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Superior dissimilar adhesive joint of mild steel and aluminium using UDM processed epoxy based TiO_2 nano-filler composite adhesive

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ABSTRACT

Characteristics of the lap joints of mechanically and chemically treated faying surface of dissimilar metals (Mild Steel and Aluminium) prepared by using UDM processed TiO_2-epoxy nano composite adhesive was investigated. Influence of the extent of TiO_2 nano (25–35 nm) particle reinforcement (5, 10 and 15 wt%) in the composite adhesive on the improvement of tensile lap shear strength of the adhesive joints has been studied. Effect of bond line thickness of the adhesive in lap joints of the differently treated faying surfaces of the dissimilar metals on lap shear strength of the joints has also been studied. The joints of 10 wt% TiO_2 nano-filler content epoxy adhesive shows maximum lap shear strength of the joints. Its lap shear strength was also studied at elevated temperatures in the range of 100–250 °C. In order to establish the potentials of nano composite adhesive to relatively improve the properties of the joint, the lap shear strength of the adhesive joint of neat epoxy adhesive was also studied and compared.

1. Introduction

Out of all major joining technologies, the adhesive joining has gathered wide attention in automobile and aerospace industries [1–3] due to its versatile and exclusive nature of application especially in the case of joining of dissimilar polymer and composite materials used in secondary load bearing structures. The main motive of the development of high strength adhesive joint of dissimilar metal substrates is to find superior applications in various components of aerospace (doors and roof), automotive (3D lock seam, hood, trunk and joining window outer or inner part), marine ships and civil construction [4,5]. The excellent thermal and insulation properties, good damping capacity, appreciable weight and noise reduction ability and better corrosion resistance are considered as primary advantages of adhesive joints [2–4]. The use of adhesive joint in automobile industry also often demands retaining its desired strength at elevated temperature [1,6]. Adhesive joining using thermoset epoxy adhesives particularly to assemble composite parts has several advantages in spite of its relative complexity in application over mechanical fastening [7]. Adhesive joining has become widely popular over other conventional methods of joining of relatively thin sections of metals primarily due to its ability to join dissimilar materials with higher joint efficiency index (a measure of relative strength to weight ratio of the bonded region), better stress distribution and lower fabrication costs [8–13].

Application of pure epoxy based adhesives in the fabrication of adhesive joints [14] of metals has certain limitations primarily due to its comparatively lower compressive and tensile strength and toughness along with inferior resistance to crack propagation due to its relatively brittle nature [15,16]. But, the epoxy-based composite adhesive has become a promising material for joining of various components largely because of its relatively higher modulus, strength and toughness especially at elevated temperature [17]. In this regard, different types of nano-filler have been found as quite promising reinforcing material to improve various properties of epoxy-based structural adhesives, such as reduction of shrinkage while curing along with significant increase of the strength and toughness [18,3]. However, the properties of nano-filler composite adhesives largely depend upon clustering and homogeneous dispersion of nano filler materials in the matrix.

The properties of nano-filler-epoxy composite adhesives prepared by ultrasonic dual mixing (UDM) process (patent file No. 1554/Del/2008) have been found superior [19–23] to those of the composite adhesives prepared by other techniques such as solution mixing, melt mixing, electro spinning, in-situ polymerization with chemical functionalization and ultrasonic mixing [24–31]. The considerable enhancement of physical and mechanical properties

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of nano-filler composite adhesive prepared by UDM processing primarily happens due to homogeneous dispersion of practically cluster free nano-fillers in morphologically modified epoxy matrix [19]. It is observed that the use of nano-filler adhesive enhances the shear tensile strength and fatigue properties of adhesive joints of aluminium at ambient temperature over those observed during using conventional epoxy adhesive [32,33]. However, the fracture behaviour of nano-filler composite adhesive at different temperature significantly depends upon the characteristics of the adhesive base as well as the type, size and amount of nano-filler content and its adhesive bond strength with the substrate. The adhesive bond strength largely depends upon bond-line thickness of the adhesive and curing cycle of the time and temperature [34,35]. A surface treatment of the substrate by mechanical, chemical or physical process improves adhesive joint strength by introducing a better wetting and bond strength between the substrate and the adhesive [34]. In certain cases, the surface roughness created by the treatment also introduces positive contribution to the bond strength by the creation of a large surface area of bonding and initiation of mechanical locking [35]. Thus, to improve the properties of adhesive joints, it is imperative to know about the flow characteristics of the nano-filler composite adhesive governing the bond line thickness and its wettability with the substrate influencing the adhesive joint strength [36,37].

In view of the above an attempt has been made to study the effect of UDM processed epoxy based composite adhesives containing different amount of TiO_2 nanoparticle on the characteristics of dissimilar adhesive joint of the mild steel and aluminium sheets. The characteristics of the dissimilar adhesive joint have been studied as a function of the mechanical and chemical surface treatment of the substrates and flow characteristics (bond line thickness) and wettability of the epoxy based nanocomposite adhesive containing different amount of TiO_2 with the substrate. The adhesive joints are characterized by their tensile lap shear strength at ambient and elevated temperatures. The behaviour of the dissimilar adhesive joints has been critically studied to optimise the bond line thickness of the nanocomposite adhesive containing different amount of reinforced particles to give maximum joint strength.

2. Experimental

2.1. Materials

A base of commercially available two component epoxy adhesive (EPOFINE-556), consists of epoxy resin diglycidylether of bisphenol-A and aromatic based diamine hardener (FINEHARD-5200), was used to prepare TiO_2 particulate nanocomposites adhesive. The commercially available TiO_2 nanoparticle of size in the range of 25–35 nm having purity of 99.9% and density of 3.9 g/cm^3 was procured from a certified source of M/s Nanoshel LLC, USA. The commercially pure aluminium and mild steel sheets of thickness 1.6 mm were used to prepare adhesive lap joints of dimensions confirming the ASTM D1002 standard.

2.2. Preparation of TiO_2 reinforced epoxy adhesive

Initially, TiO_2 nanoparticles were introduced into the epoxy resin at varying amount of 5, 10 and 15 wt% by mechanical spatula mixing. The epoxy resin-TiO_2 mixture was diluted to reduce its viscosity using MEK by addition in 2 parts by volume. The slurry (resin + nanoparticle + MEK) for each composition was then processed by ultrasonic dual mixing (simultaneous application of ultrasonic vibration and impeller stirring) using a Vibra Cell ultrasonic processor having capacity of maximum output power of 750 W with a constant frequency (20 kHz) of vibration introduced by a 13 mm diameter titanium alloy (Ti–6Al–4V) tip. Ultrasonic vibration at amplitude of 60% (or 450 W) was applied for 60 min at an interval of 5 s on and 15 s off. Prior to the addition of hardener the MEK was removed from the slurry by placing it into an oven at 70 °C for 2 h. The removal of MEK from the slurry was confirmed by comparing its weight to an accuracy of 0.1 mg with that of the epoxy resin-TiO_2 mixture prior to the addition of MEK.

The wettability of the epoxy based conventional adhesive and the 10 wt% TiO_2 nano-filler adhesive was studied by measuring their contact angle on the mechanically and chemically treated mild steel and Al substrates by sessile drop technique as typically shown in Fig. 1. The contact angle was measured by a built-in facility of a camera system operated through software (Drop Shape Analyzer – DSA25E).

