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Dry slide wear behavior of 601AC/B₄C Selective layered functionally graded material with particulate Ni-P coating

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Abstract

601AC/B₄C selective layered Functionally Graded Material with Nickel coating and without Nickel coating is fabricated by powder metallurgy technique. The basic metal alloy powder is made with Mechanical alloying technique. Controlled variables Number of layers (2, 3 and 5), particulate size (25 μm and 50 μm), Load (10 N, 20 N and 30 N), track diameter (60 mm, 80 mm 100 mm), and the speed of the disc (500, 750 and 1000 rpm) are selected to conduct dry sliding wear test using pin on disc apparatus. Experiments are design using Taguchi’s L36 orthogonal array. Optimization of parameters is done using Signal-to-Noise ratio. For the wear rate smaller-the-better criteria is used for analyzing the results. Analysis of Variance is done using Minitab software to find the most significant variables for the wear rate. From the results it is concluded that particle condition with 31.49% contribution, number of layers with 30.61% and speed of the disc with 10.36% contribution are the most significant factors. Linear regression equation is developed and conducted confirmation test. From the comparison a 13.5% error is observed between predicted value and actual value. Therefore a quadratic regression equation is proposed to predict the wear rate for this experiment.

1. Introduction

Every engineering problem, leads the to a new invention, minimization of thermal stresses at inter-metal-ceramic composites used in rocket engine leads to the development of new substantial material in the early 1980’s in Japan [1]. Japanese named this material as Functionally Graded Materials (FGMs) because of the variation of gradient of particulate in a particular direction. In reality the FGM concept is a gift given by nature to the human. Almost every materials irrespective of animate or inanimate, applies the concept of FGM. Exemplifying a few, layers of earth formation, structure of bones, tress trunk, nails, tissues, bamboos, snail shell etc, The structural gradient is depending on different applications, loading conditions and functional requirement [2]. Potential worldwide need of light weight metal matrix material leads to the Aluminum Alloy (AA) metal matrix composites. Among the wide variety of aluminum alloy family Al2024 and Al6061 are proved to be the best engineering materials for various applications like aircraft, automobile and structural [3, 4]. Wear resistance of AA-FGM is a significant property among all superior properties of components in the tribological field [5].

In this work, dry sliding wear behavior of 601AC/B₄C selective layered FGM is investigated using pin on disc apparatus. Powder Metallurgy (P/M) technique is adopted for the fabrication of the specimens. To increase the wettability of B₄C particles are coated with Nickel using electroless process [6]. Influence of centrifugal casting process parameters on the wear behavior of AA/SiCp were studied by A C Vieira et al [7], observed the correlation between wear behavior and the SiC distribution. Dry sliding wear behavior of hybrid MMC, Al 2219/SiCp-Gr was investigated by S Basavarajappa et al [8], abrasion mechanism is observed and estimated the wear rate under different parameters, sliding speed, load and track radius. Three body abrasion wear behavior of Al/B₄C FGM was investigated by Harsha, A P, &Tewari [9]. They used liquid route to fabrication of specimens, and conducted dry wear test in the present of silica sand AFS 50/70 as an abrasive medium. F Toptan et al [10]

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investigated the tribocorrosion behavior of Al-Si-Cu-Mg composite using B4C particles as reinforcement and in presents of NaCl solution. They came to the conclusion that there is a reduction in tribocorrosion of composite mainly due to the debris accumulation of counter material over the B4C particles. Functionally graded materials find a potential applications in automobile, defense, bio-medical, nuclear cells, and photocells. Some of the applications are listed as under. A G Arsha et al [11] fabricated FGM automotive piston made of Aluminum composite using in situ technique. They conducted hardness and dry wear abrasion test and concluded that A390 + 0.5% Mg Al FGM piston has high hardness and low wear rate. Chin-Yu Huang and Yu-Liang Chen [12] fabricated AA/Al2O3/ZrO2 FGM plates using powder metallurgy technique which are applicable to ballistic resistance. They concluded that ceramic-ceramic FGM have high toughness and hardness to resist the ballistic impacts. E Muller et al [13] fabricated thermoelectric (TE) FGM material using thermal spray of doping-graded Fesi2 applicable to sensors and thermogenertors. They concluded that, to identify optimal spatial compositional distribution, FGM concept is best suited material design tool. W Pompe et al [14] applies FGM concept for the biomedical applications. They developed knee joint replacement by building a graded structure of ultra high molecular weight polyethylene with high density polyethylene fiber reinforcement. W Z Zhu and S C Deevi [15] used chemical vapor deposition (CVD) to fabricate the Ni-ZrO2(Y2O3) cermets applicable to Solid oxide fuel cell (SOFC). They applied FGM concept to stack the layers of cermets. He Gong et al [16] proposed a potential application of FGM for proximal femoral nail (PFN), in which PFNA device can be fabricated to have material composition of nail gradually varying from more stiff (at femur) to more flexible (at distal side).

