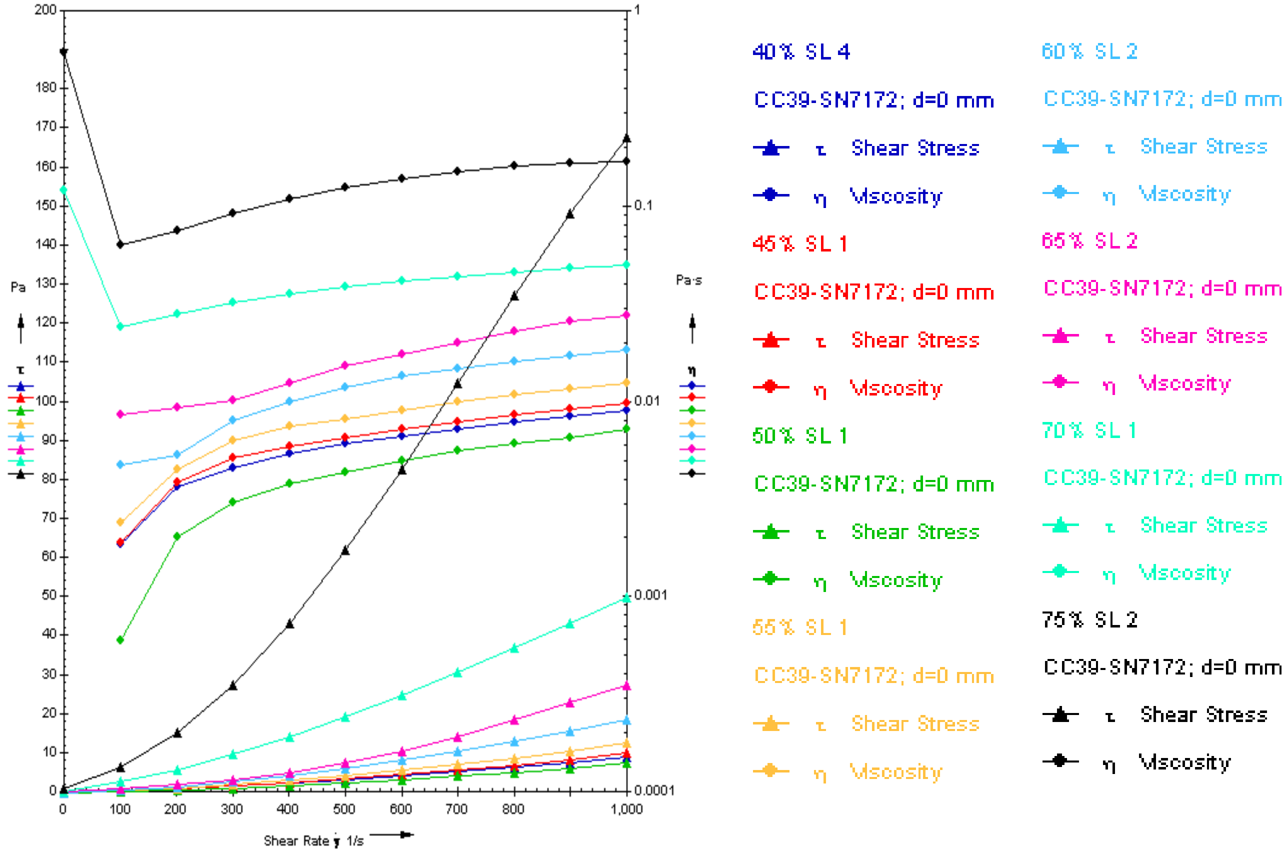


Fig. 4.3: Variation kinematic viscosity and shear stress with respect to strain rate of colloidal solution having alumina powder with different solid loading.

Figure 4.4 shows the variation of viscosity at the shear rate of 500 s^{-1}, with respect to the solid loading of both alumina and aluminium powder. It was found that as the solid loading increases from 40 to 55%, the viscosity reduces and further it increases with solid loading. Therefore 55% is most appropriate solid loading for the gelcasting from rheological point of view. But with this solid loading, shrinkage after drying was much higher, which may lead to crack formation in the green body. Therefore solid loading of 70% is accepted for casting purpose which has less shrinkage as well as acceptable flowability.

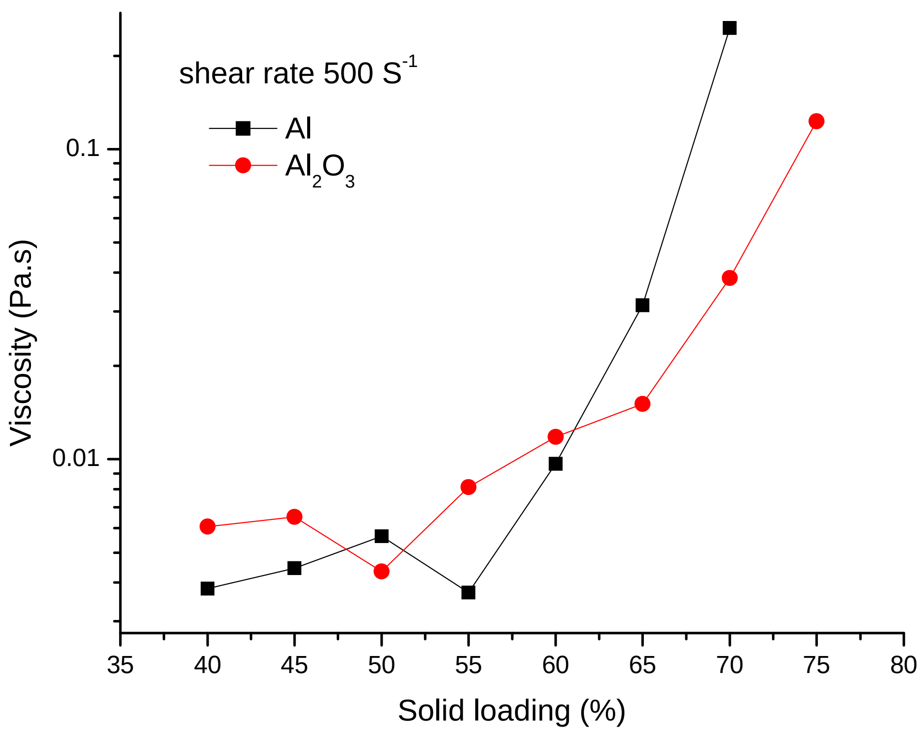


Fig. 4.4: Variation of viscosity with respect to the solid loading of both alumina and aluminium powder.

