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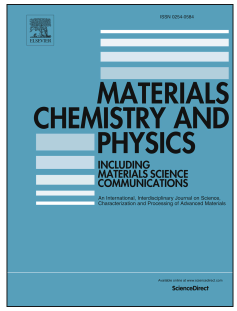


The effect of nano Zirconium dioxide and drilling on the buckling strength of epoxy based nanocomposites

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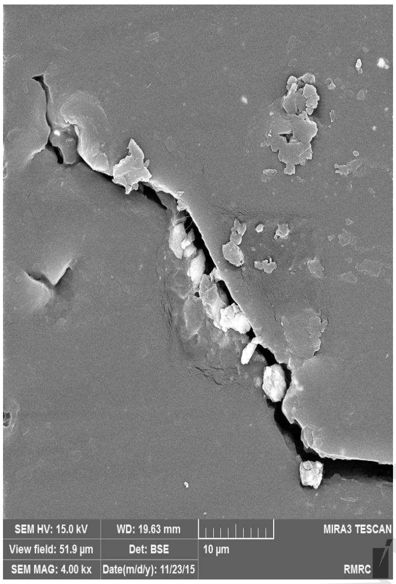
- Buckling strength of $ZrO_2$/ Epoxy nanocomposite before and after applying damage
- Applying damage with two kinds of cutting drilling tools: flat and ball end
- Studying the buckling strength and failure analysis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# The effect of nano Zirconium dioxide and drilling on the buckling strength of epoxy based nanocomposites

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**Abstract:**

In the current research, the effect of nano Zirconium dioxide ($ZrO_2$) on the buckling strength of epoxy resin based composite was studied experimentally before and after applying damage. The nano particles were added to the epoxy resin matrix with 1 to 4 wt% of the composite. The applying damage to the samples was made with two kinds of cutting drilling tools: flat end and ball end drilling tools and with 1.5 and 3 millimeter depth of hole. The buckling tests were done for neat epoxy composites without nano particles and for nano composite samples before and after drilling and compared with each other. The results obtained from tests showed a good enhancement on the buckling strength of nano composites about 50 percent for samples with 3 wt% of nano Zirconium dioxide. Also, the drilling process, was decreased the strength of composites,but the adding nano Zirconium dioxide retrieved this failure and increased the buckling strength of nano composite after drilling in comparison with the neat samples. Finally,the SEM showed a good dispersion of nano particles in the epoxy matrix and made a smooth surface.

**Keywords**: epoxy- nano composite- Zirconium dioxide- buckling strength- drilling

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**Introduction:**

Epoxy resins are very applicable and have been used in many applications such as making polymer composites. Polymer composites are important industrial materials with various applications [1-3]. Epoxy based composite materials have been used widely in various industries nowadays like adhesives, coatings, electronic, mechanical, and structural applications, because of their high and good mechanical properties like: low weight, ductility and modulus[4, 5], but the weakness of these materials is related to their brittle nature in spite of having numerous advantages this materials are fragile[1, 6, 7]. Epoxy resin is widely used as a host matrix for manufacturing Fiber Reinforced Polymers (FRP) with higher strength to weight ratio than metals [8-12]. Epoxy resin creates thermosetting matrices of advanced composites, displaying a series of interesting characteristics like good stiffness and specific strength, dimensional stability, chemical resistance, ease of processing and also strong adhesion to the embedded reinforcement[6, 13, 14]. Many researchers have been studied to enhance the mechanical properties of epoxy based composite polymers and improvement against crack development in this kind of materials. The most works related to adding micro and nano fillers to epoxy resin matrix to overcome the epoxy weakness. It has been proven that use of particulate fillers can improve the material properties of epoxy resin and make a good mixture with high improvement[5, 12, 13, 15].Use of smaller fillers (nano fillers) in comparing with the micro and macro fillers is a good solution of this problem and also retain the low weight of epoxy polymers[6, 16]. Many studies have indicated that the excellent properties of nanocomposites distinctly depend on the particle dispersion, which has a close relationship with the manufacturing method [6, 17]. Other advantages of use of nano fillers are about homogeneous distribution within the epoxy matrix that lead to the good reinforcement effect that retain the other good mechanical properties of epoxy and decrease the residual stresses[8, 18].Recently, the nano particles like: nano silica, nano clay, cnt and nano Zirconium dioxide had been used for this purpose as nano fillers to increase the mechanical properties of epoxy based composites like toughness and modulus [4, 19]. In this case, most studies have been focused to the addition of only a few weight percentages of nano materials to neat polymers due to achieve the good results[11].Hui Zhang et al. used nano silica to epoxy resin matrix to enhance the mechanical properties of epoxy composites and results showed14% enhancement on the static/dynamic modulus, micro hardness, and fracture toughness of the nanocomposites [11].

The effects of nano fillers on the properties of the epoxy based composites depend on filler type, shape and size of particles[1, 13]. Metin Sayer used nano silica and nano boron carbide to glass/epoxy composites. The results showed that the strength of the nano composite was significantly influenced by particle weight fractions, different particle sizes and different ceramic particles[20]. Among various nano sized fillers, zirconium oxide ($ZrO_2$) fillers have attracted considerable attention because of the unique physical properties as well as their low cost and extensive applications in diverse areas[19, 21].

Zirconium dioxide has excellent properties like: high strength, hardness and fracture toughness, good wear resistance, and excellent chemical resistance [7, 8]. It appears that use of nano Zirconium dioxideas nano filler in epoxy based composites can be suitable choice because of the good properties of this nano filler as told above. Rosa Medina et al. used a series of composites with varying amounts of $ZrO_2$ nanoparticles and their morphology and mechanical properties were studied. The results showed improvements of more than 37% on the modulus of the nano composites[6]. Buckling strength of polymer composite materials is an important parameter because of their low weight and thickness[1, 22-24].The buckling load of a these composites, depends on a variety of parameters like properties of reinforcement. It is important to improve the compression strength of composite polymers and preserve the weight and thickness of them simultaneously [1, 20, 25]. In addition, most of structures made of epoxy polymers like airfoils and automotive structures are prone to damage because of impacts or damage because of drilling of holes. In many structures, the holes are often drilled for join the structures to each other. These types of damages are created by drilling and make this section of structure weaker that it causes to fail at less load value. So it is seen as necessary to investigate the strength of structures after hole creation [26-28]. In this research, the effect of nano $ZrO_2$ on the buckling strength of the epoxy polymer composites before and after drilling was investigated.

## 2. Experimental procedures

### 2-1. Materials

Epoxy resin used in this research was bisphenol A, EC 130-LC provided by Altana Co. with epoxide equivalent weight 185-192 g/eqiv. The curing agent was a nominally cycloaliphatic polyamine, Aradur® 42 supplied by Huntsman Co. The nano Zirconium dioxidewas purchased from Nanosany Co. with the average size of 20 nm and purity of 99.95%.

### 2-2. Samples preparation

The procedure of reinforcing the epoxy resin was done in some steps to prepare homogenous mixture. For making the mixture of nano Zirconium dioxide and epoxy resin, the nano particles with 1 to 4 weight percentage of composite (wt%) were mixed well with the liquid epoxy resin by mechanical stirrer for 2 hours and with 1000 RPM. In the current study, the mixture was homogenized by ultrasonicating (ultrasonic SONOPLUS-HD3200, 50% amplitude, 20 KHz and pulsation; on for 10s and off for 3s) for 8 minutes. At this stage 23 per (per hundred resins) of cycloaliphatic polyamine was added as hardener based on stoichiometric ratio. The nano mixture was set at room temperature for 24 hours and then poured into the specific silicon casts and finally, were cured in oven from $50^{0c}$ to $130^{0c}$ each 2h with $20^{\circ c}$ temperature enhancement interval. All the specimens of buckling test were prepared according to the ASTM D: 6641 standard with the size of 12*140*4.5 mm in with*length*thickness and at least, 5 samples were prepared for each test.

