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# An Improved Compocasting Technique for Uniformly Dispersed Multi-walled Carbon Nanotube in AA2219 Alloy Melt

*Technology transfer for economic bulk production is the greatest challenge of the era. Production of high strength lightweight materials with nano-carbon reinforcement has attained its importance among the researchers. Property enhancement with multi-walled carbon nanotube (MWCNT) reinforcement is reported by all researchers. But effective utilization of its property remains a challenge even though it is the strongest material in the world. Achieving homogeneous dispersion especially in molten metal is a complex task. To address the same, a new approach was tried which could trigger de-bundling and make a uniform dispersion. Various metallurgical and mechanical characterizations were done. Grain refinement and the structure were studied with an optical microscope, MWCNT dispersion and structural damage was studied using field emission scanning microscope, Phase change and reactions during casting was done with XRD scan. The method remarkably facilitated 23.7% and 69.75% improvement in hardness and ultimate compressive strength respectively with the addition of MWCNT.*

***Keywords:*** *Casting, composites, aluminium, alloys, nanotube, MWCNT, nanocomposites, compocasting.*

## 1. INTRODUCTION

Since 2007 a new trend has been observed in MWCNT reinforcement with lightweight materials [1,2]. Among the lightweight materials aluminium alloy (AA) and magnesium alloys got more attention. Strong Van der Waals property of carbon nanotubes (CNT) is the major challenge in processing. Bulk material processing with low cost by liquid processing requires more attention.

Researchers have put the effort in various casting routes [3-15], but limited literature is available for aluminium (Al) – MWCNT composites by casting. Mansoor et al. [16], reported an instantaneous enhancement in tensile strength (52%), yield strength (77%), hardness (45%) and ductility (44%) on 0.2vol.% MWCNT reinforced aluminum composite, which is produced by induction melting. Abbasipour et al. [17], made use of stir casting and compo casting routes to produce aluminum alloy reinforced 2wt% MWCNT. Unlike the usual method in which raw MWCNTs are directly injected into the melt, it makes use of Ni-P electroless plating technique to deposit nanotubes on aluminium powder initially and then inject into a melt. It is reported a 21% improvement in hardness by MWCNT addition. Giridhar et al. [18], remarked that on the addition of 0.5wt% MWCNT into aluminium alloy (AA6061) melt by the ultrasonic probe exhibited 12% increment in hardness and 27% increment in tensile strength without any significant difference in ductility. Q. Li et al. [19], discovered that Al-MWCNT metal matrix composites (MMCs) by high pressure die casting with addition of only 0.05wt% reinforcement shows the elongation at fracture has increased 27% compared to pure AA; the tensile strength of CNT/Al composites increased 8% compared to pure AA. Sehyun Ko et al. [20], added a CNTs-Al powder precursor into an Al melt through the casting process to make Al-CNT MMCs. For enhancing wettability, milled powder surface was coated with Ni by an electroless plating method. But observed strength and hardness by the above researchers are comparatively lower than what was achieved in powder metallurgy (PM) route. Even if it's reported that PM [21,22] is the most adequate process for making Al-CNT composite, an effective attempt has done to achieve uniform distribution in liquid processing methods as it is the cheapest route in production of bulk composites.

## 2. MATERIALS AND METHODLOGY

The chemical composition of the AA2219 alloy used for present work is listed in Table 1. MWCNTs supplied by Redex India having purity >97%, inner diameter 5nm and bulk density 1.3 g/cm$^3$ and aspect ratio ~ 1000 as shown in fig.1 were used in the present work. Fig.1 (a, b) indicates the FESEM images of MWCNT. The entaglement of multi wall nano tubes are seen with very tight compaction. XRD image in fig.1 (c) indicates the peak at 26.140C and another peak at 44.220C and the rest of the peak to be uniform in nature which predicts it

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to be of crystalline nature. Similar peaks were obtained by Mohamed Abdel Salam et.al., [23] in their study of synthesis and charecterization of MWCNT.

