

Development of 0.2gm MWCNT reinforced Al-Carbon fiber metal laminate(CARALL)

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Abstract: Multi-wall carbon nanotubes (MWCNT) reinforced with aluminium based bi-directional carbon fiber composite is fabricated and tested for aerospace application. A 0.2gm MWCNT reinforced with three different laminates such as Al-Carbon, Al-Glass and Al-Kevlar are prepared using hand lay-up technique. Tensile and flexural specimens are fabricated using water jet cutting with the various combinations of laminate. Mechanical strength characteristics are examined and the results indicated that 0.2gm MWCNT Al-Carbon laminate has obtained maximum tensile strength of 233 MPa and flexural strength of 152 MPa in comparison with other laminates. The fabricated combination of laminate can be utilised to fabricate aircraft wing fuselage structure.

1. Introduction

Presently, composite material has received tremendous amount of interest in aerospace, marine, automotive industry due to their light weight technology. Innovations in the composite materials have allowed the industry in significant weight reduction in the structural design of the aircraft's. Composite material offers many advantages compared to metallic alloys such as high strength to weight ratio, higher fracture toughness, corrosion resistant, fire resistant, vibration damping, wear resistant. Specifically, fiber metal laminate has gained a lot of interest in aerospace application due to their fatigue resistant, impact, specific strength and less weight.

Fiber metal laminate is the hybrid combination of metals and fibers. Primarily metal is having high load bearing capacity, impact resistance, easy recovery to damage and its isotropic nature, while full composites possess excellent fracture toughness characteristics, high strength and stiffness. GLARE laminate is most important member in the FML's due to superior machinability excellent fatigue resistant, high temperature sustainability. Carbon fiber laminates (CARALL) combines the superior strength of fibers and higher impact strength of aluminium alloy results in excellent resistant to crack formation. Metals are also used in the combinations of aramid fibers (ARALL) due to higher fatigue strength and impact strength. Aramid fibers are strong synthetic fibers and heat resistant materials but poor in machinability, despite they are used widely in industries. In FML, Epoxy resin-based



laminates takes longer time to process and cure. However, they are used widely in the industries due to more than 20% weight reduction [2].

Major increase in the fatigue strength is observed in the GLARE laminate and reduction in anisotropic properties of the GLARE with the experiment of tension-tension fatigue test at room temperature with different $[0,90]$ of axis angle. It is proved by the experiment carried out by the researchers that reduction in the fatigue strength with off-axis angle increases from 0-45 [3]. The effects of thickness on the impact properties of FMLs shows significant energy absorption during impact. Lesser the thickness of aluminium layer sustainability is more in the impact, though thicker the laminate more prone to crack formation with the impact load [4]. Also, post stretching on laminate gives negative effects on impact behaviour of ARALL composite [4]. Comparative analysis of impact strength of glass fiber metal laminate and glass reinforced plastics laminate shows the higher energy absorption in fiber metal laminate [5].

Epoxy resin is most commonly used as a matrix or adhesive for composites due to its easy availability and excellent thermal, mechanical and electrical properties and resistance to chemical. These resins provide the superior adhesion, corrosion resistance and improvement in appearance in case of coating of metals or glass. The formation of brittle laminate due to use of epoxy resin and hardener can be tackled by the addition of Nano fillers. Micro-sized filler materials are used to modify the brittleness in polymers aiming at drastic improvements in toughness and rigidity. Therefore, it is possible to improve the physical, mechanical properties and electrical conductivity by adding a certain amount of CNTs to the polymers [6]. Production of laminate starts with the etching of plate to increase the surface roughness and its bonding between metal and fiber. Chemical etching primarily performed on the surface of aluminum plate [MSDS by J.T. Baker].

2. Fabrication process of MWCNT reinforced FML

Following major activities are involved in the fabrication of reinforced fiber metal laminate

- It involves preparation of tools and materials, the aluminium layer surfaces are pre-treated by hydrochloric acid or phosphoric acid or acid-based etchants, in order to improve the adhesive bonding between metallic layer and fiber.
- Selection of appropriate size of laminate and orientation of fibers.
- Reinforcement of MWCNT into resin is carried out using mechanical stirrer and amine is added to the mixture.
- During curing, at room temperature the fabricated laminate must be kept under constant load at least for 24 hrs.
- Laminate structure, reinforcement of nanofillers can be seen under microscope or XRD, SEM analysis. Load bearing capacity, tensile strength, impact strength, stiffness of the laminate can be obtained by performing various mechanical tests on it.

