DEVELOPMENT AND STUDY ON DIFFERENT PROPERTIES OF ALUMINIUM- CRYSTALLINE SILICA CERAMIC MATRIX COMPOSITES AT DIFFERENT SINTERING TEMPERATURES

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Abstract— Ceramic based composites are widely used around the world in demanding thermal, structural and electrical insulating applications. This present study aims to develop aluminium-crystalline silica ceramic based composites at different sintering temperatures with average particle sizes ranging from -150 to -200 micron. Crystalline silica matrix composites with 2.5, 5, 10, 40 Wt. % of aluminium were developed through powder metallurgy techniques. This research focus on the development of aluminium-crystalline silica ceramic based composites with optimizing its sintering temperature based on its physical as well as mechanical properties. The surface morphology of the composites were observed by optical microscopy (OM) and scanning electron microscopy (SEM). Physical property like density, porosity, shrinkage, and mechanical property like hardness were analyzed and reported for various sintering temperatures.

Keywords— Aluminium- Crystalline silica, Ceramic matrix, Powder Metallurgy, Microstructure, SEM, Shrinkage, Density, Porosity, Hardness.

INTRODUCTION

The requirement for new materials are increased to fulfill the modern engineering demands and has led to the development of composite materials. Ceramic based composites are the oldest and newest materials used in structural and high temperature applications due to its attractive properties such as high melting points, high compressive strength, and good strength at elevated temperature. Ceramic based composites are also used in power sectors as insulating materials for its excellent resistance to oxidation, good dielectric properties. These properties make it suitable for using in harsh environment applications [1-3]. But ceramic materials suffer due to lack of fracture toughness, tensile strength and brittleness. These properties can be improved by incorporating suitable ductile material as second phase [4].

Aluminium metal matrix composites are preferred and widely used in automobile industry for making engine parts. Aluminium has advantages over other metals such as good casting abilities, high corrosion resistance, low thermal expansion, wear resistant, and low density [5-9]. But low strength and low melting point are the problems for using aluminium matrix composites and its alloys [10] and makes them unable to use in high temperature applications. Metal matrix composites are also not suitable for harsh environment applications due to its chemical reactivity. These problems can be solved by using ceramic matrix composites binder with aluminium. The selection of the second phase material depends upon the application of the fabricated composites and its compatibility between matrix and reinforcement. Load carrying capacity of composites depend on the nature of matrix and reinforcement, the fabrication technique, and the sintering temperature. If the bonding between matrix and second phase is strong then crack propagation will be restricted.

In the past research have been carried out on aluminium metal matrix composites with SiC, Al_2O_3, TiC, B_4C, Ti-Aluminide, Si or SiO_2 as reinforcement [5, 11-17]. Aluminium makes good bonding with crystalline silica and as a result better adhesion takes place between matrix and reinforcement in crystalline silica aluminium composites [17]. But the selection of crystalline silica as matrix material and aluminium as second phase is rare. Here, aluminium will acts as a binder in the ceramic matrix and will overcome the excessive brittleness and lack of mechanical reliability of monolithic ceramics [4].

This paper describes about the development of Aluminium- Crystalline silica ceramic based composites by varying the Wt. % of aluminium into crystalline silica matrix for various sintering temperatures and finally optimize this sintering temperature based on its various physical and mechanical properties.

EXPERIMENTAL PROCEDURE

2.1 Development of Aluminium- Crystalline Silica Ceramic Based Composite by Powder Metallurgy Technique

The matrix material used in the present study was crystalline silica (SiO_2), extracted from natural sand and commercially pure aluminium (Al) powder (99.7 %) procured from LOBA Chemie, India. Sand was collected from the river side of Damodar at Burdwan district in West Bengal, India for producing silica. Silica was extracted by heating the natural sand at 1200^0C for 1 hour in muffle furnace (made by Nascor Technologies Private Limited, Howrah, West Bengal, India) at open atmosphere.

Then the mixture of aluminium- crystalline silica powder with composition 2.5%, 5%, 10% and 40% of aluminium (based on Wt. %) were mixed in hand driven mortar parcel until very fine powder was produced, mesh size in the range between -150 µm to -200 µm. The compact powder was uniaxially hard-pressed using a steel mold having an internal diameter of 10 mm at a pressure of 150 MPa, with a 2-ton press for 4 min from PEECO hydraulic pressing machine (PEECO Pvt Ltd, M/C NO.-3/PR-2/HP-1/07-08). Finally the samples were sintered in the same muffle furnace at temperature 1000 ºC, 1100 ºC, 1150ºC and 1200 ºC for 2 hours at a constant heating rate of 5ºC/min. After heating, samples were cooled slowly in the same furnace.

