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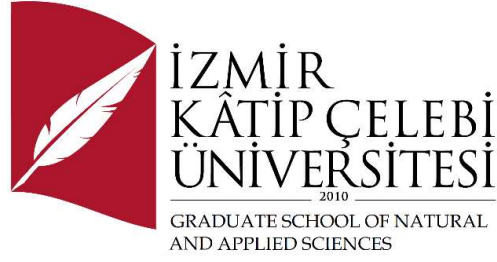
Improvement of Interface in PVC and Aluminium Structures

Department of Material Science and Engineering
Doctoral Thesis

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- Where I have consulted the published work of others, this is always clearly attributed.
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Improvement of Interface in PVC and Aluminium Structures

Abstract

There is a search for innovative hybrid products that combine different material properties in windows, doors and many other products used in buildings. PVC windows, which are widely preferred in buildings, are formed by the combination of more than one component, such as polyvinyl chloride (PVC) based profiles and steel sheets providing mechanical reinforcement.

In this study, the joining techniques of two different materials such as PVC and Aluminium, and hybrid joining, which can replace steel insertion for such applications, were investigated. The aim was to improve the interface structure for the joining of PVC and Aluminium, and the effects of mechanical surface roughness and additional processes were investigated. With the surface roughening processes carried out in different geometries on the aluminium surface, some additional processes such as pre-treatments, adhesive, plasma or emulsion PVC were applied to the PVC and aluminium hybrid joining structure. Aluminium samples with mechanically deformed surfaces were prepared and joined with PVC strips. Lap-shear, interlaminar shear strength (ILSS), 3P bending and coefficient of linear thermal expansion (CTE) tests were performed. Microstructural investigations were performed with optical microscope, scanning electron microscope and atomic force microscope.

According to the results obtained, it was determined that the mechanical surface roughness on the aluminium improved joining interface between PVC and aluminium. While the side punched and perforated samples achieved the best results in terms of geometrical variations on aluminium surface, the hybrid samples, which included pre-treatment and adhesive in the added processes, showed a considerable increase in

values. These improvements resulted in a 2-fold increase in lap-shear shear strength and 45 percent reduction in 3-point bending test results. The added process and surface treatments indicated a general positive behavior (47 percent higher) in interlaminar shear stress testing, with the best outputs obtained in samples with a deformed aluminium surface and samples with pre-treatment and adhesive application in terms of the process.

The influence of adhesion on the interface, as well as mechanical processes and process changes done on aluminium, have been measured to diminish the value significantly (about 0.15 mm/°C) in the coefficient of linear thermal expansion calculations.

On the interface of the samples, optical microscopy, SEM and AFM analyzes were performed and the cavity structures were examined. In the samples with good adhesion results, it was observed that the desired locking mechanism was formed in the hybrid structure, thanks to the abrasion on the metal surface and the filling of the holes with PVC.

According to the results obtained in this study and the Design of Experiment (DOE) analyzes made by considering different variables, it is possible to launch innovative products that can be turned into applications by help of the connection techniques developed for PVC and aluminium.

Keywords: PVC, aluminium, bonding, surface roughness, deflection, hybrid, process

PVC-Alüminyum Yapılarında Ara Yüzeyin İyileştirilmesi

ÖZ

Yapılarda kullanılmakta olan pencere, kapı ve diğer bir çok üründe, farklı malzeme özelliklerinin bir araya getirildiği yenilikçi hibrid ürün arayışları yer almaktadır. Binalarda, yaygın olarak tercih edilen PVC pencereler, polivinil klorür (PVC) esaslı profiller ve mekanik güçlendirme sağlayan çelik saclar gibi, birden fazla komponentin bir araya gelmesi ile oluşmaktadır.

Yapılan çalışmada, bu tür uygulamalar için profile çelik sac takma işleminin yerine geçebilecek, PVC ile alüminyum gibi iki farklı malzemenin birleştirilme teknikleri ve hibrid bağlantılar araştırılmıştır. Amaç, PVC ve alüminyumun birleştirilmesi için, ara yüzey yapısının iyileştirilmesi olup, mekanik yüzey işlemleri ve ilave proseslerin etkileri incelenmiştir. Alüminyum yüzeyine farklı geometrilerde gerçekleştirilen yüzey pürüzleme işlemleri ile PVC ve alüminyum hibrid birleştirme yapısına ön işlemler, yapıştırıcı, plazma veya emulsiyon PVC gibi bazı ek prosesler uygulanmıştır. Yüzeyleri mekanik olarak deforme edilen alüminyum numuneler hazırlanmış ve PVC şeritler ile birleştirilmiştir. Bindirmeli bağlantılarda kayma testi, 3 nokta eğme, kısa kiriş eğme testi (ILSS), ve ısı genleşme katsayısı (CTE) belirleme testleri yapılmıştır. Optik mikroskop, taramalı elektron mikroskobu ve atomik güç mikroskobu ile mikroyapısal incelemeler yapılmıştır.

Elde edilen sonuçlara göre, alüminyum yüzeyindeki mekanik pürüzlenmesinin PVC ve alüminyum arasındaki yapışmayı önemli ölçüde iyileştirdiği tespit edilmiştir. Alüminyum yüzeyinde yapılan geometrisel değişimlerde en iyi sonuçlar yandan kesikli ve delikli numunelerde elde edilirken, eklenen prosesler içinde ise primer ile ön işlem ve yapıştırıcının kullanıldığı hibrid numunelerin değerlerinde önemli bir

iyileşme olduğu belirlenmiştir. Bu modifikasyonlar ile bindirmeli bağlantılardaki kayma mukavemetinde yaklaşık iki kat artış, üç nokta eğme test sonuçlarında yer alan sehim değerlerinde yaklaşık % 45 azalma sağlanmıştır. Gerçekleştirilen kısa kiriş eğme testlerinde de, ilave edilen proses ve yüzey işlemlerinin genel bir olumlu davranış (% 47 artış) sergilediği gözlemlenmiş olup, alüminyum yüzeyi için deforme edilen numune ile proses açısından primerlenerek ön işlem ve yapıştırıcı uygulaması yapılan numunelerde en iyi çıktılar elde edilmiştir.

Isıl genleşme katsayısı hesaplarında, ara yüzeydeki yapışmanın etkisi ve alüminyumda yapılan mekanik işlemler ve proses ilaveleri ile genleşme katsayısı değerinin, olumlu yönde yaklaşık 0.15 mm/°C azaldığı görülmüştür.

Numunelerdeki ara yüzey üzerinde, optik mikroskop, SEM ve AFM analizleri gerçekleştirilerek, boşluk yapıları incelenmiştir. Yapışma sonuçları iyi çıktılar sağlayan numunelerde, metal yüzeyinde aşınma ve deliklerin PVC ile dolması sayesinde, hibrit yapı içinde istenen kilitleme mekanizmasının oluştuğu görülmüştür. Bu çalışmada elde edilen sonuçlar ve farklı değişkenler göz önüne alınarak yapılan DOE analizine göre, PVC ve alüminyum için geliştirilen bağlantı teknikleri sayesinde, uygulamaya dönüşebilecek yenilikçi ürünlerin pazara sunulması olasıdır.

Anahtar Kelimeler: PVC, alüminyum, bağlanma, yüzey bozma, sehim, hibrit, proses



To my family..

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List of Abbreviations

AFM	Atomic Force Microscope
ABS	Acrylonitrile-butadiene-styrene terpolymer
ACE	Acrylate polymethacrylate copolymer
CaCO ₃	Calcium Carbonate
CFRP	Carbon Fiber Reinforced Plastic
CPE	Chlorinated Polyethylene
DOE	Design of Experiments
DSC	Glass transition temperature
E-modulus	Elastic Modulus
EDC	Ethylene Dichloride
EMI	Electromagnetic Interference
EVA	Ethylene-vinyl acetate copolymer
E-PVC	Emulsion PVC
FML	Fiber Metal Laminates
HCl	Hydrochloric acid
HDPE	High Density Polyethylene
ILSS	Interlaminar Shear Strength
LDPE	Low Density Polyethylene
MS	Modified Silane
MBS	Methacrylate-butadiene-styrene terpolymer
OM	Optical Microscope

PET	Polyethyleentereftalaat
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl Chloride
PU / PUR	Polyurethane
SEM	Scanning Electron Microscopy
SiO ₂	Silicone Oxide
TiO ₂	Titanium Dioxide
TPE	Thermoplastic Elastomer
UV	Ultraviolet
VCM	Vinyl Chloride Monomer

List of Symbols

A	Atomic Weight [gram/mol]
CTE	Coefficient of Linear Thermal Expansion [$\text{m}/\text{m}^\circ\text{C}$]
F	Force [N]
k	Thermal Conductivity [Watt/mK]
m	Mass [kg]
P	Pressure [bar]
t	Time [min]
T	Temperature [$^\circ\text{C}$]
U_f	Thermal transmission of frame [Watt/ m^2K]
U_w	Thermal property of window [Watt/ m^2K]
x_i	Particle Size [μm]
η	Dynamic Viscosity [mPa.s]
σ	Surface Tension [mN/m]
ρ	Density [g/cm^3]

Chapter 1

1. Introduction

Plastics play an essential role in our lives since they are used in many different applications, including packaging, films, coverings, bags, and containers, as well as building, electrical, and electronic applications [1]. In 2012, 74.2 percent of European plastic demand was met by LDPE, HDPE, PP, PVC, PS, and PET (Figure 1.1).

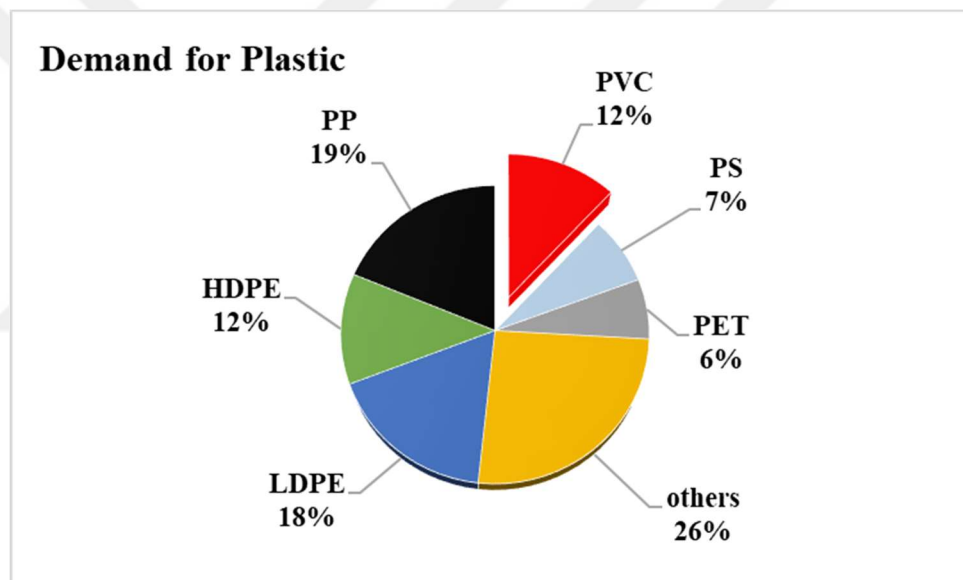


Figure 1.1: Demand for plastic in Europe by category (PlasticsEurope, 2013).

The material polyvinyl chloride (PVC) has a strong and lightweight structure that is resistant to weathering, chemical corrosion, and abrasion. It is resistant to fire. As a result, PVC is widely employed in the construction sector for a variety of purposes, including pipelines, windows, flooring, roofing, and lighter constructions. The building industry consumes 67 percent of PVC production [2] (Figure 1.2).

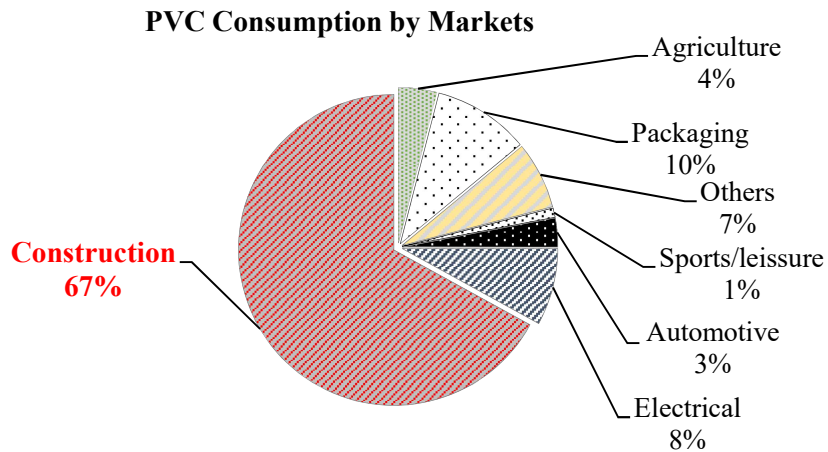


Figure 1.2: PVC consumption by market (Source: Solvin)

Because of PVC's combination of versatile performance and low price, PVC will continue to grow at about $7 \pm 3\%$ for the decade. The variability will result from new production capacity relative to world economies. The areas of highest growth are in global construction activities and export to developing nations for all applications [3].

The consumption of Polyvinyl Chloride (PVC) worldwide is expected to grow from around 47 million metric tons in 2019 to approximately 51 million metric tons by 2021 that is shown in Figure 1.3 [4].

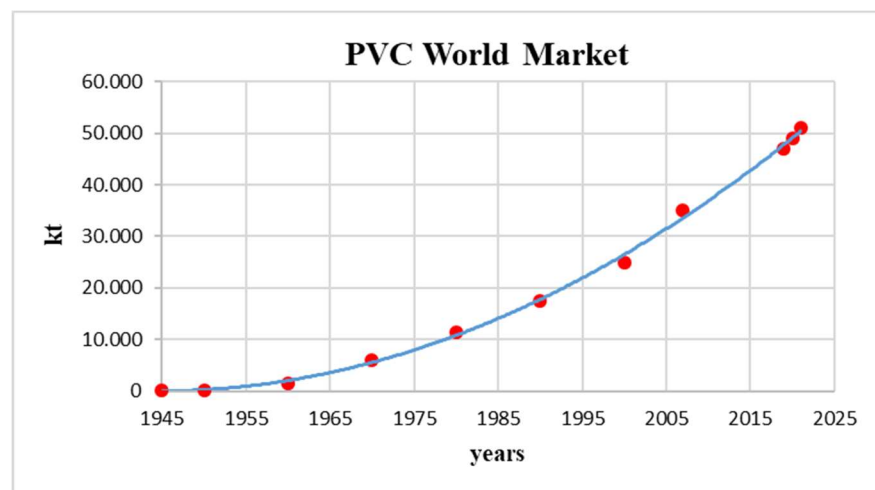


Figure 1.3: PVC world market

World PVC production capacity have more than doubled over the past couple of decades. In 1992, 22 million metric tons of PVC were manufactured, but by 2021 this is predicted to rise to 51 million metric tons, according to petrochemical consultants CMAI. The leading five global PVC manufacturers – Shin Etsu Group, Formosa Plastics Corporation, Solvay, Ineos and Oxy Vinyls [5].

The average annual growth rate of PVC has been 3.3% seen in the past eight years likely to be surpassed in future. According to forecasts PVC demand to increase at an average annual rate of 3.9% over the next years. With a roughly 53% share of global consumption, Asia-Pacific is the largest PVC consumer, followed by North America and Western Europe (Figure 1.4).

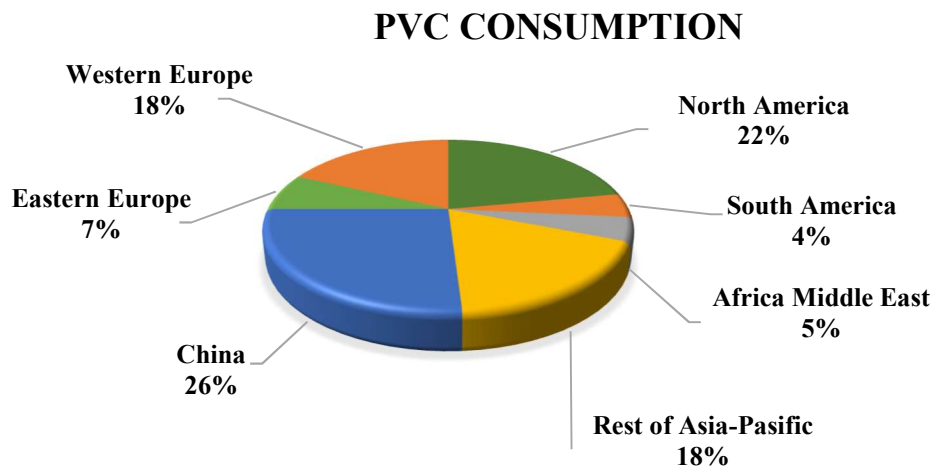


Figure 1.4: PVC consumption by region (Source: CMAI & Solvay)

Windows are available in a variety of frame materials in the construction industry, including aluminium, polyvinyl chloride (PVC), timber and cover of aluminium or PVC on timber, hybrid and composite and fiberglass. The market shares of windows with frames composed of wood, aluminium, PVC, and other materials are shown in Figure 1.5. Some observations concerning these building materials are relevant to an understanding of the PVC requirements [6].

According to market information, PVC segment is accounted more than 80 % of the windows and doors market share in 2020. Thermal properties advantage of PVC windows are main reason of having this high percentage.

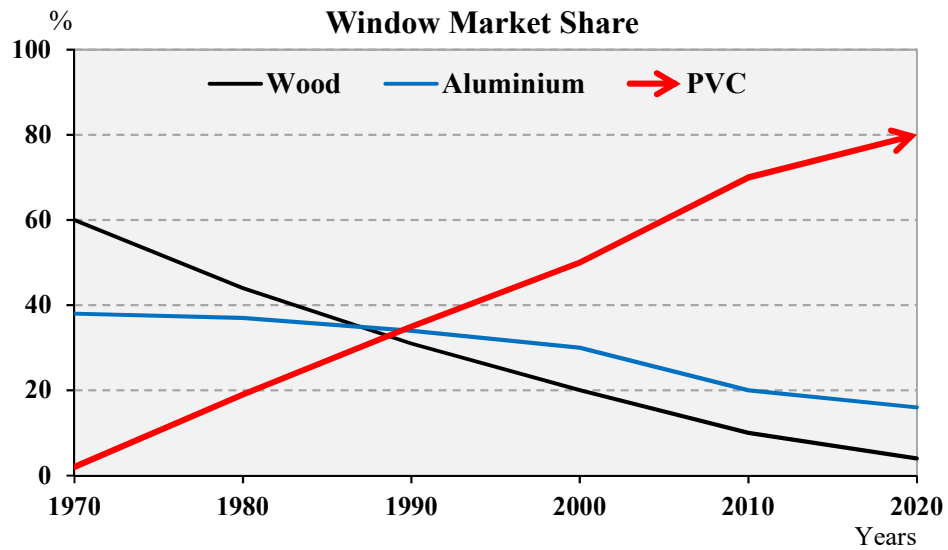


Figure 1.5: Window market distribution in years

PVC windows are strong and long-lasting. Increased new development and replacement of existing windows are driving up demand for new windows. The increasing demand for PVC windows among consumers across the globe, as they are cost efficient and provides better thermal insulation, is stimulating the PVC window profile market growth. Additionally, rising consumer preference towards more durable and cost-efficient windows is again fueling the market growth [7].