2.1. 0.2gm MWCNT reinforced Al based laminate preparation

Conventionally developed aluminum master alloy (Grade 1100) plates are used for the production of FML by hand layup technique. Orientation of the laminate is kept 45^0 - 90^0 - 45^0 fiber and top and bottom layer of Al 1100. 0.2gm MWCNT is mixed with the resin using mechanical stirrer. A mixture is kept in the vacuum chamber at set room temperature to cease bubble formations in the resin.

Chemical etching process is carried out on the aluminum plate to improve the bonding between the fiber and metal. 300x300x0.5 mm al- plate, weighs of 130gms, is used in fiber metal laminate. Curing has taken place in normal room temperature for 24 hrs. The weight of MWCNT reinforced three laminates such as carbon fiber, glass fiber, kevlar fiber are 280gm, 330gm and 315gm respectively. Tensile, flexural specimen preparation is carried using waterjet cutting.

2.2. Etching Process

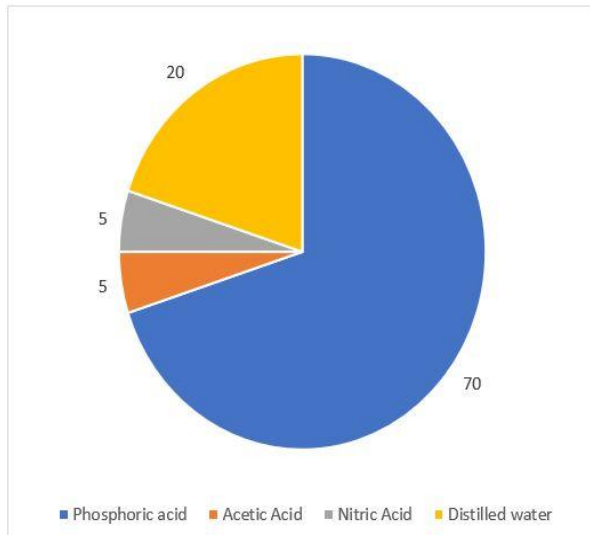


Figure 1. Etching solution



Figure 2. Aluminium etched plate

Etching treatments are useful in improving the adhesion between metal and resin. Figure. 1 shows the percentage of acids used to prepare etching solution. The solution is diluted with 20% of deionised water. The amount of nitric acid can be varied up to 1-5% and acetic acid up to 5-15% to obtained a proper etching solution. Experimental trials are performed to finalize the concentration of etching agent. Etching solutions depend upon the materials such as aluminium, copper, zinc etc [MSDS by J.T. Baker].

Performed etching process, included the roughing of the surface by grit paper followed by preheating, chemical etching and post heating. For aluminium, removing the thin oxide film with coarse sandpaper measuring 40 to 60-grit. Roughening of aluminium plate accelerated the etching of the aluminium. Figure. 2 shows the aluminium strip used in etching process. it shows the blurry area of top side which indicates the removal of thin oxide film. Chemical etching is performed at least for 20 mints per plate excluding its pre-heating and post heating at 70⁰ C for 10 mints and 20 mints resp. After chemical etching carefully removing plate from acid tub and clean it with tap water followed by cleansing with deionised water.

3. Material Selection and testing

3.1. Material selection

Materials are selected based on light weight and higher strength to weight ratio characteristics. Three different fibers are selected such as carbon fiber, glass fiber and kevlar due their higher strength, high temperature sustainability and higher impact strength resp. Multi walled nanotubes are used as a Nano filler in the laminate. LY556 resin epoxy and HY951 hardener is used as an adhesive and curing agent.

Selected materials such as bidirectional carbon fiber, glass fiber, kevlar fiber and aluminum plate are shown in Figure.3. Table I provides the data of fibers, metal, Nano-filler used in the laminate and their properties.