2.2 Testing and Characterization

2.2.1 XRD Analysis

X-ray diffraction (XRD) patterns were obtained for aluminium- crystalline silica composites using RigakuUltima III analytical difractometer (with Cu-Kα radiation, λ = 1.54059 Å) at 30 kV and 15 mA. Intensity data were plotted by the step-counting method between 10˚ and 80˚ (2θ) for each sample. The presence of various phases were selected and identified by comparing with standard JCPDS files.

2.2.2 Microstructure

For observing microstructure, the ceramic based samples were mirror polished and then microstructures were observed at 20 X magnification by LEICA Optical Microscopy model no DM-2700M Image Analyzer.

2.2.4 SEM and EDX Analysis

SEM and EDX analysis were performed for each sample using JEOL MAKE SEM model JSM 6360, operated by PCSEM software.

2.2.5 Density, Apparent Porosity and Shrinkage Measurement

Weight and dimension were measured for each sample before and after sintering. From the dimension measurement both green and sintered densities and shrinkage were calculated. Apparent Porosity was measured using the universal porosity measurement technique.

2.2.6 Micro-hardness Survey

Micro-hardness survey was carried out by using 200 gf loads with 15 sec dwell time. The hardness was measured in five different positions for each sample and finally average the hardness numbers them for getting more accuracy by using Leco Micro Hardness tester (Model LM248SAT).

