

## Research Article

# Optimization of Stir Casting Variables for Production of Multiwalled Carbon Nanotubes: AA7149 Composite

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Combining liquefied metals with multiwalled carbon nanotube-aluminium alloy 7149 composites enables the creation of intricate designs and mass production was manufactured using mechanical stir casting, thixoforming, and T6 heat treatment. Taguchi with two factorial levels was chosen to investigate the optimum parameter and affect variables such as carbon nanotube concentration, magnesium wettability, and ring mechanical stir duration were used in addition to a robust design of experiments. The response variables were the S/N ratio, hardness, and ultimate tensile strength. The fourth DOE run resulted in an optimised nanocomposite with 107.8 HV hardness and 278.1 MPa tensile strength that contained 0.75 wt.% magnesium and 1% multiwalled carbon nanotube and stirred time of 10 minutes. The as-forged AA7149 alloy had a lower hardness value (76.3%) but a higher ultimate tensile strength value (108.4%). It was demonstrated that combining thixoforming and heat treatment improves the mechanical properties of multiwalled carbon nanotube produced under mechanical stir casting conditions.

## 1. Introduction

Multiwalled carbon nanotubes have been employed in a number of investigations on composites. Load transmission, strengthening, and thermal expansion strengthening are just a few of the factors that go into making multiwalled carbon nanotubes so strong [1, 2]. The key obstacles in metal matrix composites manufacture are often achieving homogeneous distribution and adequate wetting qualities, as well as interfacial phases between reinforced particles and matrix [3]. Rather than relying on more traditional approaches, the powder metallurgy industry has opted to tackle these problems instead. Processing powder metallurgy, on the other hand, is prohibitively expensive and can only be used for simple, not elaborate,

parts [4, 5]. It is possible to use liquid metallurgy processing, a cheaper option [6], for complex designs and large-scale manufacturing. It is, however, difficult to overcome the large density difference between multiwalled carbon nanotube and aluminium alloy. Composite reinforcement requires consideration of a variety of factors, including the purification and activation of reinforced materials, the mixing of those materials, the amount of wetting agent, and other variables (sintering, extrusion, compaction, thixoforming, and heat treatment).

Taguchi is a great technique for optimising parameters for composite development conferring to [7, 8] have shown that a number of variables, including the quantity of strengthened material, processing temperatures, wettability agents, and stirring method, have an effect on the physical

TABLE 1: Chemical composition of AA 7149 by weight percentage.

Silicon	Copper	Magnesium	Manganese	Zinc	Nickel	Iron	Lead	Titanium	Aluminium
6.5	0.2	0.2	0.3	0.1	0.1	0.5	0.1	0.2	Balance

TABLE 2: Layout of L8 orthogonal array.

Ex. no	Multiwalled carbon nanotubes (wt%)	Magnesium (wt%)	Stirring time (minutes)
1	1.0	0.50	5
2	1.0	0.50	10
3	1.0	0.75	5
4	1.0	0.75	10
5	1.5	0.50	5
6	1.5	0.50	10
7	1.5	0.75	5
8	1.5	0.75	10

and mechanical characteristics of stir cast aluminium alloys. There have been numerous investigations on the mechanical characteristics of metal composites incorporating carbon nanotubes, which have been published [9, 10]. By mixing mechanically in the liquid state with a carbon nanotubes weight fraction ranging from 0.5–1.0 weight %, the hardness of the AA7149 alloy with carbon nanotubes was enhanced to 2.5 wt.%. Though the rise in compressive strength was drastically reduced in the alloy having additional than 1.0 wt% carbon nanotubes [11–13]. Addition of 4.5% carbon nanotubes to pure aluminium powder in solid-state mixing by ball milling resulted in rise in the composite’s hardness and tensile strength, respectively, from 60 HV and 123 MPa to 130 HV and 420 MPa.

With 6.0 wt% carbon nanotubes [14, 15] achieved the highest hardness of 140 HV, however, the value declined as the amount of carbon nanotubes. Two phases of rolling and melting were performed on an AA7149 alloy containing 0.5 weight % carbon nanotubes by [16]. It was shown that when carbon nanotube content was increased to more than 2% by volume, accumulation and flexible flow in the metal matrix decreased the composite’s hardness [17]. Wettability between carbon nanotubes and metal matrix was the focus of this study in order to achieve an effective interfacial area and load transmission. Surface tensions among dissimilar materials include carbon nanotubes (100–200 mN/m) and liquid aluminium (865 mN/m) and may be wetted and broken with relative ease. An indicator of this is wettability, which is the ability to soak up water [18]. Coatings, ceramic treatment, and alloying elements can all be utilised to enhance the wettability of a material, according to [19, 20]. To increase the wettability of the AMC using strengthened materials, Mg is one of the most commonly employed materials [21], for example, increased melt wettability by adding 0.75 weight % Mg. For both carbon nanotubes and other reinforcing materials, the effects of stirring have been extensively studied.

