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ORIGINAL PAPER

Morphology Studies on Mechanically Milled Aluminium Reinforced with B_4C and CNTs

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Abstract
Carbon nanotubes (CNT) with exceptional mechanical and physical properties are the prime candidate materials for metal matrix composites. But the dispersion of CNTs in the metal matrix is affected by attractive van der Waals forces. In this study, we have adopted ultrasonication and mechanical alloying to achieve homogenous dispersion of multiple reinforcements namely B_4C and CNTs within the commercial purity aluminium powder. The ultrsonication of all the starting materials was followed mechanical alloying process which was carried out in a planetary ball mill up to 480 min. The milling process was systematically studied by taking a small amount of milled powder at different milling time of 60, 120, 240 and 480 min respectively. The effect of ultrasonication and mechanical alloying on both morphology of the powders and dispersion of B_4C and CNTs was studied using scanning electron microscopy. The results showed uniform dispersion of both B_4C and CNTs within the pure aluminium powders without any agglomeration. Both the pure aluminium and hybrid nanocomposite powder showed decrease in particle size from 20.89 μm to 5.67 μm and 2.72 μm respectively after 480 minutes of ball milling.

Keywords Carbon nanotubes · Composite materials · Mechanical alloying

1 Introduction

In the recent years there has been lot of work being carried out in the area of metal matrix composites with nano-size reinforcements. With the decrease in the reinforcement size ($<$ 100nm) the mechanical properties of most the metal matrices are found to increase significantly [1]. Most of the commonly used nano-size reinforcements are carbon nanotubes [2], SiC [3], TiB_2 [4], ZrO_2 [5], TiO_2 [6] and B_4C [7]. Discovery of carbon nanotubes by Japanese scientist Iijima in the year 1991 has opened up endless opportunities in the area of materials science [8]. The carbon nanotubes (CNTs) either single walled – single tubule or mutliwalled – a centre tubule surrounded by multiple numbers of graphitic layers have large aspect ratios, high tensile strength and elastic modulus. The exceptionally high stiffness of 1 TPa and tensile strength over 150 GPa are mainly attributed to the sp^2 hybrid bond. In addition to this, these nanostructures have extremely low coefficient of thermal expansion ($\sim 0 \times 10^{-6}$/K) and very high thermal conductivity of 3000–6000 W/mK [9–11]. In order to utilise the extraordinary mechanical and physical properties, CNTs have been used as reinforcement in polymer, metal and ceramic matrices to develop high performance and multifunctional composites. In particular CNTs have been explored for reinforcing light weight metal matrices like aluminium, magnesium and titanium [12–14].

Using CNTs as a potential reinforcement for metal matrices was started by Kuzumaki et al. [15], where they reinforced aluminium matrix with CNTs. Likewise till today CNTs has been used to reinforce many metal

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matrices which includes copper, magnesium, titanium and nickel. These CNT reinforced nanocomposites have been fabricated by many processing routes, like electrochemical method, powder metallurgy, thermal spraying and melting techniques. Out of all above stated processing technique, powder metallurgy technique is the one mostly used for development of nanocomposites. Choosing the appropriate processing technique for developing nanocomposite is the most important step as its control microstructure and properties. However the major challenge in the development of nanocomposites is to obtain a uniform distribution of CNTs in the metal matrix. The CNTs tends to form clusters or bundles due to strong Van der Waals force of attraction. Mechanical alloying or Ball milling technique, molecular level mixing or growing CNTs on metal powders through chemical vapour deposition are different techniques used for ensuring better dispersion of CNTs in the metal matrices [16].

Addressing the dispersion issues, Esawi and Morsi [17] carried out detailed studies on dispersion of CNTs in aluminium powder using mechanical alloying technique. With a milling speed of 200 rpm, the aluminium and CNTs were milled up to 48 hours. The effect of milling on powder morphology and CNTs dispersion was studied using field emission scanning electron microscope (FESEM). The results revealed that the mechanical alloying technique was effective in achieving uniform dispersion on CNTs without causing much damage to the CNT structure. In another work Varol and Canakci [18, 19] opted mechanical alloying technique to disperse the two different particle size of B_4C in the Al2024 metal matrix. The morphology and structural evolution studies after 10 hours of mechanical alloying suggests that with the increase in milling time the particle size of Al2024/B_4C composite powders tend to decrease from 75 μm to 7 μm. The crystallite size of unreinforced Al2024 and composite powders with 20 wt% B_4C before milling and after 10 hours of milling was found to be 122 nm and 18 nm respectively and the grain refinement achieved was attributed to mechanical alloying technique. Yang et al. [20] report a novel approach in which nickel catalyst was deposited on aluminium powder surface by impregnation route and CNTs were grown on these aluminium particles by passing a mixture of CH_4/Ar into the CVD reactor. FESEM images showed the bamboo like structure CNTs were homogenously dispersed in aluminium powder. Cha et al. [21] developed a unique technique known as molecular-level mixing for achieving better dispersion of CNTs inside the copper matrix. The technique involved functionalization of CNTs, addition of salt containing copper ions in the stable CNT suspension, drying the solution at 100-250 °C in air and finally step was calcination and reduction of powders under hydrogen atmosphere. Transmission electron microscopy micrographs showed uniform dispersion of CNTs along with good bonding between copper powder particles. In their work, Mallikarjuna et al. [22] used ball milling for better dispersion of dual reinforcements namely CNTs and SiC in the copper matrix. The ball milling was carried out at 300 rpm with ball to powder ratio of 8:1. The TEM studies of nanocomposite showed uniform dispersion of CNTs with no visible clustering.

