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Characteristics Of Adhesive Joints Of Metals Using Inorganic Particulate Composite Adhesives

P.K. Ghosh and Sharada Kameswari Nukala
Department of Metallurgical & Materials Engineering,
Indian Institute of Technology Roorkee, Roorkee-247 667, India
E-mail: prakgfmt@iitr.ernet.in
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ABSTRACT

The characteristics of adhesive joints of similar sheets of aluminium and steel containing epoxy based particulate composite adhesives have been studied. The composite adhesives having particulate fillers as aluminium and alumina powders of varying amount (5-15wt.%) and size (75-84 to 0.8-1.2 mm) are applied to mechanically and chemically treated aluminium and mechanically treated steel substrates. The joints of the composite adhesive are characterized by their tensile lap shear strength as a function of the bond-line thickness as well as the amount, size and type of particle. For a given substrate the characteristics of the composite adhesive joint have been compared to those of the conventional adhesive joints. The bond-line thickness of the adhesive with respect to the amount and size of the aluminium and alumina particles has been optimised for maximum tensile shear strength of the adhesive joint of differently treated substrates.

1. INTRODUCTION

Adhesive joining has become widely popular over other conventional methods of joining of comparatively 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[1-6]. The excellent thermal and insulation properties, superior damping and noise reduction ability and an improvement in corrosion resistance are also appreciable advantages of adhesive joints[2,3]. Out of the commonly used structural adhesives, the epoxy-based adhesives are widely employed for joining of various components largely because of their relatively high modulus and strength. But, the application of pure epoxy based adhesives in fabrication of adhesive joints of metals has certain limitations primarily due to its comparatively lower compressive and tensile strength and toughness along with inferior resistance to crack propagation to those of other conventional joints of metals[1,4]. Thus, it threatens safety of adhesive joint and hence is not preferably used for joining of thick metallic components unless the bonded area is large.

Due to its relatively brittle nature the adhesive bond strength largely depends upon bond-line thickness of the adhesive and curing cycle of time and temperature[7]. Prior to application of adhesive a suitable surface treatment of the substrate by, mechanical, chemical and physical processes also improves strength of the adhesive joint[7-9]. The surface treatment generally improves joint properties by modification of substrate surface resulting a better wetting and bond strength between the substrate and the adhesive[9]. In certain cases the surface roughness created by the treatment also introduce positive contribution to the bond strength by initiating mechanical locking[9]. To improve various properties of structural adhesives the metallic or non-metallic powders as filler have been widely used[10]. The addition of fillers reduces shrinkage while curing as well as enhances strength and toughness of adhesive[11]. A variation in type, amount and size of powders influences the mechanical properties of powder reinforced epoxies. But hardly any systematic work has been reported so far to get a clear understanding on the criticality and mechanism of these aspects affecting the mechanical properties of structural adhesives.

In this investigation an attempt has been made to study the effect of epoxy based particulate composite adhesives on the characteristics of adhesive joints of similar sheets of aluminium and plain carbon structural steel. The characteristics of the adhesive joints have been studied with respect to the suitable use of mechanical and chemical surface treatment of the substrates and the bond line thickness of the particulate composite adhesives containing two different types of fillers as aluminium and alumina powders of varying amount and size in a commercial epoxy adhesive. The adhesive joints are characterized by their tensile shear strength. The characteristics of the adhesive joints of the particulate composite adhesive are compared to their counterpart of conventional adhesive. The behaviour of the adhesive joints under shear tensile loading has been critically studied to optimise the bond line thickness and the amount and size of the different type of reinforced particles in the particulate composite adhesive with respect to maximum joint strength.

2. EXPERIMENTAL

The lap joints of plain carbon structural steel sheets (ASTM specification SA-36) and commercial aluminium (ASTM specification SB-209 Grd. 1100) sheets of thickness 2.0 mm each were prepared using conventional epoxy adhesive and particulate composite adhesive. The chemical compositions of the steel and aluminium sheets measured by vacuum emission spectroscopy are given in Table 1. Prior to preparation of all the joints the faying surfaces of both the as extruded aluminium and as rolled steel sheets, were

Table 1

Chemical Composition of the Aluminium and Steel Sheets

Material	Chemical composition (Wt.%)							
	C	Mn	Si	P	S	Ti	Fe	Al
Aluminium	-	-	0.3	-	-	0.02	0.43	Rest
Steel	0.24	1.2	0.13	0.01	0.02	-	Rest	0.28

polished mechanically by thoroughly rubbing with 400 Grd. emery paper so that the surface contamination due to presence of excess oxide layer is practically removed and flat smooth surface all along the faying surface is physically ensured. The mechanical polishing was followed by wiping with acetone to remove any adhering dirt or grease from the surface. The selection of this grade of emery paper for mechanical rubbing of the substrate was made in agreement to an earlier observation[7] in respect to optimum strength of the adhesive joint. Prior to the joint preparation the faying surfaces of some of the aluminium sections were given chemical pickling treatment using a solution developed for this purpose containing concentrated H_2SO_4 , H_3PO_4 , HNO_3 and CrO_3 . The solution was heated up to a temperature just below its boiling point and continuously stirred for about 5 min followed by dipping of the specimen in it for about 1 min. After pickling the specimens were thoroughly washed in water.

2.1 Preparation of adhesive

The conventional and particulate composite adhesives were prepared by using a commercial adhesive containing epoxy resin Araldite AW 106 and hardener HV 953U manufactured by Huntsman Advanced Material (India) Pvt. Ltd. The adhesive was having good stability under humid environment and service temperature up to about 120ºC. The conventional adhesive was prepared by thorough mechanical mixing of the resin and hardener in equal amount by weight.

The particulate composite adhesives were also prepared on the same base of the epoxy resin and hardener of the commercial adhesive as stated above. They were prepared by thorough mechanical mixing of sparingly sprinkled particles of desired amount on the epoxy resins in about 4-6 minutes followed by further mechanical mixing of the slurry for about 4-6 minutes in the hardener with the help of a spatula. The entire mixing operation was carried out at ambient temperature and relative humidity lying in the range of 20-30ºC and 45-55% respectively. Eighteen varieties of composite adhesives were prepared by using two types of commercially available particles of aluminium and alumina of three different size ranges (75-84, 38-44 and 0.8-1.2 mm) in three different amounts (5, 10 and 15 wt.%). The laboratory grade aluminium and alumina powders were collected from the commercial sources. The particles used were practically round in shape with surface characteristics in as manufactured condition. Prior to application of both the conventional and particulate adhesives on the faying surfaces of the substrates they were freshly prepared and degassed for 5 minutes under vacuum of the order of 0.01 mbar.

2.2 Preparation of adhesive joints

The adhesive joints of all the conventional and composite adhesives were also prepared at ambient condition as stated above by applying them on the faying surfaces of the like substrates of aluminium and steel sheets and putting them together in proper position of lap joint confirming the dimensions as shown in Fig. 1. A uniform layer of adhesive of different thicknesses was obtained by rolling the freshly prepared joint with a speed of about 3 mm/min at different loads of 1 to 6 kg applied through a roller set-up. Finally these joints defined as green adhesive joints were cured at 120ºC for 60 minutes by putting them in an oven to facilitate wetting and setting of the adhesive. During curing the bond line thickness of the adhesive introduced in the joint was controlled by leaving the green adhesive joints freely with appropriate support of levelling the sheets under application of no external load on them.