Figure 4.5 shows the image of the green body made of conventional gravity casting. It is clearly seen that the aluminium particles got settled down before gelation starts and two layers were formed which resulted in non-uniform shrinkage which lead to crack formation. There a special cylindrical mould was designed and fabricated (discussed later in design section). As soon as the slurry is poured, the mould is closed air tight and rotated longitudinally at a particular rpm till gelation process is completed.

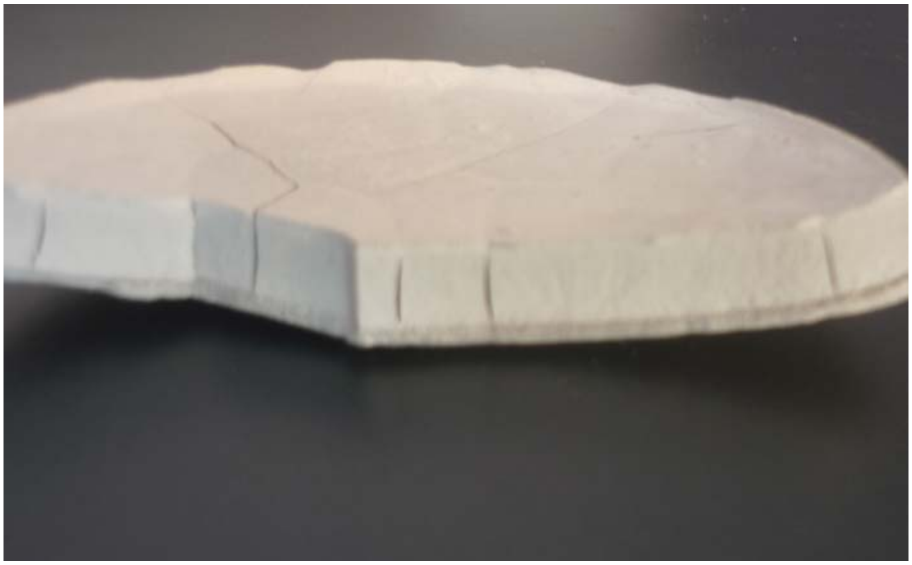


Fig. 4.5 Composite made of gelcasting in conventional way.

Figure 4.6 shows the image of green body after drying, which was produced by gelcasting way at a rotating speed at 160, 240, 400 and 580 rpm. It can be clearly observed that at 580 rpm the slurry had undergone centrifuge action resulting in the segregation of coarser particles at the outer circumference. As a result three layers are formed depending upon the particle size which can be seen with naked eyes. This formation of layer resulted in generation of cracks after drying due to the non uniform shrinkage. Sample rotated at 400 rpm also resulted in crack due to the same reason. The sample rotated at 240 rpm has uniform particle distribution, which gave the advantage of crack free green body after drying. 160 rpm is not enough to form a homogenous body as the coarser particles keep on settling inside the mould even while rotating. The sample prepared at 240 rpm gives better green gelcasted product and this rpm was fixed to prepare other compositions.

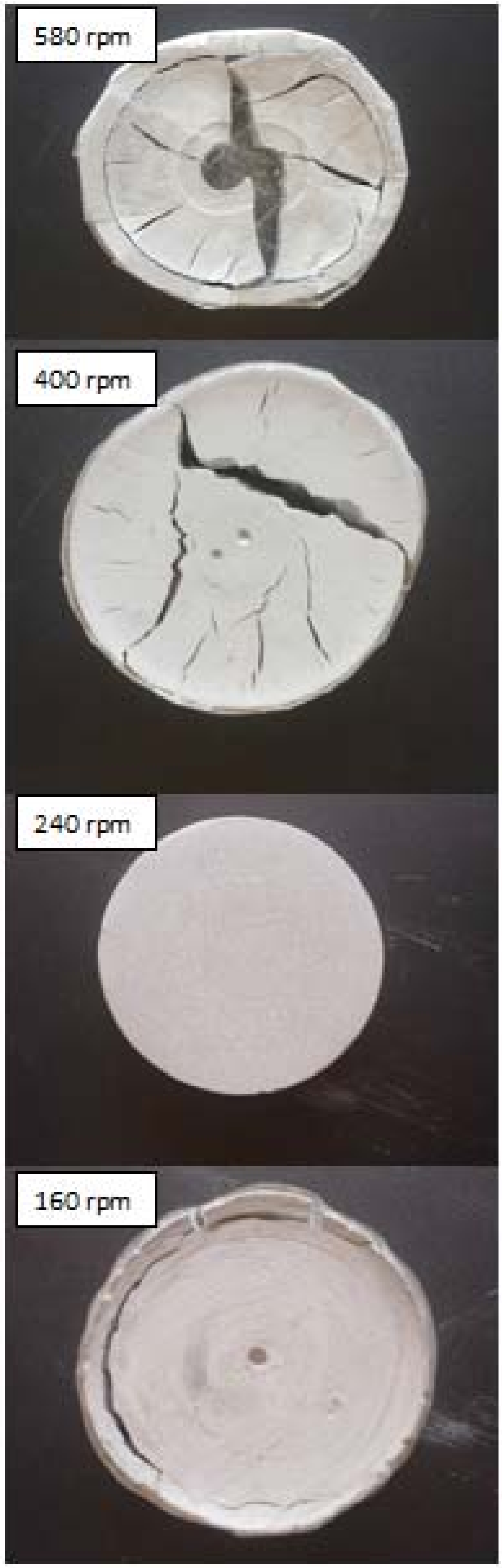


Fig. 4.6: Image of green gelcasted body after drying prepared through at different rpm.