In comparison to wooden or metal frames, UPVC is also utilized to make window and door frames since it is more energy-efficient and durable. The demand for energy conservation is heightened by a lack of supply and rising energy expenditures. As a result, enhancing the thermal insulation of buildings, particularly windows, is critical [8].

PVC profiles are fastened to aluminium surfaces in various architectural designs. The aluminium profiles have projecting fins that fit into slots in the PVC profiles, which can be solid PVC or cellular PVC. The corners of the PVC profiles are mitered, but they are not glued together and do not contribute to the joint's strength. These are essentially aluminium windows with a beautiful PVC surface that aids in energy efficiency. In alternative designs, a so-called 'thermal barrier' is created by using twin aluminium profiles separated by a rubber or flexible thermoplastics gasket glued to the

metal to reduce condensation induced by the high thermal transmission of aluminium. Another concept involves injecting polyurethane foam into hollow aluminium profiles.

A distinct combination of PVC and aluminium is employed for a different purpose. Colored aluminium covers are used to coat PVC frames for an improved color range: in some designs, the PVC is the structural component, and the aluminium is just there to launch a colored surface usually external to the building [9].

1.1. PVC

Polyvinyl chloride, is a common synthetic material which is made up of the elements chlorine, carbon and hydrogen. Chlorine atoms in the monomer of PVC material are connected to carbon atoms by covalent bonds, resulting in strong bipolar interactions between the chains consisting of these monomers. PVC is a polymer that is resistant to the effects of acids and bases, and its density ranges from 1.39-1.42 g/cm³, and the density of the material depends not only on the polymer but also on additives.

PVC resin has almost 51-57% chlorine. The remaining is made up of hydrogen and carbon, both of which come from fossil fuels/mainly natural gas and petroleum [10 , 11].

1.1.1. PVC Production

As seen in Figure 1.6, PVC is typically made from natural gas or oil and sodium chloride (salt). Chlorine is created by electrolysis of a salt solution, whereas ethylene is produced by a 'cracking' process of oil or gas. The interaction of ethylene and chlorine produces ethylene dichloride, which is then further cracked to yield vinyl chloride monomer. At room temperature and pressure, vinyl chloride, also known as vinyl chloride monomer-VCM, is a gas.

Hydrochloric acid is produced as a byproduct of the conversion of EDC (ethylene dichloride) to vinyl chloride. The molecules are then linked together to form a sludge, which is dried and centrifuged to generate PVC in the form of a white powder.

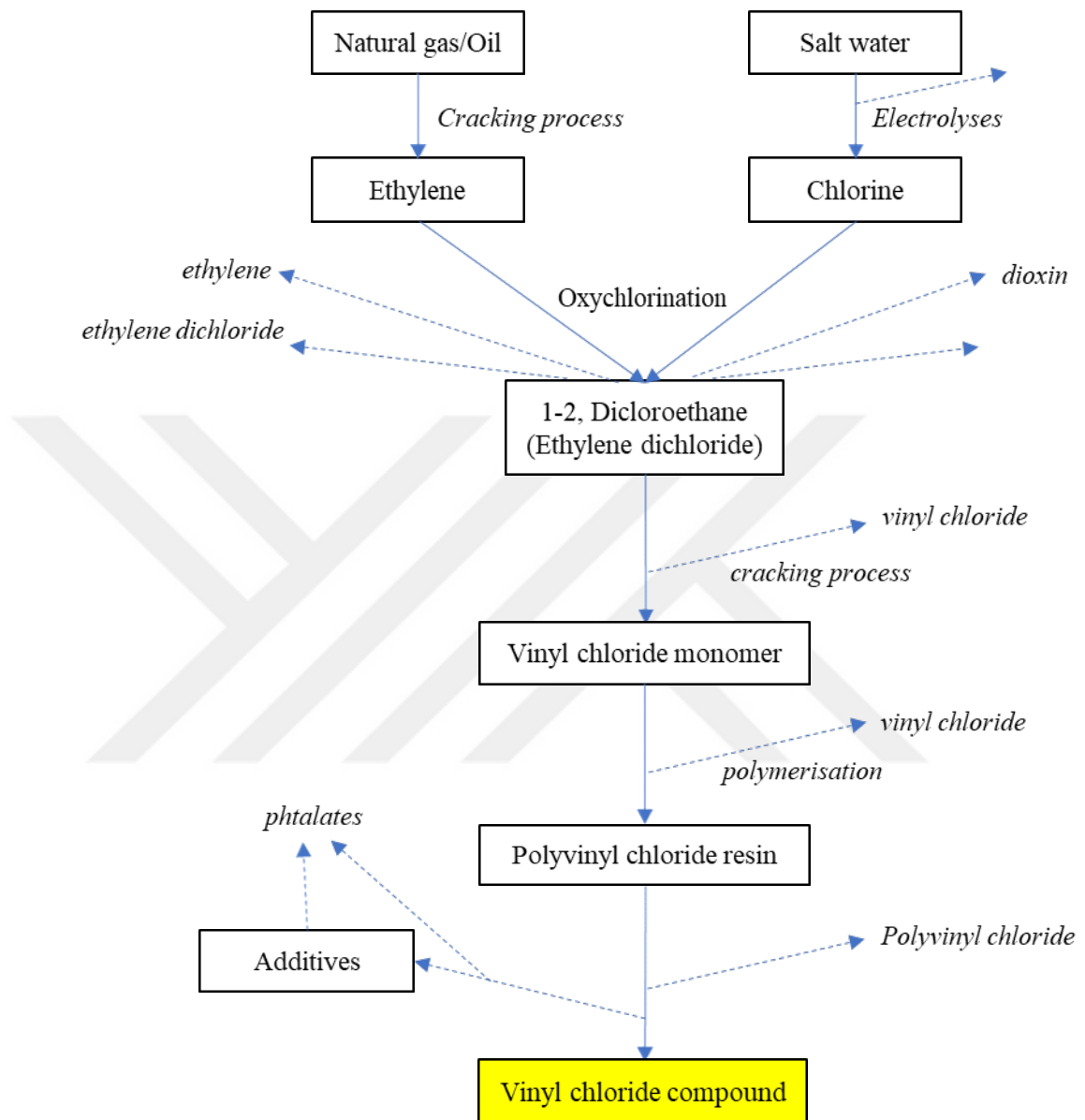


Figure 1.6: Production process of PVC [12]

There are four fundamental vinyl chloride polymerization procedures, each of which employs one of the four processes listed below: Suspension polymerization, emulsion polymerization, bulk polymerization, and solution polymerization are the four types of polymerization [13].

Suspension polymerization is the most common method for producing PVC resins, accounting for roughly 82-85 percent of overall production. PVC homopolymer is produced when only VCM is reacted. A PVC copolymer is created when a monomer, for example vinyl acetate, is combined with vinyl chloride. The productivity improvement per unit reactor volume, flexibility in terms of polymer composition and resin particle properties, and granular nature of the result are all advantages of the suspension resin process. [13].

Emulsion polymerization is the second most common method for producing PVC resins, accounting for 10-12 percent of total production. Water and emulsifier are used in the emulsion polymerization process, and control of the polymerization reaction is considerably easier than in the bulk method and is comparable to the suspension process.

Bulk polymerization is the third most important method for the production of PVC resins in terms of volume, but it only accounts for around 5% of the total. In this method, vinyl chloride monomer and initiator are charged into a first-stage polymerizer, where roughly 10% of the monomer is transformed to polymer. The simplicity of the bulk process, the consistency of resin particle size, the high porosity of resin particles, and the purity of the polymer are all advantages.

Solution polymerization is a Union Carbide Corporation-exclusive method that accounts for around 2% of total resin production. A continuous reactor system is fed with vinyl chloride monomer, comonomer, solvent, and initiator [14].

1.1.2. PVC Dryblend Compound-Additives

PVC windows are high-tech items that can only be commercially successful if the design and compound production processes are coordinated. Only the development of unique polymers, additives, and compound compositions allowed for the required performance of final PVC window profiles [9].

Many different types of additives can be used in PVC formulations to help impart a wide range of chemical and physical properties. PVC's adaptability is one of the key reasons for its commercial success as a thermoplastic [15].

Plastic additives are compounds that are placed throughout polymeric materials in order to influence their behavior during processing and improve the desired attributes of end products with little or no change in the polymer's molecular structure. Catalysts, initiators, cross-linking agents, and other fundamental chemical modifications to the polymer structure are not included in this description [16].

Additives are categorised based on their impact on fundamental properties for polymeric materials as well as their current practical applications. That idea permits additions to be divided into two groups:

1. Additives which change the plastics physical properties are included in the first group. Plasticizers, lubricants, and mold-release agents, as well as macromolecular modifiers, reinforcing fillers, reinforcing agents, and coupling agents, colorants and brightening agents, blowing agents, and antistatic agents, are among them.
2. The second group of additives contains those that protect polymers against aging and breakdown. Heat stabilizers, antioxidants, UV protective agents, flame retardants, and biocides are examples of antidegradants [17].

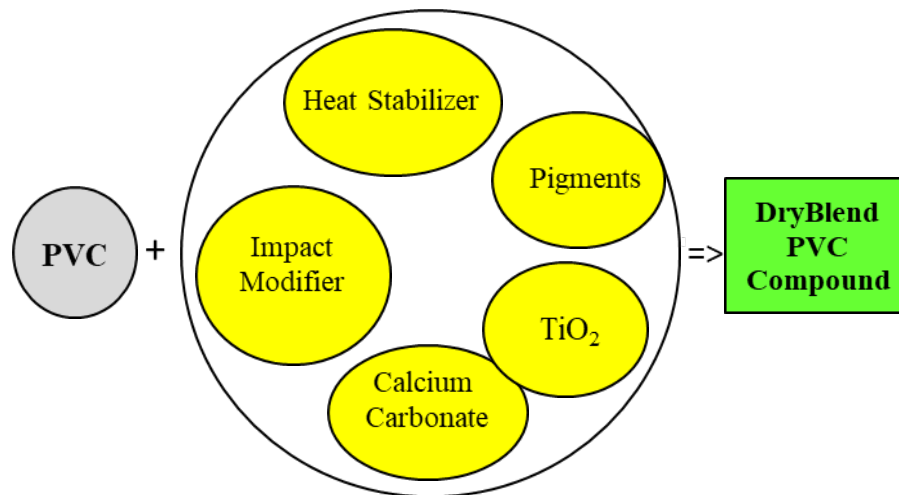


Figure 1.7: Content of PVC dryblend compound

The main additives to produce dryblend PVC compound are heat stabilizer, impact modifier, calcium carbonate, titanium dioxide and other pigments as shown in Figure 1.7.

PVC formulations contain a significant variety of additives that perform critical tasks during manufacturing and regulate the end product's performance attributes [18].

Compound which is called as dryblend , has production process in 2 steps as heating and cooling. Heating is performed at 120°C, and cooling is done at 40 °C temperatures(Figure 1.8).

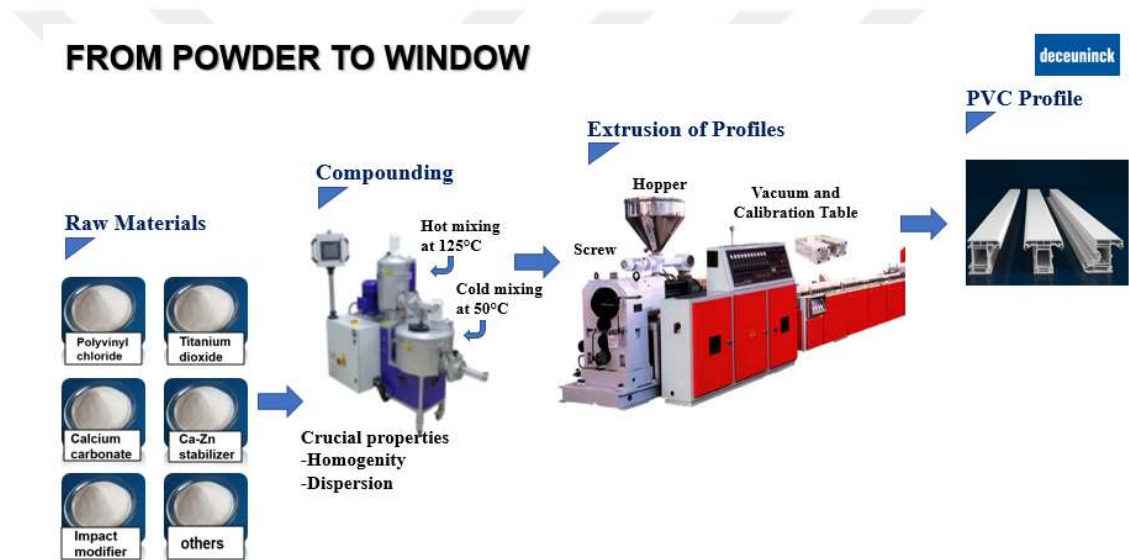


Figure 1.8: Processes of PVC compound to PVC window (Courtesy of Ege Profil)

PVC's unique polar property allows a large variety of additives to be added into the polymer. The most common types of additives and the main range of physical and chemical effects are shown in Table 1.1.

Table 1.1: Additives and physical / chemical effects

Additives	Physical / Chemical Effects
Stabiliser	Process Characteristic
Impact Modifier	Softness/ Hardness
Lubricant	Stiffness/Flexibility
Filler	Clear/Opaque
Colourant	Electrical Properties
Plasticiser	Fire Properties
Flame Retardant / Smoke suppressant	Density
Blowing agent	Cost
Processing Aid	Ductile / Brittle
Antistatic agent	Gloss/Matt
Viscosity Modifier	Chemical Resistance
Biostabiliser	Stability
Antioxidant /UV Absorber	

Thermal Stabilisers

Due to the thermal instability of PVC, a thermostabiliser is the most essential additive within a formulation. This improves its resistance to discolouration during processing, and helps maintain physical properties of the end product during service. It is fairly well established that thermal degradation leads to evolution of hydrogen chloride, development of colour (in a clear compound colour may progress from light yellow, to reddish brown to black in severe cases) and deterioration in physical, chemical and electrical properties.

Fillers

There are a number of different filler types available to the PVC compounder. The main ones are calcium carbonate and the silicates. Calcium carbonate is prepared either directly from calcite mineral or by precipitation from solution. The precipitated fine and ultrafine calcium carbonate variants offer superior performance in Some applications need good weathering and impact resistance on the outside. (such as the window profile market), particularly if stearic acid is incorporated [19].

Calcite (CaCO_3) additive is widely used in building materials, electrical cable coatings, food industry, automobile industry, building materials today. SiO_2 and glass fiber additives; they increase mechanical strength, especially wear resistance, provide Dimension stability, especially used in the production of precision measurements and plastic products that will work for a long time. It also reduces thermal conductivity. Additives that reduce product cost, such as CaCO_3 filling, increase mechanical strength and reduce thermal conductivity unless they are used excessively.

Types of calcite are talc, waste leather particles, wollastonite, aluminium fiber, aluminium hydroxide, antimony trioxide, calcium carbonate, carbon black, carbon fiber, hollow glass beads, kaolin, magnesium hydroxide, mica, sand, silica, silica carbide, wood fiber, wood flour, zeolite.

In all polymers, including PVC, calcite is the most extensively used filler. Milled, precipitated, and precipitated with a surface coating are the three main grades available. The milled grade has a mineral origin, and its quality is determined by the mineral composition as well as the milling effect on particle size distribution and shape. Because of its low price, this is the most commonly utilized grade. Because of its low packing density, it has a high plasticizer need in the plasticized composition.

The other significant disadvantage stems from its chemical structure, which lacks any groups that could interact with PVC; as a result, its use frequently reduces the product's mechanical performance. Smaller particles and a narrow distribution characterize precipitated grades. They can be customized to increase packing density, which minimizes the need for plasticizers.

Abrasion resistance, cost reduction, EMI shielding (metal and carbon fibers), electric conductivity (metal fibers, carbon fibers, carbon black), electric resistivity (mica), flame retarding properties (aluminium hydroxide, antimony trioxide, magnesium hydroxide), impact resistance improvement (small particle size calcium carbonate), improved radiation stability (zeolite), increase of density, increase of flexural modulus, impact strength, and stiffness (talc), thermal stabilization (calcium carbonate), are some of the reasons for their use (aluminium oxide, silica carbide, wollastonite).

Impact Modifiers

Unmodified PVC-U has relatively poor impact strength, i.e. poor toughness at and below room temperature. The addition of an impact modifier boosts its performance greatly.

Impact strength, air mark, chemical resistance, coloring ability, filler dispersion, easy thermoforming, good dispersion, high output, jetting, low friction, low crease whitening, low melt viscosity, low temperature applications, optical clarity, quick fusion, reduced flow marks, surface finish, uniformity of foamed cell, weather resistance are some of the reasons for its use.

The following are some examples of the various types of impact modifiers;

- Acrylonitrile-butadiene-styrene terpolymer(ABS),
- Methacrylate-butadiene-styrene terpolymer(MBS),
- Chlorinated Polyethylene(CPE),
- Ethylene-vinyl acetate copolymer (EVA)
- Acrylate polymethacrylate copolymer(ACE)

All of the above exhibit limited compatibility with PVC. It is believed that the impact improvement is achieved via the interruption of the homogeneous morphology of U-PVC into a heterogeneous structure.

Colourants

PVC compositions usually contain a colourant either in liquid form, as a dye, or, in solid form as a pigment. The criteria for a PVC colourant should include the following: good dispersability, thermal stability and lightfastness, as well as compatibility with other additives.