**Table 1. Chemical composition of the AA2219 alloy used in wt%.**

| Al | Cu | Si | Mn | Fe | Zn | Ti | Zr |
|---|---|---|---|---|---|---|---|
| Balane | 5.56% | 0.07% | 0.28% | 0.08% | 0.01% | 0.07% | 0.18% |

Homogeneous dispersion of MWCNT in a metal matrix is the major issue in achieving Al-MWCNT metal matrix composites by liquid processing route [24]. The low density of carbon nanotubes (CNTs), causes dispersion difficult in molten metal. At the melt temperature of 750°C and above, which is the processing temperature of aluminium alloy, the wetting angle between MWCNT and Al is increased above 160° [1]. The poor wettability of carbon by aluminium is the hindrance in casting route to fabricate the nanocarbon reinforced aluminium composites. The issue is a dispersion of MWCNT in metallic melt and the MWCNT stability due to strong reactivity of carbon with oxygen is also critical. All these factors make the fabrication of the CNT reinforced aluminium alloy matrix composites a difficult task resulting in poor mechanical properties. To improve the wettability of MWCNTs and to reduce their agglomeration, a new technique was used.

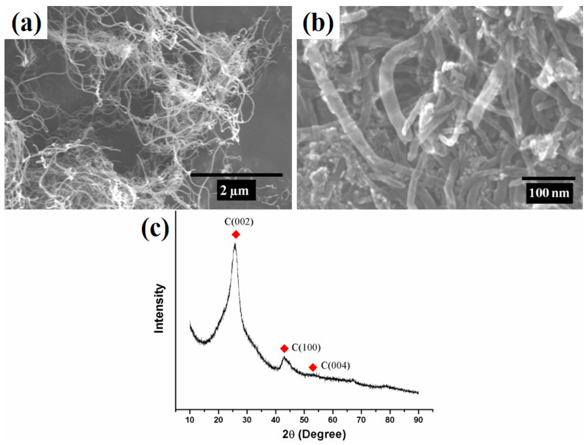


**Figure 1. (a-b) FESEM image (c) XRD scans of the MWCNT as received used in the present study.**

In the present work, MWCNT is made to deposit in AA2219 powder and form blocks (pellets) as shown in fig. 2. The aim was to deposit MWCNT pellets into molten AA2219 instead of raw MWCNT powder which helps in gradually release the MWCNTs in the molten matrix alloy during powder melting. For that as an initial step raw bundle MWCNT (as received) is added to a highly purified ethanol solution. It undergoes ultrasonication for 30 minutes, then AA2219 powder is added and allowed to sonicate for the next 30 minutes. MWCNT- AA2219 suspension undergoes magnetic stirring for next 30 minutes followed by drying in a hot air oven at 80°C. AA2219-MWCNT powder mixture undergoes high energy planetary ball milling for 30 minutes in cylindrical tungsten carbide jar with BPR 4:1 at a speed of 250 rpm. Varying amount of MWCNT (0wt.%, 0.5wt.%, 1wt%, and 2wt%) with 100 g of AA2219 powder mixture were made into blocks having a size of 25 mm x 5 mm using a Pelletizing machine as shown in fig. 2.