**2-3. drilling process and buckling test**

In this research, the buckling test was done for the neat and nano composite samples before (un-damaged samples) and after drilling (damaged samples). For this purpose, two kinds of drilling tools were used. The Cylinder radius end (ball end) and End mill (flat end) with 8 millimeter in diameter. Fig. 1(a) and 1(b) show the Cylinder radius and End mill drilling tools respectively. The depth of drilling for damaged samples was 1.5 and 3 mm in the middle of samples. Applying damage process was done by CNC milling machine with 1000 RPM of spindle and feed rate of 30 mm per minutes. Fig. 2 shows the damage process on the samples by drilling tools.

All the experiments were done at room temperature with the ASTM-150 universal testing machine (Santam Company- Iran) by applying axial compression force with a loading rate of 1 mm/min and the results of buckling tests were measured by the testing machine according to the load-displacement curves obtained from tests. Fig. 3 shows the sample under buckling test.

**3- Result and discussion:**

**3-1. Effect of nano $ZrO_2$on the buckling strength of epoxy composites before drilling**

Figures 4 and 5 show the result of buckling tests of the undamaged samples. According to the results, it was obtained that the buckling strength of the composites was increasing with adding nano Zirconium dioxide to 3 wt% and then decreasing gently for the samples with 4 wt % of nano filler. The best strength was related to the composites with the 3 wt% of nano Zirconium dioxide that had enhancement about 50 percent in comparison with the neat samples without nano filler. These results can be explained by the spherical geometrical structure of Zirconium dioxide that can fill the matrix bulk and creates strong bonding with the resin epoxy matrix that created a good adhesion particle-polymer and made arough surface, so the more stresses can be distributed by the rough surface [6, 19, 29]. Fig. 6 shows the chemical bonding of epoxy matrix and nano particles by the SEM picture. But when the nano particles increased more than 3 wt%, the matrix becomes saturated and then, the smooth surface creates that typically tends to brittle fracture[19]. The saturated mixture is shown by the SEM pictures in Fig. 7.

**3-2. Effect of drilling on the buckling strength of neat epoxy composites**

In this section, the effect of drilling on the buckling strength of neat epoxy composite was investigated. The damage process was done by two drilling tools: flat end and ball end and the depth of the damages were 1.5 and 3 millimeters on the middle of samples. Fig .8. Shows the damaged samples under the buckling test.

The results of buckling tests were shown in Figure .9 by the force-displacement plot. According to this plot, it was obtained that the applying damage by the flat-end drilling tools had more effect on the decrease of strength of the samples in comparison with the ball-end about 14.5%. The important note is that the damaged samples with the ball-end and depth of the 3mm had more strength in comparison with the samples that were damaged with the flat end and depth of 1.5 mm; though, the depth of the damage was larger in ball end.

This manner can explain by the SEM picture of the damaged zone of the sample that was shown in Fig .10. It was seen that in the damaged samples with the ball-end drilling tools, the depth and growth of the cracks at damaged areas are less than the samples that were damaged with the flat-end drilling tools, so the strength of the damaged composites with the ball-end drilling tools are more [26, 30-33]. Figures 11 (a) and (b) show the SEM picture of hole and crack growth in damaged samples with the flat end drilling tools.

**3-3- The effect of nano $ZrO_2$ on the buckling strength of damaged composites**

In this section the effect of nano $ZrO_2$on buckling strength of the epoxy composites after drilling was investigated. Fig.12 and 13 show the results of buckling tests of the damaged nanocomposite-samples with cylindrical end drilling tools and with the depth of 1.5mm and 3mm respectively. According to these plots, it was obtained that in all drilling samples, the nano $ZrO_2$ had good effect on the buckling strength of the composites and the buckling strength was increased with the increasing of the nano particles percentage to 3 wt%. In all damaged samples, the best strength was related to the epoxy composites with 3 wt% of nano $ZrO_2$with about 50% enhancement. But when the nano particles increased more than 3 wt%, the buckling strength was decreased about 30%.

This can be explained by the characterization of ZrO2 that has a higher strength than the epoxy matrix and also can create a good bonding with epoxy matrix that permits the right stress distribution into both composite phases and the nanoparticles and agglomerates act as stress concentrators[6, 34, 35]. The reduction of the buckling strength of high filler content (4 wt%) is related to the presence of the higher quantity of clusters due to the agglomeration that tend to reduce the strength of composite and also the saturation phenomenon was occurred [21, 34].

The Fig. 14 (a-d) show the SEM pictures of damaged nanocomposite samples with cylindrical end drilling tools after buckling test. The distribution of the nano particles and crack growth around the hole can be seen in these pictures.

Fig. 15 and 16 show the results of buckling tests of the damaged nanocomposite-samples with Flat-end drilling tools and with the depth of 1.5mm and 3mm respectively. In these two kind of damaged samples, the results of the test and the effect of nano $ZrO_2$ on the buckling strength of the damaged samples are nearly the same as the damaged samples with Ball-end drilling tools. In this section, the buckling strength was increased with the adding nano $ZrO_2$ particles and the samples with the 3 wt% of nano $ZrO_2$had the most buckling strength (about 55% in comparison with the neat damaged sample) and at higher contents of nano particles (4 wt%) this process became inverse, so the strength was decreased about. The effect of nano particles was the same as the previous section that told above 20% that is shown in Fig. 17 by the SEM pictures. According to these pictures it can be seen that nano particles have good distribution and good bonding with epoxy and also fill the hole and prevent crack growth some deal [5, 36-38].Also the agglomeration and saturation are shown by SEM pictures (Fig. 17 (c & d)).

**3-4. Comparison between Ball-End and Flat-End drilling tools on the buckling strength of nano epoxy composites**

In this section, the effect of two different drilling tools on the nanocomposite samples was investigated. Fig. 18 (a-d) shows the force-displacement plot of the buckling test of the damaged nano-samples. According to these plots, it can be seen that in all nano samples, the strength of the composite samples that was damaged with the Ball-End drilling tools was more than the Flat-End about 16%. It can explain by the shape and depth of the cracks that created by the drilling tools, that in the damaged samples with the Ball-End, the depth and growth of cracks were less so the strength of composite became more[39-41]. Also the nano $ZrO_2$ had a same effect for both damaged samples that had a filler role in matrix and prevent from crack growth [28, 37, 42, 43].

## 4- Conclusion

In this research, the effect of $ZrO_2$ nano particles as filler on the buckling strength of epoxy composites before and after applying damage was investigated. The nano particles were added to the epoxy matrix with 1 to 4 wt% of the composite. The damage process was done by two different drilling tools: cylindrical-end and flat-end. According to the buckling test results it was obtained that the buckling strength of the epoxy composite was increased by adding nano zirconia to 3 wt% and then was decreased slightly at the upper wt% of nano particles (4 wt%). The final results showed that the samples with the 3 wt% of nano Zirconia had the most buckling strength, about 50% by comparing with the neat samples. This procedure was same for all undamaged and damaged samples. The other aim of this paper was about the comparison between the damaged samples with different drilling tools. The results of buckling test showed that the damaged samples with the cylindrical-end drilling tools had more strength than the samples that damaged with flat-end drilling tools even in a larger depth of damage. The SEM pictures of undamaged samples showed a good distribution of nano $ZrO_2$ particles on the matrix that was created a strong bonding with epoxy that increased the buckling strength of composite. Also, the SEM pictures of damaged samples showed that the damaged area and crack growth are less in samples that were damaged with cylindrical-end drilling tools. So, in these samples, the strength of composite was more than the other damaged samples according to the reasons told above. By the way, the nano particles of $ZrO_2$ were effective parameter in the enhancement of the strength of the damaged composite as the same as the un-damaged samples. Also, the strength of the damaged nano composite was more than the neat (without nano) undamaged sample.