Antraquinone, azo, carbon black, diarylide yellow, dosazo, fluorescent, metallic flakes, monoazo orange, naphthol red, optical brighteners, perylene, phthalocyanine, quinacridone, titanium dioxide, zinc oxide, and others are examples.

Coloring and UV protection are two reasons for using carbon black and titanium dioxide.

Titanium dioxide – UV absorbers

Because it provides UV protection, titanium dioxide (TiO_2) is the most important pigment for PVC outdoor products. It is crucial to ensure that titanium dioxide is in the rutile form and has a surface coating tailored to reduce its catalytic influence on PVC degradation when used for practical applications such as coloring or UV protection. Zirconium and/or silica, as well as aluminium, are commonly used in these coatings. Coated rutile should be utilized in Table 1.2 since titanium grades are evaluated according to their photoactivity [20].

Table 1.2: Effect of composition of TiO_2 on its photocatalytic activity [21]

Pigment description	Photoactivity index, %
70/30 anatase/rutile (photocatalyst)	100
Anatase	47
Anatase with Al/Si coating	2.2
Rutile with Al coating	3.1
Rutile with Al/Zr coating	1.9
Rutile with Al/Si coating	0.62
Rutile with Al/Si/Zr coating	0.76
Rutile with Al/Si coating (exterior UPVC)	0.38

Although particle additives are often used in polymer materials to reduce cost, the mechanical, thermal, electrical and chemical properties of the composite materials produced are improving with the use of superior particulates. For example, by adding particles such as glass balls, wear-resistant polymer composites can be produced. Silicon dioxide filled PVC is used in household appliances, building materials, automobile industry, electrical industry with its low density.

1.1.3. PVC Extrusion

The extrusion process transforms a solid plastic feedstock material into a molten viscous fluid, which is then transformed into a completed solid or flexible film product. A plasticizing extruder, die assembly, cooling assembly, and hauloff or winding equipment make up extrusion equipment. In contrast to moulding, which is a cyclic process, extrusion is a continuous process. Many sorts of continuous plastic goods that have a uniform exterior shape and can be coiled, sliced, or wound can be extruded. The mechanical shearing action of a rotating screw and the heat generated by electrical resistance heaters fastened to the outside of the extruder barrel and die turn a solid plastic feedstock material into a molten viscous fluid in the extruder barrel. The solid plastic feedstock is transformed into a hot molten material by the combination of the screw's mechanical, spinning shearing action and the heat from the electrical heaters [22].

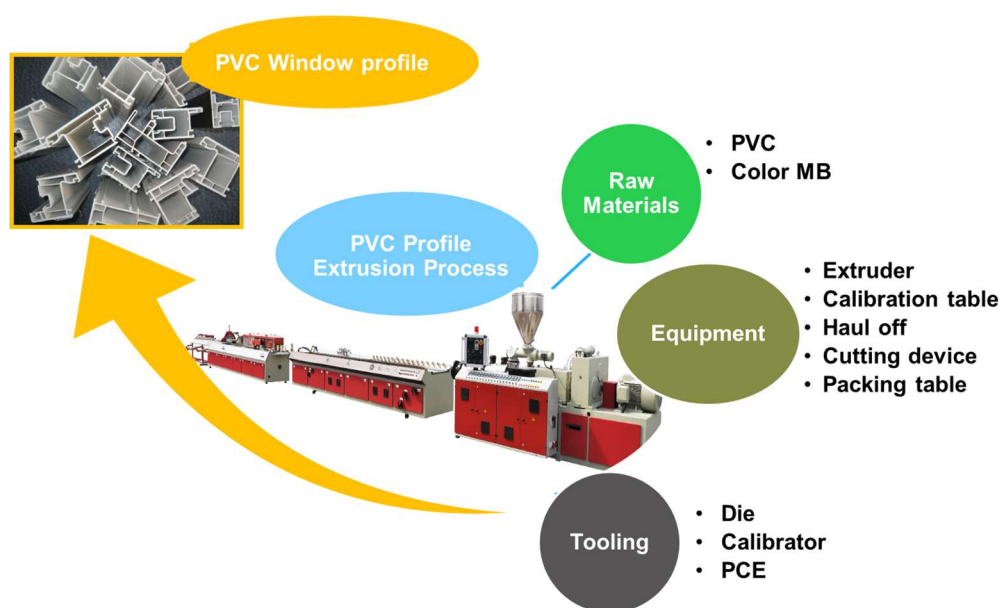


Figure 1.9: Extrusion process of PVC profile

The dryblend compound is fed into extruder which has process temperature as approximately 190 °C, and calibers for cooling. Then, depending on tool shape, PVC window profiles are extruded (Figure 1.9) as 6 meter lengths.

1.1.4. PVC Applications

PVC (polyvinyl chloride) is one of the most widely used plastic compounds in the world, with a wide range of applications. Different additives, such as organic or inorganic fillers, can be used to change the mechanical, physicochemical, and processing properties of PVC. Unplasticized Polyvinyl Chloride (UPVC), often known as stiff PVC, has piqued construction industry attention as a low-maintenance material [23].

Table 1.3 gives an overview of the general applications of PVC. During the manufacture of PVC [24], a variety of additives are used to improve the mechanical qualities of the material and expand its applications [25].

Table 1.3: General applications of PVC [25]

General Application	Specific Examples
Construction	Window frames and doors; Portable and gravity pipes; Gutterings; Cables ducting and conduits; Flooring; Wall coverings; Reservoir linings; Sheets and panels; Roller shutters; Sports stadium seats
Medical	Pharmaceutical blisters; Blood bags; Plasma bags; Tubing; Infusion kits
Electrical	Keyboards; Computers; Power tools; Electrical cords; Phone systems; Cellular phones; Component housings
Automobile	Interior trims (sun visors handbrake and gearbox lever parts carpets etc.); Window encapsulation; Dashboard skins; Coated fabrics (seat coatings door panels etc.); Wire harness systems (cable sheaths grommets etc.); Sealant (underbody insulation etc.)
Packaging	Food packaging (thermoformed cups); Mineral water bottles; Cling films; Non-food packaging (trays cosmetics and detergents containers etc.); Bottle caps
Cards	Credit cards; Smart cards; Identity cards; Telephone cards
Leisure and Sports	Toys; Footballs; Swimming rings; Garden hoses; Liferafts; Ship construction
Office	Office supplies (folders ring binders rulers etc.); Computer cases; Computer keyboards; Printing applications
Clothing	Raincoats; Lifevests; Shoe soles; Rubber boots; Imitation leather

PVC is commonly used in construction market as window frames and doors, cables, flooring and shutter applications. Windows are among the most sensitive parts in a building envelope, and they are significant not only for their effects on the interior environment, but also for the structure's energy performance, due to their multi-disciplinary role. The frame and the glazing unit are the two main components of a window. The fixed frame and the sash are the two elements of the frame. The material used to make the frame determines not only the physical features of the frame, such as thickness, weight, and durability, but it also has a significant impact on the window's thermal performance. The frame qualities have a considerable impact on the overall performance of the window because the sash and frame account for 10 to 30% of the total area of the window unit.

Based on good thermal properties, PVC window frames have big advantages to have more energy saving in buildings. Normally, 25-30 % of the heat loss in the building occurs through windows and doors which is high ratio. Mainly, PVC windows consist of 3 different materials as PVC frame, steel reinforcement and glass as shown in Figure 8. The chambers in frame profiles, provide better thermal property to window. The geometry of PVC profile and steel reinforcement defines thermal transmission of frame (U_f -Watt/m²K) . Depending on window sizes, the thermal property of window (U_w -Watt/m²K) is calculated with thermal transmission of frame (U_f), glass (U_g) according to ISO EN 10077-2 [26], EN 12412-2 [27].

The components of window section are PVC frame, sash profiles, steel reinforcements, TPE gasket (Thermoplastic Elastomer), glazing bead and glazing (Figure 1.10).

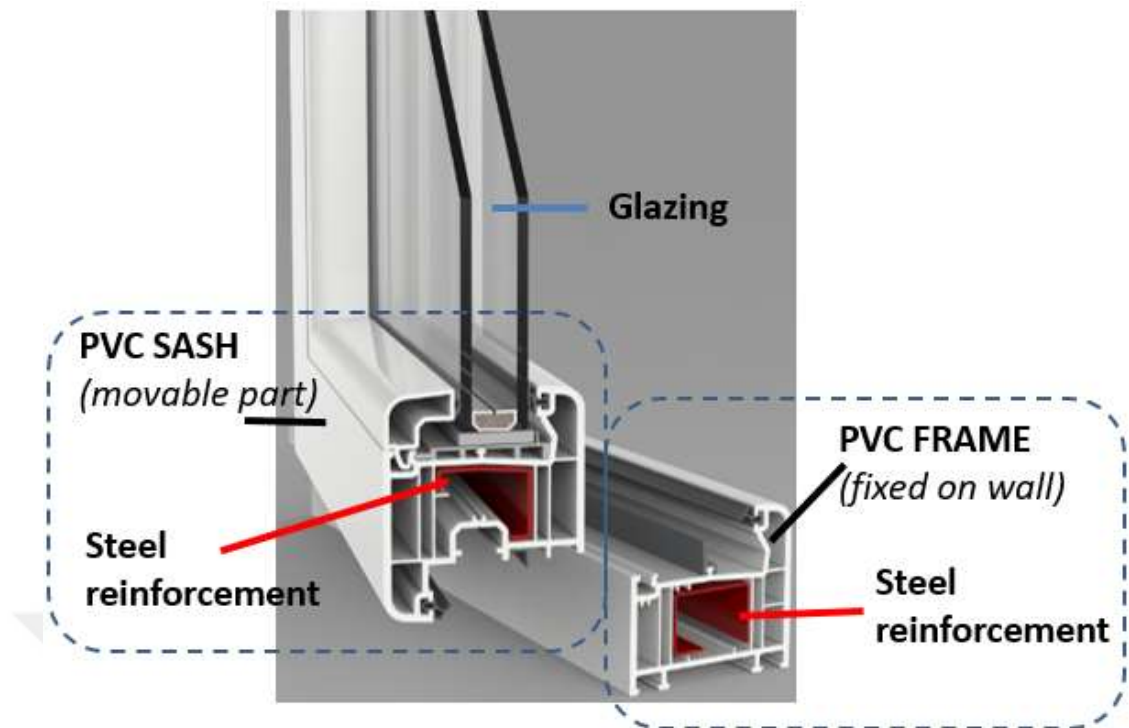


Figure 1.10: PVC frame and steel reinforcement combination in PVC window.

1.2. Aluminium

Aluminium is a metal that cools easily and absorbs/transmits heat, especially in the cooling industry, because it is cheaper and easier to process than copper because it is an alternative material, it is widely used. General properties of aluminium are summarized below : Of the Earth's crust 7.5%-8,1 abundant with a rate of aluminium, atomic number is 13 and in the third group of the periodic table, Al, icon, color 2 matte silver, usually in a pure form in nature as there is not any metal that is found in bauxite ore. The atomic diameter is $1.43 \text{ \AA}$, the ion diameter is $0.86 \text{ \AA}$ and the atomic weight is 26.97 g/mol . It has a surface-centered cubic structure, is found in a solid state under normal conditions, and the density of aluminium, the lightest metal after magnesium and beryllium, is 270 kg/m^3 . Melting point is $660.32 \text{ }^\circ\text{C}$, boiling point is 2519°C . Its thermal conductivity is as high as 237 W/m-K .

Due to their favorable properties, such as high strength-to-weight ratio, ease of fabrication, high degree of workability, considerable ductility, excellent thermal conductivity, high corrosion resistance, and attractive appearance in their natural

finish, the use of aluminium alloys as structural material has increased in recent years. The building industry today consumes 25% of worldwide aluminium production [28].

In advanced industries, aluminium extrusion technology is still the subject of research and its new applications are being discussed in various fields. In the construction and automotive sectors, small machine parts, load-bearing profiles and especially in the aerospace industry, the use of aluminium extrusion products and the demand for these products has increased significantly, and competition in this sector is quite high [29]. As of today, the extrusion industry has a history of more than a hundred years. In terms of physical, chemical and mechanical behavior, aluminium is also a valuable material in the metal class, such as steel, bronze, copper, zinc, lead or titanium.

Properties of Aluminium Alloys

Aluminium is a metal that is usually not used in a pure state and is used by alloying. Because the mechanical properties of pure aluminium are poor; it is soft and about three times lighter than iron. Its density decreases due to the increasing degree of purity. For example, at 20°C ambient temperature, the specific gravity of 99.25% pure aluminium is 2727 kg/m³, while the specific gravity of 99.75% pure aluminium is 2703 kg/m³. While its density increases very little when alloyed, there are significant increases in its mechanical strength. An important feature of aluminium is its electrical conductivity.

Wrought and cast aluminium alloys are the two types of aluminium alloys. The former is made up of alloys that have been melted in a furnace and then poured into molds, whereas the later is made up of alloys that have been treated solidly. Heat-treatable and non-heat-treatable aluminium alloys can be classed based on the strengthening operating conditions. The Aluminium Association Inc. divides wrought alloys into nine series using a four-digit method, with each series containing different alloying addition combinations. The first digit (Xxxx) denotes the main constituent alloy, whereas the second digit (xXxx) denotes the original alloy's changes. The last two digits (xxXX) are arbitrary numbers that can be used to identify a specific alloy in the series. As a result, the material qualities might change, providing a variety of application alternatives. In terms of structural response, research on aluminium alloys has primarily focused on wrought alloys, particularly the 5xxx and 6xxx series, which

are the most appealing for structural engineering applications because to their mechanical properties. In order to provide more information about the manufacturing treatment, the alloy classification is followed by the temper indication. The temper designation is made up of five basic tempers: F, O, H, W, or T, with extra digits indicating further information about the manufacturing treatment [30 , 31].

5052 alloy of aluminium belongs to the family of 5xxx series which has magnesium as the major alloying element. Aluminium alloy 5052 is a non-heat treatable alloy and hardened for higher strength by cold work. Alloy 5052 has excellent characteristics with a high fatigue strength it is used for structures which are subject to excessive vibrations [32 , 33].

Composition specification of aluminium alloy H5052 is described in Table 1.4.

Table 1.4: Composition specification of aluminium alloy H5052 [32]

Alloy	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Others	
									Each	Total
5052	0.25	0.40	0.10	0.10	2.2- 2.8	0.15- 0.35	0.10	-	0.05	0.15

The properties of 5052 aluminium-alloy sheet are given in Table 1.5, which are specified for flat rolled product(plate, sheet and coil) in ASTM B209M [34].

Table 1.5: The properties of 5052 aluminium-alloy sheet based specified thickness[34]

Temper	Specified thickness mm		R _m MPa		R _{p0.2} MPa		Elongation min %	Hardness HBW
	over	up to	Min	Max	Min	Max	A50 mm	
H12	0.2	0.5	210	260	160	-	4	63
	0.5	1.5	210	260	160	-	5	63
	1.5	3.0	210	260	160	-	6	63

1.2.1. Aluminium Applications

Aluminium and aluminium alloys have many outstanding attributes that lead to a wide range of applications, including good corrosion and oxidation resistance, high electrical and thermal conductivities, low density, high reflectivity, high ductility and reasonably high strength, and relatively low cost [35].

Aluminium can be used in a wide range of products, from food, chemistry, construction industry to household goods and decoration materials used in daily life.

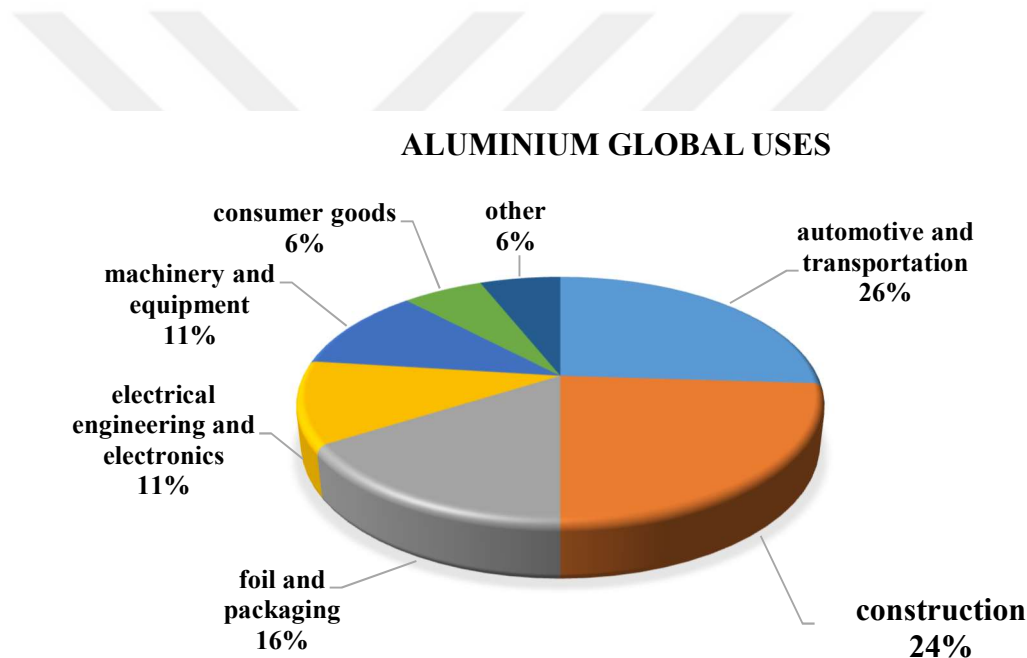


Figure 1.11: Aluminium global uses, 2019 (www.nrcan.gc.ca)

The pie bar graph in Figure 1.11 shows the major global uses of aluminium in 2019. The largest use was for automotive and transportation (26%), followed by construction (24%), foil and packaging (16%), electrical engineering and electronics (11%), machinery and equipment (11%), consumer goods (6%) and other applications (6%) [36].

CONSTRUCTION	OTHER MARKETS
WINDOW FRAMES	CAR BODY PANELS
CURTAIN WALLS	TRUCK & TANKER BODY PANELS
GUTTERS & PIPES	COMPUTER CHIPS
ROOFINGS	ENGINES
AWNINGS	

Figure 1.12: Aluminium application examples in construction and other markets

Aluminium is widely acknowledged as one of the most energy-efficient and environmentally friendly building materials. With aluminium's durability, high strength-to-weight ratio, design flexibility and contributions to energy savings, it is the material of choice for architects and designers. There are many parts in different markets that prefer aluminium in their products as car body panels, window frames, engines, tanker and truck body panels, computer chips, ladders (Figure 1.12).

