Study on formability and strain hardening index: influence of particle size of boron carbide (B₄C) in magnesium matrix composites fabricated by powder metallurgy technique

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Abstract

In the present investigation, Magnesium composites have been fabricated with boron carbide (B₄C) as reinforcement by powder metallurgical technique. Two different particle sizes—micro and nano B₄C particles with weight percentage of 0%, 5% and 10% has been studied. The green compacts were prepared by cold pressing and then sintering the specimens before being subjected to cold upsetting under triaxial stress state condition in order to study the phenomenon of workability and instantaneous strain hardening index. Powder characterizations are discussed using x-ray Diffraction peaks, Scanning Electron Microscope images and Energy Dispersive Spectrum analysis. Cold upsetting has been preferred to investigate the performance of the composites. The values of formability stress index factor ($\beta_\sigma$), various stress ratio ($\sigma_{th}/\sigma_{eff}$, $\sigma_m/\sigma_{eff}$ and $\sigma_z/\sigma_m$) parameters and instantaneous strain hardening index ($n_i$) are observed for increase in % of B₄C particles and its sizes. The experimental results were analyzed pertaining to relative density. The results reveal that Mg-10% nano B₄C composite has higher relative density, formability stress index factor and hence high workability than the other composites. The addition of B₄C particles as reinforcement affects the strain hardening index due to geometric and work hardening of the composites.

1. Introduction

Metal Matrix Composites (MMCs) which comprise wide choice of materials were meant to achieve predominant properties than unreinforced monolithic metals have seen a lot of improvement for a long time because of their promising propelled properties [1]. Although the matrix may be of an alloy or a metal, mechanical properties being the primary objective can be improved by selecting the light structural metals for hybrid metal matrix composites and improvements has been made in the reinforcements used [2].

The lightest among metals—Magnesium and its alloys, which possess low density, high specific strength, modulus, stiffness, better castability and weldability, become the appealing material for applications in aerospace and automobile sectors and also machinability can be improved with the utilization of discontinuous particle reinforcements. The density of magnesium is relatively lower than aluminium and furthermore lower than steel [3–5]. Anyhow, Mg alloys have deficient high temperature strength and worst corrosion resistance which limits their applications that can be enhanced by reinforcing the particles in the magnesium matrix [6]. Generally different grades of Magnesium such as AZ31, AZ61, AZ91 and ZM21 are used as base metals and the reinforcements such as nitrides, carbides, oxides and borides can be used for preparing magnesium composites [7–9]. Emerging need for lightweight materials with particular properties catalysed considerable interest towards development of numerous high performance composite materials. Reinforcements usually comprise of...
particles or whiskers with even small volume fractions greatly improve the strength and stiffness of the composites [10].

Among various reinforcements used with magnesium, boron carbide (B₄C) is the best because of its low density combined with high hardness, fracture toughness, superior elastic modulus and tremendous wear resistance [11, 12]. Because of its better properties, it has extensive applications in nuclear, automobile and aerospace sectors and high skilled applications such as light weight shields, fast-breeders, abrasive grit, and nozzles, cutting and grinding tools and so on [13–15].

The popularity of magnesium matrix composites in day-to-day life is delayed because of the cost, which mainly involves the cost of reinforcement particles and the method of fabrication [16]. Hence the potential of magnesium matrix composites with wide variety of reinforcing materials in advanced functional and structural materials needs attention in processing techniques and their features in order to select the suitable fabrication technique for that particular composite material. To fabricate magnesium matrix composites, three well-known processing techniques namely Powder Metallurgy (P/M), squeeze casting and stir casting are available [17]. The P/M technique is attractive among others because particle reinforced were evenly distributed in the matrix thereby regulating the microstructure and improving the structural and mechanical properties [18].

Al–SiC composites subjected to mechanical, machinability and metallurgical studies reveals that reducing the size of the particle reinforcement increases the life in low cycle fatigue because of cyclic hardening [19]. Relative density of the composite materials increases monotonically with pressure [20]. Cyclic stress response of the composite materials relies on the selection of reinforcement’s weight percentage and its particle size. Al–SiC composites subjected to upsetting test reveal that the formability of the composites was better compared to pure aluminium. Decreasing the aspect ratio of the composites gives better formability stress index rate because of the high densification [21].

Workability is defined as the capability of the composite material to resist the deformation sustaining the interior stresses before the crack initiation takes place leading to failure during cold upsetting [22]. Studies on powder metallurgy composites for its workability behaviour [23] reveals the investigation of the influence of relative density with formability stress index (βσ) to describe the influence of the effective and mean stresses with the help of Kuhn-Downey and Whang-Kobayashi theories. Strain hardening is a process of permanent plastic deformation triggered by the phenomenon of slip then by the dislocations generated and its interactions. The behaviour of strain hardening of the sintered powder metallurgy composites Al–Al₂O₃ and Al–Fe during cold upsetting preformed under uniaxial, biaxial and triaxial stress state conditions was investigated by Narayanasamy et al [24–28]. Jabbari-Taleghani et al [29] investigated hot workability behaviour of AZ91 Mg alloy and reveals that it has high hardness (133 HV) and crystallite size around 150 nm. Zhou et al [30] observed the hot deformation behaviour of the stir casted Mg-SiC particulate composites and investigated with processing maps which has lots of applications in aerospace and automobile parts due to its good tensile and compressive strength.

