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Investigating the Effects of Different Sintering Temperatures on the Microstructure and Mechanical Properties of AL-AL$_2$O$_3$ Nano-Composites by Powder Metallurgy

[1] N D V Satya Prasad, [2] M N V Alekhya
[1] Assistant Professor, [2] Assistant Professor
[1][2] Department of Mechanical Engineering
[1] Eluru College of Engineering & Technology, Eluru, [2] Sir C R Reddy College of Engineering, Eluru

Abstract: -- **This research focused on the study of the influence of different sintering temperature of physical and mechanical behavior of Aluminum Metal Matrix Composites (Al MMC) reinforced with Nano alumina (Al2O3). Al MMC reinforced with rigid ceramic particulates have become increasingly important for structural applications in aerospace, automotive and other transport industries, because of their high specific strength and modulus, good wear resistance as well as ease of processing. In this work, the influence of sintering temperature was investigated on its mechanical and microstructure properties. These Al MMCs have been traditionally fabricated by powder metallurgy (PM) method. The experiments were performed on 5%, 10% weight percentage of Nano Al2O3 on different sintering temperature. In this study, the specimens of the composite were sintered with different temperature which are 5800C 6000Cand 6200C. Then, the influence of different sintering temperature on physical and mechanical behavior of the composite was studied. The specimens were investigated on their mechanical and microstructure properties.**

Keywords— Aluminum Metal Matrix Composites (Al MMC), Nano alumina (Al2O3), blending, compacting and sintering, micro hardness and mechanical properties

1. INTRODUCTION

All Aluminum and its alloys are among the interested metals for producing metal matrix composites .This causes aluminum matrix composites to find extremely attention in the last two decades [1, 2].

Recently, researchers results denote, nano-sized reinforcement particles would increase strength, ductility and toughness [2, 3, 4, 5]. Segregation and non-uniform distribution of reinforcements are of cast method defects [2,6]. One of the main challenges inherent to this technique is to obtain a homogenous distribution of the reinforcement in the metal matrix[7,8]. Powder metallurgy can produce metal matrix composites in the whole range of matrix reinforcement compositions without the segregation phenomena typical of the casting process [6, 7, 8].

The first requirement for a composite material to show its superior performance is the homogenous distribution of the reinforcing phase [9]. The agglomeration of the reinforcement particles deteriorates the mechanical properties of the composite [4,7-10]. Differences in particle size, densities, geometries, flowing or the development of an electric charge all contribute to particle agglomeration [6,9]. Recently, high – energy ball milling has been used to improve particle distribution throughout the matrix [1,11-13].

Sintering is the key stage of a powder metal part which has been used to weld powder particles are together and a strong metal part is produced.

According to literature survey there are many articles which have been focused on production of composite materials and the past research efforts related to processing of characterization of aluminum MMC. The main process included milling the raw material (powder) which is Aluminum powder and Alumina powder (Al2O3), compacting milled powder and sintering process. A review of other relevant research studies is also provided. However, little information can be found on integrated durability evaluation methods. The main goal of this study is to investigate the process of al-al$_2$o$_3$ nano-composites by powder metallurgy and evaluation of the effect of sintering temperature on the microstructure and mechanical properties.

II. EXPERIMENTAL METHODS

A Materials

The Aluminum powder purchased are having the particle size of 20-40 μm and the Alumina particle size was about 30-50 μm. Dry ball milling is carried out for about 30 hours for producing nano particles of alumina using Fritsch

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High energy planetary ball mill. The particle size of nano powder produced is about 100 nm. The powder in 5% and 10 % of alumina are weighed and they are mixed using ball milling for about an hour.

The porosity of the sintered compacts was determined by the equation

$$\textbf{\% porosity} = \frac{(\rho^{th} - \rho)}{\rho^{th}} \times 100 \longrightarrow \quad (1)$$

Where ρ is the measured sintered density

ρ^{th} is the theoretical density.

B. Milling of materials and sample preparation

Materials whether hard and brittle or soft and ductile is of prime interest and of economic importance to the P/M industry. A ball mill, a type of grinder, is a cylindrical device used in grinding (or mixing) materials like ores, chemicals, ceramic raw materials and paints. Ball mills rotate around a horizontal axis, partially filled with the material to be ground plus the grinding medium.