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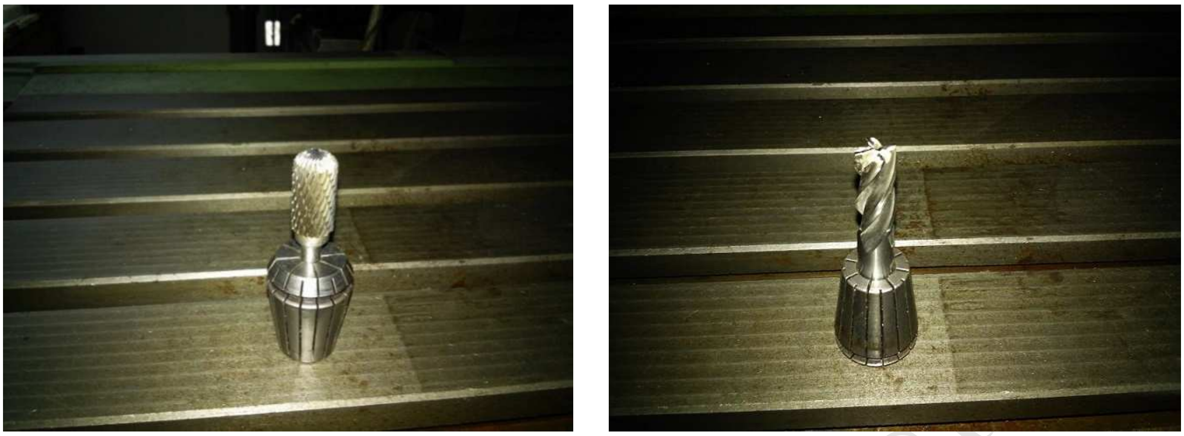


(a) (b)

Fig. 1. (a) Cylinder radius and (b) End mill drilling tools

(a) (b)

Fig .2. Applying damage process with (a) Cylinder radius-end and (b) flat-end drilling tools.