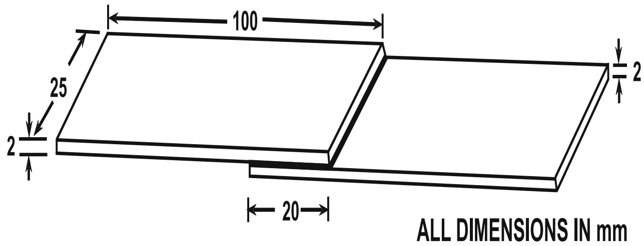

Fig. 1 : Schematic diagram of the lap adhesive joint.

2.3 Optical microscopy

The transverse sections of the adhesive joints were thoroughly polished up to metallographic standard and studied under optical microscope to measure the bond line thickness of adhesive with the help of a micrometer mounted in it. The fracture behaviour of the specimens failed under the tensile lap shear tests was studied under optical stereomicroscope to find out the mode of fracture in different specimens.

2.4 Tensile lap shear test

The tensile lap shear test of the adhesive joints was carried out on a universal testing machine at a crosshead speed of 1 mm/min. The ultimate shear tensile load carrying capacity of the joints was recorded and lap shear strength of the joints was estimated on dividing it by the area of lap joint and compared. Reproducibility of the joint properties was checked by testing at least three specimens for each type of joints and average of the result has been correlated with various characteristics of the adhesive.

3. RESULTS AND DISCUSSION

3.1 Bond line thickness

The bond line thickness of adhesive in an adhesive joint largely depends upon its flow characteristics in between two faying surfaces of the joining substrates. The flow characteristics of an adhesive can be primarily understood as a function of the mode and magnitude of pressure applied on it, surface characteristics of the substrate and most importantly the viscosity of the adhesive. The bond line

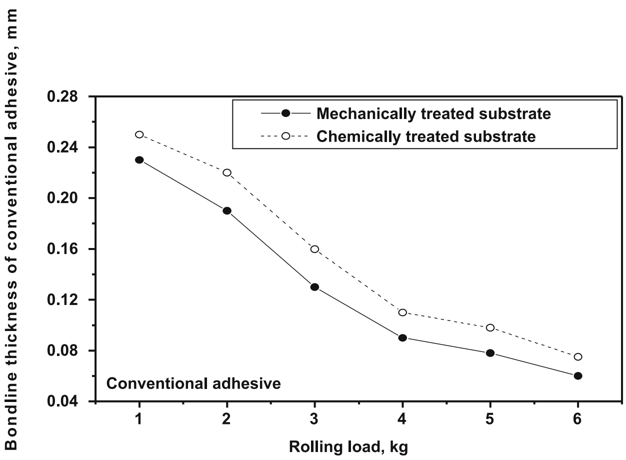

Fig. 2 : At different surface treatment effect of rolling load on bond line thickness of conventional adhesive in aluminium substrate.

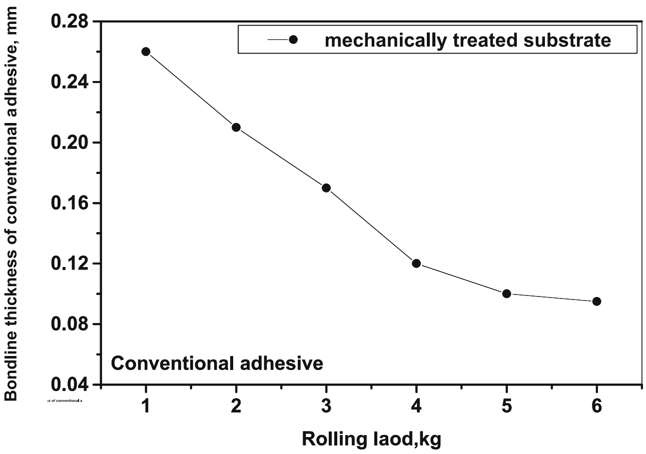

Fig. 3 : At mechanically treated steel substrate effect of rolling load on bond line thickness of conventional adhesive in steel substrate.

thickness in case of the two substrates of aluminium and steel sheets has been found to be significantly affected by the rolling load as well as the surface treatment of the substrate and viscosity of the adhesive. The viscosity of adhesive was varied due to addition of alumina and aluminium particles in different amounts and sizes. At a given surface treatment the effect of rolling load on bond line thickness of the conventional adhesive in aluminium and steel substrates has been shown in Figs. 2 and 3 respectively. Both the figures demonstrate the bond line thickness in mechanically treated substrates when the Fig. 2 also depicts the bond line thickness in chemically treated aluminium substrate. At the mechanically and chemically treated aluminium substrate the effect of rolling load on bond line thickness of the composite adhesive containing varying amount (5, 10 and 15 wt.%) of alumina particles has been shown in Figs. 4 (i), (ii) and (iii), where the particles are present in three different size ranges of 78-84, 38-44 and 0.8-1.2 mm respectively. Similarly at those surface treatment of the same substrate the effect of rolling load on bond line thickness of the composite adhesive containing varying amount (5, 10 and 15 wt.%) of aluminium particles has been shown in Figs. 5 (i), (ii) and (iii), where the

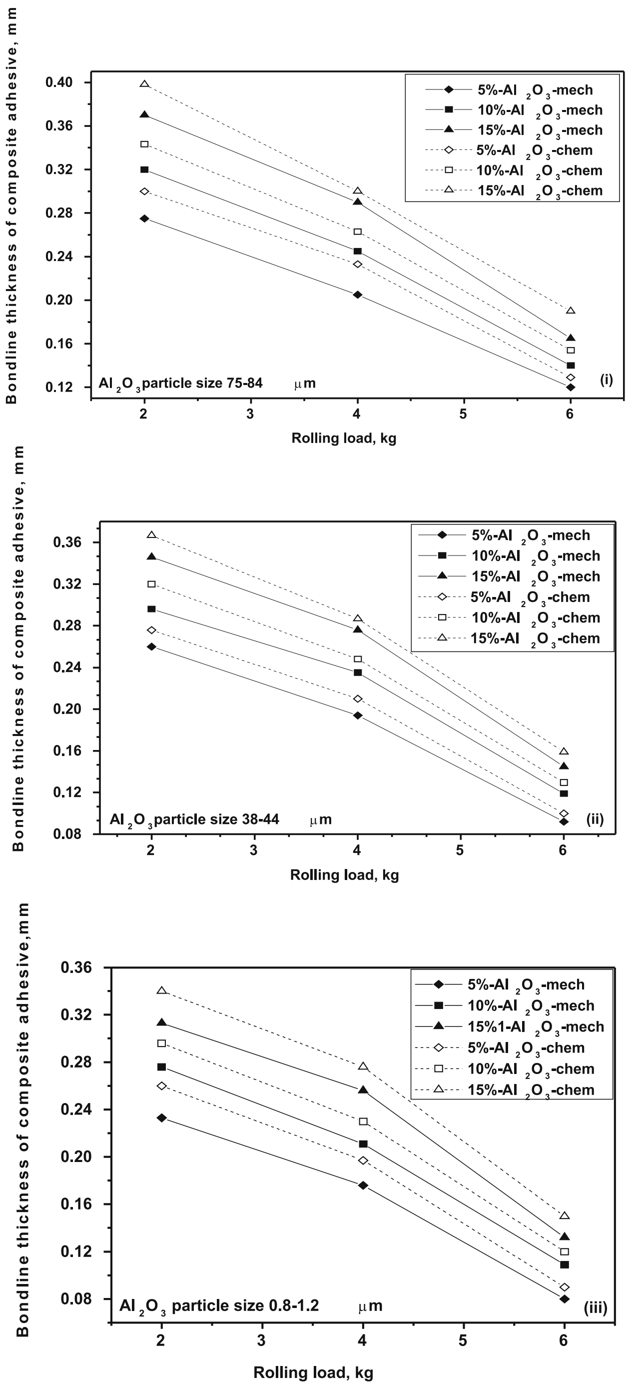

Fig. 4 : At different surface treatment of aluminium substrate effect of rolling load on bond line thickness of composite adhesive containing varying amount of alumina particles in different size ranges of (i) 78-84, (ii) 38-44 and (iii) 0.8-1.2 μm.

particles are present in three different size ranges of 78-84, 38-44 and 0.8-1.2 mm respectively.

