Intrinsic Parameters for Fabrication of Ceramic Grains & Pads for Pulveriser Grinding Media

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***Abstract*—A novel method of fabricating ceramic metal matrix composite (CMMC) insert is established by finalizing the intrinsic process parameters and composition of ceramic grains followed by the shape and properties of ceramic pads to become integral part of the bowl mill grinding roll of Pulveriser to achieve higher wear resistance properties. Initially, experiments were done to fabricate spherical ceramic grains of Al_2O_3 and found that samples with 60 wt% Al_2O_3 (Alumina-Sodium Alginate Slurry) sintered at 1650^0C for 5 hours dripped into $CaCl_2$ solution through 1mm dia nozzle resulted in high green density (61 g/cc) and high hardness (1615HV) spherical grains with size range 1.5-3.0 mm dia. Accordingly, ceramic pads, using ceramic grains in the size range 1.5-3.0 mm dia as a mixture of ZrO_2 (30 wt%) toughened and TiB_2 (6 wt%) hardened Al_2O_3 (60 wt%) with minor addition of Co (4%), were made adopting the same method. Then these ceramic pads were positioned at designated location in the CO_2 mould in to which high chrome metal is poured to make ceramic metal matrix composite inserts. Grinding rolls, fabricated using these inserts embedded in the outer (working) layer, could give enhanced average running life of bowl mill roll by 2000 hrs.**

***Key Words*—Ceramic Beads, Ceramic Metal Matrix Composite, Gel Casting, Grinding Roll, High Chrome.**

I. INTRODUCTION

Thermal power plants running on steam require boilers to generate steam which in turn use fossil fuel (coal) to generate heat. Coal has to be fed into the boiler in very fine form without any other impurities. Hence, the efficiency of thermal power plant is mainly dependent on the running life of coal grinding media of Pulveriser to enable uninterrupted coal supply to boilers at least to match to the time duration between two consecutive scheduled mill shut down time for maintenance purpose.

There has been continuous effort by researchers which led to the evolution of enhanced wear life of grinding media, especially for bowl mill applications (grinding roll and bull ring segments), starting with Mn-Steel to Ni Hard and then to high chrome metal. Presently, ceramic metal matrix composite (CMMC) is considered to be the best amongst all types.

The ever decreasing trend of coal quality in Indian conditions, demands the necessity of developing high wear resistant grinding roll and bull ring segments for bowl mill applications having ceramic metal matrix composite inserts made of ceramic grains with high hardness. This grinding roll is made of two layers, the outer layer is high wear resistant ceramic metal matrix composite material whereas the inner layer is made of spheroidized graphite (SG) Iron. Running life of grinding roll is solely dependent on the wear resistance of outer layer (working surface) which in turn is dependent on ceramic pads embedded in high chrome. It has been observed that the wear life of any metal is directly proportional to the hardness of the material. But at the same time, in the process of increasing the hardness of metal, the impact strength of the material gets reduced drastically which has an adverse effect on the wear life. Hence, it is required to attain higher hardness without compromising on the toughness/ impact values. This led to the innovation of ceramic metal matrix composites (CMMC) which strikes a balance between abrasion resistance and impact strength.

Accordingly, lot of research has been done to improve upon the existing fabrication techniques or to innovate newer methods of fabricating ceramic beads/ grains of desired shape and strength. Some of the research works related to the present study are described below:

Christian J et al [1], have produced Alumina beads using a vibrational approach in order to explore the spread of stress waves in granular material. The procedure creates beads with the desired shape (such as oblate, prolate, and tri-axial ellipsoid), microstructure (level of porosity), and size (5mm to 3cm in diameter) using an environment friendly method of processing beads that involves creating a slurry with a small amount of PVA (Poly Vinyl Alcohol) binder and dispersant. The dried pre-formed material is a shear-reversible soft-solid that flows with little strain recovery while under stress. This rheological behavior is enough to process the alumina paste into rounded shapes with a small shaker table and to keep the alumina beads in their proper shape during sintering.

