

# Effect of nano hybrid additives on low velocity impact responses of aramid composite plates: example of CNT and ZrO<sub>2</sub>

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**Abstract:** Aramid reinforced composites are advanced materials that are widely used in many industrial applications thanks to their combination of high strength and lightness. Nano additives are of great importance for improving the mechanical properties of aramid reinforced composites and reducing costs. In this paper, multi-walled Carbon Nanotube (CNT) and Zirconia (ZrO<sub>2</sub>) nano hybrid additives were used to determine the effect on the mechanical characterization of aramid composite plates. Therefore, low velocity impact responses of aramid fiber reinforced composites were investigated by adding ZrO<sub>2</sub> and CNT nano hybrid additives to the Polives 701 polymer vinyl ester resin matrix. Low velocity impact tests were carried out at 10 J and 15 J. As a result of the experiments, the effects of nano hybrid additives on the impact absorption properties of aramid composite plates were determined. By determining the maximum force, displacement and time values, the effect of CNT and ZrO<sub>2</sub> nano hybrid additives on the impact resistance of the composite plates was analyzed. In addition, it contributed to the development of composite materials used in industrial applications by providing information on increasing the performance of composite materials by using nano additives. As a result of this study, it was determined that the strength of the composite material increased proportionally when the CNT additive was used, and the material became embrittled when the ZrO<sub>2</sub> additive was used.

**Keywords:** aramid fiber, carbon nanotube, low velocity impact, zirconia.

## 1. Introduction

Fiber-reinforced composite materials are widely used in many industrial applications due to their advantages such as high strength, low weight, and good corrosion resistance [1]. Such composites consist of a combination of fiber reinforcement and polymer matrix and are usually produced using matrix resin. However, continuous research is carried out to further improve the mechanical properties of these materials and reduce their costs. In recent years, nano additives have been found to be a potential solution to improve the properties of fiber-reinforced composite materials. Nano additives are composed of particles on the nanometer scale and can significantly affect the mechanical, thermal, and electrical properties of composite materials. Therefore, nano additives have been an important research area for optimizing the properties of composite materials and for a wider range of applications. In recent years, significant progress has been made in improving the mechanical properties of fiber-reinforced composites with nano additives and reducing costs. For example, has been observed

that graphene oxide additives increase the mechanical strength of composite plates and at the same time reduce their weight. Mahesh V.P. et al. has been stated that polymer matrix composites are the best choice in terms of properties such as thermal conductivity, protection against moisture and good strength at the same time [2]. Brabazon mentioned that polymer matrix composites are used in applications such as aerospace and water treatment systems that contain external factors that will adversely affect the material [3]. The study by Esmaeilzadeh et al. shows that when multi-walled CNT are added to the material at a certain rate, they increase the hardness and fracture resistance of the material, and when the additive ratio increases, mechanical properties such as hardness are negatively affected [4]. Bocanegra et al. found high fracture toughness and short dimensional cracks relative to single-walled CNT when they added double-walled CNT in their experiments that sintered the ZrO<sub>2</sub>-saturated alumina composite for one hour at 1520 °C [5]. Shadakshari et al. found that the electrical and thermal conductivity decreased as a result of increasing the weight

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of multi-walled CNT, and they observed with SEM, XRD, EDS and TEM tools that the decrease in thermal conductivity was due to inter-nanotube bending due to high temperature as a result of sintering and heat treatment [6]. Li R. et al. concluded that when  $ZrO_2$ , which is known for its biocompatibility in dental applications, is added to the composite to increase the shear bond strength, it is very beneficial in the maintenance and repair stages in the sector, and the silaning process as a bonding agent has played an important role in increasing this bond strength and durability [7]. Muthusamy et al. determined that vinylester resins are an ideal resin type with better erosion performance than other polymer resins in this type of studies due to their affordability, properties and easy availability [8]. Thooyan et al. discussed that the use of basalt fiber as a reinforcing element in vinylester matrix composites indicates poor adhesion, and as a result, low mechanical properties occur, to prevent this negative by adding a filler additive containing nano- and micro-level SiC composition [9]. When Bonsu et al. investigated the mechanical behavior of a polymer matrix composite in an artificial seawater environment, they preferred vinylester resin because it was in the middle of epoxy and polyester in terms of moisture resistance and price [10].