Workability assesses the performance capacity to absorb the generation of internal stresses against the crack initiation and propagation leading to failure and furthermore its plastic deformation. Increasing the relative density increases the plastic deformation of the composites [31, 32]. Workability of the composites subjected to triaxial stress state of condition can be accessed from effective stress and mean stress whereas the mean stress can be calculated from hoop and axial stress values [33]. The workability relies upon the reinforcement particle size, its weight percentage in the matrix and also the aspect ratio of the composites. Further the relative density, stress-strain rate also influences the workability [34, 35]. The crack initiation relying on mean stress [36] was proved from the correlation between the triaxial stresses and its strain and formability stress index factor (βσ) also determined to analyse the influence of mean and effective stress (σm and σeff) [37].

Increasing the percentage by weight of particle reinforcements enhances the compressibility of the composites [38]. Selvakumar et al [22, 39] reveals that the workability of the composites subjected to triaxial stress state condition by analysing the influence of various stresses pertaining to relative density and concluded that increasing the percentage of particle reinforcements increases the workability because of the increase in relative density. Preforms that possess high workability have high relative density and low aspect ratio. Different tests like hardness, tensile etc, were conducted on the composites to investigate its mechanical behaviour. Reinforcement type and its composition play a vital role in the strain hardening of composite materials [40].

To the authors’ knowledge, literature related to workability during cold upsetting of Mg-B₄C nano composites subjected to triaxial stress state of condition by varying the size of the reinforcement particles and its weight percentage are not available. In this present investigation, Mg-B₄C preforms with different particle size and weight percentage has been made by cold upsetting and discussed the influence of varying the size of the particle reinforcement and its weight percentage on the behaviour of workability and instantaneous strain hardening subjected to triaxial stress state of condition. Relative density and its relationship with various stress ratios and its effect with particle size and weight percentage also been discussed.
2. Experimental details

2.1. Materials
In this investigation, the matrix is Magnesium (Mg) AZ91 alloy and it is reinforced with boron carbide (B₄C) of two different particle sizes about 60 microns and 38 nm respectively. The elemental details of both Mg and B₄C are given in tables 1 and 2 respectively. The Mg alloy powder has an average particle size of 70 microns. The weight percentage of B₄C powder is selected as 5% and 10% respectively and the preforms were prepared by powder metallurgy route.

2.2. Material characterization
Magnesium alloy (AZ91) and B₄C powder has been characterised for morphological study using Scanning Electron Microscope (SEM) images. Figure 1(a) shows the SEM image of Mg AZ91 alloy. It has ellipsoidal and spherical structure with particle size of around 70 μm. Figure 1(b) shows the SEM images of B₄C powder. It looks like a polygonal structure.

The crystal structure, phase identification and the presence of particle is measured and confirmed using x-ray Diffractometer (XRD) peaks. The x-rays was produced from anode material CuKα1 which has a wavelength of 1.540 60 Å to obtain the diffraction patterns of the Mg AZ91 alloy and B₄C powder. Figures 2(a) and (b) illustrates the XRD patterns and peaks of Mg AZ91 alloy and B₄C powder. The measurement conditions of the test have 2θ angle with scan angle from 10°−80° with step size of 0.017°. The average crystal size of nanoB₄C powder obtained from ball milling was calculated from Deybe-Scherrer equation as 38 nm. The crystal peaks are also identified from the XRD analysis. The XRD patterns were drawn between 2θ and intensity (arbitrary unit). The crystalline structure of the as received Mg alloy powder has been determined as shown in figure 2(a) which indicates the high peaks for particular intensity at 2θ values of 32.51°, 34.77°, 37.02°, 48.35°, 57.89°, 63.61°, 70.37° and 73.10° with crystal planes (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3) and (2 0 1) respectively. The lattice parameters was determined to be a = 3.1768 Å and c = 5.1781 Å which reasonably agrees with the standard JCPDS card number 89-5003 with a = 3.208 Å and c = 5.209 Å of hexagonal structure. Similarly, the crystalline structure of the as received B₄C powder has been determined from figure 2(b) which shows the high peaks for particular intensity at 2θ values of 19.73°, 22.10°, 23.53°, 31.96°, 34.99°, 37.82°, 39.18°,
53.50°, 61.79°, 63.68°, 64.63° and 66.79° with crystal planes (101), (003), (110), (104), (021), (113), (205), (303), (125), (018) and (220) respectively. The lattice parameters almostmatch with the standard JCPDS card number 35-0798 with a = 5.600 Å and c = 12.086 Å having a rhombohedral crystallographic structure. The element peaks and its chemical composition of Mg AZ91 alloy and B₄C powder are confirmed through Energy Dispersive Spectrometer (EDS) analysis. Figures 3(a) and (b) illustrates the elemental (EDS) analysis of Mg AZ91 alloy and B₄C powder. It shows the existence of Mg at an intensive signal of around 1.25 kev. Boron and carbon has an intensive signal at 0.2 kev and 0.30 kev respectively. Boron has higher elemental composition, hence it has highest peak than carbon.

