



Research Article

On the fracture of nano-silica/epoxy bonded single-lap steel joints

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ABSTRACT

This research aims to investigate the influence of nano-silica inclusion on the adhesive properties of an epoxy resin, and its capability in increasing the failure load of the joint with steel substrates. For this purpose, single-lap joints manufactured from Stainless Steel 304 (SS) adherends bonded together with a thin layer of the silica-reinforced epoxy resin (as the adhesive) are investigated. To improve the epoxy, silica nanoparticles (SNPs) are utilized in three different concentrations (i.e. 1, 2 and 3 wt. %). Failure loads of the joints are extracted by uniaxial tensile loading for the joints including either neat or reinforced epoxy resins. Scanning Electron Microscope (SEM) are used to examine the dispersion quality of nanoparticles into the polymer as well as the hindrance effect of the nano-silica inclusions on the micro-cracks' propagation or/and their unification. In addition, the fracture surfaces of the joints were investigated so as to relieve the failure type of the joints; whether cohesive or a combination of both adhesive and cohesive types. It was found out that the joint including silica-reinforced epoxy resin at 2wt.% best improved the failure load and changed the debonding mechanism to a fully cohesive type compared to that of the joint comprising the neat resin.

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1. INTRODUCTION

Adhesive joints are widely used in different industries, (e.g. aeronautical, automotive and maritime), owing to their special characteristics in bonding both similar and dissimilar materials. They are of several merits, including high fatigue performance, low added weight, limited stress concentrations and high bonding strength, which makes them a practical method in bonding similar and dissimilar materials [1, 2]. The applicability of such bonding method makes it highly employed in different industries, due to which adhesive joints experience several kinds of loading comprising static, fatigue and creep [3-5].

The growing use of such bonding method as well as the need for enhancing its strength brings the idea of utilizing improved adhesives to mind. Over a few years, several stu-

dies have been conducted to investigate the effects of different nanoparticles on the features of different adhesives [6-8]. Nano-silica (SiO₂) is one of the most common type of nanomaterials which reflects promising results while adding to the epoxy-based adhesives, however, fumed silica is also used to improve the mechanical properties of the adhesive [9, 10]. Nasser et al. [11] studies such nanoparticle in two different concentrations and sizes. They reported that in the same conditions, nano-silica outweighs the alumina nanoparticles when they are used to improve the adhesives. In addition, it was shown that increasing the concentration from 2.5 to 5 wt. %, caused a drastic reduction in the strength of the joints. They reported that several parameters affect the failure load of the joints with reinforced adhesive such as the concentration and size of the nano-silica.

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Improving bonding strength of composite adherends was investigated by Hassanifard and Paygozar [12], in which the joints experienced 22% greater failure load. Also, Ayatollahi et al. [13] used tinier nanoparticles (10-15 nm) and represented that the concentration of nano-silica responsible for maximum joint strength enhancement is 0.8 wt.%. Investigating the causes of improvement were also shown in a few studies. In general, improvement of the mechanical properties of the adhesive or epoxy polymers can be attributed to the reduction of the contact angle thereby improving wettability of the adhesive, improving chemical compatibility between adherend surfaces and the adhesive, and creation of hydrogen bonds [14, 15]. For instance, Tutunchi et al. [16] attributed the rise in the glass transition temperature (T_g) of the matrix to the strong interactions among the polymer and nano-silica. They also measured the contact angles in the silica-added polymer. It was shown that a measurable reduction occurred in the water equilibrium contact angle in the adhesives improved by nano-silica. They concluded that adding silica nanoparticles impressively enhance the interfacial wettability (resulting from contact angle reduction) and chemical compatibility of acrylic adhesives with adherends made of steel and glass/epoxy, due to which the shear and tensile strengths of the joints can rise. Similarly, Bahattab et al. [17] reported that the hydrogen bonds created between the silanol groups (located on the fumed silica surface) and polyurethane adhesive accounted for the improvements in the thermal properties, surface and adhesion features of the polyurethanes. Another research [18] were also took the hydrogen bonding into account and reported the same results in improving the mechanical properties of the improved-adhesives due to them.

Another cause of the improvement happens in the joints with the reinforced adhesives, goes to the creation of micro-cracks in the adhesives before joint failure. This lead to more energy absorption, thereby enduring high levels of failure load via the joints. In this respect, Razavi et al. [19] reported that shear bands' formation by plastic deformation can account for the delay of the micro-crack growth, by which the failure loads of the joints are augmented.