At 500 revolutions per minute, [22] mixed SiC in a pure aluminium matrix in their study. The interfacial reaction

was made stronger and more resistant to cracking thanks to the stirring process. To ensure an even dispersion of Al<sub>2</sub>O<sub>3</sub> in the AA7149 matrix alloy, [23, 24] used stirring times of 10 and 15 minutes, respectively. Induction stir casting for multiwalled carbon nanotubes and pure Al composites improved hardness and ultimate tensile strength by 45% and 52%, respectively, according to [25, 26]. Making a multiwalled carbon nanotube, aluminium alloy is therefore viable and has drawn substantial interest from the MMC community. Mechanical stirring, wetting agent, and an optimised multiwalled carbon nanotube concentration have all been tested, but their effects on the composite are still unknown. Multiwalled carbon nanotube and AA7149 aluminium alloy were synthesised utilising mechanical stir casting, thixoforming, and T6 heat treatment in this study [27]. There were two factorial levels of Taguchi technique employed to optimise 3 variables, like carbon nanotubes, magnesium, and mechanical stirring duration, while all other components remained fixed. When it comes to mechanical qualities like as hardness and the ultimate tensile strength, bigger S/N values were shown to be more advantageous. On the other hand, we also talked about how the microstructures have evolved. These factors and their impact on a multiwalled carbon nanotube-strengthened alloy matrix composites were shown in detail in this study.

## 2. Experimental Procedures

Mechanical stir casting was used to fabricate multiwalled carbon nanotube/AA7149 alloy composites in this investigation. Table 1 shows the chemical composition of AA 7149 by weight percentage. The experiment also made use of the dependable Taguchi method and the Minitab software. AA7149 aluminium alloy and multiwalled carbon nanotube of industrial quality (Sigma-Aldrich, purity > 88 %; outside 20–40 nanometer, within 5 to 10 nanometer, with an overall length of 10–30 nanometers) were utilised as the AMC and the strengthened particles, correspondingly. A wettability ingredient was added to the mixture in the form of preweighed 1 mm magnesium pellets as it increases solid solution strengthening and adds strength to composites. Mechanical stirring time and multiwalled carbon nanotube/Mg weight percentage were both studied to see what effect they had on the results. There were two response functions: Vickers hardness (HV) and ultimate tensile strength. Taguchi balanced orthogonal arrays focus on ensuring all stages of all factors are considered fairly. At random, eight tests with two levels of primary factors and the Taguchi method L8 orthogonal array were conducted as in Table 2.

Analysis of response S/N ratios (bigger is better) was used to determine the degree of variation in the data. Table 2 shows the percentages of multiwalled carbon

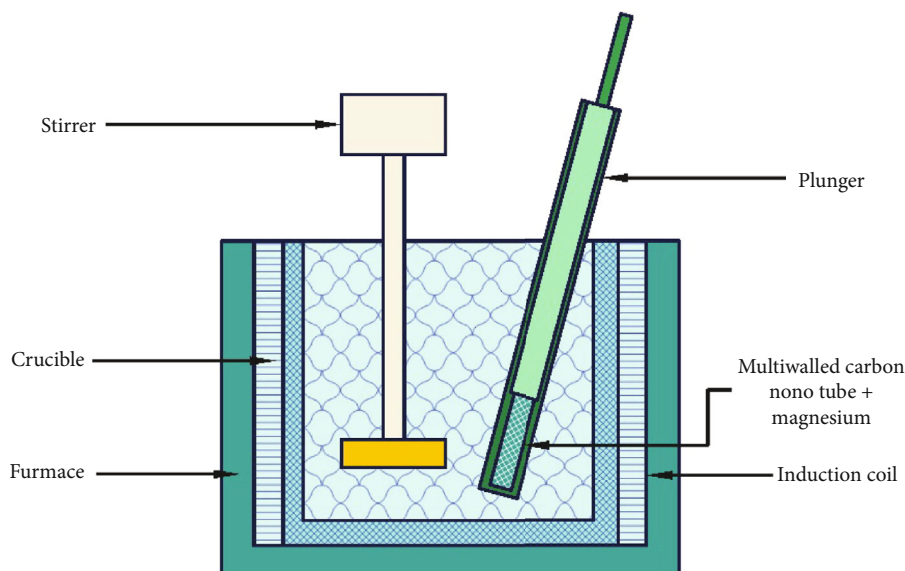


FIGURE 1: Mixing process schematic diagram.