There are many studies investigating the mechanical behavior of composites against low-velocity impact. Mantena et al. observed that the effect of low-velocity impact with weight reduction in composites is much lower than that of high-velocity impact, and the process is quasi-static [11]. Hongkarnjanakul et al. performed a very low velocity impact test using a three-point bending tester to examine matrix failure and defects such as matrix cracks opening and a small percentage of self-closing [12]. Mahdian et al. discussed that the damage can be examined more realistically and non-destructively in the low-speed impact test with acoustic emission sensor weight reduction, which is planned to be used in a similar impact test [13]. Chandekar et al. showed comparatively that realistic results can be obtained with low speed impact test analysis using LS-DYNA program on the data obtained by computer aided simulation and finite element analysis method [14]. Sevkati et al. found that the symmetrical plate with different fold directions showed the best resistance to impact [15]. It was also observed that the delamination energy due to local deformation at the contact point increased with increasing laminate thickness [16]. During impact, it complicates the simulation of impact as damage modes such as fiber breakage, delamination, matrix cracking and crushing can occur simultaneously [17]. Linking the onset of damage to a single parameter is unrealistic and also increases the margin of error of simulations. While microscopic damages represent the effects of distributed defects in terms of internal state variables, damage detection is done at the macroscopic level in some way with continuous damage mechanics. It was concluded that woven composites were superior to unidirectional composites in low-speed impact tests [18]. Bhatnagar et al., using the impact output results, found the force formed by the mass/spring mechanism and

the energy conservation law, and revealed that this force could later be used to calculate the contact time [19]. Hanif et al. performed bending tests on a multi-walled carbon nanotube reinforced epoxy/aramid composite homogenized by sonication under different forces according to ASTM D5045 standard [20]. Evci et al. argued that damage criteria can be revealed by determining the impact response, and in this context, they performed three-point bending tests to compare the dynamic structure with the static structure [18]. Wang et al. improved the matrix-fiber interface adhesion by treating bamboo-reinforced epoxy resin matrix composites produced by the resin transfer molding (RTM) process with NaOH solution in addition [21]. Increasing production temperature also plays a role in the adhesion of the composite matrix-fiber interface [22]. Wu et al., by changing the order of the chemical bonds between the layers, obtained different adhesion interfaces when additives were added according to the bonds [23]. It has been found that the use of chopped fibers between flat laminates improves the delamination strength of composites [24]. Gupta et al. concluded that epoxy resin is the matrix that uses the absorbed energy most efficiently by making the matrix epoxy or vinylester in layered composites [25]. Meyer et al. have solved this problem by reducing the molding gap after concluding that pressing at high speeds distorts the position of the stacks, but resin flow could not occur [26]. Abiodun et al., using the central composite design method, concluded that mixing should be done for 30 minutes at 643 °C and 423 rpm in order to obtain optimum results in the production of  $mTiO_2/p/Al$  7075 composite [27].

The aim of this study is to investigate the effect of CNT and  $ZrO_2$  nano-hybrid additives on low velocity impact responses of aramid composite plates. Maximum force, displacement and time values were determined by performing low speed impact tests. The obtained results were analyzed to determine the effect of nano hybrid additives on the impact absorption properties of aramid composite plates. In this study, hybrid interactions of additive ratios in 4 different composite plates, one of which was produced by using vacuum infusion method and autoclave curing, one pure resin / aramid and the other 3 using different nano additive ratios, were considered and it was aimed to find the most suitable hybridization ratio in terms of impact resistance in layered composites. The results of this study will contribute to the development of composite materials used in industrial applications by providing information on increasing the mechanical performance of composite materials with nano additives. In future studies, it is aimed to complete the studies on hybridization rates and transfer them to artificial intelligence applications by adding higher ratios of CNT and  $ZrO_2$  to the vinylester resin / aramid composite produced with the central composite design methodology.