Although a large number of research work have been conducted on mechanical alloying of aluminium with single reinforcement but very limited studies have been reported concerning with mechanical alloying of aluminium with multiple reinforcements. The aim of this paper is to study the effect of ultrasonication and ball milling on the powder morphological development and dispersion of multiple reinforcements namely, B_4C and CNTs in the aluminium matrix. The evolution of powder morphology at different milling time was studied using scanning electron microscopy.

2 Experimental Details

Information of the raw materials used in the current study is given Table 1. Figure 1a and b shows the scanning electron microscopy (SEM) micrographs of as received B_4C particles and aluminium powder and Fig. 1 shows the transmission electron microscopy (TEM) micrograph of as received multiwalled carbon nanotubes (CNTs). To ensure better dispersion of both reinforcements B_4C and CNTs, a two-step method was adopted. First the CNTs (0.25 g) were added to ethanol (500 ml) and ultrasonicated for 30 minutes. Once the CNTs were suspended in the solution, the second reinforcement B_4C (2.5 g) was added. The dual reinforcements were ultrasonicated for another 30 minutes to ensure that both them are suspended in solution rather than settling down. Finally aluminium powder was added to this slurry and the ultrasonicated for 60 minutes. The

Table 1 Details of raw material used for the development of hybrid composite powder

Raw material	Size, μm	Density, g/cm^3
Aluminium	~ 20	2.7
Boron carbide (B_4C)	~ 50	2.5
Multiwalled carbon nanotubes (CNTs)	Dia 20-40 nm and length – 10-20 μm	2.1

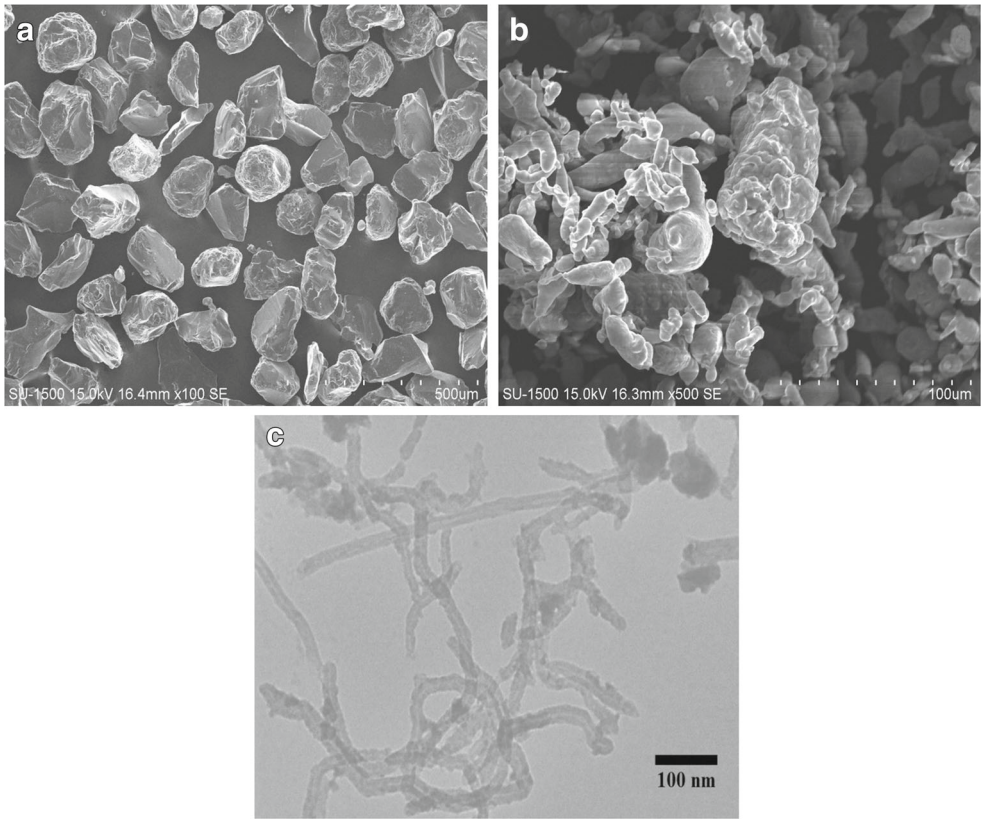

Fig. 1 SEM images of as received **a** B_4C particles, **b** aluminium powder and **c** TEM image of CNTs

ultraonicated wet mixtures of powders were dried in vacuum oven at 60 °C for 480 minutes.