There are also some parts (Figure 1.13) in building that are made from aluminium such as door frames, siding, curtain walls, window frames, roofs and entire facades.

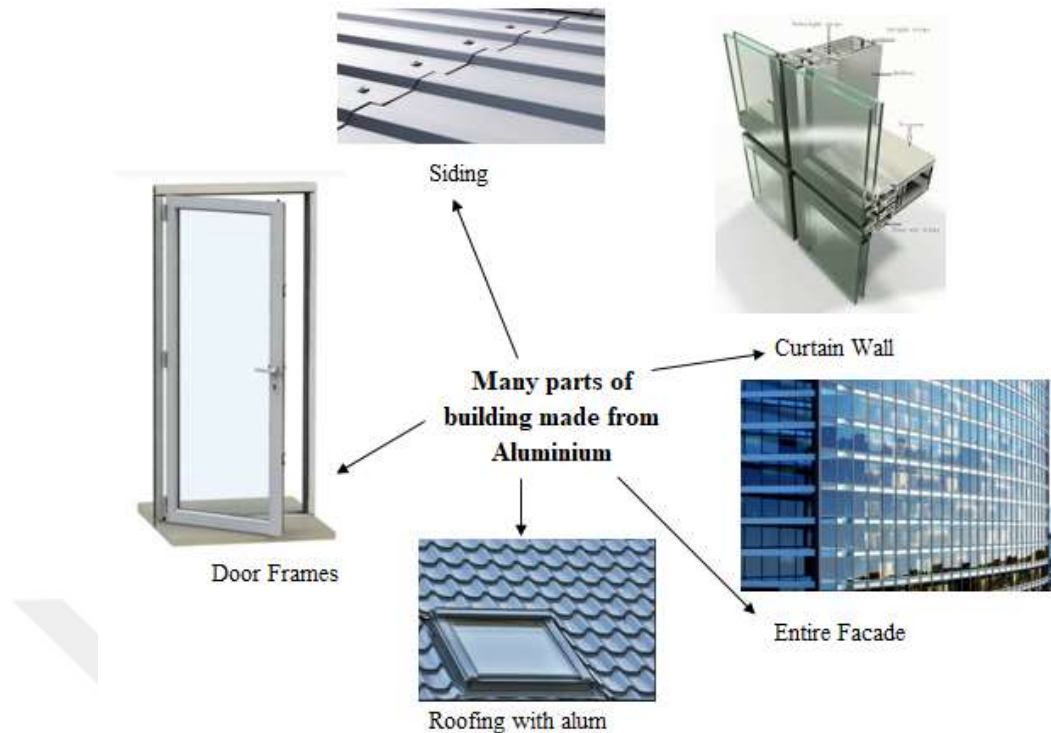


Figure 1.13: Parts of building made from aluminium

Extruded aluminium profiles are used to make ladders, doors, windows, showers, and scaffolding, among other industrial applications. Roofs, facades, panels, components, awnings, cladding gutters and downpipes, ceilings, and many more structures in the building and construction business use our coated and uncoated building goods [37].

Aluminium windows are one of the most widely used products on the market. The corner section of aluminium window has been described in Figure 1.14.



Figure 1.14: Frame section of aluminium window

The comparison of steel and aluminium are provided below;

- Unlike steel, aluminium is not magnetic and does not spark when struck [38],
- Aluminium is resistant to corrosion even when uncoated [39],
- Aluminium is ideal for applications at extremely low temperatures (it does not brittle) [40],
- Elements made of aluminium are more prone to fatigue than those made of steel, temperature makes aluminium contract and expand twice as much as steel, but the increase of stress induced by limited displacement is less (lower modulus value),
- Due to its lower modulus value, aluminium is more resistant to impulsive loads, which means that aluminium alloy constructions may absorb more deformation work and attenuate oscillations.
- Fire insulation materials for aluminium alloy constructions must be effective between 175 and 350 °C, have a low density, and have a low heat conductivity.

1.3. Hybrid Materials

Nowadays, multimaterials assemblies are widely employed into an increasing number of applications to exploit their different characteristics e.g. mechanical, thermal and physical behavior as well as aesthetical appearance. Because of the great difference among metals, polymers and composite materials, the adoption of the proper joining process is often a challenging issue to conciliate different requirements including mechanical behavior, productivity, low environmental impact, cost, automation, etc. [41].

Materials or material combinations that are difficult to join, either because of their unique chemical compositions or because of considerable disparities in physical qualities between the two materials being joined, are referred to as hybrid materials. [42]. Different joining techniques have unique strengths and limits for the joining of hybrid materials, and different joining procedures have unique strengths and limitations for the joining of hybrid materials [43]. Chemical, thermal, and physical incompatibilities (linear thermal expansion, ductility, fatigue/fracture mechanics,

elastic modulus, and so on) can cause issues with the joining process as well as the structural integrity of the joints during the product's use phase [44].

Definition: Hybrid Materials

Hybrid materials can be defined as materials with new properties created by the formation of new electron orbitals between two or more materials [45]. Many diverse systems, such as crystalline highly ordered coordination polymers, amorphous sol–gel complexes, and materials with and without interactions between the inorganic and organic units, are referred to as hybrid materials. Before the discussion of synthesis and properties of such materials we try to delimit this broadly-used term by taking into account various concepts of composition and structure in Table 1.6. The most wide-ranging definition is the following: a hybrid material is a material that includes two moieties blended on the molecular scale [46].

Table 1.6: Different possibilities of composition and structure of hybrid materials [46]

Matrix:	crystalline $\Leftrightarrow$ amorphous
Building blocks:	organic $\Leftrightarrow$ inorganic
	molecules $\Leftrightarrow$ macromolecules $\Leftrightarrow$ particles $\Leftrightarrow$ fibers
Interactions between components:	Strong $\Leftrightarrow$ weak

In general, hybrid materials are made of a casting of basic material, which is the component with the highest percentage weight fraction compared to other components, as well as other elements and compounds to strengthen the basic material and improve its qualities [47]. Aluminium and its alloys are widely used compared to other metal composites. Aluminium and its alloys allow for the use of a variety of reinforcement and enhancement materials. In comparison to materials without reinforcement, the main advantage of hybrid materials with aluminium as a foundation is:

- increased strength,
- increased stiffness,
- decreased density (weight),
- improved high-temperature characteristics,
- controlled coefficient of linear thermal expansion,

- controlled material heating
- increased and adaptable electrical conductivity,
- improved abrasion and wear resistance,
- regulated weight (particularly for ICE pistons),
- improved depreciation ability. However, aluminium alloys have a number of drawbacks, including a high coefficient of linear thermal expansion. Hybrid materials are gradually substituting traditional engineering design and building materials. Hybrid materials often outperform homogeneous material formulations in terms of stiffness and strength. Hybrid materials technology has grown at a rapid rate as a result of these driving forces [48].

In fact, one of the most difficult aspects of using hybrid materials is joining them together or with traditional structural components. This problem is compounded by the need to combine materials with anisotropic mechanical properties to materials with isotropic mechanical qualities [49].

Samples of engineering materials are frequently subjected to a wide variety of mechanical tests to evaluate their flexural strength, elastic constants, and various other engineering material properties as well as their performance under a variety of practical use conditions and environments. When joining materials with varied chemical, mechanical, thermal, or electrical properties, however, there are substantial obstacles [50].

1.3.1. Classification of Hybrid Materials

Hybrid materials have been classified in many ways based on different concepts. Hybrids with weak interactions such as van der Waal's and hydrogen bonding or weak electrostatic interactions are categorized as class I hybrids and hybrids with strong chemical bonding between the two components are categorized in Figure 1.15 as class II hybrids [51].

Structurally hybridized materials (composites), materials hybridized in chemical-bond, and functionally hybridized materials are three forms of hybrid materials that can be functionally categorised and characterized [52].

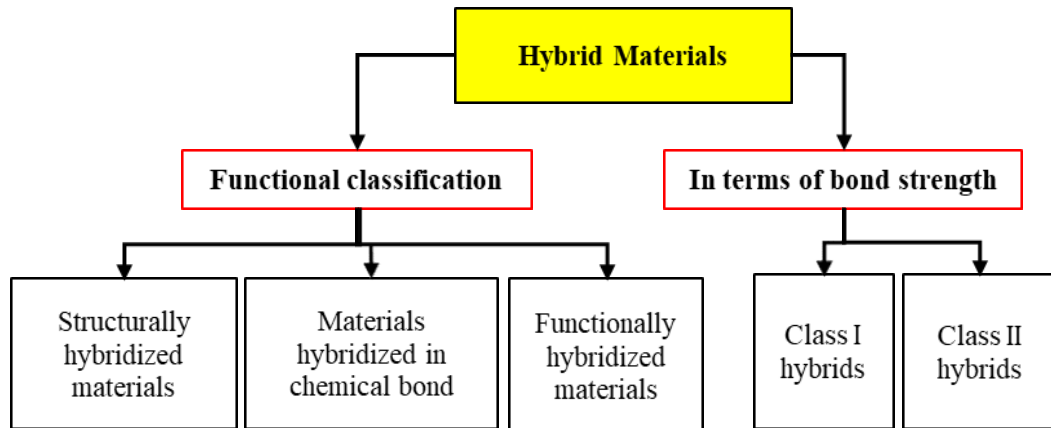


Figure 1.15: Classification of hybrid materials [52]

1.3.2. Hybrid Applications in Building Structures

Hybrid materials combine wood, concrete, and steel to provide a cost-effective and long-lasting solution for building structures, as well as possibilities for bettering building performance and design. Hybrid construction is the use of a variety of materials and techniques to create a variety of structures. A hybrid system will frequently necessitate off-site fabrication of prefabricated pieces. Because the system arrives on site when it is needed during the construction phase, prefabrication speeds up construction and provides for easier installation.

The term "smart materials" is widely used to describe hybrid materials, almost as a synonym. When subjected to external factors such as light, temperature, electrical charge, or any other physical or chemical stimulus, these materials and products undergo reversible change. Smart materials, as defined by NASA, are materials that can "remember" configurations and adjust to certain stimulation. There are numerous examples of these materials currently in use in construction (Figure 1.16). Derivatives created by mixing biological and synthetic components also have hybrid features [53].



Figure 1.16: Hybrid building systems

Hybrid Windows

Windows play a significant role in buildings and are available in a wide range of designs and frame materials. The primary contribution of windows to buildings is to incorporate daylight and to maintain interior environment at desirable comfort conditions. An important aspect of windows is their environmental impact energy consumption, natural resources depletion and environmental burden associated with their manufacture and service life [54].

There are several application types in buildings for windows that include more than one material. Depending on thermal expectations as passive house concept on windows, the hybrid materials can be solution to obtain the requirements. For example, glass fiber reinforced PVC profiles do not include any metal material that has the advantage of lower U_f value Aluminium, PVC, lumber and claddings of aluminium or PVC on timber, hybrid and composite, and fibreglass are only some of the frame materials available. Different frame materials offer their own set of advantages.

In Figure 1.17, the examples of different material used in windows are given as PVC, aluminium and wood.



Figure 1.17: Different material types for windows as PVC, aluminium and wood

1.4. Interfacial Adhesion Promoters

In comparison to other materials such as metals, polymers are known for their low surface energy and adhesive strength. This is due to their thin boundary layers, which are caused by (1) impurities formed during the polymerization process, (2) low molecular weight tails on the surface, (3) additives like antioxidants and slip agents, (4) external processing aids like mold release agents, and (5) post-fabrication contaminants. [55 , 56]. As a result, various treatments are frequently given to polymeric surfaces in order to increase adhesion performance. Mechanical treatments affecting primarily the surface topography (e.g. grit blasting, peel ply), chemical treatments involving chemical substances (e.g. solvent cleaning, primers, ultrasonic cleaning), and energetic treatments affecting primarily the surface free energy (e.g. plasma, corona discharge, flame, UV/ozone) can all be divided into three categories. [57].

Joining structure between interface layers can be performed as surface roughness, adhesive bonding, plasma and emulsion PVC applications. Having surface roughness on aluminium material can be basically more preferable in joining [58].

The underlying mechanisms that account for the success of any surface treatment are a complicated and contentious topic. The following are some of the reasons given for increased bonding performance as a result of a surface treatment:

- improved wetting of low-energy surfaces;
- chemical modification, such as the introduction of polar chemical groups or coupling agents onto the surface, which are then available for bonding;
- removal of 'weak boundary layers' at the surface, such as contaminants, oxidized layers, lowmolecular-weight species, and loose, crumbly surfaces;
- increase in surface roughness giving rise to improved mechanical interlocking or increased bondable surface area [59].

The goal of the surface treatment is to change the chemistry or morphology of a thin surface layer while leaving the bulk properties unchanged. The efficiency of any

surface treatment is determined by the substrate and the treatment's scope. Solvent cleaning, for example, will eliminate surface impurities from some substrates, but the same solvent may interact with the surface and bulk of other substrates, resulting in morphological changes. At modest treatment levels, etching treatments such as acid or laser procedures may produce chemical modification, but at high treatment levels, substantial surface texture changes may occur. Because electrical discharge treatments can create a temperature rise on a surface, surface texture alterations owing to localized melting can occur [60].

1.4.1. Surface Roughness

Material qualities and joint geometry (adhesive thickness, adherend thickness), surface roughness, and other factors influence the mechanical performance of adhesive bonded joints [61]. As a result, in industrial applications, these criteria are critical for achieving maximum strength. These bond strength-related aspects must be taken into consideration when designing adhesively bonded connections. Several studies have looked into the impact of various parameters on the strength and durability of adhesive joints. The nature of the adherends has a significant impact on the adhesive bond's strength [62, 63]. Surface roughness is one of the important factors which influence the mechanical properties of the joints. One of the most important elements influencing the mechanical characteristics of joints is surface roughness. The connection between surface roughness and adhesion is complicated. The importance of the surface and its favorable impact on bond strength was noted by the majority of the studies. Grinding, grit blasting, mechanical etching, plasma, chemical etching, and other surface treatment procedures are available.

Critchew and Brewis (1981) investigated the effect of adherend surface roughness on adhesive bonding joint durability. The grit blasting process was used to generate surface roughness. They discovered that roughness had a considerable impact on the epoxy-Al joint's durability [55].

Surface treatment and roughness adjustment were found to improve maximum lap-shear strength and durability of adhesive bonding junctions. Environmental elements such as temperature, humidity, and other factors, on the other hand, have a significantly greater impact on the surface roughness of wood than they do on a

metallic structural material like aluminium. The lower the surface roughness value (the smoother the surface), the stronger the bond strength of the wood adherend joint [64].

1.4.2. Atmospheric Pressure Plasma

One of the most efficient plasma procedures for cleaning, activating, or coating polymers, metals (e.g., aluminium), glass, recycled materials, and composite materials is atmospheric pressure plasma pretreatment [65].

Treatment with atmospheric plasma is widely employed in a variety of industries. Plasma is created by electrically stimulating a gas to make it highly reactive [66]. This method works well for etching, cross-linking, and activating polymeric surfaces [67].

The effects of plasma on surface are the cleaning of organic contamination from the surfaces, material removal by ablation to increase surface area or eliminate a weak boundary layer, crosslinking or branching to strengthen the surface cohesively, and surface chemistry modification to enhance physical and chemical interactions at the bond strength interphase [68].

There are three types of interactions that can happen between the gas plasma and the treated surface: (1) reactions between the surface species, (2) reactions between the gas species, and (3) reactions between the gas species and the surface chemical groups. (2) plasma-induced polymerization, in which the plasma gas works as a monomer to produce a thin surface layer, and (3) the production of volatile surface products via chemical reactions and physical etching [66]. Low-pressure (vacuum) plasma and atmospheric plasma are the two main types of plasma. Because of its low cost and ease of integration into manufacturing lines, the latter is more often used. The adhesion strength of PE, PET, PA, PVDF, PP, and HDPE was increased by atmospheric plasma treatment in investigations [69].

Plasma technology is widely used in a variety of industries, most notably in the creation of electronic components (semiconductor chip manufacturing). Plasmas are also useful processing tools in the automotive and aerospace industries, as well as in lighting and, more recently, large-screen televisions [70]. Plasma-driven surface treatments have the ability to achieve a variety of surface engineering goals. Controlling the surface chemistry and material characteristics are among them.

Toughness, hardness, adhesion properties, and optical and electrical properties, for example, can all be altered by plasma procedures.

In the plasma process, the surface is exposed to ionized gases, which are normally created in a low-pressure chamber using radio frequency energy. The plasma area has a high concentration of reactive species produced from the gas, such as ions and electrons. These energetic species interact with the surface and produce chemical changes, according to several research. The chemical changes that take place are determined by the gas that was used to make the plasma. If monomers are added to plasma for the purpose of treating a surface, plasma-induced graft polymerization can occur. The monomer is polymerized and bonded to the surface chemically [71].

Non-equilibrium atmospheric pressure plasmas have [72] sparked interest due to their low capital costs, ease of integration into a continuous production line, and capacity to synthesize highly reactive chemical species at ambient gas temperatures [73]. Plasma jets, also known as plasma plumes, are atmospheric pressure gas discharges in which plasma (typically derived from a noble gas) extends beyond the plasma generating zone and into the surrounding environment [74 , 75].

Sputtering is minor in plasma processing at atmospheric pressure, and surface morphological changes are primarily due to reactive species etching. The mixture of ions, electrons, neutral and excited molecules, and photons that coexist in the plasma is responsible for the many and varied plasma applications. In the case of plasma jets, the discharged and ionized argon atoms react with the surrounding air to produce reactive species (primarily atomic oxygen, ozone, and the OH radical) [76]. These highly reactive species can etch and roughen the surface of polymers [77].

1.4.3. Primer and Adhesives

In terms of chemical treatments, the chemical bonding theory, in which covalent chemical bonds are fostered across the contact, is the adhesion theory that is most closely related to this category [56]. This method is dependent on the existence of mutually reactive functional groups in the adhesive and adherends, such as hydroxyl

and carboxyl [78]. Surface cleaning is the most basic chemical treatment, and it eliminates pollutants such as process oils, grime, waxes, mold releasing agents, and plasticizers. Methyl ethyl ketone (MEK), acetone, and methanol are all good cleaning solvents. To avoid degradation, the polymer's chemical resistance should be examined. Primers and adhesion promoters are the most commonly utilized chemical therapies. By establishing a new bi-functional layer at the interface, these multifunctional molecules create a "molecular bridge" between the adhesive and the substrate [79]. Primers, unlike adhesives, have a lower viscosity, allowing them to fill in porous and rough surfaces. Adhesion promoters can be employed on the substrate or in the adhesive formulation, whereas they are exclusively applied to the substrate.

