Microstructure, microhardness, and tensile properties of hot-rolled Al6061/TiB_2/CeO_2 hybrid composites

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Synopsis

TiB_2 and CeO_2 particle-reinforced Al6061 hybrid composites were manufactured using stir casting and hot rolling techniques. The base alloy and composites were hot-rolled at 500°C and a 50% reduction was achieved through 12 passes. The effect of varying TiB_2 and CeO_2 particle additions on the microstructure and mechanical properties of the Al6061 matrix was studied. Scanning electron microscopy showed uniform dispersion of both the reinforcements, with good interfacial bonding. Microhardness and tensile properties like yield and tensile strength were found to be higher for hybrid composite with 2.5% TiB_2 and 2.5% CeO_2 compared to Al6061 alloy and other hybrid composites. The increased tensile strength is attributed to good dispersion and interfacial bonding between the particles and Al6061 matrix. Fracture analysis using a scanning electron microscope revealed ductile fracture for the Al6061 alloy and mixed characteristics of ductile-brittle fracture for hybrid composites.

Keywords

aluminium matrix composites; hot rolling; microstructure; mechanical properties; fracture analysis.

Introduction

The development of lightweight metal matrix composites with multiple reinforcements has been extensively studied owing to their numerous advantages over single reinforcement metal matrix composites. Among all lightweight metals, aluminium and its alloys have been considered as potential candidate materials for the matrix phase (Surappa, 2003; Bodunrin, Alaneme, and Chown, 2015). Aluminium and its alloys have low density, good corrosion resistance, and moderate to high strength. In particular Al6061 alloy, which contains major alloying elements such as magnesium and silicon, is known for good formability and moderate strength (Bray, 1990). At present this alloy is used for construction and transport applications such as rail coaches, bridge railings, couplings, valves, welded structures, and helicopter rotor skins. The base Al6061 alloy can be reinforced with different filler materials to improve its properties. The most commonly used reinforcement materials are ceramic and carbon-based particles/fibres (Ram, Koppad, and Kashyap, 2014; Rajesh, Auradi, and Kori, 2016). Although the early studies focused on the development of single reinforcement-based composites, the past couple of decades have seen an appreciable amount of work on hybrid composites (Sharma, 2000; Ramesh *et al.*, 2010; Boppana *et al.*, 2020; Ghazanlou, Eghbali, and Petrov, 2021).

The newly developed hybrid composites not only have good mechanical properties, but also exhibit good physical and tribological properties (Vellingiri, 2019; Mallikarjuna *et al.*, 2020). They are capable of meeting both load-bearing and wear resistance requirements for many applications. Mummoorthi, Rajkumar, and Kumar (2019) reported high tensile strength and elongation of 421 MPa and 11.2% respectively for Al6061 hybrid composites with 5% Fe_2O_3 and 6% B_4C particles. The wear rate tends to decrease with increased hybrid reinforcement content. Prakash, Sivasankaran, and Sasikumar (2015) found that the Brinell hardness of friction-stir processed Al6061 hybrid composites increased up to 6% Al_2O_3 content, decreasing thereafter.

The performance of hybrid composites depends largely on the appropriate selection of reinforcement combinations. The addition of soft phases like graphite, graphene, or carbon nanotubes reduces the coefficient of friction as well as the wear rate of hybrid composites. Premnath *et al.* (2014) studied the hardness and wear behaviour of Al6061/Al_2O_3/Gr hybrid composites developed using stir casting. With increased Al_2O_3 particle content from 5% to 15% the hardness increased marginally. Wear testing was conducted as per the central composite design method, and ANOVA was used to obtain significant wear parameters. Load was identified as the dominant factor affecting the wear rate of the composites.

