An investigation was carried out to find the workability behaviour of a Cu-TiB₂ composite under triaxial stress-state conditions. Initially, the TiB₂ powder was prepared by using a self-propagating high-temperature synthesis (SHS) technique and the same was added to a Cu matrix in order to make Cu-TiB₂ composites. Cylindrical preforms with three different TiB₂ weight percentages (2%, 4% and 6%) with aspect ratios of 0.50, 0.75 and 1 were prepared using a uniaxial load. Then the preforms were pressureless sintered in a tubular furnace with a continuous flow of pure argon gas at 950 °C for a period of 1 h. The cold upsetting test was carried out on the sintered specimens. The relationships between the various stresses, strains and the relative density were determined. The results for the various stress-ratio parameters, namely (σ_r/σ_m) and (σ_r/σ_eff), the formability stress index (β) under triaxial stress-state conditions were systematically analysed. The formability stress index was found to increase with the increase in preform fractional density and it decreased with the aspect ratios. This was because the preform contains more pores and the porous bed height is high. A statistical fitting method was performed on the curve drawn between the axial strain and the stress-formability index. The compacts with a higher value of the aspect ratio and the initial preform density showed a very high fracture strain.

Keywords: SHS, powder metallurgy, TiB₂, workability, relative density, fracture strain
fields. It has applications in rocket nose cones for atmospheric re-entry, ballistic armour, cathodes for Hall-Heroult cells, crucibles for molten metals, metal evaporation boats, and as a coating on cutting tools.\textsuperscript{9–10} It is widely used as cutting-tool composites and wear-resistant parts.\textsuperscript{11} The TiB\textsubscript{2} powder is synthesized using the SHS method. The main feature of the SHS process is that, it utilizes the high energy released during the exothermic chemical reaction of the reactants to yield a variety of inorganic materials. Once the reactants are ignited by an external source, the reaction front propagates within the solid with a certain velocity to complete the chemical reaction.

The extent of deformation possible without failure is defined by the term “workability”. It is the ability of a material to withstand the induced internal stresses of forming before any failure occurs. It is the extent to which a material can be deformed in a specific metal working process without the initiation of cracks.\textsuperscript{12–14} Workability depends on both the material and the process parameters. The workability of dense material is better than with P/M material. The workability can be calculated by interpreting the value of hydrostatic stress and effective stress for a tri-axial state of compression, and the hydrostatic stress can be evaluated from the axial and hoop stresses. The evaluation of different stresses and the failure strain will reveal the workability limits of the P/M composites.\textsuperscript{15} M. Abdel-Rahman and E. Sheikh\textsuperscript{16} explored the effect of the relative density on the forming limit of P/M compacts during upsetting. J. J. Park et al.\textsuperscript{17} developed a constitutive relation involving the Poisson’s ratio, relative density and flow stress to predict the plastic deformation behaviour of porous metals. A mathematical equation for the calculation of the flow stress in the case of a simple upsetting of P/M sintered preforms was proposed by R. Narayanasamy et al.\textsuperscript{18} Furthermore, the authors developed a new equation for the determination of the hydrostatic stress in the case of the simple upsetting of sintered P/M compacts. Equations for the determination of the flow stress and the hydrostatic stress depending upon two factors, i.e., (i) the value of Poisson’s ratio and (ii) the relative density of the P/M preform in the case of the simple compression test were proposed in the literature.\textsuperscript{18} However, copper-based materials are hard to form as they offer resistance to the forming load due to the formation of intermetallic compounds. Thus, it is essential to investigate the deformation behaviour of the Cu-TiB\textsubscript{2}–based composite developed in the present work.

2 EXPERIMENTAL DETAILS

Cu-TiB\textsubscript{2} composite sintered preforms were selected in order to provide a reasonably wide range of study, namely, workability and work-hardening behaviour during cold upset operation. The commercially available copper powder was obtained from Alfa Aeser and the TiB\textsubscript{2} powder was produced using self-propagating high-temperature synthesis (SHS) in our lab, igniting the stoichiometric mixture of 20 g according to Equation (1), in a tubular furnace, maintaining an argon atmosphere. To investigate the particle size, shape and its distribution, copper, TiB\textsubscript{2} powders were studied using a scanning electron microscope (SEM) (Figure 1). The Cu-TiB\textsubscript{2} powders with different weight percentages of TiB\textsubscript{2}, namely, 2 %, 4 % and 6 %, blend in a mortar mixer in order to obtain a homogeneous mixture.