1.4.4. Emulsion PVC

Batch emulsion, continuous emulsion, and micro-suspension polymerisation are all used to make E-PVC. All three methods result in an aqueous latex, which is a very fine suspension of PVC polymer with mean particle sizes ranging from 0.1 to 3 μ m, which is substantially smaller than that produced by suspension polymerisation. When physically agitated in the presence of an emulsifier, liquid vinyl chloride is insoluble in water and disperses to fine droplets. Under the influence of heat, initiators, and/or catalysts, the reaction takes place in pressurized vessels.

When a high level of surface gloss and smoothness is required, E-PVC can be extruded in stiff profiles. However, E-PVC is primarily distributed in plasticiser to generate plastisol. Plastisol is applied or sprayed, then heated to cure it [80].

1.4.5. Combination Processes

Some of the combination processes are applied as Primer +EPVC, Primer +Plasma, Primer +Adhesive to the joining layer.

1.5. Design Of Experiments (DOE)

Design of Experiments (DOE) is a statistical tool used in the development and optimization of numerous types of systems, processes, and products. It's a versatile tool that may be used for a variety of tasks, including design for comparisons, variable screening, transfer function discovery, optimization, and resilient design [81].

DOE has been a very valuable instrument for improving product quality and reliability for the past two decades. DOE has becoming more widely used in various industries as part of the decision-making process for new product development, manufacturing process improvement, and quality assurance. It has been utilized in administration, marketing, hospitals, pharmaceutical, food industry, energy and architecture, and chromatography, in addition to engineering. DOE can be applied to both physical processes and computer simulation models [82].

The design of experiments (DOE) is a structured method for analyzing any scenario in which the answer varies as a function of one or more independent factors. DOE was created to deal with complex problems where more than one variable may influence a response and two or more factors may interact [83].

DOE is a statistical formal methodology that allows an experimentalist to establish statistical correlation between a collection of input variables and a chosen outcome of the system/process under investigation under some uncertainties, referred to as uncontrolled inputs. Figure 1.1 depicts this definition, where $(x_1, x_2, \dots, x_n)$ are the n input variables chosen for study; $(y_1, y_2, \dots, y_m)$ are the m possible system/process outputs from which one should be chosen for analysis; and $(z_1, z_2, \dots, z_p)$ are the p uncontrollable (the experimentalist has no influence) inputs (often referred to as noise) [84].

1.5.1. Terminology in DOE

The terms that are used in DOE have been explained below.

Response: A response is a dependent variable that correlates to the experiment's outcome or effects of interest. One or more response variables may be investigated at the same time.

Factors: A factor is a variable that influences the outcome.

Levels: The chosen conditions of the element under investigation are referred to as levels.

The visualization of DOE terminology, process and its principal stages are explained in Figure 1.18.

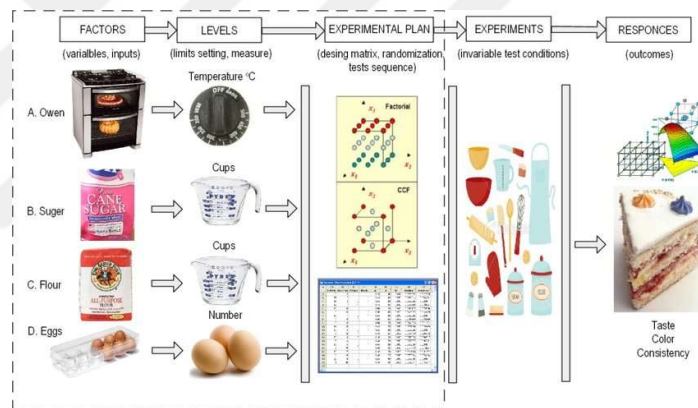


Figure 1.18: Visualization of DOE terminology, process and its principal stages [84]

1.5.2. Full Factorial Design

If the resources are available, a full factorial design is advantageous for a small number of factors. The DOE conceptual approach is discussed for two 22 and three 23 factors, as well as a general 2k factorial design, where k denotes the number of factors and 2 denotes the number of levels. Factor designation is normally done with uppercase letters A, B, C..., whereas treatments are done with lowercase letters. There are two levels for each factor: low (-) and high (+). There are four possible permutations for number 22, eight for number 23, and so on [85].

1.6. The Aim of the Study

In this study, the joining of PVC and aluminium has been investigated using variety of methods including mechanical, chemical and physical processes. According to the findings of available studies, PVC was not studied before in terms of aluminium joining structure. Therefore, the improvement of the interface layer between PVC and aluminium structures have been investigated in this thesis through experimental studies in various processes.

Aim of the study;

It aims to develop on a study that has never been studied before in PVC material by determining the combinations that will increase adhesion strength with aluminium. The overall aim of the thesis is to provide an alternative hybrid material that contains PVC material which is mechanically reinforced. In hybrid material systems, the interlayer, which creates the connecting surface between two materials, has been found to be a critical factor. As an outcome, the improvement of adhesion at the interface has been fully examined.

Objective of the study;

According to performed test results, the effects of surface roughness on aluminium surfaces were evaluated. Also, the adhesion effect on primed surface has been examined on interlayer.

Motivation of the study;

This study, chosen to be developed, was approved as an innovative product in simple and complex geometries, in line with the Tubitak project, which was supported by the state, to be presented to as a potential commercial product.

Chapter 2

2. Materials & Methods

In Chapter 2, the materials, experimental methods and studies on joining process and characterization parts has been explained. In the first part, the materials that include PVC, aluminium, interfacial agents and equipments were described. Secondly, experimental methods for preparation of samples has been identified. Then, experimental studies on joining process such as surface roughness, plasma, primer, adhesive, emulsion PVC and also some combinations of these processes were explained.

2.1. Materials

Hybrid materials are increasingly being used due to its mechanical qualities and lightweight in a range of areas, including military, aircraft, rope, construction, and automobiles. [86] In the study, PVC and aluminium were joined to each other with some additional surface roughness and processes to obtain hybrid material.

2.1.1 PVC

PVC strips have been obtained from suspension u-PVC from Petkim S65 that has the properties given in Table 2.1. These materials were prepared from extruded u-PVC window profiles.

Table 2.1: Properties of the u-PVC material (Petkim S-65)

Material	K value	Bulk density (g/cm ³)	Particle Size (μm)
u-PVC	67±2	0.55±005	0.250 μm – Max 8%
			0.063 μm – Min 90%

2.1.2. Aluminium

The alloy of 5052H18 which is the strongest temper produced through the action of only strain hardening, decided to use for preparation of aluminium strips in the study.

The material properties of used aluminium alloy is described in Table 2.2.

Table 2.2: Material properties of the aluminium alloy 5052 [87]

Material	Alloy	Density (kg/m ³)	Young Modulus (GPa)	Poisson's Ratio	Yield Strength (MPa)
Aluminium	5052	2,700	70	0.33	178

Samples are prepared in required dimensions from aluminium rolls.

2.1.3. Interfacial agents

2.1.3.1. Primer

According to pre-tests in laboratory of Henkel, the product of Technomelt PUR Primer L457 which is used for pre-treatment of aluminium surfaces, produced as polyurethane technology. The application areas for PUR primer priming of aluminium surfaces and it is also used for primer for bonding with PUR hotmelt adhesives.

2.1.3.2. Adhesive

Polyurethane adhesives are found in a variety of applications. To ensure durability and functionality, a polyurethane adhesive is required to possess good adhesive properties and thermal stability. In this study, reactive Hotmelt Adhesive based on Polyurethane was used (Table 2.3).

The product PURFact 121.10 provided by Betakimya is polyurethane based reactive hot melt adhesive includes diphenylmethane-4,4'-diisocyanate.

Table 2.3: Material properties of adhesive

Type	Product	Ingredients
Hotmelt Polyurethane Adhesive	PURFact 121.10	diphenylmethane-4,4'-diisocyanate.

2.1.3.3. Emulsion PVC

In the study, emulsion PVC, polyvinyl chloride water based dispersion Nanovin™ PVC RA007 (PVC and 2-ethylhexyl 10-ethyl-4,4- dimethyl-7-oxo-8-oxa-3,5-dithia-4-stannatetradecanoate) which is considered as nanomaterial, has been used.

The primary particles are solid, smooth surfaced spheres which are clustered into irregular shaped aggregates with a typical mean particle size of 40-50 µm with a range of 0.1-100 µm. Dynamic viscosity of the material is lower than 30 mPa.s [88].

The properties of emulsion PVC used in the study is explained in Table 2.4.

Table 2.4: Properties of Nanovin PVC RA007-Inovyn

Property	Reference standard	Unit	Typical value
Active matter content	Supplier based	%	44
Density	ISO 1183-3	kg/dm ³	1.14
- Before gelation (wet)		kg/dm ³	1.40
- After gelation (dry)			
Mean PVC particle size	Supplier based	µm	0.1
Dynamic viscosity (20 °C)	Supplier based	mPa.s	<30
Surface tension (25 °C)	Supplier based	mN/m	41
Glass transition temperature (DSC)	ISO 11357-2	°C	69

2.1.4. Equipments

2.1.4.1. Hotpress

Hotpress machine includes two metal plates as horizontally on top and bottom with heating resistance to melt the material with pressing 6 bar compression pressure (Figure 2.1). Preheated PVC and aluminium samples have been joined to each other in hotpress process.



Figure 2.1: Hotpress machine

2.1.4.2. Tensile Tests

Tensile properties of the polymer and hybride materials were measured with Zwick Roell kN 020 (Figure 2.2).

Single lap-shear , 3 point bending and ILSS tests were performed in this machine.



Figure 2.2: Lap-shear and ILSS - Zwick Roell kN020

2.1.4.3 Optical Microscope

Interface of section on hybrid samples were analysed with Eclipse LV150N of Nikon as digital imaging combined with advanced optical results (x100) in IKC laboratory. (Figure 2.3)



Figure 2.3: Optical microscope

2.1.4.4. Scanning Electron Microscope (SEM)

SEM analysis of the samples were recorded using SEM- Zeiss 300VP (Figure 20) instrument with micro(10^{-6}m) and nano(10^{-9}m) level resolution can be performed. The powdered native and quaternized samples were coated with gold and images were acquired in the magnification range of 1–5 K.

Scanning Electron Microscope (SEM) device (Figure 2.4) is based on the analysis of the scattered electrons with the help of various detectors as a result of the interaction of the accelerated electrons sent to the sample with the atoms of the sample. In addition, qualitative and quantitative elemental analysis can be performed within the sample, and the distribution of elements can be monitored with the mapping technique.

Carl Zeiss 300VP SEM device, the samples were coated to have more clear image with QUORUM Q150 RES coating device that can be used for more detailed and clearer images on non-conductive samples.



Figure 2.4: SEM Carl Zeiss 300VP and coating quorum Q150 RES

2.1.4.5. Atomic Force Microscope (AFM)

The surface morphology and roughness were measured by atomic force microscopy (AFM, Nanosurf, Flex-Axiom). The device (Figure 2.5) displays the surface properties of materials in 2 and 3 dimensions by scanning in X-Y-Z axes according to scanning tip microscope techniques. Z can also take images at different heights between 100 μm and 5 mm.



Figure 2.5: AFM - Nanosurf flex axiom

2.1.4.6. Heating Oven

The samples are heated with KD400 of Nuve heating oven that is shown in Figure 2.6. KD400 oven is the instrument of choice for routine heating and drying. Temperature range of oven is 70°C / 250°C.



Figure 2.6: Heating oven -Nuve KD400

2.1.4.7. Plasma

Openair-Plasma technology of Plasma Treat company has been preferred in the study. The RD1004 plasma rotary nozzle is used to generate cold plasma (Figure 2.7). This plasma nozzle is suitable for thermally sensitive and geometrically complex surfaces and materials.

The surface was swept several times briefly in a pulse-like manner, which is a very effective form of cleaning and activation – with very low heat input at the same time. Plasma has supplied on surface that has been cleaned of release agents and additives and sterilized (plasma sterilization), while plasma activation allows for later adherence of glues and coatings.



Figure 2.7: Plasmatrete RD2004 atmospheric plasma

2.2. Experimental Methods

2.2.1. Preparation of PVC Samples

The starting materials were PVC sample. First step was compounding production according to PVC window profile formulation with addition of stabilizers, impact modifier, filler, UV resistance and masterbatches. After compounding, PVC profiles that is the origin of the samples were produced in extrusion process. As third step, the strips were prepared from extruded PVC profiles. The thickness of PVC samples were preferred as 3.2 mm.

2.2.2. Preparation of Aluminium Samples

To obtain the patterns on aluminium surface, modelling of different patterns were studied. During this study, the resistance to applied forces (as lap-shear, 3P bending, ILSS and linear thermal expansion) on interface layer has been considered.

Surfaced deformed, which were planned to apply on aluminium surface, have been designed on CAD software. 3D modelling of patterns were created on a jig to have this effect on aluminium surface.

The several geometries of patterns for surface deforming have been studied on cad software. For having surface roughness on aluminium samples, the tool of this pattern which will be transferred to aluminium, has been prepared (Figure 2.8).

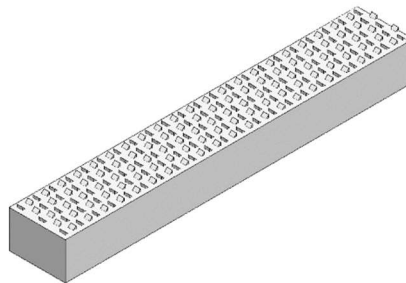


Figure 2.8: 3D model of pattern applied to have surface roughness

This tool was inserted in pressing machine to give the form to aluminium surface for deformation (Figure 2.9).

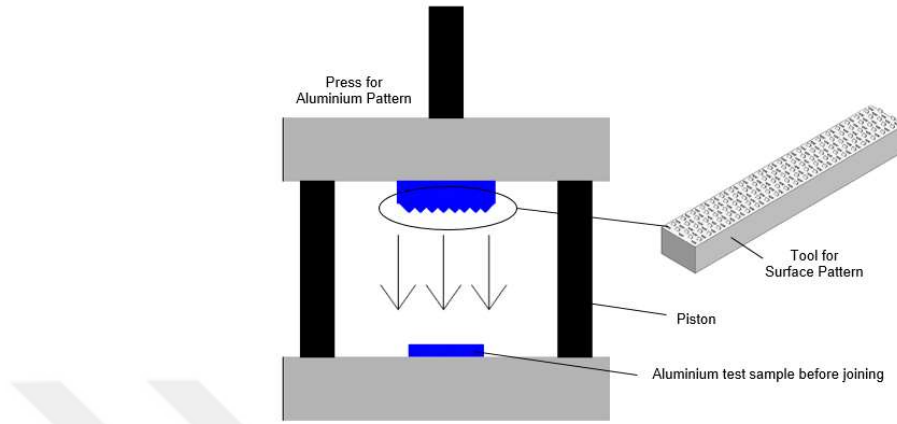


Figure 2.9: Pressing process for having patterned surface on aluminium samples

After pressing process, surfaces of aluminium prepared according to pattern on these jigs. Six different surface types were determined to use in this study. Figure 2.10 and 2.11 show the aluminium samples that are prepared according to flat, surface deformed, perforated, punched, surface deformed punched, perforated punched surfaces. The thickness of aluminium samples were 0.8 mm.

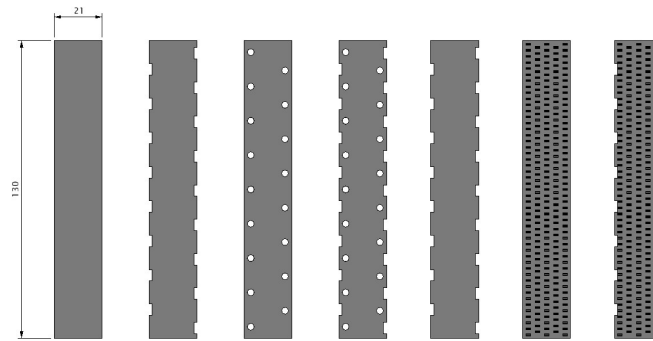


Figure 2.10: Schematic representation of aluminium surface types

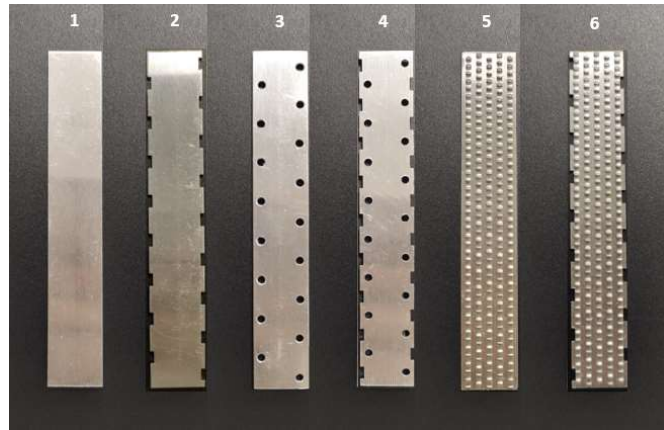


Figure 2.11: Aluminium samples, S1-Flat Surface, S2-Punched, S3-Perforated, S4-Perforated +Punched , S5-Surface Deformed, S6-Surface Deformed+Punched

In the study, the aluminium surface type definitions will be expressed with the abbreviations explained in the Table 2.5.

Table 2.5: Abbreviations of Aluminium Surface Types

#	Aluminium Surface Types	Sample Pictures	Codification
1	Flat		S1
2	Side Punched		S2
3	Perforated		S3
4	Perforated + Side Punched		S4
5	Surface Deformed		S5
6	Surface Deformed+ Side Punched		S6

2.2.3. Preparation of Hybrid Samples as PVC and Aluminium

The joined samples were prepared using hot pressing process. In this method firstly, PVC and aluminium samples were preheated and joined in hotpress machine to obtain hybrid samples.

There are several process types are investigated to apply on aluminium surfaces. Mainly plasma , primer , adhesive, E-PVC were used. Also combination of primer and plasma , primer and adhesive, primer and E-PVC were studied to see the difference on output test results.

Schematic picture of process overview was described in Figure 2.12.