As mentioned earlier, the enhancement of mechanical and tribological properties is highly dependent on the hybrid reinforcement combination, but the effect of the processing route also needs to be considered. An appropriate processing route helps in achieving good bonding between matrix and reinforcement and uniform dispersion of the reinforcement in the metallic matrix. Much research has employed thermomechanical processing to obtain good quality and enhanced properties of hybrid composites. In particular, hot rolling is used to shape cast composites into sheet or plate form, as well as improve the properties. Hot rolling is generally carried out above the recrystallization temperature and is capable of refining the microstructure significantly; it also helps in obtaining good bonding and uniform dispersion of reinforcements. Kumar *et al.* (2018) employed hot rolling to develop Al6061/ZrB_2 *in-situ* composites. The rolled composites showed a significant reduction in the grain size and improvement in the tensile properties compared to as-cast composites. The composite with 10% ZrB_2 content showed the highest tensile and yield strength values of 324 and 310 MPa. Xu *et al.* (2012) studied the effect of rolling reduction on the tensile properties of Al6061/$Al_{18}B_4O_{33}$ composites. Increased rolling reduction from 30% to 70%, improved the tensile properties. Additionally, $Al_{18}B_4O_{33}$ whiskers tended to orient themselves along the rolling direction. Nie *et al.* (2017) investigated the mechanical properties of hot-rolled Al-TiB_2/TiC hybrid composites. Rolling was carried out at 300°C with different rolling reductions of 20% to 90%. After rolling, the microstructures revealed improved particle distribution with significant grain refinement. Similarly, the hardness and strength increased with increased rolling reduction.

Exploiting the advantages of hot rolling, this work focused on the development of Al6061/TiB_2/CeO_2 hybrid composites using stir casting and hot rolling. The addition of multiple reinforcements in the Al6061 matrix can cause clustering in cast conditions. In order to break reinforcement clusters, hot rolling was used for grain refinement and to improve the mechanical properties. The effect of varying reinforcement contents on the mechanical properties of Al6061 alloy is presented.

Experimental

Materials and method

The Al6061/TiB_2 composites with 2.5 and 5 wt% TiB_2 particle content were manufactured by *in-situ* reaction between Al-3%B and Al-10%Ti master alloys. The chemical composition of the Al6061 alloy used as matrix material is presented in Table I. The detailed procedure for the synthesis of Al6061/TiB_2 composite is described in Ramesh, Pramod, and Keshavamurthy (2011). For these *in-situ* composites, CeO_2 particles at 2.5 and 5 wt% were added externally. The *in-situ* composite melt was held at 800°C, followed by gradual addition of preheated CeO_2 particles, and stirred using a mechanical stirrer rotating at 400 r/min. Stirring was continued for about 15 minutes to ensure proper mixing and good dispersions of CeO_2 particles. After stirring, hexachloroethane tablets were used to degas the molten metal to avoid casting-related defects. The degassed molten metal was then poured into permanent moulds of cast iron. After casting, all the samples were subjected to hot rolling using a two-high rolling mill (Buhler Group, Germany). Prior to hot rolling, all the samples were heated to 515°C for about one hour. After heating, hot rolling was carried out to achieve about 50% rolling reduction in a 12-pass rolling schedule. The Al6061 and hybrid composites with their designations are presented in Table II.

Characterization and testing

Hot-rolled samples were cut by electrical discharge machining (EDM). Samples for scanning electron microscopy (SEM), microhardness, and tensile tests were machined using EDM as per ASTM standards (ASTM E8/E8M, 2016). For microscopic analysis, the samples were polished using grit papers followed by 0.25 µm diamond paste. To reveal microstructures such as grain boundaries, all the samples were etched using Keller's reagent (190 ml distilled water, 5 ml HNO, 3 ml HCl, 2 ml HF). Scanning electron microscopy with energy dispersive X-ray analysis (EDX) (Hitachi, model number SU3500N) was used for the analysis of the hot-rolled microstructure, reinforcement dispersion, interfacial bonding, and composition. The micrographs were taken in backscattered (BSE) and secondary electron (SE) modes for analysis of microstructural features. Microhardness tests were conducted using a Vickers tester (Shimadzu microhardness tester) employing a load of 500 g. For each sample, about five indentations were made and the average value recorded. Tensile test samples were prepared as per ASTM E8/E8M and tests were conducted using a Universal testing machine. A schematic diagram of the tensile test sample is provided in Figure 1. For each sample, about three tests were conducted and the average value recorded. Fracture analysis after tensile tests was conducted on fractured samples of Al6061 alloy and its hybrid composites using SEM.

Results and discussion

Microstructure analysis

The microstructures of the hot-rolled Al6061 alloy and hybrid composites are shown in Figure 2. Figure 2a shows the Al6061 alloy, displaying elongation and orientation of aluminium grains along the rolling direction. Generally, in the as-cast condition Al6061 alloy had a dendritic microstructure, but during hot rolling the grains with the dendritic structure tended to break down, forming elongated grains with fine equiaxed grains.