Table 1. Material Characterization

Al1100	Al: 99% Zn: 0.10%, Si: 0.95%, Mn: 0.05% and Cu: 0.05%
Carbon Fiber Fabric	220gsm Bidirectional
Glass Fiber Fabric	200gsm Unidirectional
Kevlar Fiber Fabric	200gsm Unidirectional
MWCNT	95% purity, Out.Dia-20-45mm, Surface area-500m ² /gm

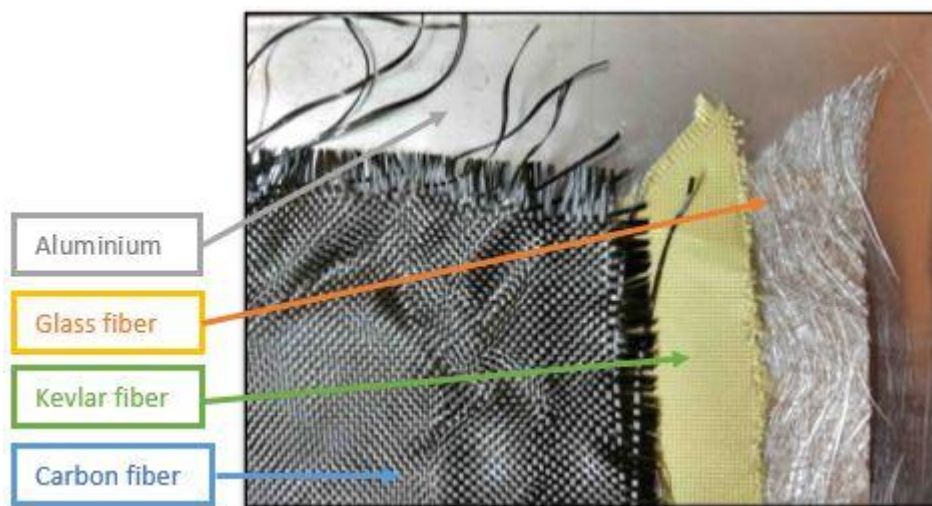


Figure 3. Fibers and metal

3.2. Layers

Table 2. Sequence and orientation of the laminate

Material	Aluminium	Resin	Fiber	Resin	Fiber	Resin	Fiber	Resin	Aluminium
Thickness (mm)	0.5	0.1	0.3	0.1	0.3	0.1	0.3	0.1	0.5
Orientation (°)	0	0	45	0	90	0	45	0	0

Design of composites commonly focusses on minimising the laminate thickness and maximising the static strength or allowable load bearing capacity of a laminate. With the optimization of laminate using analysis software's, three layers of fiber and two layers of aluminium is found as an excellent combination for FML, having an advantage of reduced weight and improved strength. Orientation angle is kept 45°-90°-45° which allowed the laminate to possess the properties in all four directions. Table 2 illustrates the thickness of the fiber, metal and its stack up sequence with orientation of fibers.

3.3. Specimen preparation



Figure 4. Tensile specimen

Water jet cutting is used for cutting the samples. ASTM D638 and D790 is followed for tensile and flexural specimen geometry. FML's are more prone to delamination, laminates are inspected to check the occurrence of delamination. Occurrence of delamination result in invariability in the strength of the material. Specimens are then subjected to mechanical tests for further studies. Figure. 4 shows the tensile specimens are fabricated using water jet cutting and tabs are made in the gripping sections. Metallography specimens are prepared by standard water jet cutting and processed by mounting, polishing and etching practice. Specimens are examined using optical microscope equipped with a digital camera.

3.4. Tensile test

Tensile test is carried out on two specimens of each three different laminates. Specimen preparation and testing procedure is followed using ASTM D638. Figure. 5 shows the UTM



Figure 5. UTM Machine

Machine, 1000KN load capacity. Tabs are prepared for the gripping section to avoid the slippage of the specimen from the machine. Test is performed in room temperature with strain rate of 0.1mm/min.

