



Mechanical, wear, and fracture behavior of Titanium Diboride (TiB₂) - Cerium Oxide (CeO₂) reinforced Al-6061 hot-rolled hybrid composites

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ABSTRACT. Development of aluminium composites by stircasting technique is an effective method for fabrication of better quality of MMCs. Stircasting technique is one of the most commonly accepted techniques. In this research work, Al6061 / TiB₂+CeO₂ hybrid MMCs have been fabricated with varying wt. % of TiB₂ (2.5%, 5%, 7.5% and 10%) particulates and constant 5% of CeO₂ particulates. The monolithic alloy and hybrid composite were hot-rolled at a temperature of 515°C. Both the monolithic and hot-rolled hybrid composites were subjected to micro-structural study, hardness and tensile test. Optical microscope analysis revealed uniform dispersal of hard particles within the base matrix in case of both of ascast and hot-rolled composites. Both ascast and hotrolled hybrid composites showed extensive enhanced mechanical behavior and high wear resistance when compared with monolithic alloy. Hot rolled MMCs showed enhancement of 25 % of hardness when compared with monolithic alloy with increasing reinforcement of 2.5 - 10 wt. % of TiB₂ content. Tensile strength increased by 53.54 % for hot rolled composites when compared to the as cast and other hybrid composites. Tensile and wear fractography outcome showed internal fractured structure of a tensile and wear specimen which was analysed using SEM analysis.

KEYWORDS. AMMCs, Hot rolling, Microstructure, Mechanical, Wear, Fracture behavior.



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INTRODUCTION

Two dissimilar materials are combined together and which makes one of a kind new material known as composite [1]. Aluminum Matrix Composites (AMC) are used worldwide in variety of mechanical and wear applications due to their superior properties like better strength-to-weight-ratio, hardness, low density, excellent wear resistance & stable properties at higher temperature [2-4]. Due to ease of recycling nature and being non-toxic aluminum alloys, they are finding extensive commercial applications in various areas such as automobile, space craft and structural industries where component weight reduction is the main objective [5-7]. Many research works have been done in developing Aluminum composites with different combinations of reinforcements. Sridhar Raja et al. [8] evaluated the effect of TiB₂ on micro-structural and mechanical characteristics of Al Composite. The outcomes revealed that, enhancement of tensile strength and hardness of the MMCs was observed with the increase in the reinforcement content. R Balachandar et al. [2] evaluated the mechanical characteristics of Al 6xxx reinforced with magnesium and rock dust using different weight % of composites produced by Stir Casting technique. From the outcomes it was observed that, addition of AZ31 led to increase in tensile Strength and rock dust content led to decrease in the density of developed composites. S. Gopalakrishnan et al. [9] revealed that wear rate increased by increasing in applied load. But, the wear rate reduced by increasing the TiC content. Xuedan Dong et al. [10] studied the effect of CeO₂ particulates on micro-structure and mechanical behavior of Al composites. The outcomes showed that the presence of the suitable wt. % of CeO₂ increased the tensile strength of the MMCs. Chao Liu et al. [11] evaluated the effects of CeO₂ on the mechanical behavior. The outcomes showed that the presence of CeO₂ led to enhancement in the mechanical behavior of the Al alloys. By increasing wt. % of CeO₂, all the composites samples showed the trend of increasing in the mechanical properties of Al but, porosities attained the maximum value. Reddappa et al. [12] observed that the specific wear loss has decreased with increasing wt. % of beryl content. A. Anilkumar et al. [13] analyzed that the specific wear rate reduced by increase in Beryl/CeO₂ content. SEM was used to study the uniform dispersal of Beryl-CeO₂ particulates in Al6061. R. Saravanan [14] in his research work focused on the studies relating to wear and mechanical properties of Al reinforced with CeO₂ and TiB₂. MMC's were prepared using stircasting technique. Here, cast parts were subjected to hot-rolling at the temperature of 515°C to reduce the thickness of developed composites from 10mm to 5mm in 12 passes. From the outcomes it was found that, the developed composite properties were enhanced by addition of reinforcement content. Amra et al. [15] evaluated the mechanical properties of CeO₂ - SiC reinforced Al Composites. This study was aimed to manufacture Al composites with improved mechanical properties by the addition of CeO₂ - SiC reinforcement particulates into the Al. Chicet et al. [16] summarized that, the inter conditioning parameters of contact fatigue in the case of a rolling and sliding contact depending on: – surface quality: micro defects; microcracks; material detachments; porosity; pitting; flaking; large detachments (spalling); plastic flows; – the appearance, from the depth to the surface, of the micro defects, microcracks; cracks; – material properties: modulus of elasticity; hardness; fatigue resistance; – lubricant properties: viscosity; degree of contamination; – load applied to the surface: load size; the meaning of application; mode of variation. Abuthakir et al. [17] SEM analysis of composites in the peak aged (T6) condition revealed that the amount of AlNi intermetallic phase formed increased with an increase in weight fraction of Ni particles since the number of heterogeneous nucleation sites were increased. It can be said that AA6061 + 1.5 Wt% Ni composite has higher amount of AlNi intermetallic reinforcements. The increased amount of AlNi intermetallic phase in the composites with increasing weight fraction of Ni particles can be attributed to dissolution of Ni particles in the peak aged (T6) condition. This resulted in higher hardness and more effective grain refinement of composites than the base alloy in the peak aged (T6) condition. Though, from the literature survey it is found that sufficient data on hot rolled hybrid composites is not available on the mechanical properties of Al6061 MMCs. So, the current research work aims at study of micro-structure, hardness, tensile strength and wear characteristics of the hot-rolled hybrid AMMCs (Al6061+TiB₂+CeO₂) having different wt. % of reinforcement. The fractography of the fractured surface of tensile and wear test specimens were analyzed through SEM study.