In second step the dried hybrid nanocomposite powder was subjected to ball milling in a planetary ball mill (Make: Insmart Systems, India). The powders were loaded in 250 ml stainless steel jars with stainless balls and milling was carried out at 200 rpm with a ball to powder ratio of 6:1. The milling was carried for duration up to 480 minutes in presence of 1.5% of process control agent (PCA). The ethanol which was used as PCA was added to avoid excessive cold welding of aluminium powders during milling process. After 30 minutes of running the ball mill was given an interim period of 15 minutes to avoid overheating. The effect of milling time on powder morphology and the particle size was studied by taking small amount of hybrid nanocomposite powder at different milling time of 60, 120, 240 and 480 minutes respectively. The powder morphology and distribution of both the reinforcement in the milled nanocomposite powders were characterised using a scanning electron microscope.

3 Results and Discussion

The average particle size of as-received commercial aluminium powder particles was found to be (D_{50}) 20.89 μm. The aluminium powder particles are having ligament and irregular morphology and the B_4C ceramic particulates were found to be angular or polygonal in shape. The as received CNTs as shown in Fig. 1c were found to be agglomerated due to strong Vander Waal's force of attraction between the CNTs. The TEM image of CNTs shows that nanotubes were having no impurities such as Fe or Ni nanoparticles. The tubular structure was clearly seen in the TEM micrograph. We will see evolution of particle size and morphology of pure aluminium and hybrid nanocomposite powder comprising of both B_4C and CNTs after different milling time of 60, 120, 240 and 480 minutes in the next sections.

3.1 Evolution of Al Powder Morphology

It is well known that during the ball milling process the powders are subjected to ball to powder collision leading to severe plastic deformation, cold welding and fracturing of powders. During ball milling the centrifugal force is produced by stainless steel vial and that by supporting disc on starting materials. The aluminium powder particles having irregular and ligament shape at the initial stage were starting changing into flakes after 60 minutes of milling. The predominant mechanism at this stage is the plastic deformation, which will start flattening of powder particles

Table 2 Particles size at different milling time

Material	Milling time (minutes)				
	0	60	120	240	480
Aluminium (μm)	20.89	19.08	15.27	10.67	5.67
Hybrid nanocomposite (μm)	20.89	17.32	13.51	7.91	2.72

which will lead to forming of flake like morphology with smooth surfaces due to impact of stainless steel balls used for ball milling. It's worth noting that the use of PCA in our work was successful in avoiding the cold welding of ductile aluminium particles. The powder yield in most of the cases is limited to 10% as observed by Ye et al. [23]. This is mainly due to adhesion of aluminium powder particles on the walls of vial and balls used for milling. In order to avoid cold welding and achieve a balance between fracturing and welding of aluminium powder particles, 1.5% of ethanol was used as PCA. The PCA get dispersed evenly as soon as the ball milling process is started and cover all the aluminium particles forming a barrier for cold welding, which prevents cold welding of aluminium powder with inner wall of stainless vial as well as stainless ball thus improving the powder yield. The average particle size of both aluminium and hybrid nanocomposite powder at different milling time is given in Table 2. The particles size after 60 minutes of ball milling was found to be reduced to 19.08 μm from 20.89 μm in the starting stage. The flake like morphology of aluminium particles were still detected even after 120 minutes of milling time (Fig. 2b) but it was observed that there was decrease in particle size to 15.27 μm. Further with the increase in milling time to 240 minutes, fracturing of flaky particles was noticed due to repetitive collusion of stainless balls and aluminium particles as shown in Fig. 2c. The mechanism operating at this stage was fracturing and initiation of formation of equiaxed particles whose size is was found to be 10.67 μm. It can be seen that the decrease in particle size was quite significant as the particle size at this stage of milling almost half of the starting stage. Further after 480 minutes of ball milling almost all flaky particles were broken. Cold welding of small fragments went on to form relatively equiaxed particles of size of about 5.67 μm which is quite evident from Fig. 2d. Here the role of ethanol is very crucial and important in reducing the particle size of aluminium particles. This is mainly due to free movement of ethanol molecule in aluminium powder particles especially through the cracks formed during milling process. This free movement of liquid ethanol all over the powder particles