3.5. Flexural test

Figure 6. shows flexural test machine. A three-point bend test carried out in room temperature. The test specimen is simply supported by two points, fixed at span length of the specimen. ASTM D790 ensures the flexural specimen geometry and the process. Applied variable load forced the specimen to bend towards the load applied and crack started forming.



Figure 6. Flexural test machine

3.6. Microstructural analysis



Figure 7. Optical microscope

Figure. 7 shows the OLYMPUS GX 21 optical microscope. Magnification ranges from 50x to 1000x. In micro structural analysis of FML, specimens are cut by using waterjet cutting and mountings are made to hold the specimen under microscope. Hot wax is used to mount the specimen in the fixture.

Specimens are polished smoothly to obtained a mirrored surface for accurate reading or examining. For FML's etching is performed on the observed surface, using solution of acetic acid for 5 mints, then ultrasonically cleaned using deionized water for 10 mints. Samples are dried at 60⁰ C for 1 hr.

4. Result and discussion

4.1. Morphological studies

Basically, these laminates consist of three fiber layers incorporated between two aluminium layers on the top and the bottom. The sequence of the layers is investigated and optimized using analysis software's. Compare to conventional 3/2 laminate, combination three fiber layers incorporated between two aluminium layers proved excellent with improved strength of about 10% and reduction in the weight of about 20%.