Thomas J. Vlach et al [2], devised a process for producing ceramic beads that are substantially spherical which involves transferring an aqueous ceramic slurry through a nozzle tip submerged in an inert fluid layer that is immiscible with water. The nozzle tip is separated from a rotating disc, which is also submerged in the immiscible fluid layer, by a predefined distance. Droplets of the aqueous ceramic slurry are forced out of the nozzle tip and into the layer of immiscible fluid by the shear force produced by the revolving disc at the nozzle tip. The droplets take on a substantially spherical form and a substantially mono-modal size distribution, once they are dislodged. The droplets are allowed to pass through the layer of immiscible fluid into an aqueous gelling solution, where they are transformed into hard beads.

The miscibility of ceramic beads in high chrome matrix has been another big challenge for the researchers to fabricate an integral unit of high strength ceramic beads/ grains embedded in high chrome metal. The related work done in this regard is described below:

Takalani Madzivhandila [3], examined the infiltration of zirconia (ZrO_2) and alumina (Al_2O_3) in high chrome cast iron. Investigations were done into how molten iron behaved when it came to wetting on ZrO_2 and Al_2O_3 surfaces. Although with varying potencies, titanium, chromium, copper, and nickel were found to increase wetting properties. Zirconia and cast iron bond more effectively than alumina.

These literature inputs have enabled to assess the existing practices and explore for probable scope of improvement or to establish a novel method of fabricating CMMC high chrome insert for grinding roll of bowl mill application of Pulveriser.

Though CMMC has fulfilled the requirement for majority of bowl mills, but matching the roll running life to duration between two scheduled mill shut down time has still remained a challenge in case of worst Indian coal.

This triggered the objective of present work where in the main focus remained in developing a novel method of fabricating ceramic grains with high hardness and spherical shape followed by fabrication of ceramic pads, with desired compactness and shape to enable perfect miscibility and mechanical bonding with high chrome metal so as to make the insert with high wear resistance which will be used as an integral part of grinding roll.

The ceramic metal matrix layer consists of ceramic pads that are embedded/interwoven with high chrome metal by mechanical bonding. Moreover, for better running life and strength, it demands the ceramic particles to be in spherical shape. It is aimed to make the grains spherical in shape, as any other shape is likely to have sharp corners and will act as stress concentrators when used as reinforced particles in the metal matrix. Also these sharp corners/ edges become the potential crack generation and propagation centers leading to premature failure of the casting.

Keeping the above factors in view, initially experiments were carried out to finalize the density, sintering cycle and methodology to achieve the spherically shaped ceramic grains/ particles with desired hardness values so that subsequently it can be used in combination with other ceramic particles to act as hard reinforced material in the high chrome metal matrix [4].Then ceramic pads were developed with desired size, shape and strength to be positioned in the designated location in the CO_2 mould to pour high chrome metal for fabrication of CMMC insert.

In the present study, the method of making the spherical ceramic grains of required density and hardness, followed by fabrication of ceramic metal matrix composite insert with high chrome metal could be established successfully.

II. EXPERIMENTAL DETAILS

Step-I (Fabrication of ceramic grains/ particles of spherical shape with required density and hardness of Al_2O_3*)*

Prior to processing, the input alumina powder is subjected to material characterization through XRD and EDS to ascertain that the selected powder does not contain any other elements except Al_2O_3 and also contains hexagonal crystal structure to confirm α phase. The diffraction spectrum obtained from the XRD technique has been analyzed using the "PANalytical X'pert Highscore plus" software (it is a database containing the peak XRD data for all the elements in the periodic table and their compounds). The composition of the phases was measured using Energy Dispersive X-Ray Spectroscopy (EDS) (Make: Oxford).