From the above literature it is evident that there is a need of much more investigation on FGMs in different area of applications. Most of the investigators used liquid route, additive manufacturing or CVD to fabricate the FGMs. A few authors attempted the simple and power full P/M fabrication method for FGM. Some of the authors used P/M to fabricate bulk FGMs in which the ceramics are reinforced directly into the metal matrix. There is a compatible problem between the two materials (ceramic and metal) due to two quite opposite material properties. This problem can be rectified by the modification of surface properties of ceramic particles. In this work an attempt is made to investigate the dry wear behavior of selective layered FGM, made of 601AC matrix reinforced by Nickel coated B4C particles.

2. Selection of material

Al 601AC comes under the family of aluminum P/M [17], alloying element composition given in the table 1, all the metal powders which are commercially available with 340 mesh (average particle size 50 μm) are selected (supplied by Shubhmets metal powder Pvt. Ltd, India). Metal alloys are prepared by Mechanical Alloy (MA) technique [18]. The elemental metal powders are mixed and blended for 2 h using bi-directional speed control blending machine to attain uniform alloy mixture. Commercially available Boron Carbide (B4C) of 340 mesh (average particle size 50 μm) (supplied by Next gen Steel & alloys, India) is used as particulate with 5%, 10%, 15% and 20% weight percentage composition at different layers of FGM. The received 50 μm B4C powder is milled for 4 h and 25 μm size B4C particles are separated.

Alloy powder Al601AB is made by blending of individual metal powders of same particle size and is blended to get a uniform mixture of all metal powders. The properties of 601AB are similar to wrought alloy Al6061, which offers good ductility, corrosion resistance, and high strength and specified for anodized parts. The basic difference between 601AB and 601AC is the way in which lubricant is used. In 601AB lubricant is mixed along compared to hard wax, parafin, Linseed oil and fisher-tropsch [20].

Boron Carbide (B4C) comes third in the list of ceramics after Diamond and Cubic boron nitride in terms of hardness (Vickers hardness 2900–3900 kgf mm−2) [21]. The additional properties of B4C are very high melting point (2450°C), good chemical resistance, low density (2.52 g cm−3) and neutron absorption properties. Because of these additional properties B4C has vast applications like dressing diamond tools, hot pressed sand blasting nozzle, ceramic tooling dies etc With its high hardness and light weight, B4C has the applications in

| Designation | Cu(%) | Mg(%) | Si(%) | Al(%) | Lubricant(%) |
|-------------|-------|-------|-------|-------|--------------|
| 601AB       | 0.25  | 1.0   | 0.6   | Balance| 1.5          |
| 601AC       | 0.25  | 1.0   | 0.6   | Balance| —            |
armor material used for military personnel and vehicles to protect from ballistic attack. Because of its neutron absorption property it is used as absorbent in nuclear power plants in the form of shielding, control rod and shut down pellets [22]. The above properties of B₄C are acquired due to its molecular structure of twelve atom icosahedral clusters linked by direct covalent bonds. These clusters are located at the corners of a rhombohedral unit cell [23].

3. Methodology

Solid route called Powder Metallurgy process is used for making the specimens by following the conventional procedure as shown in figure 1. The major problem with AA/B₄C composite is the wettability of ceramic (B₄C) with metal alloy (AA). This compatibility problem is reduced by coating the ceramic particles with metal ions [24]. Different methods like electrolysis process, electroless process, electroforming, barrel nickel plating etc, are available for coating the ceramic materials with metals [25]. Since the particulate size is in microns, the best suited coating method is electroless process [26]. This electroless plating technique was proposed by Brenner and Riddell and being used from the past two decades in may application [27]. In this process metallic salts are reduced to metal ions and these metal ions are made to deposited on ceramic particles under favorable conditions.

Electroless process includes five major steps

(i) Ultrasonic agitation
(ii) Etching
(iii) Sensitization
(iv) Activation
(v) Plating

The electroless plating technique is explained with diagrams from figures 2(a) to (e), and followed ASTM B-733 standard for Ni plating on the B₄C particles. The confirmation of Ni coating on boron carbide particle is
done with images of Scanning Electron Microscope (SEM) and Energy Dispersive x-ray Analysis (EDAX) using VEGA3 TESCAN with the magnification of 1.00kX.

