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Evaluation of the Effect of ZrB₂ (SHS) Reinforcements in the Metal Matrix Composites

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ABSTRACT: The continuous process in the path of energy efficiency in industrial applications left tasks to develop energy efficient materials in all industries. Metal matrix composites (MMC) are on such development which gives scope for vide range of combinations. In the present study, Aluminium (Al) and Copper (Cu) are used as the matrices to develop MMCs. Zirconium Diboride (ZrB₂)ceramic partices of nano size produced through self-propagating high temperature synthesis (SHS) was used as reinforcement particle in the MMCs. With 0-10wt% of reinforcements added to matrices vide range of MMCs were produced through powder metallurgy (PM) route. Microstructures and hardness of all the compositions were analyzed to evaluate the effect of reinforcements in the matrix and are reported here.

KEYWORDS:Self-propagating high temperature synthesis, Powder Metallurgy, Metal matrix composite

I. INTRODUCTION

The highly competitive powder metallurgy PM process have many advantages over conventional manufacturing processes such as casting, forging, machining and stamping to make components to cater all these industries discussed above. PM proved to me mare rapid in production and highly economic for high volume production of producing components according to the industry needs[1]. Ultra-high temperature ceramics (UHTC) are the advanced borides, carbides and nitrides which form a class of non-oxide ceramic compounds. These materials possess high melting point, extremely high hardness and wear resistance, low specific gravity, and in addition holds mechanical properties and chemical inertness at elevated temperatures [2], [3].

Titanium diboride (TiB_2) and zirconium diboride (ZrB_2) , its composites are important ceramics, they possess properties which are desirable. Borides having properties like high melting points, high hardness, low thermal conductivities, and high seebeck coefficients etc[4]–[7]. The monolithic UHTC composite based on ZrB_2 offer potential solution. The ZrB_2 based materials indicate great dimensional stability in low-pressure supersonic aerothermal heating environments, where different materials fizzle by dissolving, spalling, pyrolyzing and so forth [3]–[6], [8]–[12]. The effect of ZrB_2 in Al matrix on the microstructure, mechanical properties and the formability behavior has been reported [13]–[18].

II. LITERATURE REVIEW

The metal precursors Al, Cu and Self-propagating high temperature synthesized ZrB_2 are the precursors for the preparation of Al and Cu composites. Al (> 99 % pure and the particle size < 44 µm, Sisco Research Laboratories, India). Copper (Cu) (>99.9% pure and the particle size < 44 µm, Alfa aesar) as shown in fig.1 . The ultra-high temperature ZrB2 ceramic powder particles are produced through self-propagating high temperature synthesis (SHS) process. H3BO3, ZrO2 and Mg are the raw materials used in the process of producing ZrB_2 particles. The resultant in this process is leached in Hydrochloric acid to tap the ZrB_2 nano sized powder particles. The tapped ZrB_2 particles exist as agglomerates of nano size fig. 2. The particle size, shape and morphology of the powders were studied by scanning electron microscope (SEM, VEGA TESCAN) by scattering sufficient amount of powder on the adhesive carbon tape and then subjected to electron beam. The metallographically polished fine mirror finish surface sample is used in SEM to see the morphology of the sintered preforms. The X-ray diffraction patterns (XRD, PAN ANALYTICAL) of powders are detected by subjecting to X-rays on flattened powder surface, while a mirror surface finish polished preform is used in case of sintered preforms.



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The Al-ZrB₂ and Cu-ZrB₂ MMCs were fabricated using powder metallurgical cold uniaxial pressing and sintering technology. Aluminium and Copper powders were blended with abrasive grade ZrB_2 particles to form a mechanical mixture of Al-ZrB₂ and Cu-ZrB₂ powder with varying percentage of ZrB_2 (from 0%-10%) powder by weight are mixed to form a homogeneous mixture. Following the blending operation, the mixture is then pressed at room temperature in a die punch arrangement made up of stainless steel at pressures which make the powders adhere to each other. This process is called cold compaction. The blended powders must be compacted into a 'green compact form', with appropriate density typically by cold isostatic pressing. About 10-15 gm of the powder mixture was taken in a die of 30 mm diameter adopting a method of coning and quartering for compaction. For each component, approximately 10-15(generally higher order is taken in case of copper as of its density) gm of powder was measured out and poured into the die cavity. The equipment used for this machine is cold uniaxial pressing machine (25 TONS MANUAL PELLET PRESS MFG: KIMAYA ENGINEERS).

Sintering was carried out within a sealed unit, in an atmosphere of argon at pressure of 1 bar at different temperatures for Al-MMC and Cu-MMC. The green samples are sintered at an elevated temperature but just below the melting point of main component for an ample amount of time. A batch of thirty six green samples, each six of which different composition from (0% to 10%) of ZrB_2 for Al-MMC and so as for Cu-MMC. Al-MMC sintered at a temperature of 550°C with a heating rate of 10°C/minute and a soaking time of 1 hour after reaching a temperature of 550°C. Cu-MMC sintered at 950°C with heating rate of 10°C/minute and soaking time of 1 hour is given after reaching at temperature of 950°C with a supply of pure argon (XL grade). The aluminium particle is always surrounded by an oxide layer.