Table I

Chemical composition of the Al6061 alloy

Element	Al	Mg	Si	Cu	Mn	Fe
Weight %	Balance	1.08	0.63	0.32	0.52	0.17

Table II

Composition of hybrid composites and sample designations

No.	Sample composition	Designation
1	Al6061 alloy	AA1
2	Al6061 + 2.5%CeO_2 + 2.5%TiB_2	HB1
3	Al6061 + 2.5%CeO_2 + 5.0%TiB_2	HB2
4	Al6061 + 5.0%CeO_2 + 2.5%TiB_2	HB3-

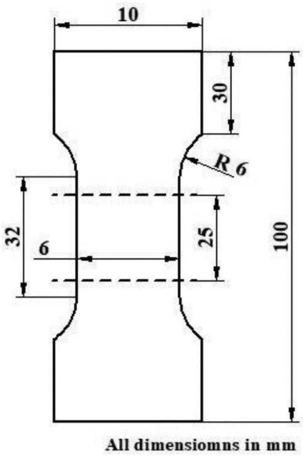

Figure 1—Schematic of a tensile test sample

Figure 2b shows the micrograph of HB1, which contained a fairly uniform dispersion of TiB$_2$ and CeO$_2$ particles. This is the main advantage of hot rolling because it breaks up inhomogeneous dispersions formed during casting by fragmenting them into individual particles and forming good bonding due to the application of compressive forces (El-Sabbagh *et al.*, 2013). Figure 2c shows HB3, which had an elongated microstructure with many fine equiaxed grains between the elongated grains. Figure 2d shows HB3 with fairly uniform dispersion of both TiB$_2$ and CeO$_2$ particles. The reinforcements were located at the grain boundaries and few were within the grains. Generally, the as-cast composites showed inhomogeneous dispersion of reinforcements and large particle-free zones. The clusters of reinforcement were very few in the hot-rolled composite, as seen in Figure 2d. This suggests that due to the application of compressive forces most of the reinforcement clusters were broken up. In addition to reinforcement declustering, the hot rolling redistributed and oriented the grains in the rolling direction. The particle-free zones were quite narrow, showing good distribution of both reinforcement particles. The absence of defects suggests that the compressive forces applied during hot rolling were sufficient to eliminate them. The compressive forces, along with elevated temperature, helped to close the porosity and improve the interfacial bonding between reinforcements and the Al6061 matrix. The clean interfaces, without any processing defects, showed that good bonding was achieved between the constituents of hybrid composites. This is important because the formation of any reaction products at the interface can have detrimental effects on the mechanical properties. Figure 3a shows that for the HB1 hybrid composite there was good interfacial bonding between the two reinforcements and the Al6061 matrix. The good bonding can be inferred from the fact that there were no defects at the reinforcement/matrix interface.

In general, the cast aluminium composites suffer problems with molten aluminium wetting the reinforcements, especially the ceramic particulates. The unwetted particles trap gases, which leads to the formation of gas voids at the interface, and numerous gas voids may form a network of interconnected voids. In addition, there is a high possibility of clustering of reinforcements due to gas adsorption by the unwetted particles. Alternatively, detrimental products may form at the interface, which could change the composition of the matrix and affect the final properties of the composites (Hashim, Looney, and Hashmi, 2001; Gawdzinska, Chybowski, and Przetakiewicz, 2016). In the present case, the interface was continuous with no interfacial products, gas voids, or crack formation due to the coefficient of thermal expansion mismatch. Also, neither TiB$_2$ nor CeO$_2$ particles reacted with the Al6061 matrix, which could have led to the formation of brittle reaction products at the interface. The absence of any kind of defects or interfacial products indicates that the bonding between reinforcements and Al6061 matrix was good. The EDX peaks, as shown in Figure 3b, indicate that most of the elements detected correspond to constituents of the hybrid