RAW MATERIALS

Matrix and Reinforcements

Aluminium 6061 is a precipitation-hardened alloy whose major alloying element is silicon (Si) and magnesium (Mg). Alloy 6061 sets the standard from the other alloys on account for its lightweight, medium-to-high strength and it provides an excellent corrosion resistance when exposed to atmosphere and sea water [9, 17].



Titanium Diboride (TiB_2) is a non-lustrous metallic grey colour material which is an excellent heat conductive material and is a very hard ceramic [18]. It exhibits oxidation stability and also resistance to mechanical erosion. Due to it being a reasonable conductor, it can be used as a cathode material in aluminium smelting [19].

Cerium oxide (CeO_2) is an oxide from the family of rare-earth metal called cerium. Its crystal structure is that of cubic packing. The appearance of cerium oxide is that of a pale yellow-white colour powder and plays a vital role in purification of ores by performing as an intermediate content. The stand out point of this material is in its ability to undergo reversible conversion to a non-stoichiometric oxide [20].

METHODOLOGY

Preparation of composites

For the fabrication of hybrid composites, liquid metallurgy technique was selected [21]. The required content of raw materials for the fabrication of composites was obtained in the form of ingots for Al6061 and powder in the case of TiB_2 and CeO_2 . Al6061 alloy was first melted at $775^\circ C$ using an electrical furnace. Graphite crucible was used to melt the matrix material. Generally, this graphite crucible helps to withstand the high temperature, and provides good resistance to thermal shock and chemical erosion [22, 23]. Pre-heated reinforcements such as TiB_2 (2.5%, 5%, 7.5% and 10%) and CeO_2 (5%) were added to the molten metal. While adding of required quantity of preheated reinforcements, stirring action was maintained continuously for 3 minutes at the speed of 300 rpm [24]. The continuous stirring action generally enables to attain the uniform dispersal of reinforced particulates within the base matrix. Finally, ready molten metal was poured continuously in to metallic die. After solidification, the castings were removed from the metallic die. Then, cast parts (as shown in Fig. 1(a)) were subjected to hot rolling at the temperature of $515^\circ C$ to reduce the thickness of developed composites from 10mm to 5mm in 12 passes (as shown in the Fig. 1(b)). The developed composite samples were pre-machined by using wire EDM. Hardness, tensile and wear test samples (specimens) were prepared based on the standards (ASTM). These test specimens were subjected to study the micro-structure, mechanical, wear and fracture behavior. The test samples of developed hybrid composites are shown in the Fig. 1 (c - e).