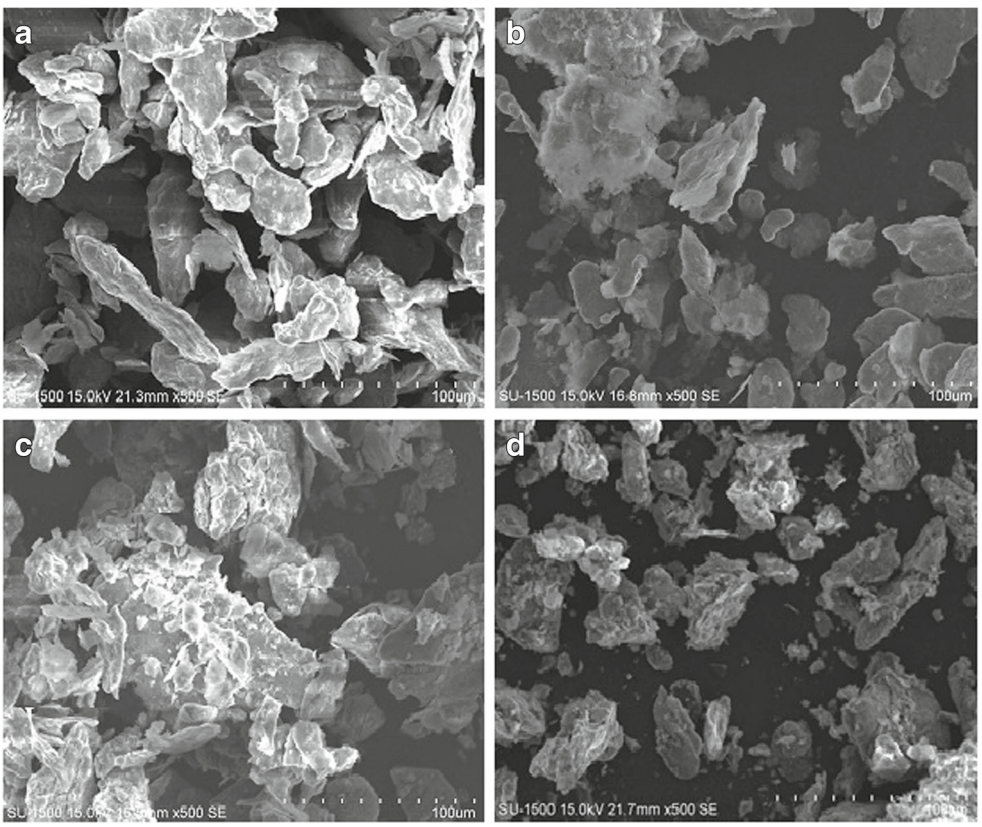

Fig. 2 SEM images of aluminium powder particles after a milling time of **a** 60 minutes, **b** 120 minutes, **c** 240 minutes and **d** 480 minutes

make sure that the there is no excessive cold welding of ductile aluminium particles. However as the milling proceeds from 240 minutes to 480 minutes the possibility of evaporation of ethanol is high since its melting point is close to that of 159K, so we can observe cold welding of particles leading to the formation relatively equiaxed morphology. Further at higher milling times, there occur a state of equilibrium between fracturing and cold welding at which milling would be stopped [24].

Figure 3a and b shows the elemental mapping of aluminium powder after 60 and 480 minutes of ball milling to find out if there is any kind of contamination or formation of oxides. The elemental mapping was done exactly on the aluminium particle to cheque the presence of any foreign element which could iron from the milling media such as stainless vial or the balls or it could oxygen from the milling atmosphere. Either the case both the elements are detrimental for both mechanical and physical properties especially thermal and electrical conductivity. It can be observed from the Fig. 3a and b that in both the cases only aluminium element mapping is seen and no foreign element are observed which explains the ball milling parameters used were efficient enough to avoid any kind contamination either from atmosphere or milling media. Here the use of ethanol as process control agent not only serve in reducing the particle size but also avoid possible oxidation of aluminium powder particles during milling process.

3.2 Evolution of Hybrid Composite Powder Morphology

A uniform dispersion of both the reinforcements B_4C and CNTs in the aluminium matrix is crucial to achieve hybrid nanocomposites with improved mechanical properties. In case of ball milling, the milling time and the concentration of both the reinforcements have huge influence on the powder morphology and particle size of the hybrid nanocomposite powders. During the initial stage of milling process the aluminium powder particles started flattening and morphology changed to flaky similar to that of monolithic aluminium. However, the B_4C particles did not undergo any noticeable change and retained their original polygonal shape up to 60 minutes of ball milling as shown in Fig. 4a. Since the coarse B_4C particles were not deformed, most of them were clearly visible were seen in between the flaky aluminium particles rather than embedding in them (indicated with an arrow in Fig. 4a). The same phenomenon was observed by Varol and Canakci in their work on aluminium 2024-B_4C composite [25]. The presence of CNTs and B_4C particles decreased the weldability of aluminium particles resulting in the formation of small flakes of aluminium. Figure 4b shows the hybrid nanocomposite powder after 240 minutes of milling. It is observed that the presence of hard ceramic particles increases the local plastic deformation of the