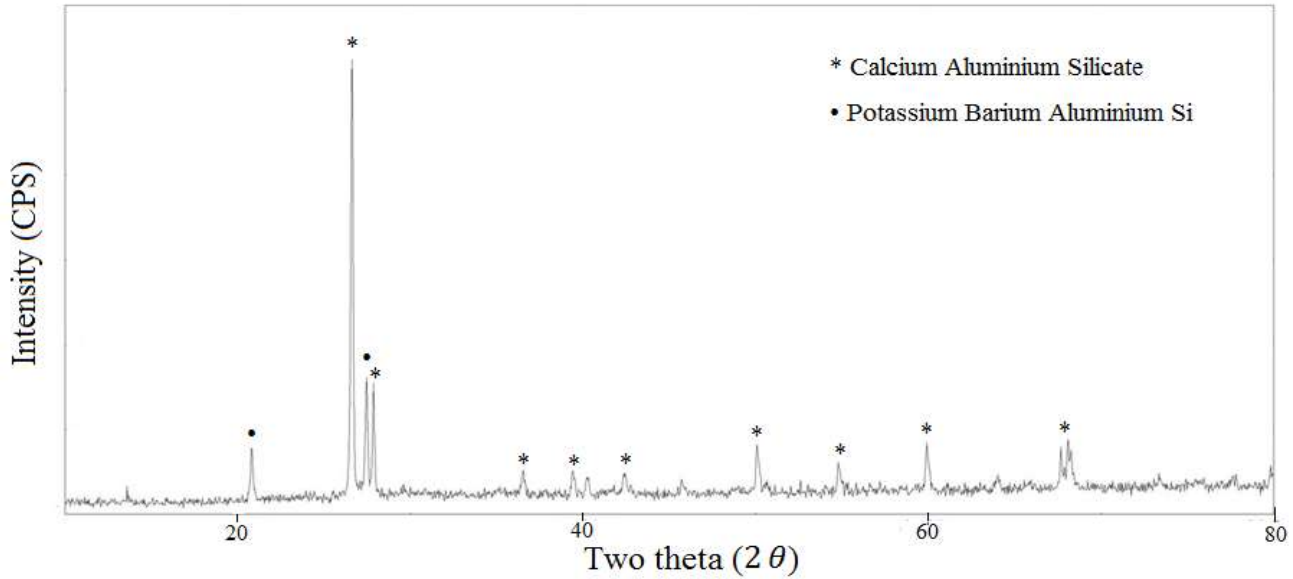## 3. RESULTS AND DISCURSION

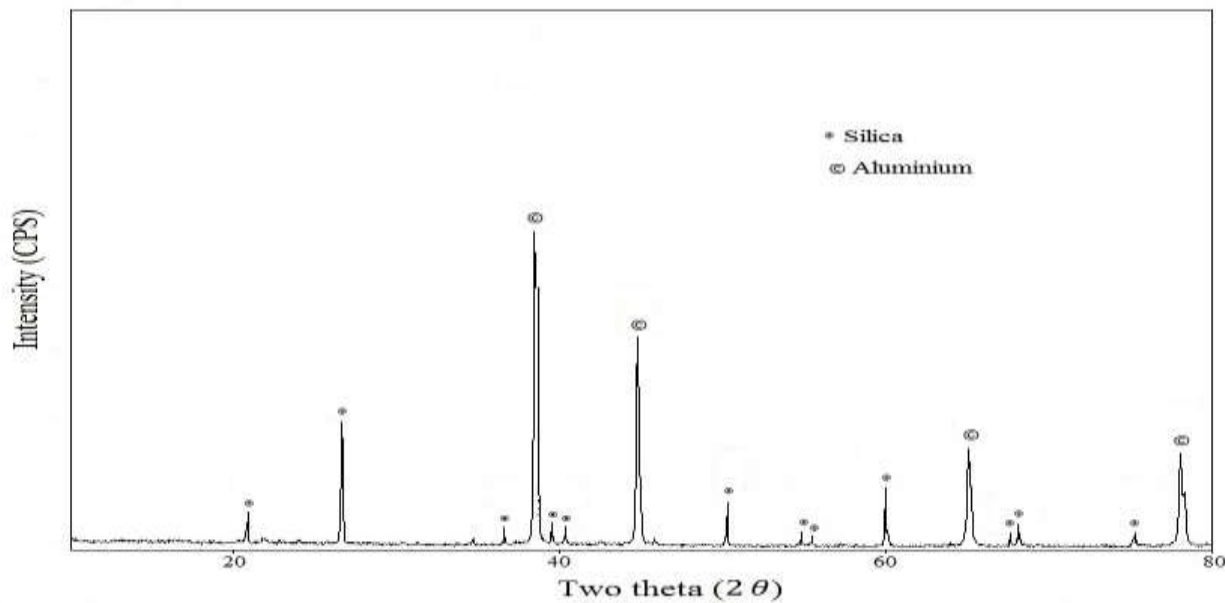## 3.1 XRD Analysis

Fig. 1 XRD analysis of natural sand

Fig. 2 XRD analysis of Aluminium- Crystalline silica composites after sintering

In the present study, here silica was extracted from natural sand. The XRD pattern for the natural sand is shown in fig.1. Calcium Aluminium Silicate was the main constituent component in this sand, present at 2θ= 26.600 (d= 3.3482), 27.880 (d= 3.1973), 36.520 (d= 2.4583), 39.400 (d= 2.2850), 42.360 (d= 2.1319), 50.080 (1.8199), 54.800 (d= 1.6737), 59.920 (d= 1.5424), 71.440 (d= 1.3193), 71.600 (d= 1.3168). A crystalline phase of Potassium Barium Aluminium Si is present at 2θ= 20.800 (d= 4.2669) and 27.440 (d= 3.2476) in the same sand as shown in fig.1.

The XRD patterns of sintered aluminium- crystalline silica composites at $1150\,^{\circ}C$ temperature are shown in Fig. 2. The composite sample contains two crystalline phases – Aluminium and Crystalline silica; the characteristic peaks of Aluminium were observed at 2θ= 38.493°(d= 2.3369), 44.739° (d= 2.0240), 65.012° (d= 1.4334) and 78.019° (d= 1.2238); and crystalline silica was present at 2θ= 20.802° (d= 4.2668), 26.622° (d= 3.3456), 36.551° (d= 2.4564), 39.482° (d= 2.2805), 40.291° (d= 2.2366), 50.152°