At the mechanically treated steel substrate the effect of rolling load on bond line thickness of the composite adhesive containing varying amount (5, 10 and 15 wt.%) of alumina particles in three different size ranges of 78-84, 38-44 and 0.8-1.2 mm has been shown in Figs. 6 (i), (ii) and (iii) respectively. Similarly at this surface treatment of the same substrate the effect of rolling load on bond line thickness of the composite

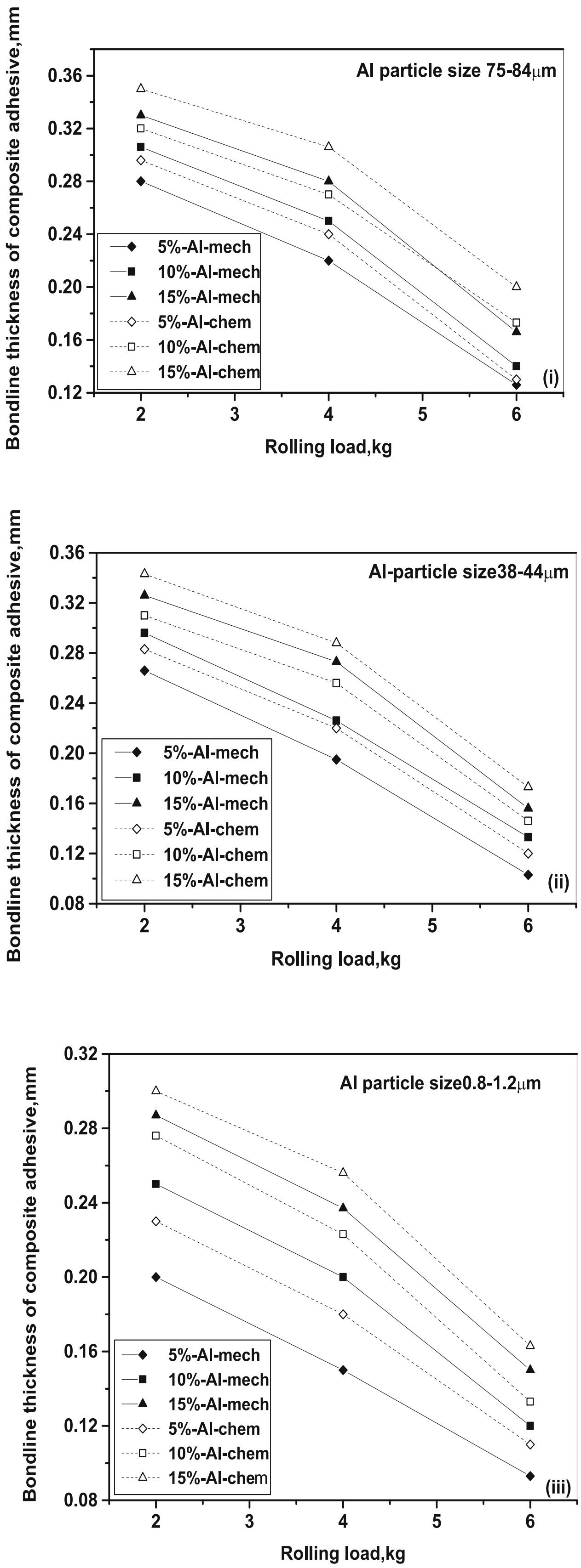

Fig. 5 : At different surface treatment of aluminium substrate effect of rolling load on bond line thickness of composite adhesive containing varying amount of aluminium particles in different size ranges of (i) 78-84, (ii) 38-44 and (iii) 0.8-1.2 μm.

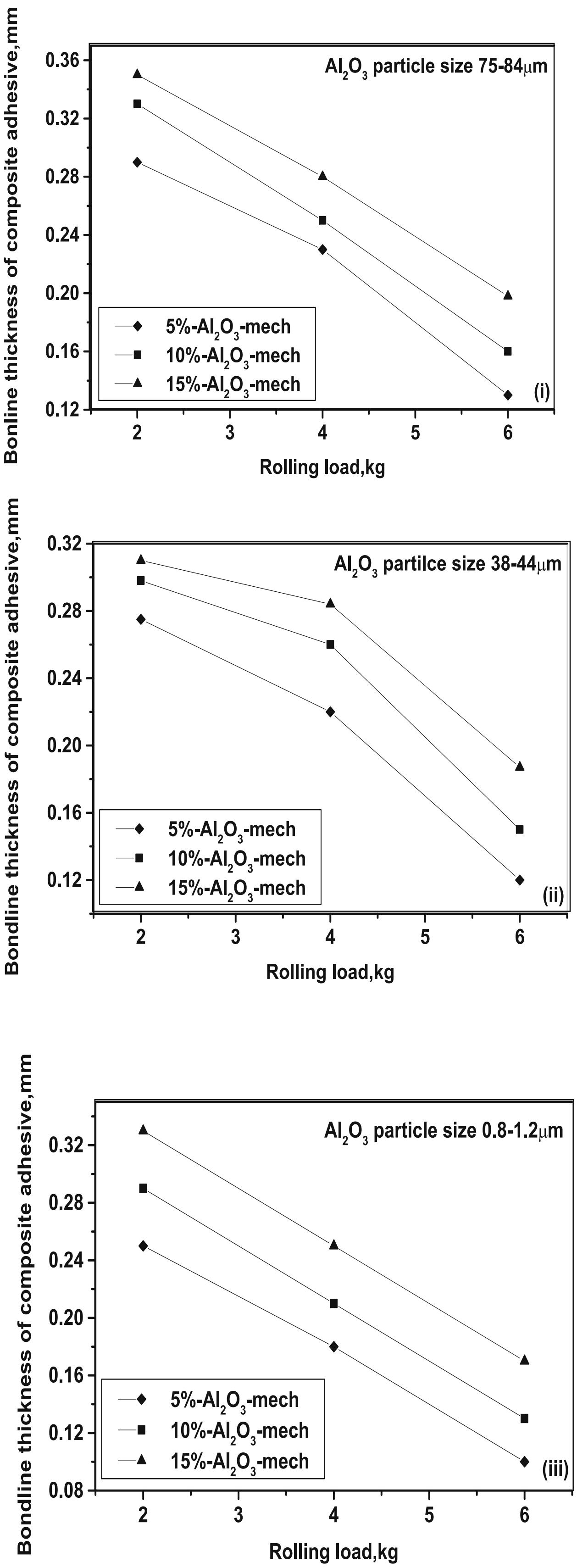

Fig. 6 : At mechanically treated steel substrate effect of rolling load on bond line thickness of composite adhesive containing varying amount of alumina particles in different size ranges of (i) 78-84, (ii) 38-44 and (iii) 0.8-1.2 μm.

adhesive containing varying amount (5, 10 and 15 wt.%) of aluminium particles has been shown in Figs. 7 (i), (ii) and (iii), where the particles are present in three different size ranges of 78-84, 38-44 and 0.8-1.2 mm respectively.