2.3. Preparation of faying surface of substrates

Mechanical abrasion was employed in order to remove dirt, oxide layers or any other contaminants from the faying surfaces of the mild steel and aluminium substrates. In the light of the earlier observations, the emery papers of grade 220 [38,39] and 400 [40] were consecutively used for effective surface abrasion of mild steel and aluminium respectively. Abrasion is employed also to enhance the available surface area along with possible mechanical interlocking [41] and to increase surface energy [42] for bonding which in turn enhances the bond strength. It reduces void formation and increases surface tension [43] to improve wetting which is an important requirement for increased bond strength.

The faying surfaces of the mild steel and aluminium substrates were also chemically treated using the solutions given in Table 1. Prior to chemical treatment both the mild steel and aluminium surfaces were mechanically polished by emery paper as stated above and cleaned by acetone. The mechanically polished mild steel and aluminium substrates were immersed in the respective chemical solution (Table 1) for 2 min followed by consecutive washing with tap water and rinsing in DI water for 2–3 min. The chemically treated surfaces were given a further chemical treatment to neutralize the presence of any acidic ions on them by rinsing in a solution (100 ml DI + 2 gm NaOH pellets) for 30 s. Then the surfaces were washed under tap water and cleaned by flowing acetone. The substrates were air dried at 60–65 °C.

Characteristics of the mechanically and chemically treated surfaces of mild steel and aluminium have been studied under a profilometer (Mitutoyo SJ 400), field emission scanning electron microscope (FESEM) and X-ray diffraction (XRD). The FESEM studies were carried out at an acceleration voltage of 15 kV while the XRD analysis was performed with CuK_α radiation at $\lambda = 1.5418$ Å on an area of 20 × 20 mm^2. With the help of the profilometer the surface roughness of the mechanically and

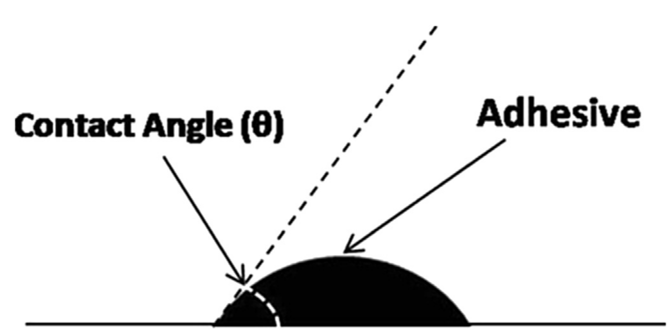

Fig. 1. Systematic diagram of contact angle measurement.

Table 1
Chemistry of the solution used for chemical treatment of Mild steel and Aluminium substrate.

Mild steel substrate	Chemistry	H_2SO_4	Oxalic acid	Distilled water
	Amount (wt%)	10	10	80
Aluminium substrate	Chemistry	H_2SO_4	$Na_2Cr_2O_7$	Distilled water
	Amount (wt%)	27.5	7.5	65

chemically treated mild steel and aluminium substrates, defined by the average roughness (Ra) and mean roughness depth (Rz), was measured.

2.4. Preparation of adhesive joints

Single lap dissimilar joints of the steel and aluminium sheet substrates were prepared using neat adhesive and adhesive containing nano filler TiO_2 at varying amount of 5, 10 and 15 wt%. The adhesive joints were prepared by applying adhesive on differently treated virgin faying surface of the substrates followed by putting both the steel and aluminium sheets together in the proper position of lap joint confirming the dimensions as shown in Fig. 2. A uniform layer of adhesive at bond-line of the joint was obtained in between the substrates by applying a rolling pressure at a speed of 5 mm/min under different load varied to 2.2, 4.2 and 6.2 N. The green adhesive joints were put inside an air oven at 120 °C for 2 h followed by 160 °C for 6 h for curing the adhesive. After curing, fillets of adhesive from all edges of the specimens were carefully removed with a knife and emery paper followed by polishing to measure the bond-line thickness of the adhesive. The bond-line thickness of the joints was measured under an optical microscope.

2.5. Lap shear testing

Mechanical properties of the single lap dissimilar adhesive joints of mild steel and aluminium sheets were studied by tensile lap shear test. The test was carried out at ambient and elevated temperatures on an electro-hydraulic universal testing machine (Hounsfield H25K-S) operated at a cross-head speed of 1 mm/min. The elevated temperature lap shear test was carried out on adhesive joint of neat epoxy and 10 wt% TiO_2 containing composite adhesive prepared at optimized 4.2 N rolling load. The test at elevated temperature was performed inside a split furnace at four different temperatures of 100, 150, 200 and 250 °C. The lap shear strength (σ_s) of the adhesive joint was estimated by using the expression $\sigma_s = F/W \times L$, where F is the failure load in Newton (N), W is the width of adhesive joint in mm and L is the length of adhesive joint in mm [40,44]. During tensile lap shear test, the stress–strain plot was recorded till fracture of the adhesive joint.

3. Results and discussion

3.1. Characteristics of treated substrates

The appearance of surface morphology of the mechanically and chemically treated mild steel and aluminium substrates studied at different magnifications of FESEM has been typically shown in Figs. (3 and 4) and 5 respectively. The images at relatively low and high magnifications clearly reveal respectively the overall characteristics and specific features of the matrix in detail. The mechanically treated mild steel surface shows (Fig. 3 (a1) and (a2)) the presence of peaks and valleys of long scratches along with the presence of relatively rougher patches of oxides having micro-cracks in them (Fig. 4). However, the chemically treated surface of mild steel shows (Fig. 3 (b1)) the presence of all over rough reacted matrix along with the pit formation all through it as more clearly visible in Fig. 3 (b2). Similarly Fig. 5 (a1) and (a2) shows the presence of directionally oriented scratches of peaks and valleys on the matrix of mechanically treated aluminium substrate. Whereas, Fig. 5 (b1) shows no such directional scratches on the surface rather the presence of all through relatively rough chemically reacted product in the matrix of aluminium. It is also noted (Fig. 5 (b2)) that the rough chemically reacted surface is having a significant amount of elongated cavities in the matrix.

The surface roughness of the mechanically treated mild steel and aluminium substrate was measured as (Ra = 0.24 μm and Rz = 2.1 μm) and (Ra = 0.57 μm and Rz = 5.1 μm) respectively. The roughness value for chemically treated mild steel and aluminium substrate was increased and measured as (Ra = 0.81 μm and Rz = 5.5 μm) and (Ra = 1.17 μm and Rz = 9.3 μm) respectively. This is in agreement to the observed relatively rougher morphology of the chemically treated surface than the mechanically treated one with comparatively higher severity in case of aluminium than mild steel as discussed with Figs. 3 and 5.

The XRD analyses of the mechanically and chemically treated surfaces have been shown in Fig. 6 (a) and (b) respectively. The figure shows the presence of an insignificant amount of oxide and hydroxide on both the mechanically and chemically treated mild steel (Fe_3O_4, $Fe(OH)_3$) and aluminium ($Al(OH)_3$) substrates. However, it is noticed that the presence of oxide and hydroxide is relatively more significant in the case of chemical treatment of both the substrates.