## 2. Material and Methods

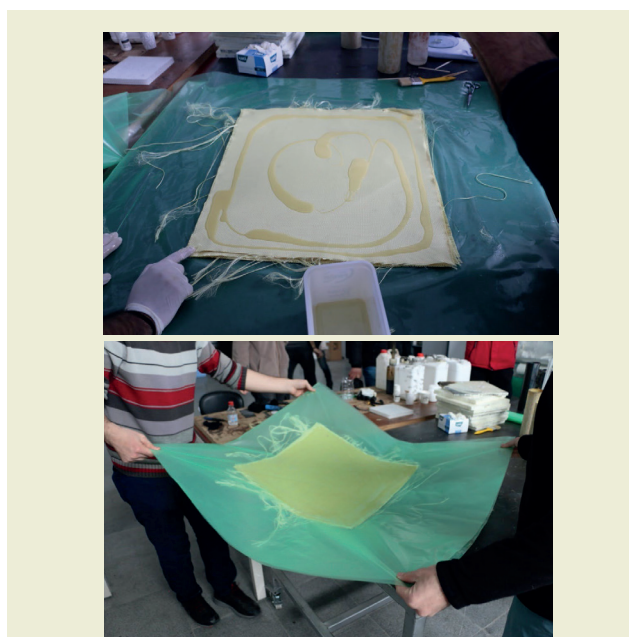
Ballistic para-aramid fabric with product code Kevlar 29 CT736 was used as fiber reinforcement material in the

study. Aramid fiber fabric basket type is 2x2 braided, 0.62 mm thick, 410 g/m<sup>2</sup> density and 150 cm wide. Vinylester Polives 701 was used as matrix material. Multi-walled CNT and ZrO<sub>2</sub> nanopowders were used as nano additives. The properties of nano additives are shown in Table 1.

**Table 1.** Properties of CNT and ZrO<sub>2</sub>

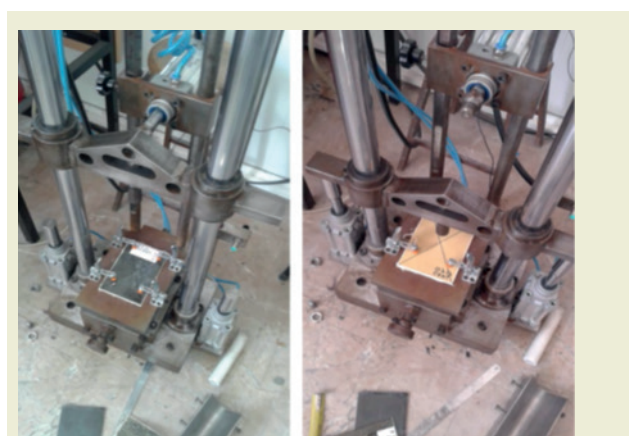
Nano materials	Properties	Values
CNT	Purity	92%
	Intensity	2.4
	Outer diameter	8-10 nm
	Inner diameter	5-15 nm
	Electrical conductivity	98 S/cm
ZrO <sub>2</sub>	Size	20 nm
	Purity	99.8%
	Morphology	Spherical
	Surface area	20-60
	Intensity	5.89

Aramid fabric was cut with special scissors in dimensions of 400 mm x 400 mm. New production vinylester resin hardener 0.2% by weight and 6% cobalt accelerator 0.02% by weight were prepared for hand lay-up. Six layers of nano-additive-free fabric were laid on the vacuum nylon and impregnated with a resin roller brush with hardener and accelerator between each layer. Trial studies were carried out with the resin to wet six layers of fabric and the amount that completely wetted the fabrics was determined as 280 g. In order to prevent any gelation of the vinylester resin during the process with a hardener and accelerator with a roller brush, the airflows in the workshop were turned off and the work was carried out at 20 °C (ambient temperature). After the material was placed in a vacuum bag in a vacuum of -0.6 Bar and placed in a press heated at 30 °C, the temperature was increased to 70 °C, and it was left to cure under 5 Bar pressure for 2 hours and left to cool naturally for 24 hours after the press was closed. After cleaning the sample, it was prepared for impact test by cutting with water jet in 100 mm x 150 mm dimensions according to ASTM D-7136 standard. In the sample with additives, it was first mixed into the CNT resin and cooled intermittently up to 40 °C by mixing for 15 minutes in the mechanical mixer and for 10 minutes in 2 periods in the ultrasonic mixer, for a total of 20 minutes. Then, ZrO<sub>2</sub> was added and mixed for 15 minutes in the mechanical mixer and in 2 periods of 10 minutes in the ultrasonic mixer, for a total of 20 minutes, and it was prepared by cooling to 40 °C. Hardener and accelerator were added to the prepared resin and applied on the aramid fabric. In this way, homogeneous dispersion of nano materials into the resin is ensured. All production processes were applied separately to the fabrics for each nano parameter (3 different parameters) as stated above, and nano-doped samples were obtained and made ready for the experiment. Sample preparation is shown in Figure 1.