Nickel coated Boron Carbide particles are used as particulate in the Aluminum alloy matrix for making the Functionally Graded Materials. Three different FGM specimens, two layered, three layered and five layered were fabricated. In the two layered FGM bottom layer is with 0 wt% and the top layer is with 5 wt% of Nickel coated B₄C. Three layered FGM is with 0%, 5% and 10 wt% composition with the matrix material. Five layered FGM is fabricated with 0%, 5%, 10%, 15% and 20% of Nickel coated B₄C. For comparing the wear behavior of Ni coated B₄C reinforced layered FGM, specimens were fabricated B₄C particulate without Ni coating [28]. The gradient
patterns of the specimen are showing in figures 3(a)–(c). The prepared composition of calculated weight is staked inside the die set and compressed to a load of 600 kN (325 MPa) using hydraulic compression testing machine. Sintering is done at 530°C temperature using muffle furnace with the capacity of 1000°C by following the sintering process as shown in figure 5. The size of the all layered disk is of 50 mm diameter and 10 mm thickness as shown in figure 7(a.), figure 7(b) shows the marking to cut the wear specimens. Square cross section specimens with the dimensions 25 X 10 X10 mm are cut from the center of the disk as shown in figure 7(c).

Figure 4 shows the hydraulic compacting machine used for compacting of metal powders in the dies. The specifications of the machine are shown in the diagram as shown below.

Figure 5 shows the muffle furnace used in sintering process. Atmospheric conditions are maintained inside the furnace. The specifications of the furnace are mentioned along with the diagram. Figure 6 shows the sintering process followed, in term of time (min) and temperature (°C)

### 3.1. Pin on disc experimentation

After screening the controllable variables are with their levels are given below (table 2).

The number of factors is six, of which two are with two levels and four are with three levels. For the range of mixed level design $2^{1-11}$ and $3^{2-12}$, L36 orthogonal array suited. The experimental design for conducting abrasive wear test using pin–on-disc apparatus is given in the table. Different controlled variables are set with the corresponding levels according the design table and the experiment is run for 15 min Wear test is conducted according to ASTM G99-95a standard on standard steel disc (EN-31) with roughness value 0.14 to 0.15 μm. Before conducting each experiment the initial weight of specimen is noted up to the 4th decimal with high precision electronic weighing machine. After each experiment the final weight of the specimens are taken and the weight loss $[29]$ is calculated with the equation (1).

$$\Delta w = w_1 - w_2$$ (1)

Where $\Delta w =$ Weight loss in gm

$w_1 =$ Weight of the specimen before test (gm)

$w_2 =$ Weight of the specimen after test (gm)

| Compression Testing Machine specification |
|------------------------------------------|
| Make                               | AIMIL, India |
| Max. capacity                        | 1000 kgf    |
| Accuracy                            | 1 kgf       |
| Piston diameter                      | 15 cm       |
| Cylinder height                      | 18 cm       |
| Calibrated                           | 2018        |

| Muffle furnace specification         |
|--------------------------------------|
| Make                                 | INDFURR, INDIA   |
| Heating mode                         | SiC heating elements |
| Max. Temperature                     | 1400°C          |
| Accuracy                             | 1°C              |
| Sensor type                          | Platinum/Rhodium |
| Power rating                         | 5 kW             |
Volume loss ($\Delta V$) of the test specimen is calculated using equation (2)

$$\Delta V = \frac{(W_1 - W_2)}{\rho} \times 1000$$  \hspace{1cm} (2)

Where $\rho$ = Experimental density of the specimen

Specimen wear rate ($W$) is computed using equation (3)

$$W(\text{wear rate}) = \frac{\Delta V}{S_d} \text{ (mm}^3/\text{m})$$  \hspace{1cm} (3)

Where $S_d$ is the sliding distance in m.

Experimental densities of the specimens are obtained by Archimedean principle [30] and are listed in table 3 and figure 8 is the computer integrated density measuring machine.

The reliability of the experimental densities is confirmed by the theoretical calculation of density of the FGM specimens using rule of mixture.

$$\rho_{FGM} = \text{Avg} \left( \sum_{n}(V_{fm} \times \rho_m + V_{fr} \times \rho_R) \right)$$  \hspace{1cm} (4)

Where 'N' is the number of layers, $V_{fm}$ is the volume fraction of matrix, $\rho_m$ is the matrix density, $V_{fr}$ is the volume fraction of reinforcement, $\rho_R$ is the reinforcement density.
Considering the compositions of Al 601 AC given in table 1, and the densities of corresponding alloying elements the theoretical density of Al 601 AC is 2.70381 g cm$^{-3}$.