Figure 4.7 shows the sample made of gelcasting which was tested for its machinability. The gelcasted body was found to be machinable in lathe drilling machine. A hole of 3.3mm was made and it was given an internal thread using tap drill of M4 size.

Fig. 4.7: Machinable green gelacted product

Figure 4.8 (a) shows the green product of Al-Al_2O_3 composites and fired in vacuum at 620 °C for 2 hours (Fig. 4 b). It wasobserved that the samples are not been sintered to its full density. The porosity in the sample is high which resulted in less strength.

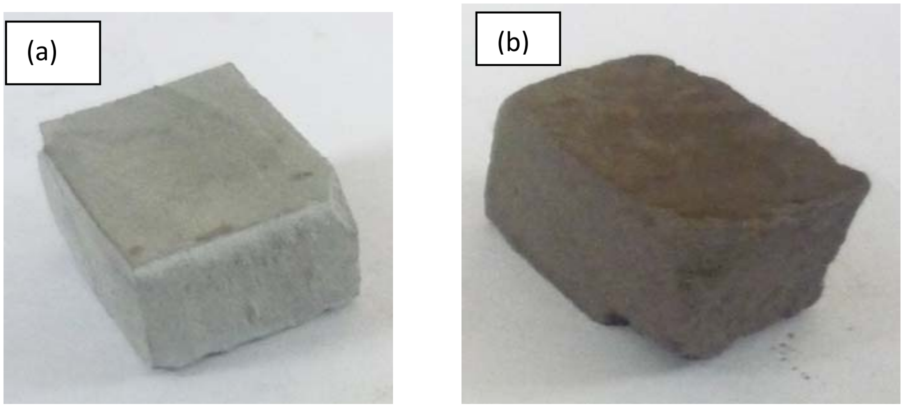


Fig. 4.8 (a) and (b) shows the green and fired gelacted product.

Figure 4.9 shows the SEM image of (a) green body and (b) sintered samples made of gelcasting. It was seen that the aluminium is dispersed uniformly along the aluminium matrix in the green body. There are no such distinct particles in the fired sample, XRD analysis (discussed later) shows no sign of oxidation of aluminum. It can be assumed that the aluminium would have artially mented and covered the alumina content in the cast. A large number of pores are also visible which justifies the argument for the low strength of the sintered body.

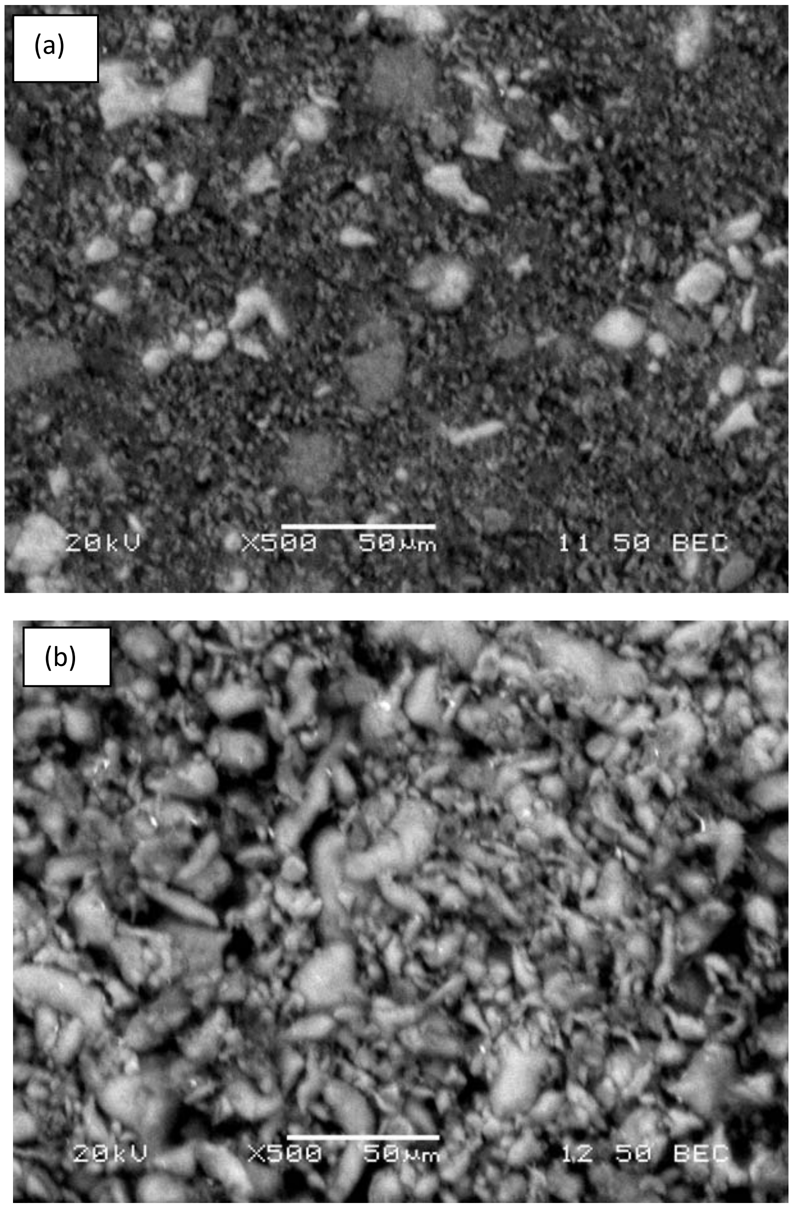


Fig. 4.9: SEM image of (a) green body and (b) sintered $Al\text{-}Al_2O_3$ product made by gelcasting.

Figure 4.10 shows XRD pattern of the green and fired Al-Al_2O_3 product, prepared by gelcasting way. All the peaks are identified with wither Al (JCPDS file no: 04-0787) or Al_2O_3 (JCPDS file no: 81-1667). The volume percentage of Al and Al_2O_3 was calculated and it was nearly 80: 20.