2.3. Preparation of composites

2.3.1. Blending

The primary matrix (Mg) and as received reinforcement particles (B₄C) were blended using planetary ball mill which has 10 mm diameter tungsten carbide balls and hybridize each other to distribute the secondary phase particles homogenously on the matrix. Since Magnesium alloy is highly reactive with atmosphere, the ball milling was performed under protective argon atmosphere to prevent oxidation during the process and the powder was handled carefully during loading and unloading. In the present investigation, reinforcement is added in the weight percentage of 5% and 10% respectively. Each blending has been done at 150 rpm for two hours. Initially, the planetary ball mill was used with the ball to powder ratio of 20:1 at 200 rpm for 30 h to synthesise B₄C powder separately. Milling was done intermittently to overcome the frictional heat by using toluene as the process control agent. Finally after 30 h of milling, the size of the particles was measured and characterized using SEM and XRD. The final mean size of the particle is 38 nm. This B₄C powder particle is then blended with Mg as said above with the same weight percentage of 5% and 10% respectively.

Figure 2. (a) XRD results of Mg alloy powder. (b) XRD results of B₄C powder.

Figure 3. (a) EDS of Mg alloy powder. (b) EDS of B₄C powder.
2.3.2. Compaction
The blended powders were heated to 120 °C for 2 h in the furnace to eradicate the unstable matters present in it. Then the dried blended powder is filled into the die and then compacted by gradually applying the uniaxial compressive load in the hydraulic press up to a pressure of 550 MPa and then removed the green compact specimen safely from the die. Zinc stearate has been used for die wall lubrication before each run. The green compacted specimen was prepared with diameter of 10 mm and height 10 mm respectively for all the combinations.

2.3.3. Sintering
The green compacts are then subjected to sintering process. Sintering was done in a controlled atmosphere tubular furnace at the temperature of 520 °C with a dwell time of 1 h and allowed to get cooled inside the furnace itself. The sintered samples cannot be used directly for characterization study because of its hard surfaces. Hence the surfaces should be polished for microscopic analysis. The sintered composite specimens are shown in the figure 4.

2.4. Characterization of composites
To study about the characterization of composites, the sintered composite specimen end surfaces were cleaned and polished in succession with the SiC abrasive papers of grit sizes 600, 800 and 1000 respectively in the disc polishing machine to get a mirror-like surface finish. Then the polished composite specimens were etched as per the metallographic study and examined for its characterization.

The SEM images of the sintered composites with Mg – 5% B₄C and Mg – 10% B₄C shown in the figures 5(a) and (c) respectively illustrate the even distribution of reinforcement particles in the Magnesium matrix. The structure of the composites shows the mixture of flake shapes along with polygonal shapes of different sizes because of the clustering of the B₄C reinforcement particle with Mg matrix.

The EDS analysis of the same specimen’s is shown in the figures 5(b) and (d) respectively. From the images the presence of magnesium (Mg) and boron carbide (B₄C) particles are confirmed and shows that the elemental peaks of Mg occur at 1.25 keV, boron (B) and carbon (C) at 0.2 keV and 0.30 keV with respect to the cps/eV value. Also the Mg has highest peak than boron and carbon particles due to its high percentage weight than others. Increasing for 5% to 10%, the corresponding peaks of boron and carbon also increases in the composites.

X-ray diffraction analysis is also made on the composites to confirm the intensity peaks of the particles. Figures 5(e) and (f) confirms the presence of magnesium (Mg) with higher intensity peaks due to its high weight percentage. The intensity peaks of the B₄C particles increases by increasing its weight percentage (figures 5(e) and (f)). Hence the presence of Mg and B₄C particles were confirmed through XRD analysis and it is identified that increasing the weight percentage of B₄C particles the intensity peak increases. There are no other intermetallic compounds formed during sintering. This was clearly identified from the XRD analysis. This may be attributed to the controlled inert atmosphere maintained in the tubular furnace during sintering.

