# Optimization of Process Parameters of Zirconia Reinforced Aluminium by Powder Metallurgy using Response Surface Methodology

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***Abstract*—Powder metallurgy process is one of the types of fabrication of composites in which both matrix as well as reinforcement exist as solid powders. Optimizing the process parameters of powder metallurgy in the fabrication of aluminium-zirconia composite used in the bone marrow implants has been done for the desirable physical properties. In this research paper, process parameters such as composite ball milling hours, composition of zirconia and compaction pressure were evaluated using RSM. The properties such as particle density and porosity are studied and tabulated for the proposed composite and the influence of different process parameters over these properties were studied by framing 17 experimental runs using Box-Behnken method. Tests were done by considering three factors and two levels. The parameters which significantly affect the properties were identified using ANOVA. The results indicate that the composition of zirconia predominantly influences the physical properties compared to other factors.**

***IndexTerms- Powder Metallurgy, Optimization, Zirconia, Compaction Pressure, Density***

## I. INTRODUCTION

Powder metallurgy is an important composite forming technique in which a metal and a non metal, both in powdered form are joined together in specific proportion to enhance the mechanical properties so that the properties of the fabricated composite specimens are superior when compared to the individual metal. Metal matrix composites which are fabricated using powder forming process are found to have increased applications in automobile, shipping and aircraft industries.

In our study, we are going to replace the bone joints which are generally made up of corrosion resistant steel with aluminium-zirconia composite. Generally ceramics have the properties such as high strength, hardness, corrosion resistance, least wear rate and bio-inertness[1]. But they are vulnerable to brittleness and slow crack growth. Phase stabilized zirconia is having the unique property of excellent fracture toughness and good adhesive property[2]. It is also having least particle weight when compared to other ceramics that makes it to readily combine with the aluminium powder. Aluminium, on the other hand has good strength-to-weight ratio and workability characteristics that makes it the most popular and inevitable material on earth.

When zirconia powder is mixed with aluminium powder, it forms a perfect blend. Thus this composite can act as a bio-degradable implant to fuse the bones inside human body[3]. This composite acts as a catalyst in the speedy growth of broken bone joints as it degrades and disappears due to the flow of blood over a period of time. Hence this composite proves to be a bio-compatible material to be used as an alternative for the implants[4][5].

In the present study, the objective is to determine the optimum process parameters for the preparation of test specimen with respect to the determined responses.

## II. MATERIALS AND METHODS

### A. *Selection of Materials*

#### a) *Matrix:*

Aluminium alloy(Al6061) metal powder of size 50µm is selected as the major constituent. The uniform size can be obtained by means of physical separation processes such as filtering through various size sieves. The chemical composition and physical properties of aluminium powder is given below:

TABLE I. CHEMICAL COMPOSITION OF ALUMINIUM

| Constituents | Percentage |
|---|---|
| Manganese (Mn) | 0.15% |
| Iron (Fe) | 0.70% |
| Copper (Cu) | 0.15 - 0.40% |
| Magnesium (Mg) | 0.15% |
| Silicon (Si) | 0.4 - 0.8% |
| Zinc (Zn) | 0.25% |
| Chromium (Cr) | 0.4 - 0.35% |
| Others (Total) | 0.05- 0.15% |
| Aluminium (Al) | 95.8-98.6% |

TABLE II. PHYSICAL PROPERTIES OF ALUMINIUM

| Properties | Unit | Value |
|---|---|---|
| Density | g/cm$^3$ | 2.7 |
| Melting point | °C | 582-652 |
| Brinell Hardness | | 45 |
| Ultimate Tensile Strength | MPa | 130 |
| Properties | Unit | Value |
| Yield Strength | MPa | 276 |
| Modulus of Elasticity | MPa | 68.9 |
| Thermal conductivity | W/m-K | 167 |
| Coefficient of Thermal Expansion | m/°C | $23.6\times10^{-6}$ |

### b) *Reinforcement:*

The reinforcement material which is to be added in minor proportion with the matrix is yttria stabilized zirconia of size 5µm. The general properties of zirconia are tabulated below:

TABLE III. GENERAL CHARACTERISTICS OF ZIRCONIA

| Properties | Units | Value |
|---|---|---|
| Density | g/cm$^3$ | 5.74 |
| Melting point | °C | 2815 |
| Vickers Hardness | | 1170 |
| Fracture toughness | MPa√m | 12.0 |
| Elastic Modulus | GPa | 205 |
| Tensile Strength | MPa | 380 |
| Thermal conductivity | W/mK | 3 |
| Coefficient of thermal expansion | m/°C | 10.2 |

### B. *Fabrication process*

In this research work, both powders are charged into the ball mills and pulverized together for hours in order to obtain uniform particle size. They are then mixed together and blended with suitable additives for different compositions of aluminium and zirconia.

Now the powder blends are poured into the circular die cavity of specified dimensions. Then the blends are compacted to green compacts as pressure is exerted by punch on the top of the compact against the cylindrical walls of the die. The entire setup is placed in between the compression chamber of Universal Testing Machine which supplies force to the punch as shown in figure 1.

Fig. 1. Universal testing machine

The as prepared green compacts of diameter 12mm and length 80 mm are sintered at a temperature of 500ºC with 5ºC raise in temperature per minute in the muffle furnace as shown in the figure 2.

Fig. 2. Muffle Furnace

### C. *Determination of Responses*

The particle density and porosity of the sintered specimens are determined using Archimedes' principle.

Experimental density or particle density of the composite specimen is calculated using Archimedes' principle.Initially for measuring the density,test samples of different compositions are cut from the specimen, grinded and highly polished before weighing. They are made into perfect cubes having dimension (lxbxt) 10x10x10 mm. The mass of the composite is measured using a highly accurate digital mass balance of unit weight 0.01g as shown in figure 3.

Fig. 3. Determination of density

$$\rho = (m_A/(m_A - m_{su}))\text{x}\ \rho_{water} \quad (1)$$

where,

$\rho$ = density of the sample in g/cm$^3$
$m_A$ = mass of the sintered sample in air in g.
$m_{su}$ = mass of the sample suspended in distilled water in g.
$\rho_{water}$ = density of water (≈ 1 g/cm$^3$)

The porosity indicates the void or holes inside the material and is generally expressed in percentage.

$$\%\Phi = (m_{dip}- m_A)/(m_{dip} - m_{su}) \quad (2)$$

where,

$m_{dip}$ = mass of the sample dropped inside the water in g.

## III. EXPERIMENTAL PROCEDURE

### A. *Experimental Design by RSM – Box Behnken Method*

RSM is a collection of both scientific and mathematical techniques used for evaluating problems in which a particular response is influenced by variety of factors. Here a standard technique in RSM called Box-behnken Design is opted to study density and porosity of the sintered compacts[3]. Design for three factors namely ball milling time, composition of zirconia and compaction pressure each with two levels are formed in Design of Experiments. We are going to do multi objective optimization function by maximizing the density and minimizing the porosity that gives optimal solution which suits bone implants[5].

All the samples are prepared according to the test runs developed by DESIGN EXPERT 7. The controlling parameters are given in Table IV.

TABLE IV. CONTROLLING FACTORS AND THEIR LEVELS OF STUDY

| Symbol | Factors | Experimental values | |
|---|---|---|---|
| | | Low level | High level |
| A | Ball milling time (hours) | 2 | 6 |
| B | Composition of $ZrO_2$ (WT. %) | 5 | 15 |
| C | Compaction Pressure (MPa) | 100 | 130 |

Table V gives the list of experimental runs in the design of process parameters using Box-behnken design and the values of the responses.