This work mainly aims to investigate the improvements in the failure load of the joints including silica-improved adhesives prepared at three different concentrations. The quality of the nanoparticles' dispersion as well as fracture surfaces are also to be studied. Changing debonding mechanism from a combination of adhesive and cohesive types to the only cohesive type is investigated as a potential cause of improvement, followed by observing the crack propagation and its hindrance by nanoparticles through scanning electron microscope (SEM) images.

2. EXPERIMENTAL WORK

To do the experiments of the study, several steps were taken, including preparation of the silica-reinforced epoxy

resin, manufacturing of the joints as well as uniaxial tensile testing of the joints. Afterwards, some images were prepared via SEM to check the quality of the dispersion and the reasons of any improvement in the failure load of the joints with reinforced epoxy resin. Debonding mechanism, either cohesive or a combination of adhesive and cohesive types, was also investigated.

2.1. Nano-Silica/Epoxy Preparation

To prepare the reinforced epoxy resin, silica nanoparticles (SiO₂ amorphous, US-Nano), with an average size of 20-30 nm, were added in three different concentrations (i.e. 1, 2 and 3 wt.%) into the resin part of a two-component epoxy-resin system (EPL1012/EPH112) including resin and hardener which should be mixed together in the weight ratio of 100:12, respectively. Prior to this process, the resin was diluted by acetone to facilitate the dispersion of the nanoparticles in order to have a well-dispersed mixture [20]. Then, the mixture was blended in a constant speed in a magnetic stirrer (700 RPM) for a few minutes and subsequently was put in a sonication bath for a short period of time. Eventually, the vacuum apparatus was utilized in order for the acetone to disappear. All equipment used to prepare the silica-reinforced epoxy resin were shown in detail in the previous study [20].

2.2. Bonding Process

To manufacture the joints, the contamination removal process of the bonding areas of the stainless steel 304 substrates [21] was done through several steps, as listed in [22]. After surface preparation step, two adherends (120 mm×20 mm×2 mm) of each joint were attached together by one thin layer (30 mm×20 mm×0.1 mm) of the silica-reinforced resin epoxy of different weight percentages as an adhesive layer. To avoid any potential sources of errors, the joints of improved epoxy resins were manufactured in three batches. The joints manufactured in one batch of this work, were shown in Figure 1. The joints prepared from both neat and silica-improved epoxy resins (1, 2 and 3 wt.%) would be tensile tested in order to extract the load-displacement diagrams. Hence, each end of the joints was adhered to a tap in order for the alignment of the joints while being put into jaws of the tensile testing machine.

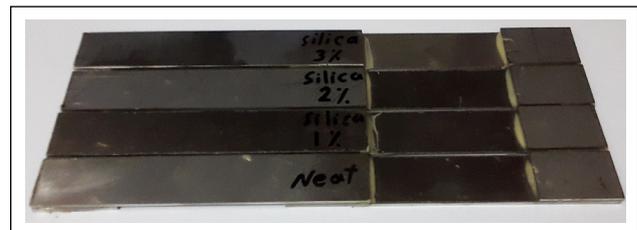


Figure 1. The specimens tested in the study. The samples (batch 2) prepared by neat and silica-reinforced resin epoxies including 1, 2, and 3 wt.%.

2.3. Tensile Loading

All twelve samples of the study, manufactured by both neat and silica-reinforced epoxy resins, were tested in a universal tensile testing machine (SCHUTZ + LICHT Pruftechnik, 70 KN, Germany). The specimens were tensile loaded with a crosshead speed of 1 mm/min.

3. RESULT AND DISCUSSION

Comparing the outcomes from the tensile testing, the concentration at which the joint experiences a higher failure load was found. To guarantee the accuracy of the process of the nano-silica/epoxy preparation, SEM images were taken to check the dispersion quality, followed by some other SEM images taken from the fracture surfaces of the joints after debonding.

3.1. Dispersion Quality

To approve the accuracy of the obtained results some SEM images were taken via a scanning electron microscope (MIRA3 FEG-SEM, Tescan, Czech Republic) in order to illustrate the quality of the nanoparticles dispersion into the epoxy resins, despite the fact that several measures were taken into account while preparing the reinforced epoxy resins. Figure 2 indicates the dispersion quality of the nanoparticles into epoxy resin improved by 2 wt.% nano-silica, with two different magnifications. The figures show almost well-dispersed epoxy resins which guarantee the quality of the preparation process.