**Figure 1.** Sample preparation

Impact tests were performed with free weight drop. The impactor has a hemispherical barrel with a diameter of 12 mm and a mass of 5.6 kg. Force changes were obtained with a millivolt piezo electrical sensor. Sensor signals were acquired via a computer-mounted data acquisition card. The variation of the interaction force between the impactor and the samples over time was obtained with the NI Labview Signal Express (Figure 2) software. The sampling rate of the data acquisition system is 25 kHz. The tests were applied separately from three samples with the same parameter to samples with four different parameters at two different speeds, 10 J and 15 J, and a total of 24 results were obtained. The force data received were calculated as described in the ASTM D-7136 standard. The velocity with the first integration of acceleration and the displacement values with the second integration are obtained [28]. When the impactor hits the samples, its kinetic energy is partially transferred to the material and the remaining kinetic energy is recovered as recoil energy. The impact test unit has a rebound mechanism to catch the impactor after the initial impact is complete.



**Figure 2.** Impact testing device

### 2.1. Data Analysis and Preprocessing

Data analysis involves the process of examining, modeling and interpreting data. Data preprocessing is the process of making raw data suitable for analysis. In this process, steps such as data cleaning, transformation and feature engineering are performed. The data set obtained from experimental studies consists of CNT, ZrO<sub>2</sub>, time, applied energy, displacement, obtained energy and force features. A number of adjustments were made using the data pre-processing method for the missing and erroneous data that occurred on the data of these features. Feature statistics of the dataset are shown in Table 2.

### 3. Results and Discussion

Figures were obtained using force-time data under 10 J and 15 J energy levels. The interactions of low-rate CNT and ZrO<sub>2</sub> nano additives on the strength of the composite were discussed. Comparisons at 10 J and 15 J energy levels were made with reference to the undoped sample. The undoped vinylester resin/aramid composite sample was taken as the reference material. When the force/time graph obtained from the experimental study performed at 10 J energy level was examined, it showed a normal distribution in the form of a bell curve to the impact response of the undoped vinylester resin/aramid composite plates.

It was observed that the force increased rapidly in a short time and reached its maximum value (Figure 3). The oscillations observed in all graphs showed that the damage started with delaminations and matrix fractures in the impact region. With the addition of 0.5% ZrO<sub>2</sub>, a decrease in maximum force and an increase in pulse duration were observed. It was determined that the damage was in the form of delaminations and partial fiber fractures. When 0.5% CNT was added, it was observed that the maximum strength of the material increased partially, and the damage occurred in the form of high rates of delamination and fiber breaks. When 0.5% CNT / 0.5% ZrO<sub>2</sub> was added, it was determined that the damage was severely intensified with fiber breaks and delaminations because of the increase in the maximum force.

These results emphasized that CNT and ZrO<sub>2</sub> nano additives have significant effects on the impact strength of aramid composite plates and that nano additives contribute to the potential to improve the impact strength of

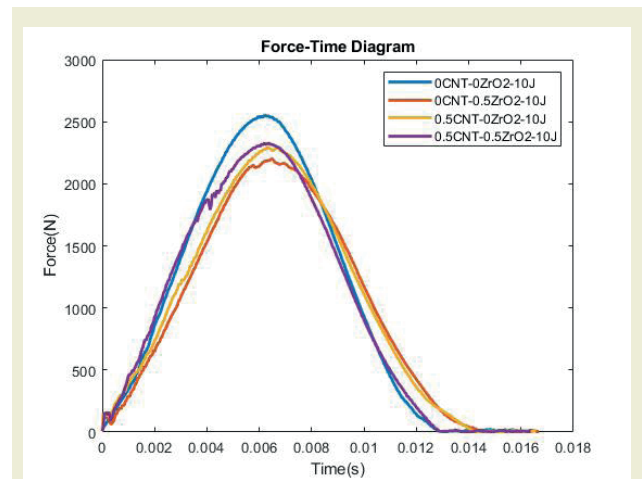


Figure 3. Force-time diagram under 10 J energy

composite materials.

