Effect of Surface Pretreatment on Hot Press Lap joining of High Density Polyethylene to Stainless Steel Alloy AISI 304L

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Abstract. Hot press bounding technique was used to join a polyethylene sheet of 4mm thickness to stainless steel AISI 304L of 1mm thickness. Three different process parameters were used during the joining process: pressure of 4, 6, 8 and 10 bar, processing time of 1.5, 2, 2.5 and 3 min. and temperature of 115, 120, 125 and 130 °C. The steel surface was treated with two methods: mechanical (scratch) and electrochemical (electro-polishing) treatment. A shear tensile, microstructure, scanning electron microscope and energy dispersive spectrometry tests were used to examine the joint specimens. The effect of process parameters on the joint properties was analyzed by the Minitab program. The scratched and electro-polished joint specimens exhibited a maximum shear force of 1112 and 1022N respectively. The tested joints were failed by an interfacial shear and necking in the polymer side with a ductile fracture. The joining process was occurred by a mechanical interlocking between the molten polymer and pores of the treated surface of steel specimen. The average thickness of the joining line for the scratched and electro-polished specimens was 5 and 4µm.

Keywords: Hot press, hybrid joining, polyethylene, electro-polish.

1. Introduction

At this time of rapid technological progress, there is no single material which collects all the needed exceptional properties, such as light weight, high strength, high corrosion resistance, high conductivity and high toughness [1]. The joining process of dissimilar materials between polymeric and metallic materials is important in engineering applications. Engineering structures such as those used in automobile, aerospace and medical and biological industry are made from a hybrid parts of light weight dissimilar materials, such as stainless steel, aluminum or magnesium alloys which joined with polyethylene, polyvinyl chloride and fiber reinforced polymers. The hybrid joints exhibited a combined mechanical, physical and chemical properties between the metal and the joined polymers [2,3,4]. The metal part is used in sections where high strength and stiffness can be exploited, while the plastic material begin the balance of stiffness alongside with effect resistance and moreover enables functional integration across the complex shapes formation in the molding process [5]. There are many processes for joining metal to polymer such as hot press bounding process [6], laser direct joining [7] and friction spot welding [8]. The joining of Metal to polymer is difficult due to the differences in the mechanical and physical characteristics [9]. Light weight dissimilar materials techniques for joining, particularly metals and polymers, increasingly were becoming important in the manufacturing of hybrid joints parts and components for engineering applications [10]. The hybrid aluminum to polyamide joints have been bonded by a novel laser beam method. Surface pre-treatment of the metal surface by electro chemical, mechanical (scratch) or laser had a special effect on the joint shear strength [11]. Metals and polymers are joined mechanically by interlock or chemically bonded with adhesives. So, to enhance the strength, surface roughness is very important to be taken in consideration due to the size and density of the surface roughness and the size and depth of the pores at the surface of the metal [12]. Short carbon fiber reinforced poly-ether ether ketone (SCF/PEEK) and aluminum alloy AA2060-T8 were joined by friction stir welding with co-controlling shape and performance, the mechanical interlocking and the chemical bonding refer to the main bonding mechanisms [13]. Laser direct joining process of glass fiber reinforced polyamide polymer to steel was carried out to estimate its influence on the joint’s mechanical
performance [14]. AZ31 Mg alloy and carbon fiber reinforced plastic (CFRP) was joined via hot press bounding process. Two groups of specimens were used: one as received and the other were annealed. The oxide layer was grown into the CFRP near the joint interface [15]. A hot press binding process was used to join the polyamide 6 (PA6) with metal. At the elevated temperature of metal, the polymer surface is melted and wet the surface of the metal [16]. F. Balle, et. al., (2007) performed ultrasonic metal welding process of aluminum alloy (AA5754) to carbon fiber reinforced polymer (CFRP) has been carried out. Both of the adhesive bonding and mechanical interlocking are outset in the weld zone [17]. Seiji Katayama and Yousuke Kawahito, (2008) carried out a laser direct joining of metal and plastic was investigated to join a polyethylene terephthalate (PET) polymer of 2 mm thick and 30 mm width overlapped on to stainless steel type (AISI 304) of 3 mm thickness. Close contact between the metal and polymer was obtained by physical, chemical bonding and mechanical interlocking occurring at the interface [18]. A two sealants were used in the investigation of the refill friction stir spot joining process (RFSSJ): phenol formaldehyde resin based adhesive and epoxy resin based tape. The joining process has been done by single lap joint of (RFSSJ) [19]. Yongxian Huang et. al., ( 2018) attained the hybrid joint between the aluminum alloy AA6061-T6 and poly ether ether ketone (PEEK) using friction stir lap welding via a tapered thread pin with the triple facets [20]. The tapered thread pin and the triple facets had potential and feasible to this joining process of metal and polymer on the one hand of enhancing mechanical interlocking [20]. The mechanism of the bonding of the metal to polymer hybrid joints was achieved by a mechanical interlocking [21].