The theoretical densities of the FGM specimens without Nickel in the table are calculated using equation (4). Since the amount of nickel deposited on to the boron carbide particles is of nanometer level, the volume fraction of nickel is unknown. Assuming the theoretical density of FGM specimen with nickel and without nickel are the same, the error term of theoretical and experimental density is calculated. The error between theoretical and experimental density indicates the porosity of the FGM specimens fabricated by powder metallurgy. From the table 4 it is observed that the porosity of FGM specimens with nickel deposition is less when compare to porosity of FGM specimens without nickel. The decrease in the porosity is due to the filling of gaps between boron carbide particles and the 601 AC matrix in terms of Nickel layer deposition by electroless process. The five layered FGM specimen with nickel is having high density compare to the theoretical density. This may be due to high composition of boron carbide in layer four (15%) and five (20%), which includes excess amount of nickel (density 8.908 gm cm$^{-1}$ c.).

4. Results and discussion

4.1. Characterization of powder particles

Figure 9(a) shows the SEM image of Boron carbide particles after clinching with acetone. Most of the particles are dendrite in shape. The SEM image of Boron carbide particles after Nickel coating with electroless process is as shown in figure 9(b). Further confirmation of Nickel on Boron Carbide particles, EDAX is taken for the full area of the powder sample.

Figure 10 shows the EDAX of B$_4$C powder particles after clinching, weight% of boron is observed as 13.49 and the weight percentage of carbon is observed as 60.69 and the remaining is oxygen. Figure 11 shows the EDAX of B$_4$C after nickel coating. The present of nickel on the particles is observed in SEM image as a white clusters on each particles (shown in figure 9(b)), and is confirmed in EDAX, since the amount of nickel deposited on B$_4$C particles is in nano level, the weight percentage for nickel is observed as 1.13. Figure 12 shows comparative XRD graph of B$_4$C and B$_4$C + Ni. The (h k l) values of all peaks are marked and compared with the standard XRD patterns of B$_4$C (JCPDS No. 35-798 and 06-0555) and for Nickel (JCPDS No. 75-787 and 04-850).

4.2. Characterization of FGM layers

Figures 13(a) to (d) shows the SEM images of FGM layer with 5% B$_4$C, 10%, 15% and 20% B$_4$C reinforcement. From the figures it is observed that the number of particles present in a particular area is increasing from 13 (a) to 13 (d). It is also evident that there is no cluster formation of particles and they are uniformly distributed. Therefore it is concluded that the blending process of alloying powder and B$_4$C reinforcement is done properly.

Figure 14(a) gives the XRD of Al 601 AC in which the alloying element Al, Mg, Si, and Cu are observed as peaks and marked with symbols, these peaks are compared and confirmed with JCPDS(04-0787 for Al, 27-1402 for Si, 04-0836 for Cu, 78-0430 for Mg), figure 14(b) show the XRD of Al601 AC + B$_4$C + Ni. The extra peaks observed in figure 14(b) show the present of boron carbide and nickel. Figure 15 shows the comparative XRD of all the layers with Al 601 AC. From this it is observed that the extra peaks present in 5%, 10%, 15%, 20% B$_4$C layers are related to reinforcement B$_4$C and coated nickel. There are no impurities observed in all the XRD graphs.

4.3. Wear test analysis

Using Taguchi’s L36 orthogonal design and abrasion wear test the wear rates are obtained and are show in table 5. The wear test is conducted by setting the controllable variable shown in table 5 using pin on disc apparatus and the wear rate is calculated using equation (3). For each run two trails have conducted and the average value of wear rate is listed in table 5. From the table 5 it is evident that the least value of wear rate is observed for run 9, with the controlled variables of B$_4$C without nickel, 25 microns of particle size, specimens having 5 layers, load of 30 N and a speed of 750 rpm. Highest value of wear rate is observed for run 22 for which the test conditions are B$_4$C with Nickel coating, 25 micron particle size, specimen with 2 layers, with load of 20 N on the pin and the rotational speed of 1000 rpm. The mean and S/N ratio plots for wear rate are generated using MINITAB software tool. The response for the wear test is to minimize the wear rate, therefore the criteria for S/N ratio is smaller is the best. Equation (5) is used to evaluate the S/N of each run. Figure 16 shows the main effect plot for S/N ratio considering data mean, higher the value of S/N ratio indicates the particular variable at the particular level has most influencing the response [31]. From the figure it is observed that FGM specimens without Nickel coated B$_4$C and with nickel coating B$_4$C have the variation of S/N of 4.5%. For the second factor, particle size condition there exist a variation of 1.6% S/N ratio. As the particle size decreases the mechanical and tribological
properties are increases [32]. The controllable factor, number of layers is also having highest variation of S/N ratio of about 5%. More number of layers leads to the more number of separating boundaries of different gradients, which gives more strength to the FGM component [33].

\[
\frac{S}{N} \text{ ratio } = -10 \log \left( \sum \frac{y^2}{n} \right)
\]  