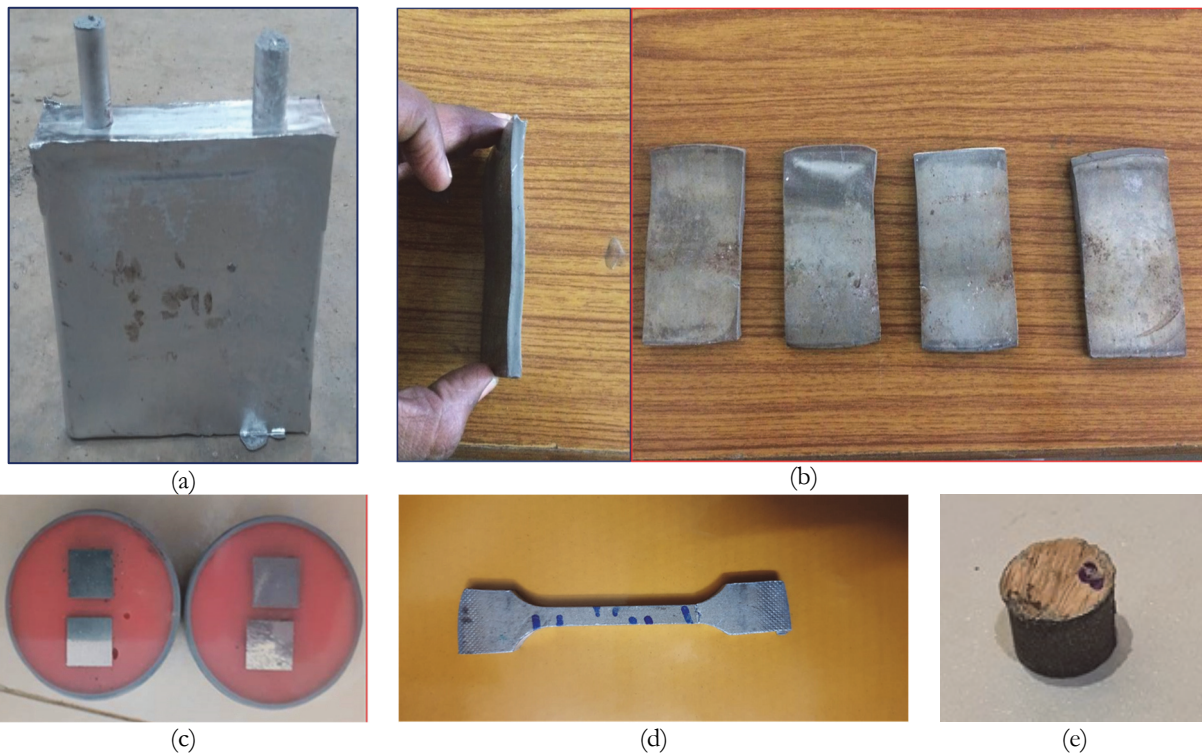


Figure 1: (a) Cast part, (b) Hotrolled composite sample, (c) Hardness specimen, (d) Tensile specimen and (e) Wear specimen



RESULTS AND DISCUSSIONS

Micro-structural Analysis

Hot-rolling of Al-6061 alloy and hybrid composites (Al-6061 + TiB₂+CeO₂) was carried out successfully at 515°C. The surfaces of the micro structure test specimens were polished using emery papers with 400 grit size with diamond paste. Then the specimens were polished using velvet disk polishing machine to get fine finish on the surface. Uniform dispersal of TiB₂ and CeO₂ particulates was studied by microscopic examination. Uniform dispersion of reinforcing particulates showed better impact on the mechanical properties of MMCs [25-27]. An optical microscopic image of Al6061 and Al6061 / TiB₂ + CeO₂ hybrid composites prior to the rolling process is shown in Fig. 2 (a-c).

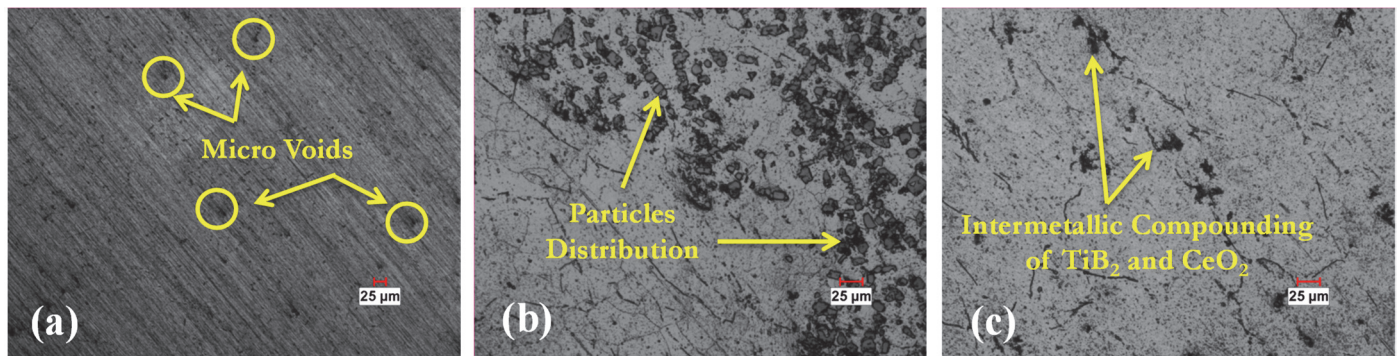


Figure 2: Optical Micrograph of (a) Pure Al alloy (b) Al 6061 reinforced with 7.5% TiB₂ and 5% CeO₂ (c) Hot rolled hybrid composite with 7.5% TiB₂ and 5% CeO₂

From the figure it is seen that reinforcement particulates have been found along with the grain boundaries [28]. It is also revealed that hard ceramic TiB₂ particulates are uniformly dispersed in the Al6061 alloy. Results reveal that, after hot rolling, most of the reinforcement particulates have been aligned within the direction of metal flow [29, 30]. When compared to the Al6061 alloy, the hybrid MMCs show smaller sized grains due to presence of hard particles generally, which contributes to better grain refinement [31, 32]. The presence of TiB₂, a grain refiner, plays a vital role in grain refining of hybrid MMCs. Fig. 2 (a) depicts the micro-structure images of Al6061 alloy, Fig. 2 (b) depicts the micro-structure images of hybrid composites before hot rolling and Fig. 2 (c) depicts the micro-structure images of hybrid composites after hot rolling. From the outcomes, it is observed that, the micro structural changes have occurred during hot rolling. The grain morphology has changed in the direction of rolling to an elongated grain structure. There is nucleation of the new grains within the grain boundaries of grains; the microscopic image clearly reveals less porosity in both matrix material and in hybrid MMCs after hot rolling. In addition to this, the hybrid composites show a better bond between the base matrix and TiB₂ particulates which may be attributed to the better wettability of particulates, refinement of reinforcement and uniform dispersion of reinforcements within the base matrix alloy leading to the enhancement in strength of the developed hybrid composites.