3.1.1 Effect of rolling load

The Figs. 2-7 show that at any surface treatment of mechanical polishing or chemical etching the increase of

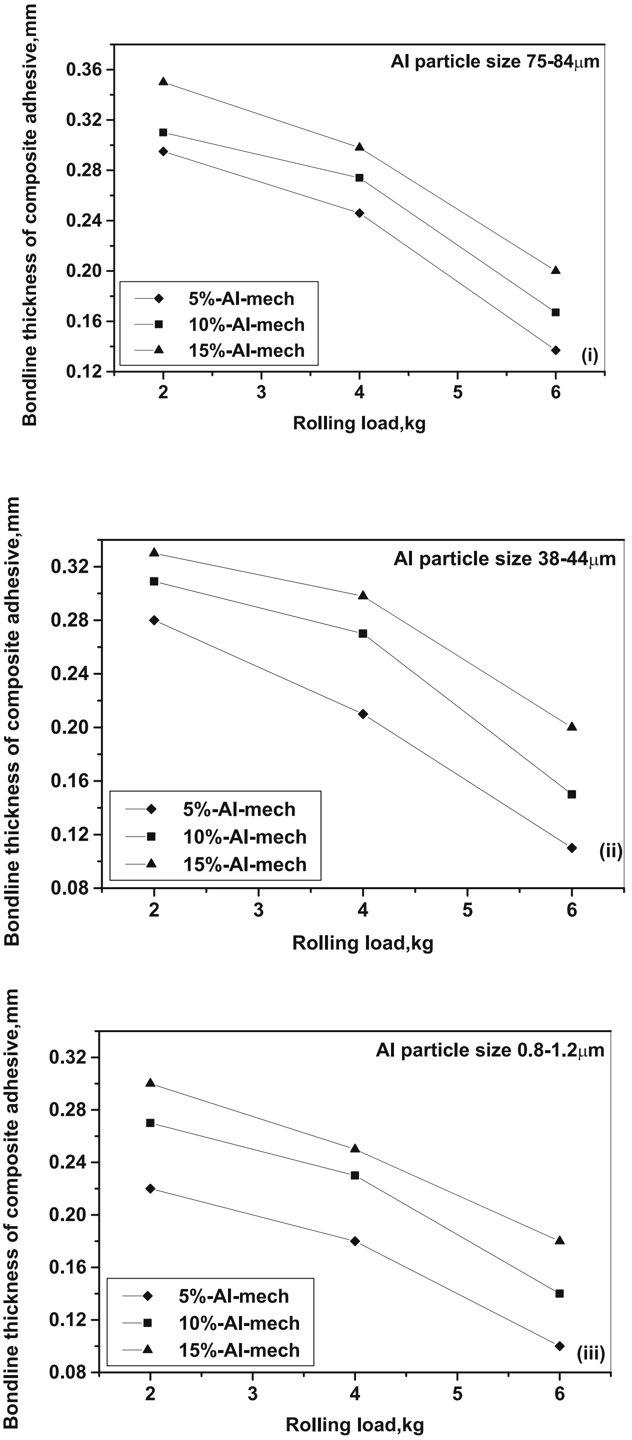

Fig. 7 : At mechanically treated steel substrate effect of rolling load on bond line thickness of composite adhesive containing varying amount of aluminium particles in different size ranges of (i) 78-84, (ii) 38-44 and (iii) 0.8-1.2 μm.

rolling load from 1-6 kg reduces the bond line thickness of both the conventional and composite adhesives. However, at a given rolling load, the bond-line thickness of the composite adhesive (Figs. 4-7) has been found comparatively higher than the conventional adhesive (Figs. 2 and 3). It is also interestingly observed that the nature of reduction in adhesive layer thickness with the increase of rolling load varies with the type of adhesive as the conventional (Figs. 2 and 3) or particulate composite (Figs. 4 and 5), being comparatively sharper in case of the former one up to the rolling load of 4 kg. Whereas, at a higher rolling load beyond it the reduction in adhesive layer thickness reacts more sharply to the increase of rolling load in case of the particulate composite adhesive. The lowering of bond line thickness with the increase of rolling load primarily happens due to squeezing out of the adhesive under the pressure in between the substrates resulting from the enhanced rolling load. However, at a given rolling load the observed higher bond line thickness of the composite adhesive than that of the conventional adhesive may have caused by comparatively higher resistance to flow of the composite adhesive imposed by the presence of particle and its amount.

The variation in mode of reduction in bond line thickness of the conventional and composite adhesives with the increase of rolling load is largely governed by the thixotrophy and rheological behaviour[12] affecting the nature of the complex flow dynamics of the composite adhesives especially under the heterogeneous fluid adhesive in the confined space between the metal sheets. The flow characteristics of the conventional and composite adhesives may have also further influenced by surface tensional force of the substrate up to certain extent. However, at a larger layer thickness the flow of adhesive is predominantly governed by the viscosity of the adhesive, but at a lower layer thickness the flow of adhesive may be largely controlled by the interaction of the substrate with the adhesive. The substrate-adhesive interaction offers a significant resistance to the flow of adhesive and thus it impairs the spreading of adhesive with the increase of rolling load lowering its response to reduction in bond line thickness of adhesive. This phenomenon has been found effective at a bond line thickness of adhesive at least of the order of 0.1-0.08 mm as revealed in case of conventional adhesive shown in Figs. 2 and 3. In case of using particulate composite hardly any situation of such a low bond-line thickness of adhesive has been resulted during the increase of rolling load up to 6 kg to activate an effective interaction of the substrate with the adhesive restricting its flow in between the substrates.

3.1.2 Effect of surface treatment

The Figs. 2, 4 and 5 also interestingly depict that at a given rolling load the bond line thickness of adhesive, in the joints of the conventional and particulate composite adhesives of different composition, is relatively higher in case of the chemically pickled substrate (aluminium) than that observed in case of the mechanically polished substrate. However the trend of reduction in bond line thickness with the increase of rolling load up to and beyond 4 kg has been found similar to that discussed above in case of both the surface treatment of the substrate. The relatively higher bond line thickness of the adhesive in case of using the chemically pickled substrate may have primarily attributed to roughening of the surface by formation of large number fine etch pits in newly introduced coating (possibly chromic oxide) by removing the readily available aluminium oxide layer from the substrate [10]. The surface roughening may have introduced a resistance to flow of adhesive resulting into its observed increase of bond line thickness.

3.1.3 Effect of amount of particle

The adhesive joints of aluminium and steel, prepared by using any size of Al_2O_3 or Al particle reinforced composite adhesives at a given rolling load, shows increase of bond line thickness with the increase in amount of particle content from 5-15 wt% (Figs. 5-8) irrespective of any surface treatment of the substrate. The enhancement of bond line thickness of the composite adhesive with the increase in amount of particle reinforcement is primarily attributed to the increase in thixotropy and resistance to flow of the epoxy adhesive [10].