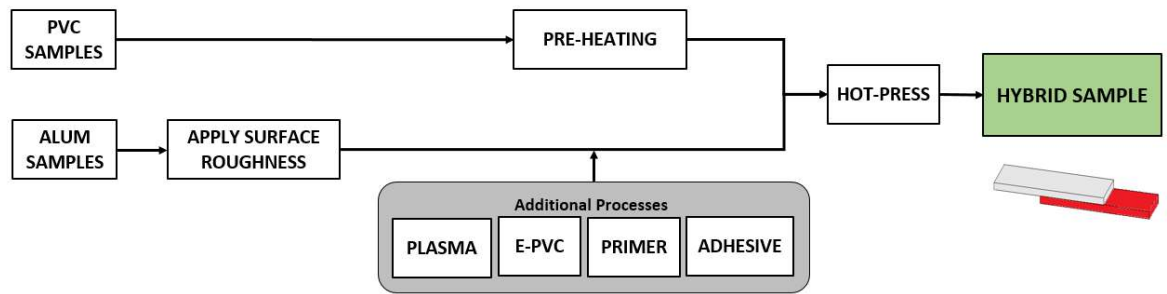


Figure 2.12: Schematic picture of the sample preparation process

180, 190 and 200 °C temperatures have been studied for preheating of rigid PVC samples to optimize the plastification properties. The temperature (180, 190, 200 °C), pressure (6 bar), preheating time (10,15,20 min) and compression time (3, 4, 5 min) were varied to find the optimum joining properties in hotpress machine (Figure 2.13).

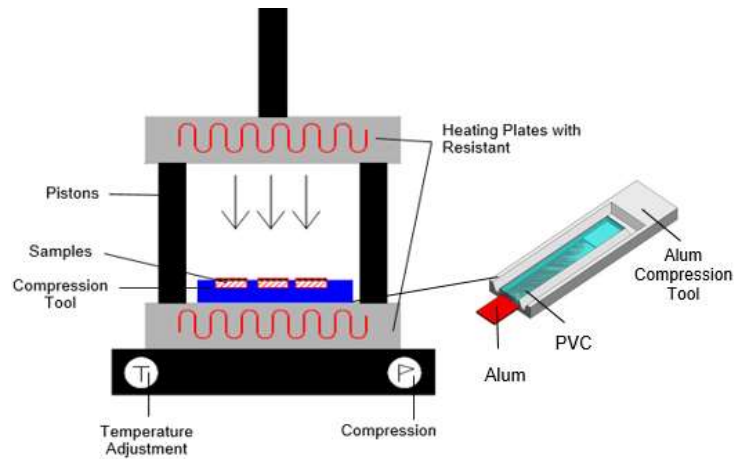


Figure 2.13: Schematic representative of hotpress machine

In these optimization tests, some observations, which were not good, had been obtained for combinations of some parameters as the sample visually burned or not joined. Therefore, at the end, the compression pressing conditions were optimized to 190 °C, 10 minutes for preheating and 3 minutes for compression time.

PVC and aluminium samples were placed on customized tool as shown in Figure 2.14.

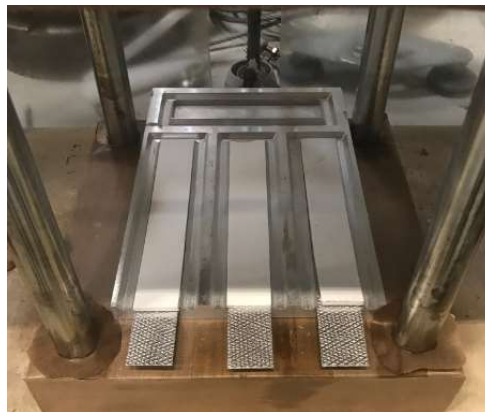


Figure 2.14: PVC and aluminium samples on customized tool

2.3. Experimental Studies on Joining Process

Seven process types were studied. These additional processes are Plasma, E-PVC, Primer, Primer+E-PVC, Primer+Plasma, Primer+Adhesive. The tests which are performed based on processes are described as an overview in Table 2.6. The results of samples were compared with no additional process samples.

The process type definitions on aluminium surfaces were expressed with the abbreviations explained in Table 2.6.

Table 2.6: Abbreviations of process types

#	Process Types	Codification
1	No Additional Process	P1
2	Emulsion PVC	P2
3	Primer	P3
4	Plasma	P4
5	Primer + EPVC	P5
6	Primer + Adhesive	P6
7	Primer + Plasma	P7

Test table for characterization has been prepared to show the overview of process and tests types in Table 2.7. Lap-Shear, 3P Bending, ILSS, CTE, Optical Microscope, SEM and AFM tests are performed for the aluminium specimens (S1,.....,S6).

Table 2.7: Testing methods and characterisation

Codification	Test Table						
P1 (S1,...S6)	Lap-shear	3P bending	ILSS	CTE	OM		
P2 (S1,...S6)	Lap-shear	3P bending	ILSS	CTE		SEM	
P3 (S1,...S6)	Lap-shear	3P bending	ILSS	CTE		SEM	
P4 (S1,...S6)	Lap-shear	3P bending	ILSS	CTE		SEM	
P5 (S1,...S6)	Lap-shear	3P bending	ILSS	CTE	OM	SEM	AFM
P6 (S1,...S6)	Lap-shear	3P bending	ILSS	CTE	OM	SEM	AFM
P7 (S1,...S6)	Lap-shear	3P bending	ILSS	CTE	OM	SEM	AFM

2.3.1. Surface Roughness

Six different types of roughness were applied on aluminium surface. These are Flat, Perforated, Side Punched, Surface Deformed, Side Punched+Surface Deformed, Side Punched+Perforated surfaces which are abbreviated as S1 to S6 shown in Table 2.5.

3D-modelling of hybrid material with different Surface Roughness on Aluminium;

To investigate of effects of different surface roughness for joining, 3D models for each aluminium surface types were created NX-Nastran software. The purpose of the analysis is to compare and interpret the maximum displacements and stresses of different samples against bending. Linear static solver in NX Nastran software was used for these analysis.

During simulation the set-up aluminium and PVC materials are glued on joined surfaces. As boundary conditions, 2 supports were applied to both ends of the sample. One is sliding bearing and other is built-in bearing. The line force which cause the bend, has been applied at the middle of the PVC surface.

2.3.2. Emulsion PVC

PVC emulsion has been prepared with Solvin Nanovin emulsion PVC – liquid 50% H₂O and 48% PVC as particles – 2 % emulsifier. This process has been described as P2.

2.3.3. Primer

According to cooperation with laboratory of Henkel, the product of Technomelt PUR Primer L457 (*Purmelt Primer L457*) which is a primer used for application of pre-treatment of aluminium surfaces. L457 has been decided to use in the study.

L457 primer has been designed for bonding with PUR hotmelt adhesives and priming on aluminium surface as cleaning and preparing to joining.

In this process, the aluminium surface of samples were pre-heated to 50 °C, then primer has been applied and priming process has been abbreviated as P3.

2.3.4. Adhesive (PU)

In the study, PURFact 121.10 was applied by melting it in the nozzle application unit of the machine in the melting chamber designed for reactive hot melt adhesives. PUR hotmelt adhesive begins to harden during cooling and continues to chemically cure as it comes into contact with moisture in the air.

As the PUR hotmelt adhesive cures, it will form a strong and permanent bond with the substrate.

2.3.5. Atmospheric Plasma Technology

The effects of plasma technology which has been considered to improve wettability and increase the bonding strength between PVC and aluminium surface were studied. During the process, plasma — partially ionised gas — initiates a multitude of physical and chemical processes that treat the surface, removing contaminants. The electrons

and ions in plasma is accelerating to very high energies and collide with gas molecules to produce short-lived, chemically active species, such as atomic hydrogen, nitrogen and oxygen species, hydroxyl radicals. These species are disinfect, clean, modify and functionalise a range of surfaces to prepare them for adhesive bonding.

Plasma has been applied during the extrusion process on aluminium surface (Figure 2.15).

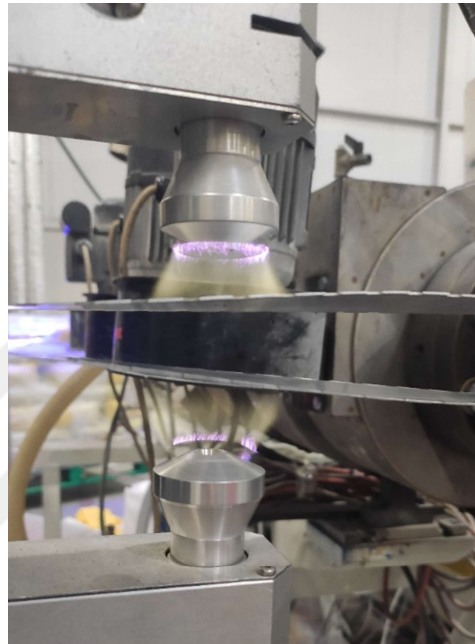


Figure 2.15: Plasma- rotary nozzle on aluminium surface (Experimental Setup)

2.3.6. Combination of Processes

Multiple process combinations were prepared and their effects on the test results were examined.

Process combinations are abbreviated as P5, P6 and P7 for Primer+E-PVC, Primer+Adhesive and Primer+Plasma shown in Figure 2.16.

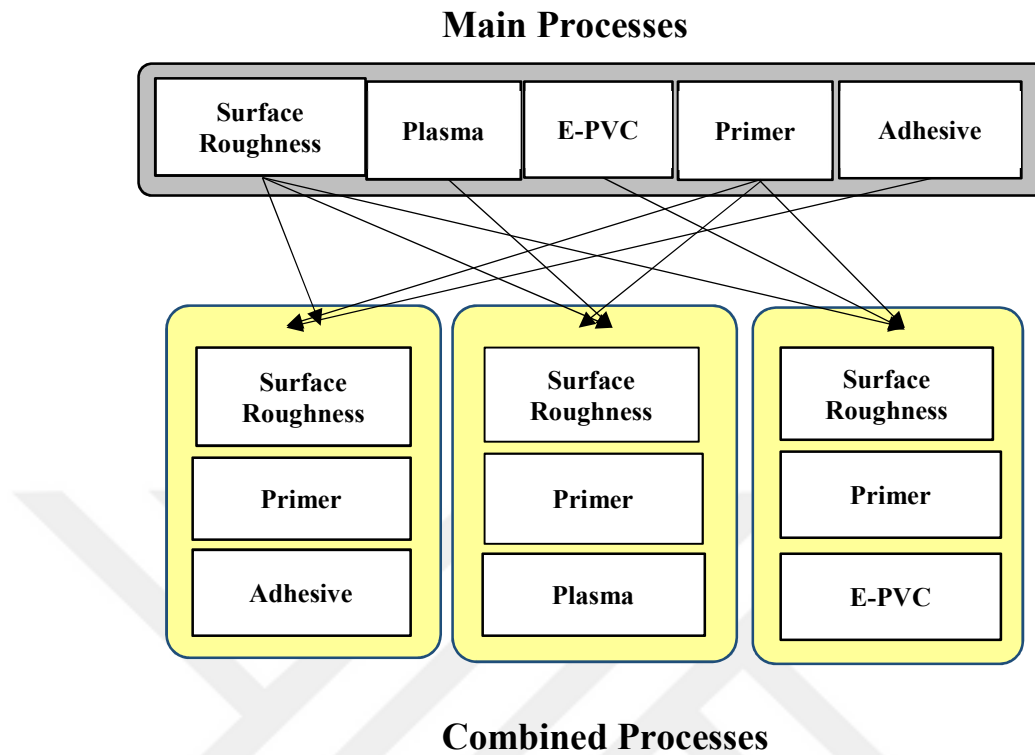


Figure 2.16: Process combinations

2.4. Characterization

2.4.1. Mechanical tests

2.4.1.1. Lap-Shear

The lap-shear test is determining comparative shear strengths of joined structures. It is the most commonly used as test method carried out to observe the effect of different surface behaviors due to its ease of assembly and simple testing method. Lap-shear test consists of two rectangular pieces joined and subjected to tension forces from the opposite ends.

The lap-shear tests were conducted on a Zwick mechanical testing equipment with a load cell of 20 kN. During the tensile/shear testing, the displacement control mode with a rate of 1 mm/min was utilized. After the peak loads were registered, the experiments were halted.

The test specimen in the lap-shear arrangement is formed by two rectangular pieces with an overlapping area large enough to cause failure. The subsequent tensile testing determines the lap-shear strength, which is given by:

$$\sigma = \frac{P}{b.L} \quad (2.1)$$

where,

P – Maximum load (N),

b- Joint width (mm),

L- Joint length (mm),

σ - Stress (N/mm²)

However, there are a number of influencing parameters for determining shear strength values, the most important of which is the sample preparation technique, which includes surface treatment, adhesive application, and thickness [89].

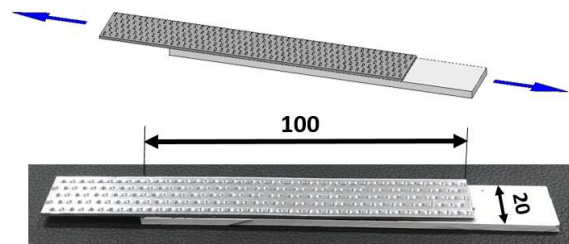


Figure 2.17: Test sample of lap-shear

Tensile properties of the polymer and composite materials were measured with Zwick Roell kN020. Samples dimensions were 20x100 mm as shown in Figure 2.17. The measurements were done at 22 °C. The maximum strength was obtained from the stress–strain curves. For each composites at least five samples were tested.

2.4.1.2. 3-Point Bending

In engineering mechanics, the flexure or bending determines the behavior of a long cylindrical structural element subjected to an externally applied load applied perpendicularly to a longitudinal axis of the element.

A flexural strength test imposes tensile stress in the convex side of the specimen and compressive stress in the concave side.

This creates an area of shear stress along the midway [90] to ensure the primary failure comes from tensile or compression stress. In the present test set up, a specimen is simply resting over the two pins held parallel to each other at the same level as in Figure 2.18.

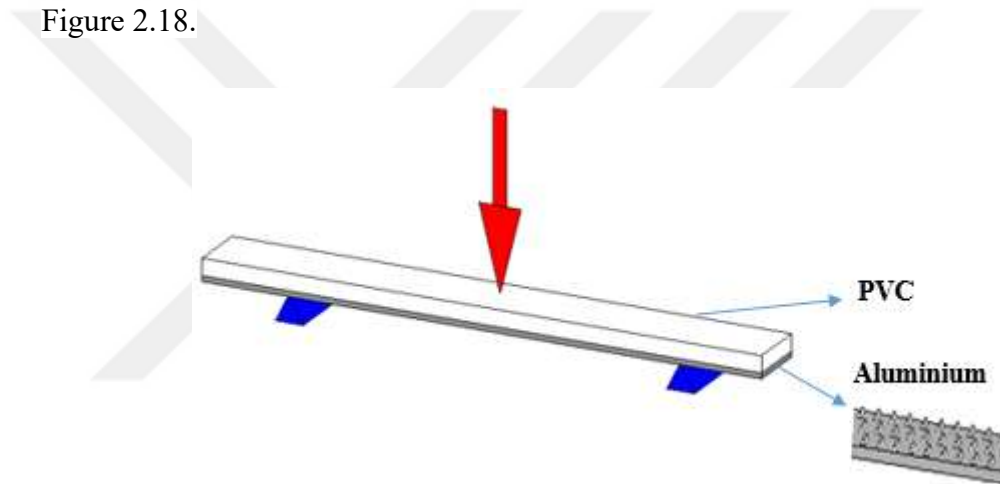


Figure 2.18: Material before bending (compression/tension)

A vertical downward load is applied at midspan, over the specimen using a loading pin. The supporting pins and loading pins allow free rotation about its own axis. This test helps to determine the deflection and flexural properties of material [91]. The flexural test measures the force required to bend a specimen under 3-point loading conditions. Flexural modulus is used as an indication of a material's stiffness when flexed.

In that bending, testing is used to find material characteristics like shear strength, modulus of elasticity etc. These test methods cover the determination of flexural properties of reinforced polymer and high modulus composites in the shapes of rods, plates, rectangular bars and other moulded shapes. These test methods are generally applicable for both rigid as well as semi rigid materials. But the flexural strength cannot be determined for those materials which do not break or do not fail in the outer surface

of the test specimen. Flexural properties of materials with linear stress - strain behavior are used for engineering design calculations [92]. For nonlinear characteristics, the flexural properties are observed to be only nominal. The test method for conducting the 3-point test usually involves a specified test fixture on a universal testing machine.

2.4.1.3. Inter Laminar Shear Strength (ILSS)

Interlaminar shear failure is one of the major failure modes for laminated hybrids. The resistance against shear delamination is characterized by the interlaminar shear strength (ILSS) .

The apparent inter-laminar shear strength (ILSS) of hybrid materials can be calculated, provided that inter-laminar shear fractures the specimen during the test. A specimen is loaded in a three-point bending arrangement for this purpose, and a rise in shear stress and inter-laminar failure is achieved by selecting a proper ratio of specimen thickness to support span. The fracture surface is assessed after the specimen is fractured, and the kind of failure is determined.

The short beam shear (SBS) test subjects a beam to bending, the beam is very short relative to its thickness. The short beam strength test of high modulus reinforced composite materials is determined in ASTM D 2344. ASTM D 2344 specifies a supports pan length to specimen thickness ratio (s/t).

The specimen thickness defines other dimensions of the sample according to definitions in ASTM D2344.

$$\text{Specimen length, } L = \text{thickness} \times 6 \quad (2.2)$$

$$\text{Specimen width, } b = \text{thickness} \times 2 \quad (2.3)$$

The example of test sample dimensions is given in Figure 2.19 which has the sizes as 4x24x8 mm (*thickness x length x width*).

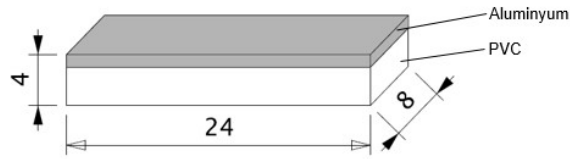


Figure 2.19: Sizes of ILSS samples (4x24x8)

The objective is to minimize the flexural (tensile and compressive) stresses and to maximize the induced shear strength. The ILSS values were evaluated from the short beam shear test according to the following relation:

$$\text{ILSS} = 0.75 \times P_b / (b \times d) \quad (2.4)$$

Where P_b = breaking load (N), b = width (mm) and d = thickness (mm) of the specimen.