Hardness

The microhardness of developed composites was tested as per E92-ASTM standards by using Vickers Micro Hardness testing apparatus. Diamond shape indenter was used under the constant load of 5 kg for a time period of 15 seconds. Hardness tests trials were carried out at 27°C (room temperature) and the hardness of each sample was evaluated at 3 different zones on the test specimens to find the average hardness value. The microhardness of as-cast, hybrid composites and hot rolled hybrid composites are depicted in Fig. 3.

It is found that the presence of TiB₂ and CeO₂ particulates has enhanced the hardness of hybrid composite when compared with base matrix. During the solidification process in cast composites, TiB₂ elements cause increase in the dislocation of density [33-36]. Xuedan Dong et al. [10] observed that, when the CeO₂ content was increased, the mechanical properties of the composites were enhanced. Since, Stircasting technique is adopted for the purpose of fabrication of composites, this leads to obtain uniform bonding among reinforcement and matrix. This is helpful in improving the material properties of developed hybrid MMCs. Generally, the reinforcing of ceramic particles with in the soft matrix will carry the load and also offer better resistance [38]. In present research work, hard ceramic TiB₂ particulates act like load bearing elements and also take the maximum applied load for the plastic deformation and lead to increase of



hardness in developed hybrid composites [35]. TiB_2 being hard particulates allow the material to flow with-out undergoing the deformation. And when it exceeds the critical values it will cause fracture without any further deformations. Based on the concept of Hall-Petch equation, the hardness will be enhanced by decrease in size of grain [39]. The grain refinements can also be an effective reason for improving the hardness in TiB_2 and CeO_2 reinforced hybrid composite. Finally, the hard ceramic particulates such as TiB_2 and CeO_2 content have better influence in increasing the strength of developed hybrid MMCs [40]. When the wt. % of reinforcements exceeds, the agglomeration of hard particulates was increased. During fabrication of MMCs with stircasting process, 10% of TiB_2 led to agglomeration. Generally, it occurs due to the less wettability of TiB_2 particulates in the matrix material. Due to this, agglomeration forms in MMCs reinforced with 10% of TiB_2 . The internal structure of the agglomerated particulates was loose and which usually could not efficiently withstand the stresses. The capacity to transfer the stress will also reduce. The agglomerated particulates will be irregular/uneven in shape which leads to crack initiation in the development of plastic deformation. It is also found that, agglomeration leads to reduce the hardness of hybrid composites at higher wt. % of reinforcements [37]. Whereas, in the hot rolled MMCs, the bonding between matrix and reinforcements is very good as compared to the as-cast composites. Due to better bonding between matrix and reinforcements, the stircasted hot-rolled MMCs exhibited enhanced microhardness. One more reason for enhancement in hardness is restraint to dislocations of movement by TiB_2 & CeO_2 particulates. The hard particulates such as TiB_2 & CeO_2 in the base matrix act like a barrier to the movements of dislocation and also increase the stresses essential for the purpose of movement of dislocation. Uniformly distributed TiB_2 & CeO_2 particulates in the base alloy will enhance in overall stress which leads to increasing in microhardness. In addition, it is observed from the micro-structure study, the amount of defects is less in the hot-rolled MMCs which leads in enhancement of microhardness. This is generally due to the existence of minor flaws and be restored which are rectified during the hot rolling process [41, 39]. From Fig. 3, it is observed that, hardness tends to increase as the indent size decreases in small scale. It is purely based on the ductility of the tested materials. The developed composites reinforced with hard particles have high influence on the hardness of the materials. The obtained results indicated that, there was an increase in hardness (25 %) of hybrid composites with increasing of 2.5 - 10 wt. % of reinforcement content. The indentation in 7.5 % of TiB_2 reinforcement shows minimum indentation size when compared to the other samples. It reveals that highest hardness (83 VHN) was achieved in 7.5 % of TiB_2 reinforced hot rolled hybrid composites.