3.2. Wettability of adhesives on different substrates

The average contact angle (θ) of the neat and 10 wt% TiO_2 nano-filler epoxy adhesives with the mechanically and chemically treated aluminium and mild steel substrate are typically shown in Fig. 7. Contact angle of the neat epoxy adhesive with the mechanically and chemically treated mild steel has been found as 64° and 60.2° respectively whereas, the same of the mechanically and chemically treated aluminium has been found as 65.2° and 62.4° respectively. It shows that the chemical treatment provides relatively better wettability of epoxy adhesive to the mild steel and aluminium substrate than that found in case of mechanical treatment of the substrates. It is interestingly observed that the chemically treated mild steel and aluminium substrate gives further significantly lower contact angle of 60.9° and 60.7° with the 10 wt% TiO_2 nano-filler epoxy based adhesive. Thus, it infers that the UDM processed 10 wt% TiO_2 nano-filler epoxy based adhesive is having higher wettability than the neat epoxy adhesive on the chemically treated aluminium and mild steel substrate. The improvement in wettability enhances thorough wetting of the surface to increase the adhesion area even by penetrating the pores created on the chemically treated aluminium and mild steel substrates which provides higher bond strength and consequently improved adhesive joint strength.

However, the bond strength of adhesive with the substrate not only depends upon the area of contact but also becomes appreciably encouraged by the characteristics of the faying surface that may promote a chemical interaction with the adhesive resin and contribute to enhancement of the adhesion strength [45–47]. In this regard, the oxide of aluminium present on the aluminium substrate plays a more significant role over the oxide of iron to chemically react with the epoxy resin [47].

3.3. Bond-line thickness analysis

The variation in wettability of the neat and nano-filler adhesive on the differently treated aluminium and mild steel substrate

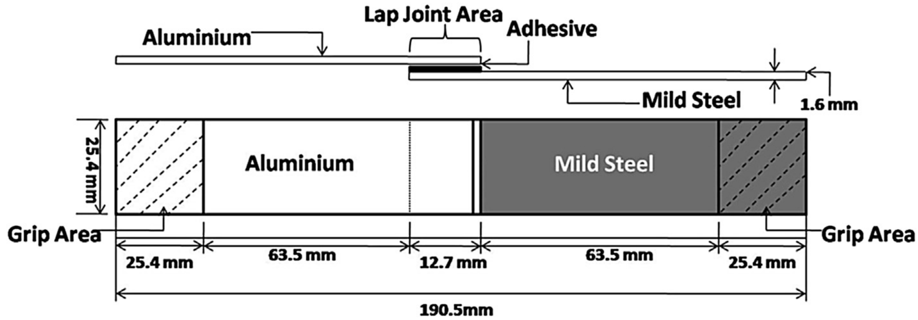

Fig. 2. Schematic diagram of single lap dissimilar adhesive joint of mild steel and aluminium sheet.

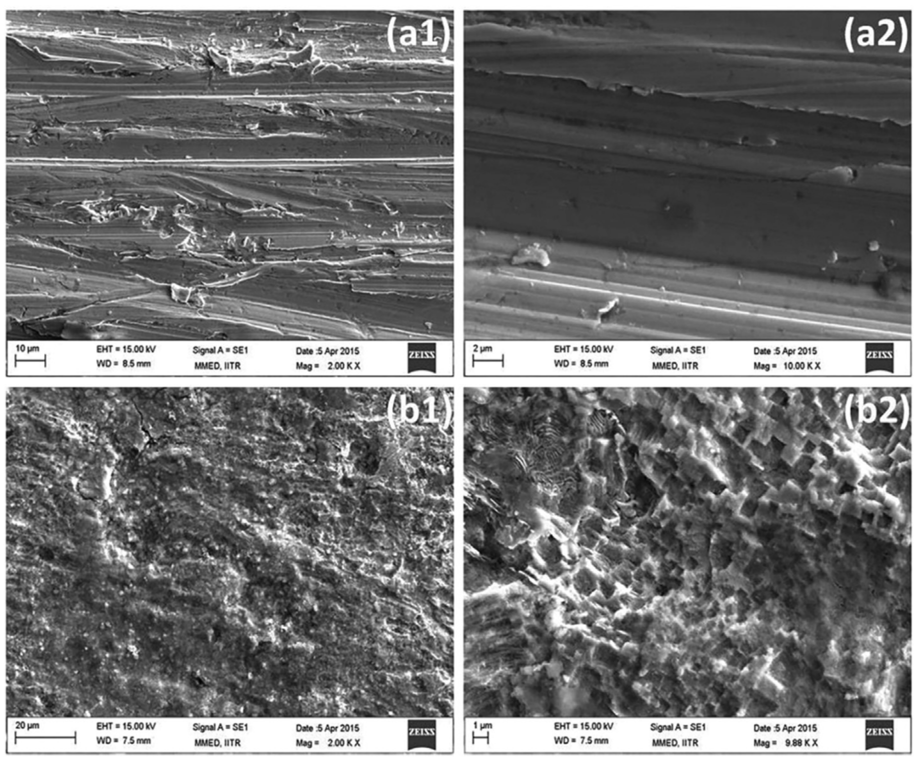

Fig. 3. FESEM images of differently treated faying surfaces of mild steel at different magnifications; (a1) and (a2) mechanically treated and (b1) and (b2) chemically treated.

significantly influences the bond line thickness of adhesive at a given load of rolling during preparation of lap joint. However, in this regard the viscosity of the adhesive affecting its flow characteristics also plays a significant role. Bond-line thickness of the adhesive in the lap joints prepared by using the neat epoxy and TiO_2 nano filler composite adhesives was studied under optical microscopy. The typical optical microscopy images of variation in bond line thickness of epoxy based nano composite adhesive containing 5, 10 and 15 wt% TiO_2 nanoparticles as a function of the rolling load has been shown in Fig. 8. The variation in thickness of adhesive in bond-line primarily depends upon flow characteristics of adhesive as a function of viscosity of the adhesive, characteristics of faying surface and load applied on the lap joint. The nature of variation of the bond-line thickness of neat epoxy and different amount of TiO_2 content epoxy nano composite as a function of rolling load applied in the case of preparation of lap joint of mechanically as well as chemically treated mild steel and aluminium substrates has been shown in Figs. 9 and 10 respectively.

It is observed that the bond-line thickness always significantly decreases with the increase of rolling load from 4.2 to 6.2 N in case of both the neat as well as composite adhesives. Further at a given rolling load the bond-line thickness of composite adhesive increases with the increase of its nanoparticle content from 5 to 15 wt %. This may have primarily happened due to enhancement of viscosity of the neat epoxy with the increase of its nano particle content. But, the effect of particle content on bond-line thickness of the nano composite adhesive gradually becomes insignificant at a higher rolling load of 6.2 N (Figs. 9 and 10). This may have primarily happened due to predominant influence of high force over the other factors as stated above influencing the flow characteristics of the adhesive. The enhancement of the bond line thickness of the composite adhesive having nano particle content is primarily attributed to rheological behaviour and viscoelastic characteristics of the adhesive that provide resistance to its flow [48,49]. The increase in bond line thickness occurs due to higher resistance to flow of the composite adhesive imposed by the presence of amount and distribution of nano particle in the epoxy base.