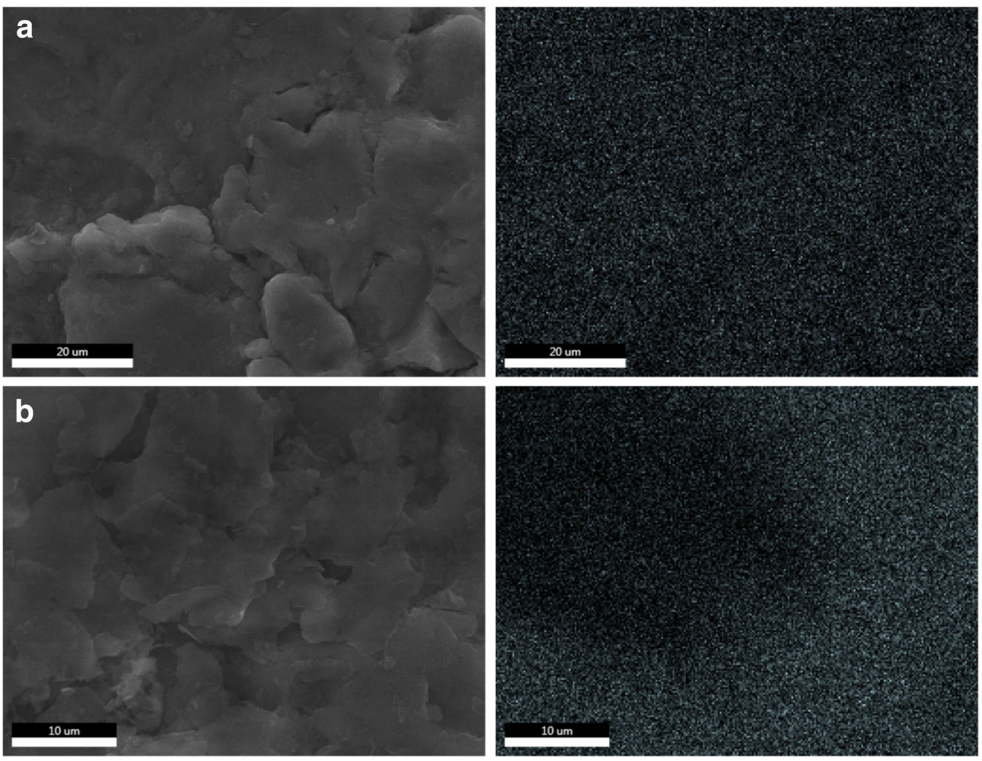

Fig. 3 Elemental mapping of aluminium powder particle after **a** 60 minutes and **b** 480 minutes of ball milling

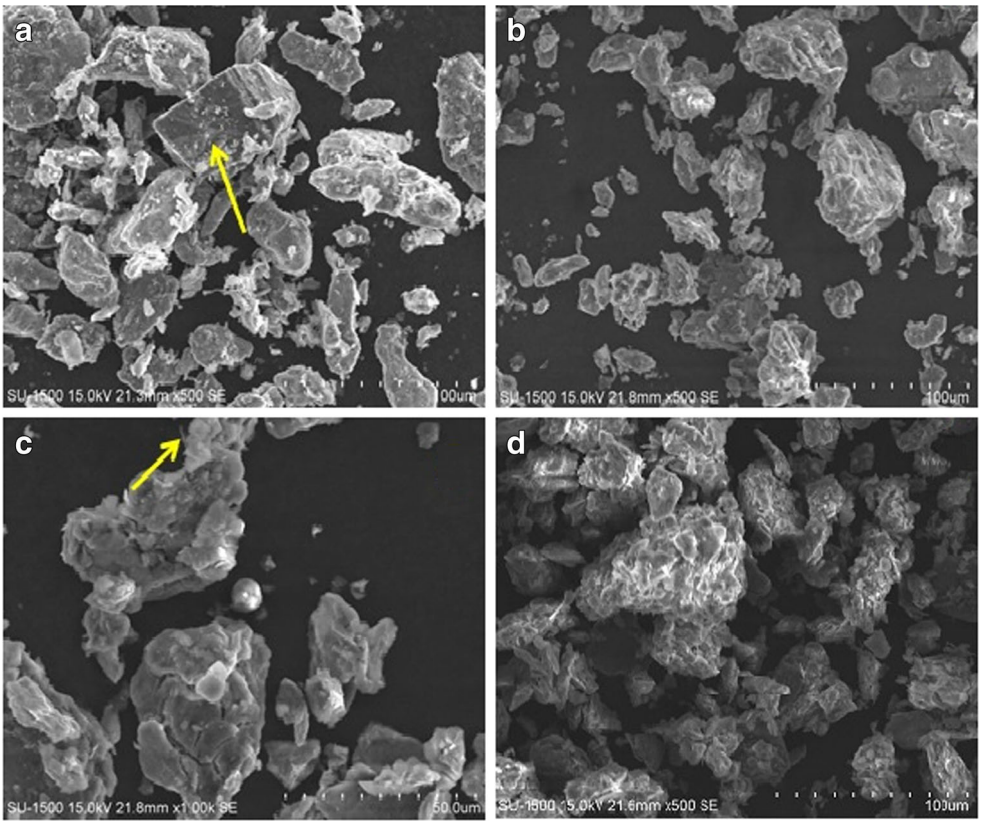

Fig. 4 SEM images of hybrid nanocomposite powder particles after a milling time of **a** 60 minutes, **b** 120 minutes, **c** 240 minutes and **d** 480 minutes