3.2. Failure Load

To obtain the tensile response of the joints with neat and reinforced epoxy resins, all the twelve specimens were tensile tested. The results comprising the values of the failure load and the elongation at failure were gathered in Table 1. Given the failure loads of the corresponding joints in the batches, the joint indicating higher failure load was chosen, for which the load-displacement curves are shown in Figure 3. It can be observed that the failure load of the joint with 2wt.% nano-silica improved by about 25.7%, whereas this enhancement for the epoxy resin improved by 1 wt.% nano-silica is of a lower amount as 14.1%. It is worth noting that the joints including the epoxy resin added by 3 wt.% of SNPs reflected a reduction in the failure load (-6.4 %). Moreover, the elongation at failure was extracted for all the joints. It was shown that the elongation at failure rose as a function of nano-silica concentration. In spite of the failure load, for which 2wt.% reflected better improvement, elongation at failure raised almost linearly when raising the weight percentage of the nano-silica incorporated.

3.3. Debonding Mechanism

Debonding mechanism of the epoxy resin joints is of high importance owing to its effects on the amount of the failure load. It generally can be either of purely adhesive type, cohesive type or a combination of both. In this regard, fracture surfaces of the joints with neat and 2 wt.% silica-reinforced resin epoxies were photographed in detail (Fig. 4). Demonstrations pointed out that with adding NSPs into the epoxy resin, the debonding mechanism changed from

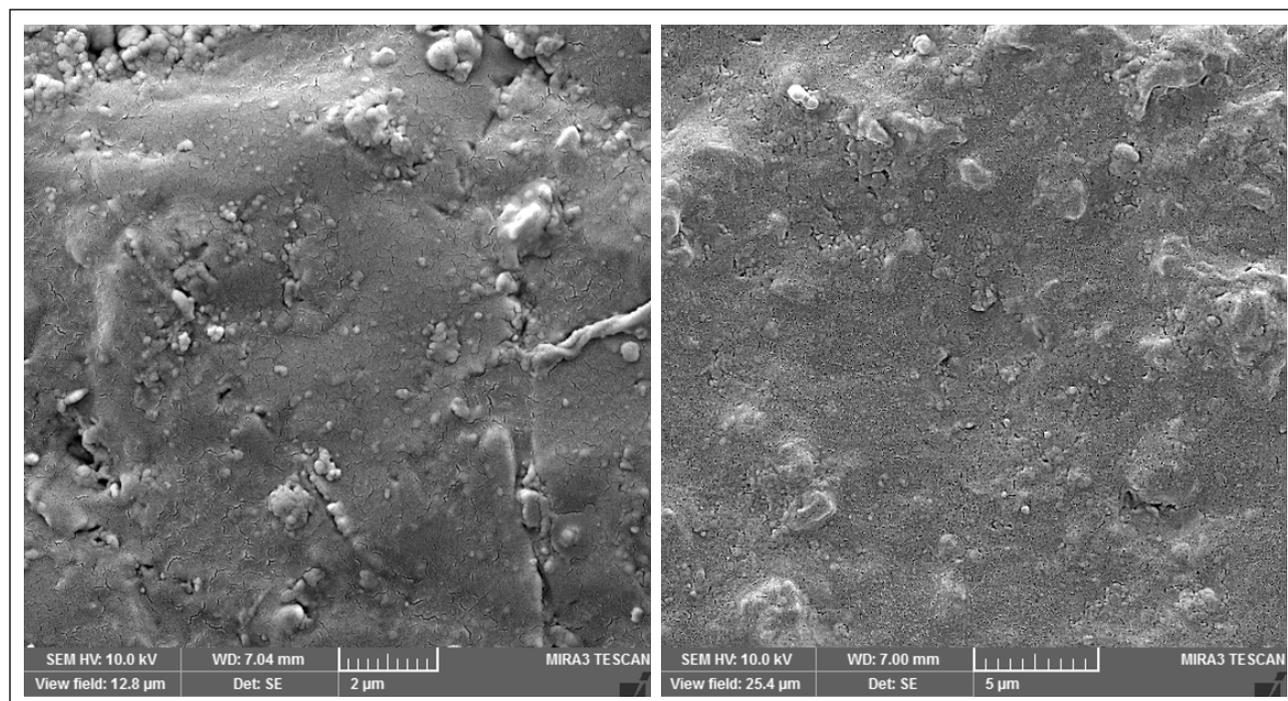


Figure 2. Dispersion quality of the silica-reinforced resin epoxy with 2 wt.% nano-silica, demonstrated in two different magnifications.