The ball to powder ratio was about 10:1 and they were ball milled at a speed of 60 Rpm for about one hour the balls used were tungsten carbide of 5mm diameter.

The milling speed can have an important influence but this varies with the type of mill. For the higher speeds the temperature of the milling chamber may reach a high value. It will be in the range of 50-400 RPM.

C. Powder compaction

Die compaction represents the most widely used method and is considered as the “conventional” technique. It allows pressure to be applied only in the axial (vertical) direction to one or both ends of the powder mass. Involves relatively slow speeds, and no liquid is used to suspend the powder, although various additives may be added for specific purposes. Both low density and high density structural parts over a very broad size range are made by this technique.

A source of energy or pressure, usually a mechanical or hydraulic mechanism. A Single action compaction of hydraulic equipment is used

D. Sintering

Sintering is “the thermal treatment of a powder or compact at a temperature below the melting point of the main constituent, for the purpose of increasing its strength by bonding together of the particles.” In this stage, the part acquires the strength needed to fulfill the intended role as an engineering component.

By using a push-puller mechanism, compacts are moved to high temperature sintering zone and held until an optimum sintering temperature in the range of 550-620°C is reached. Thereafter, compacts are left undisturbed at constant sintering temperature under inert atmosphere to ensure the movement of atoms within solid crystalline material, for 45 minutes. Finally, compacts are pushed to cooling zone and are allowed to cool to room temperature by stopping any further addition of heat to furnace.

E. Characterization

The amount of porosity in the sinter mass, as indicated by increasing green density or compaction pressure values, increases the degree of bonding. Based on this consideration and also a number of trial tests, a compaction pressure that would result in 90% theoretical density in green compact, has been selected.

F. Archimedes’s Principle

Experimentally this appears in the fact that the submerged object apparently weighs less by an amount equal to the weight of the liquid displaced. The buoyant force can be expressed as

$$F_b = W_{air} - W_{liq} = dgv \longrightarrow \quad (2)$$

G. Hardness measurement

Vickers hardness measurement was performed with 10kg load i.e; 440HV10.

Micro hardness can be calculated by using the formula i.e.

$$HV = 1.854F/d^2 \longrightarrow \quad (3)$$

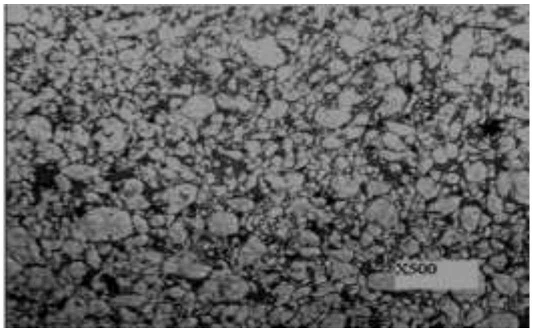
H. Compression test

Universal tester materials testing machine TUE-600(C) or materials test frame, is used to test the tensile stress and compressive strength of materials.

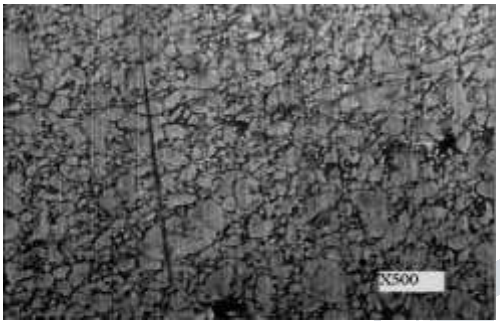
III. RESULTS AND DISCUSSION

A. Milling time effect on the microstructure

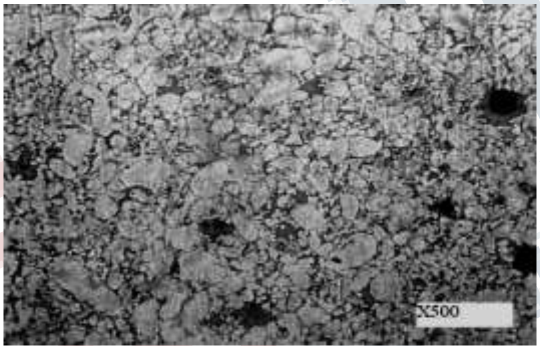
Figures 1(a) to 1(c) show the SEM microstructures taken from pure aluminum sintered at 580°, 600° and 620° at different milling timings. Figures 1(d) to 1(f) shows Al-5% Nano Al_2O_3 composite sintered at 580°C, 600°C and 620°C and Figures 1(g) to 1(i) shows Al-10% Nano Al_2O_3 composite sintered at 580°C , 600°C and 620°C which are mixed using ball milling for about 1 hour. Figure 1(j) shows SEM image of Nano Al_2O_3 .