TABLE 3: Parameters of T6 heat treatment.

Quenching	Treatment solution	Artificial ageing
27°C at room temperature	540°C for 1 hour	180°C for 2 hour

nanotube, and Mg used to make the composites, which were then wrapped in aluminium foil. The alloy was molten in an induction furnace at a temperature of 650 degrees celsius after being melted at temperatures as high as 700 degrees celsius (400 was completely consumed). It was wrapped in foil, placed in the crucible, and mechanically churned at 200 revolutions per minute using a three-blade propeller for specific periods of time (Table 2). To create new metal alloys in the semisolid state, thixoforming is a novel process. Thixoforming techniques rely on customised casting process. The thixo feedstock billet was formed by immediately pouring the mixed composite into a mould. Use of T30-80 KHz thixoforming machine was used as in Figure 1. It was heated to 580°C (semisolid temperature) with the aid of pneumatic ram and then smashed into hot work tool steel mould with a forging load of 5 tonnes at a speed of 1 m/s inside induction coils. After being removed from the mould, the billet was allowed to cool to ambient temperature.

The MT6 heat treatment process was sped up. The samples were heated to 540°C and cooled to room temperature before being quenched with water. In the Nabertherm furnace, the specimens were aged at 180°C for one hour at 30–3000°C. Following treatment for 5 minutes by [28] and it is represented in Table 3, particles will developed and hardness comparable to ASTM B917 can be attained after 20 minutes, according to this study. Because the samples had previously undergone thixoforming prior to MT6, a quicker treatment time was chosen.

The samples were sectioned, ground (400, 600, 800, and 1200 grits), polished (6  $\mu$ , 3  $\mu$ , and 1  $\mu$  with diamond solution), and etched with Keller's solution before and after thix-

forming/heat treatment. To establish the mechanical qualities, we used the VH testing machine (with a load of 1 kgf and a dwell time of 10s) and the universal testing machine (with tensile tests). According to ASTM E8, the tensile test samples were machined and shown in schematic as Figure 2. Calculated on the basis of experiment results, a follow-up experiment was conducted. Reliable findings could only be achieved by conducting tests on at least three samples per step.

### 3. Result and Discussion

**3.1. Mechanical Responses.** It is seen in Table 4 that the average hardness (HV) and ultimate tensile strength of the runs are summarised. In run 4, hardness and ultimate tensile strength were optimised using 1 weight % multiwalled carbon nanotube, 0.75 weight % Mg, and ten minutes of mechanical stirring. The hardness HV and tensile strength of the AA7149 cast ingot arrived in the laboratory at 59.5 HV and 132.9 MPa, correspondingly. A hardness of 104.2 HV and a tensile strength of 271.5 MPa were measured in the confirmation studies.

The S/N response graphs were created using minitab software as in Figures 3(a) and 3(b). The number of carbon nanotubes was found to be the second most important determinant of hardness, after mechanical stirring. When it came to composite hardness, magnesium content had the least impact. Although carbon nanotubes and magnesium were present, mechanical stirring time had the greatest impact on the composite's strength. Comparing run 4 to as-cast AA7149 alloy, the hardness and hardness of the composite improved by 76.3% and by 108.4%, respectively. In the following subchapter, we will go into more detail about this topic. It was shown that when the rheocast alloy was compared to the gravity cast aluminium alloy AA7149, the ultimate tensile strength was 73.5% greater after a 50% decrease in compaction. After thixoforming and T6 heat