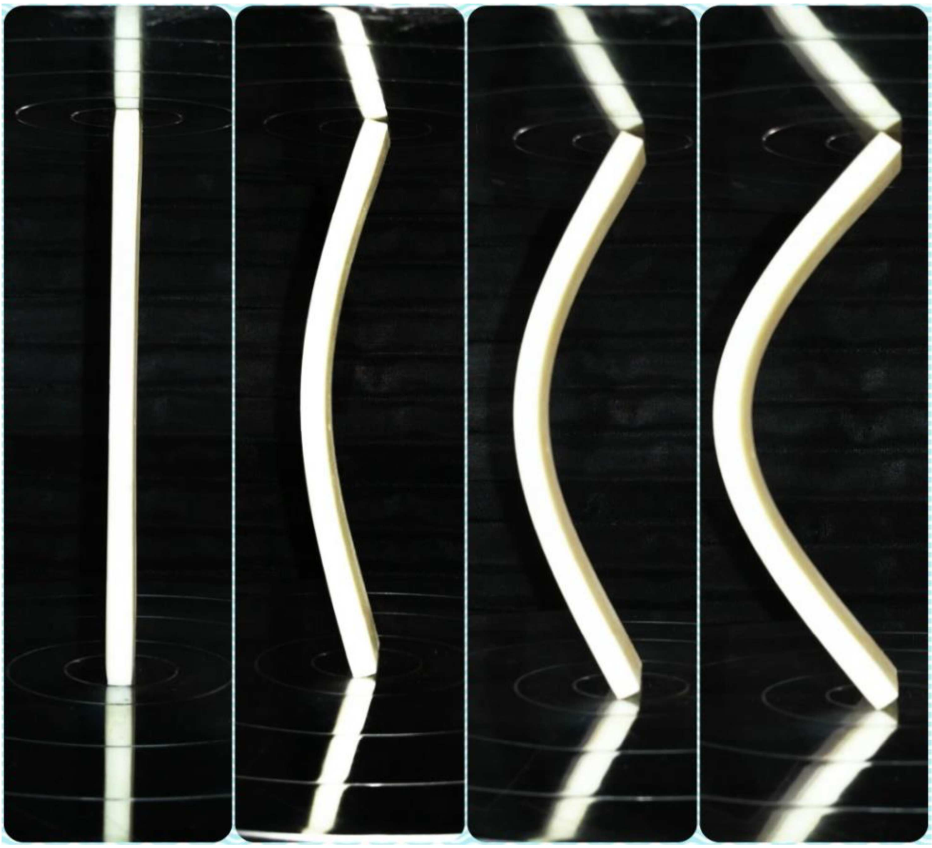


Fig 3. Buckling process of laminate

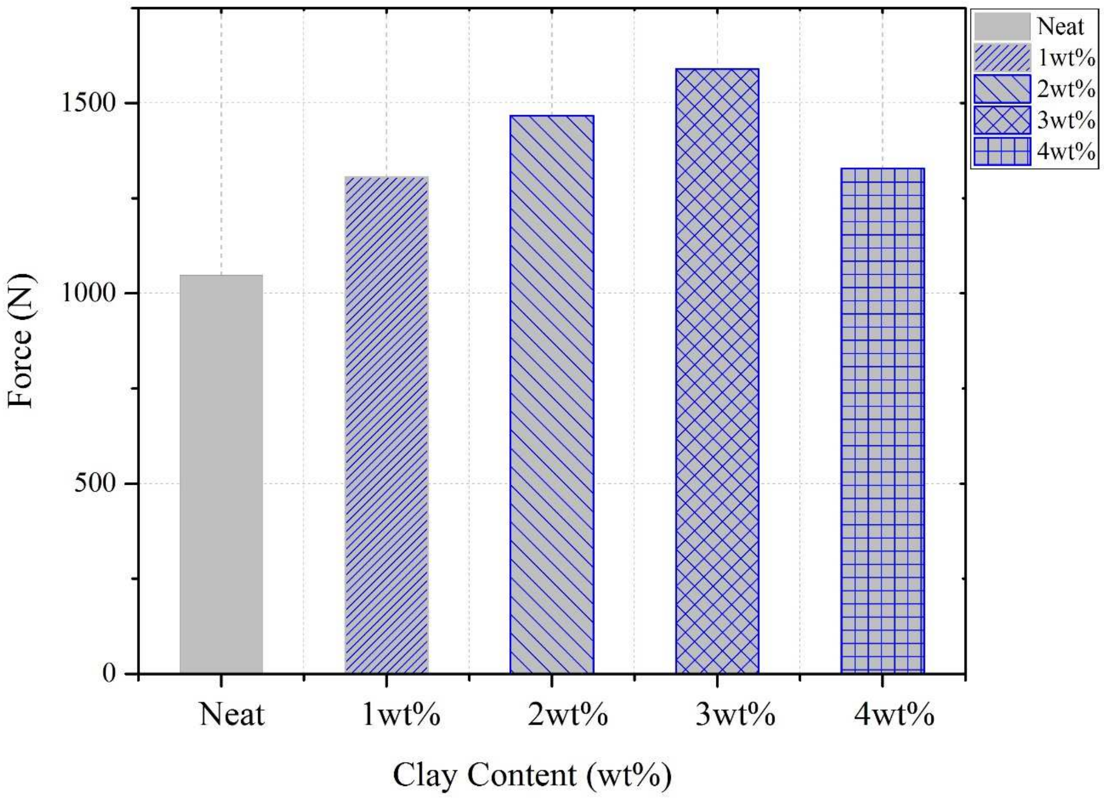



Fig .4. the results of buckling test of undamaged epoxy composites

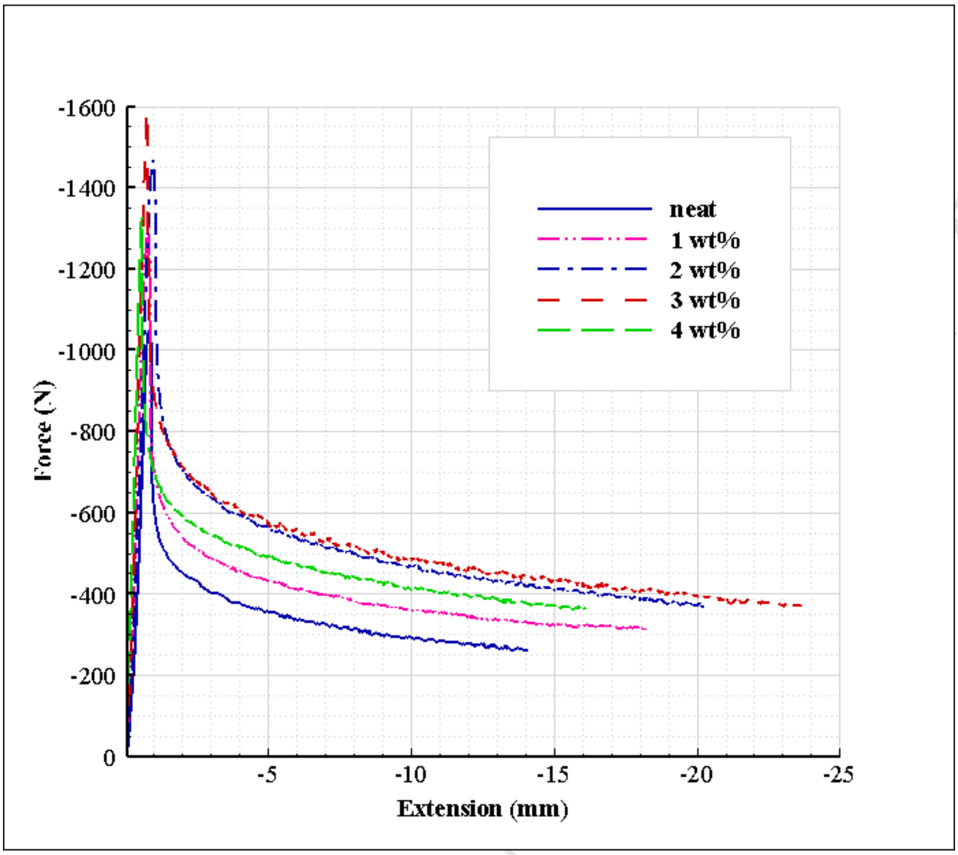



Fig .5. force-extension plot of buckling test of epoxy composite before drilling

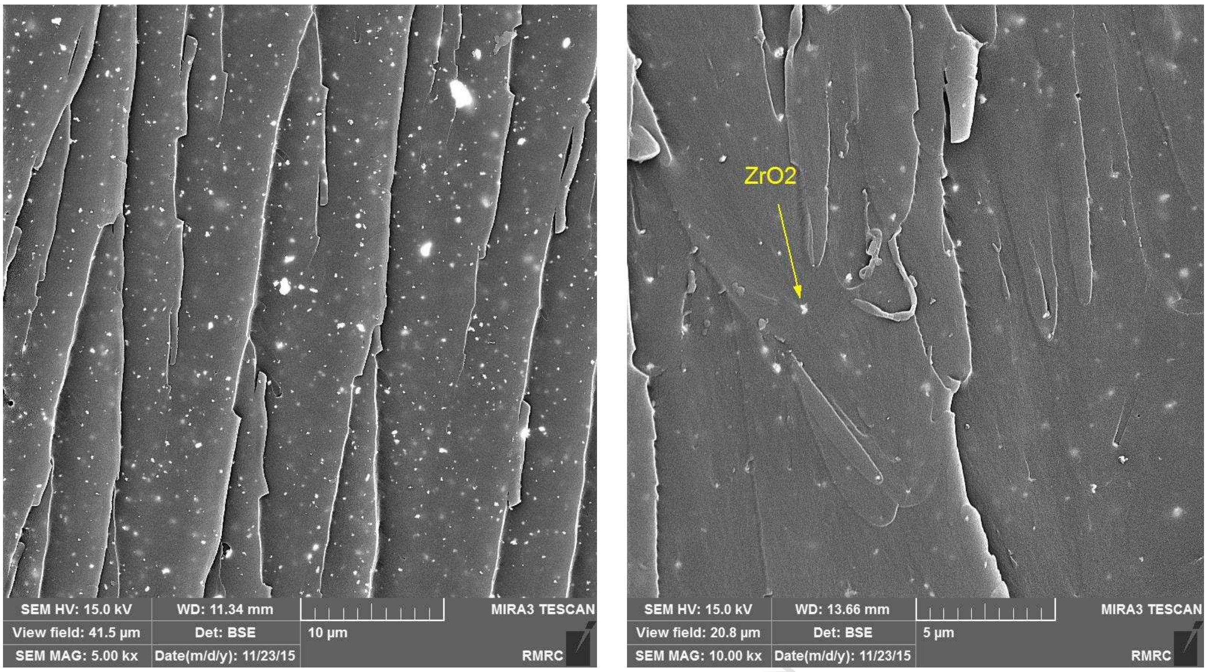