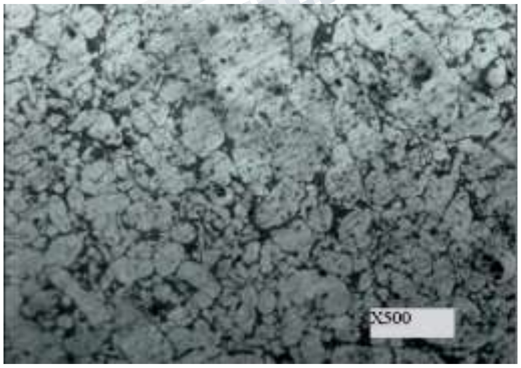
1(a). Pure aluminium sintered at 580^{0}C



1(b). Pure aluminium sintered at 600^{0}C

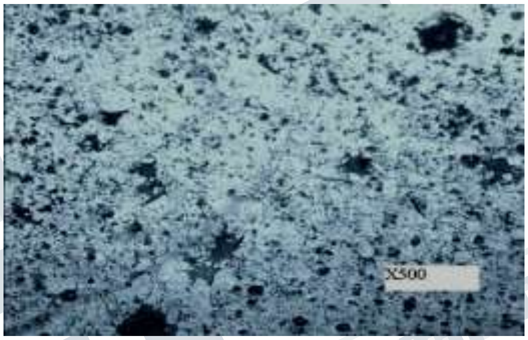


1(c). Pure aluminium sintered at 620^{0}C

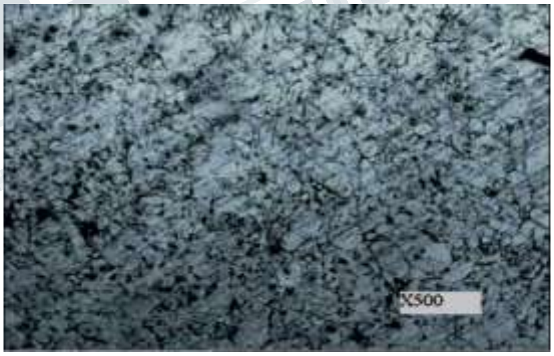


1(d). Al-5%NANO AL$_2$O$_3$ COMPOSITE SINTERED AT 580^{0}C

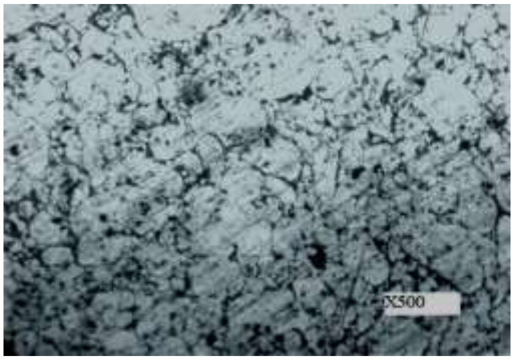
1(e). Al-5%NANO AL$_2$O$_3$ COMPOSITE SINTERED AT 600^{0}C



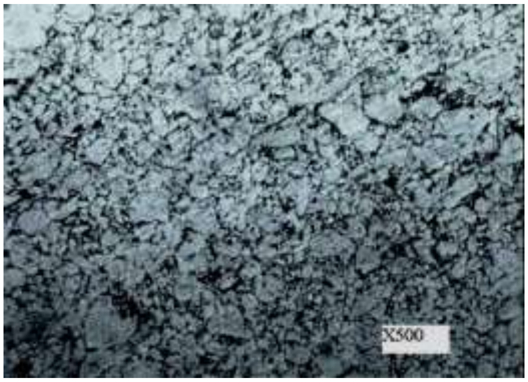
1(f). Al-5%NANO AL$_2$O$_3$ COMPOSITE SINTERED AT 620^{0}C