Table 1. Consequences of the joints tested

Epoxy resin type	Failure load (N)			Elongation at failure (mm)		
	Batch 1	Batch 2	Batch 3	Batch 1	Batch 2	Batch 3
Neat	3095	3100	3175	1.97	2.00	2.04
Silica- improved (1 wt. %)	3623	3554	3597	2.19	2.20	2.17
Silica- improved (2 wt. %)	3864	3991	3943	2.51	2.69	2.61
Silica- improved (3 wt. %)	2901	2971	2946	3.10	3.07	3.08

The values shown in bold refer to the joints of higher failure loads.

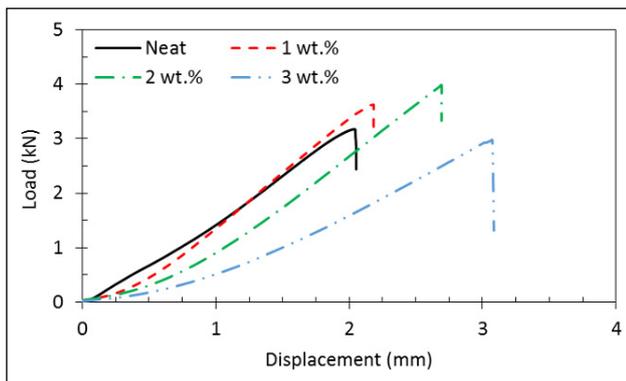


Figure 3. Failure loads of the joints with and without silica-modified adhesives. For each kind of silica-reinforced epoxy resin, the curve was plotted for the joint of the highest failure load chosen among the corresponding joints.

a mixture type- comprising both adhesive and cohesive types- to a fully cohesive type. This improvement itself can be a cause of improving failure load in the joint with epoxy resin including NSPs at 2 wt.%. However, another reason was mentioned in the following sections.

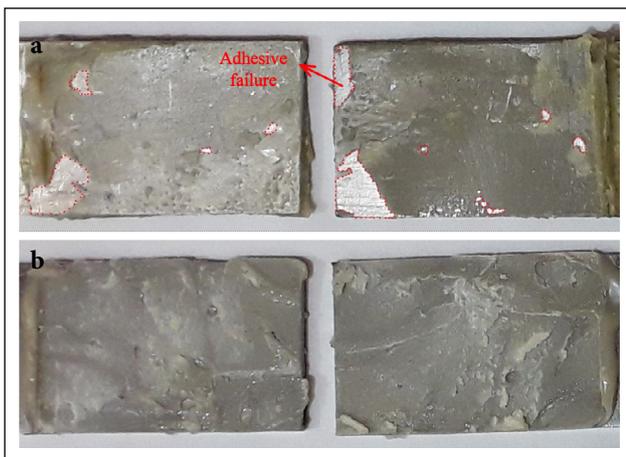


Figure 4. Fracture surfaces of the joints with (a) neat epoxy resin, and (b) silica-improved epoxy resin at 2 wt.%, indicating the failure mechanism. The locations circumscribed by red dots indicate the locations of the adhesive failure occurrence.

3.4. Fracture Surfaces

The fracture surfaces of the epoxy resins in the chosen joints were studied by SEM images, as well. As shown in Figure 5-a, it can be found out that the modified adhesive by 2wt.% nano-silica has rougher surface which can be attributed to its failure type and the higher load borne at failure. Rougher fracture surface can also implies a better interaction between the nanoparticles and polymer which lead to higher bonding strength. On the other hand, the epoxy resin improved by lower amount of NSPs depicted a smoother fracture surface which can be a reason of smaller failure load of such joints (Fig. 5-b).

3.5. Crack Growth Postponement

In general, failure mechanisms of the silica-reinforced epoxy resins include shear failure of the matrix, interfacial debonding of the particles from the matrix and crack pinning and bowing [23-26]. The latest can be explained through describing the crack propagation inside the matrix. In the polymers reinforced by nano-particles, a macro-crack always initiates and propagates gradually as the result of expansion and coalescence of micro-cracks [27, 28]. Then, the crack front reaches the agglomerations of the nanoparticles, due to which it is hindered and consequently continue to propagate into the matrix in different directions (see Fig. 6-a). In brief, the agglomerations hamper the crack propagation and bridge it over the crack faces [29, 30]. With further loading, the particles-resin interface would be debonded and the crack face would gradually open, thereby continuously expanding the area of particle bridging in the vicinity of the crack tip. The crack deflection or its path perturbation, as a consequence of particle debonding, causes the area of the fracture surface to rise. This process creates some hemispherical holes into the matrix and increases the roughness [31-33]. This process can be considered as the most effective factor in dissipating the energy in the matrices reinforced by nano-particles.