The aim of this work is to join a stainless steel AISI 304L to polyethylene by a hot press bounding process. The effect of steel surface pre-treatment was taken into account to study its effect on the joint properties. Two type of surface pre-treatment were used: electro-polish and scratched methods. In order to examine the joint strength, a tensile shear tests were a achieved for each specimen. The joining mechanism was analyzed by microstructure, scanning electron microscope (SEM), Energy Dispersive Spectroscopy (EDS). A design of experiment method was used to investigate the effect of process parameters on the joint performance.

2. Experimental work

2.1. Materials

Two types of materials were used in the joining process: stainless steel alloy type AISI 304L and high density polyethylene (HDPE) of melting point $T_m=139{\degree}C$ and the molecular weight was $0.19\times 10^6$ g/mol. The chemical and mechanical properties of stainless steel alloy are presented in Tables 1 and 2 respectively.

| Element wt% | C% | Mn% | Si% | P% | S% | Cr% | Ni% |
|-------------|----|-----|-----|----|----|-----|-----|
| Standard [22] | 0.03 | 2 | 1.00 | 0.045 | 0.030 | 18-20 | 8-12 |
| Actual | 0.027 | 1.1 | 0.501 | 0.005 | 0.005 | 19.7 | 10 |

Table 1. Chemical composition of AISI 304L.

| Property | $\sigma_u$ (MPa) | $\sigma_y$ (MPa) | % Elongation |
|----------|----------------|----------------|--------------|
| Standard [22] | 564 | 210 | 58 |
| Actual | 605 | 290 | 62 |

Table 2. Mechanical properties of AISI 304L.
2.2. Specimens Preparation

The specimens of AISI 304L are prepared from a sheet of thickness 1mm and the specimens of HDPE are prepared from a sheet of thickness 4mm. The specimens of AISI 304L and HDPE have the same dimensions of width 25mm and length 100mm. The specimens were manufactured according to the standard specification AWS C1.1M/C1.1:2012 spot welding standard, as shown in Figure 1 [23].

![Figure 1. Schematic of AISI 304L/ HDPE with lap joint of 25 * 25 mm²](image)

2.3. Surface Pretreatments

2.3.1 Scratch surface pre-treatment

The scratch surface pretreatment had been done according to the following:

The specimen was rinsed with warm water and liquid dish detergent. The specimen was washed by sponge and water. Taking into account the areas in which the grime and grease was accumulated. After that the specimen was washed with water to clear the suds. The specimen was wiped and dried with a clean lint-free towel. So as, a small amount of a liquid abrasive cleaning product was poured in a soft scouring pad (be sure the cleaning product is suitable for stainless steel). Then, the surface of the stainless steel was rubbed back and forth over using a suitable grade of emery paper. washing process was achieved to remove the grits remaining on the surface of the stainless steel. Dried the scratched area with a suitable hand pressure. A different size of emery paper grit (P100, P220, P320 and P600) was used to achieve a variable surfaces roughness.