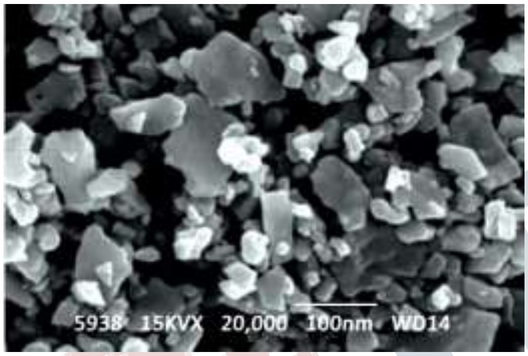
1(g). Al-10%NANO AL$_2$O$_3$ COMPOSITE SINTERED AT 580^{0}C



1(h). Al-10%NANO AL$_2$O$_3$ COMPOSITE SINTERED AT 600^{0}C



1(i). Al-10%NANO AL_2O_3 COMPOSITE SINTERED AT 620^0C



1(j). SEM image of Nano Al_2O_3

Cylindrical compacts were obtained by single action die compaction at ambient temperature using a 200 ton Hydraulic Press with the load range of about 5 tons for about 5 min. Stearic acid was used as the die wall lubricant. The aluminum -matrix Alumina particulate composites by powder compact dimensions were 10 mm diameter and 12 mm length. The compacts made were sealed in Electrical tube furnace under argon atmosphere and sintered at 580-620^0C in a tubular furnace for a period of 1 hour.. Metallographic examination of sintered compacts was carried out using optical microscopy (fig 1(d) to 1(i)).

Fig 2. Die used for compaction

The green density was evaluated as a function of alumina weight percent. Density of the components was determined using Archimedes principle, porosity, and hardness of the sintered compacts of different percentages of alumina were also determined as a function of different sintering temperatures using Vickers micro hardness testing equipment. Green and sintered densities were determined by Archimedes principle. For evaluation of compressive strength of the nano composites, compression testing is done on universal testing machine TUE-600(C).

In Planetary Ball Mills, the combination of the material to be ground takes place primarily through the high-energy impact of grinding balls. Conventionally, the term fine milling is used for size range below 100 µm and the ultrafine (or very fine) milling for particles size less than 10 µm. The material used for the milling media (milling chamber, vial, balls) is important due to impact of the milling balls on the inner walls of the milling chamber.

Material	**Main composition**	**Density (g cm^{-3})**	**Abrasion resistance**
Agate	SiO2	2.65	Good
Corundum	Al_2O_3	>3.8	Fairly good
Zirconium oxide	ZrO_2	5.7	Very good
Stainless steel	Fe, Cr, Ni	7.8	Fairly good
Tempered steel	Fe, Cr	7.9	Good
Tungsten carbide	WC, Co	14.7-14.9	Very good

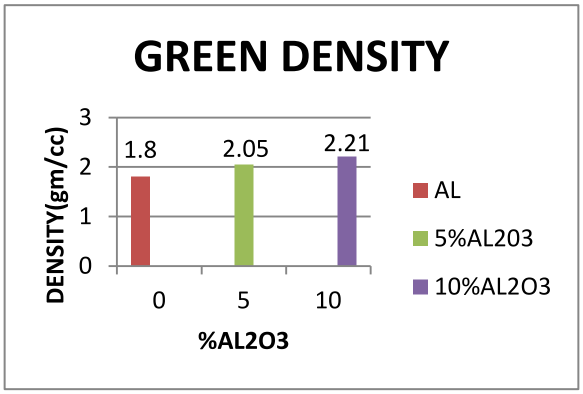


Fig 3.GREEN DENSITY

Fig3. Shows that the green density increases with the increase in the percentage of Nano Al_2O_3 content in the composite. Green density for 10% addition of AL_2O_3 is higher of about 2.21gm/cc.