Figure 17 shows the main effect plots of data means which is the direct reading of variation of wear rate with respect to different controllable factors [34]. From the figure it is evident that B₄C particles without nickel coating is giving less wear rate compare to B₄C particles with nickel coating. For the second factor particle size 50 microns give less wear rate compare to particle size of 25 microns. FGM specimen with five layers shows less

![Table 4. Reliability of Exp. densities with rule of mixture.](image)

| FGM specimen (code) | Experimental density (gm/c.c.) | Theoretical density (gm/c.c.) | Error (%) |
|---------------------|--------------------------------|-----------------------------|----------|
| 2 layered FGM 612(without Ni) | 2.556 | 2.69921 | 5.305 |
| 622 (with Ni) | 2.623 | 2.69921 | 2.823 |
| 3 layered FGM 613(without Ni) | 2.326 | 2.69615 | 13.72 |
| 623 (with Ni) | 2.432 | 2.69615 | 9.797 |
| 5 layered FGM 615 (without Ni) | 2.624 | 2.68849 | 2.398 |
| 625 (with Ni) | 2.712 | 2.68849 | −0.87 |

![Table 5. Wear test response table for L36 orthogonal array.](image)

| Run | Particulate condition | Particulate size (µm) | Number of layers | Load(N) | Track diameter(mm) | Disc speed(rpm) | Wear rate (mm³/m) |
|-----|----------------------|-----------------------|-----------------|---------|--------------------|----------------|------------------|
| 1   | B₄C                  | 25                    | 2               | 10      | 60                 | 500            | 0.00548          |
| 2   | B₄C                  | 25                    | 3               | 20      | 80                 | 750            | 0.004236         |
| 3   | B₄C                  | 25                    | 5               | 30      | 100                | 1000           | 0.003066         |
| 4   | B₄C                  | 25                    | 2               | 10      | 60                 | 500            | 0.004527         |
| 5   | B₄C                  | 25                    | 3               | 20      | 80                 | 750            | 0.003366         |
| 6   | B₄C                  | 25                    | 5               | 30      | 100                | 1000           | 0.002918         |
| 7   | B₄C                  | 25                    | 2               | 10      | 80                 | 1000           | 0.004302         |
| 8   | B₄C                  | 25                    | 3               | 20      | 100                | 500            | 0.00443          |
| 9   | B₄C                  | 25                    | 5               | 30      | 60                 | 750            | 0.002712         |
| 10  | B₄C                  | 50                    | 2               | 10      | 100                | 750            | 0.003625         |
| 11  | B₄C                  | 50                    | 3               | 20      | 60                 | 1000           | 0.004712         |
| 12  | B₄C                  | 50                    | 5               | 30      | 80                 | 500            | 0.003926         |
| 13  | B₄C                  | 50                    | 2               | 20      | 100                | 500            | 0.004638         |
| 14  | B₄C                  | 50                    | 3               | 30      | 60                 | 750            | 0.003702         |
| 15  | B₄C                  | 50                    | 5               | 10      | 80                 | 1000           | 0.00315          |
| 16  | B₄C                  | 50                    | 2               | 20      | 100                | 750            | 0.004358         |
| 17  | B₄C                  | 50                    | 3               | 30      | 60                 | 1000           | 0.003334         |
| 18  | B₄C                  | 50                    | 5               | 10      | 80                 | 500            | 0.003282         |
| 19  | B₄C + Ni             | 25                    | 2               | 20      | 60                 | 1000           | 0.005463         |
| 20  | B₄C + Ni             | 25                    | 3               | 30      | 80                 | 500            | 0.005943         |
| 21  | B₄C + Ni             | 25                    | 5               | 10      | 100                | 750            | 0.004358         |
| 22  | B₄C + Ni             | 25                    | 2               | 20      | 80                 | 1000           | 0.006659         |
| 23  | B₄C + Ni             | 25                    | 3               | 30      | 100                | 500            | 0.005621         |
| 24  | B₄C + Ni             | 25                    | 5               | 10      | 60                 | 750            | 0.003992         |
| 25  | B₄C + Ni             | 25                    | 2               | 30      | 80                 | 500            | 0.006321         |
| 26  | B₄C + Ni             | 25                    | 3               | 10      | 100                | 750            | 0.004937         |
| 27  | B₄C + Ni             | 25                    | 5               | 20      | 60                 | 1000           | 0.004038         |
| 28  | B₄C + Ni             | 50                    | 2               | 30      | 80                 | 750            | 0.005031         |
| 29  | B₄C + Ni             | 50                    | 3               | 10      | 100                | 1000           | 0.004125         |
| 30  | B₄C + Ni             | 50                    | 5               | 20      | 60                 | 500            | 0.003769         |
| 31  | B₄C + Ni             | 50                    | 2               | 30      | 100                | 1000           | 0.005324         |
| 32  | B₄C + Ni             | 50                    | 3               | 10      | 60                 | 500            | 0.004962         |
| 33  | B₄C + Ni             | 50                    | 5               | 20      | 80                 | 750            | 0.004884         |
| 34  | B₄C + Ni             | 50                    | 2               | 30      | 60                 | 750            | 0.003956         |
| 35  | B₄C + Ni             | 50                    | 3               | 10      | 80                 | 1000           | 0.004263         |
| 36  | B₄C + Ni             | 50                    | 5               | 20      | 100                | 500            | 0.004898         |
wear rate compare to 3 layered and 2 layered FGM. Forth factor, load has a mixed effect on the wear rate. The basic mechanism of wear occurring in pin on disc experiment is adhesive type of wear due to plastic deformation of the matrix \[35\]. As the load increases from 10 N to 20 N, the frictional force between the test specimen and the disc increases, thereby the wear rate increases. Further increase in the load from 20 N to 30 N, the ceramic particles comes in contact with disc and restricts the wear for the pin. Opposite pattern is observed for the speed of the disc. At lower speed of 500 rpm, the wear rate is more. As the speed increased from 500 rpm to 750 rpm, the wear rate decreased due to the contact of B4C particles with the disc. As the speed increased from 750 rpm to 1000 rpm, the trend of wear rate is increasing, this is due to chopping of hard ceramic particles to the rotating disc. The controllable factor track diameter follows the same pattern as load.