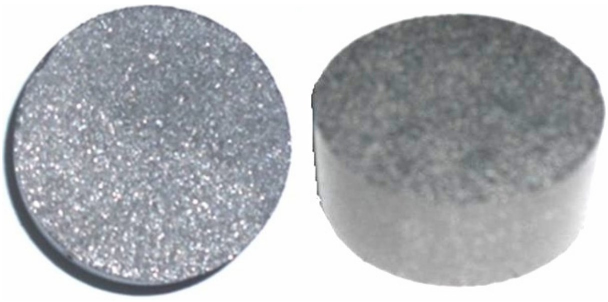

**Figure 2. AA2219 with MWCNT blocks (pellet).**

AA2219 in required quantity is cut into different pieces from a plate. It is then cleaned and weighed for four different samples. AA2219 alloy plates were kept in furnace and the temperature was raised to 750°C. High temperature inside the furnace enhances the wettability of the reinforcement particles in the matrix metal. MWCNT with AA2219 blocks (pellets) was preheated separately at a temperature of 250°C for 2h to improve wettability and eliminate dampness. Preheating of the reinforcement prevents thermal mismatch and results in improvement of wettability. Alloy sheet was heated to a temperature of 750°C±30°C (above the liquidus temperature of the alloy) and mechanically stirred at 350 rpm to ensure the alloy melts completely. 0.5wt% magnesium (help in improving wettability) and coverall (degasser) was added, which help in improving wettability. The preheated MWCNT pellets were charged into the molten metal at this temperature (750°C±20°C) and stirred by mechanical stirrer at 500 rpm for 5 min. Due to the temperature difference between molten alloy and reinforcement, a semi-solid molten metal is formed in crucible. Stirring a semi-solid molten material improves the dispersion of clustered reinforced particle with obtained shear force [25]. This semi-solid melt can mechanically entrap the reinforcing particles, prevent their gravity segregation and reduce their agglomeration [4,17,26], hence a better distribution of the reinforcement particles. The semi-solid composite mixture was superheated to 800 °C±20°C to make sure composite was in fully liquid, and stirred rotationally along with up and down using an automated mechanical stirrer. Before casting into the molds, the mechanical stirring was performed at 750 to 800 rpm for 10 min. Molten composite is poured into preheated (250°C) mould of the required shape. Schematic representation of the casting process is shown in fig. 3.

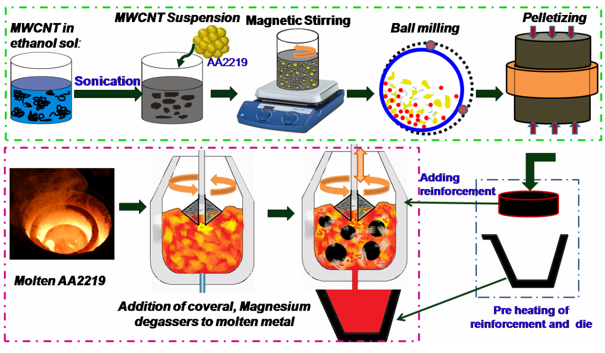


**Figure 3. Schematic representation of the casting process.**

Optical microscope (De-wintor inverted trinocular metallurgical microscope) and field emission scanning electron microscope (FESEM) (FESEM-SUPRA 55 - CARL ZEISS, GERMANY) was used to analyze morphology and MWCNT dispersion. X-ray diffraction (XRD) (using Panalytical, Netherlands) was used for phase analysis. The mechanical properties were investigated with microhardness tester under ASTM standard E384-16 using Vickers microhardness tester (Shimadzu microhardness tester, HMV - G20). Short Solid Cylindrical Specimens for Compression testing on cast samples was made as per ASTM E 9-89a. The specimen has a diameter (D) of 13.0 ±0:2 mm, Length (L) of 25 ±1 mm, and an Approx L/D Ratio of 2.0. Testing was done at room temperature using a SHIMADZU AG-X 50KN capacity universal testing machine. The crosshead speed was 0.254 mm/min, and the data were recorded automatically for load and strain every second. The reported property was the average of five tests.