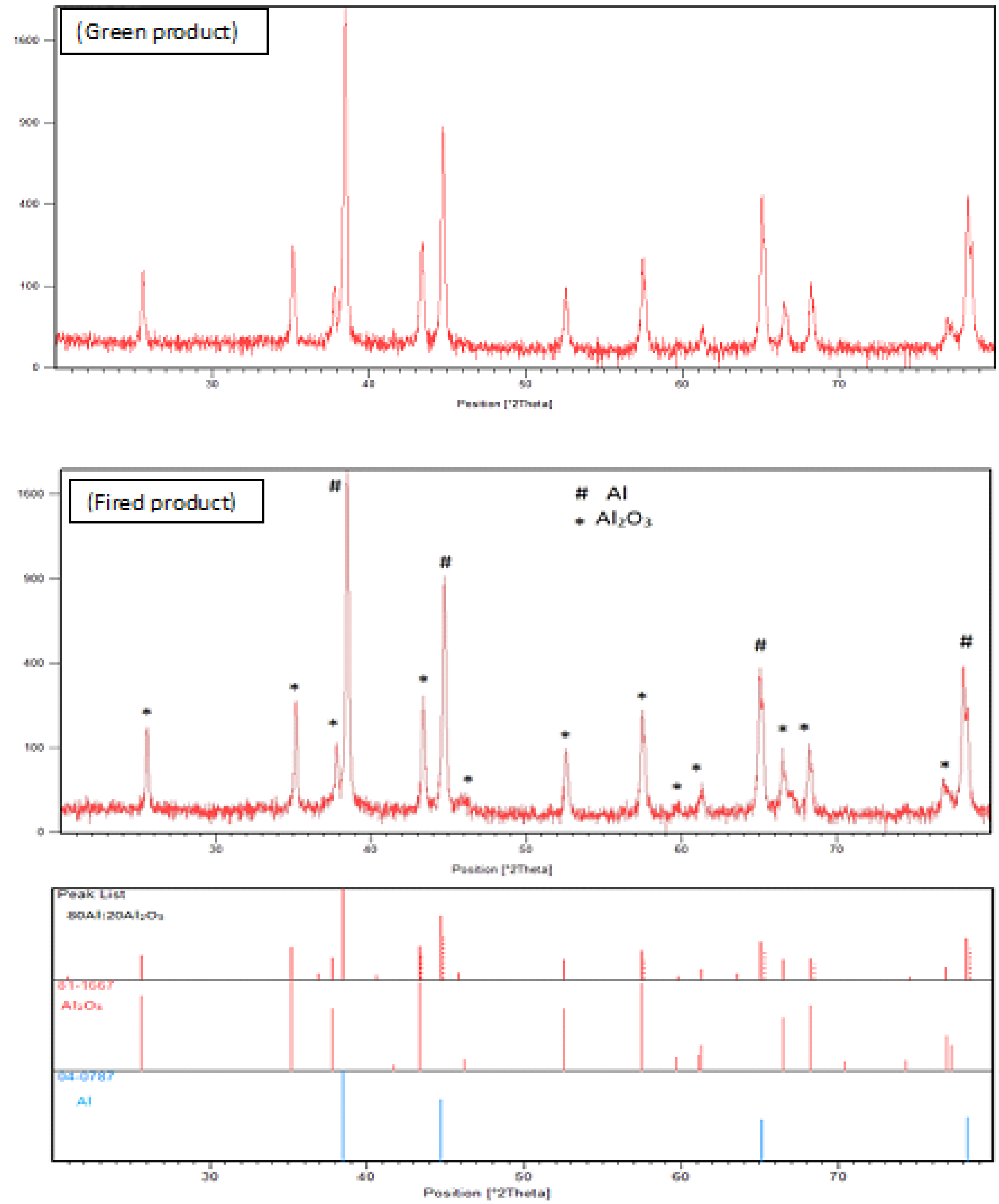


Fig. 4.10: XRD pattern of green 80Al-20Al_2O_3 product and fired product.

Remarks

Green body of Al/Al_2O_3composite was successfully made using gel-cast, in which the matrix and the reinforcements are homogenously distributed. Certain aspects prevent the sintering of the sample, resulting in large pores which result in low strength of the composite. Further studies are required in order to overcome these limitations, and to implement the technique for the manufacturing of Metal Matrix Composites, which could be a breakthrough in the manufacturing of composites using gelcasting. Further, the product was prepared through powder metallurgy process.

4.2 Fabrication of Al-Al_2O_3 composites using powder metallurgy technique

Figure 4.11 shows the green (a) and sintered (b) product of Al-Al_2O_3 composites with different compositions and fired in vacuum at 620 °C for 2 hours.

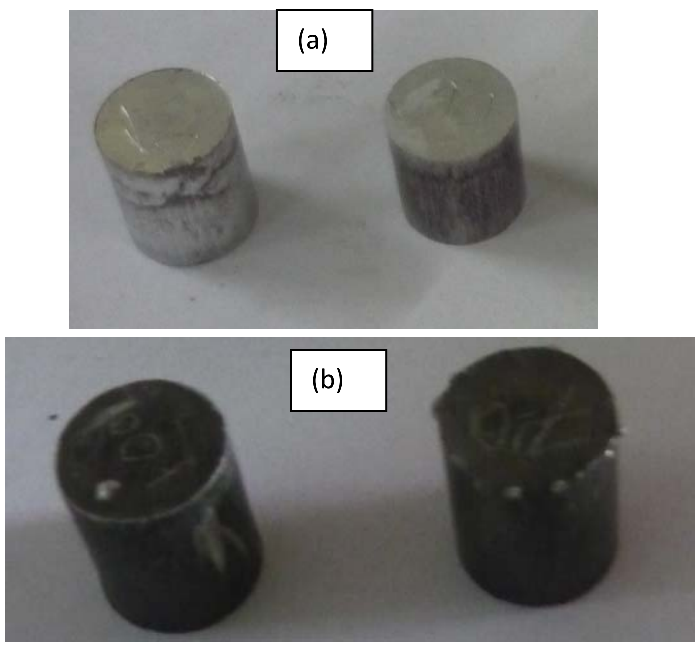


Fig. 4.11: Image of green (a) and sintered (b) body prepared through powder metallurgy process.