Figure 18, shows the interaction effect of different factors on the wear rate. Only some of the factors have the interaction effect as the graph corresponding to different levels crosses with each other. As the interaction plot indicates factors AB, AD, AE, AF, BC, BD, BE, CB interaction plots are crossing with each other. The first column of the figure 18 shows the interaction effect of particle condition with respect to the change in the levels of other controllable variables. Since the slope of the lines in all cells are dropping down, it means that changing the level of particle condition from B4C to B4C with Nickel coating has negative effect on minimization of wear rate.
### Table 7. Analysis of variance for transformed response.

|                     | DF | Seq SS  | Contribution | Adj SS  | Adj MS  | F-Value | P-Value |
|---------------------|----|---------|--------------|---------|---------|---------|---------|
| Particle condition  | 1  | 0.51475 | 31.49%       | 0.51475 | 0.51475 | 39.08   | 0.000   |
| particle Size (microns) | 1  | 0.03048 | 1.86%        | 0.03048 | 0.03048 | 2.31    | 0.141   |
| Number of Layers    | 2  | 0.50036 | 30.61%       | 0.50036 | 0.25018 | 19.00   | 0.000   |
| Load (N)            | 2  | 0.05732 | 3.51%        | 0.05732 | 0.02866 | 2.18    | 0.135   |
| Track diameter (mm) | 2  | 0.03300 | 2.02%        | 0.03300 | 0.01650 | 1.25    | 0.303   |
| Speed of the Disc (rpm) | 2  | 0.16936 | 10.36%       | 0.16936 | 0.08468 | 6.43    | 0.006   |
| Error               | 25 | 0.32927 | 20.14%       | 0.32927 | 0.01317 |         |         |
| Lack-of-Fit         | 22 | 0.28337 | 17.34%       | 0.28337 | 0.01288 | 0.84    | 0.663   |
| Pure Error          | 3  | 0.04591 | 2.81%        | 0.04591 | 0.01530 |         |         |
| Total               | 35 | 1.63454 | 100.00%      |         |         |         |         |

### Model Summary for Transformed Response.

| S          | R-sq  | R-sq(adj) | PRESS     | R-sq(pred) |
|------------|-------|-----------|-----------|------------|
| 0.114765   | 79.86%| 71.80%    | 0.682780  | 58.23%     |
Figure 9. (a) SEM image of B₄C figure 9 (b) SEM image of B₄C with Nickel.

Figure 10. EDAX of B₄C particle after clinch.

Figure 11. EDAX of B₄C after Nickel coating.
Figure 12. XRD comparisons of \( \text{B}_4\text{C} \) and \( \text{B}_4\text{C} + \text{Ni} \).

Figure 13. (a) SEM of 5% \( \text{B}_4\text{C} \) FGM layer (b) SEM of 10% \( \text{B}_4\text{C} \) FGM layer. (c) SEM of 15% \( \text{B}_4\text{C} \) FGM layer (d) SEM of 20% \( \text{B}_4\text{C} \) FGM layer.
rate [36]. Second column of interaction plot is related to interaction effect of particle size with respect to the changes of other parameters. All the cells in the second column are showing increase in the slope of the lines. That is there is a positive effect on minimizing the wear rate if the particle size is changed from 25 microns to 50 microns. The third column shows the interaction effect of number of layers of the FGM with the other parameters. As observed from the third column there are interaction plots with particle condition and particle size. As the slope of the lines shows increase in slope, there is a positive effect on the response by changing the number of layers from two to five [37]. The forth column is related to load on the pin is showing mixed effect on the response as the slope of the line is first decreased when the load is changed from 10 N to 20 N and then the slope is increased when the load is changed from 20 N to 30 N. Same analysis can be applied to the other two variables that is track diameter and speed of the disc.