**Table 2. Sample compositions and identification number.**

| Sl No: | Composition | Sample ID |
|---|---|---|
| 1 | Cast AA2219 with 0wt,% MWCNT (as received) | C AA 0CNT |
| 2 | Cast AA2219 with 0.5wt,% MWCNT | C AA 0.5 CNT |
| 3 | Cast AA2219 with 1wt,% MWCNT | C AA 1CNT |
| 4 | Cast AA2219 with 2wt,% MWCNT | C AA 2CNT |

## 3. RESULTS AND DISCUSSION

Optical microstructure analyses of AA2219 alloy with and without MWCNT processed under casting are presented in fig. 4. The presence of very huge amounts of solute in the alloy (5.56% Cu) and non-equilibrium cooling conditions lead to dendritic solidification and segregation to grain and subgrain boundaries. As shown in fig. 4 casted samples show dendritic microstructure and indicate grain refinement with and without reinforcement. No structural or size changes were observed between monolithic alloy and MWCNT reinforced composites during casting. Castings exhibit a dendritic structure that is well-organized (uniform) and homogenized. Addition of MWCNT doesn't make any drastic change in macrostructure. Usually, it is observed that the metal strength increases with a reduction in the grain size. From the Hall–Petch equation, it is evident that yield strength is proportional to reciprocal of the square root of grain diameter. But in the case of cast metals, the strength and the grain size need not always follow the Hall–Petch equation. The relation between the strength and grain size is not fulfilled if the amount of microporosity is increased with the production of small grain [27,28].

The factors that play a crucial role in the final properties of the cast metals are the size and shape of those dendrites. It is possible to change or break the dendritic morphology using some ways in the agitation of melt during the initial growth, thus resulting in semi-solid processing in which alloys are processed in the partially liquid and partially solid state. No breakage in dendrite structure was observed which indicates the efficiency in liquid processing. FESEM images of MWCNT reinforced samples (fig. 5) indicate the presence of MWCNTs. In the grain and dendrite boundaries, a considerable amount of $CuAl_2$ segregates in large eutectic particles (white colour regions showed in fig. 5(a). A similar surface of cast monolithic alloy was observed for the samples even for increased concentration of MWCNT. But concerning an increase in reinforcement concentration, some amount of MWCNT was observed inside the porous region. From 0.5wt.% MWCNT reinforced sample (fig. 5 a) no MWCNT could be observed on the surfaces even in the porous region. In casting the MWCNT clusters were absent on grain boundaries as like reported [21] in PM route. But MWCNT was observed inside the walls of porosity having a reinforcement concentration above 0.5wt%. Figure 5(b-e) shows cast AA2219 alloy with 1wt.% and 2wt.% MWCNT reinforcement, where MWCNT could be observed after casting.