Figure 4.12 shows the chart showing the variation of apparent porosity and bulk density of the green body. Apparent porosity of sample with 0% alumina is found to be 1.672%. As the alumina percentage increases the apparent porosity increases to 17.41 %. This was due to the pores present in the ceramic particle. Bulk density in the acceptable range was formed in every composition.

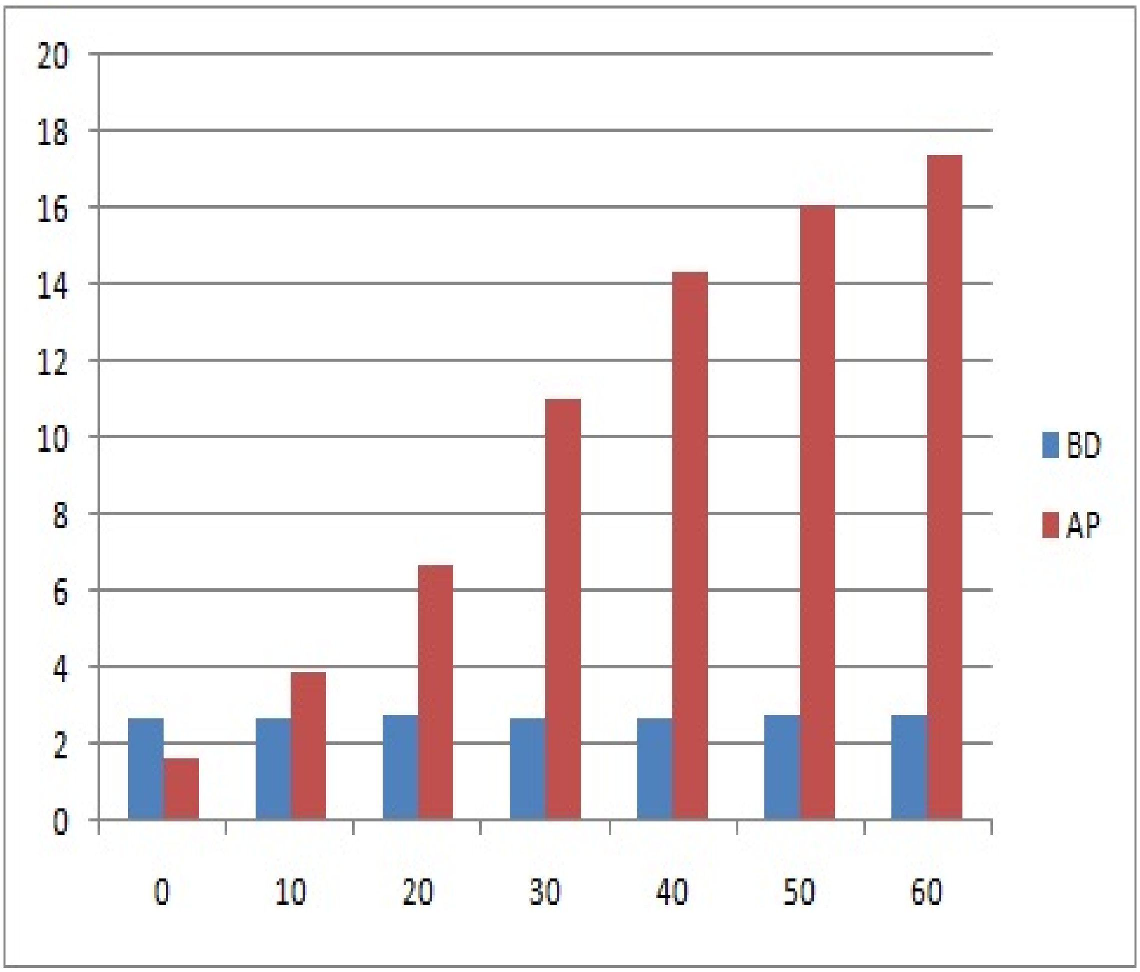


Fig. 4.12: Chart showing the variation of apparent porosity and bulk density of the green body.

Figure 4.13 shows the chart of apparent porosity/ bulk density respectively for the sample sintered in vacuum atmosphere. When compared with the AP/BD of the green body, it is evidently proved that the samples are sintered to high density. There was reduction in the apparent porosity in every composition.