(d= 1.8175), 54.902^o (d= 1.6710), 55.244^o (d= 1.6614), 59.918^o (d= 1.5425), 67.567^o (d= 1.3853), 68.091^o (d= 1.3759) and 75. 5° (d= 1.2582) as shown in fig. 2. Figure 2 shows nearly same peak for the composites sintered at 600^{0}C [17]. As aluminium and silica gives individual peak and they form no compound, there was no reaction between aluminium and crystalline silica in this sintering environment.
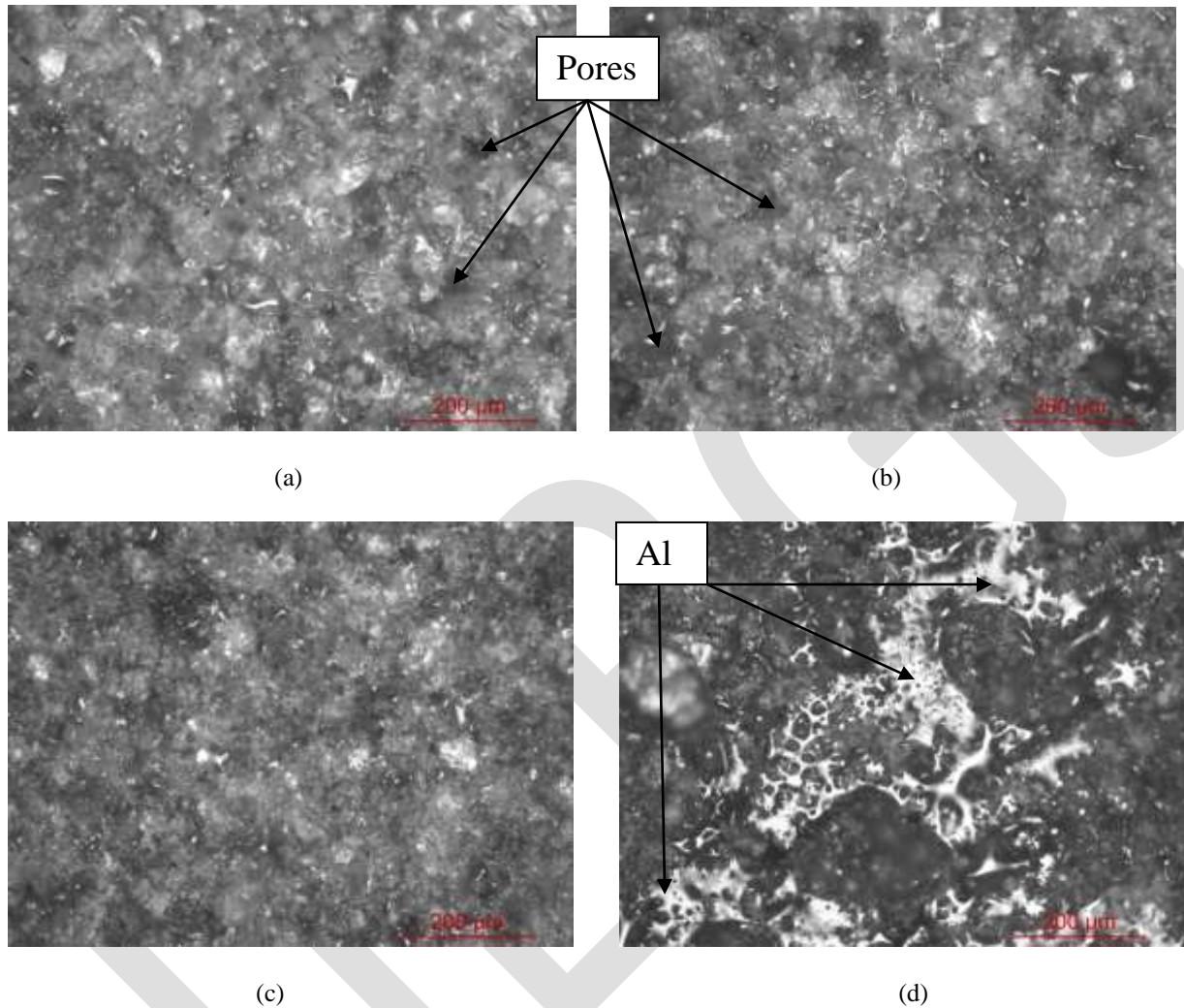
3.2 Microstructure

(a)   (b)

(c)   (d)

Fig. 3 Aluminium- crystalline silica composites sintered at 1150^{0}C for (a) 2.5 Wt. % Al (b) 5 Wt.% Al, (c) 10 Wt.% Al, (d) 40 Wt.% Al