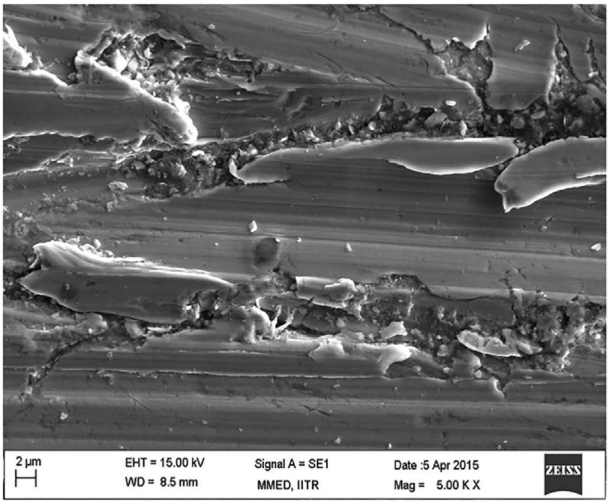

Fig. 4. FESEM image of mechanically treated mild steel substrate.

3.4. *Lap shear strength analysis*

Effect of rolling load on the lap shear strength of the adhesive joint of the neat epoxy adhesive and composite adhesive containing different amount of TiO_2 nano-particles applied on mechanically treated surface (MTS) of steel and aluminium has been shown in Fig. 11. Similarly, the effect of rolling load on the lap shear strength of the adhesive joint of the same adhesives applied on the chemically treated surface (CTS) of the steel and aluminium has been shown in Fig. 12. It is found that the use of the composite adhesive instead of the neat epoxy adhesive is always beneficial to improve the strength of the adhesive joint. Both Figs. 11 and 12 reveal that irrespective of any treatment (MTS or CTS) of faying surface and the use of neat or composite adhesives containing different amount of nanoparticles the lap shear strength of the dissimilar adhesive joint becomes maximum at the rolling load of 4.2 N that gives optimum level of bond line thickness (Figs. 9 and 10). A relatively lower strength of the adhesive joint at a comparatively thicker bond line thickness of the adhesive at a lower rolling load primarily happens because it is more sensitive to cohesive fracture of adhesive especially under the higher possible presence of matrix defects [50–52] like pores. It is reported that in the case of thinner bond line thickness of the epoxy adhesive at higher rolling load, the presence of cavitation ahead of the crack tip may precede plastic flow. Thus, the fracture is predominated by high triaxial stresses and the stress concentration near the crack tip, causing the increase in extent of the plastic zone at it, resulting in a lower strength of the adhesive joint for a thinner bond line [50,52].

It is also noted (Figs. 11 and 12) that in both the cases of the surface treatment (MTS and CTS) the lap shear strength of adhesive joint with composite adhesive considerably increases with the increase of its TiO_2 particle content from 5 to 10 wt% followed by a significant decrease in it with further increase of particle content to 15 wt%. It is marked that at any conditions of rolling load or the characteristics of adhesive the adhesive joint of the chemically treated surface of the mild steel and aluminium shows considerably higher lap shear strength than that observed in case of the mechanically treated substrates. This has primarily happened due creation of appreciably larger surface area of contact between the adherents and adhesive by roughening of the faying surface as well as the formation of pits and elongated cavities (Figs. 3(b1) and (b2) and 5 (b1) and (b2)). Such features of the faying surface primarily enhance the bond strength due to increase of bonding area and mechanical interlocking. In this regard it may also be noted that the chemical treatment, in agreement to an earlier work [45], also enhances the wettability of metallic substrate which contributes to better spreading of adhesive at every location of the rough faying surface as discussed above with enhanced adhesive bond strength. In the line of the studies on wettability, it is clearly observed that the adhesive joints of 10 wt% TiO_2-epoxy adhesive on chemically treated surface prepared at 4.2 N rolling load shows maximum enhancement of lap shear strength compared to that observed in case of the adhesive joint of mechanically treated substrate. The

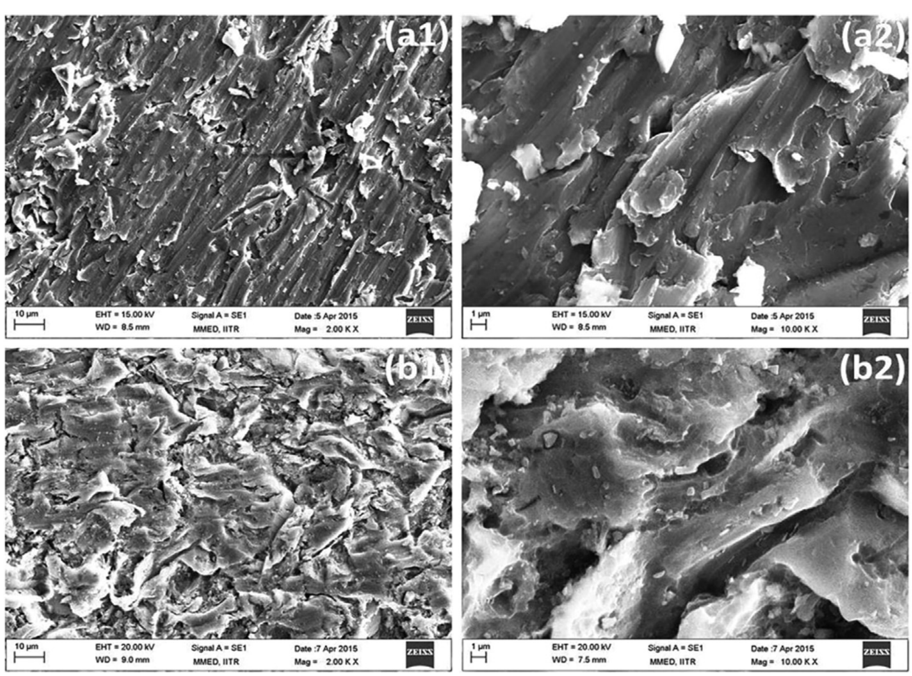

Fig. 5. FESEM images of differently treated faying surfaces of aluminium at different magnifications; (a1) and (a2) mechanically treated and (b1) and (b2) chemically treated.

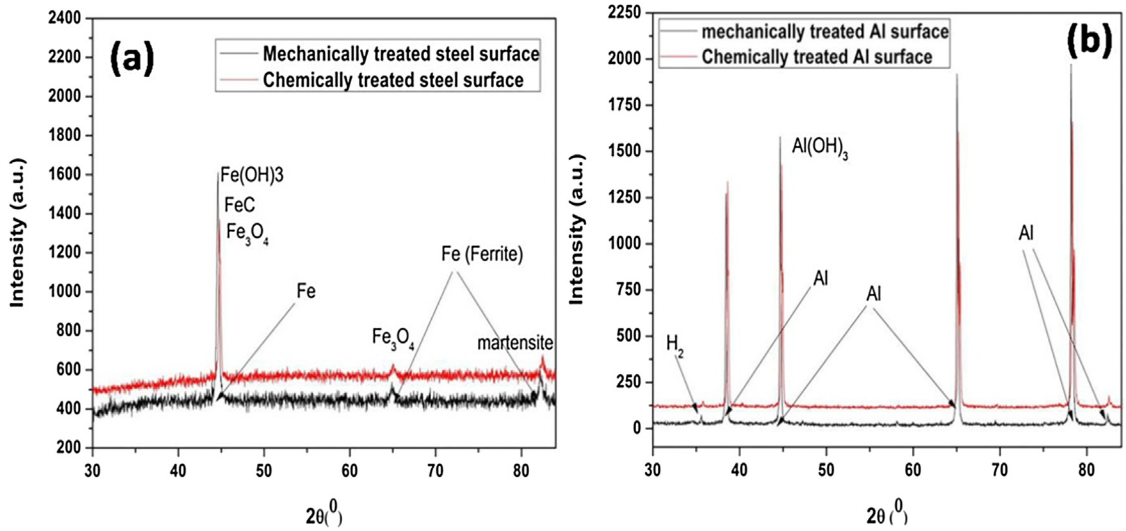

Fig. 6. XRD pattern of mechanical and chemically treated (a) mild steel and (b) Aluminium surface.