While similar properties to the reference material were observed with only 0.5% ZrO<sub>2</sub> contribution at 15 J energy level (Figure 4), fiber fractures intensified in the impact region and damage developed early and the material exhibited brittle behavior. Since nano material agglomeration occurred in the sample with only 0.5% CNT addition, sudden strength drops were observed. It was observed that fiber fractures intensified, and layer separation occurred in the 0.5% CNT / 0.5% ZrO<sub>2</sub> added sample. The results highlighted that different nano doping ratios can trigger different damage mechanisms and the importance of an optimized additive composition selection.

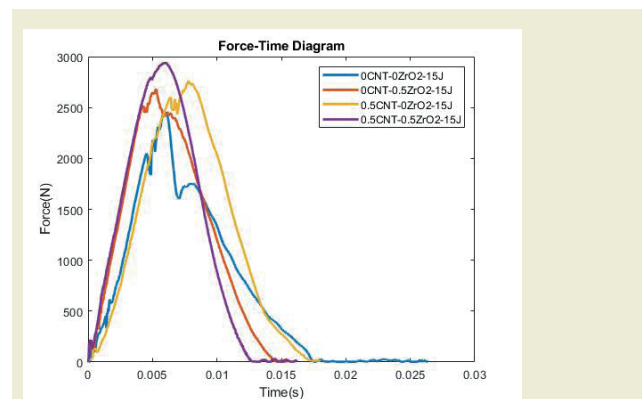


Figure 4. Force-time diagram under 15 J energy

Table 2. Feature statistics of the dataset

Feature	Mode	Mean	Median	Dispersion	Min.	Max.	Missing
CNT	0.5	0.268	0.5	0.928	0	0.5	0
ZrO <sub>2</sub>	0.5	0.258	0.5	0.964	0	0.5	0
Time	0.004	0.011	0.011	0.505	0.00001	0.041	0
Applied Energy	15	12.720	15	0.195	10	15	0
Displacement	7.55e-08	0.016	0.015	0.549	7.55e-08	0.047	0
Obtained Energy	4.21e-10	6.099	7.161	0.846	4.21e-010	15.332	0
Force	2.755	1058.41	904.711	0.869	2.755	2941.03	0

Figures were obtained using force-displacement data under 10 J and 15 J energy levels. Comparisons at 10 J and 15 J energy levels were made with reference to the undoped sample. In the sample containing only 0.5% ZrO<sub>2</sub> at 10 J energy level, the maximum force decreased while the amount of displacement increased. Maximum force and displacement value of the material increased only in 0.5% CNT added sample. A more balanced situation was observed with 0.5% CNT / 0.5% ZrO<sub>2</sub> additives, and there was no significant difference in force and displacement ratios. Graphs of force-displacement values is shown in Figure 5.