Since the experimentation is based on design of experiments, it is assumed that every observation is mutually independent and homoscedastic. Observations should give different unknown results. Normality check is inappropriate for this kind of data. For verifying statistical validity of data residuals of each experiment is plotted against wear rate. Zero residuals are expected from the observation, but due to uncontrolled variables the residuals are obtained in the observations. Normal probability curve is generated for the L36 observations and is shown in figure 19, from the figure it is observed that the residuals are distributed on both sides of the normal line, which is necessary condition for any experimental data. Most of the residual points obtained by the difference of predicted model and experimental values are closure to the zero value and spreading either side of the zero up to ± 0.005. The data points in the figure 19 are following the normality line which is evident that the predicted model is adequate and acceptable within the limits.

Figure 14. (a) XRD of Al 601AC. (b) XRD of Al601AC + B4C + Ni.

Figure 15. XRD of all layers of the specimens.
Table 6 shows the response table for signal to noise ratio of wear rate. The delta values in the table are obtained as the difference between maximum to minimum value of S/N with different levels of each factor. Rank 1 is for number of layers in the FGM for which the delta value is 2.30 which is the maximum compared to other variables. The sequence of factors influence on the response (wear rate) based on S/N ratio is number of layers (1), Particle Condition (2), speed of the disc (3), Load acting on the specimen (4), Track diameter (5) and Particle size (6).

Table 7, shows the Analysis of Variance for the response. F values which are the ratio of adjusted mean square of the sample and population are observed from the table. If the value of F is closure to 1, null hypothesis is
accepted and is rejected with F values farther from 1. Null hypothesis is that all the samples are having equal means \[39\]. For the variables speed, Matrix, track diameter, coating condition and number of layers are having larger F value. Therefore the observations by the variation of levels of these variables are statistically significant.

**Figure 18.** Interaction plot for S/N ratios.

**Figure 19.** Normal Probability plot for wear rate.
Generally for MINITAB software, F values are calculated for confidence level of 95% and 5% of significance level [40]. That is there is a significant variation of means of the observations from the mean of population if its P-value is less than 0.05. From the table 6, it is observed that the P-value for the variables Particle condition (A),...
Number of layers (B), Speed of the Disc (C) is less than 0.05. Therefore these factors are most significant factors on the wear rate. The above Analysis of Variance is run for the linear model, i.e., it includes only main effect. The table includes the lack of fit condition with the F-value of 0.84 and P-value of 0.663. According to the general concept of lack of fit for the linear model, if the P value is less than the F-value, the model is accepted.

4.4. Worn surface analysis

Figures 20(a) to (f) shows the SEM images of worn surfaces for the scale of 200 μm. The left side figures that is 20(a), 20(c) and 20(e) are related to 2 layered, 3 layered and 5 layered worn surfaces (612, 613 and 615 specimen codes) of specimens without nickel coating and the right side figures that 20(b), 20(d) and 20(f) are related to 2 layered, 3 layered and 5 layered worn surfaces of specimens with nickel coating (622, 623 and 625). The specimens with B4C without nickel shows deep scratches (plough), removal of material in the form of abrasion, plastic deformation due to high stresses created between specimen and the disc. Deep scratches in these specimens are due to the weak bond between the Al601 AC alloying element and B4C particles.

In the right side figures i.e. 20(b), (d), (f) are showing the SEM images of specimens reinforced with nickel coated B4C. From the figures it is observed that the scratches are narrow and no abrasion takes place. This is due to the decrease in porosity and metallic bond between Al 601 AC and Nickel that is deposited on the surface of boron carbide particles.

Figure 21 shows the EDAX of worn out surface of Al601 AC, which includes all the alloying elements Mg, Si, Cu and a small amount of Zn and Fe.