2.5. Experimentation
2.5.1. Cold upsetting
To study about the cold workability behaviour of the Mg-5% B₄C composites and Mg-10% B₄C composites, cold deformation test has been carried out. The workability may be defined as the capability of the P/M composite to endure the crack initiation during cold upsetting by means of distortion measurement [22, 37]. Initial specimen dimensions such as diameter (Dₒ), height (hₒ) and theoretical density of the fully dense material (ρₒth) are measured. By means of Archimedes principle, the initial density (ρₒ) of the composites is measured. Then the cold upsetting of the composites has been done in the universal testing machine with 1 MN capacity. Each specimen undergoes the compressive load increasing in the order of 0.01 MN during upsetting. The dimensional changes such as bulged diameter (Dₜ), contact diameter at the top (DₜC) and bottom (DₜCb),
specimenheight ($h_i$) and density of the composites after deformation ($\rho_f$) are measured. The incremental load is applied until the initial crack has been detected on the free surface of the specimen. The contact diameter ($D_C$) and the relative density ($R$) of the specimens are also calculated. The formability stress index factor ($\beta_\sigma$) is calculated from the triaxial hydrostatic and effective ($\sigma_h$ and $\sigma_{eff}$) stresses which defines the workability behaviour of the composites. The schematic illustration of an upset forming- before and after deformation has been explained in the figure 6 [41].

Figure 5. (a) SEM image of Mg-5%$B_4C$ composite. (b) EDS of Mg-5%$B_4C$ composite. (c) SEM image of Mg-10%$B_4C$ composite. (d) EDS of Mg-10%$B_4C$ composite. (e) XRD results of Mg-5%$B_4C$ composite. (f) XRD results of Mg-10%$B_4C$ composite.
3. Hypothetical investigation

3.1. Measurement of density
Density measurement is needed to study the workability of the composites. The rule of mixture has been used to find out the theoretical densities of the fabricated composites ($\rho_{th}$) using the equation (1).

$$\rho_{th} = [(\rho_{Mg} \times \text{wt}\%_{Mg}) + (\rho_{B4C} \times \text{wt}\%_{B4C})]$$

where, $\rho_{Mg}$, $\rho_{B4C}$ refers to the theoretical densities of the completely dense material, magnesium (Mg) and boron carbide (B$_4$C) respectively in terms of g/cc and wt% Mg, wt% B$_4$C refers to the weight percentage of magnesium (Mg) and boron carbide (B$_4$C).

In keeping with Archimedes principle, the densities of the specimen before ($\rho_o$) and after ($\rho_f$) deformation are measured as per the ASTM: B962-13 test procedure using a high precision digital balance. The relative density ($R$) is defined as the ratio of the density of the deformed composite ($\rho_f$) to its true density ($\rho_{th}$) and it can be computed through the equation (2).

$$R = \frac{\rho_f}{\rho_{th}}$$

3.2. Triaxial stress state
Specimens with composition of Mg-5%B$_4$C and Mg-10%B$_4$C are tried to undergo normal axial stresses acting in three mutually perpendicular directions to cause volumetric deformation in triaxial state. In case of uniaxial stress state, only one axial stress acts and all other stresses are zero whereas in biaxial stress state, two axial stresses act in two directions and the remaining stresses are zero due to the deformation of the shape. In cold upsetting process, uniaxial as well as biaxial stress conditions cannot be applied since they influence the mechanical flow characteristics of the composites. Therefore, the triaxial stress state conditions are used to study the properties of the composites.

The contact area of the deformed composites after upsetting ($A_C$) can be calculated using the formula as given in equation (3).

$$A_C = \frac{\pi D_C^2}{4}$$

where composites’ contact diameter, $D_C$ can be calculated using the formula as given in equation (4).

$$D_C = \frac{(D_{CT} + D_{CB})}{2}$$

where, $D_{CT}$ and $D_{CB}$ refers to the contact diameter at the top and bottom of the composites respectively after cold upsetting.

3.2.1. Stresses and strains referred to triaxial stress state
The several stresses, for example, true axial stress ($\sigma_z$), effective stress ($\sigma_{eff}$), true hoop stress ($\sigma_\theta$) and mean stress ($\sigma_m$) can be calculated to determine the workability behaviour of the prepared composites. True axial stress is direct stress acting on the specimen caused by the application of the axial load which makes deformation and can be expressed as given in equation (5).
The three types of strains true axial strain ($\varepsilon_{\alpha}$), true hoop strain ($\varepsilon_{\theta}$) and conventional hoop strain ($\varepsilon'_{\theta}$) are essential for theoretical investigations of workability studies. The strain calculations are followed as per the standard [44].

The true axial strain ($\varepsilon_{\alpha}$) represents the deformation of composites caused by axial stress. In case of forming of cylindrical specimen, $\varepsilon_{\alpha}$ was calculated from the following expression.

$$\varepsilon_{\alpha} = \ln \left[ \frac{H_o}{H_f} \right]$$

where, $H_o$ and $H_f$ are the height of specimen before and after deformation respectively.

The true hoop strain ($\varepsilon_{\theta}$) caused by hoop stress is defined as the ratio of change in diameter of cylindrical specimen to actual diameter. The formula of true hoop strain ($\varepsilon_{\theta}$) for a specimen is given in equation (7) as specified by Narayanasamy et al [25].

$$\varepsilon_{\theta} = \ln \left[ \frac{D_c}{D_o} \right]$$

Conventional hoop strain ($\varepsilon'_{\theta}$) is calculated from the equation (8) as given below:

$$\varepsilon'_{\theta} = \ln \left( \frac{2(D_h^2 + D_c^2)}{3D_h^2} \right)$$

where $D_o$ and $D_c$ are the initial and average contact diameter of the composites and $D_h$ is the bulged diameter of composites after deformation.