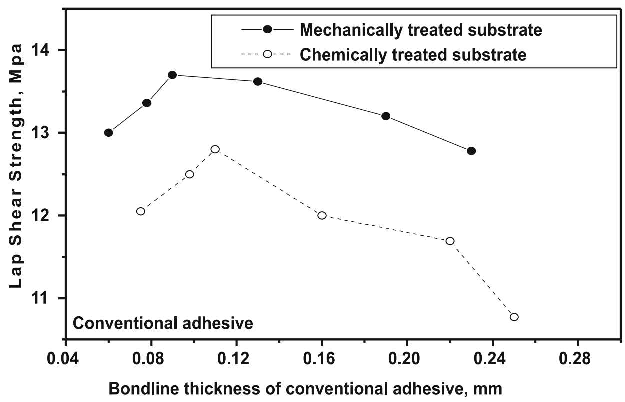

Fig. 8 : At different surface treatment effect of bond line thickness of conventional adhesive on tensile lap shear strength of the adhesive joint of aluminium.

3.1.4 Effect of particle size

At a given rolling load, surface treatment and amount of particle reinforcement the increase in particle size from (0.8-1.2) to (78-84) mm has been found to enhance the bond line thickness of the composite adhesives containing Al_2O_3 or Al particles in the adhesive joints of both the aluminium and steel substrates as revealed in Figs. 4 and 7 respectively. This may have primarily happened because the finer particle cooperates more to the component of flow of adhesive but with the increase of its size it starts imposing resistance to the flow by creating a significant stress field around it, causing enhancement in the bond line thickness of adhesive.

3.1.5 Effect of type of particle

In case of the aluminium substrate it appears that at a given surface treatment, particle amount and particle size the Al_2O_3 containing particulate composite adhesive (Fig. 4) results a comparatively higher bond line thickness than the Al containing particulate composite adhesive (Fig. 5) especially at lower rolling load of 2 kg. But, at higher rolling load of 6 kg this behaviour has been found relatively insignificant. However, this phenomenon has not been found valid in case of the adhesive joint of steel (Figs. 6 and 7) having adhesive containing higher size of particle beyond 0.8-1.2mm. The observed insignificant influence of the type of particle at higher lolling load of 6 kg on the bond line thickness of adhesive possibly attributed to predominant influence of the substrate surface at low bond line thickness. The influence of the oxide (Al_2O_3) and metallic (Al) particles governing The variation in bond line thickness in presence of the oxide (Al_2O_3) and metallic (Al) particles is possibly attributed to the flow characteristics of the adhesive dictated by the surface roughness of the particles, where the generally found comparatively rougher surface of the oxide particle may have exerted relatively higher resistance to flow of adhesive resulting a comparatively higher bond line thickness. However, the basic mechanism of this phenomenon may be studied further in detail, which is beyond the scope of the present investigation.

3.2 Tensile lap shear strength

The lap shear strength of adhesive joint largely depends upon bond line thickness of the adhesive and surface treatment of the substrate. However, in case of the adhesive joint of particulate composite adhesive the amount, size and type of reinforced particle plays a significant role on mechanical properties of the joint primarily due to their considerable influence on fracture characteristics.

At a given surface treatment of mechanical polishing or chemical pickling the effect of bond line thickness on lap shear strength of the conventional adhesive joints of aluminium or steel substrates has been shown in Figs. 8 and 9 respectively. At the mechanically and chemically treated aluminium substrate the effect of bond line thickness on lap shear strength of the composite adhesive containing varying amount (5, 10 and 15 wt.%) of alumina particles has been shown in Figs. 10 (i), (ii) and (iii) where the particles are present in three different size ranges of 78-84, 38-44 and 0.8-1.2 mm respectively. Similarly at those surface treatment of the same substrate the effect of bond line thickness on lap shear strength of the composite adhesive containing varying amount (5, 10 and 15 wt.%) of aluminium particles has been shown in Figs. 11 (i), (ii) and (iii) where the particles are present in three different size ranges of 78-84, 38-44 and 0.8-1.2 mm respectively. At the mechanically treated steel substrate the effect of bond line thickness on lap shear strength of the composite adhesive containing varying amount (5, 10 and 15 wt.%) of alumina particles in three

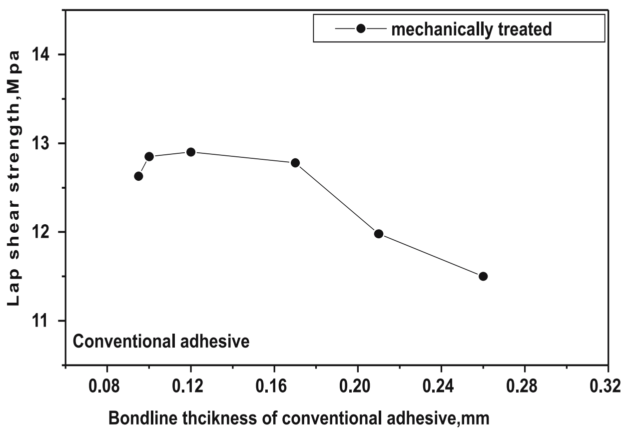

Fig. 9 : Effect of bond line thickness of conventional adhesive on tensile lap shear strength of the adhesive joint of mechanically polished steel.

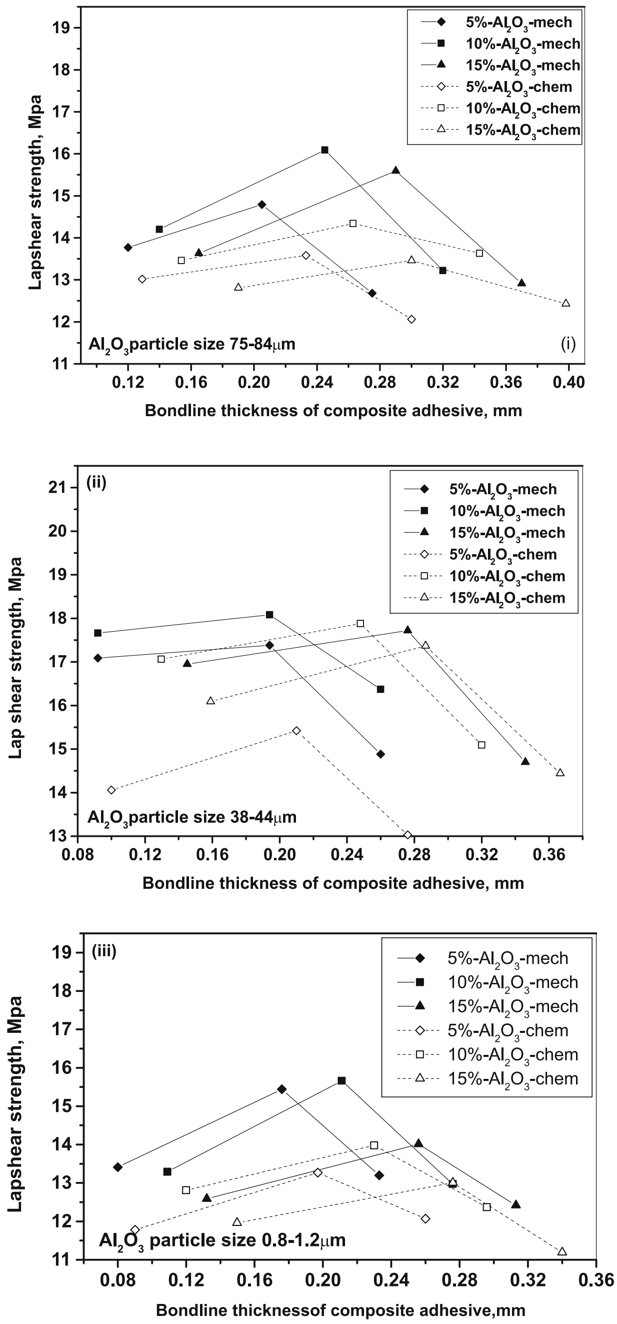

Fig. 10 : At different surface treatment of aluminium substrate effect of bond line thickness on tensile lap shear strength of composite adhesive joint containing varying amount of alumina particles in different size ranges of (i) 78-84, (ii) 38-44 and (iii) 0.8-1.2 μm.