aluminium enhancing the work hardening rate. When the work hardening of these particles reaches a critical value the fracturing process starts. This indicates these B_4C particles are acting as milling agents, starting the fracture process much earlier than observed in milling of monolithic aluminium powder [26]. In addition to this, in early stage of milling of process both agglomerates of CNTs and B_4C particles are adhered to the aluminium matrix. Later during milling the cold welding leads to trapping of both reinforcements in the matrix. These trapped CNTs and B_4C particles help the cracks to propagate easier into the matrix. The initiation and propagation of cracks within the powder particles results in fracture and exposing the fresh fractured metallic surface. The fresh fractured surface of aluminium particles with CNTs and B_4C pinned on them would weld together with other surfaces due to repeated cold welding. At this stage of milling most of CNTs and B_4C particles were entrapped between cold welded aluminium powder particles. One thing to be noticed that the extent of cold welding is very less due to presence of ethanol as process control agent whilst in other cases were the ball milling is carried the extent of cold welding is very high [17]. It can be observed that there is significant decrease in particle size of hybrid nanocomposite powder after 60 and 120 minutes of ball milling. The particle size dropped from 20.89 μm in the starting stage to 17.32 μm and 13.37 μm after 60 and 120 minutes of ball milling respectively.

The dispersion of CNTs appeared to uniform without any noticeable damage to their structure after 240 minutes of milling as shown in the Fig. 4c. Similarly no evidence of agglomeration of B_4C particles was observed indicating homogenous distribution in the aluminium matrix. At this stage of milling process hybrid nanocomposite particles begin to have equiaxial morphology demonstrating fracturing as a dominant mechanism. The formation of partially equiaxed particles indicate that due to the presence of CNTs and B_4C particles, the milling process is progressing towards the equilibrium state at much shorter milling time. Few individual CNTs were seen near the edge of the fractured hybrid nanocomposite powder (as indicated by arrows) [27]. Unlike in other works [17, 28] were with the increase in ball milling time the agglomeration of particles was noticed, the present work shows no such agglomeration due to the use of PCA. The decrease in the particle size observed can also be attributed to CNTs and B_4C which retards the formation of large particle size. As it observed in other works the CNTs and B_4C does influence the particle size by hindering the dislocations movement at the grain boundaries and inhibiting the cold welding process during further milling process. The observations on 480 minutes milled hybrid nanocomposite powder revealed the formation of equiaxed morphology of particles suggesting sufficient milling time has already reached the steady state equilibrium. It can be observed from the SEM micrograph that

none of the two reinforcements were visible on the surfaces hybrid nanocomposite particles, which implies that both of them have been incorporated inside the aluminium matrix as result of high energy milling [29]. Since the absence of any free CNTs or B_4C particles, it can be presumed that a good bonding between matrix and reinforcements is achieved. These uniformly dispersed reinforcements in the aluminium matrix will ensure improved densification of the hybrid nanocomposites. This confirms that full hybrid nanocomposite powder can be obtained with the use of two