The powders were compacted in a 25-ton manual pellet press with the closed die set assembly technique. Compacts of 15-mm diameter were prepared with aspect ratios of 0.50, 0.75 and 1. The aspect ratio is the ratio of the height to the diameter of the sample. The approxi-
mate initial preform density is 70% of the theoretical density. These densities were achieved after sintering. Then the preforms were sintered in a tubular furnace at a temperature of 950 °C for a period of 1 h. To avoid oxidation the preforms were heated in an inert argon atmosphere. After the sintering schedule, the compacts were cooled in the furnace itself. The sintered preforms were cleaned and the dimensional measurement was made before the deformation.

The upsetting tests (Figures 1a and 1b) were conducted on a hydraulic press having a capacity of 50 tons. Extreme care was taken to place the cylindrical specimen within the platens, concentric with the central axis of the hydraulic press (loading direction). Cylindrical preforms were cold upset between the flat platens. Each preform was subjected to an incremental compressive loading in steps until the appearance of visible cracks on the free surface.

Immediately after each incremental loading, the contact diameter at the top ($D_{CT}$), the contact diameter at the bottom ($D_{CB}$), the bulged diameter ($D_B$), the height of the preforms ($h$) and the density ($\rho$) were recorded. Before upsetting, the initial diameter ($D_i$), the initial height ($h_o$) and the initial preform density ($\rho_o$) of the specimens were measured. Moreover, the density measurements of the preforms were carried out using the Archimedes principle. Using the load, the dimensional parameters and density, the different true stresses (i.e., $\sigma_z$, $\sigma_\theta$, $\sigma_m$ and $\sigma_{\alpha}$) and the different true strains (i.e., $\varepsilon_z$ and $\varepsilon_\theta$) and the workability parameters ($\beta$) were determined using the expressions specified below for the triaxial stress-state condition.

For the present investigation, the TiB$_2$ powders were synthesized in-house as explained by the authors in a previous study. The mixture of titanium oxide (TiO$_2$), boric acid (H$_3$BO$_3$) and magnesium was taken as per the stoichiometric reaction (Equation 1). The powders were mixed in a mortar mixer for about 20 min. A mixture of 20 g was then taken in a stainless-steel boat and was kept in a tubular furnace (Systems control, Chennai). The complete process was carried out in a highly pure argon atmosphere in order to maintain an inert atmosphere. The furnace was then heated up to 800 °C with a constant heating rate. It was observed that the reaction was taking place with an explosive sound at an approximate temperature of 680 ± 15 °C. The furnace is then left to cool to room temperature.

$$
\text{TiO}_2 (s) + 2\text{H}_3\text{BO}_3 (s) + 5\text{Mg} (s) \rightarrow \text{TiB}_2 (s) + \text{MgO} (s) + 3\text{H}_2\text{O} (g) + \Delta H
$$

After cooling, the synthesized powder was taken out. It was observed that the reacted mixture is formed of black lumps, and some amount of white surface layer was seen on the lumps. The powder is then taken out and was crushed into fine powder before going to the leaching process, in order to make the leaching process effective. The leaching process was carried out in diluted HCl, with normality of 2 N. The solution was mixed.
with the crushed powder and heated up to 120 °C. The process was continued while the solution boils for about 10 min, and then the solution was separated using filter paper. The resulting powder, which was taken after the leaching process, was then dried in an oven for 1 h. The resulting powder is used in the present study.

The XRD patterns of the sample produced by the SHS process after leaching shows the presence of TiB₂ as major phase with TiO₂ as minor phase in Figure 2. The TEM image of the synthesized powder is shown in Figure 3. The TEM images show the formation of spherical and hexagonal TiB₂ particles.