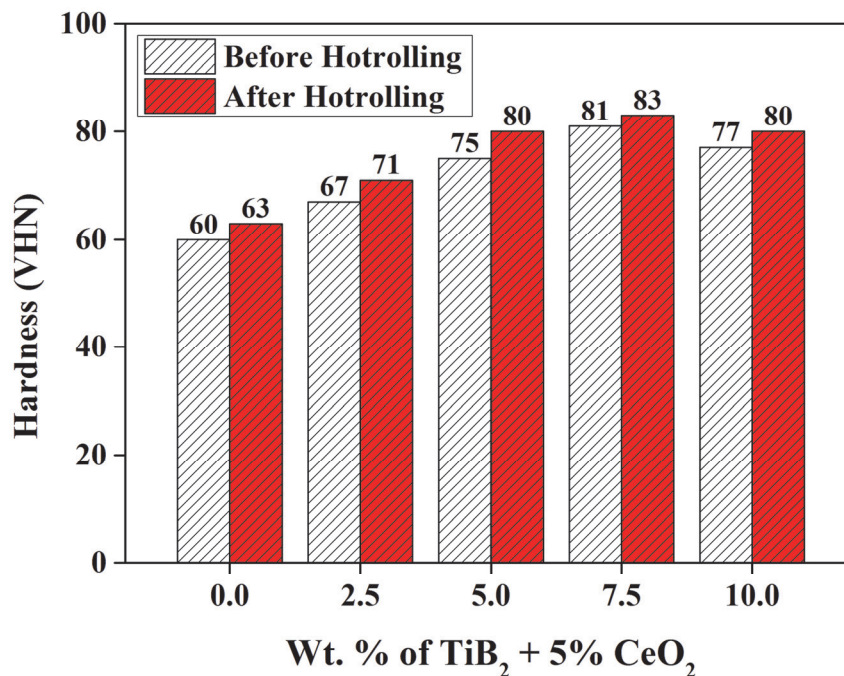


Figure 3: Hardness results of monolithic, hybrid MMCs and hot-rolled hybrid MMCs

Tensile strength

The test samples were machined according ASTM-E8 standards [42, 43] and tension tests were conducted on UTM which has maximum load capacity of 400 KN. Fig. 4 depicts ultimate tensile strength of ascast and hot rolled hybrid MMCs.

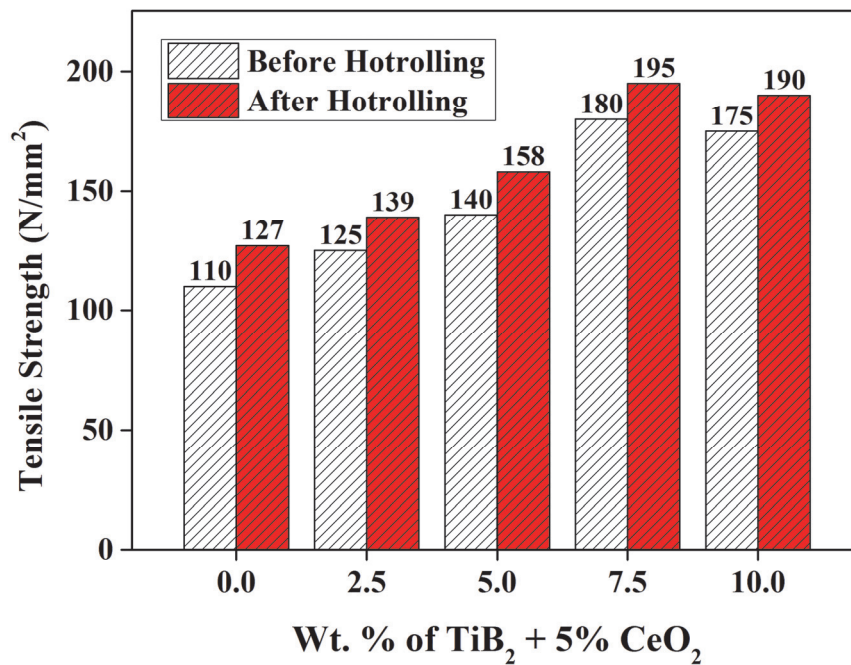


Figure 4: Tensile strength of Al-6061, hybrid MMCs and hot-rolled hybrid MMCs

The presence of TiB_2 and CeO_2 particulates enhanced the overall mechanical strength of developed composites in both ascast and hot rolled conditions. Better enhancement in tensile strength after hot rolling shows an effective forming method in densifying the ascast and MMCs. Improved ultimate tensile strength may be due to the existence of elongated grains within the direction of hot roll [41]. There are several factors which lead to improve the mechanical behavior of hybrid MMCs, and these include dispersion of particles, grain size and interfacial bonding among matrix and reinforcements. Generally, hot rolling does not only lead to decrease the grain size by the strain-hardening, but also increases the dispersal of reinforcement/s more uniformly within the base matrix. The fresh interface formed at the time of casting, the bonding among TiB_2 and CeO_2 particulates and matrix alloy increase further due to hot rolling process. Enhancement in the tensile strength can also be ascribed to strengthening of mechanisms such as grain refinements, dislocation, load transfer and “Orowan” strengthening. It is identified that there must be stability among the matrix material and hard reinforcement particulates during load on a bulk material for the load transfer. Interface with better bonding among the matrix and reinforcement particulates confirm that the load transferred is more effectively from the matrix alloy to hard particulates (TiB_2 and CeO_2) [28]. The strengthening mechanism takes place by recrystallization at the time of thermo-mechanical processing. The hard particulates do not deform at the time of hot-rolling and strain with in the base material forms the deformation region around the particulates. Particulates deformation region is formed nearby hard particulates because of mismatching between them and base material. Thus, the recrystallization takes place easily in these deformation regions due to the particulates stimulated nucleation. And resultant grains are formed because of recrystallization with in hot rolled hybrid MMCs compared to ascast Al6061 or hybrid MMCs. The grain refinements by particulates stimulated nucleation leads to increase in material strength of hotrolled hybrid MMCs according to the Hall-Petch concepts. The existence of higher dislocations density with in hot rolled hybrid MMCs results in significant improved strength. It revealed that, “Orowan-Strengthening” mechanism also leads to improvement in tension strength (tensile) of hybrid MMCs from preventing movement of dislocation by finely distributed hard particulates like TiB_2 and CeO_2 [39]. The formed hard particulates are uniformly dispersed within the base matrix and act like obstacle for dislocations. This is mainly because the particulates are very hard and also non-deformable because it is very difficult for the dislocations and also to cut them. Fig. 5 depicts the ductility of Al6061 alloy, hybrid composites and hot rolled hybrid MMCs.