Sintering furnace set-up explains first zone of the furnace is meant to heat the green compacts rather slowly to a moderate temperature in the order of 150^{0}C. The volatilization and elimination of the admixed lubricant usually present is one of the prime functions of this burn-off zone. A slow rate of heating (5^{0}C/min) is necessary to avoid excessive pressures within the compact a possible expansion, spalling and fracture. Slow heating in this zone is also needed to avoid too rapid expansion of entrapped gases in the compacts. In the high temperature zone, the actual sintering of the material takes place. In this zone, the samples are properly heated at a heating rate of 10^{0}C/min until the desired temperature is reached. Because of the necessity to have a reducing atmosphere present during sintering, a gas tight tube furnace is utilized in this work the cooling zone consists of two sections:

1) A short insulated section that permits the parts to cool down from the high sintering temperature to 150^{0}C at a slow rate (5^{0}C/min) so as to avoid thermal shock in the compacts as well as to the furnace, and

2) A relatively long water jacketed section providing cooling to a temperature low enough to prevent oxidation of the material upon exposure to the air.

Fig 4 . SINTERED DENSITY

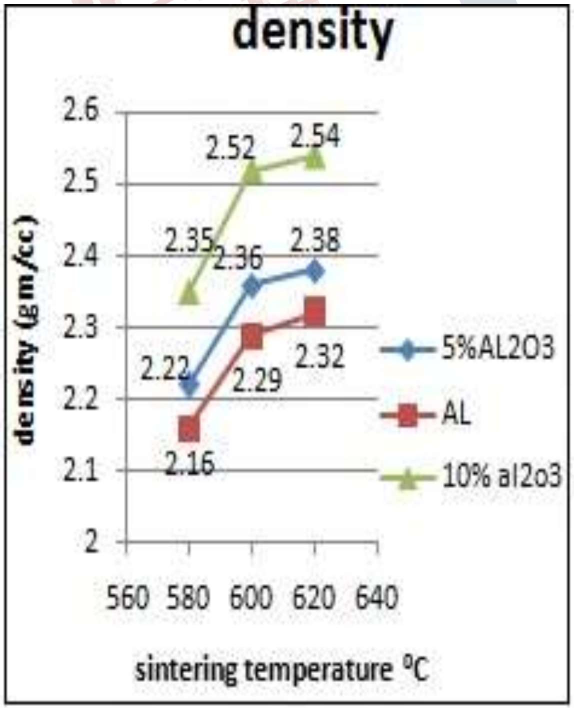


Fig4. Shows Sintered density increases with the increase in the sintering temperature and even with the increase in the percentage of Al_2O_3. The composite with 10%Al_20_3 sintered at

620 ^{0}C yielded a highest density.

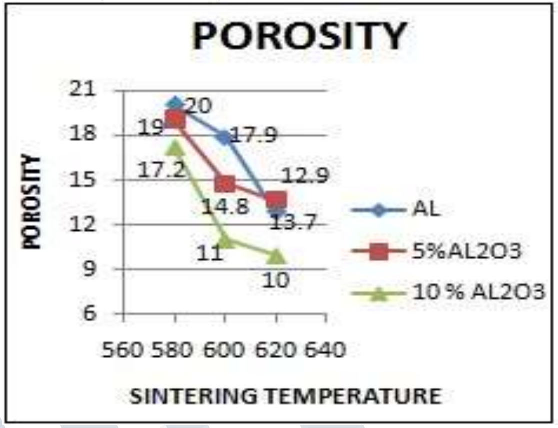


Fig 5. SINTERED POROSITY

Fig 5. Shows Porosity of composites for different sintered temperature is calculated using the the theoretical density and sintered density. There is a fall in the porosity as the sintering temperature of the composite increase. The composite with 10% AL2O3 sintered at 620 0C has a lesser porosity.

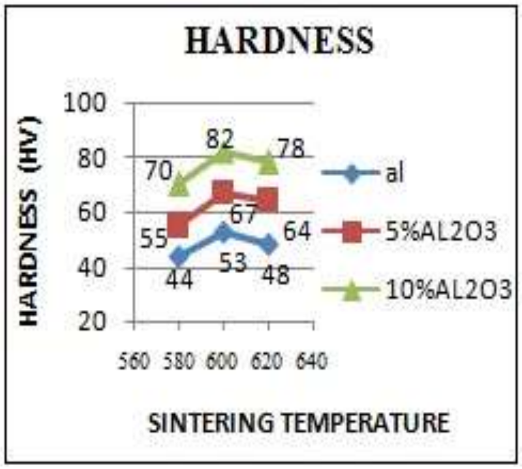


Fig 6. SINTERED HARDNESS

Fig 6. Shows initially there is an increase in the hardness as the sintering temperature is increased and there is a slight fall in the hardness value as the sintering temperature is further increased. The composite with 10% AL2O3 sintered at 6000C has the highest hardness of about 82 HV 10.