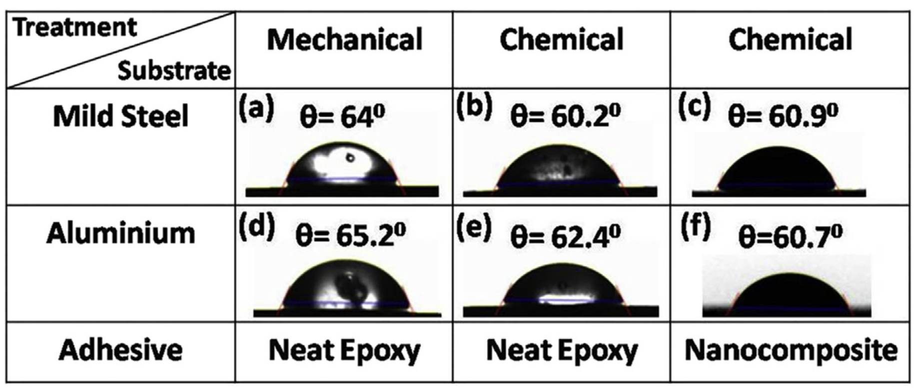

Fig. 7. Typical images of contact angle measurement of neat epoxy and 10 wt%TiO_2 nano-filler composite adhesive on mechanically and chemically treated substrates.

dissimilar adhesive joint of mechanically and chemically treated surface using 10 wt% TiO_2-epoxy nano particulate adhesive enhances the lap shear strength up to about 59% and 110% respectively as compared to that observed in the respective adhesive joints of neat epoxy adhesive.

The stress–strain curves of tensile lap shear test of the dissimilar adhesive joints prepared at optimized load of 4.2 N applied on neat and nanocomposite adhesives have been shown in Figs. 13 and 14 where the substrates are treated mechanically and chemically respectively. The figures also show the effect of TiO_2 nano particle content of the epoxy adhesive on the stress–strain curves of the adhesive joints. It is observed that the use of composite adhesive significantly increases the ductility (strain %) of adhesive joint under the lap shear test over that found in case of using the neat adhesive. It is clearly noticed that the presence of 10 wt% TiO_2 nanoparticle in adhesive introduces maximum ductility to the adhesive joint and it is considerably more effective (about 425%) in case of the chemically treated substrate than that of the mechanically treated one. This is in the line of understanding [50,52] as stated above regarding the formation of cavitation and plastic flow in reference to the crack tip influencing the strength of the adhesive joint.

In case of the lap shear test of adhesive joint under tensile loading the fracture may take place in the mixed mode of interfacial debonding from the substrate and cohesive failure of adhesive where, the occurrence of the latter one provides relatively higher strength to the joint. This is because the cohesive fracture happens when a sufficiently strong interface, prior to its debonding, transmits enough force to the adhesive to attain its fracture stress [53,54]. In both the cases of surface treatment (mechanical and chemical) and type of adhesive (neat or nanocomposite) the fracture of lap joint has always been found to occur predominantly by interface failure from the mild steel. The lap shear fracture surface of both the mechanically and chemically treated aluminium and mild steel substrate with neat as well as nanocomposite adhesive containing 10 wt% TiO_2 particle has been shown in Fig.15 and Fig.16. In case of the adhesive joint of TiO_2 nano-particle containing adhesive the mixed mode of fracture of the joint appears to predominantly occur by the interface failure from the mild steel followed by cohesive fracture of the adhesive, while the interface failure from the aluminium is practically insignificant.

The epoxy resin, which is one of the most common structural adhesives, particularly for the use in automotive and aircraft industries, has a good affinity for the aluminium alloy surfaces and the oxide layers produced during surface preparation. The interfacial bond strength between the epoxy adhesive and the metal sheet vary with the type and characteristics of the oxide present on the metal substrate [46,47]. The most common components in these epoxies are phenol compounds. Phenol compounds form ionic bonds with their surfaces providing a very strong metal epoxy

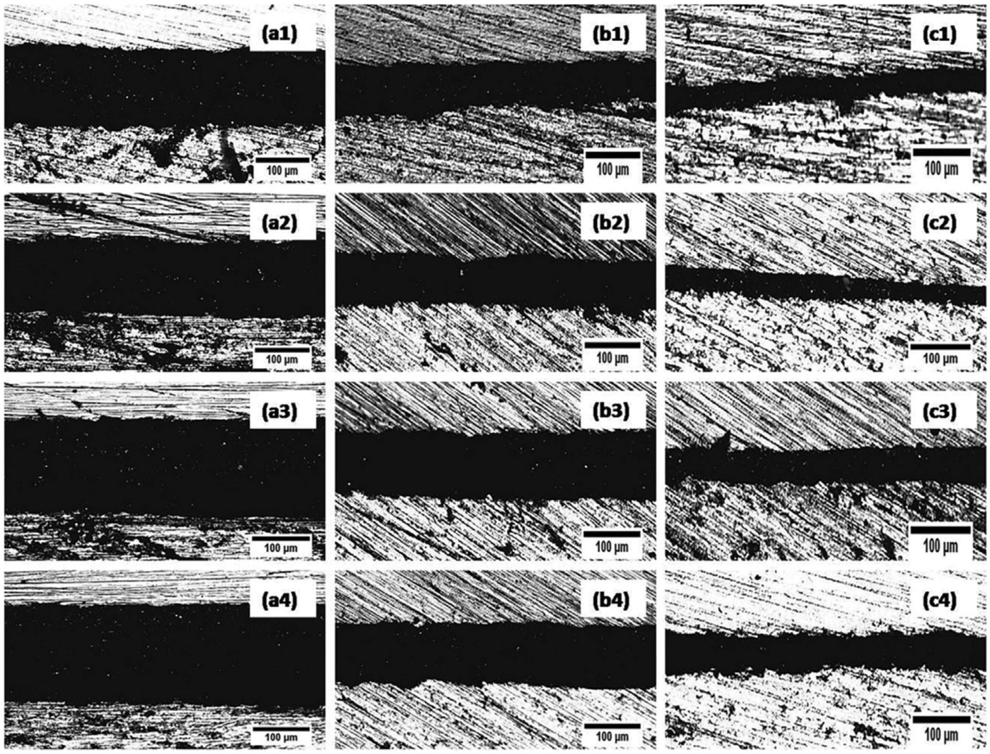

Fig. 8. Bond-line thickness of nano composite adhesive as a function of rolling load and TiO_2 nano filler content of epoxy used in dissimilar lap joints of mechanically treated substrate.