2.3.2 Electro-Chemical surface pre-treatment (Electro-polishing)

An electro-chemical rig was used to pre-treatment the steel surface as shown in Figure 2. The electro-chemical rig consisted the following parts: a glass beaker which used as an electro-polishing cell, power supply in which the AC is converted to DC with a voltage range 0-60 volt and current range 0-15 Amp. The main benefit of this part is to produce a uniform DC and as a result the anodizing current will be pass uniformly through the electro chemical cell. The anode was connected to a multimeter to read the processing current. A typical electro-polishing solution consisted of an equal volume mixture of 97% mass fraction of sulphuric-acid and 85% phosphoric acid. The applied current density was 8 Amp. with 12 volt which applied according to the ASM Metals Handbook, Vol. 05 [24]. The process has been achieved according to the following:

The specimen was rinsed with warm water and liquid dish detergent. A wet sponge was used to wash the specimen to remove the dust, grime and grease. After that, the specimen was washed with clean water, wiped and dried to remove the suds. Now, immersing the specimen into the electrolyte solution at a suitable distance between cathode and anode during a time of 8 minutes. Then, the specimen was rinsed in warm water and dried. The purpose of post treatment in nitric acid is to dissolve the film of chemical by-products that forms as the electrochemical reactions take place.
2.4. Hot Press Equipment and the Joint Process

The main goal of this device is to join the pretreated stainless steel specimen to high density polyethylene via the applied pressure and heat generated within a known time. The pressure and temperature were regulated by the hydraulic press and temperature controller respectively. The device system is assembled as shown in Figure 3. This equipment consisted of a hydraulic press (10 ton capacity), heater and heating control ($T_{\text{max}} = 380^\circ\text{C}$) and sample die fixture. The heating device consisted of lower and upper plates with a heating source which placed between them. The thermocouple was used to measure the temperature at the surface of the metal. To achieve a mutual centre line for each specimen, a suitable die fixture was built from two parts: aluminum was the first one and the maica was the second. Specimen of stainless steel is placed on the side of aluminum die through the slot; while, the polymer specimen was placed on the side of the maica die. So, the heating can be easy transfer by the conductivity from the heater to the stainless steel specimen through the side of this aluminum die. The polymer specimen is pressed on the surface pre-treatment of the AISI 304L specimen.

Figure 4 presents a sample of the joint specimens.

Figure 2. Sketch of Electro-chemical rig

Figure 3. (a) hot press rig (b) specimen of fixture die (c) heater

The hot press process parameters were temperature, pressure and time. The temperature was 115, 120, 125 and 130$^\circ\text{C}$, while the pressure was between 4, 6, 8 and 10 bars. The pressing time was 1.5, 2, 2.5 and 3 minutes. The design of experiment was used to analyze the effect of those parameters. Figure 4 presents a sample of the joint specimens.
2.5. Experimental Tests

The samples which were tested by shear test, optical microstructure, scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). The tensile shear has been achieved according to the standard specification (AWS spot welding C1.1M/C1.1:2012) [23]. The tensile test was achieved such that two shims were placed in the opposite sides to prevent sliding and bending during the test as shown in Figure 5. The cross head velocity was 10mm/minute for the shear test.

![Figure 5. tensile test setup](image)

According to ASTM E 407-99 the microstructure inspection of the samples has been done by grinding the surface with emery papers of SiC with a different grain size (P400, P600, P800, P1000, P1200 and P2000) [25]. Polishing process was used with different particle size of alumina 3, 0.3 and 0.05 µm. The specimen was washed with water and alcohol and dried with air.

An energy dispersive spectroscopy test was used to analyze and examine the joint details and indicate the percentage of elemental distribution. A scanning electron microscope was used to examine the microstructure of the cross section joint and indicate the interface between metal and polymer.

The design of experiment method was used to study the effect of the processing parameters on the shear force of specimens.

3. Results

3.1. Shear tensile test of joints

All the hot pressed specimens were joined successfully according to the experimental joining process. The shear tests indicated that all the joined specimens have a specific shear force value for each one. Figure 6 and Table 3 indicates the results of the shear forces for the lap joint specimen of stainless steel AISI 304L to Polyethylene (HDPE). The minimum shear force value was found in specimen no.1 with boundary conditions; pressure = 4bar, time = 1.5 min., temperature = 115 °C and scratching grade = p100, because of the low temperature and minimum time which is not enough to complete the joining. While the maximum shear force in this group was found in specimen no.5, in which, the applied pressure, temperature, time and scratching grade were; 6 bar, 120 °C, 1.5 min. and p320 respectively, which has a
suitable temperature and pressure also a good scratch grade. All the electro-polished surface treatment samples of AISI 304L to HDPE were tested by shear test which indicated that the samples were failed by shearing the polymer layers at the lap joint region. Figure 7 and Table 4 illustrates the resulted data from the shear force. The maximum and minimum shear force values were obtained in the specimens No.7 and 1 respectively.