Fig .6. SEM picture of epoxy composite with 3 wt% of Zirconium dioxide

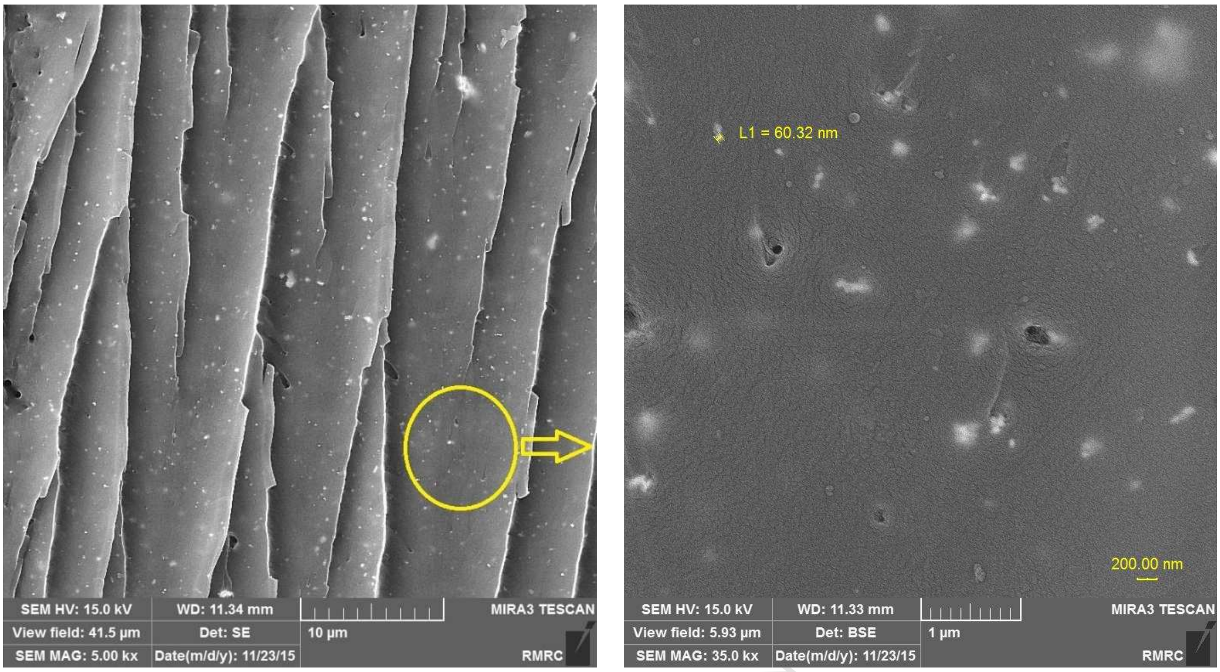



Fig .7. SEM picture of saturated mixture of nano particles and epoxy

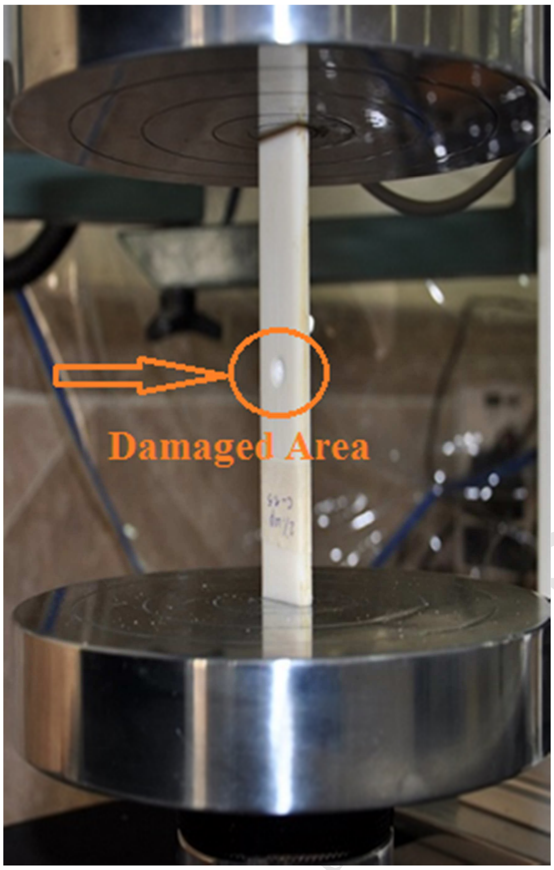



Fig 8. Damaged laminate under buckling test

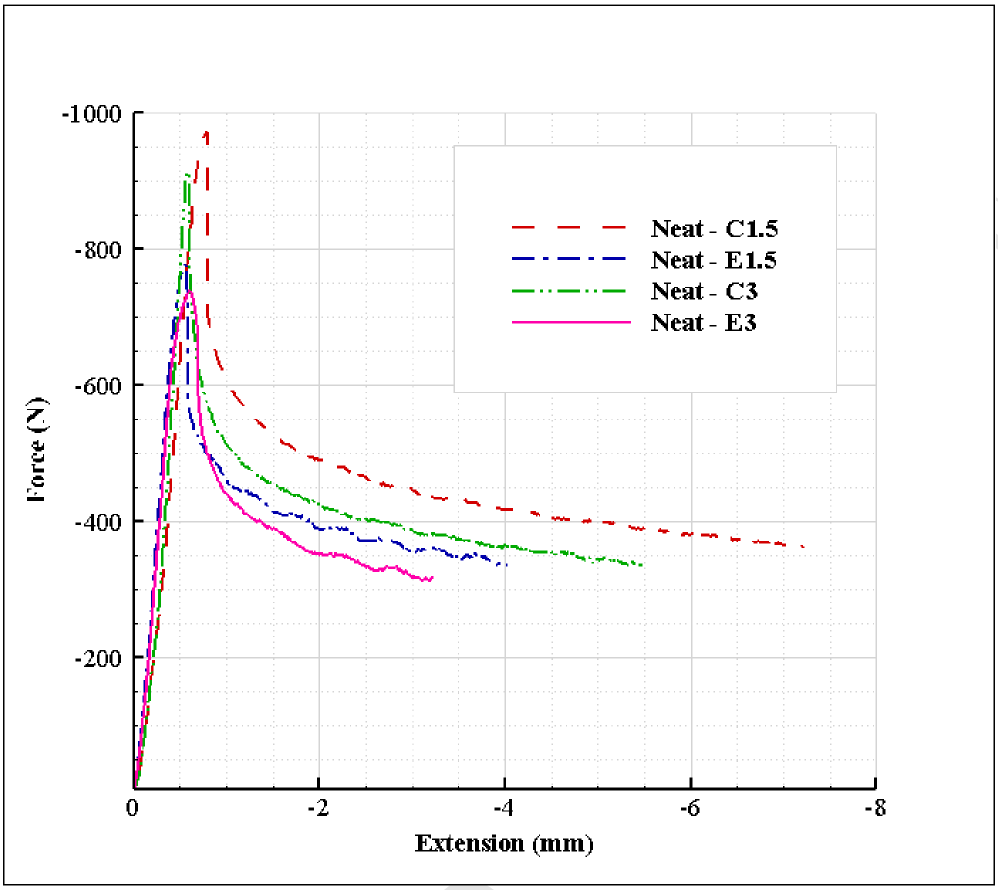
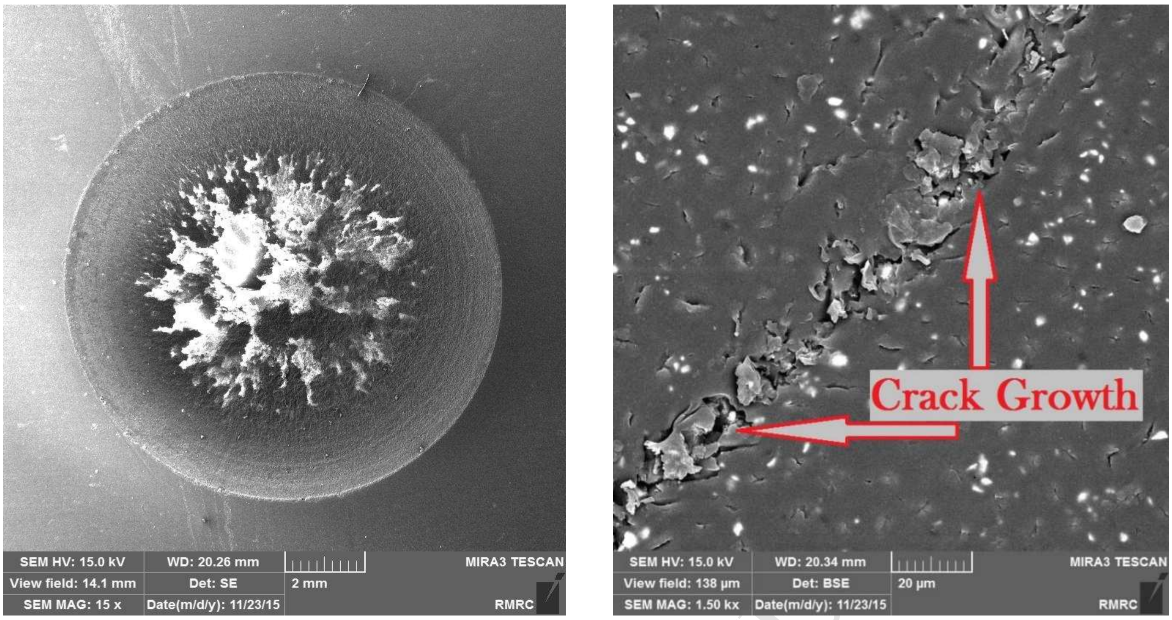
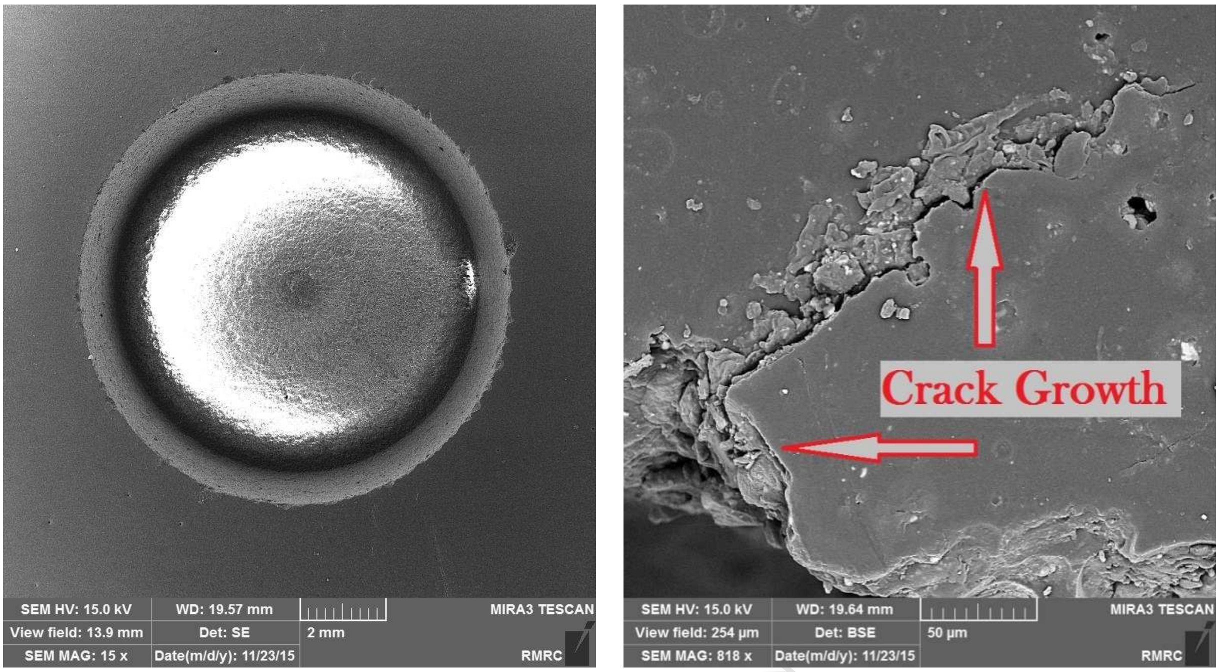