The XRD pattern of pure Cu and Cu-6 (6 % of mass fractions of TiB₂) is shown in Figure 4. The pattern shows the presence of TiB₂ as small peaks and Cu as a major phase. This indicates there is no interaction between the Cu and TiB₂ during the pressureless sintering. It is because of the smaller weight percentage of TiB₂ in the Cu matrix.

The scanning electron microscope images of the Cu-TiB₂ samples are shown in Figures 5a to 5d. Pure Cu is shown in Figure 5a. Cu-2 (2 % of mass fractions of TiB₂), Cu-4 (4 % of mass fractions of TiB₂) and Cu-6 (6 % of mass fractions of TiB₂) are shown in Figures 5b to 5d, respectively. The SEM images reveal the surface morphology of the sintered samples. The images show the porosity, the distribution of the powder particles and the sintering behaviour.

3 THEORETICAL ANALYSIS

In the upsetting of P/M parts, the height decreases, the average density increases, and the various stresses increase. The expressions for the normal stress (σ₁), normal strain (ε₁), hoop stress (σ₃), hoop strain (ε₃), hydrostatic stress (σ₃), effective stress (σₑ), and effective strain (εₑ) were taken from N. Selvakumar et al. and Narayanasamy et al.

![Figure 6](image-url)

**Figure 6:** a) Relative density (R) versus axial strain (ε₁) for triaxial stress state condition, b) relative density (R) versus axial strain (ε₁) for triaxial stress-state condition (power-law curve-fitting results) and c) relative density (R) versus axial strain (ε₁) for triaxial stress-state condition (parabolic curve-fitting results)

![Slika 6](image-url)

**Slika 6:** a) Odvodnost relativne gostote (R) od osne napetosti (ε₁) pri trisnem napetostnem stanju, b) odvodnost relativne gostote (R) od osne napetosti (ε₁) pri trisnem napetostnem stanju (rezultati urejanja potenčne krivulje) in c) odvodnost relativne gostote (R) od osne napetosti (ε₁) pri pogoju trisnega napetostnega stanja (rezultati urejanja parabolične krivulje)
Triaxial Stress State Condition:

\[ \sigma = \frac{A}{B} \]  

\[ A = (2 + R^2) \sigma_0 - R^2 (\sigma_0 + 2 \sigma_0) \]  

\[ B = (2 + R^2) \sigma_0 - R^2 (\sigma_0 + 2 \sigma_0) \]  

Hoop stress, \( \sigma_0 = \left[ \frac{2 \alpha + R^2}{2 - R^2 + 2 R^2 \alpha} \right] \sigma_0 \)  

Hydrostatic stress, \( \sigma_m = \frac{\sigma_0 + 2 \sigma_0}{3} \)

Effective stress,  
\[ \sigma_{eff} = \left[ \sigma_0^2 + 2 \sigma_0^2 - R^2 (\sigma_0^2 + 2 \sigma_0 \sigma_m) \right]^{\frac{1}{2}} \]

Relative density, \( R = \frac{\rho_f}{\rho_0} \)  

\( \rho_f \) is the final density of the compact after deformation and \( \rho_0 \) is the theoretical density of the compact.

Formability Stress Index, \( \beta = \frac{3 \rho_m}{\sigma_{eff}} \)

**4 RESULTS AND DISCUSSION**

Figures 6a to 6c show the relationship between the relative densities attained and the axial strain for the Cu-TiB₂ preforms. The compaction load was kept constant for all the samples compacted with different proportions of TiB₂ and copper. It is observed that the initial densification achieved is better for the preforms prepared with copper alone and its relative density is around 75%. This reduces as the percentage of TiB₂ addition increases. The strain to failure was found to be low for the preforms with 6% TiB₂ and it was found to increase as the TiB₂ decreases. Moreover, it can also be inferred that the strain to failure is low for the low initial relative densities.

A statistical curve-fitting technique was adopted for the drawn curves and the prediction equation developed from the curves was checked for its applicability by comparing the correlation coefficient \( R^2 \) values. These values can be used for modelling purposes and can also serve as prediction equations. In the present study, two
different curve-fitting techniques, i.e., the power law and parabolic curve fitting, were used.