TABLE V. PROCESS DESIGN LAYOUT

| Run | Ball milling time | Composition of $ZrO_2$ | Compaction Pressure | Particle Density | Porosity |
|---|---|---|---|---|---|
| No. | Hours | Wt % | MPa | g/cm$^3$ | % |
| 1 | 4 | 5 | 130 | 2.72 | 3.14 |
| 2 | 4 | 10 | 115 | 2.78 | 4.12 |
| 3 | 4 | 10 | 115 | 2.81 | 4.89 |
| 4 | 4 | 15 | 130 | 3.02 | 7.12 |
| 5 | 4 | 10 | 115 | 2.83 | 4.6 |
| 6 | 2 | 5 | 115 | 2.77 | 3.56 |
| 7 | 4 | 10 | 115 | 2.83 | 5.33 |
| 8 | 6 | 10 | 100 | 2.66 | 5.02 |
| 9 | 2 | 15 | 115 | 2.98 | 7.06 |
| 10 | 2 | 10 | 130 | 2.85 | 4.87 |
| 11 | 2 | 10 | 100 | 2.81 | 3.98 |
| 12 | 6 | 5 | 115 | 2.68 | 2.89 |
| 13 | 6 | 15 | 115 | 2.87 | 6.33 |
| 14 | 4 | 5 | 100 | 2.66 | 2.7 |
| 15 | 4 | 10 | 115 | 2.87 | 4.09 |
| 16 | 6 | 10 | 130 | 2.84 | 5.12 |
| 17 | 4 | 15 | 100 | 3.11 | 7.23 |

TABLE VI. ANOVA FOR RESPONSE SURFACE LINEAR MODEL FOR DENSITY

| Source | Sum of squares | Df | Mean square | F value | p-value Prob>F |
|---|---|---|---|---|---|
| Model | 0.19 | 3 | 0.062 | 14.94 | 0.0002 (significant) |
| A-ball milling time | 0.016 | 1 | 0.016 | 3.90 | 0.0698 |
| B-composition of $ZrO_2$ | 0.17 | 1 | 0.17 | 39.83 | <0.0001 (most influencing) |
| C-compaction pressure | 4.513E-003 | 1 | 4.513E-003 | 1.09 | 0.3161 |
| Residual | 0.054 | 13 | 4.150E-003 | | |
| Lack of Fit | 0.050 | 9 | 5.515E-003 | 5.11 | 0.0656 (not significant) |
| Pure Error | 4.320E-003 | 4 | 1.080E-003 | | |
| Cor Total | 0.24 | 16 | | | |

Std. Dev.=0.064; Mean=2.83; C.V%=2.28; press=0.11; R-Squared=0.7752; Adj R-Squared=0.7233; Pred R-Squared=0.6972; Adeq Precision=12.080

From the Table VI, it is observed that the Model F-value 14.94 implies that the model is significant. There is only 0.02% chance that this large value could occur due to noise. Values of Prob>F less than 0.0500 indicates that model terms are significant. In this case B-composition of $ZrO_2$ is more significant. Values greater than 0.1 indicate model terms are not significant. The Lack of Fit value 5.11 implies that low probability (<10%) is troubling. The Pred R-Squared value of 0.6972 is in reasonable agreement with Adj R-Squared value 0.7233. Adeq precision measures signal to noise ratio. The ratio of 12.080 is greater than 4 and indicates an adequate signal.

The regression equation in terms of coded factors for calculating the density of proposed material is given below:

$$\text{Density} = +2.83-0.045A+0.14B+0.024C \tag{3}$$

TABLE VII. ANOVA FOR RESPONSE SURFACE LINEAR MODEL FOR POROSITY

| Source | Sum of squares | Df | Mean square | F value | p-value Prob>F |
|---|---|---|---|---|---|
| Model | 30.06 | 3 | 10.02 | 41.46 | <0.0001 (significant) |
| A-ball milling time | 1.512E-003 | 1 | 1.512E-003 | 6.259 E-003 | 0.9381 |
| B-composition of $ZrO_2$ | 29.84 | 1 | 29.84 | 123.47 | <0.0001 (most influencing) |
| C-compaction pressure | 0.22 | 1 | 0.22 | 0.90 | 0.3598 |
| Residual | 3.14 | 13 | 0.24 | | |
| Lack of Fit | 2.03 | 9 | 0.23 | 0.82 | 0.6341(not significant) |
| Pure Error | 1.11 | 4 | 0.28 | | |
| Cor Total | 33.20 | 16 | | | |

Std. Dev.=0.49; Mean=4.83; C.V%=10.19; press=5.27; R-Squared=0.9054; Adj R-Squared=0.8835; Pred R-Squared=0.8411; Adeq Precision=17.582

ANOVA test carried out for predicting the influence of process parameters in porosity of the composite samples is tabulated above.