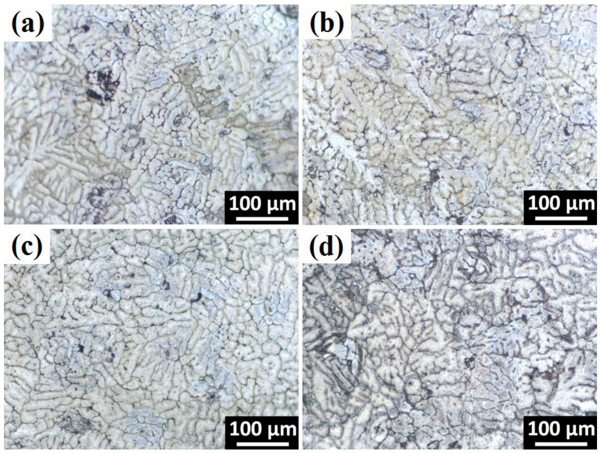


**Figure 4. Optical microstructure of (a) C AA 0CNT, (b) C AA 0.5CNT, (c) C AA 1CNT, (d) C AA 2CNT.**

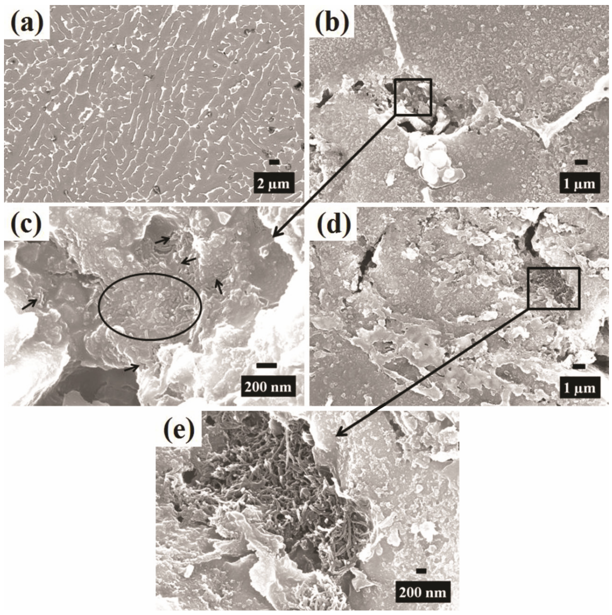


**Figure 5. FESEM image of cast (a) C AA 0CNT, (b) C AA 0.5CNT, (c) C AA 1CNT, (d) C AA 2CNT.**

While increasing the MWCNT concentration, 2wt% reinforced sample (fig. 5d-e) showed an increased amount of MWCNT inside the porous region. The FESEM images indicate even dispersion of MWCNT in the Al matrix without any noticable cluster formation at

0.5wt%. Utilization of new technique of casting process, has led to proper wettablity of MWCNT in the developed Al composite. However, even dispersion with MWCNT accumulating in pores when added at 1wt% and 2wt% was noticed. This method has also helped in the infringement of MWCNT clusters [29]. Addition of 1wt% and 2wt% of MWCNT that gave rise to agglomerates in pores may be attributed to greater amount of MWCNT that affected the metal infiltration during formation of blocks. This in turn has led to the accumulation of MWCNT inside the α grain and near the eutectic structure, that results in deterioration of desired properties. In a similar study, it was noted that incrased addition of MWCNT to Al alloy increases the agglomeration percent [30].

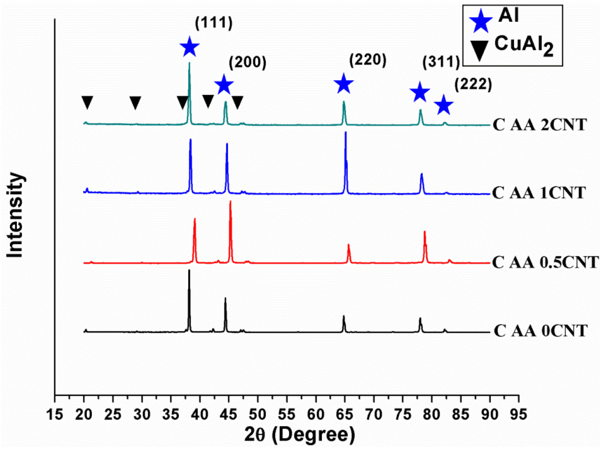


**Figure 6. XRD scan of AA2219-MWCNT composites by casting**

XRD scans of cast AA 2219 with MWCNTs for varying concentrations are shown in fig. 6. XRD scans could show a considerable amount of impurities, as there is a chance of its presence in the processing environment. It also does not divulge casting defects like surface oxides. The pure alloy and MWNT reinforced composites showing the same XRD peaks can be seen in fig. 6. XRD scan indicates the absence of Al4C3 in our composites, even at higher MWCNT content of 2wt%. The contact time of the molten aluminium alloy with the MWCNT is limited before the composite gets solidified in a relatively cold die, which is has the temperature of 300°C. The improved mechanical properties of the composites resulted in the absence of carbide formation and due to the addition of MWCNTs. Even though there might have been some presence of the impurities, it could only have a quantity below detection level (i.e., 2%) of XRD. It is evident from the x-ray diffraction analysis that there is no presence of interfacial reaction/chemical products (aluminum carbide phase) at the interface between CNT and aluminum matrix. This clearly demonstrates the absence of chemical interaction between CNT and Aluminium alloy. The reason for absence of interfacial chemical products may be attributed to favourable processing conditions in particular temperature and time. Fabrication temperature and time plays vital role in deciding interfacial characteristics of aluminum CNT composites. The clean interface between CNT and Aluminum is beneficial in achieving better physio-mechanical properties in nano-composites with approving process temperature and time adopted in present approach. On the other hand, physical contact is established in CNT reinforced aluminum composites as a result of compressive residual stresses generated during fabrication process. These residual stresses are formed due to difference in the coefficient of thermal expansion between CNT and aluminum alloy. The strong physical interfacial bonding is extremely favourable for enhanced load transfer efficiency of the nano-composites from matrix to reinforcement [31-33].