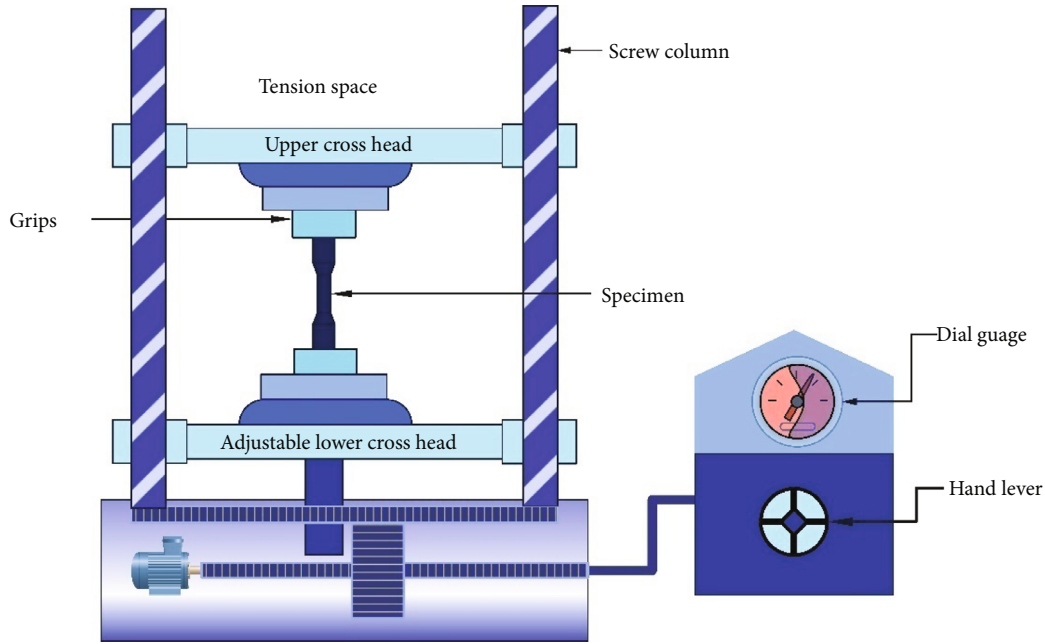


FIGURE 2: Shows schematic view of universal testing machine.

TABLE 4: Results for hardness and ultimate tensile strength.

Run	Hardness	Ultimate tensile strength
1	103.5	180.5
2	105.2	263.6
3	99.6	164.8
4	107.8	278.1
5	105.6	196.7
6	101.2	232.4
7	102.9	208.5
8	104.7	244.3

treatment, the ultimate tensile strength of an AA7149 alloy containing 6% copper was determined to be 361 MPa by [29, 30]. When silicon particles in the AA7149 alloy, [31] found that solution treatment decreased ultimate tensile strength somewhat.

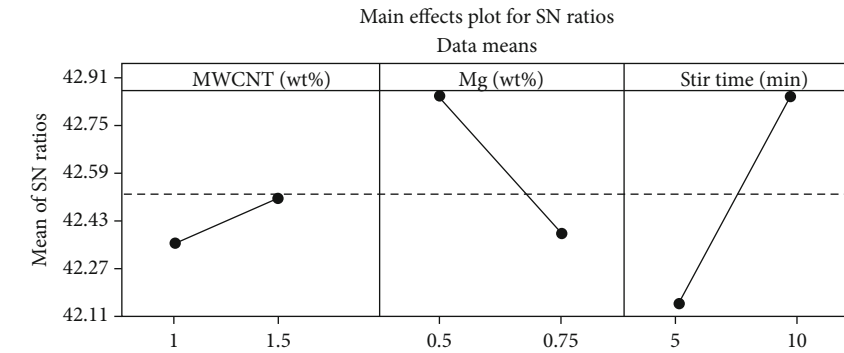
**3.2. Homogeneity and Wettability of Multiwalled Carbon Nanotubes.** A study of the fracture surfaces of tensile specimens exposed that the recommended manufacturing method produced fracture surfaces that were uniformly wettable. The matrix's multiwalled carbon nanotubes structure also showed no signs of heat deterioration. They found that wet shake mixing and cold compaction/hot extrusion achieved similar results for 1% multiwalled carbon nanotubes and 1.5% carbon nanotubes in pure aluminium matrix, respectively.

In spite of the lack of interfacial phases, such as  $Al_4C_3$ , which could be attributed to technical limitations, there were discovered to be favourable conditions for the nanotubes to spread and bridge across the grains, indicating adequate wet-

ting. Reinforced strengthening may also be a factor in the increased ultimate tensile strength characteristics of base and composite alloys. There was also evidence of multi-walled carbon nanotubes clumping in the sample from run 5. There was a correlation between the composite's mechanical properties and the reinforcing amount.