The hoop stress ($\sigma_{\theta}$) can be calculated from the Poisson’s ratio or stress-strain increment ($\alpha$), Poisson’s ratio ($\alpha$) is the ratio of the changes in hoop strain to the corresponding axial strain [42]. Form the Poisson’s ratio ($\alpha$), the hoop stress ($\sigma_{\theta}$) can be obtained from the equation (9) [45].

$$\alpha = \frac{d\varepsilon_{\theta}}{d\varepsilon_{\alpha}} = \frac{(2 + R^2)\sigma_{\alpha} - R^2(\varepsilon_{\alpha} + 2\sigma_{\theta})}{(2 + R^2)\varepsilon_{\alpha} - R^2(\varepsilon_{\alpha} + 2\sigma_{\theta})}$$

where, $R$, $\sigma_{\alpha}$ and $\sigma_{\theta}$ refers to the relative density, true axial stress and hoop stress respectively.

Hoop stress ($\sigma_{\theta}$) also called as circumferential stress acting along the lateral surface area of composites will typically be greater than the true axial stress ($\sigma_{\alpha}$). By calculating Poisson’s ratio ($\alpha$), hoop stress ($\sigma_{\theta}$), can be obtained from the expression as given in equation (10).

$$\sigma_{\theta} = \frac{(2\sigma_{\alpha} + R^2)}{(2 - R^2 + 2R^2\alpha)} \varepsilon_{\alpha}$$

The mean stress ($\sigma_m$) is the mean of three mutually perpendicular axial principal stresses. The mean stress ($\sigma_m$) formulae for a cylindrical specimen forming is given in equation (11).

$$\sigma_m = \frac{(\sigma_{\alpha} + \sigma_{\theta} + \sigma_f)}{3}$$

Since the axial load applied during cold upsetting is compressive, the true axial stress, $\sigma_{\alpha}$ and the other two stresses hoop ($\sigma_{\theta}$) and radial ($\sigma_f$) will be of compressive and tensile in nature respectively. Narayanasamy et al [45] investigated that hoop stress ($\sigma_{\theta}$) and radial stress ($\sigma_f$) are identical for the specimens subjected to axisymmetric load under triaxial stress state of condition during cold upsetting. Thus the equation (11) becomes

$$\sigma_m = \frac{(\sigma_{\alpha} + 2\sigma_{\theta})}{3}$$

During yielding, the stress increases gradually to a critical point, effective stress ($\sigma_{eff}$). The expression for effective stress in case of cylindrical preforms [44] is given in the equation (13).

$$\sigma_{eff} = \left[ \frac{\sigma_{\alpha}^2 + 2\sigma_{\theta}^2 - R^2(\sigma_{\alpha}^2 + 2\sigma_{\theta} \sigma_{\theta})}{2R^2 - 1} \right]^{0.5}$$

3.3. Formability stress index factor ($\beta_{\sigma}$)

Formability is the capability of any composite materials to form or deform without failure. The formability stress index factor ($\beta_{\sigma}$) depends on the influence of mean and effective stresses during cold upsetting of the composites. Vujovic et al [46] proposed the expression for formability stress index factor ($\beta_{\sigma}$), as given in equation (14):
\[ \beta_n = \frac{3\sigma_m}{\sigma_{eff}} \]  

(14)

### 3.4. Instantaneous strain-hardening index \((n_i)\)

The strain-hardening index \((n)\) is calculated from the conventional Ludwik equation

\[ \sigma = K \varepsilon^n \]  

(15)

where \(K\), \(\sigma\), and \(\varepsilon\) refer to the strength coefficient, true effective stress and strain respectively. In this work, this index is calculated by rewriting the above equation (15) as shown in the equation (16), keeping that the successive compressive loads are specified as \((1, 2, 3 \ldots (m-1), m)\) [32]. This helps to calculate the strain-hardening parameters under triaxial stress state conditions for the prepared composites.

\[ n_i = \ln \left( \frac{\sigma_m}{\sigma_{m-1}} \right) = \ln \left( \frac{\varepsilon_m}{\varepsilon_{m-1}} \right) \]  

(16)

### 4. Results and discussion

#### 4.1. Influence of cold deformation test

The outcomes of the Mg alloy, Mg-5% micro B\(_4\)C, Mg-10% micro B\(_4\)C, Mg-5% nano B\(_4\)C and Mg-10% nano B\(_4\)C composites subjected to cold deformation was discussed by plotting various graphs for the values obtained through parameters theoretically evaluated [43, 44].