Fig .9. the force-displacement plot of neat epoxy composite after drilling




(a) (b)

Fig .10. SEM pictures of (a) hole and (b) crack growth on damaged samples with the ball-end drilling tools before test




(a) (b)

Fig .11. SEM picture of hole (a) and crack growth (b) at damaged samples with the flat end drilling tools before test

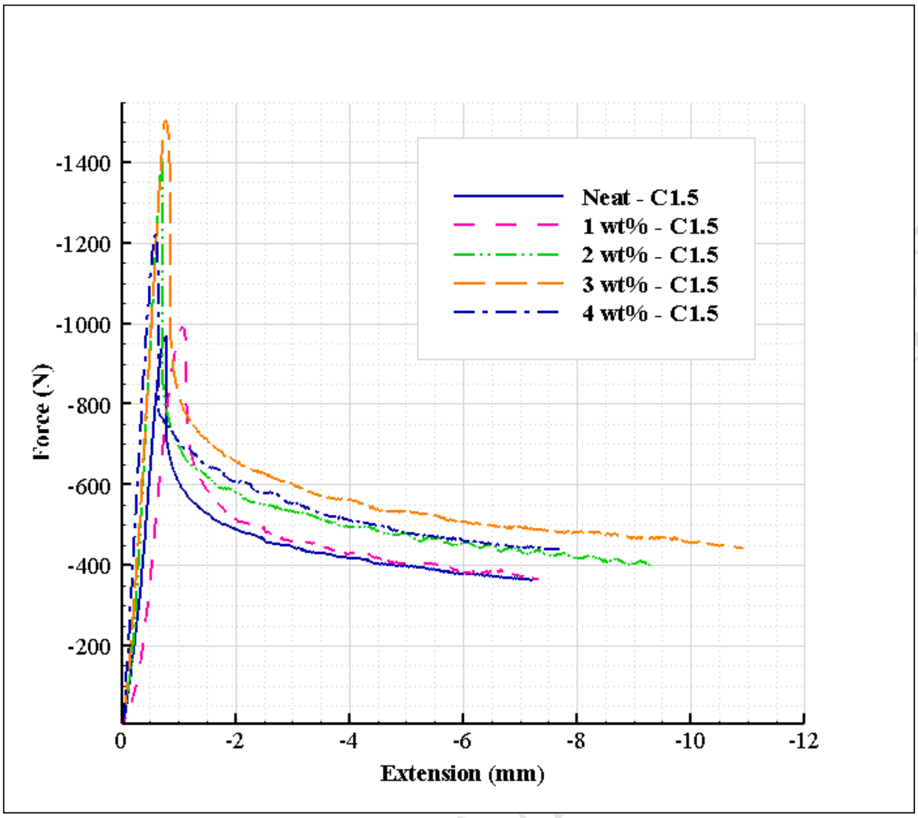



Fig. 12. The Force-Extension plot of the buckling test of the damaged nanocomposite samples with cylindrical end drilling tools and depth of 1.5 mm.

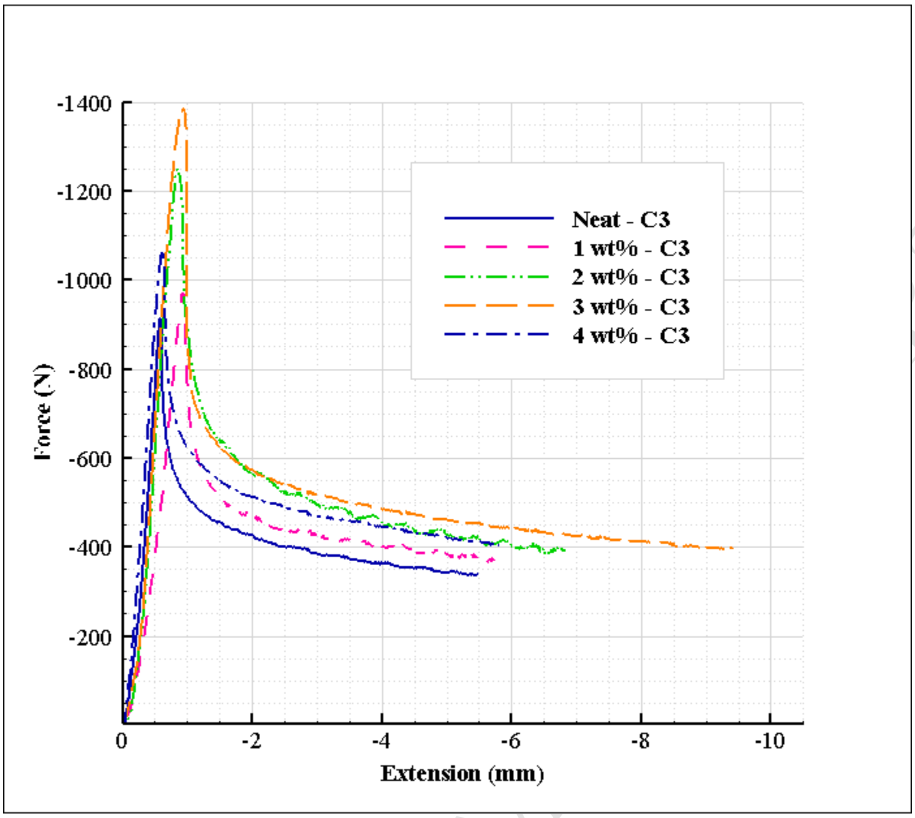



Fig. 13. The Force-Extension plot of the buckling test of the damaged nanocomposite samples with cylindrical end drilling tools and depth of 3 mm.