From the graph, it is observed that, the increasing TiB_2 and CeO_2 particulates content in Al-6061, marginally reduces the ductility in ascast and hot rolled composites [44, 45]. When compared to ascast material, it is found that the hot rolled matrix and its MMCs show improved ductility. This is generally due to the existence of defects in casting like solidification shrinkages and porosities which act like a crack nucleation due to the load which aids in propagation of cracks and nucleation. Whereas, in the case of hot-rolled hybrid MMCs the defects are reduced because of the plastic deformation at a higher temperature. The grain refinement due to the hard particulates also leads to better ductility in hybrid composites.

There is a reduction of ductility by increased wt. % of TiB₂ and CeO₂ particulates due to existence of cracks nucleating sites mainly at interface among reinforcement and base alloy in large number and also due to the existence of micro-porosities. Hard particulates of TiB₂ and CeO₂ change the direction of crack propagation and it results in bridging of cracks, branching and deflection in the direction along with the direction of tension load. This needs high amount of the energy. And also it leads to high resistance to the cracks propagation causing in the higher ductility and fracture toughness [46]. High wt. % of cluster (agglomeration) in TiB₂ and CeO₂ particles leads to high debonding of reinforced particulates from interface of matrix and reinforcements during tensile loading, and this leads to decreased ductility [41]. Tensile fractography images of hybrid composites and hotrolled hybrid composites with 7.5% TiB₂ and 5% CeO₂ are shown in Fig. 6.

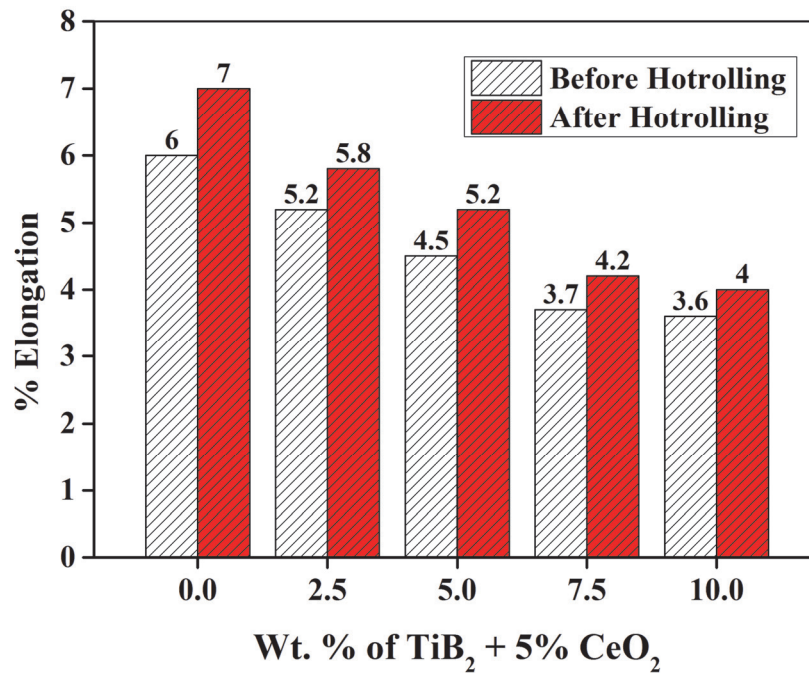


Figure 5: Ductility of monolithic, hybrid composites and hot rolled hybrid MMCs

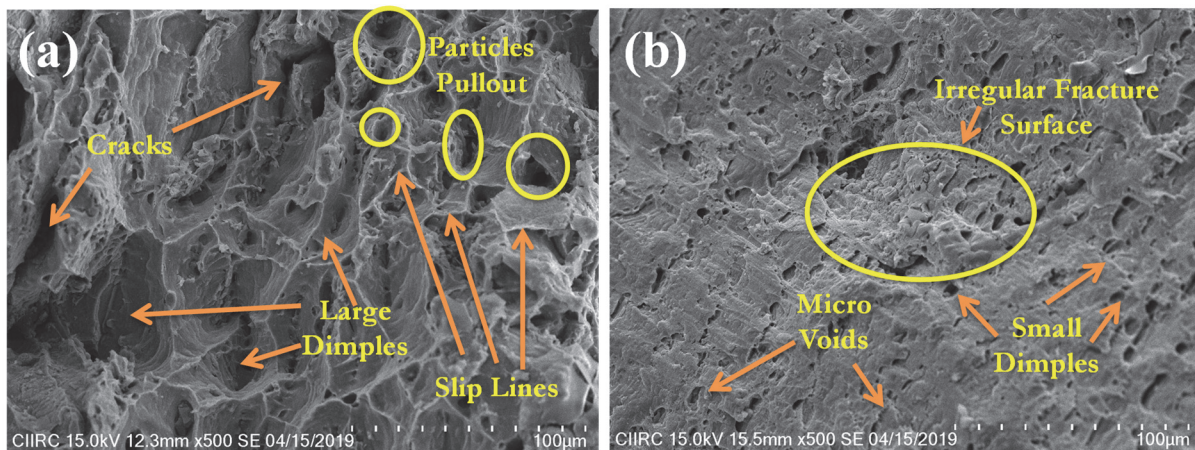


Figure 6: SEM images of fractured surface of tensile test samples (a) 10% TiB₂ and 5% CeO₂ hybrid composite (b) 10% TiB₂ and 5% CeO₂ hot-rolled hybrid composite.