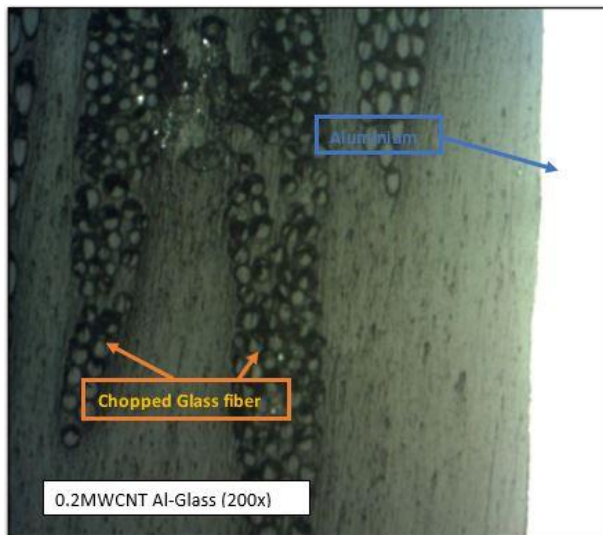


Figure 8. 0.2MWCNT Al-Glass 200x

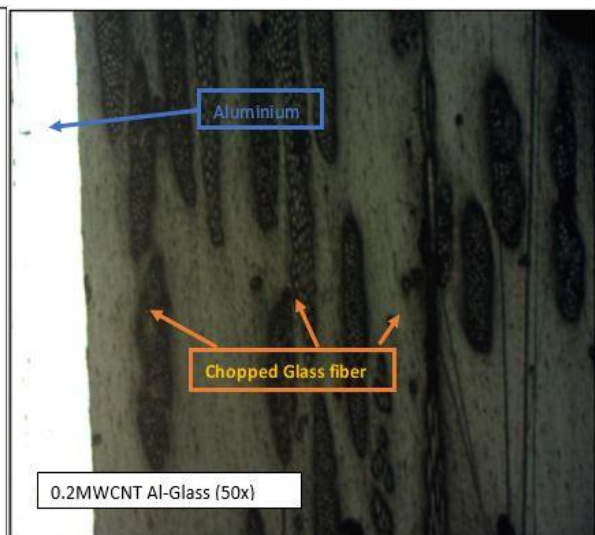


Figure 9. 0.2MWCNT Al-Glass 50x

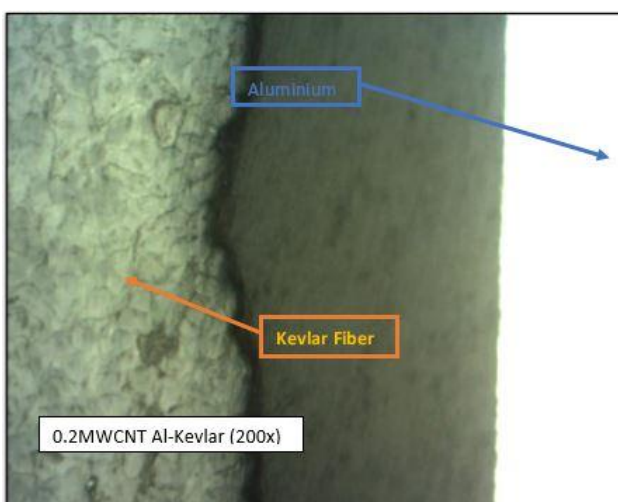