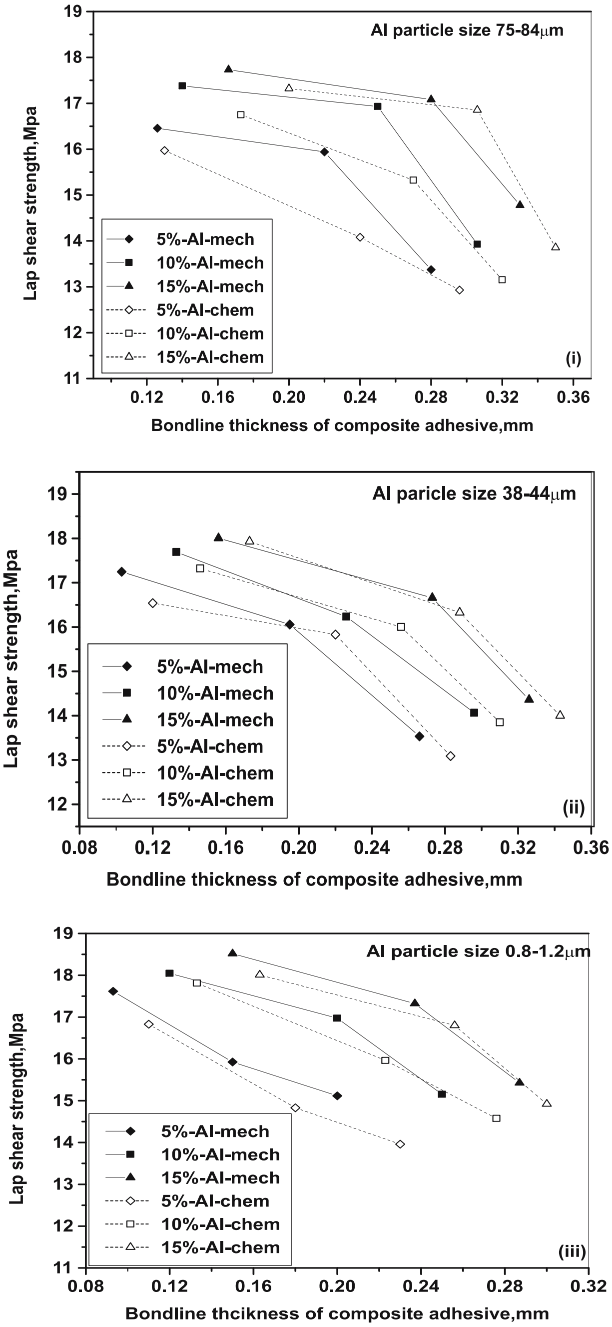

Fig. 11 : At different surface treatment of aluminium substrate effect of bond line thickness on tensile lap shear strength of composite adhesive joint containing varying amount of aluminium particles in different size ranges of (i) 78-84, (ii) 38-44 and (iii) 0.8-1.2 mm.

different size ranges of 78-84, 38-44 and 0.8-1.2 mm has been shown in Figs. 12 (i), (ii) and (iii) respectively. Similarly at this surface treatment of the same substrate the effect of bond line thickness on lap shear strength of the composite adhesive containing varying amount (5, 10 and 15 wt.%) of aluminium particles has been shown in Figs. 13 (i), (ii) and (iii), where the particles are present in three different size ranges of 78-84, 38-44 and 0.8-1.2 mm respectively. The results presented in Figs. 8-13 show that irrespective of the substrate and its surface treatment by chemical pickling or mechanical polishing the adhesive joints prepared by applying composite adhesive give comparatively better strength than that of the conventional adhesive joint. However, the tensile shear strength of the adhesive joints of the particulate composite adhesive varies with the bond line thickness and surface treatment as well as the amount, size and type of particle reinforcement in the adhesive.

3.2.1 Effect of bond line thickness

In case of the adhesive joints of both the mechanically polished aluminium and steel substrates the bond line thickness of the conventional adhesive of the order of about 0.09-0.1 mm has been found to give rise to maximum joint strength followed by a reduction in it with a further increase

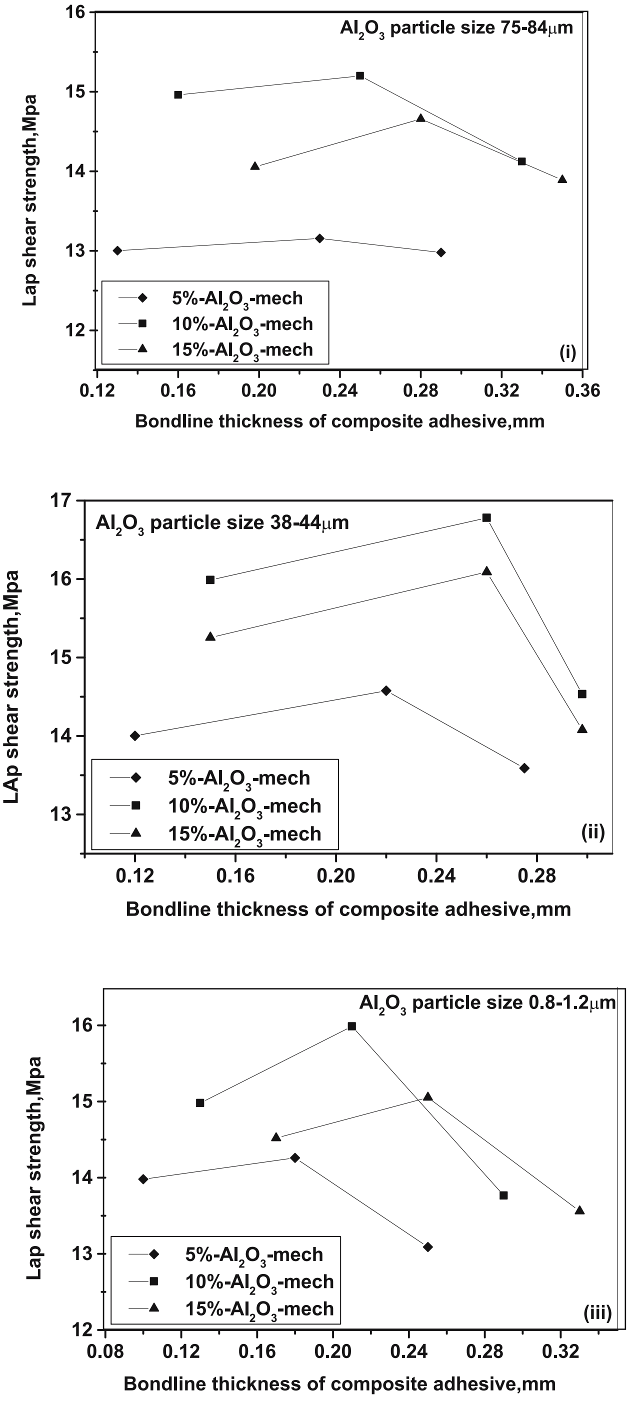

Fig. 12 : At mechanically treated steel substrate effect of bond line thickness on tensile lap shear strength of composite adhesive joint containing varying amount of alumina particles in different size ranges of (i) 78-84, (ii) 38-44 and (iii) 0.8-1.2 μm.