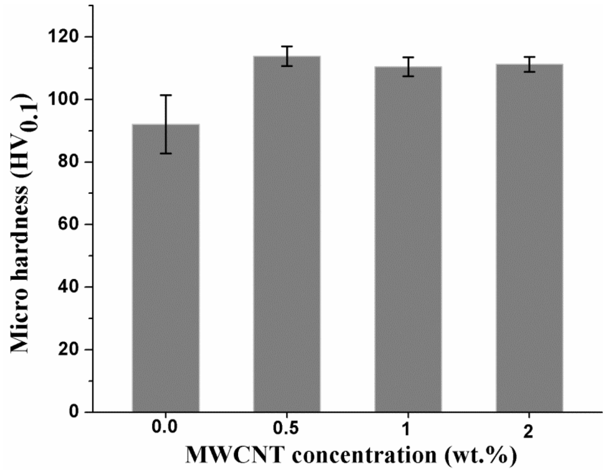


**Figure 7. Microhardness variation in cast AA2219 with various MWCNT concentrations**

Researchers report that [1,2] MWCNT/graphene improves the mechanical properties of cast composite samples. However, increased MWCNT in cast samples did not show an increase in hardness. This indicates homogeneous dispersion can be achieved only with the low concentration MWCNTs, by increasing MWCNT reinforcement that will tend to agglomerate rather than disperse in the matrix. 0.5wt% reinforcement shows uniform dispersion and has a 23.7% improvement in hardness. Further increasing concentration of MWCNT, samples didn't show improvement in hardness. MWCNTs addition above 0.5wt% leads to a considerable agglomeration of MWCNT and has no variation in hardness. FESEM images (fig. 5) support the above observations. Microhardness variation in cast AA2219 with various MWCNT concentrations is shown in fig. 7. Minimum scattering of the hardness data over reinforced cast was observed. Increased concentrations also have the same dispersion as of lower concentration, but the excess quantity of reinforcement was still present as clusters and cause the formation of porosity and voids inside cast which results in retardation of the properties. Fig. 8 shows the pores and voids along with MWCNT clusters after machining.

The compression test is used for measuring the mechanical properties for MWCNT reinforcedAA2219 cast composites. Fig. 9 shows characteristic stress strain curves obtained by compression tests of AA2219 having various MWCNT concentrations. Ultimate compressive strength was increased with MWCNT addition. But porosity and voids inside the cast due to MWCNT agglomeration limit the MWCNT reinforced cast composites in structural applications. It is also to be noted that there is a considerable change in mechanical properties for the AA2219-MWCNT composites with an addition of 0.5wt% MWCNTs. 0.5wt% addition shows 69.75% improvement in ultimate compressive strength from pure cast alloy, where the ductility got the reduction. With 1wt% and 2wt% shows an equal percent of improvement (~ 21.7%) from monolithic alloy. Nevertheless, as the MWCNTs content reaches beyond 0.5wt% compressive strength is reduced, but better than base alloy. Consequently, mechanical properties did not improve with increase in MWNCTs amount due to large agglomeration. Increasing the reinforcement concentration above 0.5wt% shows no obvious changes and shows equal amount of compressive strength due to lack of uniform distribution over improved concentration. Table 3 shows the mechanical property analysis of cast AA2219-MWCNTcomposites. The ultimate compressive strength and strain at fracture properties at varying MWCNT concentration on AA2219 are plotted for comparison in Fig. 10 (a and b) respectively.