Figure 10. 0.2MWCNT Al-Kevlar 200x

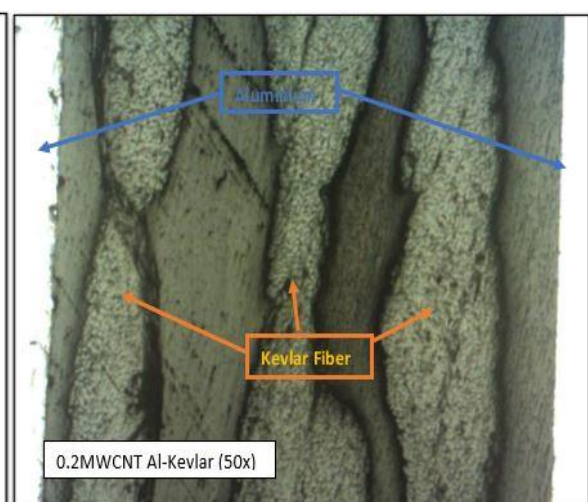
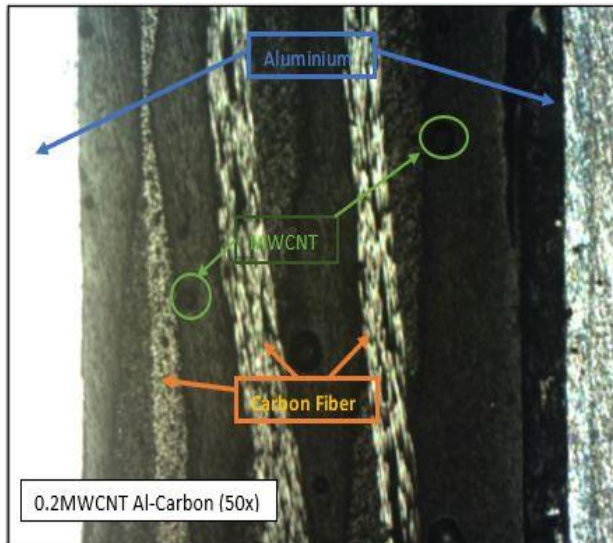
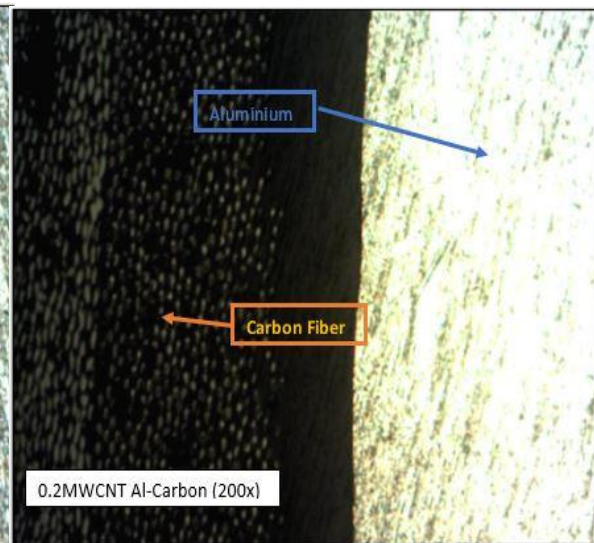
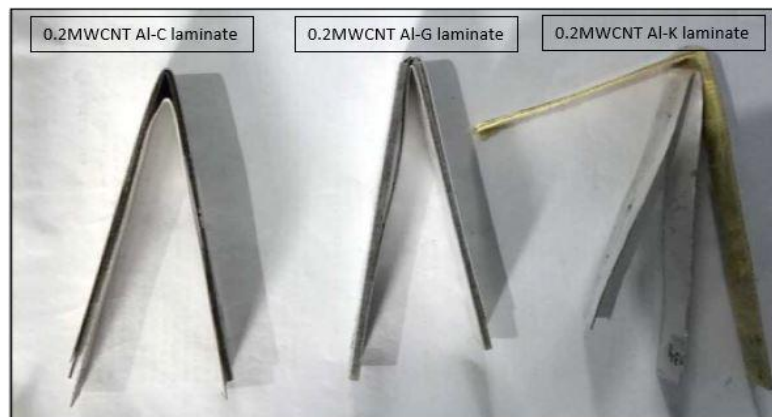