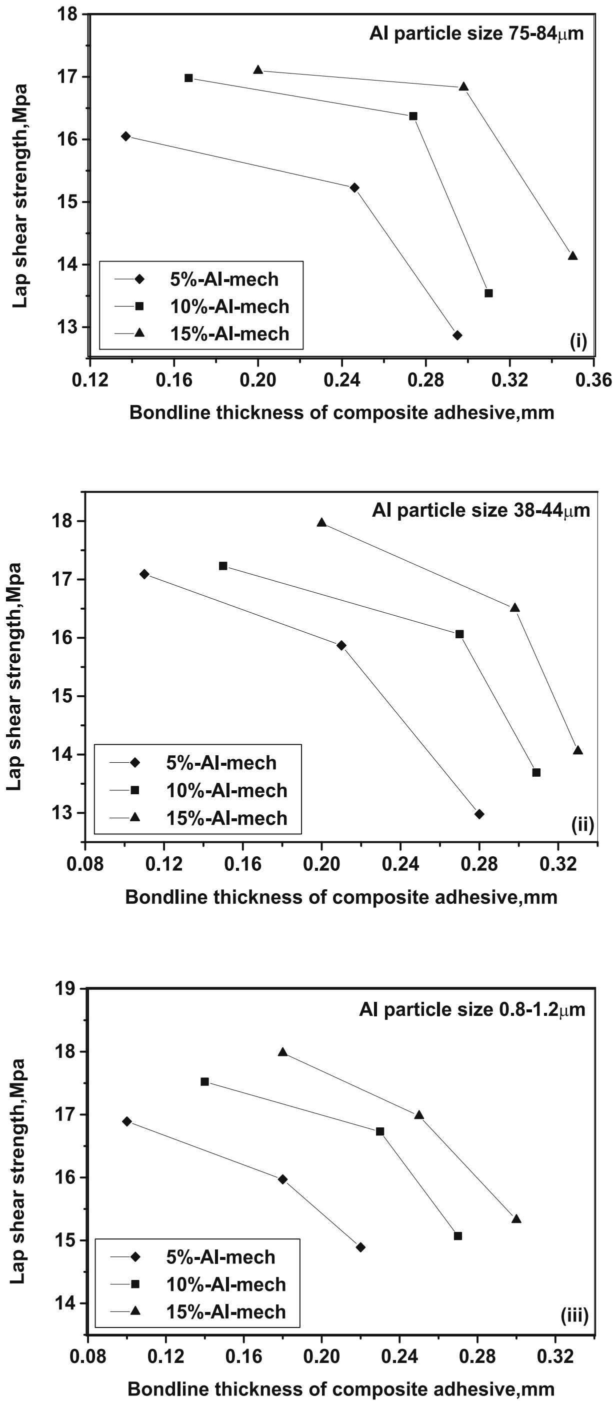

Fig. 13 : At mechanically treated steel substrate effect of bond line thickness on tensile lap shear strength of composite adhesive joint containing varying amount of aluminium particles in different size ranges of (i) 78-84, (ii) 38-44 and (iii) 0.8-1.2 μm.

in bond line thickness as shown in Figs. 8 and 9 respectively. However, the adhesive joint of the chemically pickled aluminium has been found (Fig. 8) to give maximum joint strength at a relatively higher bond line thickness of the conventional adhesive of about 1.1 mm.

In case of the adhesive joint of the composite adhesive, irrespective of any surface treatment or type of substrate (Figs. 10-13) the joint has been found to achieve maximum strength at certain bond line thickness followed by a reduction in it with a further increase of bond line thickness. The figures also interestingly depict that the optimum bond line thickness giving maximum strength to the adhesive joint of any substrate enhances with the increase in amount of particle reinforcement in the composite adhesives. However, in agreement to that found in case of the conventional adhesive the optimum bond line thickness of the composite adhesive giving maximum strength to the adhesive joint of aluminium (Figs. 8, 10 and 11) has been found relatively lower in case of the mechanically polished substrate than that observed in the chemically pickled one. The figures also depict that at a given bond line thickness the application of mechanical polishing to the substrate results a comparatively higher joint strength than that happens in case of the chemically pickled one. A comparison of the Figs. 8 and 9 with the Figs. 10-13 reveals that for maximum joint strength the bond line thickness of adhesive joint of any composite adhesive is in general significantly higher than that of the conventional adhesive joint of irrespective of the substrate and its surface treatment. In agreement to earlier observation [13] the comparatively higher bond line thickness of the composite adhesive than that of the conventional adhesive giving maximum joint strength shows that the particle reinforcement enhances resistance of the adhesive to cohesive fracture.

A low joint strength of both the conventional and composite adhesives at thin bond-line thickness of adhesive may have resulted from the presence of a significant amount of unbonded surface area due to tearing of too thin film of adhesive resulting in discretely presence of accumulated adhesive film on the substrate[14]. However, at higher bond-line thickness of adhesive with uniform spreading at the interface a competitive nature of predomination of the strengths of three regions of an adhesive joint, such as the interface/interphase bond strength, shear strength of adhesive adjacent to its interface and cohesive strength of the adhesive, primarily dictates the joint strength[14]. In case of the conventional adhesive it is reported[10,14,15] that the interphase region of adhesive adjacent to the interface with the substrate possesses maximum strength out of the three regions as stated above and the performance of the bond depends upon net work structure of the adhesive influenced by the surface treatment of metal like aluminium[16-18]. A comparatively high strength of the adhesive adjacent to its interface with the substrate may arise from a relatively larger resistance to deformation of this region under the influence of its good interfacial strength. But, the situation can be different in case of the composite adhesives, where the cohesive strength of the adhesive under the influence of reinforcement may primarily dictate the joint strength. This phenomenon has been marked as cohesive fracture of the adhesive joint containing 15wt.% of 0.8-1.2 mm Al particle with certain amount of porosity. However, to characterise the adhesive joint of the composite adhesive for its maximum strength the mechanism of fracture should be studied further, which is beyond the scope of the present investigation.

The chemical pickling of aluminium in the solution as stated above may have improved the strength of its adhesive joints primarily by roughening of the faying surface through pitting and oxide coating[10]. But it may not serve the critical requirements of surface roughening defined by the size of peaks and valleys as created in mechanical polishing. The comparatively higher strength of the adhesive joint of the mechanically polished aluminium substrate than that of the chemically pickled one may have primarily occurred due to interlocking between the substrate and adhesive giving rise to maximum strength at optimum level of surface roughness[7]. This phenomenon is true to any substrate of aluminium or steel. However, the generally observed comparatively higher joint strength of the mechanically polished aluminium joints (Figs. 8,10,11) than the mechanically polished mild steel joints (Figs. 9,12,13) of both the conventional and composite adhesives may have primarily caused by the formation of aluminium oxide with deficiency of oxygen[5,9,16-21] at the polished surface of the substrate prior to application of adhesive on it, which becomes more reactive to the adhesive and provide stronger bonding with the adhesive and the substrate giving rise to improved joint strength.