The high temperature sintering process causes this aluminium surrounded oxide layer in the particle melt and expand in volume to rupture due to high sintering temperature. Then the aluminium melt from one particle come in contact with melted aluminum escaping from neighborhood particles and welding takes place in between them. The presence of Zirconium di- -boride particles obstructs the aluminum melt from one particle to weld melt from another. So, increasing silicon carbide content increases the sintering temperature needed to achieve high strength composite. Also, the oxide layer fragmented into small shell pieces disrupted in the aluminum matrix restraining the increment in strength and the movement of dislocation. Then furnace is left to cool to room temperature for a time span of 24 hours. The average thickness and diameter of AL pellets are 11 mm and 28 mm respectively, and of Cu pellets are 8mm and 21mm respectively.

Hardness of Al-MMC and Cu-MMC was measured after sintering followed by polishing (mirror like polishing by emery paper of grade 1/0, 2/0, 3/0, and 4/0 followed by cloth polishing). Vickers' micro-hardness test was carried out using Shimadzu micro-hardness tester (model: HMV-2000). A load of 150g and dwell time of 15sec was applied on the sample (Al-ZrB₂ and Cu-ZrB₂ pellets), and allowed to make indentation. An average of 5 readings for every sample has been recorded and average of the readings with error has been reported here.

III. RESULTS AND DISCUSSION

The SEM images of pure Al and Cu shows that the powder particle size were in the range of $10-50\mu m$ while in case of ZrB₂ they were in nano scale range but appeared in the form of agglomerates fig.1 and fig.2.



Fig.1: (a) Pure Aluminium, (b) Pure Copper



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Fig. 2: Scanning electron microscope image of SHS synthesized ZrB₂ powder.

The X-Ray diffraction analysis of the SHS produced ZrB_2 powder shows that there are traces of precursor ZrO_2 . The presence of traces of ZrO_2 is because of the unreacted ZrO_2 during SHS. Leaching the products of SHS for 2-3 times in HCL solution will keep the amount of ZrO_2 in the final products to minimal levels. The ZrB_2 powder particles produced in SHS process were about 97% pure.

The relative density of Al-ZrB₂ after (cold compaction and sintering) of composites is found to be 83.5, 81.6, 77.4, 75.7, 71.2 and 69.9% for 0, 2, 4, 6, 8 and 10 weight % of ZrB₂. The relative density of Cu-ZrB₂ the relative density after (cold compaction and sintering) is found to be 87.36, 86.94, 81.76, 77.5, 76.78 and 74.10% for 0, 2, 4, 6, 8 and 10 weight% of ZrB₂. The relative density decreases with increase in weight % of ZrB₂ due to increase in porosity as abrasive nature of ZrB₂ when it is reinforced in Al-X% ZrB₂ and Cu-X% ZrB₂ (X=0, 2, 4, 6, 8 and 10 weight%) it does not allow the composite to get pressed as it reaches to higher weight % while cold compaction is done.



Fig.3: X Ray analysis of (a) Pure Aluminium & (b) Pure Copper powder



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Fig. 4: X-Ray analysis of SHS synthesized ZrB₂ powder.

The SEM images of the Al-MMCs shows the homogenous distribution of the ZrB_2 particles in the matrix fig. 5. The increased porosity with the increase in the amount of ZrB_2 is evident in the SEM images. The elongated scratches appeared in the composites when compared to monolithic pure Al preform. This is because of the pull out of the ZrB_2 particles during metallographic polishing. The dark spots on the surface of the Cu-MMC were the ZrB_2 particle pullout spaces as shown in fig.6. Comparatively Cu-MMcs exhibited higher relative densities when compared to to Al-MMCs.





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Fig. 5: SEM images of Al-ZrB₂ composites



Cu-8ZrB₂

Cu-10ZrB₂

Fig. 6: SEM images of Cu-ZrB₂ composites

The hardness of the composites increased with amount of ZrB_2 in the matrix. This is due to the presence of ultra-hard ceramic ZrB_2 in the matrix which inhibits the ability to get compacted during cold axial compaction. This



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results in more porosity in the composites with higher percentages of ZrB_2 . The hardness is found to be higher in case of Cu-MMC than Al-MMC when composites are tested on Vickers Hardness testing machine(an average of six values were evaluated). This is due to high density of pure copper (8.96gm/cm³) than that of pure aluminium (2.70 gm/cm³) fig. 7 and fig. 8.



Fig. 7: Hardness of Al-ZrB₂ Composites.



Fig.8: Hardness of Cu-ZrB₂ Composites.

IV. CONCLUSIONS

• The ZrB2 particles produced by SHS are of nano scale but appeared in agglomerates.

• The relative densities of the Cu-MMCs appeared to be on higher side when compared to Al-MMCs. This is due to the inherent density nature of the respective monolithic metal matrices.

• The relative densities of the composites decreased with increase in the amount of ZrB2 in both the metal matrices. This trend in decrease of the relative densities is due to the inhibiting nature of the ZrB2 powder particles to get uniaxial compressed during compaction.

• The Hardness of the composites increased with the amount of ZrB2 particles in the matrix. Upon comparison the Cu-MMCs appeared to have higher hardness to their counterparts in Al-MMCs. This can be attributed to the hardness of the matrix elements hardness.

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