In the current research, in order to survey the potential causes of the betterments occurred in the failure load of the joints of silica-modified epoxy resin, especially those that improved by 2wt.% nano-silica, two other SEM images of larger magnifications were taken from the fracture surfaces (Fig. 6). One of the possible reasons for improving the fai-

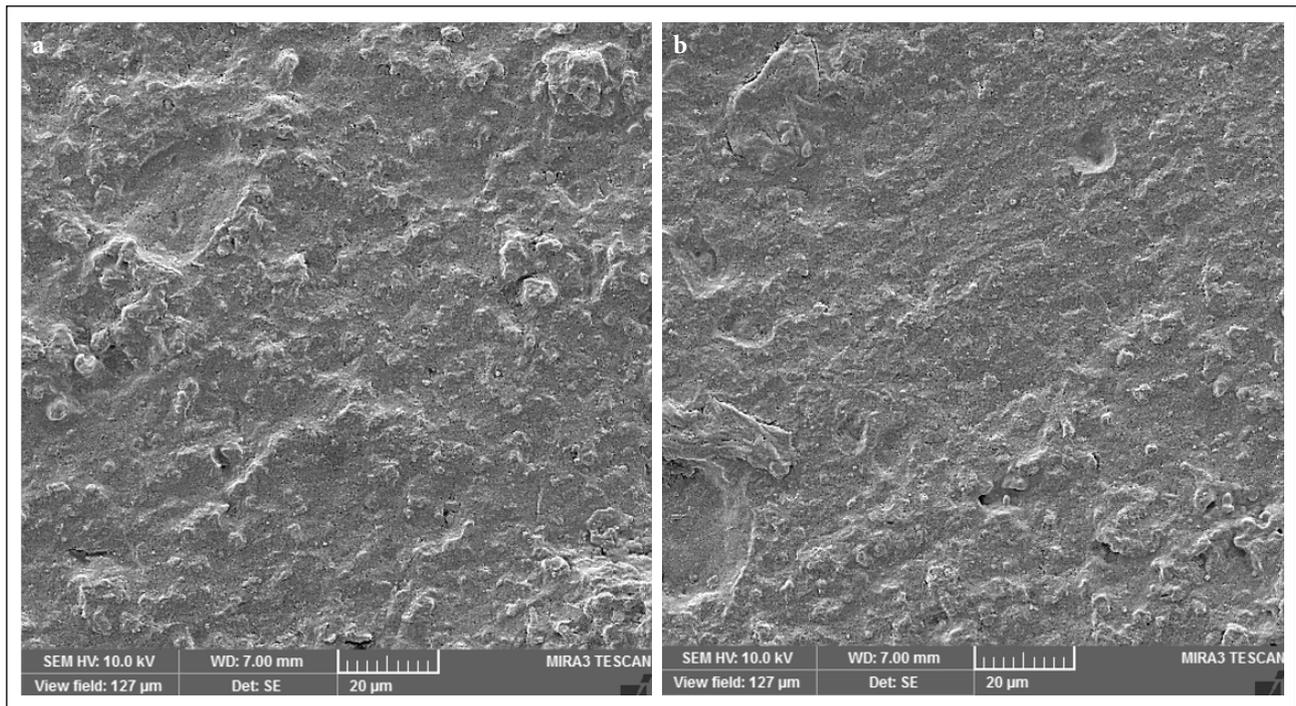


Figure 5. Fracture surfaces of the joints including silica-reinforced epoxy resins of, (a) 2 wt.% and (b) 1 wt.%.