In the fig. 3, black portion indicates crystalline silica while white portion represents aluminium. Aluminium is well distributed throughout the matrix. Here, aluminium acts as binder and helps to reduce porosity hence improves strength of the material.
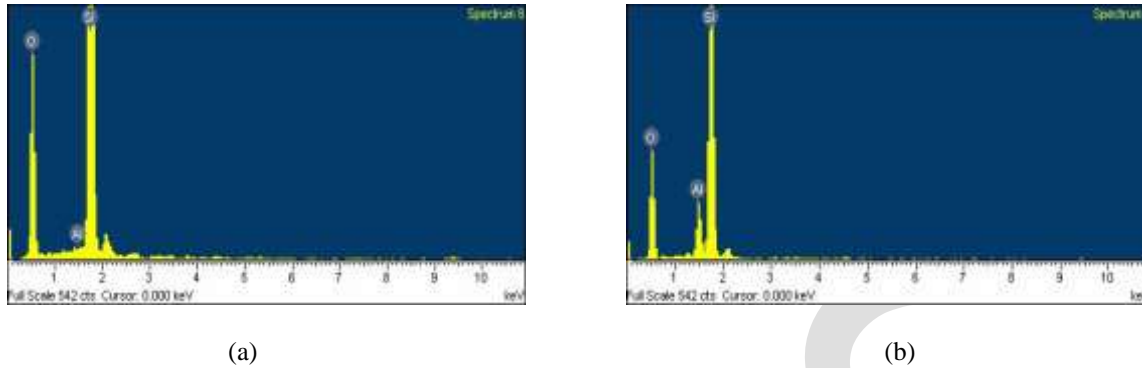
4.3 EDX Analysis

(a)   (b)

Fig. 4 Aluminium- crystalline silica composites sintered at 1150^0C for (a) 2.5 Wt. % Al, and (b) 40 Wt.% Al

From EDX analysis, there is 2.24 % and 38.9 % Al (based on Wt. %) for 2.5 % and 40 % aluminium- crystalline silica composites respectively as shown in fig. 4. Here, EDX analysis was carried on each composite sample for understanding the contamination, purity of the element, and about the reaction between ceramic matrix and second phase. EDX data show that there are no other element produced other than Si, O, and Al.
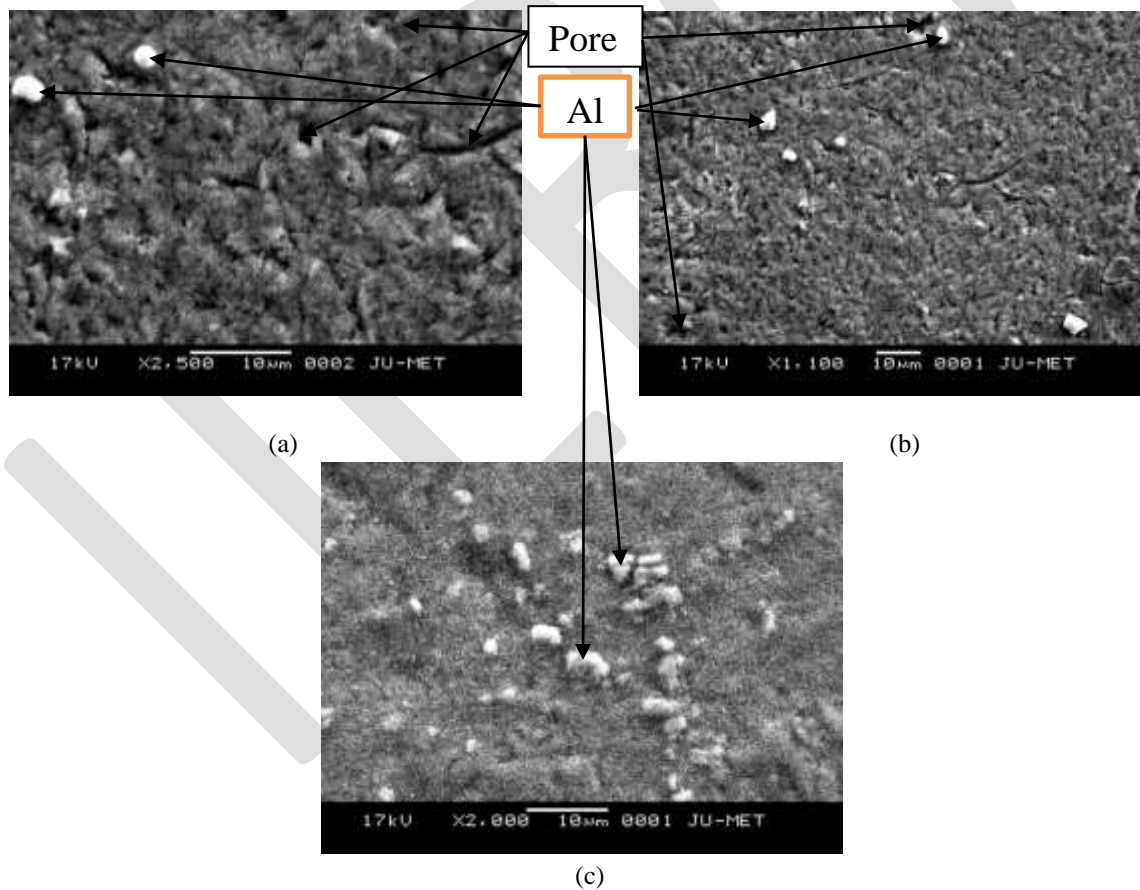
4.4 SEM Analysis