**Figure 8. Show the AA2219 MWCNT powder cluster inside the machined cast samples.**

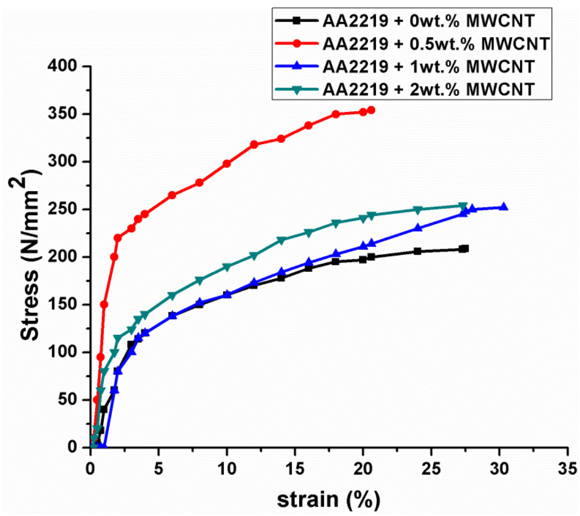


**Figure 9. Stress-strain curve of cast AA2219-MWCNT composites.**

An important factor contributing to obvious strengthening enhancement of the AA2219-0.5wt% MWCNT composite is that MWCNTs in the composite can maintain their integral structure and have distribution in molten AA2219. Proper distribution with good interfacial bonding helps in effective load transfer. The reinforcement in the form of pellets is added into the solidifying melt during vigorous agitation. It is observed that the semisolid slurry has primary solid particles in it and the reinforcing particles are entrapped mechanically by these primary solid particles thus avoiding gravity segregation and decrease their agglomeration. This results in improved distribution of the reinforcement particles. We can conclude that the improvement in composite strength is contributed by all these factors.

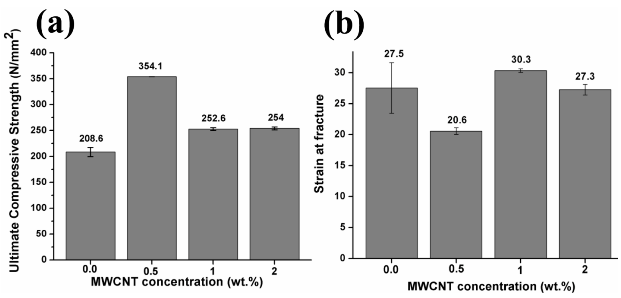


**Figure 10. Shows variation in (a) ultimate compressive strength and (b) strain at fracture of composites on increasing MWCNT concentration**

**Table 3. Mechanical properties analysis of AA2219-MWNT composites fabricated by casting route**

| Sample | Micro-hardness | Compressive strength ($N/mm^2$) | Strain at fracture (%) |
|---|---|---|---|
| CAA 0CNT | 92 | 208.6 | 27.5 |
| CAA 0.5CNT | 113.8 | 354.1 | 20.6 |
| CAA 1CNT | 110.5 | 252.6 | 30.3 |
| CAA 2CNT | 111.3 | 254 | 27.3 |

The improvement of the mechanical properties of the composites is due to the excellent properties of MWCNT. During the dispersion process, as the MWCNTs amount increases, a few MWCNTs are restricted from separating to individual MWCNTs. Furthermore, during the machining in lathe (com-pression test sample preparation) it could be observed that by increasing the amount of MWCNTs, few pellets added during casting still present inside cast as MWCNT clusters (fig. 8). It is observed that the amount of MWCNTs dispersed in the molten matrix is similar for lower concentration (0.5 wt%) and higher concentration (1 and 2wt%). Excess amount of nanotubes remains as an agglomerated MWCNT itself. This results in the formation of micro voids and more porous region as larger agglomerates get integrated into the composite and help in the initiation and propagation of cracks to earlier failure. Thus compressive strength remains the same for all the increased MWCNT concentration reinforced composites, and also poses lower properties than uniformly dispersed lower concentration reinforcement due to increased porosity and void formation.