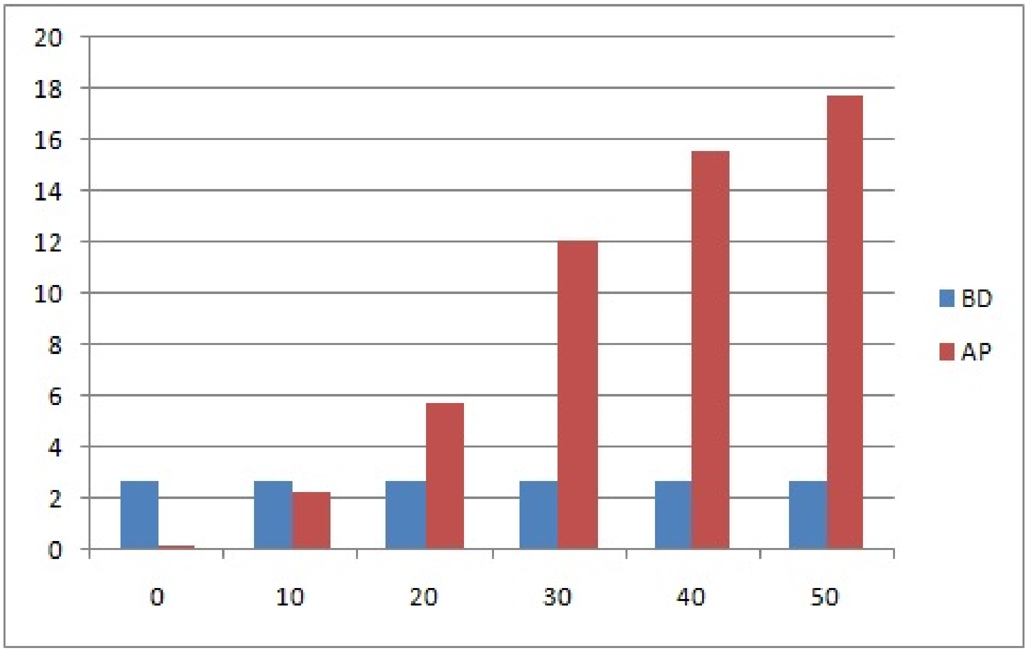


Fig. 4.13: Chart showing the variation of apparent porosity and bulk density of the sintered body.

Figure 4.14 shows the variation in the Vickers hardness number with respect to the percentage composition of alumina. Solid aluminium has a hardness of 25 in Vickers scale. Same value is obtained using powder metallurgy process. As the alumina content is increased hardness of the composite is also increased to a maximum value of 35 at 20 % of alumina. This shows that an excellent bond is formed between alumina and aluminium matrix up to 20%. Beyond 20% the hardness is reduced which may be due to the week bond between the matrix and the reinforcement. The ceramic particles would be in direct contact resulting in week bond between matrix and the ceramics.

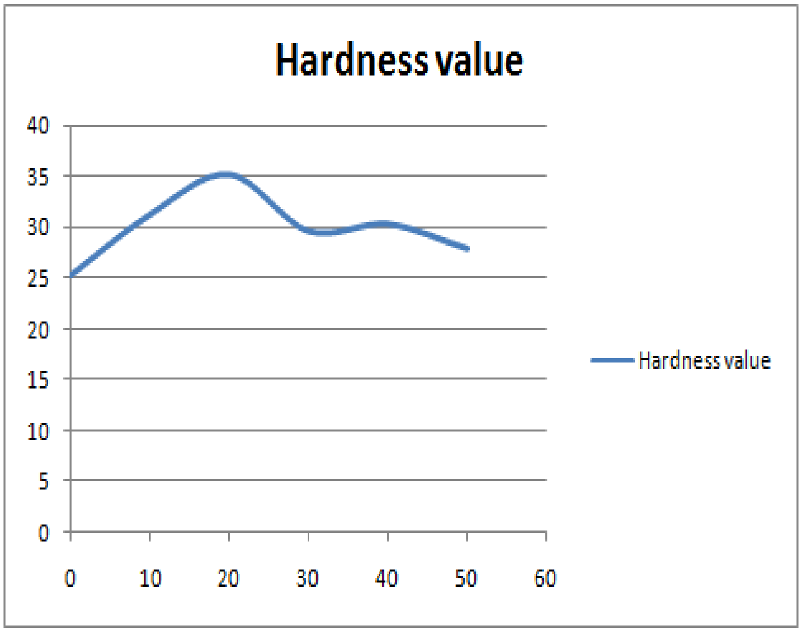


Fig. 4.14: Vickers hardness with respect to the percentage composition of alumina.

Figure 4.15 shows graph of bending stress vs. bending strain of the composite at various compositions. The test result shows that with 0% alumina, the material shows plastic and elastic property to a higher extend. As the alumina is added the plasticity is reduced drastically. At 10, 20 and 30% of alumina the elastic property is more or less same as pure aluminium and average plasticityis obtained. Therefore composite having this composition can be used for certain applications like engine casing, push rod, rocker arm etc. At 40 and 50 % alumina the plasticity and the elasticity both are reduced and hence it cannot be used for structural application having bending load.

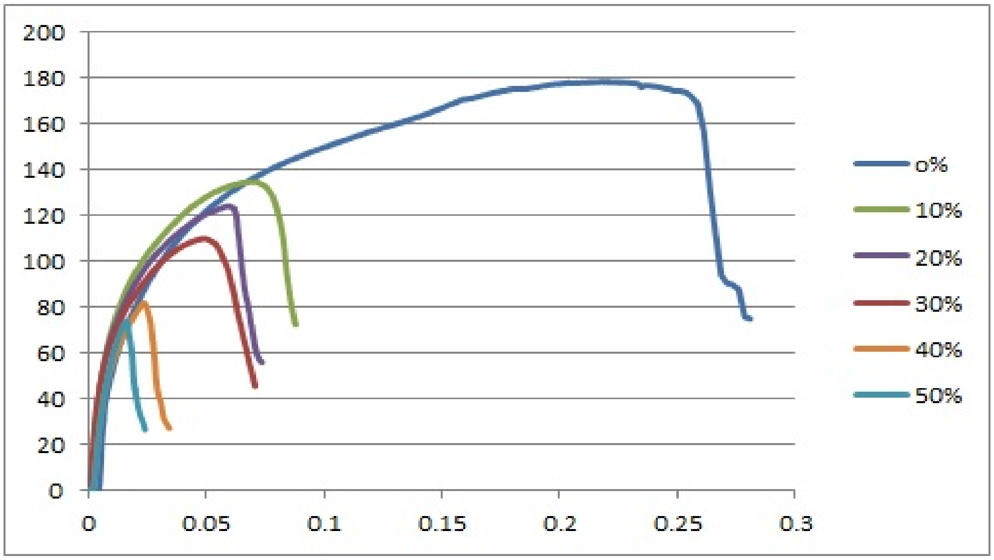


Fig. 4.15: Bending stress versus bending strain of the composite at various compositions.