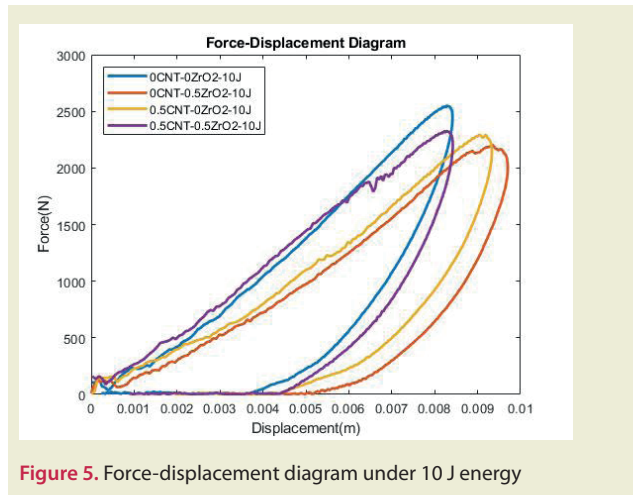


Figure 5. Force-displacement diagram under 10 J energy

Serious damage modes occurred at the 15 J energy level in the pure sample, and the irregularity in the material rebound in the graph is thought to have occurred due to nano agglomeration has been detected. This shows that the impact strength of the undoped sample is limited, and its mechanical performance needs to be optimized. It was determined that only 0.5% ZrO<sub>2</sub> added sample material reached the maximum strength and serious fiber damage occurred with low displacement rate in the impact region. These results showed that the ZrO<sub>2</sub> additive increased the impact strength of the material, and the material became brittle when the damage intensified. It was observed that the impact force and displacement amount of the material were maximized in the sample with only 0.5% CNT additive, but the amount of energy consumed was reduced. These results showed that the CNT additive increased the energy absorption capacity of the material and the deformation caused by the impact was absorbed more effectively. With 0.5 CNT / 0.5% ZrO<sub>2</sub> additives, it has been determined that the material maintains the maximum force ratio and the amount of displacement is at the lowest level. It has been shown that the use of CNT and ZrO<sub>2</sub> additives together increases the impact resistance of the material and controls the amount of displacement. Graphs of force-displacement values is shown in Figure 6.

Figure 7 and Figure 8 were obtained using energy-time data at 10 J and 15 J energy levels, and comparisons were made with reference to the undoped sample. At the 10 J energy level, the pure sample gave back 60% of the energy

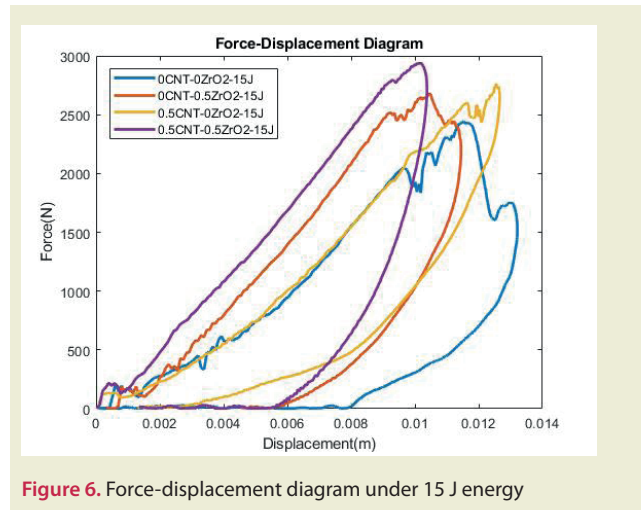


Figure 6. Force-displacement diagram under 15 J energy

as recoil energy and absorbed the rest. The recoil energy increased only in the 0.5% ZrO<sub>2</sub> added sample. As a result, the material was less damaged and partially ductile. It was concluded that only 0.5% CNT additive delayed the recoil energy and increased the energy absorption in the sample. With 0.5% CNT / 0.5% ZrO<sub>2</sub> additives, it did not cause any increase in the energy absorption ability of the sample.

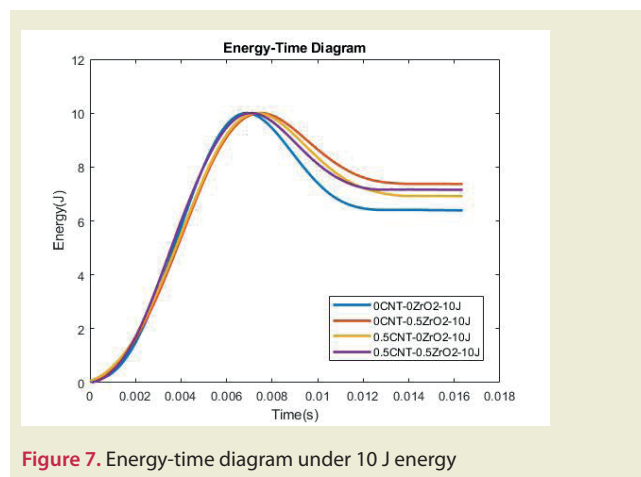


Figure 7. Energy-time diagram under 10 J energy

It was observed that 0.5% CNT / 0.5% ZrO<sub>2</sub> additive at the 15 J level provided a slight increase in the energy absorbing ability of the material. This gives an idea about the low rates of nano additives. The effect of energy increase at this level is clearly visible. The return energy level in the undoped material increased, but the return energy in the 0.5% ZrO<sub>2</sub> doped sample decreased. This can be explained by the fact that as the energy level increases, the material's ability to absorb energy increases. It can be concluded that with the effect of only 0.5% CNT additive, the recoil energy decreases, absorbs more energy, and increases the impact strength.