![Figure 6. shear forces of the scratched surface specimens](image)

Table 3. Parameters of stainless steel to HDPE joint of scratched surface treatment.

| Sample No. | pressure (bar) | time (min) | temp. (°C) | scratch grades |
|------------|----------------|------------|------------|----------------|
| 1          | 4              | 1.5        | 115        | p100           |
| 2          | 4              | 2          | 120        | p220           |
| 3          | 4              | 2.5        | 125        | p320           |
| 4          | 4              | 3          | 130        | p600           |
| 5          | 6              | 1.5        | 120        | p320           |
| 6          | 6              | 2          | 115        | p600           |
| 7          | 6              | 2.5        | 130        | p100           |
| 8          | 6              | 3          | 125        | p220           |
| 9          | 8              | 1.5        | 125        | p600           |
| 10         | 8              | 2          | 130        | p320           |
| 11         | 8              | 2.5        | 115        | p220           |
| 12         | 8              | 3          | 120        | p100           |
| 13         | 10             | 1.5        | 130        | p220           |
| 14         | 10             | 2          | 125        | p100           |
| 15         | 10             | 2.5        | 120        | p600           |
| 16         | 10             | 3          | 115        | p320           |
| 17 (Optimum condition) | 4 | 1.5 | 130 | p600 |
Figure 7. shear forces of the electro-polished surface specimens

Table 4. Parameters of stainless steel to HDPE joint of electropolished surface treatment.

| Sample No. | pressure (bar) | time (min.) | temp. (°C) |
|------------|----------------|-------------|------------|
| 1          | 4              | 1.5         | 115        |
| 2          | 4              | 2           | 120        |
| 3          | 4              | 2.5         | 125        |
| 4          | 4              | 3           | 130        |
| 5          | 6              | 1.5         | 120        |
| 6          | 6              | 2           | 115        |
| 7          | 6              | 2.5         | 130        |
| 8          | 6              | 3           | 125        |
| 9          | 8              | 1.5         | 125        |
| 10         | 8              | 2           | 130        |
| 11         | 8              | 2.5         | 115        |
| 12         | 8              | 3           | 120        |
| 13         | 10             | 1.5         | 130        |
| 14         | 10             | 2           | 125        |
| 15         | 10             | 2.5         | 120        |
| 16         | 10             | 3           | 115        |
| 17 (Optimum condition) | 4   | 1.5       | 130        |

3.2. Design of experiments (DOE) analysis

Effect of the joining parameters for the lap joint of scratched surfaces on the joint shear force was analysed by the design of the experiments using the MINTAB program and presented in figure 8. The main effect plot showed that shear tensile force exhibited a maximum value at 4 bar pressure, 2.5 minute
time, 120 °C temperature and p320 scratch grades. The Pareto chart showed that the joining time has the highest effect on the shear tensile force of lap joint as compared to other parameters.

Effect of the joining parameters for lap joint of electro-polished surfaces specimens was presented in figure 9. The main effect plot showed that the shear force have the maximum value at 6 bar pressure, 2.5 minute time and 120 °C temperature. The Pareto chart showed that the temperature has the greatest effect on the shear force of joint, while the processing time exhibited the minimum effect on the shear force of this type of joint.

![Figure 8. Main effects plot and Pareto chart for scratched surfaces joining parameters.](image1)

![Figure 9. Main effects plot and Pareto chart for electro-polishing surface joining parameters.](image2)