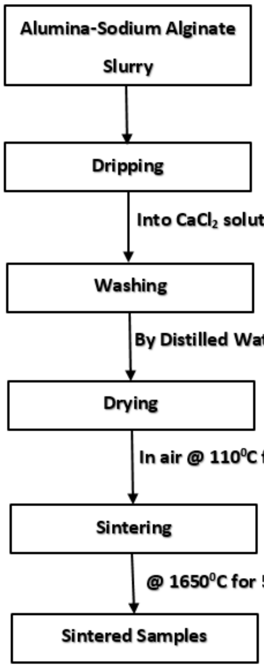


Fig. 1 Diagram showing the process flow chart for fabrication of Al_2O_3 spherical grains

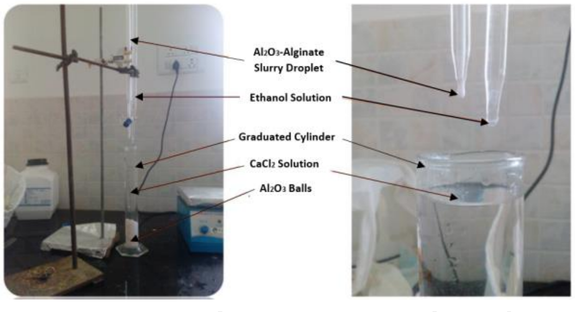


Fig. 2 Experimental Setup to prepare Al_2O_3 spheres

Then to fabricate spherical shaped Al_2O_3 particles with optimum density and hardness, following process is followed [5, 9]:

Sodium Alginate, a natural biopolymer extracted from algae, is used as a gelling additive to Alumina powder (α phase). It forms a gel and the sodium ion is substituted by a multivalent cation such as Ca (II) to build a cross sectional network between the polymer chains. This characteristic property is used for fabricating ceramic (Al_2O_3) grains of spherical shape, the process flow chart is given in Figure 1 above.

Thoroughly mixed Alumina-Sodium Alginate slurry was dropped into the calcium chloride solution through the nozzle dropwise to form spheres. As the droplets fell through the reacting solution contained in the reaction column, semi rigid gelled Alumina spheres were formed and these are collected at the bottom of the reaction column.

Stabilization of alumina/alginate slurry was achieved by the addition of appropriate dispersant, i.e. ammonium polyacrylate [6], which provided low viscosity and ensured that the alumina particles would not settle within a short period of time. Ammonium polyacrylate, dispersant with ethanol as the absorbent [7, 8], was employed for the stabilization of the slurries with 40 and 60 wt % solid contents at a fixed concentration of 0.5 wt % of sodium alginate

The gelation reaction is expected to be [9]:

$$CaCl_2+2(C_6H_7O_6Na) \longrightarrow 2NaCl + (C_6H_7O_6\text{-}Ca\text{-}C_6H_7O_6) \qquad (1)$$

Collected Alumina spheres were cleaned by using distilled water and dried at 1100C for 2hrs. Thermal treatment was applied to the dried spheres. Finally, dense Alumina spheres were obtained.

The experimental setup to prepare Alumina spheres is shown in Figure 2. Here burette is used to drip the slurry into the reacting solution and graduated cylinder is used to collect the semi rigid Alumina spheres.

In order to make the ceramic pads for the grinding roll (Bowl Mill Type XRP1043), a thoroughly blended mixture of 60 wt% Al_2O_3, 30 wt% ZrO_2, 6 wt%TiB_2 and 4 wt% Co was considered for the above treatment and obtained the spherical ceramic grains of size range 2.5 to 3 mm. The above composition of ceramic mixture is finalized by the authors in their previous work [10]. These grains were further processed to get the ceramic pads of desired geometry and shape as given below.

Step-II (Fabrication of ceramic metal matrix composite high chrome insert)

A metallic die (as shown in Figure 3) is fabricated using CNC machine along with a metallic encapsulation such that the cavity formed, by closing the metal encapsulation and placing the die at the center, is exactly of the desired ceramic pad dimension and shape as per the drawing/ pattern finalized.