bonding [55]. Strong adhesion of metal oxide layer with the neat and composite adhesive is responsible to enhance the lap shear strength of the mechanical as well as chemically treated dissimilar metal adhesive joint. The adhesive joint of the chemically treated metallic surface shows the maximum enhancement in lap shear strength due to the better interfacial interaction between the metal oxide layers and adhesive. The formation of porous structure in chemical etching of metal substrate has also contributed to enhance the lap shear strength. From Figs. 15 and 16, one can see that the neat and composite adhesives have raised relatively stronger interfacial bonding with the mechanically as well as chemically treated aluminium substrate as compared to that happened with the similarly treated steel substrate. This may have

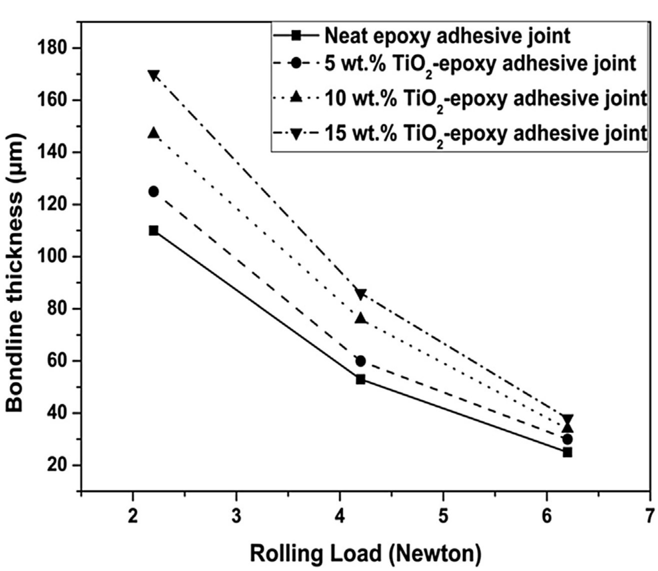

Fig. 9. Bond-line thickness of adhesive joints with neat epoxy and different amount of TiO_2 nano filler content epoxy composite adhesives on mechanically treated dissimilar metal substrate.

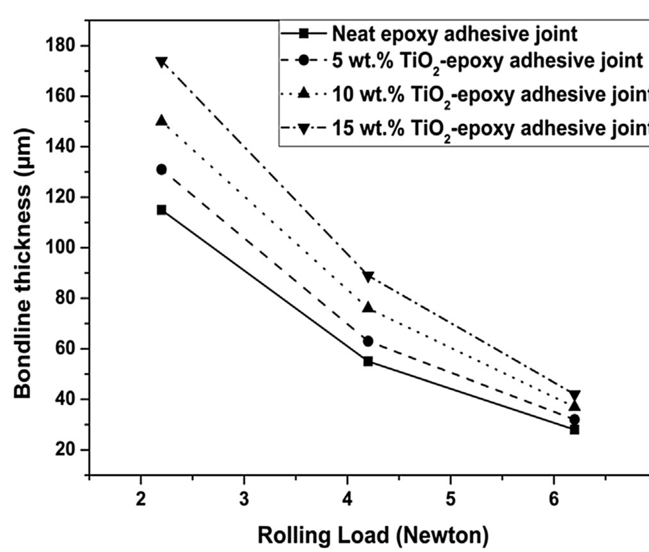

Fig. 10. Bond-line thickness of adhesive joints with neat epoxy and different amount of TiO_2 nano filler content epoxy composite adhesives on chemically treated dissimilar metal substrate.

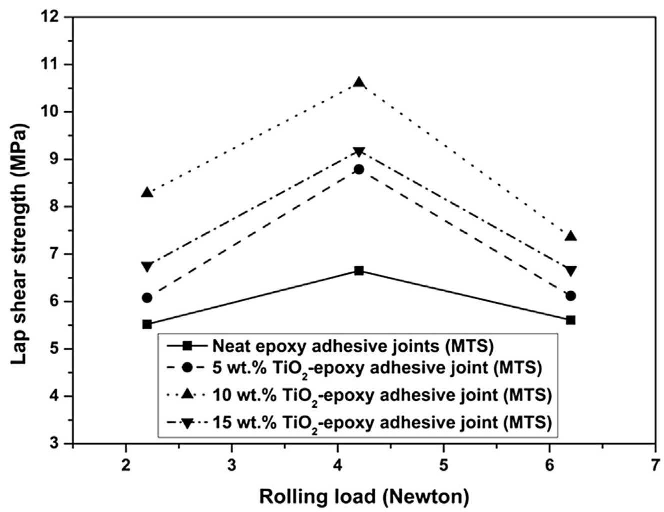

Fig. 11. Effect of rolling load on lap shear strength of dissimilar adhesive joints of neat and varied nano filler content epoxy adhesives applied on mechanically treated substrate.

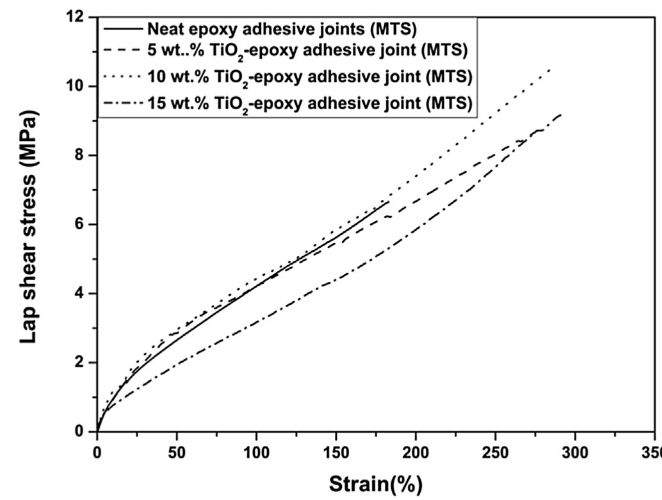

Fig. 13. Lap shear stress–strain curves of dissimilar adhesive joints prepared on mechanically treated substrate at rolling load of 4.2 N using neat and different amount of nano TiO_2 content epoxy adhesives.

largely attributed to the inherent nature of iron oxides to develop the faults and micro cracks within it due to appreciable volume change. Whereas the oxides of aluminium carries a strong nature of adherence with the substrate without developing any fault in it. Besides their nature of chemical reaction with the epoxy contributing to bonding, the mechanical factors as discussed above may have significantly affected the adhesive joint strength of the aluminium and mild steel.