(a)   (b)

(c)

Fig. 5 Aluminium- Crystalline silica composite sintered at 1150^0C for (a) 2.5 Wt.% Al, (b) 5 Wt. % Al and (c) 40 Wt.% Al

SEM was done for each sample for observing the surface morphology like the distribution of aluminium into crystalline silica, presence of pore, and nature of crystal formation. Fig. 5 shows that aluminium is distributed throughout the entire ceramic

matrix. The tendency of pore formation decreases with increment of aluminium percentage and better adhesion occurs between ceramic matrix and second phase.

4.5 Shrinkage Measurement

(a)   (b)
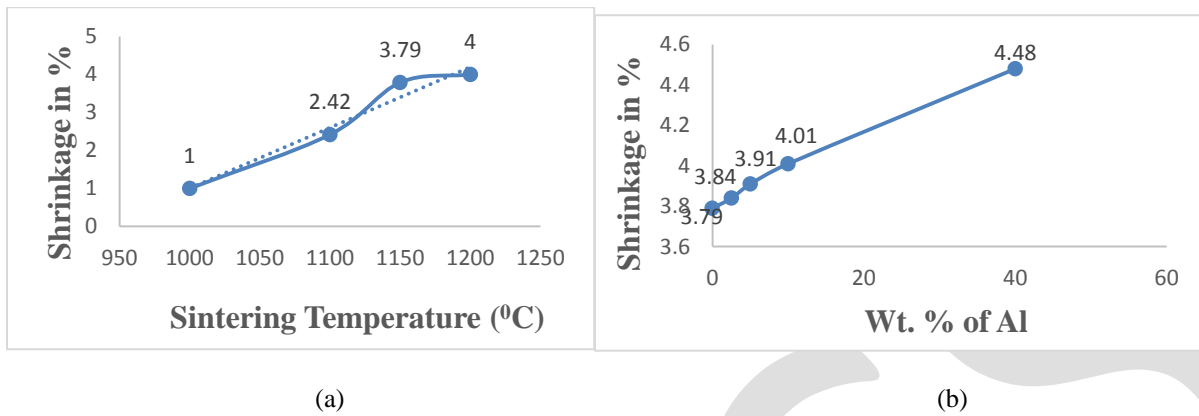
Fig. 6 Shrinkage for (a) crystalline silica sintered at different temperature, and (b)Aluminium- Crystalline silica composites

From fig. 6 (a), it is observed that maximum shrinkage value obtained at temperature 1200^0C, and which is almost same for the sample sintered at temperature 1150^0C. From fig. 6 (b), it is shown that the shrinkage value increases gradually with the increment of aluminium percentage in aluminium-crystalline composites.
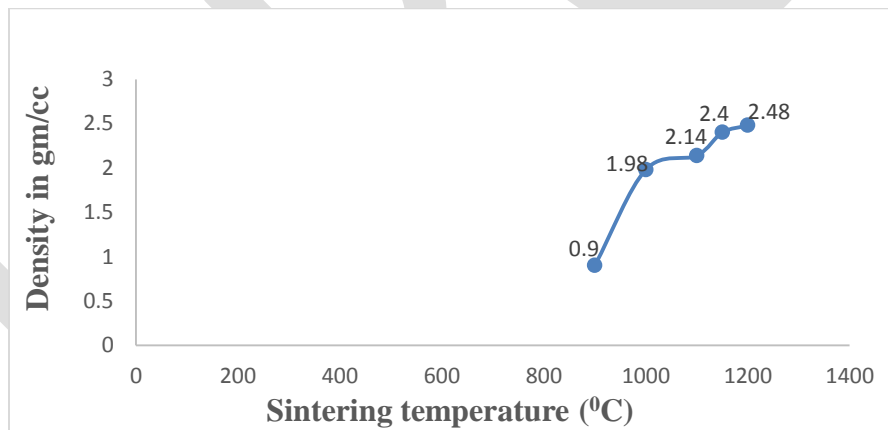
4.5 Density Measurement