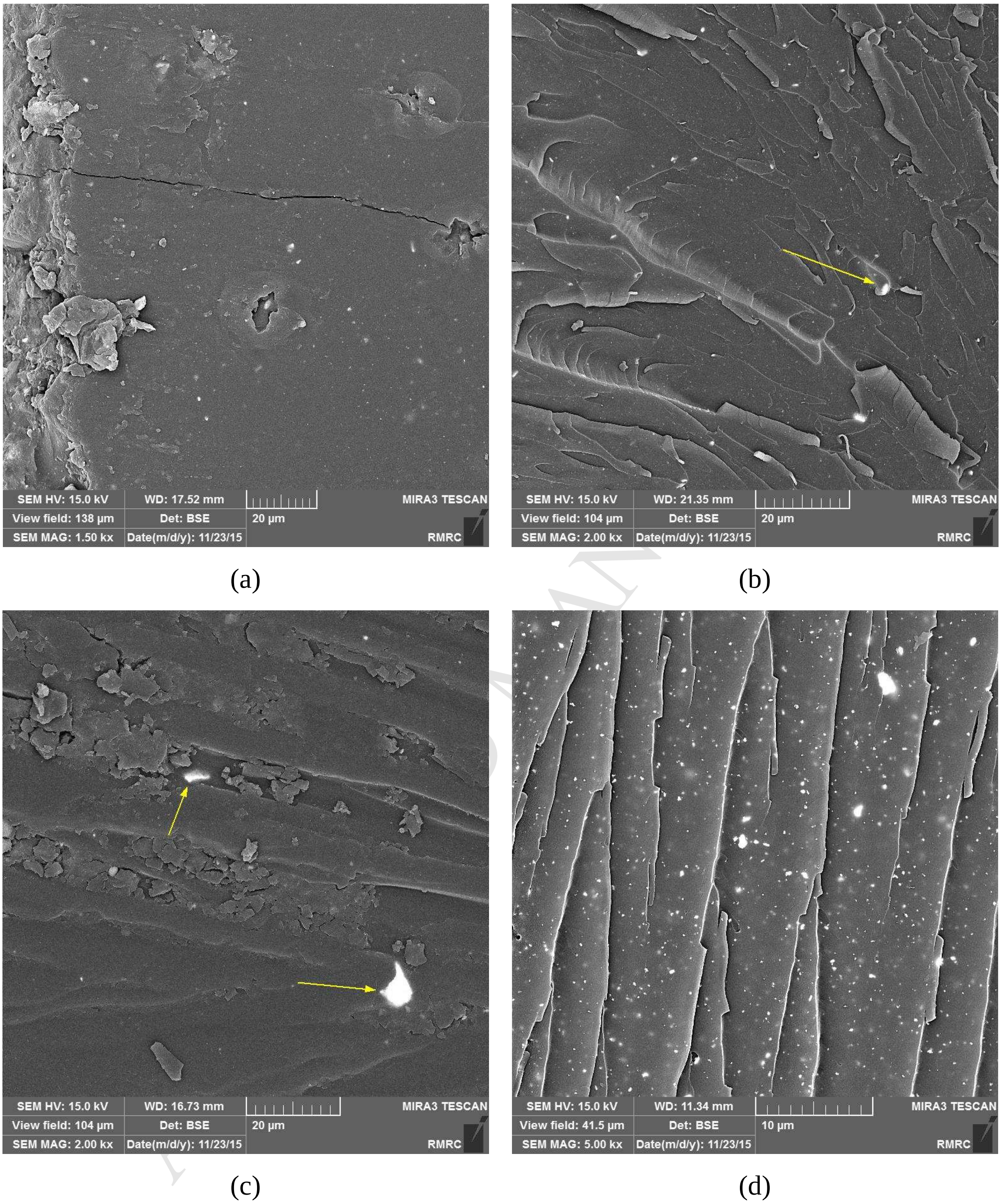



Fig 14. SEM picture of damaged samples with ball-end drilling tools and the distribution of nano $ZrO_2$in matrix. (d):Saturated mixture

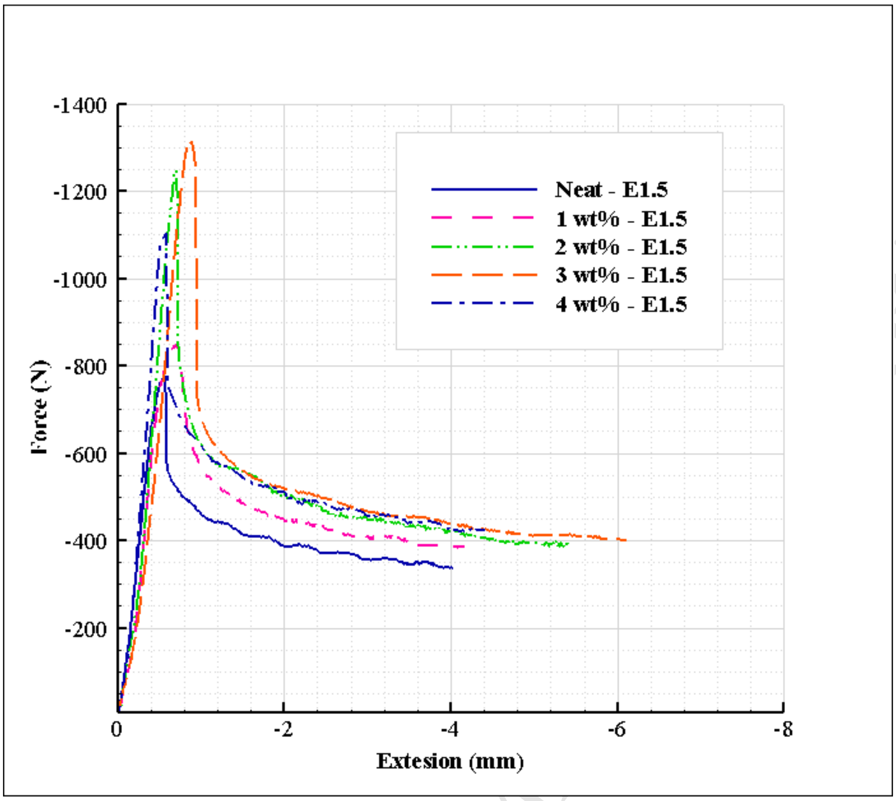



Fig 15. The Force-Extension plot of the buckling test of the damaged nanocomposite samples with Flat-end drilling tools and depth of 1.5 mm.

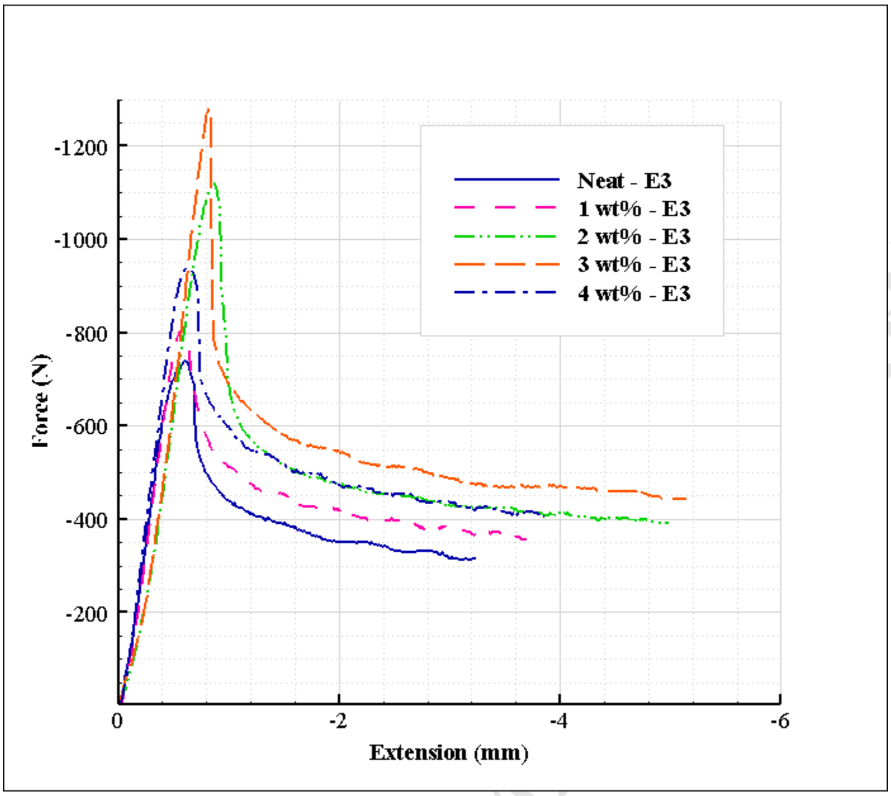



Fig 16. The Force-Extension plot of the buckling test of the damaged nanocomposite samples with Flat-end drilling tools and depth of 3 mm.