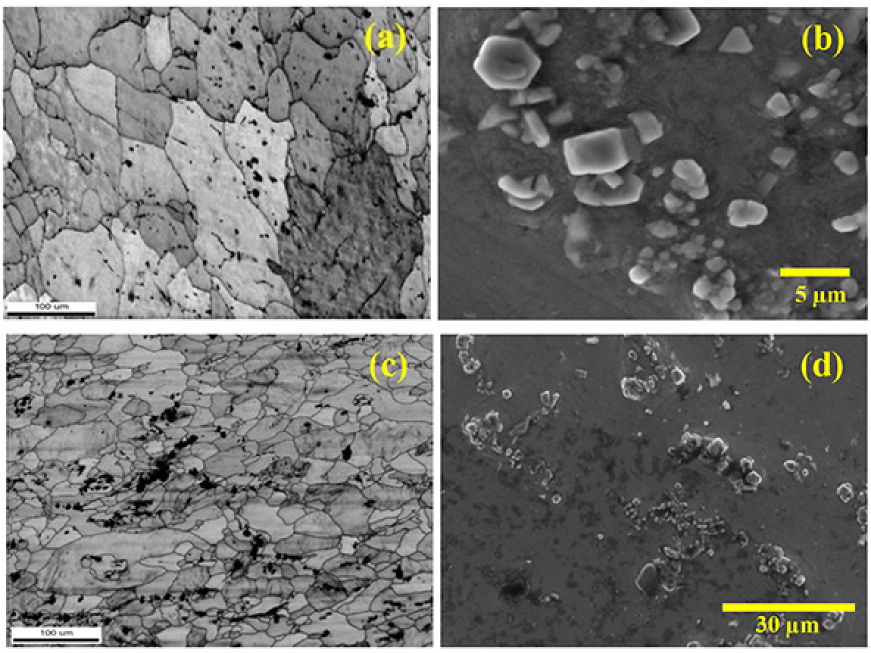

Figure 2—SEM micrographs of hot-rolled specimens. (a) AA1 taken in BS mode, (b) HB1 taken in SE mode, (c) HB3 showing grain structure, taken in BS mode, and (d) HB3 showing reinforcement dispersion, taken in SE mode

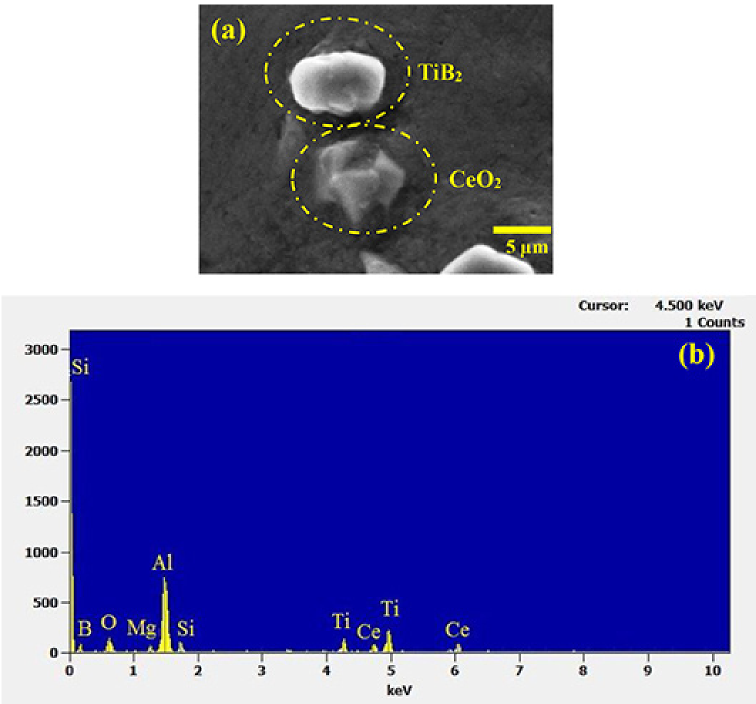

Figure 3—Hot-rolled HB1 (Al6061+2.5%CeO_2+2.5TiB_2 hybrid composite). (a) SEM-SE micrograph showing good interfacial bonding between reinforcements and Al6061 matrix, and (b) EDX analysis of the complete area

composite. The presence of Mg and Si in the pattern corresponds to the major alloying elements of the Al6061 matrix. On the other hand, Ti, B, Ce and O correspond to both the reinforcements CeO_2 and TiB_2 particles. From the EDX analysis it was quite clear that no detrimental interfacial products formed during casting or hot rolling and the interfaces were clean and continuous.