Figure 4.16 shows the wear rate in g/m of sample of composition at different speed of 2.62, 3.93, 5.24 m/sec and with a varying load of 1 kg, 2kg and 2.5 kg. The Wear rate is increased as the load is increased. This is due to the face that frictional force is increased due the increase in the load. This leads to higher wear rate in the composite. At higher load of 2.5 kg the surface was chipped off from the surface and stick on the surface of pin on disc, for the composite having 0% and 10% alumina. Therefore these compositions were not considered further testing at that load. The wear rate first decreases with sliding speed and then increases. This is due to the fact that under low sliding speed and load, abrasion wear mechanism becomes dominant. Under low loads, the hard alumina particles support the normal pressure on the surface resulting in increased wear resistance with greater presence of the alumina in the composite. This is similar to the results of Amro M. Al-Qutub, Ibrahim M. Allam, M. A. Abdul Samad [10] who suggested that this effect diminishes at higher sliding speeds and loads due to the fragmentation of particulates. The wear rate was better in 80Al-20Al_2O_3 composites in almost all conditions. Maximum hardness was obtained at this composition. Comparing both these results it can be stated that at this composition alumina particles are firmly held by aluminium. These alumina bears the load to a great extend and decreases the wear rate.

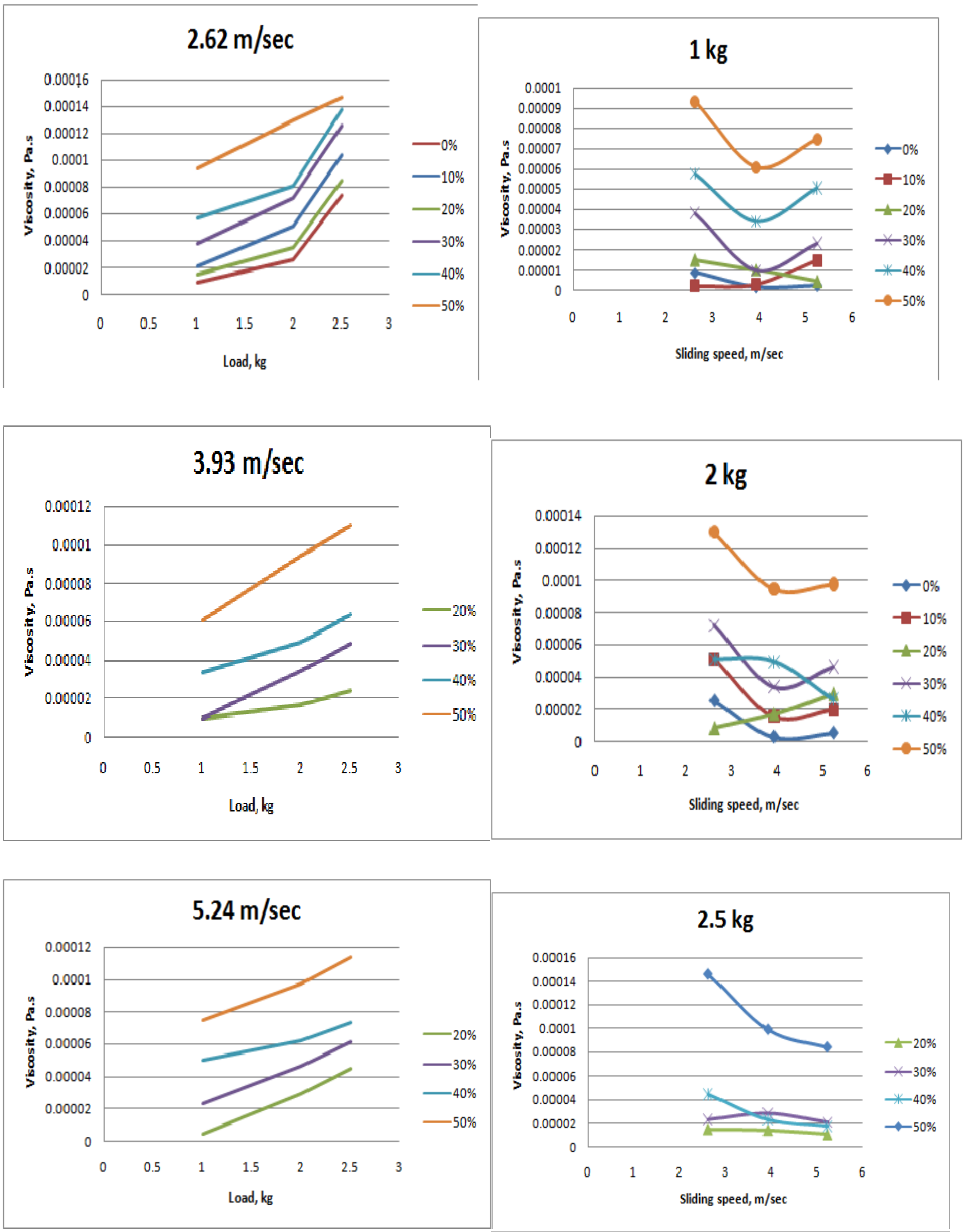


Fig. 4.16: Wear rate (in g/m) of different compositions of Al-Al_2O_3 composites under different load and different speed.

Figure 4.17 shows the SEM image of sintered composites containing 60 Al and 80 Al made of powder metallurgy process. It was seen that the aluminium was nearly elliptical shape and well dispersed in the sintered body. The dark gray phase in Fig. is Al_20_3, while the light gray phase is aluminum. Here again, no other phases are identified, except alumina and aluminum. The dark black areas are pores.