Fig. 3 Metallic Die and Metallic Encapsulation for fabrication of Ceramic Pad

Once the die set up is assembled and made ready, then the cavity is filled with liquid silicon rubber [11] and allowed to cure. This cured rubber mould can subsequently be used for making ceramic pads for regular mass production as shown in the Figure 4.

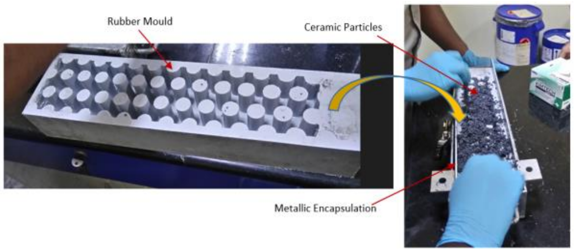


Fig. 4 Silicon Rubber Mould for Fabricating Ceramic Pads

In order to have dimensional accuracy of the ceramic metal matrix inserts so that they can perfectly be fitted in the centrifugal die to cover a depth up to 30mm to 50mm from the working surface inwards, right across from the bottom part of the roll to the top, the patterns were made by creating 3D model of the same followed by 3D printing (Creality 3D CR-10S Printer Cura Repeater) by using hard plastic material with programming for high density packing.

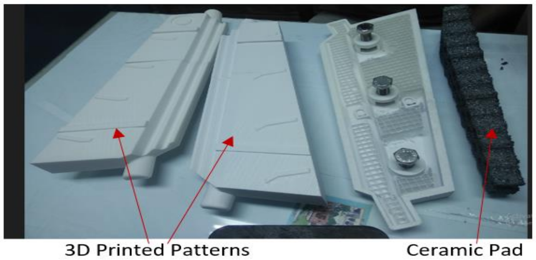


Fig. 5 Ceramic Pad and 3D Printed Plastic Pattern for Mould Making

The typical two-part 3D printed pattern made for grinding roll insert is shown in Figure 5 which are fitted with the bolts on the follow board for moulding, the provision for bolting has been created while printing itself.

Moulds were made by CO_2 sand moulding process and two coats of spirit based Zircon paint was given on the mould surface. Once the moulds were ready for assembly with ceramic pads, they were heated by torching the burners to a minimum temperature of 100^0C and at the same time the ceramic pads are preheated to a temperature of 120^0C in a drying oven for 1 hour and then assembled in the mould for pouring of liquid high chrome metal at a temperature of 1500^0C ($\pm 15^0$C), within 15 minutes of mould closing. After pouring, the casting is kept in the mould for minimum 48 hours and then removed for knockout. The typical arrangement for mould assembly is depicted in Figure 6:

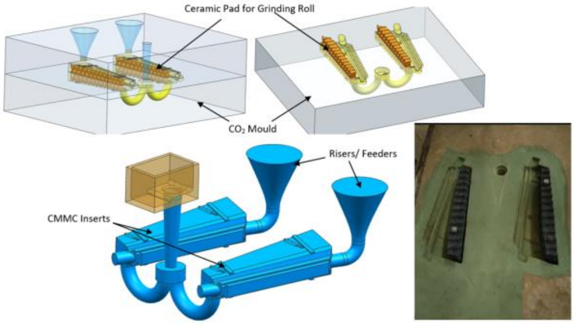


Fig. 6 Schematic & Photograph of CO_2 Moulding with Positioning of Ceramic Pad in the Mould Cavity

The chemical composition of high chrome and SG of grinding roll are maintained as given in Table 1 and Table 2 respectively, which were finalized by the authors in their earlier works [12, 13 & 14]:

Table1: Chemical Composition of High Chrome Metal of the Insert

Description	C	Si	Mn	S	P	Cr	W	Mo	Fe
Actual	2.81	0.78	0.67	0.05	0.05	18.02	1.25	1.5	Bal.
Target	2.6 - 3.0	0.5 - 1	0.5 - 2	≤ 0.1	≤ 0.1	15 - 22	1 - 2	1 - 2	Bal.

