Powder metallurgy process towards functional gradation of Al-Al$_2$O$_3$ metal ceramic mixture samples

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Abstract. Functionally graded materials (FGM) belong to the class of advanced materials whose properties vary along one or more coordinate. In the present study, an attempt has been made to manufacture FGM beams using a powder metallurgy process. The Al and Al$_2$O$_3$ in powder form are mixed to different volume fraction according to the rule of mixture. These samples are ball milled to form a proper blended mixture and to reduce the particle size for 1 hour at 250 rpm with tungsten carbide balls. A cold compacted die made of AISI D2 steel is used for preparing the sample. Different composition of Al and Al$_2$O$_3$ with Al$_2$O$_3$ varying from 0 to 10% is laid in the die using ethanol as the binder. This sample is compressed using hydraulic press with a load varying from 50-70 ton. The compressed sample is removed using a compression moulding machine, then it is sintered to a temperature of 650°C using a muffle furnace to form a smooth graded Al-Al$_2$O$_3$ FGM beam. Various defects such as surface cracks and bending were experienced during the preparation of samples; these defects were overcome by varying the parameters such as loading and sintering temperature.

1. Introduction

FGM is basically an advanced class of composite in which the volume fractions of two or more materials are varied continuously. Two types of gradation can be conceptualized: a continuous gradation and a stepwise gradation. Human bone is a best example for continuous gradation which is formed by variation in porosity and composition. Spark plug and gears, in which gradient is formed by changing its composition from a ceramic to metal comes under step wise graded material [2].

Different manufacturing process such as vapor deposition technique, plasma spray coating, powder metallurgy, centrifugal method and solid freeform fabrication method has been developed to produce the FGM [1]. Selecting the suitable manufacturing process depends on the volume of FGM production. Powder metallurgy is commonly used in mass production primarily being economical compared to other production process.

This paper gives an overview of manufacturing FGM beam samples using powder metallurgy process, steps involved such as ball milling the powder mixture, design and preparation of a die, powder layup into the mold, compaction of the layered sample, sintering and cooling. A stepwise gradation method is adopted and is shown in the figure 1.
2. Processing of Functionally graded beam

Schematic representation of the manufacturing process is shown in figure 2.

2.1. Features of powder compaction die

AISI D2 material is used for machining the die for cold compaction of powder samples. The die is made of three parts namely top die, middle die and bottom die as shown in figure 3.
Top die can also be defined as a punch block of 210mm×60mm×21mm in dimension. It has a projection of 150mm×20mm×25mm and also has three holes through its thickness. The mid die is of 210mm×60mm×25mm and it has a cavity along its length where different powder compositions are laid. The dimension of cavity is 150mm×20mm×20mm. The cavity is provided with 2° draft angle at inner walls for easy removal of green compact. The bottom most part of the die is of 210mm×60mm×15mm in dimension.

2.2. Powder mixing

Commercially available aluminium and aluminium oxide powders are used for preparing FG beam samples. These powders are mixed according to required volume fraction proportion with regards to different layers and are blended at 250 rpm for 1 hour in high energy ball mill for proper mixing of aluminium and alumina oxide. The powder is placed into cylindrical jar consisting of 50 tungsten carbide balls having 10 mm size. The tungsten ball to powder weight ratio is 10:1. Particle sizes of ball milled powders according to their different volume fraction are measured with the help of scanning electron microscope.

2.2.1. Determination of powder mass

Powder mass measurements are taken according to the thickness of beam. In the present study samples with thickness 2.25 mm and 2.5 mm are fabricated. Thickness of each layer can be found out by dividing thickness of complete beam with number of layer. The volume of each layer is calculated according to the dimension of cavity and thickness of each layer. The density of each powder layer can be calculated with the help of law of mixture which is given by

\[
\rho_{\text{eff}}(z) = \rho_m V_m(z) + \rho_c V_c(z)
\]

Where

\[
V_m + V_c = 1
\]

\[
\rho_{\text{eff}} = \text{effective density}, \quad \rho_m = \text{metal density (Al)}, \quad \rho_c = \text{ceramic density (Al}_2\text{O}_3), \quad V_m = \text{metal volume}, \quad V_c = \text{ceramic volume}
\]

The mass of the Al-Al\textsubscript{2}O\textsubscript{3} for preparation of the FGM beam depends on the dimensions of the beam i.e. length (l), width (b) and thickness (t). In the present study 2 different thicknesses of the beams are prepared. The dimensions and the mass of the beam prepared are shown in table 1. The percentage weight composition of Al\textsubscript{2}O\textsubscript{3} is given in table 2.

2.3. Die filling

Die filling is done manually with the help of commercially available sieve. The stability of powder flow and apparent density is critical for better consistency in fabrication of samples. Various distinctive conditions such as humidity and temperature plus storage duration can all influence the performance of a powder mixture. It has also been found that environmental conditions during the compaction process can influence the performance of powder mixtures [3]. Hence for this purpose binders are used for better green compact strength in powder metallurgy products. In this study ethanol is used as binder medium for mixing the powder (Al- Al\textsubscript{2}O\textsubscript{3}).