3.5. Elevated temperature lap shear strength analysis

In view of the considerable influence of using the optimum 10 wt% TiO_2 nano particulate epoxy adhesive on enhancement of lap shear strength of the dissimilar adhesive joints of mild steel and aluminium, its effect on strength of the adhesive joint at elevated temperature has been studied. Similarly, the lap shear strength at elevated temperature of the adhesive joint using net epoxy has also been studied in order to realise the fraction of improvement in joint strength due to the use of optimum nano composite adhesive. Effect of temperature on the lap shear strength of the dissimilar adhesive joint of net epoxy and the 10 wt% TiO_2 filled nano particulate epoxy adhesive applied on the mechanically and chemically treated substrates with 4.2 N rolling load has been shown in Fig. 17. The figure shows that irrespective of the nature of surface treatment the elevated temperature strength of the adhesive joint of the neat and nano composite adhesives is appreciably retained up to 150 °C followed by a significant reduction in it with a further increase of temperature. However, at 150 °C the strength of the adhesive joint has been found considerably higher in case of using the nano composite adhesive than the neat epoxy adhesive. In this regard it is also interestingly noted that the use of CTS is comparatively more favourable than using MTS. The relatively higher strength of the adhesive joint observed in case of using the composite adhesive over the neat epoxy adhesive on the CTS rather on the MTS may be again understood as they provide better wettability and

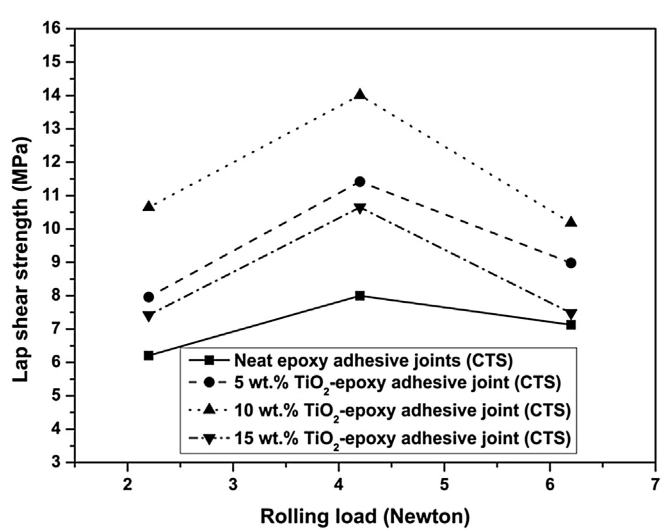

Fig. 12. Effect of rolling load on lap shear strength of dissimilar adhesive joints of neat and varied nano filler content epoxy adhesives applied on chemically treated substrate.

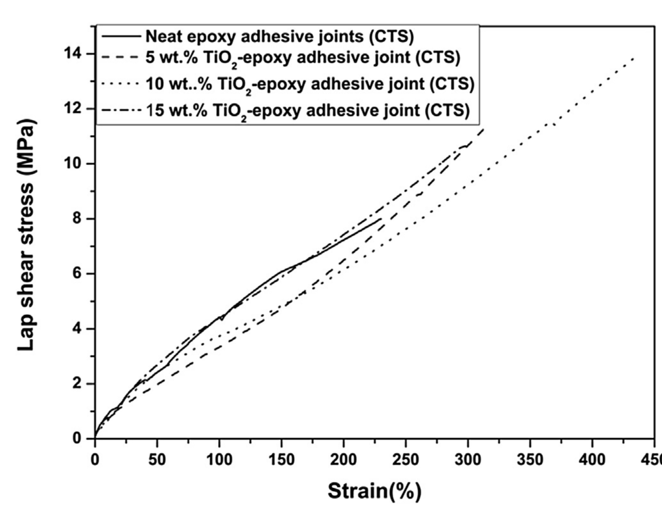

Fig. 14. Lap shear stress–strain curves of dissimilar adhesive joints prepared on chemically treated substrate at rolling load of 4.2 N using neat and different amount of nano TiO_2 content epoxy adhesives.

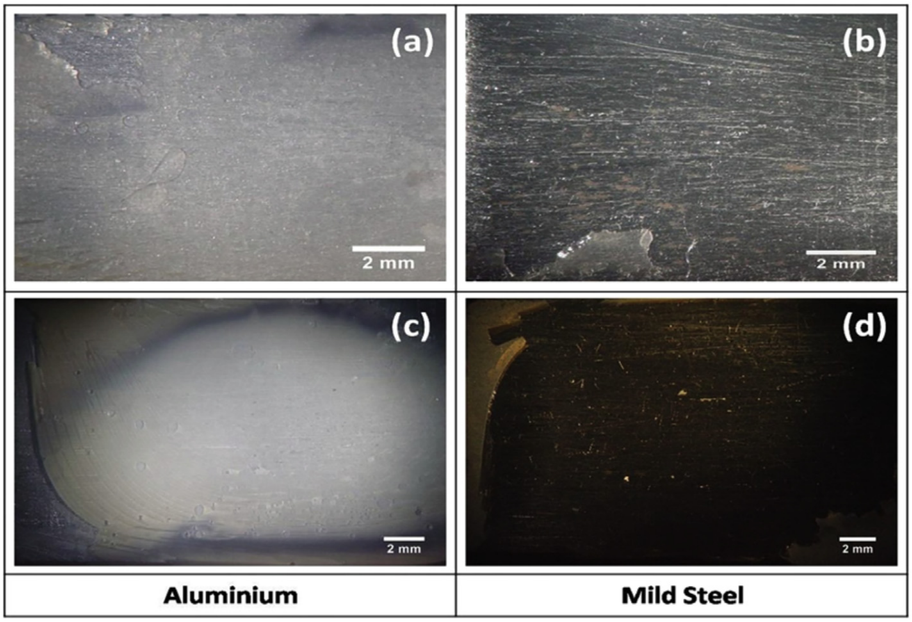

Fig. 15. Typical lap shear fracture surface of both the counterpart of a mechanically treated aluminium and mild steel joint with neat epoxy (a and b) and nanocomposite adhesive containing 10 wt% TiO_2 particle (c and d).

consequently improved bond strength with the substrate as discussed above. At a given temperature of 150 °C the adhesive joints of neat epoxy and 10 wt% TiO_2 nano filler composite adhesive applied on differently treated substrates show the lap shear strength of similar order to their ambient temperature strength (Fig. 12).

At 150 °C the lap shear stress–strain curves of the dissimilar adhesive joints of neat epoxy and 10 wt% TiO_2-epoxy adhesive applied on differently treated substrates have been shown in Fig. 18. However, the figure interestingly shows that at the given