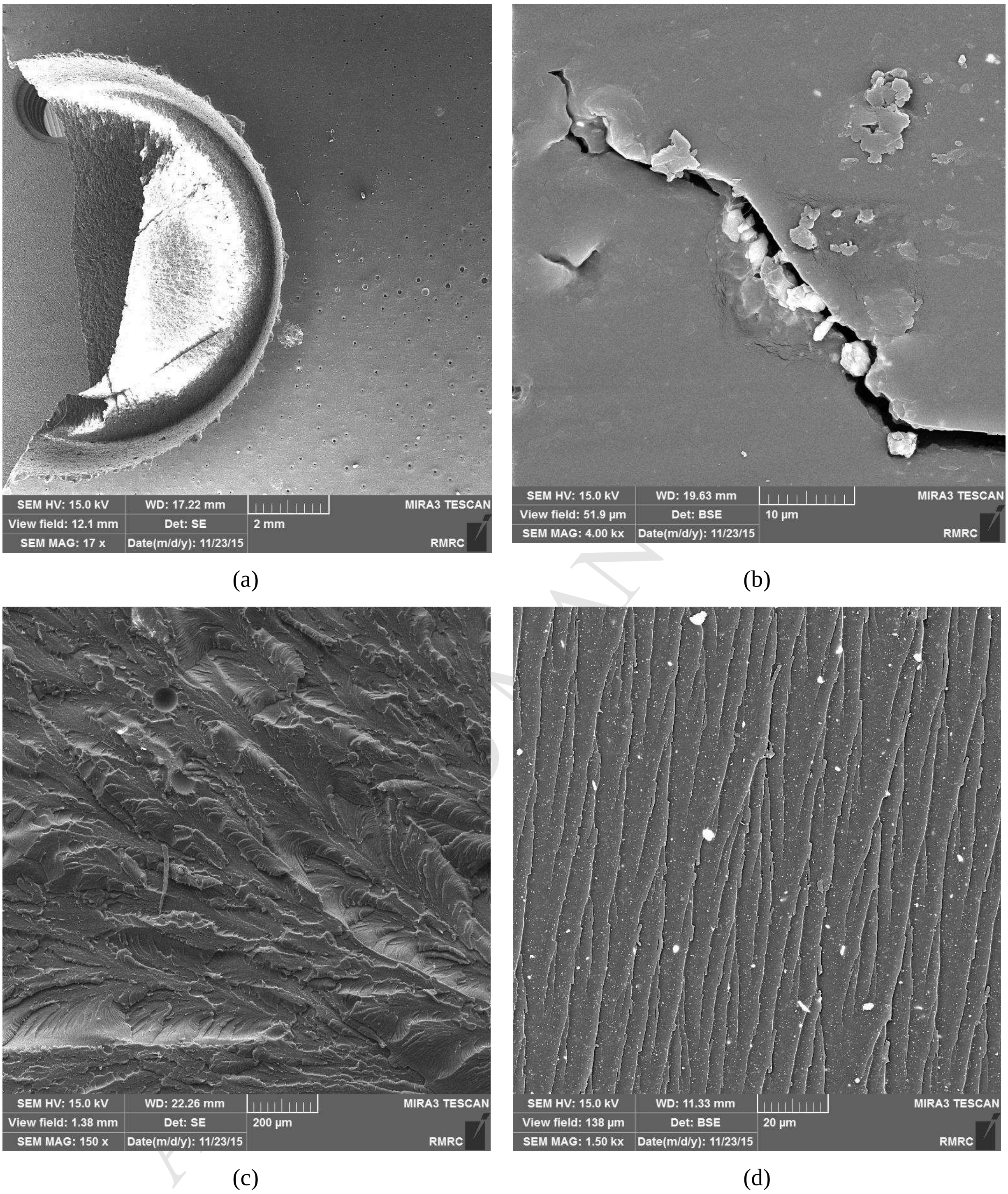



Fig 17. SEM picture of damaged samples with Flat-end drilling tools and the distribution of nano $ZrO_2$ in matrix. (c):Agglomeration (d):Saturated mixture

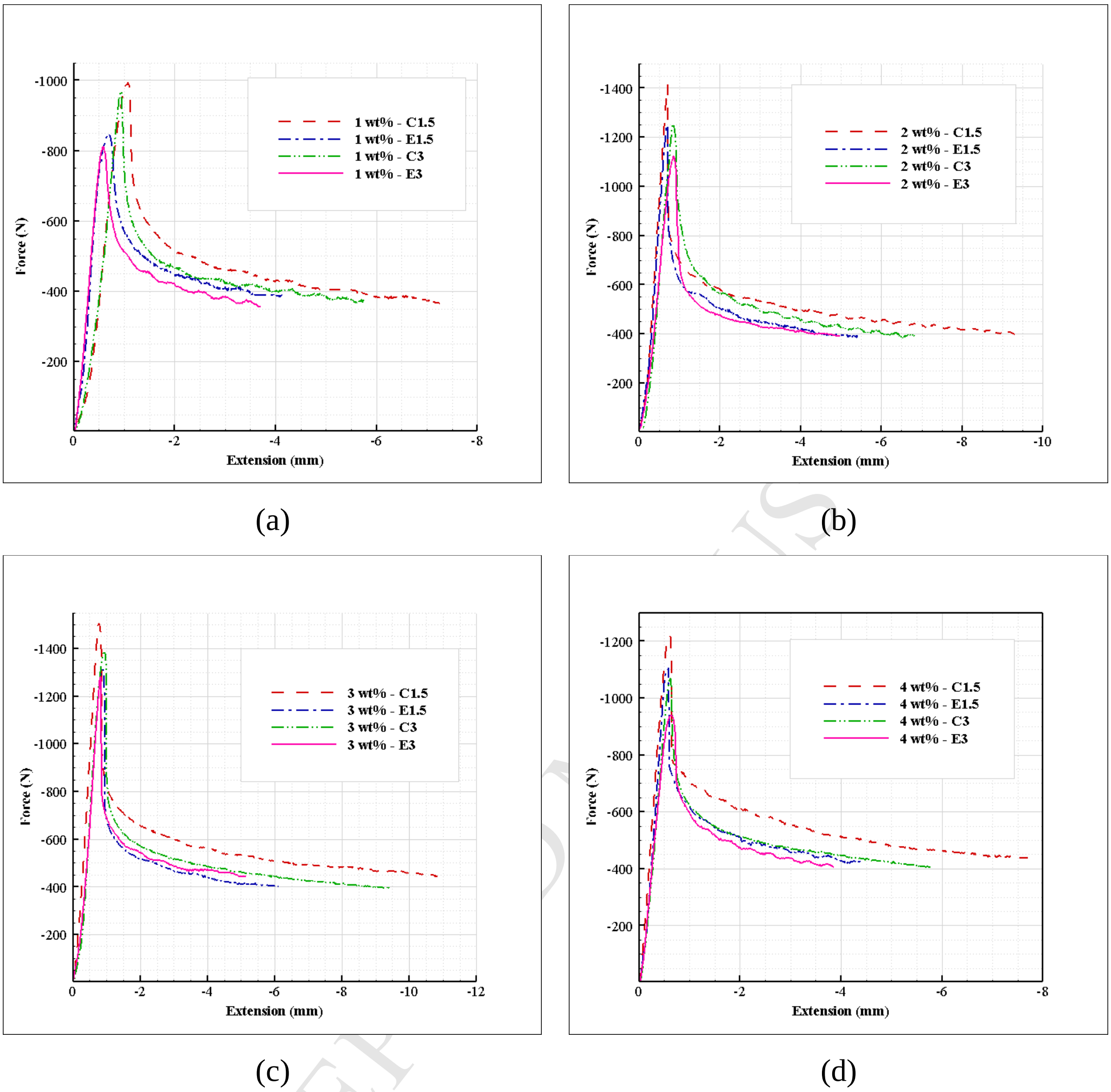



Fig. 18. The comparison between buckling strength of damaged samples with flat and Ball End drilling tools and with equal nanoparticles contents.