3.3. Scanning electron microscope (SEM) and optical microscope test

A microstructure inspection was used to investigate the type of interface between the two materials AISI 304L and HDPE. SEM test was achieved to determine the thickness of the joining layer between the two materials and explain the behavior of the joining mechanism at the interface. A mechanical interlock mechanism was the basic joining mechanism between the polymer and metal without cavities. The average thickness of the interlock line between the AISI 304L with HDPE for types of scratched surfaces and electro-polishing surface were 5 and 4 µm respectively as shown in Figure 10 and 11. Hence, the bonding mechanism was achieved by a Vander Waals force [27]. The higher generated temperature and applied pressure resulted in built in the two materials along the interface line without gaps. There were no cavities founded in the joined line which indicate that all the molten polymer was penetrated through the scratched and electro-polished surface of AISI 304L with a good efficiency. The SEM images indicated to a two bonding reaction between the molten polymer and the electro-polished
surface of AISI 304L: adhesive and mechanical interlock. The bonding mechanism of the joint was occurred due to the combination of the chemical, physical and mechanical interlock. The chemical interaction was occurred between the carbon atoms of the polymer and the oxide film of metal. A Vander Waal forces represents the physical phenomena.

Figure 10. SEM images of the optimum condition for scratched surfaces AISI 304L – HDPE interfaces at (a) 10 µm magnification (b) 50 µm magnification.

Figure 11. SEM images of the optimum condition for electro-polishing surface AISI 304L/ HDPE interfaces at (a) 50 µm magnification (b) 10 µm magnification.

The microstructure of the joined specimen AISI 304L/HDPE at 130°C with a scratched grade p600. Two boundary conditions were tested, Pressure: 4 and10 bar, time: 1.5 and 3 min. as shown in figure 12. The heat affected zone (HAZ) was clearly observed in each section for the joints of scratched surfaces. As well as, the resoldificated polymer was clearly observed. The thickness of the joined line was increased by decreasing both of the applied pressure and processing time.

The cross section microstructure examination of the joints for the electro-polished AISI 304L/HDPE was shown in Figure 13. The images indicated that the joining between polymers and the metal was achieved by a mechanical interlock between the molten polymers and porous of metal surface. The thickness of the interface line was increased by increasing the applied pressure and time.
3.4. Energy Dispersive Spectroscopy (EDS) analysis test

EDS line scan was carried out to determine the elements distribution across the joint of the stainless steel to polymer bonding. The EDS examination exhibited three different elements, Iron (Fe), Carbon (C) and oxygen (O). Figure 14 and 15 show the EDS spectra line scan across the base material and the bonding zone which explained the element identification of metal/HDPE joints for type of scratched and electro-polished surfaces respectively. The presence of oxide film and the carbon atoms of polymers showed the chemical bonding between them in addition to the mechanical interlocking and Vander Waals forces. It clearly observed that the iron element was found at the region of stainless steel and fall down through the joined zone until reach the a zero value which stabilized at this value through the polymer region. The joined zone exhibited a uniform increasing in the carbon content (polymer) starting from the edge of stainless steel towards the polymer edges. Approximately, the oxide layer of the stainless steel has a constant value through the zone of polymer and metal. The oxide layer represented the zone of the electro-polished layers of the AISI 304L as shown in Figure 15.
Figure 14. EDS map line scan of scratched surfaces AISI 304L to HDPE joint (a) Iron (b) Carbon (c) Oxygen.
4. Conclusions

AISI 304L sheets were joined with HDPE polymer using a hot press process. Two types of the surface treatment were achieved on the surface of stainless steel. The following conclusions were summerized:

The hot press technique was a successful method to join different materials; AISI 304L with HDPE polymer in the case of metal surface treatment (scratching and electro-polishing). The SEM examination of joint cross indicated that the joining process between the metal and polymer was achieved.
by mechanical interlock between the molten polymer and metal surface. Electro polishing of AISI 304L surface increased the pores size and rearranged its distribution which resulted in penetrate the molten polymer through it homogenously. The EDS examination indicated that the interaction line between the two materials contained three elements: O, C and Fe. The shear test indicated that all the tested specimens were failed by shearing the polymer layers at the lap joint. The applied temperature was the highest effective parameter on the shear strength of joint type (AISI 304L- electro polishing / HDPE). The scratch grade was the lowest effective parameter on the shear strength of joint type AISI 304L- scratch / HDPE. The processing time exhibited the lowest and highest effect on the shear force of joint AISI304L- electro polishing / HDPE and AISI 304L- scratch / HDPE respectively. Increasing the applied pressure resulted in decreasing the shear force of decreasing the shear force of joint type AISI 304L- scratch / HDPE.

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