2.4.2. Thermal Tests

2.4.2.1. Coefficient of Linear Thermal Expansion (CTE)

The coefficient of linear thermal expansion (CTE, α or α_1) is a material property that is indicative of the extent to which a material expands upon heating. Different substances expand by different amounts. Over small temperature ranges, the linear thermal expansion of uniform objects is proportional to temperature change. The coefficient values of linear expansion for PVC and aluminium are given in Table 2.6.

Table 2.8: Values of coefficient of linear thermal expansion for materials

Material	Coefficient of Linear Thermal Expansion ($1/^\circ\text{C}$)
PVC	50.4
Aluminium	23.1

The test specimens were marked as l_0 at T_0 , after the test, l_f has been recorded at temperature T_f (Figure 2.20).

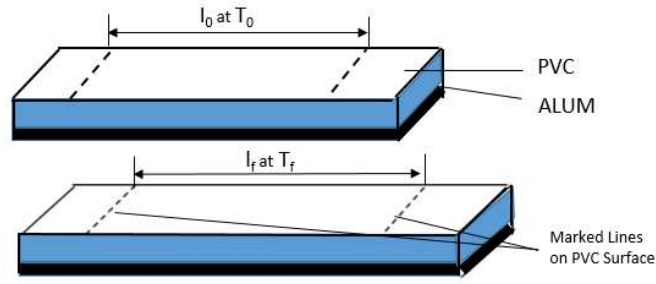


Figure 2.20: Marking on joined samples

By measuring marked lengths at room temperature, the linear expansion and the temperature difference, α value can be calculated as formulation given below.

$$(l_f - l_0) / l_0 = \alpha l \times (T_f - T_0) \quad (2.5)$$

$$\Delta l / l_0 = \alpha l \times \Delta T \Rightarrow \alpha l = 1 / l (dl/dT) \quad (2.6)$$

where l_0 and l_f indicate, respectively, where l_0 and l_f represent, the original and final lengths with the temperature change from T_0 to T_f . The parameter αl has units of reciprocal temperature (K^{-1}) such as $\mu m/mK$ or $10^{-6}/K$.

The length of the marked sample is measured at room temperature, and again when it has been heated up. Test has been performed at 20 °C (T_0) to 70°C (T_f) with 10°C increasing temperature intervals.

2.4.3. Microscopic Analysis

For the microscopic tests, the Optical Microscope, SEM and AFM analysis were performed on hybrid samples.

2.4.3.1. Optical Microscope

Interface layer of sections of hybrid samples were analysed in Optical Microscope. Eclipse LV100ND model microscope in Nikon which is motorized one with episcope/ diascope illumination were used for these analysis. The section of joined samples were prepared and then the microscopic analysis have been performed for 100X magnifications.

2.4.3.2. Scanning Electron Microscope (SEM)

SEMs with high magnification ratios are used to learn morphological, structural, and elemental information, topographic composition, and composition of specimens, as well as to determine the interaction between the materials. The measurements are taken at 100X and 250X magnifications.

2.4.3.3. Atomic Force Microscopy (AFM)

In recent years, atomic force microscopy (AFM) has become a very essential tool in the investigation of surfaces and nanoobjects. The procedure is deceptively simple: a cantilever with a sharp tip at one end, commonly made of silicon or silicon nitride with tip diameters on the order of nanometers, is brought close to a sample surface. The cantilever deflects due to the van der Waals interaction between the tip and the sample. A laser beam is reflected from the top of the cantilever into an array of photodiodes, which is used to measure the deflection. If the tip is scanned at a consistent height, there's a chance it'll collide with the surface, resulting in damage. The sample is usually put on a piezoelectric holder in all three dimensions (x and y for scanning the sample, z for maintaining a constant force) [93].

The topography of the sample is represented by the $s(x,y)$ map that results. Contact mode, non-contact mode, and dynamic contact mode are the three main modes of operation for an AFM. The force between the tip and the surface is kept constant while scanning in the contact mode operating by keeping a constant deflection. The cantilever is externally oscillated at or near its resonance frequency in the non-contact mode. The tip-sample interaction forces change the oscillation; these variations in oscillation relative to the external reference oscillation offer information about the sample's attributes. The cantilever is oscillated in dynamic contact mode until it makes contact.

Compared to other nanoanalysis techniques, such as the electron microscope, the AFM approach has significant advantages. Unlike other approaches, AFM produces a three-dimensional surface profile of a material. Furthermore, materials seen by AFM do not require any special treatment, such as high vacuum, and it is a non-destructive

technique, unlike electron microscopy images, which frequently destroy the organic sections of a hybrid sample due to high energy electron beams.

However, compared to SEM, AFM can only reveal a maximum height of a few micrometers and a maximum area of around 100 by 100 micrometers, and the scanning speed is slow.

According to tests explained in this study, overview of test performed samples is shown in Figure 2.21.

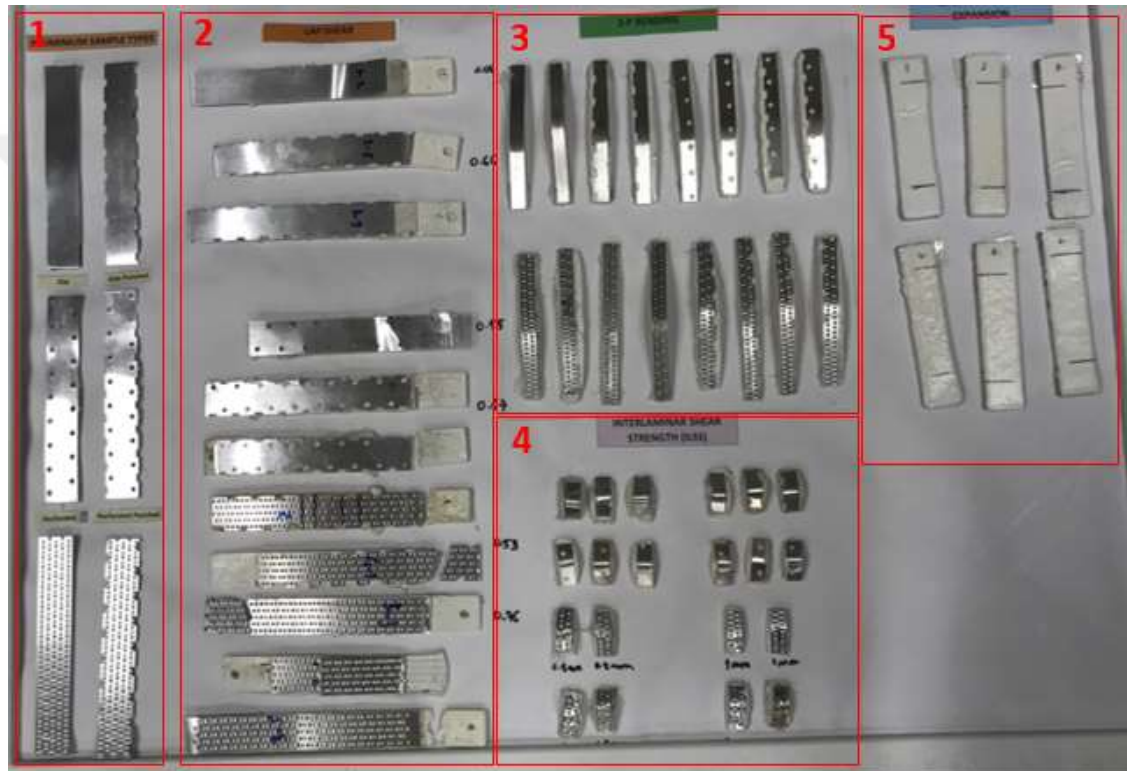


Figure 2.21: Overview of test samples 1-Aluminium sample types; 2-Lap-shear samples; 3-3P Bending samples; 4-ILSS samples; 5-Coefficient of Linear Thermal Expansion samples

Chapter 3

3. Results And Discussions

In chapter 3, the results of design of experiments, mechanical and thermal tests, microscopic analysis were explained.

3.1. Design of Experiments (DOE)

In order to analyze the effect of the design of the experimental parameters, the aluminium surface roughness and the process types were used for the improvement of two material joining. MINITAB software was used to examine the main effect and interaction plots.

In the study of Rafidah et al.(2014) on “Comparison Design of Experiment (DOE): Taguchi Method and Full Factorial Design in Surface Roughness” , they compared the effectiveness of the Taguchi and full factorial design methods on surface roughness using both Taguchi and full factorial design techniques [94]. According to the obtained results in their paper, the full factorial design looks better DOE technique than the Taguchi method, since the mean square error is lower, and the parameter design of the full factorial design provides a simple, systematic, and efficient methodology for optimizing the process parameter. When the available technics in DOE analysis were evaluated, the full factorial design has been decided to use in current study.

DOE which is shown in Figure 3.1; the dependent variables (responses) consist of the test results as lap-shear, 3-point bending, interlaminar shear strength (ILSS), coefficient of linear thermal expansion (CTE) performed as a result of the study. The independent variables (factor) were determined as aluminium surface roughness and process types. The determined independent variables are classified as levels and described in Minitab software. There are 6 levels for aluminium surface roughness and

they are described as S1 to S6. The process types of joining methods are outlined as 7 levels like P1 to P7.

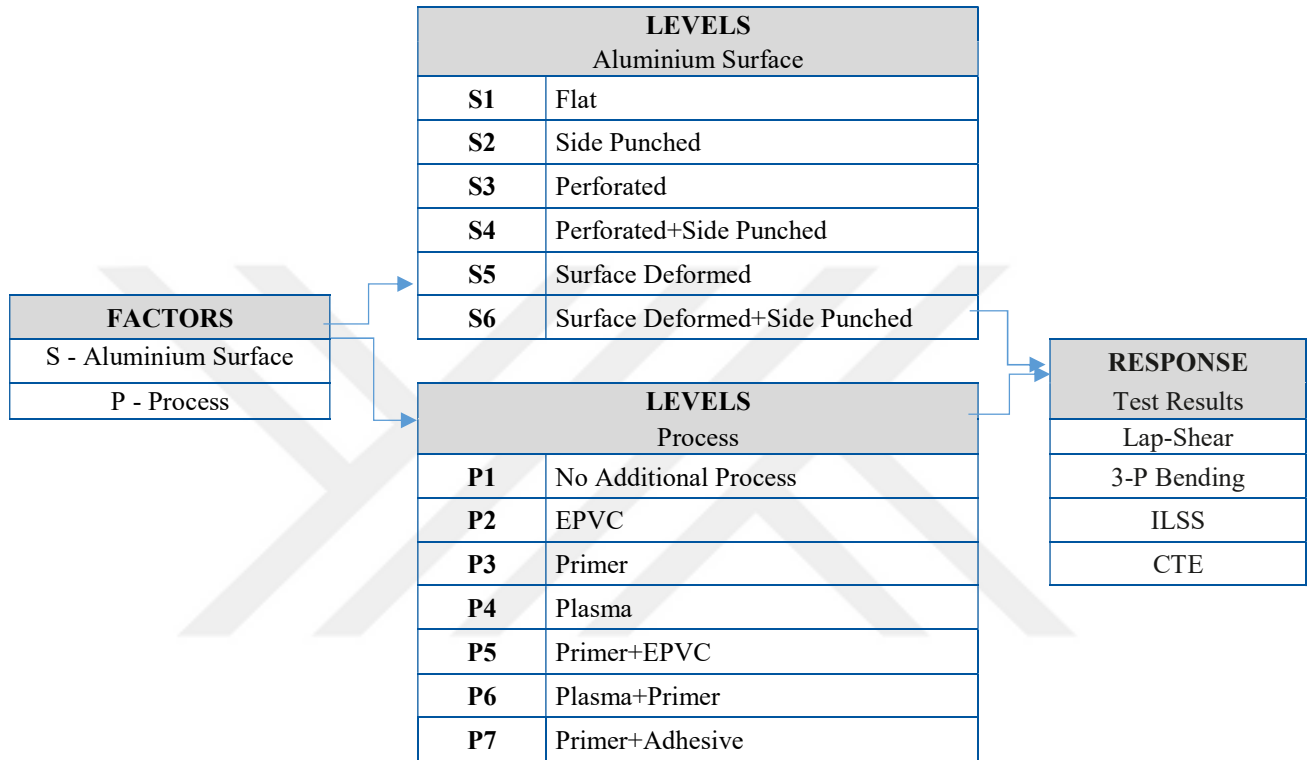


Figure 3.1: DOE analysis in the study (factors, levels and responses)

3.1.1. Main Effects Plots

Main effect plots provide the information about which is most influencing factor and classic relationship between availability, performance and quality rate [95].

In this study, the main effect plots describe the relations of levels between aluminium surface roughness and process types. For each level, the mean values of test result were shown on the main effect plots. Therefore, these plots provide a good overview of the data.

3.1.1.1. Main Effects Plot of Lap-shear

The results of the lap-shear relationship between the different samples were explained in the first main effect plot. These plots show that the mean value of that parameter based on different roughness and process types. According to the given results in Figure 3.2., addition of roughness and process types have positive effects on the results. While S1 (untreated flat surfaces) have the lowest value, the additional processes and mechanical operations on surface have an impact on the values as variations. Mechanically, S2, S3 and S4 have been resulted as higher than 2 MPa. From the process view, P3, P5 and P6 processes have provided an increase in obtained values which is shown in the main effects plot (Figure 3.2).

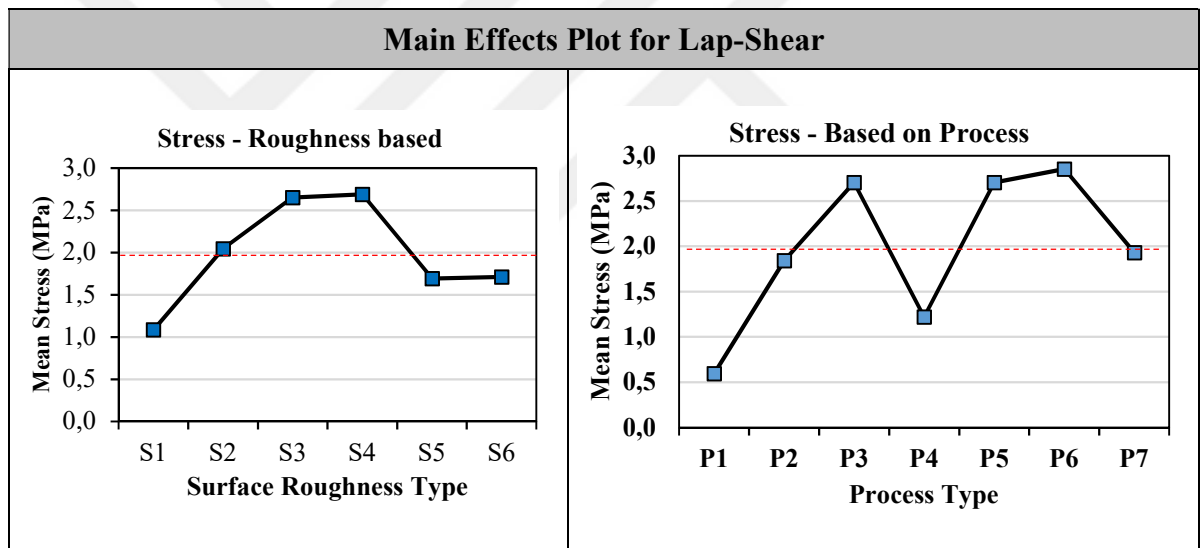


Figure 3.2: Main effects plot for lap-shear

Jung et al.2013 studied on “Atmospheric pressure plasma treatment of polypropylene to improve the bonding strength of polypropylene/aluminium composites”. According to their results after eight treatments, peel and shear strengths were improved by 42 percent and 44 percent, respectively with plasma treatment [96]. The current results show that the plasma process does not have similar improvement on the interface layer in hybrid structure shown in main effects plot for lap-shear (Figure 3.2). Process conditions of plasma in present study needs to more optimization to well design, therefore the further studies were planned to work at different plasma parameters.

3.1.1.2. Main Effects Plot of 3P Bending

3P bending main effect plot explains the measured deflection on test samples. The highest deflection value were measured in S1 and P1, which was decreasing roughly from 2.5 mm to lower than 1.0 mm with surface roughness and adding extra treatments. P5, P6 and P7 specimens have low displacements as desired which are shown in Figure 3.3.

The experimental results indicated that, the pretreatment on aluminium surface with primer provided the lower displacement. S4, S5 and S6 samples in roughness group have been better resulted than others.

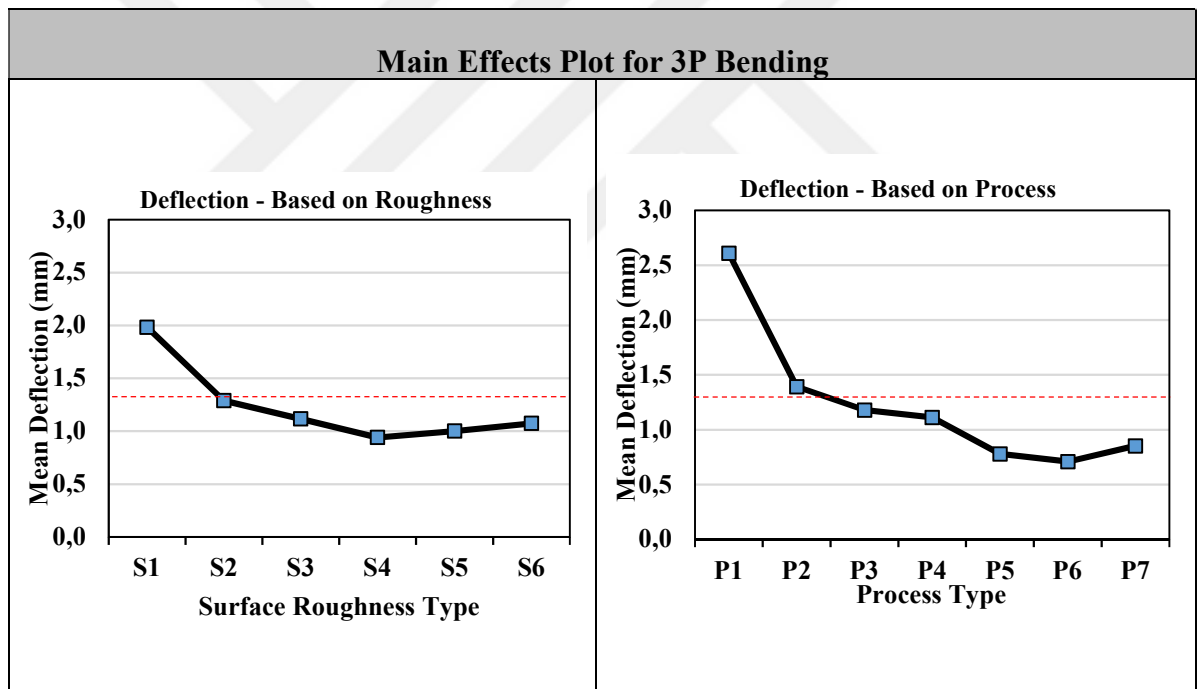


Figure 3.3: Main effects plot for 3-P bending

3.1.1.3. Main Effects Plot of ILSS

The results of the ILSS test indicated that the surface roughness on the aluminium surface has an effect in positive direction on the values. The highest stress values have been performed on P3, P5 and P6 samples (Figure 3.4).