When the damage images in Figures 9 and 10 were examined, partial delamination's at 10 J energy level and fiber fractures in the first layer were detected in all samples. At 15 J energy level, it was observed that the amount of delamination increased, and fiber fractures occurred in

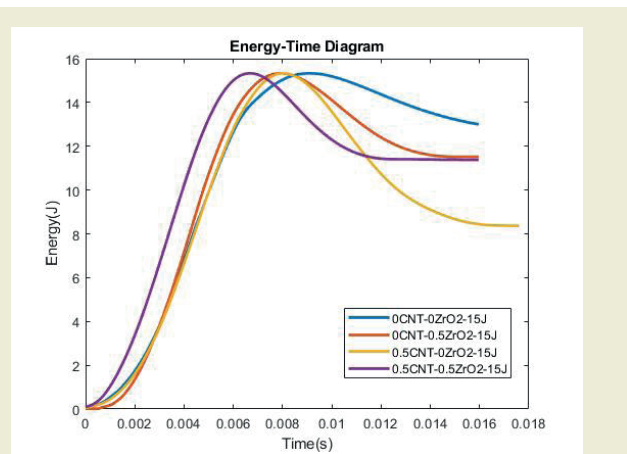


Figure 8. Energy-time diagram under 15 J energy

a wider region. As the energy level increases, the level of these damages should be expected to increase.

It was determined that the 0.5% CNT and 0% ZrO<sub>2</sub> doped sample developed at 10 J energy level in the opposite di-

rection of the impact force, and the delamination's developed towards the layer separation and the material damage occurred in the thickness direction. When the energy level was increased to 15 J, the damages were more intense in the first layers. Although a small number of partial delamination and fiber fractures occurred at 10 J energy level in the 0% CNT and 0.5% ZrO<sub>2</sub> doped sample, the damages increased and intensified due to the embrittlement effect of the ZrO<sub>2</sub> addition.

In the 0.5% CNT and 0.5% ZrO<sub>2</sub> doped sample, it was observed that delamination's resulted in layer separation at 10 J energy level and fiber fractures increased towards the layer depth. At the 15 J energy level, damage occurred due to the separation of the middle layer and the intensification of fiber fractures in the middle layer. It was concluded that the additives of CNT and ZrO<sub>2</sub> at these rates made the material embrittle.