**4.1.1. Stress variations pertaining to axial strain**

Graphs have been plotted as shown in the figures 7(a)–(e) for the Mg alloy, Mg-5% micro B\(_4\)C, Mg-10% micro B\(_4\)C, Mg-5% nano B\(_4\)C and Mg-10% nano B\(_4\)C composites with an aspect ratio of one subjected to cold upsetting at triaxial stress state condition to study the various tri-axial stress behaviours like true axial \((\sigma_z)\), hoop \((\sigma_\theta)\), mean \((\sigma_m)\) and effective \((\sigma_{eff})\) stresses pertaining to axial strain \((\varepsilon_z)\). The true axial stress \((\sigma_z)\), compressive in nature during cold upsetting is negative whereas other stresses are tensile and hence positive.

During cold upsetting with gradual application of load, all the stresses gradually increases with respect to strain because of the resistance against deformation. With increase in load, the stress increases because of the growing relative density \((R)\) value by reducing porosity. The stress-strain curve increases gradually till the crack initiation on the composites. By adding the micro B\(_4\)C and nano B\(_4\)C particles to the magnesium alloy, the relative density of the composites increases owing to the enhanced load transfer capability of the reinforcement to the matrix and hence decreases the porosity.

It has been observed from the results that, addition of reinforcement B\(_4\)C particle increases the true axial stress \((\sigma_z)\) and the stress further increases by increasing the percentage of B\(_4\)C. As the size of the reinforcement B\(_4\)C particle gets reduced, the true axial stress \((\sigma_z)\) and strain \((\varepsilon_z)\) increases due to the decrease in pore sizes by the reinforcement B\(_4\)C particles and its uniform and effective particle distribution which also increases the relative density under the same compacting pressure. The true axial stress \((\sigma_z)\) remains higher than the hoop stress \((\sigma_\theta)\). The mean stress \((\sigma_m)\) was minimum and the effective stress \((\sigma_{eff})\) was maximum because of the better densification. Hence among these composites the Mg-10% nano B\(_4\)C composite withstands more stresses for deformation. The maximum values of stresses \((\sigma_z, \sigma_m, \sigma_\theta\) and \(\sigma_{eff}\) for all the composites were given in the table 3.

**4.1.2. Influence of axial stress \((\sigma_z)\) pertaining to relative density \((R)\)**

The influence of axial stress \((\sigma_z)\) on relative density \((R)\) has been observed by plotting the graph for the Mg alloy, Mg-5% micro B\(_4\)C, Mg-10% micro B\(_4\)C, Mg-5% nano B\(_4\)C and Mg-10% nano B\(_4\)C composites with an aspect ratio of one as revealed in the figure 8. At the beginning of cold upsetting the relative density \((R)\) increases rapidly due to the closure of pores which will be high at that stage but the axial stress \((\sigma_z)\) is slowly increased because of low resistance against deformation. As the deformation progresses, the porosity reduces and hence the relative density \((R)\) is slowly increased but axial stress \((\sigma_z)\) begins to increase marginally. It continues till the initial crack was observed on the specimen.

Hence comparing the results of all these composites, the Mg-10% nano B\(_4\)C composite withstands high relative density and hence axial stress. This is because of the B\(_4\)C reinforcement, which reduces the porosity and increases the densification of the composites and therefore deformation requires high axial stress \((\sigma_z)\). The maximum values of relative density \((R)\) and true axial stress \((\sigma_z)\) for all the composites was given in the table 4.
4.1.3. Influence of formability stress index \((\beta_{\sigma})\) pertaining relative density \((R)\)

The relation among formability stress index \((\beta_{\sigma})\) and relative density \((R)\) for magnesium alloy and its composites containing 5% and 10% B\textsubscript{4}C particles of micro and nano sizes is depicted in figure 9. Table 5 shows the

### Table 3. Maximum values of stresses.

| S. no. | Stress (MPa)   | Mg alloy | Mg-5% micro B\textsubscript{4}C | Mg-10% micro B\textsubscript{4}C | Mg-5% nano B\textsubscript{4}C | Mg-10% nano B\textsubscript{4}C |
|--------|----------------|----------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|
| 1.     | Max. Axial stress | 265.73   | 308.79                        | 335.70                        | 321.18                        | 342.78                        |
| 2.     | Max. Hoop stress  | 224.86   | 279.16                        | 314.73                        | 309.63                        | 333.91                        |
| 3.     | Max. Mean stress  | 61.33    | 83.18                         | 97.92                         | 99.36                         | 108.35                        |
| 4.     | Max. Effective stress | 516.99   | 603.13                        | 660.70                        | 635.04                        | 680.27                        |