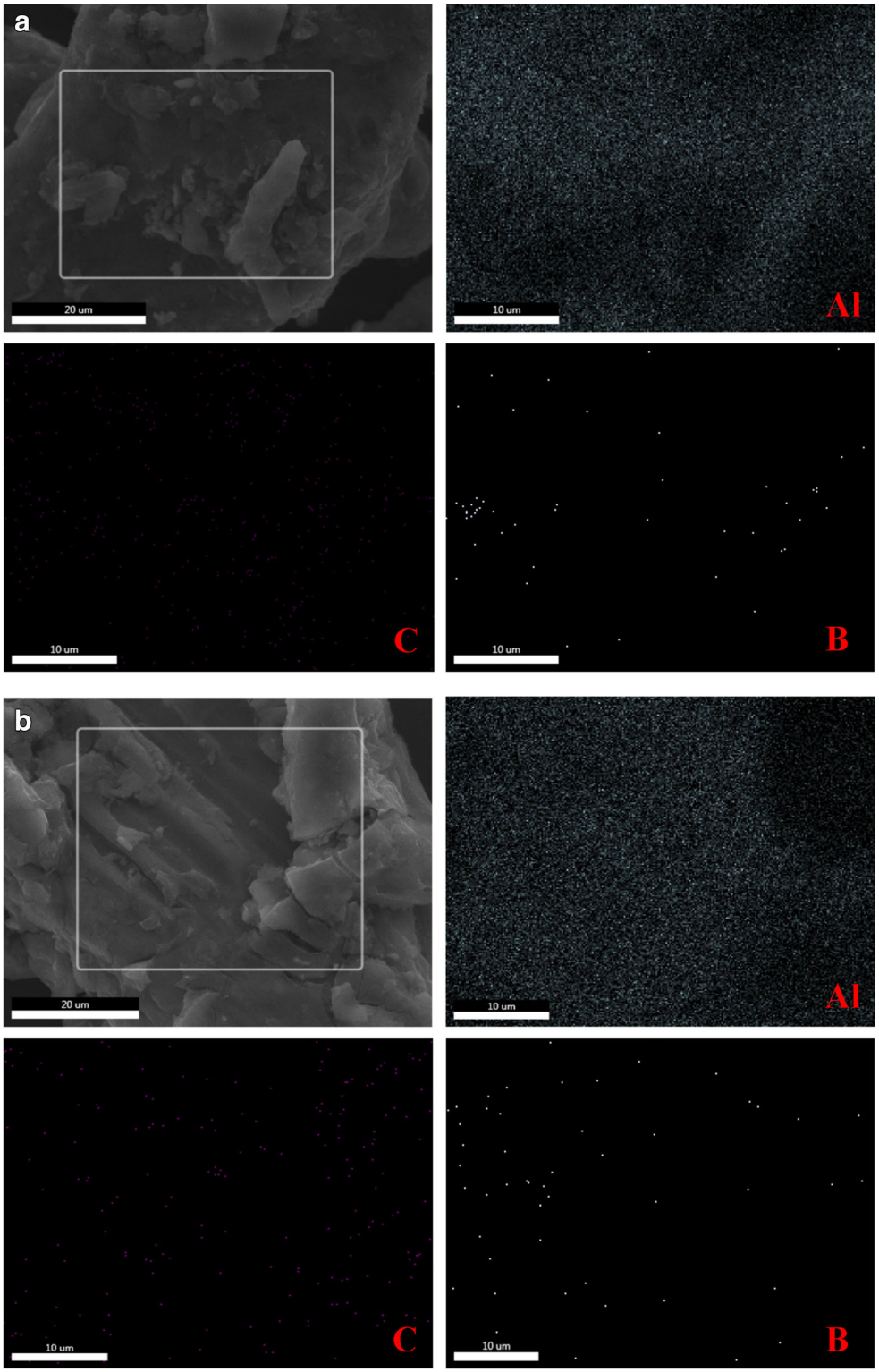

Fig. 5 Elemental mapping of hybrid nanocomposite powder particle after **a** 60 minutes and **b** 480 minutes of ball milling

step method. Further the particle size of hybrid nanocomposite powder dropped to 7.91 µm and 2.72 µm after 240 and 480 minutes of ball milling. The decrease in particle size can be attributed due to increase in fracture tendency from hard B_4C particles and the process control agent ethanol. Further the ball to powder ratio chosen in the present work was found to be appropriate since significant reduction in particle size in both the cases was observed without any overheating or contamination. Both the reinforcements were found to be dispersed in aluminium matrix uniformly without formed any clusters. This is mainly because high ball to powder ratio can result low impact energy of milling media as well as leads to agglomeration of reinforcements.

The elemental analysis carried out on hybrid nanocomposite powder particle after 60 and 480 minutes of ball milling is shown in Fig. 5a and b. It can be observed that the elemental mapping consists of three main elements corresponding to aluminium, boron and carbon. Both the carbon and boron elements which correspond to the reinforcements CNTs and B_4C were found to be uniformly distributed on the aluminium particle surface. In both Fig. 5a and b, the presence of carbon and boron in confirmed but the concentration of both the reinforcements was low. It is interesting to note that the oxygen and iron were absent in the elemental mapping at both starting and end stage of ball milling indicating that no contamination has occurred. Further Fig. 6a and b shows the X-ray diffraction pattern of the pure aluminium and hybrid nanocomposite powder after 480 minutes of ball milling. It can be observed that both the X-ray patterns showed five peaks corresponding to that of aluminium. These five peak corresponds to Al (1 1 1), Al (2 0 0), Al (2 2 0), Al (3 1 1) and Al (2 2 2) FCC phase of aluminium. However in case of Fig. 6b, the hybrid nanocomposite, no peaks corresponding to B_4C