From the Table VII, it is observed that the Model F-value 41.46 implies that the model is significant. There is only 0.01% chance that this large value could occur due to noise. Values of Prob>F less than 0.0500 indicates that model terms are significant. In this case B-composition of $ZrO_2$ is more significant. Values greater than 0.1000 indicate model terms are not significant. The Lack of Fit value 0.82 implies that low probability (<10%) is troubling. Non significant lack of fit is good. The Pred R-Squared value of 0.8411 is in reasonable agreement with Adj R-Squared value 0.8835. Adeq precision measures signal to noise ratio. The ratio of 17.582 is greater than 4 and indicates an adequate signal.

The regression equation in terms of coded factors for calculating the porosity of proposed material is given below:

$$\text{Porosity} = +4.83-0.014A+1.93B+0.17C \tag{4}$$

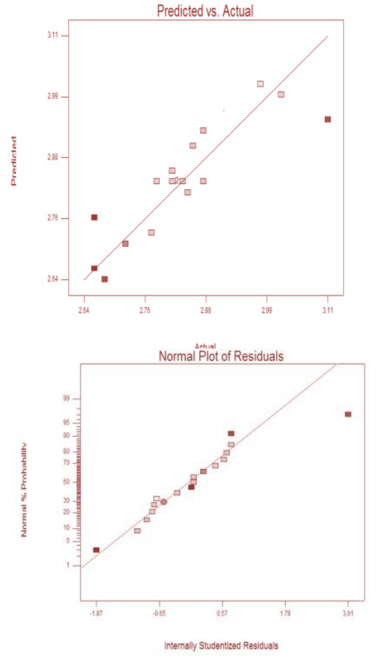
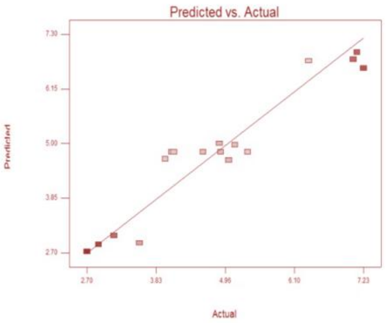


Fig. 4. Predicted vs Actual plot and Normal plot for density



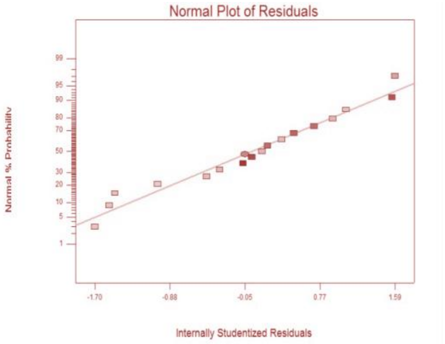


Fig. 5. Predicted vs Actual plot and Normal plot for porosity

It is desirable to increase the density and reduce the porosity of the material using the data and it is observed using the Design Expert built in optimization tool. The optimal results are given in Table VIII.

TABLE VIII. OPTIMIZED VALUES OF THE PROCESS PARAMETERS AND FACTORS

| | |
|---|---|
| Ball milling time (hours) | 2 |
| Composition of $ZrO_2$ (wt %) | 8.75 |
| Compaction pressure (MPa) | 130 |
| Density (g/cm$^3$) | 2.8616 |
| Porosity (%) | 4.52193 |

## IV. CONCLUSION

In the above study, zirconia reinforced aluminium metal powder composite is fabricated using powder metallurgy technique. Three process parameters namely ball milling time, composition of zirconia and compaction pressure are selected as factors with two levels. The influence of these factors are studied using RSM. The responses are two physical properties namely density and porosity.From the above mentioned results it is clear that the factor significantly affecting the responses is percentage composition of zirconia.

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