Figure 11. 0.2MWCNT Al-Kevlar 50x

**Figure 12.** 0.2MWCNT Al-Carbon 50x**Figure 13.** 0.2MWCNT Al-Carbon 200x

of MWCNT because of its improper dispersion of MWCNT in the resin. Figure. 12 and Figure. 13 shows the 0.2MWCNT Al-Carbon laminate structure. In Figure. 12 Al-carbon laminate with 50x magnification is presented, the presence of MWCNT is detected in the image. According to Figure. 13 carbon fiber and aluminium plate has magnified at 200x and it is clearly seen that bonding between metal and fiber. No crack and no porosity are detected between bonding. Detected reinforcement was lied on the side face of specimen. Porosity can be detected in the resin but it has no effect on the bonding of metal and fiber.

4.2. Flexural test

**Figure 14.** Tested flexural specimens

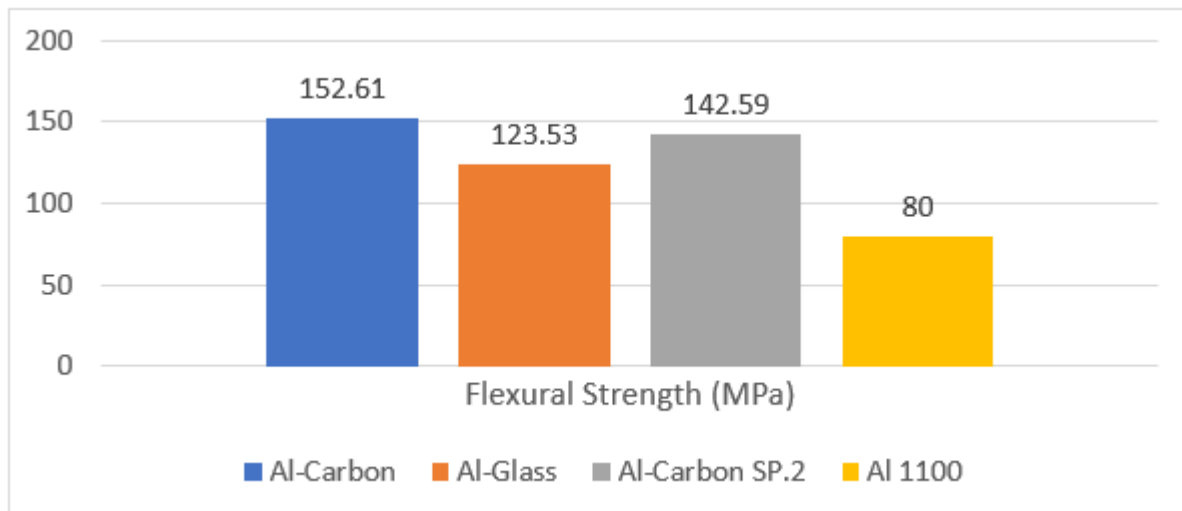


Figure 15. Flexural strength

Typical flexural behaviour obtained from the three-point bending test is shown in Figure. 15. Also, it shows the data collected from the experiment conducted on UTM machine with different laminates. Figure. 15 presented the result data indicating 0.2MWCNT Al-Carbon fiber metal laminate compare to 0.2MWCNT Al-Glass and conventional aluminium. From the Figure 15, highest flexural strength identified in 0.2MWCNT Al-Carbon fiber metal laminate is 152.61 MPa. Experiment performed on the three different fibers is shown in Figure. 14. It shows the specimens of 0.2MWCNT Al-Carbon fiber, 0.2MWCNT Al-Glass fiber, 0.2MWCNT Al-Kevlar fiber with same stack-up sequence of layers and orientation. It is clear that from the Figure. 14 carbon fiber metal laminate and glass fiber metal laminate the crack formation is extended along the internal layers between the aluminium and FRP layer i.e. these laminates acted as a single entity during crack formation. While in the case of 0.2MWCNT Al-Kevlar fiber as observed in the Figure. 14, is delaminated due to intervention of interlaminar stresses due to mechanical loading. Interlaminar stresses are viewed as a shear and normal stresses but they tend to separate laminae from each other. Also, few fibers burst out of the laminate while testing.