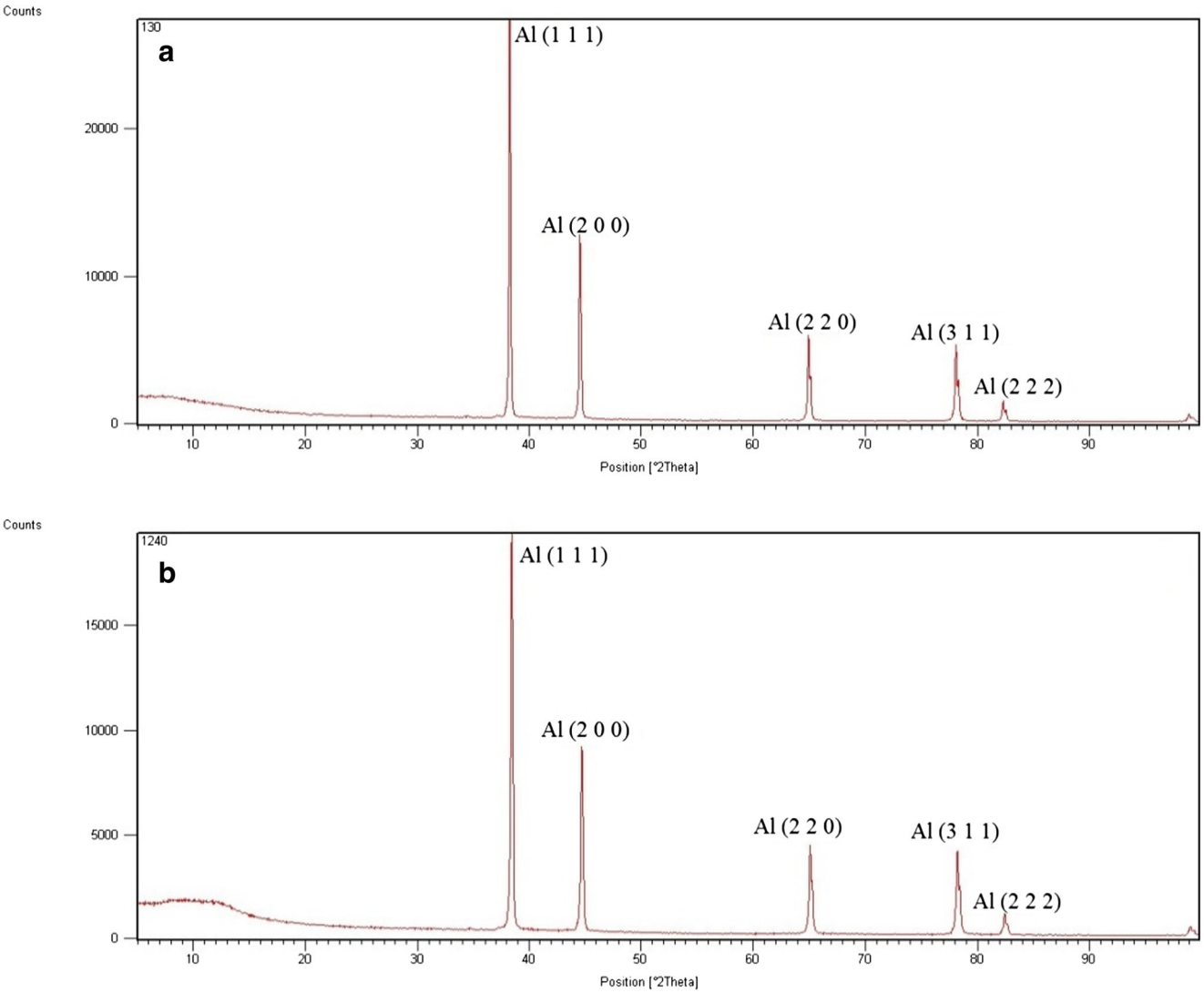

Fig. 6 X-ray diffraction patterns of **a** pure aluminium and **b** hybrid nanocomposite powder after 480 minutes of ball milling

and CNTs were observed. The absence of peaks related to B_4C and CNTs can be mainly due to their low concentration. Along with this both the reinforcements are embedded in between the aluminium particles which pose difficulties in obtaining their corresponding peaks. Similar observation was reported Basariya et al. [29] where they could observe peaks related to aluminium only but none related to CNTs which was due to its low concentration. The X-ray diffraction also confirms that no compound related to Al_2O_3 or Al_4C_3 are formed after a prolonged ball milling of aluminium or hybrid nanocomposite powder. However Pérez-Bustamante et al. [30] observed formation of Al_4C_3 in Al2024/CNT composites after a milling duration of 30 hours. The formation of such carbides/oxides usually results in the poor mechanical properties of composites.

4 Conclusions

A two-step method involving ultrasonication and ball milling technique was employed to achieve a uniform dispersion of two reinforcements namely CNTs and B_4C particles inside the aluminium matrix. The agglomerations of both the reinforcements was avoided and were found to be embedded inside the aluminium matrix without any defects by selected milling intensity of 200 rpm and ball to powder ratio of 6:1. In addition to cold welding of hybrid nanocomposite particles was prevented by the use of process control agent which in turn improved the yield of powder particles after the completion of milling process. The increase in the milling times the particle size of both aluminium and hybrid nanocomposite powder was found to decrease. The decrease in the particle size in case of pure aluminium was attributed to presence of ethanol as process control agent and in case of hybrid nanocomposite powders the presence of reinforcements which increase the fracture tendency by increasing the work hardening rate help in decreasing the particle size. Finally the X-ray diffraction studies showed that the unwanted oxides/carbides like Al_2O_3 or Al_4C_3 are not formed after ball milling of both aluminium and hybrid nanocomposite powder.

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