In the die filling process firstly, die walls are cleaned by zinc stearate with acetone [4]. Zinc stearate is used to avoid the sticking of powder mixtures to die wall. Then different powder mixture stocks are mixed with ethanol binder and deposited in the die. The deposition starts with 10% Al\textsubscript{2}O\textsubscript{3} and 90% Al layer and the last layer is of 100% Al composition. After laying one layer of certain powder mixture the powder layer is pressed with the help of top die so that layer is flattened to receive the next layer of mixture.
Table 1. Parameters involved in FGM beam preparation.

| Parameters(mm) | 100% Al | 98.75% Al | 97.5% Al | 95% Al | 90% Al |
|----------------|---------|------------|----------|-------|-------|
| l=153,b=21,t=2.25 | 3.7582  | 3.7765     | 3.7948   | 3.8315| 3.9047|
| l=153,b=21,t=2.5  | 4.514   | 4.5405     | 4.5665   | 4.6189| 4.7234|

Table 2. Mass of Al2O3 in FGM beam preparation.

| Parameters(mm) | 100% Al | 98.75% Al | 97.5% Al | 95% Al | 90% Al |
|----------------|---------|------------|----------|-------|-------|
| l=153,b=21,t=2.5 | 0       | 0.2571     | 0.3373   | 0.4982| 0.8196|

2.3.1. **Cold compaction of FGM samples**

After completion of the filling of die cavity with powder mixture, it is taken to a compaction machine. For this purpose a universal testing machine of 100 ton capacity is used. 60 tons load is applied gradually for 5 minutes, after which sample is removed by compression molding machine. Samples removed by this process are termed as green compacts they possess sufficient strength to maintain the shape of beam.

2.4. **Sintering process**

The green compact samples which are obtained after compaction cannot withstand mechanical load. The green compacts are therefore kept in a furnace under controlled condition to obtain samples with better strength. Studies on the effect of various parameters such as particle size, sintering temperature and duration of sintering on physical properties of Al-Al2O3 based alloys fabricated with the help of powder metallurgy process have been reported [5]. They found highest relative density of 99.95% around 600°C. Hence sintering temperature was set up in between 600°C and 650°C for sintering of FGM beam samples. Studies on effect of step-wise sintering temperature increment on the properties of Al-SiC based alloys were reported by [6].

Based on these literatures the maximum sintering temperature was set as 650°C. During sintering the furnace temperature was gradually increased at a rate of 10°C per minute. Once the maximum temperature was attained, the samples were retained in the furnace for 4 hours. Further, samples were allowed to cool inside the furnace itself to make sure there were no cracks developed due to sudden cooling or air quenching. It was seen while operation that furnace nearly takes 1 to minute of time to reach 10°C of temperature.

3. **Analysis of FGM beams**

Various parameters such as load, binder quantity, re-compaction load and temperature were varied to manufacture a good FGM beam samples. The optimum load, temperature, binder required is shown in the table 3.

Table 3. Parameters involved in FGM beam preparation.

| Parameters          | Equipment used    | Units  |
|---------------------|-------------------|--------|
| Compression load     | Universal testing machine | 60 ton |
| Binder               | Ethanol           | 4-5 drops |
| Sintering temperature| Muffle furnace    | 650°C  |
3.1. Defects in the sample

All the samples prepared were of 5 layers. The composition of each layer is as given in the table 1. During initial stages of sample preparation lot of defective samples were obtained. The main defects of the FGM samples were top face peeling and cracks in the sample. The reasons for crack are due to the loading effect. Initially the load applied on the sample was around 40-50 tons which was found to be not sufficient for the proper binding of the powder particles [6]. So this defect was overcome by increasing the compression load. Face peeling is also because of the quantity of binder used. So this defect was overcome by re-compacting the sample after removal around 10-15 tons. 15 FGM samples were prepared and the brief analysis is given below:

Figure 4 FGM sample with face peeling.

Figure 5 FGM sample with powder erosion and crack.

Figure 4 and 5 gives the FGM samples with defects such as face peeling, irregular edges, surface cracks before sintering and powder erosion after sintering due more usage of ethanol respectively.

Figure 6 is the sample which is obtained after maintaining all the optimum parameters. Minor defects such as warping during sintering were overcome by keeping the sample between two ceramic tiles to reduce the thermal stresses which led to buckling [8, 9 and 10].

The reason for restricting the maximum percentage of $\text{Al}_2\text{O}_3$ to 10% is because as the ceramic content is increased it was observed there was no proper binding between the powder particles and there was surface peeling on the ceramic side [8, 9 and 10].

From the SEM analysis of the FGM sample as shown in Figure 7 it can be seen that $\text{Al}_2\text{O}_3$ dispersion is sparse in the matrix of Al. The large white spots are the $\text{Al}_2\text{O}_3$ whose percentage is increasing layer by layer.

Aluminum-steel FGM were fabricated using powder metallurgy process [4] with 100% Al and 100% steel, these samples were having fewer defects. In current work the FGM is fabricated using metal ceramic mixture (Al-$\text{Al}_2\text{O}_3$), more the content of the ceramic more will be the defects. Thus the volume fraction of $\text{Al}_2\text{O}_3$ is limited to 10%. The various processing techniques and the problems faced during the fabrication process were discussed by [11 and 12]. It was observed that the preparation of FGM manufacturing is a costly process and mass production is not effective. Whereas the powder metallurgy process is the cost effective and the mass production is possible.
Figure 7 SEM image of the FGM sample.

4. Conclusions
It can be concluded that due to large difference in coefficient of thermal expansion of aluminium and alumina, the maximum volume fraction of alumina in aluminium is limited to 10% in order to avoid warping during sintering. The microstructure study of FGM powder mixtures shows the uniform distribution of Al₂O₃ in Al. The micrographs of FGM beam samples indicate the layer-wise gradation of aluminum and alumina powders.

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