4.3. Tensile test

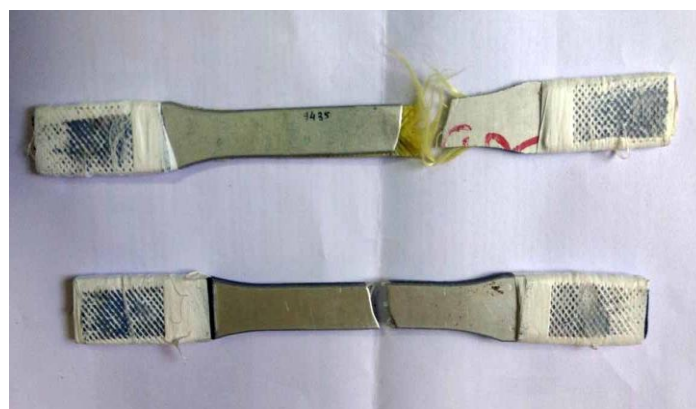


Figure 16. Tested tensile specimens

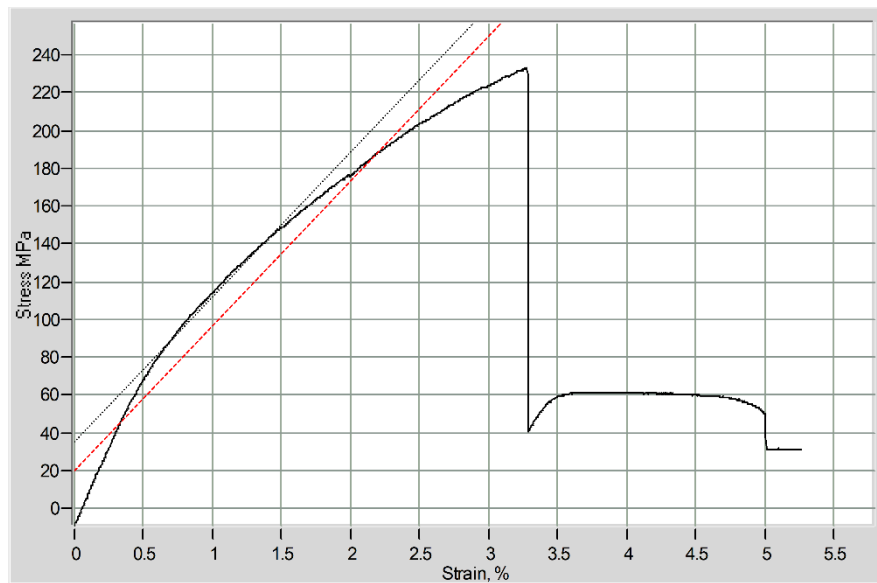


Figure 17. Stress-Strain curve for 0.2MWCNT Al-Carbon laminate

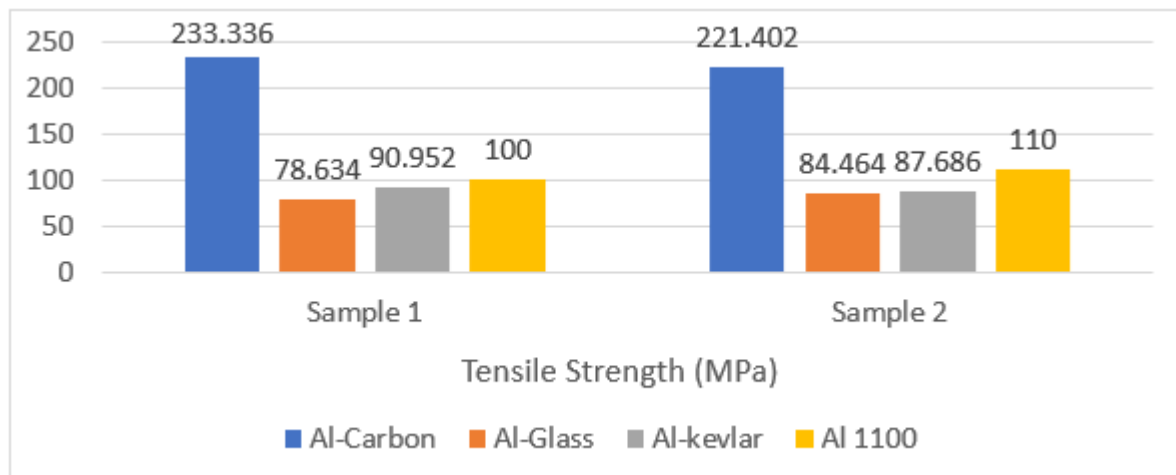


Figure 18. Tensile strength

Stress-Strain curve for 0.2MWCNT reinforced Al-Carbon is presented in Figure. 17. Strain rate remained constant for all the specimens is 0.5mm/min. Figure. 16 shows the tested tensile specimens for MWCNT reinforced carbon and glass. It is observed that failure of carbon fiber laminate specimen is in between the critical region of tensile specimen. It shows the positive impact on reinforcement of MWCNT and incorporation of woven carbon fiber with a aluminum alloy. Zigzag fractured observed in the case of carbon fiber laminate due to aluminum plate and CFRP shares uneven load. Non-uniformity caused due to difference in elasticity between metal and fiber. It resulted in zigzag nature of fracture. Mode of failure is observed as long splitting gauge middle. Few bursting of fibers observed during the test with kevlar fiber, failure mode is observed as explosive, fibers dispersed out from the laminate due to the density of kevlar laminate but fractured in the critical region. In case of glass fiber laminate fractured took place in critical region and failure observed as natural straight cut. Ultimate tensile strength is observed highest in 0.2MWCNT Al-Carbon fiber laminate as 233.36 MPa compare to 0.2MWCNT Al-Kevlar laminate and 0.2MWCNT Al-Glass laminate tabulated in Figure. 18.

5. Conclusion

In this work, reinforcement of MWCNT into resin and use of carbon fibers, glass fibers and aramid fibers with aluminum 1100 are tested for its mechanical performance. Amino functionalized MWCNTs is incorporated into epoxy resin fiber metal composite with different orientation and sequence of layers to enhance mechanical properties. 0.2gm MWCNT Al based carbon fiber, glass fiber, Kevlar fiber laminate is fabricated using hand lay-up process. Incorporation of MWCNT into resin and use of woven carbon fiber increased tensile strength of the laminate with the substantial reduction in the weight of about 20-30%. 0.2gm MWCNT incorporation and 0.375 fiber volume fraction in the laminate increased flexural properties of composite. Flexural test indicated that maximum strength and strain energy is achieved in 0.2MWCNT carbon fiber metal laminate and glass fiber laminate. It is observed that addition of bidirectional woven carbon and 95% pure MWCNT to the Al 1100 alloy gives better tensile strength, flexural strength and load carrying capacity compare to Al 1100 alloy, 0.2 MWCNT Al-Glass laminate and 0.2 MWCNT Al-Kevlar laminate. Morphological studies of different fiber metal laminates confirm the presence of MWCNT in the laminate and the absence of delamination. The combination of 3 fiber layers and 2 aluminum layers in the laminate causes weight reduction of about 30% observed in 0.2MWCNT Al-Carbon fiber metal laminate and 20% in 0.2MWCNT Al-Glass, 0.2MWCNT Al-Kevlar laminate compared to conventional aluminum alloy.

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