Table2: Chemical Composition of Spheroidized Graphite Iron

Description	C	Si	Mn	S	P	Residual Mg	Fe
Actual	3.65	2.22	0.67	0.008	0.05	0.05	Bal.
Target	3.5-3.8	2.2-2.4	0.1-0.7	≤ 0.01	≤ 0.05	0.05 -0.07	Bal.

Chemical composition was analyzed through Spark Emission Spectroscopy (SES) method using SPECTROMAXx machine.

The ceramic metal matrix high chrome inserts after knock out are subjected to stress relieving at 296^0C for 8 hours with rate of heating @ 50^0C (max) followed by air cooling after soaking. Then the inserts are ground and polished to carry out visual inspection for defects (if any) and then subjected to further processing to fabricate grinding roll for bowl mill

III. RESULTS AND DISCUSSION

Step-I (Fabrication of ceramic grains/ particles of spherical shape with required density and hardness of Al_2O_3)

Material Characterization:

The diffraction spectrum obtained from the XRD technique has been analyzed using the “PANalytical X’pert Highscore plus” software. XRD peaks of Al_2O_3 are given in Figure 7. Almost all peaks are found to be matching with reference code 98-004-0004 ICSD data base and identified as the peaks corresponding to Al_2O_3 with hexagonal crystal system and it is observed that starting powder is alpha Alumina with no other materials or other phases were present in the starting powders within XRD detection limit.

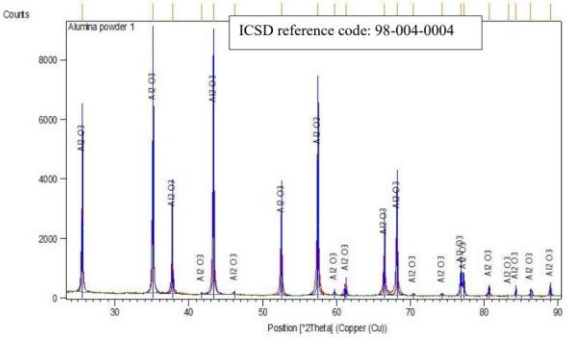
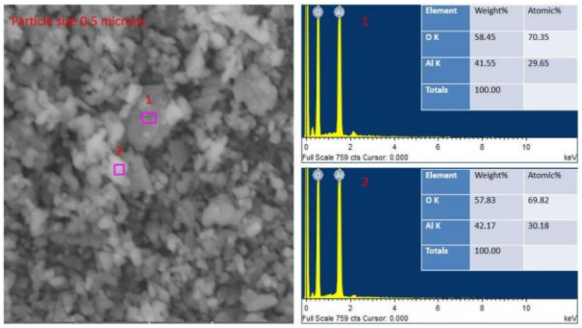


Fig. 7 XRD Plot of Starting Powder

Elemental Analysis:

EDS analysis has been done on the initial starting powder (Figure 8). From the obtained data, it is clear that only Aluminium and Oxygen elements were present in the starting powder with no other elements within the detection limit of EDS spectra.



1

Element	Weight%	Atomic%
O K	58.45	70.35
Al K	41.55	29.65
Totals	100.00	

2

Element	Weight%	Atomic%
O K	57.83	69.82
Al K	42.17	30.18
Totals	100.00	

Fig. 8 EDS image of Starting Powder

Alumina Spheres Characterization:

Alumina spheres of size 1.8 mm to 3.2 mm were synthesized by drip casting method.