Fig.7 Density of crystalline silica at different sintering temperature

Figure 7 shows that density of crystalline silica increases with the sintering temperature and crystalline silica sintered at 1200^0C shows maximum density. It is also observed that the sample sintered at 1150^0C shows nearly same density with the sample sintered at 1200^0C.
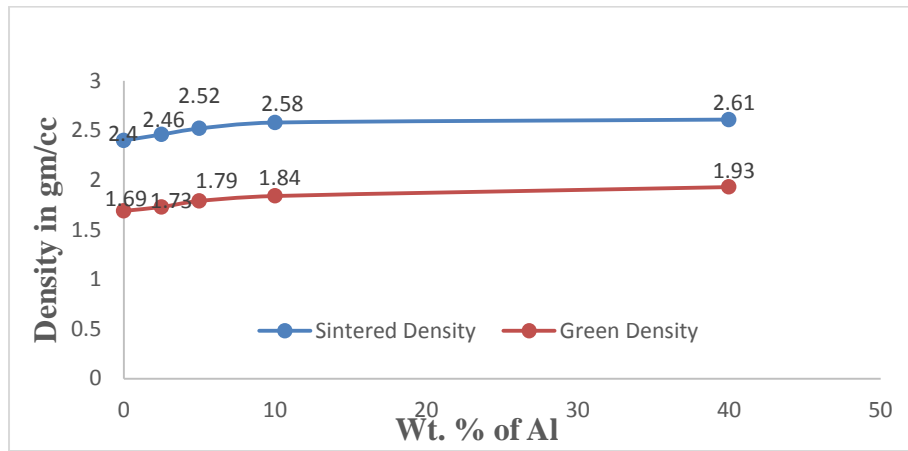
Fig. 8 Sintered &Green density of aluminium- crystalline SiO_2 composites

Figure 8 shows both green and sintered density of aluminium- crystalline silica composites. From fig.8, density is increased significantly after sintering for all samples. On the other, density, hence weight of the composites are increased slightly with increment of aluminium percentage into crystalline silica matrix.

4.6 Apparent Porosity
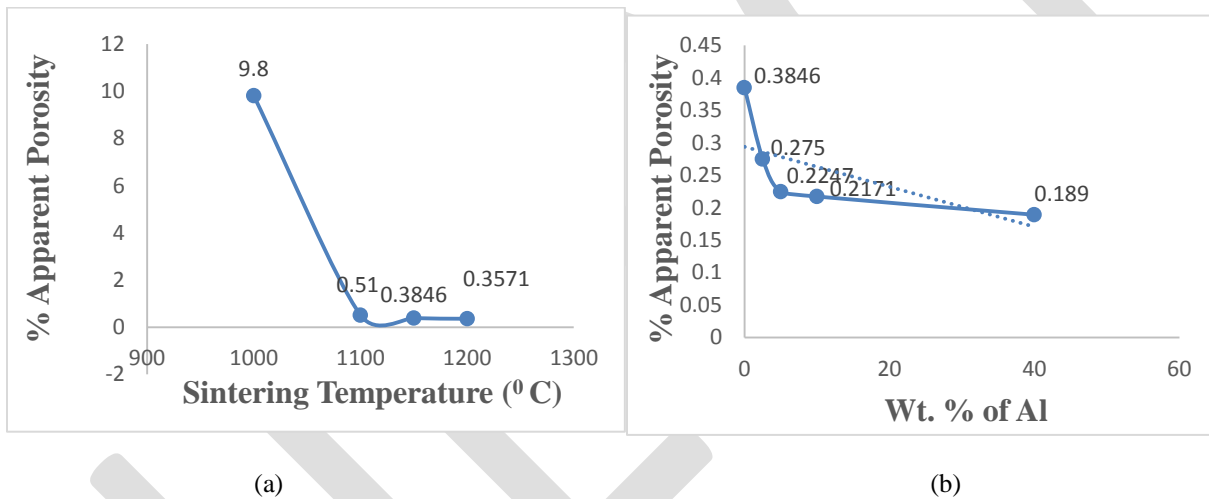

| (a) | (b) |

Fig.9 Apparent Porosity for (a) crystalline silica sintered at different temperature, (b) Aluminium-Crystalline silica composites

Now from the apparent porosity data obtained, it is seen that apparent porosity value decreases with the increasing of sintering temperature as shown in fig. 9 (a). Sintering was carried on number of steps with crystalline silica and the sample which was sintered at temperature 900^0C, gives 20.98 % porosity while the sample sintered at temperature 1200^0C gives minimum porosity. From fig 9 (a), it is also seen that the sample sintered at temperature 1200^0C gives almost same apparent porosity as that of the sample sintered at temperature 1150^0C.