In the hybrid composites sample shown in Fig. 6 (a), fractured surface reveals the existence of dimples showing ductile nature of fracture. But, the size of micro voids formed after the fracture show debonding of Al grains, which are placed adjacent to micro-porosities and agglomeration of hard particulates [32]. The existence of hard particulates such as TiB₂ and CeO₂ on the fractured surface shows better bonding with the base alloy due to the clean interface. Whereas, in hot

rolled hybrid composites depicted in Fig. 6 (b), micro voids and dimples in small size are formed over the fracture surface. Fracture begins with development of coalescence and micro-voids occur by increasing in applied load. When the load reaches the high ultimate value, then the micro-voids become bigger size to nucleate the cracks which leads to the fracture [47]. Close investigation of fracture surface exhibited fracturing of hard particulates rather than the de-bonding. The de-bonding of reinforced particulates usually occurs in MMCs where it is very hard to achieve better bonding among base material and the reinforced particulates [31]. So, the application of applied load led to development of cracks near the interface as well as removal of the whole reinforcement/s in that region. But in current research work, because of better bonding attained via stircasting of TiB_2 and CeO_2 particulates and hot rolling results in good interface. The results of better interface are revealed due to the witnessing of particulates fracturing.

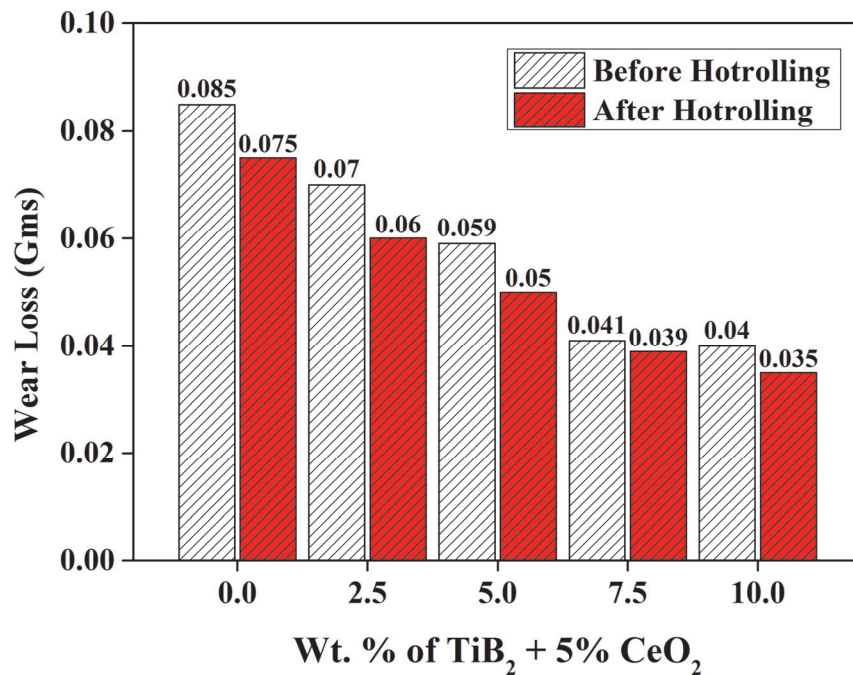


Figure 7: Wear loss of monolithic, hybrid composites and hot rolled hybrid MMCs

Wear Loss

Wear test was conducted as per the standards (ASTM). In the present research the tests were executed at the sliding-speed of 2 m/s and constant load of 30 N against steel disc (Grade: EN-32). The test samples of 5 mm thickness and 8 mm of diameter were prepared by wire EDM process. The wear loss of ascast, hybrid and hot rolled hybrid composites was determined by weight loss method. Fig. 7 depicts the wear behavior of TiB_2 and CeO_2 reinforced Al hybrid composite. The addition hard particulates increase the van-der-wall forces inside the matrix material. Generally, this leads to reduction of the dislocations and results in high wear resistance [36]. The decrease in wear loss may lead to capacity of higher load bearing of hard particulates. It can be observed that the hybrid composites exhibits lower weight loss indicating the effect of addition of TiB_2 and CeO_2 particles. It reveals that hot rolled hybrid composites show lesser wear loss when compared to monolithic and un-rolled hybrid composites. This is due to increase in hardness of the developed MMCs with the extent of hot rolling and also the due to the abrasive nature of TiB_2 and CeO_2 particulates. The finer dispersal of the hard fragmented particles generally strengthens the hybrid composite [48]. The wear resistance of the hybrid MMCs and hot rolled composite with 7.5% and 10% of TiB_2 is almost same. It is due to high hardness of MMCs experiencing high wear resistance in 7.5% and 10% of TiB_2 . The sliding wear tracks on the test samples were examined by SEM analysis. The SEM analysis of the wornout surfaces was done to study the effect of the hard particulates on the wear characteristics of the developed MMCs and hot rolled hybrid composites. The wornout surfaces of the SEM images of test samples (10% TiB_2 and 5% of CeO_2) of both hybrid and hot rolled hybrid composites are depicted in Fig 8.