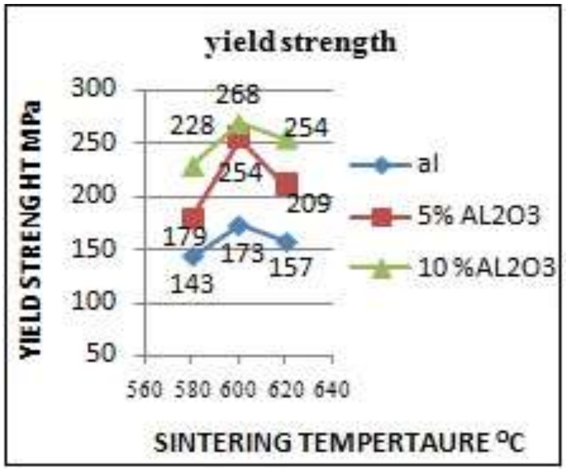


Fig7. YIELD STRENGTH

Fig7. Shows Yield strength of the composites is obtained from hardness values calculated by Vickers micro hardness testing. Initially there is an increase in the yield strength as the sintering temperature is increased and there is a slight fall in the yield strength value as the sintering temperature is further increased. Composite with 10% **AL_2O_3 SINTERED** at 600^0C yielded a better result.

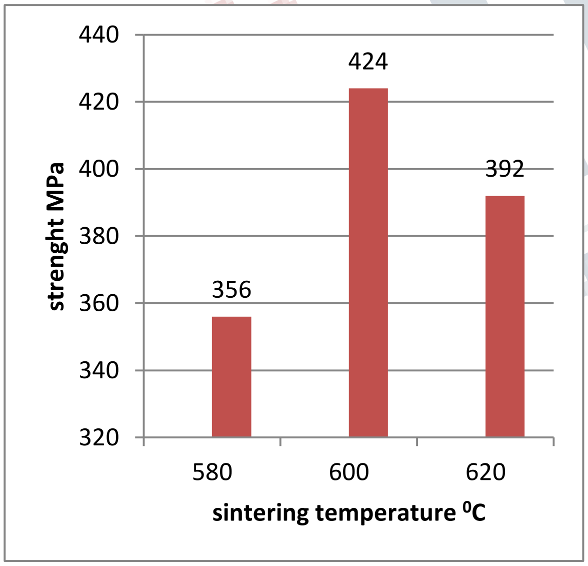


Fig8. COMPRESSIVE STRENGTH

Fig8. Shows the variation of compressive strength for different sintering temperature. The composite with 10% AL_2O_3 is being tested and the composite sintered at 600^0c has the highest compressive strength value of about 424Mpa

Figures 8(a), 8(b) and 8(c) shows compressive strength results for 10% Al_20_3 sintered at 580°C, 600°C and 620°C