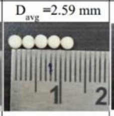
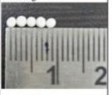
Alumina balls	40wt% Alumina spheres prepared with 1mm dia nozzle		60wt% Alumina spheres prepared with 1mm dia nozzle	60wt% Alumina spheres prepared with 0.5 mm dia nozzle
Green balls		D_{avg} =2.65 mm 	D_{avg} =3.18 mm 	D_{avg} =2.17 mm 
Sintered balls(1650°C/5h)		D_{avg} =2.27 mm 	D_{avg} =2.59 mm 	D_{avg} =1.87 mm 

Fig. 9 Alumina spheres before and after sintering

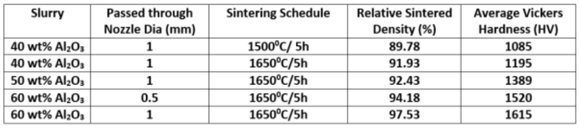
The pictures taken before and after sintering cycle of Alumina spheres is shown in Figure 9. The green balls which were produced by using 1 mm diameter nozzle with 40 wt% alumina, having a relative green density of 58% and average diameter of 2.65 mm whereas the green balls which were produced by using 1 mm diameter nozzle with 60 wt% alumina having a relative green density of 61% and average diameter of 3.18 mm, those which were produced with 0.5 mm diameter nozzle having average diameter of 2.17mm.

Table3: (Density and average diameter of Alumina spheres)

Parameter	40 wt% Alumina Spheres (1mm dia nozzle)	40 wt% Alumina Spheres (1mm dia nozzle)	50 wt% Alumina Spheres (1mm dia nozzle)	60 wt% Alumina Spheres (1mm dia nozzle)	60 wt% Alumina Spheres (0.5mm dia nozzle)
Sintering Schedule	1500^0C/ 5Hr	1650^0C/ 5Hr	1650^0C/ 5Hr	1650^0C/ 5Hr	1650^0C/ 5Hr
Diameter before sintering (mm)	2.65	2.65	2.41	3.18	2.17
Diameter after sintering (mm)	2.36	2.27	2.04	2.59	1.87
Green Density (g/cc)	58	58	59	61	61
Relative Sintered Density (%)	89.78	91.93	92.43	97.53	94.18

Table 3 represents relative green and sintered density, average diameter of the balls before and after sintering. The synthesized spheres were sintered at different sintering schedules and optimum sintering schedule was found to be 1650°C for 5hours. As the sintering temperature raised from 1500^0C to 1650^0C, samples became densified. It is because, metallurgical bond formation is higher at higher temperature.

Table 4: (Vickers hardness of sintered samples measured at 2 Kg load with 10 sec dwell time using Vickers hardness tester (Shimadzu, Japan)):

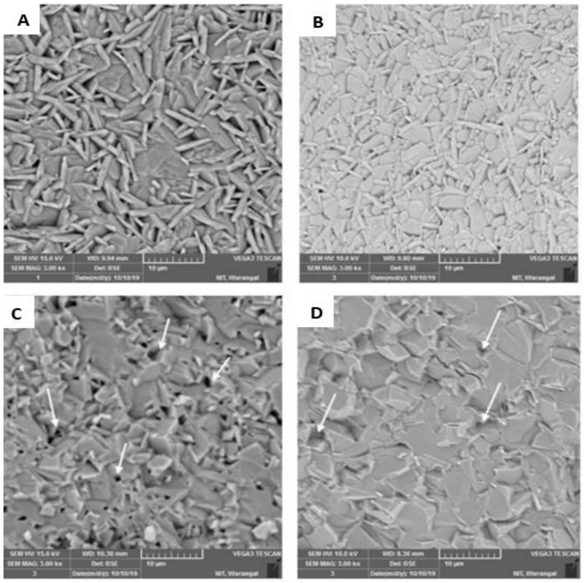


Slurry	Passed through Nozzle Dia (mm)	Sintering Schedule	Relative Sintered Density (%)	Average Vickers Hardness (HV)
40 wt% Al_2O_3	1	1500^0C/ 5h	89.78	1085
40 wt% Al_2O_3	1	1650^0C/5h	91.93	1195
50 wt% Al_2O_3	1	1650^0C/5h	92.43	1389
60 wt% Al_2O_3	0.5	1650^0C/5h	94.18	1520
60 wt% Al_2O_3	1	1650^0C/5h	97.53	1615