## 4. CONCLUSION

Current research establishes a new approach in advancement to improve nanodispersion in the metal matrix which could be applicable for nanomaterials with higher potential. Composite processed through stir casting process was evaluated and proves Al-MWCNT by liquid processing doesn't have any value addition on higher concentration reinforcement. The mechanical properties of AA2219-MWCNT composite improve only at an optimal MWCNT concentration (0.5 wt%), superfluous addition will deteriorate the properties. Compressive strength and micro-hardness reached 354 $N/mm^2$ and 114 HV0.1 which is 69.75% and 23.7% of improvement respectively. Enhancement in mechanical property is achieved mainly by the role of MWCNTs in grain refining and grain boundary reinforcing, along with proper load transfer mechanism. Even though MWCNT is the strongest material in the world, it is evident that complete potentials of reinforcement were not achieved in cast composites. Thus, future researches should inculcate the need for achieving uniform nanodispersion in molten metal and to utilize all potentials from nano reinforcement.

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## NOMENCLATURE

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|---|---|
| MWCNT | Multi-Walled Carbon Nanotube |
| XRD | X-ray diffraction |
| AA | Aluminium Alloy |
| CNT | Carbon Nanotubes |
| MMCs | Metal Matrix Composites |
| PM | Powder Metallurgy |
| AA2219 | Aluminium Alloy 2219 |
| BPR | Ball to Powder Ratio |
| RPM | Rotation Per Minute |
| FESEM | Field Emission Scanning Electron Microscope |
| ASTM | American Society for Testing and Materials |
| wt.% | Weight Percentage |
| $Al_4C_3$ | Aluminium carbide |
| Ni-P | Nickel- Phosphorus |
| $Cu\,Al_2$ | Copper Aluminide |
| Al | Aluminium |
| Ni | Nickel |
| Mg | Magnesium |
| Cu | Copper |
| Si | Silicon |
| Mn | Manganese |
| Fe | Iron |
| Zn | Zinc |
| Ti | Titanium |
| Zr | Zirconium |

## ПОБОЉШАНИ ПОСТУПАК КОМПО-КАСТИНГА ЗА ДОБИЈАЊЕ ЈЕДНОЛИКО ДИСПЕРГОВАНЕ ВИШЕЗИДНЕ УГЉЕНИЧНЕ НАНОЦЕВИ У РАСТОПУ ЛЕГУРЕ АА2219

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Трансфер технологије представља највећи изазов данашњице за економичну масовну производњу. Производња материјала мале тежине и велике чврстоће са нано-угљеничним ојачањем добија на значају у истраживањима.
Истраживачи наводе да су постигли побољшање својства ојачања код једнолико дисперговане вишезидне угљеничне наноцеви (MWCNT). Али ефикасно коришћење овог својства и даље представља изазов иако је то најјачи материјал на свету.
Постизање хомогене дисперзије нарочито у растопљеном металу је сложен задатак. У том циљу је примењен нов приступ за добијање једнолике дисперзије. Извршена је анализа различитих механичких и металуршких карактеристика. Пречишћеност зрна и његова структура су утврђени помоћу оптичког микроскопа, дисперзија и структурно оштећење MWCNT помоћу FESM, а фазне промене и реакције током изливања коришћењем XRD скенера. Применом наведеног метода постигнуто је побољшање од 23,7% односно 69,75% код тврдоће и притисне јачине додавањем MWCNT.