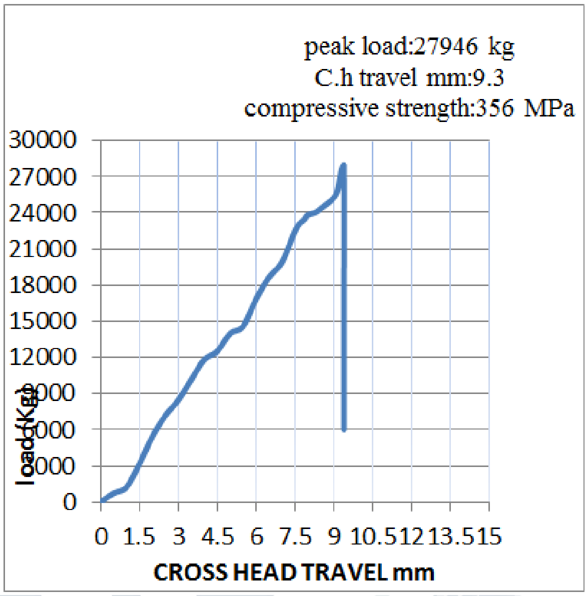


Fig8(a) compressive strength results for10% Al_20_3 sintered at580^0C

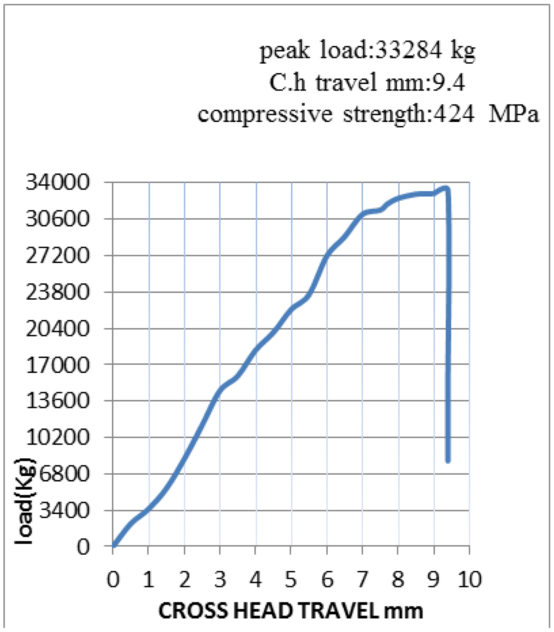


Fig 8(b) compressive strength results for10% Al_20_3 Sintered at 600^0C

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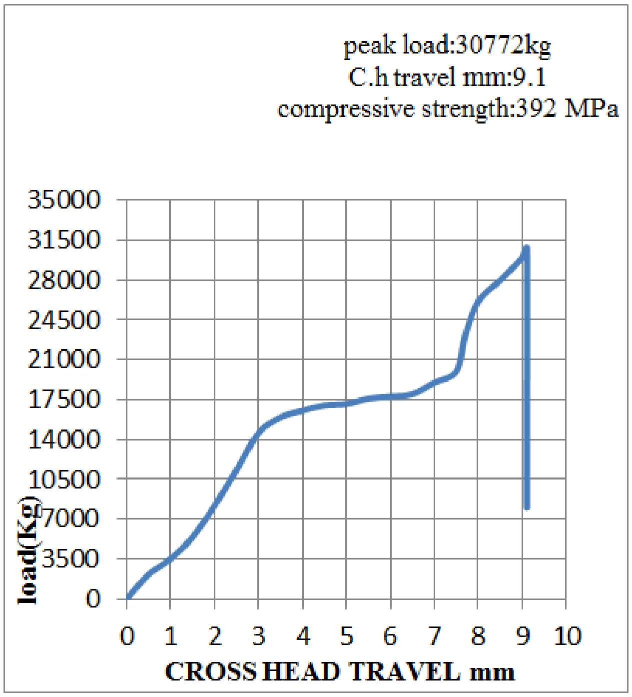

Fig8(c) compressive strength results for10% Al_20_3 sintered at 620^{0}C

The Vickers test is often easier to use than other hardness tests since the required calculations are independent of the size of the indenter and the indenter can be used for al materials irrespective of the material. the basic principle as with all common measures of hardness is to observe the questions material ability to resist plastic deformation from a standard source. The Vickers test can be used for all metals and has one of the widest scales among hardness test. The unit of hardness given by the test is known as the Vicker Pyramidal Number (HV) or diamond pyramid hardness(DPH). The hardness number can be converted into units of Pascal's but should not be focused with the pressure which also has units of Pascal. The hardness number is determined by the load over the surface area of the indentation and not the area normal to the force and is therefore not the pressure. The Hardness number is not really a true property of the material and is empirical value that should be seen in conjunction with the experimental method and the hardness scale used .when doing the hardness test the distance between indentation must be more than 2.5 indentations apart to avoid interaction between the work hardening region.

IV CONCLUSION

From this investigation, following conclusions may be drawn.

1. The composites can be prepared by powder metallurgy technique, involving pressing with pure aluminum and nano Al_2O_3 followed by sintering.
2. Green density is increased with the increase in the percentage of nano Al_2O_3 since the density of Al_2O_3 is greater than the aluminum powder.
3. Sintered density of the composite produced is increased with the increase in the sintering temperature this is due to the flow of materials in to voids causing in the reduction in volume of the composite, Because of which there is an increase in density and reduction of porosity takes place. Initially there is an increase in Hardness value, yield strength with the sintering temperature and by still further increase in temperature resulted in decrease in the hardness and yield strength. at last we can conclude that the composite with 10% nana Al_2O_3 sintered at 600^{0}C yielded a better results.

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