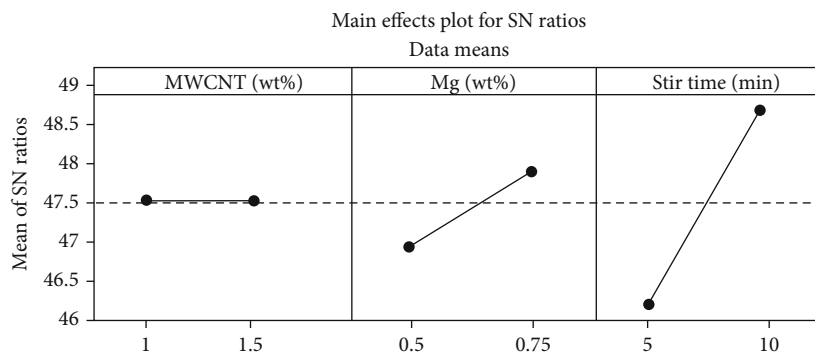
**3.3. Effect of Mechanical Stirring.** As multiwalled carbon nanotube particles were injected into the melt, the matrix's density and surface tension caused some of the particles to float and aggregate immediately. However, when mechanical stirring was introduced, the particles began to dissipate and were incorporated into the melt. A layer of multiwalled carbon nanotube particles had formed on the surface after 5 minutes of agitation, and this layer was gradually eroding in size. It is possible that the stir ring helped disseminate the particles and avoided density segregation in the molten matrix, which has been observed before. Disruption in viscoplastic behaviour was made possible by the shearing action of whirling blades that deagglomerated and homogenised molten alloy components. As observed by [19], a vortex is created when metal is vigorously stirred during liquid metallurgy processing, aiding in the transfer of nanoparticles and keeping them suspended.

The composite's hardness and ultimate tensile strength decreased as a result of the stirring process, which created voids and porosities. Therefore, thixoforming and heat treatment were required as secondary operations in order to minimise these effects. As the temperature rose, so did the grain boundaries, until they were unbroken. These operations had a direct impact on the matrix alloy. Both of the highest-response samples showed a similar significant reduction in porosity in all samples. It is assumed that a low porosity results in increased hardness.



Signal-to-Noise: Larger is better

(a)



Signal-to-Noise: Larger is better

(b)

FIGURE 3: Main effects of S/N ratio for (a) hardness and (b) ultimate tensile stress responses.

**3.4. Effect of Carbon Nanotubes and Magnesium Contents.** It was found that carbon nanotube content had a greater impact on hardness than tensile characteristics, according to the DOE data. By utilising carbon nanotubes with a lower content of 1%, hardness and tensile strength were both improved. When the entire metal matrix density is greater than 0.5 weight %, low porosity and high-density dislocations might arise. Additionally, stirring aided to enhance the grain's microstructures. It is because of the inclusion of an agglomerated reinforcing material, the carbon nanotube content had a smaller impact on ultimate tensile strength than on hardness in this investigation.

Compared to 0.5 wt.% of Mg in the matrix, 0.75 wt.% contributed to greater and more consistent hardness and ultimate tensile strength values. Carbon nanotubes can penetrate the grain boundaries of the molten matrix thanks to the wettability of pure Mg, which functions as a lubricant. Even though the additional 0.75 weight % of Mg in the AA7149 alloy may have altered the composition, it was still within the alloy's maximum Mg range given that 0.2 weight % of Mg had been added to it. Multiwalled carbon nanotubes and magnesium concentrations in the metal matrices could not be determined with any precision. Reinforced particles are still difficult to quantify, despite earlier research on varied percentages of these particles in their matrix.

## 4. Conclusions

Hardness and ultimate tensile strength of the multiwalled carbon nanotubes—AA7149 composite was studied in this analysis. The manufacturing parameters were optimised using Taguchi's robust L8 orthogonal array with two layers of major factors. Using the S/N ratio responses, we were able to determine how much the hardness and ultimate tensile strength varied from one response to the next. As a result of the uniform distribution and pullouts of multiwalled carbon nanotubes across grain boundaries, it was possible to produce a multiwalled carbon nanotubes—AA7149 composite via processing. Despite this, a small number of multiwalled carbon nanotube particles clumped together in the MWCNT sample (1.0 wt%). It was subjected to a modified short-solution treatment after thixoforming and heat treatment (MT6). Using a mixture comprising 1 weight % multiwalled carbon nanotubes, 0.5 weight % magnesium, and mechanical stirring for 10 minutes, we were able to attain T6 conditions.

A 76.3% increase in the composite's hardness compared to the AA7149 alloy resulted in an increase in its ultimate tensile strength (108.4%). According to Taguchi analysis, mechanical stirring duration was the most critical factor in increasing the mechanical properties of the composite.



There was a greater correlation between the amount of multiwalled carbon nanotubes and the material's hardness and its ultimate tensile strength. The amount of magnesium in the composite had the least impact on its two attributes. The current findings have shown nanoparticles are distributed and suspended throughout the matrix as a result of vortex activity during stirring, which breaks surface tension. The increased wettability was partially attributed to the magnesium level as well, but this contribution was small. After thixoforming and MT6, the size of Al remained constant and grew. Porosity reduced by 11% after MT6, leading to an increase of hardness and ultimate tensile strength. Thixoforming and heat treatments can improve the composite's mechanical characteristics even further.

### Data Availability

The data used to support the findings of this study are included within the article. Further data or information is available from the corresponding author upon request.

### Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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