Vickers micro hardness test carried out on the alumina spheres (data given in Table 4) that were produced with different wt% of alumina. The alumina spheres, which were produced with 40wt% alumina and sintered at 1500^0C/5Hr, having hardness value of 1085 HV, which is far less than the hardness of samples which were prepared with 60 wt% Al_2O_3 sintered at 1650^0C/5Hr. It is because of high porosity and less hardness of 40wt% alumina samples than 60wt% alumina. The alumina spheres which were synthesized with 50wt% alumina and sintered at 1650^0C/5h having hardness value of 1389 HV. The alumina spheres that were synthesized by using 0.5 mm diameter nozzle and 1 mm diameter nozzle with 60wt% alumina and sintered at 1650^0C/5h having hardness values 1520 HV and 1615 HV respectively.

Microstructure Analysis:

SEM analysis has enabled to find surface morphology and characteristics of the Alumina spheres. SEM images (Figure 10) were taken for samples sintered at 1650^0C/5Hr with 40 wt% Al_2O_3 and 60 wt% Al_2O_3. Images A and B of Figure 10 were taken on the surface of the alumina microsphere, it shows the bimodal distribution of particles of spheres, and a needle-like shape.



1650ºC-5h-40 wt% Al_2O_3 1650ºC-5h-60 wt% Al_2O_3

Fig.10. SEM images of 40 wt% (A & C) Al_2O_3 and 60 wt% (B & D) Al_2O_3

Similarly, images C & D of Figure 10 are the fractographic images which clearly indicate that as the wt% of alumina increased from 40 to 60 there is decrease in porosity (shown by white arrows), it is mainly because of higher cohesiveness between the 60 wt% slurry alumina particles compared to 40 wt% slurry alumina particles, as it caused increase in relative density from 91.93 to 97.53% at 1650^0C - 5Hr The above analysis has confirmed that 60 wt% Alumina-Sodium Alginate slurry and subsequent sintering at 1650^0C for 5 Hrs through 1mm nozzle has yielded the best hardness along with density. Hence, for fabrication of the ceramic pads, the same procedure is adopted to fabricate ceramic grains by thoroughly blending 60 wt % mixture of ceramic powder, containing 60 wt% Al_2O_3, 30 wt% ZrO_2, 6 wt% TiB_2 and 4 wt% Co, with Sodium Alginate to obtain the optimum combination of hardness and toughness to produce high wear resistant material. Moreover, the methodology of fabricating spherical shaped ceramic grains/ beads could be successfully established.

Step-II (Fabrication of ceramic metal matrix composite high chrome insert):

During the initial stages, CMMC inserts were made with manually made patterns and also the grooves required in the mould formed due to these patterns were not in exact position leading to dislocation of pads from the designated location, especially due to metallostatic pressure of the liquid metal. Hence, 3D printed patterns were made so that the exact location of the groove with proper strength could be achieved which resolved the issue.

Moreover, in the beginning inserts were made with directly blended mixture of ceramic powder due to which particles with irregular shapes used to form and because of which many inserts were cracking during stress relieving stage itself or few during the actual running of mill leading to catastrophic failure of the rolls.

However, the spherical shaped ceramic grains had not only removed the possibility of stress concentrations, but provided additional strength to the overall matrix thereby enhancing the toughness while retaining the strength.

IV. CONCLUSIONS

[1] The maximum relative density of the alumina samples was 97.53%, which was synthesized

with 60 wt% alumina sodium alginate slurry, sintered at 1650^0C for 5Hrs using 1mm nozzle which has offered highest hardness (1615 HV) due to minimal porosity in the matrix.

[2] The spherical shaped ceramic grains used for fabrication of ceramic pads for CMMC insert has not only resolved the cracking tendency of the insert/ roll, but enhanced the wear life of the roll.

[3] The average running life of grinding roll could be enhanced by minimum 2000 hours in worst Indian coal conditions (i.e. from 10000 hrs to 12000 hrs)

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