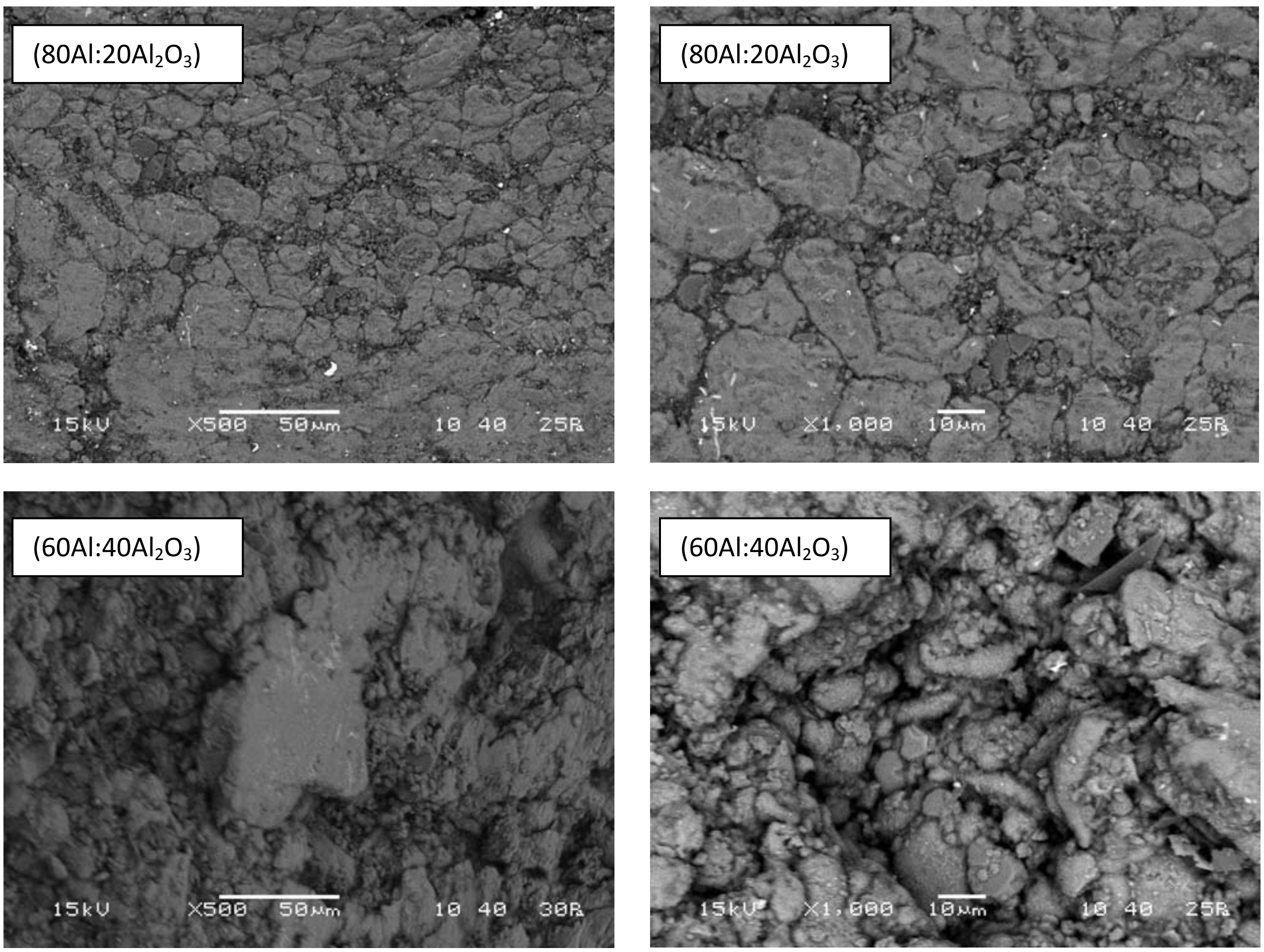


Fig. 4.17: SEM images of $80Al:20Al_2O_3$ and $60Al:40Al_2O_3$ composites.

Figure 4.18 shows the sample made of powder metallurgy process, which was tested for its machinability. The body was found to be machinable in lathe drilling machine. A hole of 3.3mm was made and it was given an internal thread using tap drill of M4 size.

Fig. 4.18: Machinable sintered product prepared through powder metallurgy process.

Remarks

It was found that powder metallurgy is a good technique for the manufacturing of Al/Al_2O_3 composite. Homogeneous distribution of Al and Al_2O_3 was attained. Design and arrangements made for vacuum sintering was found to be successful as no sign of oxidation is found in XRD analysis. Composition having 20% alumina shows better property that pure aluminium and other compositions. This is due to the fact that, alumina particles are firmly held by the aluminium matrix and a good bond is obtained between them. At higher percentage of alumina, the particles are close to each other with limited quantity of aluminium between them to hold the particles together, thus leads to poor strength. The machinability is found to be excellent for the composite which significantly reduce the machining time and cost when implemented in component production.

Chapter – 5

Conclusions

Conclusions

Green body of Al: Al_2O_3 Composite of different compositions were successfully made using both gelcasting and powder metallurgy technique. Samples made by powder metallurgy were sintered successfully under vacuum condition.

- A homogeneously distributed green body was obtained using gelcasting. The mould design and the processing route was therefore suits the purpose. The green body was easily machinable.
- Thus the mould can be used for the manufacturing of any composite using gelcasting, provided that the speed of rotation should be optimized according to the particle size and the density.
- Certain aspects prevent the sintering of the sample, resulting in large pores which result in low strength of the composite. Further studies are required in order to overcome these limitations, and to implement the technique for the manufacturing of Metal Matrix Composites, which could be a breakthrough in the manufacturing of composites using gelcasting.
- The vacuum attachment for sintering purpose worked properly and was able to produce a negative pressure of 5.6×10^{-6} mbars inside the chamber.
- Composite made using powder metallurgy was sintered successfully in vacuum condition, which shows excellent mechanical properties compared to unreinforced aluminium.
- Hardness was improved from 25 HV to 35 HV by adding 20 wt % of alumina into aluminium.
- Bending strength was reduced adding alumina into aluminum. Plastisity of composite with 10, 20, and 30% alumina is more acceptable for certain specific application.
- Excellent wear resistance was noticed in the composite having 20% alumina which makes it suitable for manufacturing components which are having sliding contact in the system.
- Excellent machinability was observed in the composite, which makes it easy manufacturing which helps in reducing the manufacturing cost to a greater extend.

Thus it has been concluded that Al: Al_2O_3 composite prepared by powder metallurgy technique and sintered under vacuum condition gives better mechanical properties and this material could replace conventional materials in various automotive, aerospace, and automobile applications.

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