It is observed in Fig. 8 (a&b) that, the existence of grooves of different sizes was seen on the SEM images wornout surface. The worn-debris particulates are likely to act like a third body abrasion particulates. The TiB_2 and CeO_2 particulates trapped among the test samples and counter face caused micro ploughing on contact surface of the developed hybrid MMCs. The wear surfaces were characterized by the significant transfer of the material among the sliding surfaces. TiB_2 and CeO_2 spreading inside the matrix material with good bonding results in higher wear resistance. High debris can

be observed in the tracks of hybrid composite in Fig. 8 (a) while Fig. 8 (b) shows uniform sliding wear track with the reasonable lower debris. The lower wear loss of hot rolled hybrid composites may be due to the fact that material is denser, good interfacial bond between the particulates and the base matrix than in the un-rolled hybrid composites samples. Also it is found that in the hot rolled hybrid composite reinforced up to 10wt. % TiB_2 and 5wt. % CeO_2 with a constant load and sliding speed, there is less fracture initiation at the matrix and particulates interfaces [48].

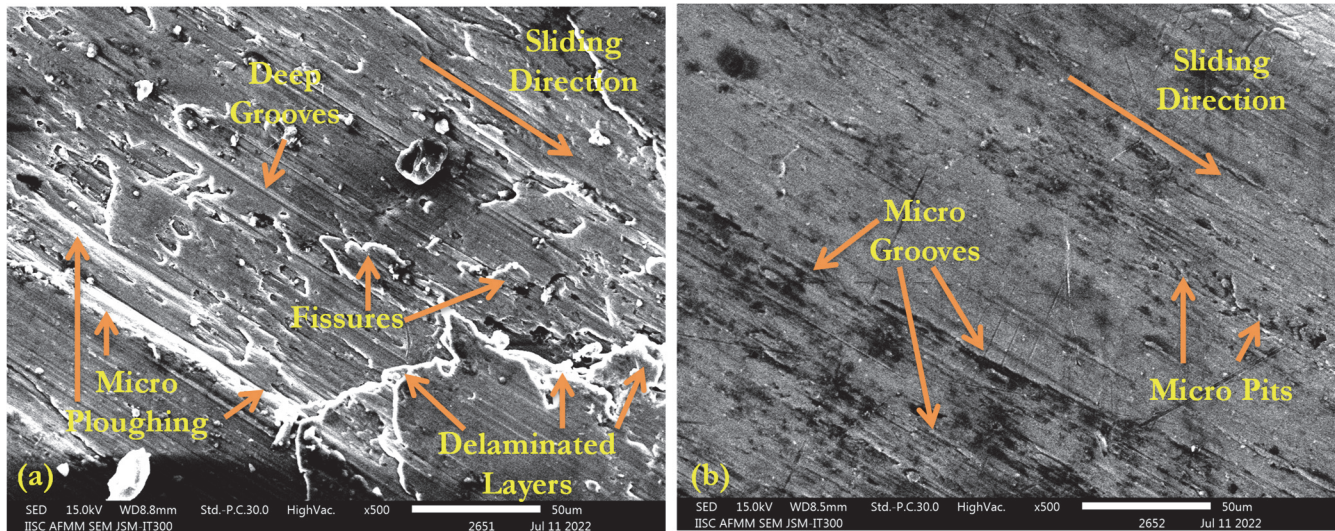


Figure 8: SEM fractography of worn-out surface of (a) 10% TiB_2 and 5% CeO_2 hybrid composite (b) 10% TiB_2 and 5% CeO_2 hot rolled hybrid composite.

CONCLUSIONS

The research works represents an investigation of micro-structure, mechanical and wear characteristics of monolithic, ascast and hot rolled hybrid MMCs. The outputs from the present work are as follows:

- Al-6061+ TiB_2 + CeO_2 hybrid MMCs have been manufactured by stircasting method and also effectively hot-rolled under the temperature of $515^\circ C$.
- Microstructure study reveals the uniform dispersal of TiB_2 + CeO_2 particulates with better bonding between reinforcement and matrix under both ascast and hotrolled conditions.
- Hotrolled MMCs showed enhancement of 25 % of hardness when compared with monolithic alloy with increasing reinforcement of 2.5 - 10 wt. % of TiB_2 content.
- Tensile strength increases by 53.54 % for hot rolled composites when compared to the as cast and other hybrid composites.
- Ductility decreased with the increasing in wt. % of TiB_2 and CeO_2 in both ascast and hot rolled conditions. Hot rolled hybrid MMCs show better enhancement in the ductility as compared to monolithic alloy.
- High wear resistance is found in both MMCs and hotrolled MMCs as compared to monolithic alloy.
- Fractography outcome shows the internal fractured structure of a tensile and wear specimen which was analysed using a SEM analysis.

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