Figures 22–24 shows the EDAX of worn surface of the 2 layered, 3 layered and 5 layered specimens. Selective area for the element analysis is shown in the red colored rectangular box. From the figures it is evident that the all the alloying elements including B4C and Nickel are present each area. And small amount of Fe in this area may be the debris of disc surface due to abrasion of hard B4C particles on rotating steel disc.
A linear regression equation is generated by running the regression analysis, and is given in equation (6). Since the objective function of this test is to minimize the wear rate, the parameters in the equation with the negative coefficient have the minimization effect on the response.

\[
\text{Wear rate}(\text{mm}^3/\text{m}) = 0.004726 + 0.0 \text{ Particle condition}_B + 0.001032 \text{ Particle condition}_C + 0.0 \text{ Particle size (microns)}_{25} - 0.000346 \text{ Particle size (microns)}_{50} + 0.0 \text{ Number of Layers}_2 - 0.000488 \text{ Number of Layers}_3 - 0.001224 \text{ Number of Layers}_5 + 0.0 \text{ Load (N)}_{10} + 0.000371 \text{ Load (N)}_{20} + 0.000088 \text{ Load (N)}_{30} + 0.0 \text{ Track diameter (mm)}_{60} + 0.000376 \text{ Track diameter (mm)}_{80} + 0.000121 \text{ Track diameter (mm)}_{100} + 0.0 \text{ Speed of the Disc (rpm)}_{500} - 0.000720 \text{ Speed of the Disc (rpm)}_{750} - 0.000520 \text{ Speed of the Disc (rpm)}_{1000}
\]

The predicted regression model developed is tested by the confirmation tests, which includes the experimental setup other than the experimental design given by L36 OA. The experimental results obtained by conducting the dry wear test using the selective parameters are compared with the values of wear obtained by the regression model. Table 8 shows the confirmation test experimental design along with the observed wear and predicted wear. From the comparative results it observed that there exists an error 7% between the experimental and predicted values. Therefore the regression model is accepted by 93% confident level. But for the run 1, that is with the parameters B4C particle condition, particle size of 25 \( \mu \text{m} \), 2 layered FGM, load 20 N, track diameter 60 mm, and the disk speed of 500 rpm, the error between the experimental wear and predicted wear is about 13%. The range of errors of the predicted wear model is lying in between 3% to 13%, which is not acceptable for 95% of confident level. The model has to modify with the quadratic terms of the parameters [41].

![Figure 23. EDAX of 3 layered FGM.](image)

| Element | Weight% | Atomic% |
|---------|---------|---------|
| B       | 1.90    | 0.43    |
| C       | 3.83    | 0.43    |
| Fe      | 0.21    | 0.10    |
| Ni      | 0.95    | 0.02    |
| Cu      | 0.44    | 0.10    |
| Zn      | 0.43    | 0.17    |
| Mg      | 1.76    | 1.07    |
| S       | 0.52    | 0.49    |

![Figure 24. EDAX of 5 layered FGM.](image)

![Image of EDAX graphs](image)
Table 8. Confirmation test results.

| Exp. No | Particle condition | Particle size (μm) | Number of layers | Load (N) | Track diameter (mm) | Speed of the disc (rpm) | Expt.l wear | Predicted wear | Error  |
|---------|--------------------|--------------------|------------------|----------|--------------------|------------------------|-------------|---------------|-------|
| 1.      | B₄C                | 25                 | 3                | 20       | 100                | 1000                   | 0.003278    | 0.003295     | −5%   |
| 2.      | B₄C + Ni           | 25                 | 2                | 20       | 100                | 1000                   | 0.006376    | 0.006335     | +6.5% |
| 3.      | B₄C + Ni           | 50                 | 5                | 20       | 100                | 1000                   | 0.001960    | 0.001971     | −5.6% |
test is also evident that for the particle size of 50 microns and FGM specimen with five layers shows the minimum wear rate under the constant level of other parameters.

5. Conclusion

601AC/B₄C functionally graded materials with and without nickel coating to the particulate is successfully fabricated using powder metallurgy technique. Controlled variables Number of layers (2, 3 and 5), particulate size (25 μm and 50 μm), Load (10 N, 20 N and 30 N), track diameter (60 mm, 80 mm 100 mm), and the speed of the disc (500, 750 and 1000 rpm) are selected to conduct dry sliding wear test using pin on disc apparatus. Taguchi’s L36 orthogonal array is selected to conduct the experiments. From the analysis of variance to response wear rate, the following conclusion are derived,

- Particle condition that is B₄C with coating and without nickel coating is the most influencing factor to reduce the wear rate with the contribution of 31.49%.
- Number of layers used to FGM also significant to the wear rate, 5 layered FGM gives less wear rate compared to 2 layered FGM.
- Speed of the disc has a mixed effect on the wear rate i.e., for the less speed, wear rate is less, increase in the speed up to 750 rpm leads to increase in wear rate, but further increase in the speed to 1000 rpm, wear rate is in decreasing trend. Speed of the disc is also a significant factor with the %contribution of 10.36.
- Linear Regression equation is developed which has the 13.5% error between predicted and confirmation values.
- A quadratic Regression equation is proposed to predict the wear rate.
- Worn surface analysis is done with optical microscope observed improve wettability of Nickel coated B₄C particles compared to B₄C without Nickel coating.

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