From fig. 9 (b), the apparent porosity value decreases with increment of Wt. % of aluminium and hence 40 Wt. % aluminium-crystalline silica composite shows minimum porosity.
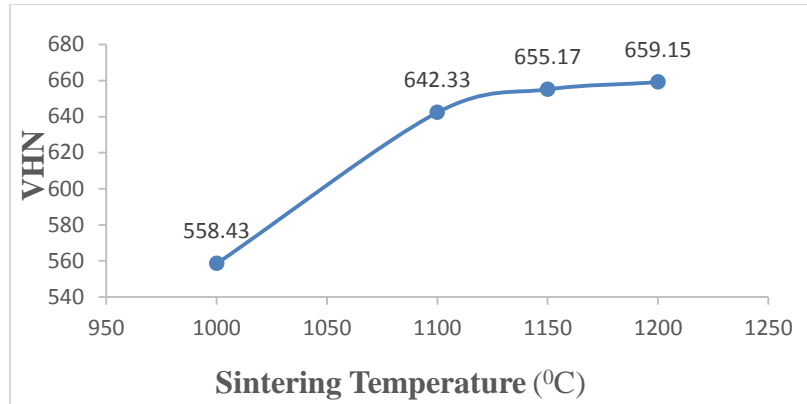
4.7 Hardness

Fig. 10 Micro-Hardness Graph for silica sintered at different temperatures

From the micro – hardness data obtained, it is seen that hardness value increases with the increasing of sintering temperature as shown in fig. 10. Sintering was done on number steps with same composition of crystalline silica, i.e. at temperatures 900^0C, 1000^0C, 1100^0C, 1150^0C and 1200^0C. But due to high porosity and brittleness of the sample which was sintered at 900^0C, micro hardness test could not be performed. From fig. 10, it can be seen that the sample which was sintered at temperature 1200^0C gave almost same hardness as that of the sample sintered at temperature 1150^0C.
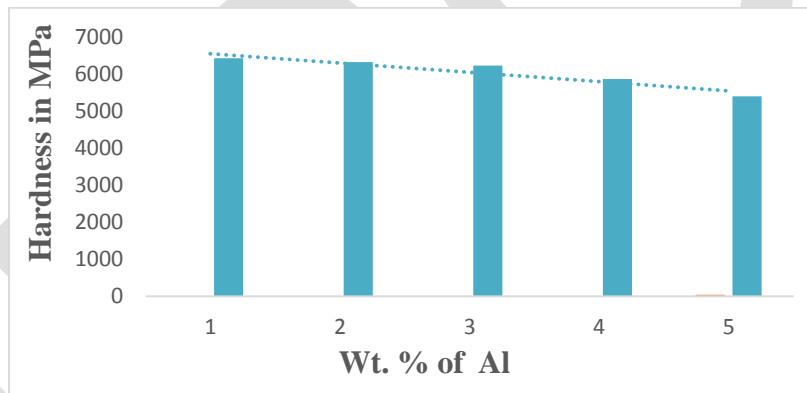

Fig. 11 Micro-Hardness Graph for Aluminium-Crystalline silica composites

Fig. 11 shows the variation of micro-hardness with the variation of the percentage of aluminium in aluminium- crystalline silica composites sintered at 1150^oC for 2 hours. As the aluminium is a ductile material, when it is mixed with the crystalline silica, reduces the micro-hardness value. Hence, hardness value decreases gradually with aluminium content.

CONCLUSION

The significant conclusions of the studies on aluminium- crystalline silica composites are as follows:

➢ From all the experimental data obtained, it is found that by analyzing different parameters like hardness, shrinkage, apparent porosity, etc. optimum sintering temperature for aluminium-crystalline silica composite is obtained which is 1150^0C.
➢ It is seen that hardness value of crystalline silica increases with the increasing of sintering temperature.
➢ For aluminium- crystalline silica composites micro – hardness value decreases with the increase of aluminium percentage. This indicates the improvement of ductility.
➢ Due to increment of shrinkage, apparent porosity decreases with the increase of aluminium percentage.

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