Microhardness

The microhardness values of hot-rolled Al6061 alloy and its hybrid composites were studied to ascertain the effect of multiple reinforcements. Sample AA1, with no reinforcement, had a microhardness value of 57±2 VHN. With the addition of 2.5% CeO_2 and 2.5% TiB_2, sample HB1 showed a microhardness value of 83±3 VHN – compared to AA1, about 46% increment in microhardness value was recorded for sample HB1. The other hybrid composites, HB2 and HB3, had microhardness values of 66±2 and 71±4 VHN respectively, representing increments of 16% and 25% compared to AA1. Addition of CeO_2 and TiB_2 particles resulted in a significant enhancement in microhardness of the Al6061 matrix. However, a higher reinforcement content led to decreased microhardness in hybrid composites HB2 and HB3. Overall higher microhardness values for hybrid composites indicate that both hot rolling and reinforcements increased the resistance to deformation. The increment in microhardness is attributed to densification of the microstructure due to hot rolling and the addition of hard reinforcing phases like CeO_2 and TiB_2 particles.

There is a very high probability of pore formation after casting of hybrid composites. These pores adversely affect the mechanical properties of both Al6061 alloy and hybrid composites, whereas hot rolling tends to improve the properties by eliminating pores. The plastic deformation caused by compressive forces at high temperature was the main reason for the reduction of pores in these samples (Guo *et al.*, 2017). The other main factor contributing to enhanced hardness was resistance to plastic deformation due to the presence of hard reinforcing phases. The uniformly dispersed CeO_2 and TiB_2 particles tended to resist plastic deformation caused by the indenter of the Vickers hardness tester. The resistance to plastic deformation was due to inhibition of dislocation movement by CeO_2 and TiB_2 particles (Shin and Bae, 2015). Further grain refinement caused by the hot rolling tends to increase the grain boundary volume. This inhibited the movement of dislocations, thereby contributing to improved microhardness values.

Tensile properties

The tensile strength, yield strength (0.2% proof stress), and elongation values of hot-rolled Al6061 alloy and its hybrid composites were studied. Sample AA1 showed yield and tensile strength values of 78±5 MPa and 130±4 MPa respectively. The hybrid composite HB1, which contained 2.5% CeO_2 and 2.5% TiB_2, showed yield and tensile strength values of 124±4 and 208±3 MPa respectively. The increment is quite significant compared to the unreinforced AA1 sample. Increments of 60% and 59% in yield and tensile strength values for HB1 demonstrated the synergistic effect of hot rolling and strengthening by CeO_2 and TiB_2 particles. The HB2 sample showed yield and tensile strength values of 115±7 MPa and 183±6 MPa, while HB3 showed 104±5 MPa and 169±7MPa respectively. All hybrid composites displayed significant enhancement in strength. However, the best reinforcement combination for obtaining high yield and tensile strength was that for HB1, constituting 2.5% CeO_2 and 2.5% TiB_2 particles. The strength increase of hybrid composites may be due to the following factors.

i. The hot rolling used for densification of the microstructure not only improves density and closes the casting-related pores, but also helps in uniform dispersion of the reinforcements. Furthermore, the compressive forces during rolling improve the bonding between both the reinforcements and Al6061 matrix. The uniformly dispersed CeO_2 and TiB_2 particles with good bonding are capable of bearing the load from the matrix because the good bonding helps in the efficient transfer of load from the Al6061 matrix to both CeO_2 and TiB_2 particles.

ii. The difference between the coefficients of thermal expansion of CeO_2 (approx. 10.7×10^{-6}/K), TiB_2 (approx. 6×10^{-6}/K) and Al6061 (approx. 23.4×10^{-6}/K) is quite large. Due to this, dislocations form around the reinforcements. Uniform dispersion of reinforcements helps in dispering the dislocations uniformly in the matrix. The dislocations formed due to thermal mismatch become entangled with the dislocations already present (Poza and Llorca., 1999), thus increasing the dislocation density, which is another factor contributing to strength enhancement.

As well as strength values, the elongation of Al6061 alloy and its hybrid composites was studied. For Al6061 alloy the elongation was 17.2%. In the case of the hybrid composites, HB1, HB2, and HB3, the elongation values were 9.2%, 13.9%, and 10.1% respectively. The relatively high elongation value for HB2 is due mainly to the high proportion of *in-situ* formed TiB_2 particles (5%). This agrees with the findings of Han, Liu, and Bian (2002) on TiB_2 particle-reinforced Al-Si alloy composites, who reported increased ductility as the TiB_2 particle proportions increased from 1% to 7%. The grain refinement and improved interfacial bonding of reinforcements with the matrix from hot rolling were the main reasons for the increment in elongation values for hybrid composites.