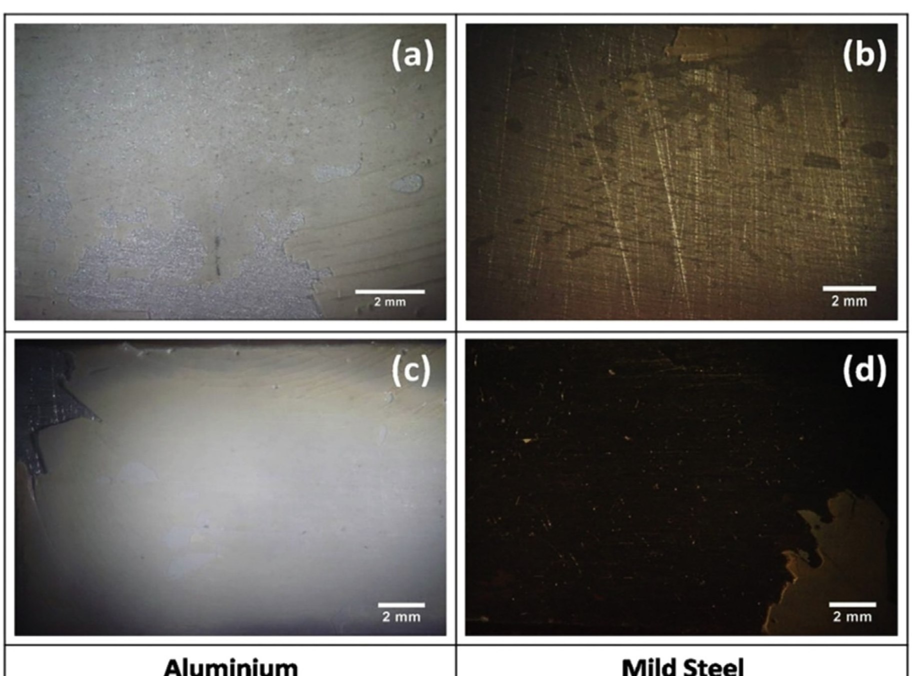

Fig. 16. Typical lap shear fracture surface of both the counterpart of a chemically treated aluminium and mild steel joint with neat epoxy (a and b) and nanocomposite adhesive containing 10 wt% TiO_2 particle (c and d).

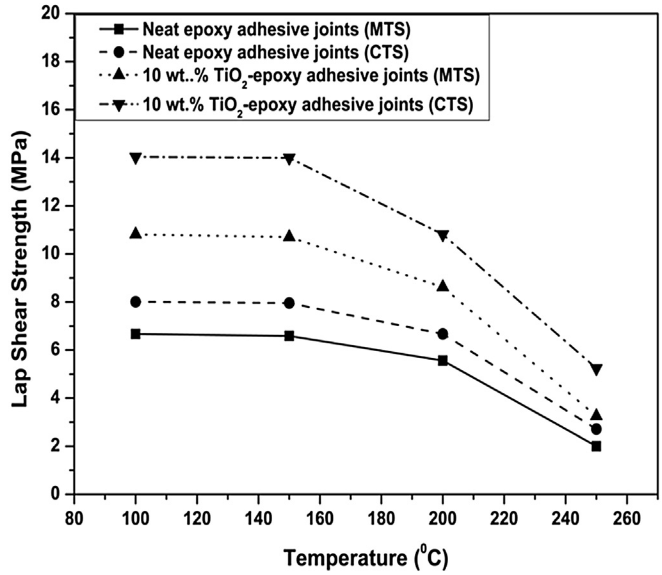

Fig. 17. Elevated temperature lap shear strength of dissimilar adhesive joints of differently treated substrates using neat epoxy and 10 wt% TiO_2-epoxy adhesive.

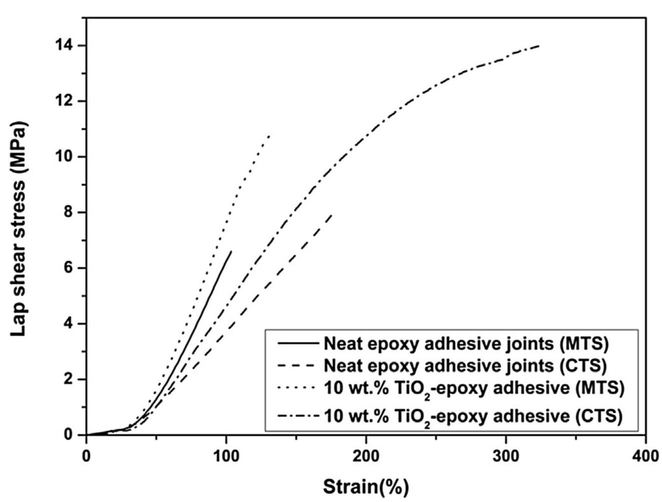

Fig. 18. At 150 °C lap shear stress–strain curves of dissimilar adhesive joints of differently treated substrates using neat epoxy and 10 wt% TiO_2-epoxy adhesive.

temperature of 150 °C the ductility of both the neat and composite adhesives goes down considerably with respect to that observed at ambient temperature (Fig. 14).

The lap shear fracture surface of the high strength (at 150 °C) joint of the chemically treated aluminium and mild steel substrates prepared by using the nanocomposite adhesive containing 10 wt% TiO_2 particle has been shown in Fig. 19. The figure shows that the mixed mode fracture of the joint has occurred by a symmetrical response of interface failure from the mild steel and aluminium substrate along with cohesive fracture of the adhesive. This may have possibly happened due to weakening of the interfacial bond strength and cohesive strength of the adhesive at elevated temperature. In order to understand the mechanism of such fracture with the priority of its initiation and propagation to final fracture a systematic study has to be carried out further. In view of the superiority of nano-particulate composite adhesive containing 10 wt% TiO_2 in producing adhesive joint of significantly improved mechanical properties, its performance under dynamic loading along with its life in different exposures should be studied in reference to different service conditions based on applicability of such joint. It was beyond the scope of the current studies on development of advanced nano composite adhesive and its application on differently treated substrate.

4. Conclusion

Lap shear strength of dissimilar adhesive joint of mild steel and aluminium significantly depends upon the bond line thickness of the adhesive. The optimum bond line thickness which is relatively higher in case of nano composite adhesive than that of the neat adhesive is obtained at the rolling load of 4.2 N. Use of chemical treatment on aluminium and mild steel faying surfaces significantly improves the lap shear strength of adhesive joint over that happens in case of the mechanical treatment of the substrates. In this regard, the use of TiO_2 nano filler epoxy based composite adhesive is highly beneficial over the use of neat epoxy adhesive, where the optimum level of its presence has been found as 10 wt%. The 10 wt% TiO_2 nano filler composite adhesive also retains its lap shear strength at an elevated temperature up to 150 °C. Such an optimum nano filler epoxy based composite applied on the chemically treated substrate not only gives maximum lap shear strength but also significantly enhances its ductility. Whereas irrespective of any condition the ductility of the lap adhesive joints significantly reduces at elevated temperature.

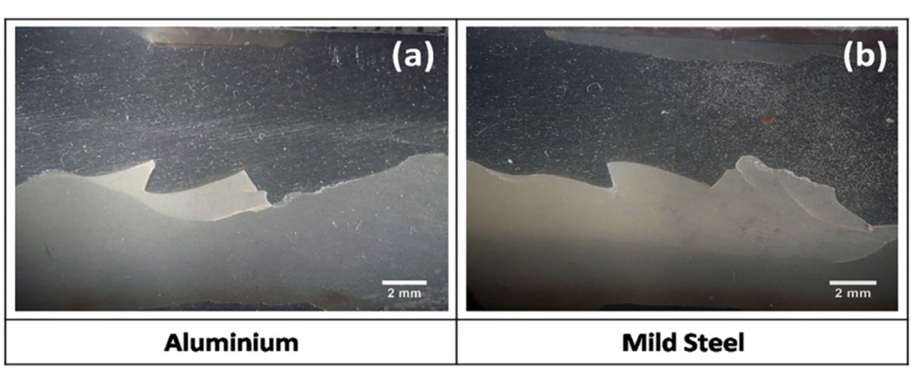

Fig. 19. Typical lap shear fracture surface of both the counterpart of a chemically treated aluminium and mild steel joint with nanocomposite adhesive containing 10 wt% TiO_2 particle (a and b) at 150 °C.

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