As the aspect ratio increases, the fracture strain increases (Figure 7). The fracture strain decreases with the addition of TiB₂. Irrespective of the TiB₂ content, the fracture strain is less for 0.5 aspect ratio preforms. The decrease in the fracture strain indicates that the composite has attained a higher strength level with the addition of TiB₂, with less sacrifice in the strain values. The addition of TiB₂ to a preform with an aspect ratio of 1 has increased the strength with very little loss of fracture strain.

Figures 8a to 8c show the plot drawn between the axial strain and the formability stress index (b). A statistical fit is made using the polynomial function and the power-law function. It is found that the power law related the parameters with higher accuracy. The addition of TiB₂ decreased the strain further.

For preforms with a higher aspect ratio and a lower initial relative density, the formability stress-index value moves closer to the minimum value. The reason is that this preform contains more pores and the porous bed height is larger or greater. The increase in relative density with increasing deformation is less in this case compared to lower aspect ratio preform. A parabolic curve-fitting technique was applied to relate the formability stress index and the axial strain for a varying aspect ratio and relative density. The polynomial equations obtained for each aspect ratio and relative density along with its regression co-efficient value are presented in Table 1, where it is observed that the constant value decreases with a decreasing amount of relative density, irrespective of the aspect ratio.
Figures 9a to 9c give the plot of the relative density with the stress ratio. The change of density along the axial and hoop stress directions was analysed. Figure 8a shows the variation of the relative density with the mean stress ratio. It is found that Cu with 4% TiB₂ and an aspect ratio of 0.5 yields high density values with a high load-bearing capacity. The same was true for the axial stress ratio and the hoop stress ratio. The hoop stress is responsible for the initiation of cracks in the preforms. Thus, it is clear that the addition of 4% TiB₂ improves the density of the preforms and postpones the initiation of cracks. As the relative density increases the stress ratio parameter also increases.

It was found that the relative density increases as the stress-ratio parameter increases. The effect of the aspect ratio on the stress-ratio parameter is found to be minimal for the lower initial preform density preforms. However, as the initial preform density increases, a higher stress ratio parameter is observed for higher initial preform densities with a lower aspect ratio. This shows that the formability increases for the preforms with lower aspect ratios and higher initial preform densities.

The Figures 10a to 10c show plots of the axial stress (σz) against the relative density R. The experiment was done with preforms that have initial densities ranging from 0.6 to 0.75 and aspect ratios ranging from 0.5 to 1. The axial stress is found to increase rapidly during the initial stage of densification, and thereafter continue to increase with a lesser rate. The increase in stress due to the forming load is followed by the closure of pores in the preform, leading to its densification. This densification is attributed to the combined effect of the geometric and the matrix work-hardening. The preforms with a lower TiB₂ content were found to attain a higher stress value than the TiB₂ preforms. Along with the densification, the load-bearing capability of the preforms also increases, as is evident from the higher stress values in the plot (Figures 10a to 10c).

It was found that the preform with 6% TiB₂ and a 0.5 aspect ratio densified more. Preforms with a high initial preform density had a higher load-bearing capacity and a longer strain to failure. This is due to the presence of a smaller number of pores. At the same time, the dislocation density increases rapidly during plastic deformation, thereby resulting in a steep axial stress regime with a smaller increase in the corresponding relative density.

5 CONCLUSION

The formability behaviours of sintered Cu-TiB₂ composite preforms were studied. The formability stress index increased with an increase in the initial preform fractional density and decreased with the aspect ratios. A statistical fitting method was performed on the curve drawn between the axial strain and the stress formability index, and the parabolic curve fitting was found to give better predictive results. For the compacts with a higher value of the aspect ratio and initial preform density, the initiation of the crack appeared at a very high fracture strain.

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6 REFERENCES

5. A. Nekahi, S. Firooz, Effect of KCl, NaCl and CaCl₂ mixture on volume combustion synthesis of TiB₂ nanoparticles, Materials


14 G. Dieter, Handbook on workability analysis and applications, ASM publications 1, 2002