3.2.2 Effect of amount of particle

At a given bond line thickness and surface treatment the tensile shear strength of the adhesive joints of both the aluminium and steel substrates prepared by using the composite adhesive has been mostly found to enhance (Figs. 10 and 12) with the increase in amount of alumina particle reinforcement up to 10 wt% followed by a decrease in it with a further increase in particle reinforcement to 15 wt% irrespective of the particle size. However, the situation has been interestingly found (Figs. 11 and 13) different in case of the adhesive joints of aluminium particle reinforced composite adhesive, where the increase in amount of particle reinforcement from 5 to 15 wt% has been found to steadily enhance the tensile shear strength of the adhesive joint irrespective of the particle size.

The increase in adhesive joint strength of both the aluminium and steel substrates (Figs. 10 and 12) with the increase of alumina particle content in the adhesive from 5 to 10 wt. % may be primarily happened due to well known resistance to fracture provided by the particle with the help of crack blunting mechanisms[22,23]. It happens through suitable modification of stress distribution in the matrix under an effective inter-particle distance affecting the localised shear yielding and damage formation due to crack diversion and particle-matrix debonding[24,25]. But in agreement to an earlier observation[13] the relative decrease in joint strength with the increase of particle content to 15 wt % possibly primarily attributed to adverse interaction amongst the particles resulting from lowering of inter-particle spacing beyond a critical level in the matrix. However, in this regard the adverse influence of surface roughness of the oxide along with significant gas content[26] on its bonding with the adhesive may not be ignored. The surface roughness of untreated oxide may provide gas pocket and thus impair soundness of its bonding with the adhesive. Thus, it can play a significant role in determining the critical inter-particulate spacing for maximum adhesive joint strength, which enhances with the weakening of the particle matrix bonding causing reduction in joint strength at comparatively low particle content. This phenomena is further conformed in case of the adhesive joints of both the aluminium and steel substrates (Figs. 11 and 13) prepared by using aluminium particulate composite adhesive where the joint strength has not been found to decrease up to the particle content of 15 wt.%. Rather at a given amount (wt.%) of reinforcement the comparatively more effective contribution of Al than Al_2O_3 powder on strengthening of the adhesive joint is primarily attributed to availability of more number of particles in case of the earlier one due to its comparatively lower density, which reduces the inter-particle distance within the critical limit of its positive contribution on matrix strengthening. The resistance to fracture offered by the particle with the help of crack blunting mechanisms also justifies the maximum strength of adhesive joints (Figs. 10-13) at higher bond line thickness of composite adhesive containing larger amount of particle.

3.2.3 Effect of particle size

In case of the adhesive joints of a given amount (in the range of 5-15 wt.%) of alumina particle reinforced composite adhesive of both the mechanically and chemically treated substrates the joint strength has been found (Figs. 10 and 12) to increase with the decrease in particle size from 78-84 to 38-44 ìm followed by a decrease in it with a further decrease in particle size to 0.8-1.2 µm. This is in agreement to an earlier observation [27] where the approximately round shape untreated 35 ìm size glass particle filled epoxy adhesive has been found to result maximum adhesive joint properties specially with respect to strength and fracture toughness. However, the strength of the adhesive joint of a given amount (in the range of 5-15 wt.%) of aluminium particle reinforced composite adhesive of both the mechanically and chemically treated substrates has been found (Figs. 11 and 13) to enhance steadily with the decrease in particle size from 78-84 to 0.8-1.2 ìm.

At a given amount (wt.%) of particle reinforcement the decrease in particle size enhances the number of particles in the matrix, which increases the strength of the adhesive joint by reduction in interparticulate spacing due to on setting of resistance to fracture with the help of crack blunting mechanisms as stated above. Moreover, at a given amount the finer particle reinforcement provides more surface area of contact with the adhesive and the substrate, which may also positively contribute to the improvement in strength of the adhesive joint. However, in the line of the discussions given above the decrease in inter-particulate spacing below a critical level with the decrease in particle size, which may have achieved easily in case of the reinforcement of comparatively rough alumina particle of size in the range of 0.8-1.2 µm, possibly reduced the strength of the adhesive joint. The surface roughening of alumina containing appreciable amount of gas in it may have also impaired the positive influence of surface area on the joint strength than that observed in case of the joint containing aluminium particle reinforced adhesive.

3.2.4 Effect of particle type

At a given state of surface treatment of the substrates including the amount and size of particle reinforcement within the range as stated above the strength of the adhesive joints containing composite adhesive having aluminium particle reinforcement (Figs. 11 and 13) has been found comparatively higher than that of the adhesive joints of the alumina particle reinforced composite adhesive (Figs. 10 and 12). It is also interestingly observed that the aluminium particle reinforcement within the amount and size in the ranges of 5 to 15 wt.% and 78-84 to 0.8-1.2 ìm respectively dose not maintain any criticality of its effect on strength of the adhesive joint showing (Figs. 11-13) maximum strength at higher amount and lower size of particle at thinner bond line thickness. Whereas the alumina particle reinforcement within the amount and size in the ranges of 5 to15 wt.% and 78-84 to 0.8-1.2 µm respectively shows (Figs. 10-12) criticality in attaining maximum joint strength at the amount and size of reinforced particle of 10 wt.% and 38-44 ìm respectively.

From the above discussions on the role of amount and size of particle on the strength of adhesive joints of both the substrates of aluminium and steel it is understood that the surface characteristics of the particle play significant role on it. Although a rough surface improves strength of the adhesive joint up to a certain extent providing more area of bonding but, for a given adhesive it may considerably put forward an adverse influence on the particle-adhesive bonding by entrapment of gases in the pores and valleys depending upon the contour and degree of surface irregularities[9,10] as well as gas content of the particle. The better strength of adhesive joint of particulate composite adhesive containing aluminium than alumina particles may have primarily attributed to comparatively smooth surface of aluminium powder with less gas content in it than that of its oxide.

4. CONCLUSIONS

The lap shear strength of the adhesive joints of similar sheets of aluminium and steel is a function of bond line thickness of epoxy based particulate composite adhesives as well as its type of filler as aluminium and alumina powders and their amount (5-15wt.%) and size(75-84 to 0.8-1.2 mm). The use of particulate composite adhesive considerably improves the strength of the adhesive joints with respect to that of the conventional epoxy adhesive joints. The composite adhesive having aluminium particle of size 0.8-1.2 mm in 15 wt.% most effectively enhances the joint strength especially in case of the aluminium substrate. Whereas the alumina particulate composite adhesive provides maximum adhesive joint strength to both the substrates with the particle of size of 38-44 mm in 10 wt.%. In general the adhesive joint of the mechanically polished aluminium is comparatively stronger than adhesive joint of the mechanically polished steel. For any size and amount of particle generally the optimum bond line thickness for maximum joint strength is comparatively lower in case of the composite adhesive containing aluminium (0.15-0.18mm) than alumina (0.2-0.25mm) particle. The use of chemical pickling instead of mechanical polishing relatively enhances the optimum bond line thickness for maximum joint strength of the adhesive joint, when mechanical polishing becomes more effective than the chemical pickling to strengthen the adhesive joint of aluminium substrate.

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