Figure 7. (a) Stress variations pertaining to true axial strain—Mg alloy. (b) Stress variations pertaining to true axial strain—Mg-5% micro B\textsubscript{4}C composite. (c) Stress variations pertaining to true axial strain—Mg-10% micro B\textsubscript{4}C composite. (d) Stress variations pertaining to true axial strain—Mg-5% nano B\textsubscript{4}C composite. (e) Stress variations pertaining to true axial strain—Mg-10% nano B\textsubscript{4}C composite.
maximum formability stress index ($\beta \sigma$) values for all composites. It has been observed that reinforcing the B$_4$C particles into the Mg alloy increases the relative density which in turn reduces the porosity. The same has been observed by reducing the size of the B$_4$C particles. Thus the formability stress index ($\beta \sigma$) increases uniformly with relative density ($R$) with rise in weight percentages of B$_4$C particles and also by reducing its particle sizes. Relative density increases the workability of composites. During cold upsetting, the relative density is increased because of the increase in density of the composites by reducing porosity. Results shows that Mg-10% nano B$_4$C composite has high relative density ($R$) and hence high formability stress index ($\beta \sigma$) than the other composites.
4.1.4. Influence of relative density ($R$) pertaining to axial strain ($\varepsilon_z$)
Figure 10 shows the relation between relative density ($R$) and axial strain ($\varepsilon_z$) for magnesium alloy and its composites containing 5% and 10% B$_4$C particles of micro and nano sizes by using parabolic curve fitting technique of second order polynomial equation. It has been observed that the relative density ($R$) increases due to the increasing axial strain ($\varepsilon_z$) for all the composites. Initially the relative density increases and becomes consistent with the development of axial strain ($\varepsilon_z$). The Mg-10% nano B$_4$C composite has higher axial strain ($\varepsilon_z$). For a particular axial strain ($\varepsilon_z$), the relative density increases with the addition of B$_4$C particles and further improved by reducing the particle sizes. The maximum value of axial strain ($\varepsilon_z$) for all the composites are given in the table 6.

| S. no. | Composites          | $\varepsilon_z$ |
|-------|---------------------|-----------------|
| 1     | Mg alloy            | 0.5816          |
| 2     | Mg-5% micro B$_4$C  | 0.6597          |
| 3     | Mg-10% micro B$_4$C | 0.7340          |
| 4     | Mg-5% nano B$_4$C  | 0.6616          |
| 5     | Mg-10% nano B$_4$C | 0.7361          |

4.1.5. Impact of formability stress index ($\beta_\sigma$) pertaining to axial strain ($\varepsilon_z$)
Formability stress index ($\beta_\sigma$) graph is plotted against axial strain ($\varepsilon_z$) as shown in the figure 11. for magnesium alloy and its composites containing 5% and 10% B$_4$C particles of micro and nano sizes by using parabolic curve fitting technique of second order polynomial equation. It has been observed that with increasing axial strain ($\varepsilon_z$), relative density ($R$) increases and hence formability stress index ($\beta_\sigma$) increases for all the composites, which is attributed due to the reduction of pores during cold upsetting. The highest values of the formability stress index ($\beta_\sigma$) attained for the Mg alloy, Mg-5% micro B$_4$C, Mg-10% micro B$_4$C, Mg-5% nano B$_4$C and Mg-10% nano B$_4$C composites are given in the table 5 which shows that Mg-10% nano B$_4$C composite has the highest value of 0.4719.

| S. no. | Composites          | $\beta_\sigma$ |
|-------|---------------------|----------------|
| 1     | Mg alloy            | 0.3559         |
| 2     | Mg-5% micro B$_4$C  | 0.4245         |
| 3     | Mg-10% micro B$_4$C | 0.4493         |
| 4     | Mg-5% nano B$_4$C  | 0.4524         |
| 5     | Mg-10% nano B$_4$C | 0.4719         |
4.1.6. Influence of stress ratio parameters pertaining to relative density (R)

The stress ratio parameters ($\sigma_\theta/\sigma_{\text{eff}}$, $\sigma_m/\sigma_{\text{eff}}$ & $\sigma_z/\sigma_m$) have been drawn against the relative density (R) for magnesium alloy and its composites containing 5% and 10% B$_4$C particles of micro and nano sizes by using parabolic curve fitting technique of second order polynomial equation as shown in the figures 12–14. The result shows that the mean stress ($\sigma_m$) and hoop stress ($\sigma_\theta$) rises through the increase in relative density (R) as compared to effective stress ($\sigma_{\text{eff}}$). With gradual increase in the load during cold upsetting, the resistance against deformation increases due to the volume required to close the pores gets reduced. Therefore bulging takes place with increase in $\sigma_\theta$ & $\sigma_m$ and hence the stress ratio parameters ($\sigma_\theta/\sigma_{\text{eff}}$ & $\sigma_m/\sigma_{\text{eff}}$) increase with increase in relative density (R). Since the relative density (R) is incremental, the stress ratio parameters ($\sigma_\theta/\sigma_{\text{eff}}$ & $\sigma_m/\sigma_{\text{eff}}$) increases compared with previous level which leads to formation of initial crack by damaging the pores on the composites. Composites with larger value of relative density yield the highest stress ratio parameters ($\sigma_\theta/\sigma_{\text{eff}}$ & $\sigma_m/\sigma_{\text{eff}}$). The relative density (R) of Mg-10% nano B$_4$C composite is higher than all other composites for the particular stress ratio parameters.

On the other hand, when the stress ratio parameter of ($\sigma_z/\sigma_m$) is drawn pertaining to relative density (R), the behaviour has been reversed. This is due to the compressive nature of the true axial stress ($\sigma_z$). The maximum and minimum values of stress ratio parameters ($\sigma_\theta/\sigma_{\text{eff}}$ & $\sigma_m/\sigma_{\text{eff}}$ are given in the table 7.