#### 4. Conclusion

In this study, the effect of different amounts of CNT and

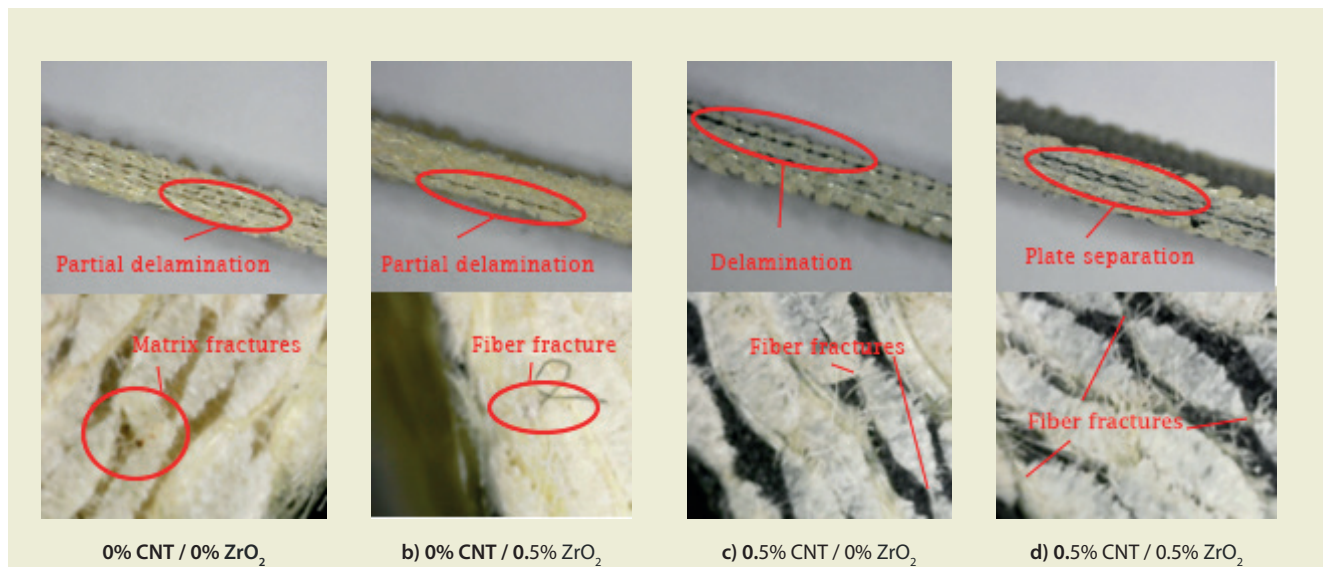


Figure 9. Impact zone damage mechanisms at 10 J energy level

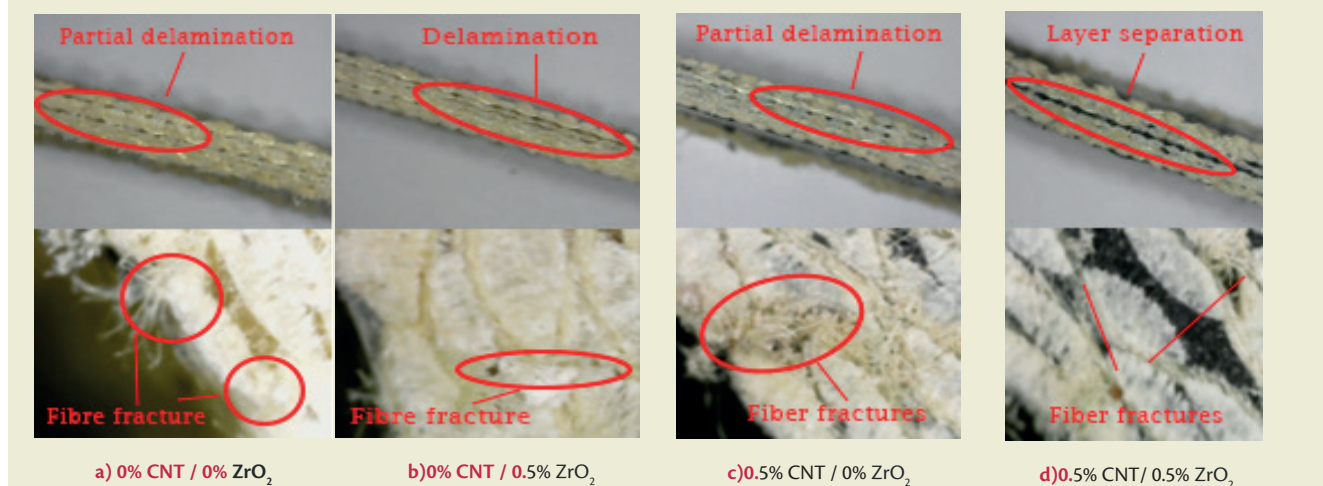


Figure 10. Impact zone damage mechanisms at 15 J energy level

ZrO<sub>2</sub> additives on the mechanical properties of aramid composite plates was investigated. As a result of the experimental studies and analyzes, the following results were obtained:

- Impact force and displacement amount were homogeneous in unadulterated samples. However, significant damage modes and nano-agglomeration were observed. The maximum force/displacement ratio was determined at a low level.
- Only 0.5% ZrO<sub>2</sub> addition enabled the material to reach its maximum strength level and serious fiber damage occurred in the impact zone. The amount of displacement was realized at a lower level. This indicates a decrease in material flexibility.
- Only 0.5% CNT addition increased the impact force and displacement amount of the material. This shows that the durability of the material has increased. However, sudden force drops due to nanomaterial agglomeration were observed.
- The 0.5% CNT / 0.5% ZrO<sub>2</sub> additive preserved the maximum force ratio of the material and kept the displacement amount at the lowest level. This showed that the use of CNT and ZrO<sub>2</sub> additives together increases the durability of the material and controls the displacement. This shows that the use of CNT and ZrO<sub>2</sub> additives together can affect the mechanical properties of the material in a balanced way. This information is of great value for the design and application of composite materials.

The results showed that suitable nano doping ratios can increase the impact resistance of the material and provide an optimized structure for the desired mechanical properties. In future studies, the determination of the optimum additive ratio by using higher nano additive ratios and the application of artificial intelligence using the data we have, and the mechanical effects of different ratio nano additives on the composite material will be focused on in more detail.

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