Failure load of the joints of silica-modified epoxy resins can be hindered by the propagation of the existing micro-cracks, and consequently the initiation of new micro-cracks, thereby absorbing more energy. This results in improving the capacity of the joint to endure a higher failure load. Fi-

gure 6-a shows the micro-cracks growth hindered by silica nanoparticles. In addition, the propagation and unification of the existing micro-cracks in the absence of nanoparticles were demonstrated in Figure 6-b. It can be found out that the micro-cracks propagate and join together to create lar-

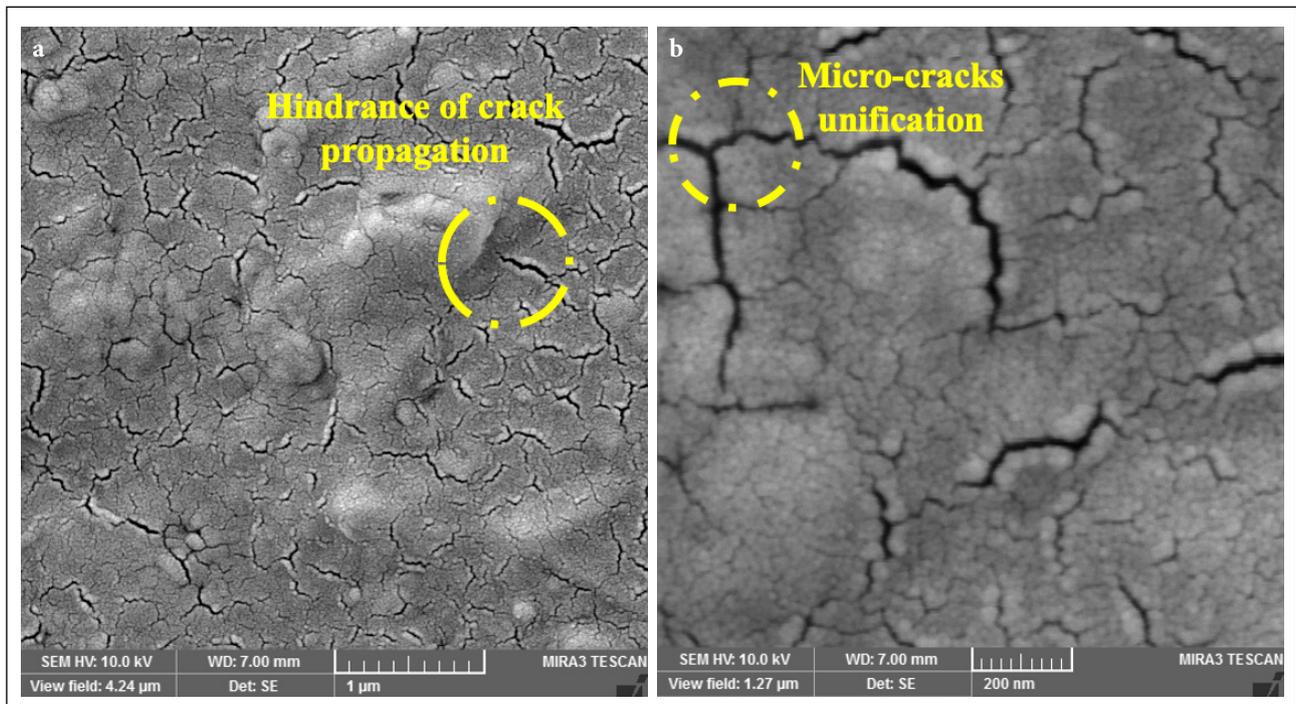


Figure 6. Illustration of, (a) crack propagation postponement in the silica-reinforced epoxy resin at 2wt.% due to the presence of the nano-silica particles, and (b) micro-cracks unification in the neat epoxy resin leading to the joint failure.

ger cracks, by which epoxy resin may fail rapidly. Taking both images into account, implies that the nanoparticles by hindering the propagation of the present micro-cracks as well as their unification, cause the material to tolerate more loading levels by absorbing more energy owing to the initiation of new micro-cracks. It is worth mentioning that, in two relevant studies [34, 35], crack growth deviation was similarly reported as an effective mechanism which lead to improvement in the bonding strength of the single-lap epoxy resin joints.

4. CONCLUSION

Silica nanoparticles were utilized to modify an epoxy-based epoxy resin in three different weight percentages. It was concluded that the concentration of 2wt.% better improved the characteristics of the epoxy polymer while using in the single-lap steel joints when being tensile tested. In addition, the postponement of the micro-cracks propagation was investigated by SEM images as an effective factor in improving the failure load of the joints including silica-improved epoxy resins. Moreover, it was observed that the use of nano-silica changed the debonding mode of the epoxy resin joints from a mixture type to a purely cohesive type, leading to the augmentations of the load endured by the joints.

AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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