4.1.7. Influence of instantaneous strain hardening index ($n_i$) pertaining to relative density (R)

Graph shown in figure 15 has been drawn for the instantaneous strain hardening index ($n_i$) pertaining to relative density (R) for magnesium alloy and its composites containing 5% and 10% B$_4$C particles of micro and nano sizes as it has been observed that the instantaneous strain hardening index ($n_i$) drops rapidly with increase in

![Figure 11. Influence of formability stress index ($\beta$) pertaining to axial strain ($\varepsilon_z$) for Mg alloy, Mg-5% micro B$_4$C, Mg-10% micro B$_4$C, Mg-5% nano B$_4$C and Mg-10% nano B$_4$C composites.](image1)

![Figure 12. Influence of stress ratio parameter ($\sigma_\theta/\sigma_{\text{eff}}$) pertaining to relative density (R) for Mg alloy, Mg-5% micro B$_4$C, Mg-10% micro B$_4$C, Mg-5% nano B$_4$C and Mg-10% nano B$_4$C composites.](image2)
relative density from 0.79 to 0.84 in case of Mg alloy. For Mg-5% micro $B_4C$ the drop takes place from the relative density 0.82 to 0.90 and for Mg-5% nano $B_4C$, it takes place from the relative density 0.86 to 0.93. Similarly for Mg-10% micro $B_4C$ the drop takes place from the relative density 0.85 to 0.91 and for Mg-10% nano $B_4C$, it takes place from the relative density 0.87 to 0.94. This is due to the phenomenon of pore closure during the load application. Hence the curve continues to decrease because of strain softening up to that relative density. Beyond that, with further increase in deformation the matrix work hardening increases and hence instantaneous strain hardening index $(n_i)$ begins to increase slightly. It continues to rise up until the flow softening occurs towards the

Table 7. The maximum values Stress ratio parameters $(\sigma_{\theta}/\sigma_{\theta})$ pertaining to relative density $(R)$ for Mg alloy, Mg-5% micro $B_4C$, Mg-10% micro $B_4C$, Mg-5% nano $B_4C$ and Mg-10% nano $B_4C$ composites.

| S. no. | Composites       | $\sigma_{\theta}/\sigma_{\theta}$ (Max. Value) | $\sigma_{\theta}/\sigma_{\theta}$ (Max. Value) | $\sigma_{\theta}/\sigma_{\theta}$ (Min. Value) |
|--------|------------------|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|
| 1.     | Mg alloy         | 0.4349                                        | 0.1186                                        | 4.3329                                        |
| 2.     | Mg-5% micro $B_4C$ | 0.4629                                       | 0.1379                                        | 3.7125                                        |
| 3.     | Mg-10% micro $B_4C$ | 0.4763                                       | 0.1482                                        | 3.4283                                        |
| 4.     | Mg-5% nano $B_4C$ | 0.4876                                        | 0.1565                                        | 3.2325                                        |
| 5.     | Mg-10% nano $B_4C$ | 0.4900                                        | 0.1593                                        | 3.1637                                        |
end of deformation because of the influence of both geometrical as well matrix work hardening. As compared to Mg alloy, instantaneous strain hardening index ($n_i$) increases with the inclusion of B$_4$C particles and further increases with the reduction of its particle size.

5. Conclusion

The experimental investigation of formability and strain hardening index of the Mg alloy, Mg-5% micro B$_4$C, Mg-10% micro B$_4$C, Mg-5% nano B$_4$C and Mg-10% nano B$_4$C composites has been studied using cold deformation test and the following conclusions were made:

- The powder characterization of the composites through SEM confirms the presence of B$_4$C and its bonding with Mg alloy and the XRD and EDS results further confirms the existence of both Mg and B$_4$C in the composites with different peaks.
- It is evident that increasing the weight % of B$_4$C reinforcement reduces the porosity of composites. Further reducing the B$_4$C reinforcement particle size increases the densification of the composites because of the better load distribution. This leads to the higher formability index value ($\beta_\sigma$).
- The results of the cold upsetting reveals that increasing the B$_4$C particles increases the relative density ($R$) due to the low porosity. Among all the composites, Mg-10% nano B$_4$C composite has high strength and formability ($\beta_\sigma$) behaviour than other composites.
- Stress ratio parameters ($\sigma_0/\sigma_{eff}$ & $\sigma_m/\sigma_{eff}$) for Mg-10% nano B$_4$C composite is higher than the other composites due to the better densification and low porosity and the stress ratio parameter ($\sigma_0/\sigma_m$) decreases for the composites compared to Mg alloy because of the higher mean stress ($\sigma_m$) combined with low porosity.
- As compared to Mg alloy, the inclusion of the B$_4$C particles increases the instantaneous strain hardening index ($n_i$). This parameter further increases with reduced particle size of B$_4$C reinforcement due to the better load distribution and high densification.

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