Fracture analysis

Figure 4 shows the SEM micrographs of tensile fractured samples of Al6061 alloy and the hybrid composites. Unreinforced AA1(Figure 4a) underwent ductile-type fracture. The fracture surface had large numbers of dimples, indicating plastic deformation. Figures 4 b, c, and d show the fracture surfaces of hybrid composites HB1, HB2, and HB3. As seen in Figure 4b the fracture surface of HB1 again had a large number of dimples due to plastic deformation of the Al6061 matrix. However, some regions in the fracture surface of hybrid composite HB1 had no dimples at all, but displayed cleavage facets that formed due to the addition of hard CeO_2 and TiB_2 particles to the Al6061 matrix. Such cleavage regions showed some brittle fracture, and these hybrid composites exhibited mixed features of ductile-brittle fracture (Manikandan *et al.*, 2020). As observed in Figure 4c, the fracture surface of hybrid composite HB2 was covered with numerous dimples, indicating that the sample had undergone plastic deformation, which is consistent with the highest elongation value of 13.9%. The presence of multiple reinforcements and application of hot rolling resulted in the fine equiaxed grains in the hybrid composites. In addition, the dispersion of reinforcements was quite uniform and bonding with the matrix was good, which is why the dimples were present all over the fracture surfaces. Hybrid composite HB3 had a similar fracture surface to HB1, indicating failure due to the combined effect of ductile-brittle fracture (Figure 4d). The dimple-covered fractured surfaces as seen in Figures 4b to 4d indicate that the hybrid composites were not adversely affected by the addition of the reinforcements and were able to display sufficient ductility (Xie *et al.*, 2020).

Conclusions

The effect of hot rolling and addition of multiple reinforcements in the form of CeO_2 and TiB_2 particles on microstructure, microhardness and tensile properties of Al6061 alloy were evaluated.

i. SEM analysis showed uniform dispersion of CeO_2 and TiB_2 particles in the Al6061 matrix. The improved bonding between matrix and reinforcements is attributed to hot rolling.

ii. The microhardness of the hybrid composites was significantly better than that of unreinforced Al6061 alloy. The hybrid composite with 2.5% CeO_2 and 2.5% TiB_2 had the best microhardness, at 83±3 VHN.

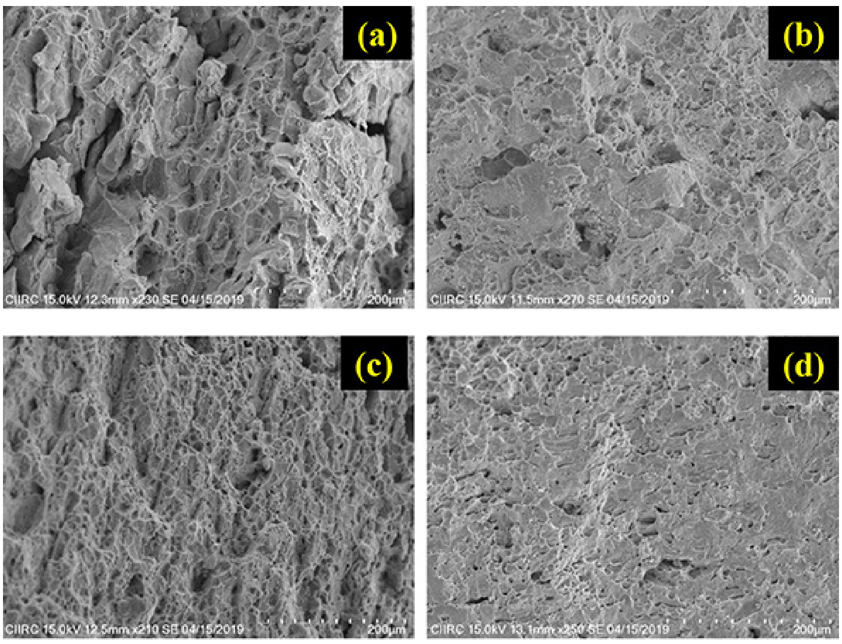

Figure 4—SEM-SE micrographs showing tensile fracture surfaces of (a) AA1, (b) HB1, (c) HB2, and (d) HB3

iii. Tensile testing revealed significant increases in yield and tensile strength of hybrid composites with the addition of CeO_2 and TiB_2 particles. The optimum reinforcement combination for attaining strength was 2.5% CeO_2 and 2.5% TiB_2 particles.
iv. Fracture analysis conducted using electron microscopy revealed that Al6061 alloy underwent ductile fracture, while the hybrid composites exhibited mixed ductile-brittle fracture.

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