In contrast to expectations, the outputs of plasma-treatment and primed adhesive specimen P4 and P7, ILSS test have been resulted with low outcomes. The parameters of plasma like the treatment time and distance must be re-evaluated. From aluminium surface types, S4 and S5 have higher values in their range. Small sizes of ILSS specimen needs to prepare the sampling more sensitive. It is the one reason for these deviations on the results. If location of the roughness was not centered correctly on specimen, it creates some deviations on unexpected direction.

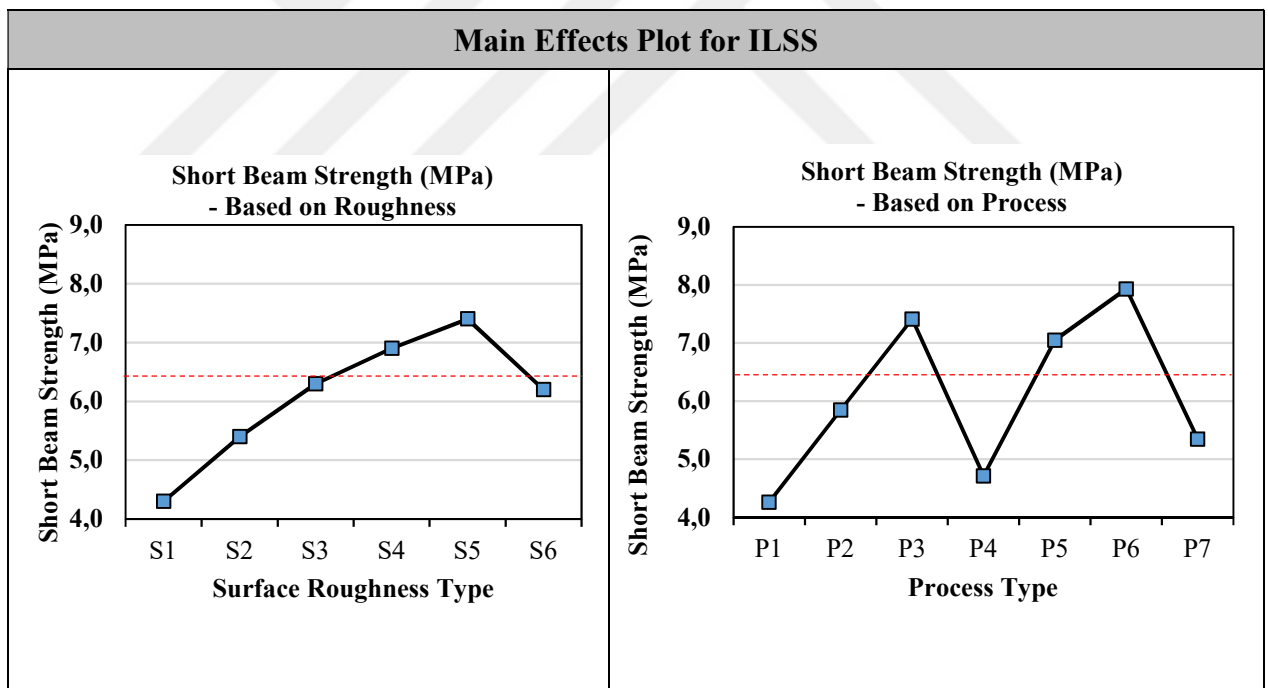


Figure 3.4: Main effects plot for ILSS

3.1.1.4. Main Effects Plot of CTE

In Figure 3.5, the main effect plot for the coefficient of linear thermal expansion (CTE) results have been indicated. The measured results for CTE show that the roughness on aluminium surface has an effect on expansion property. The lowest values in S range, which are identical for hybrid structure were obtained in S2, S4 and S5 samples.

The additional processes P3 and P6 have provided decrease on CTE values. Plot indicates that CTE can be decreased from 0,27 to 0,10 mm/°C when the additional treatment process has been involved.

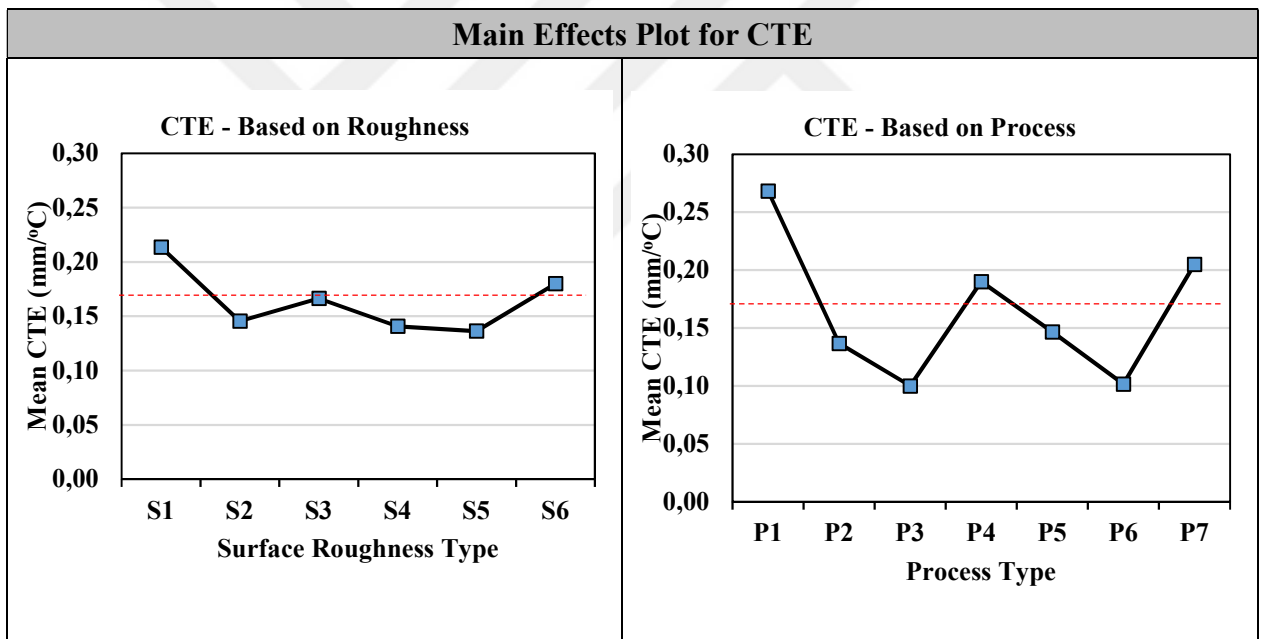


Figure 3.5: Main effect plot for CTE

According to the plots of main effect in DOE studies, the surface roughness examined on the aluminium surface contributes positively to joining interface. Additional processes are also resulted positively, when they are compared untreated process. DOE study results show that S2,S4 and S5 specimens achieved the better results among all samples. Similarly, when the results are evaluated based on process, P3 and P6 applications were found to be more successful in joining.

3.2. Mechanical Tests

3.2.1. Lap-Shear Results

Kwon et. al has researched on “Comparison of interfacial adhesion of hybrid materials of aluminum/ carbon fiber reinforced epoxy composites with different surface roughness” in 2019. They have modified the surface of aluminium using some sanding processes. The lap-shear tests were performed on the modified samples which have effect of surface roughness and adhesions. The results show that increasing sanding time as higher surface roughness, was improving mechanical locking. In other words, the increased energy of surface results in improved mechanical adhesion with higher lap-shear values [97]. Similar to these results, the different roughness types have improved the results positively in our study. Because of locking mechanism, PVC material filled the deformations on aluminium surface, the results were obtained identically higher than S1-flat surface.

Hamdi et. al (1995) studied on “Improving the adhesion strength of polymers: effect of surface treatments” on PVC, ABS and EPDM materials. According to their outputs on graphs for lap-shear tests, PU application on PVC surface has lower effect than other adhesives which selected as silicone and modified silane. On the other hand, the samples which were cleaned and primed ones have better results than not treated samples [98]. Pretreatment had also similar influence on the outcomes of present study specimens. The samples which were primed as a pretreatment on aluminium surface, has higher values on lap-shear tests.

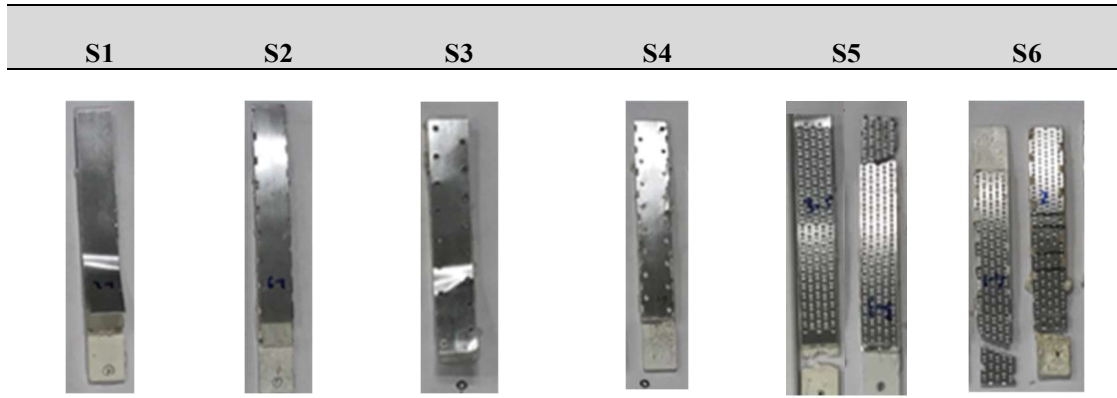
The surface treatment of effect on aluminium was studied by Boutar et.al (2016) for automotive applications. They have aimed to determine the effect of surface roughness and wettability on the strength of single lap joints on three abraded surface. The results indicate that shear strength appears to increase from not abraded to polishes surface with abrasive paper that provides 0.6 μm surface roughness. But, after having maximum shear strength, the surface roughness degraded the bond strength of adhesive which means reducing in shear strength at higher roughness. They have

summarized that the joint durability can not be provided by higher surface roughness, it also depends on characterisation of the interphase and its formation mechanism [99]. In the present thesis, the customized tool has been designed to deform and increase the surface area like roughness. While the values were expected to go up, the results were actually worse than expected, because of air in the gap remained closed, it had a negative impact on the outcomes. Equivalent to the their study, depth of roughness must be optimized. Otherwise, the roughness can have a negative effect on the results.

“Plasma treatment for improved bonding” has been studied by Liston et al.(1989) with performing some lap-shear tests. The polyphenylene sulfide/glass fiber material were treated to improve the bonding with plasma effects of cleaning of surface, ablation, crosslinking (when the surface exposed to plasma, free radicals were created in the polymer). The datas that they have obtained indicates optimum treatment time for the polymer was between 5 and 7 minutes not an immediately. As a result of this treatment, the lap-shear bond strength improves by a factor of three. A further increase in bond strength were obtained at 15 minutes of treatment time. At 30 minutes, the slight decrease was appeared in bond strength which was not statistically significant [68]. This study shows that optimum treatment time with plasma which is critical for bond strength, should be minimum 5 minutes. In current process, since the plasma was applied during the extrusion which is continue process, the maximum plasma treatment time could be 15 seconds which is lower than optimum declaration. Therefore, the test results for plasma specimens were lower than other additional processes. Plasma must be studied again with keeping longer treatment time in further studies.

The lap-shear tests on six different roughness were examined to measure maximum stress in Zwick Roell Z020 (Table 3.1).

Table 3.1: Samples after lap-shear test



According to the results, the surface roughness on aluminium samples have affected as an increase in outcomes. While the lap-shear test result value is 0.05 MPa for S1 (flat surface), a result above 2 MPa were obtained even on a flat surface with additional processes.

In the case of comparing lap-shear results in re-formed surfaces, the highest values were resulted in S3 (perforated) as 3.94 MPa and S4 (side punched perforated) as 3.86 MPa (Figure 3.6).

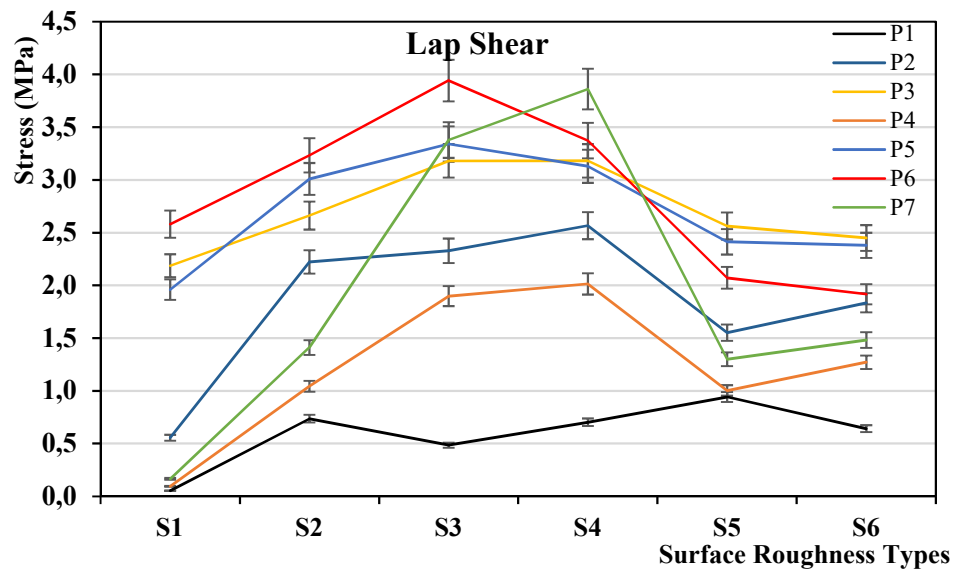


Figure 3.6: Measured max stress of lap-shear based on surface roughness

Process type comparison for the results can be done in Figure 3.7, which indicates lap-shear values based on process. This graph shows that the overall average of primered adhesive samples achieved the highest values.

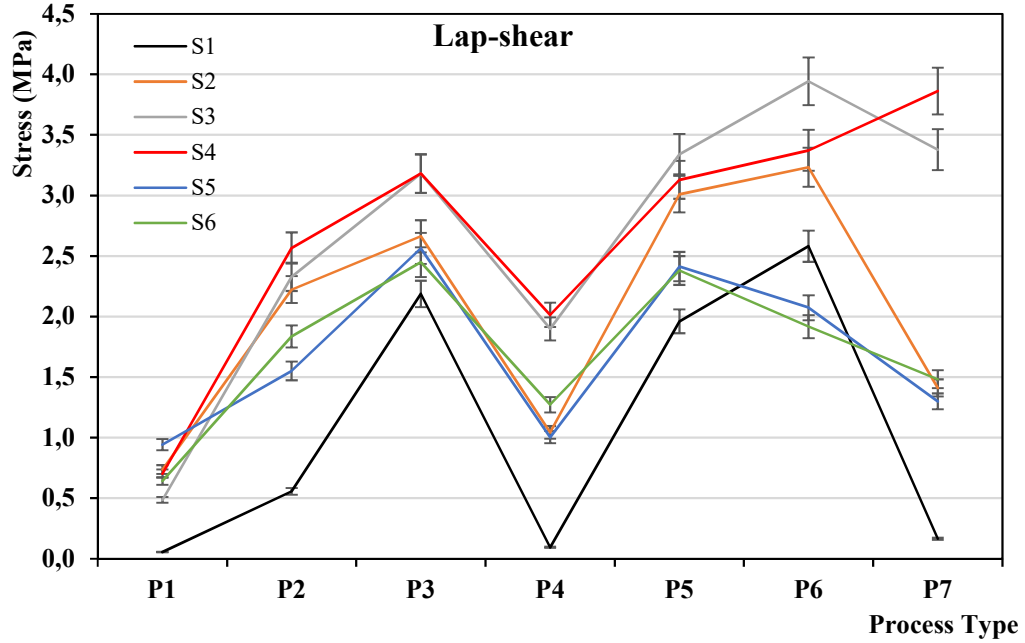


Figure 3.7: Measured max. stress of lap-shear testing based on process type

The detailed overall data of Lap-shear test results are available in Table 3.2.

Table 3.2: Results of lap-shear stress (MPa)

	P1	P2	P3	P4	P5	P6	P7
S1	0.05	0.56	2.19	0.09	1.96	2.58	0.17
S2	0.74	2.22	2.66	1.04	3.01	3.23	1.41
S3	0.49	2.33	3.18	1.90	3.34	3.94	3.38
S4	0.70	2.57	3.18	2.01	3.13	3.37	3.86
S5	0.94	1.55	2.56	1.00	2.41	2.07	1.30
S6	0.64	1.84	2.45	1.27	2.38	1.92	1.48

Similarly, the results that were obtained in present study for only primer and primered adhesive samples, were performed as the highest value. Silicon can also be considered in future studies for this process.

3.2.2. 3-Point Bending Results

Lee et al.(2016) studied on a carbon fiber reinforced plastic/AI5052 hybrid samples. Microsurface roughness has been provided with sandblasting process, and its mechanical properties were investigated through three-point bending test. When the flexural stress of the composite was measured in relation to the surface roughness, it was found that if the specimen surface was treated with sandblasting, the flexural stress remained relatively constant regardless of the surface roughness; nevertheless, it was lower when the specimen surface was not treated. This shows that surface treatment improved the flexural strength of the material. The specimen had a flexural stress of 480–500 MPa after sandblasting, whereas it was only 220 MPa in the absence of surface treatment [100]. In the present study, the roughness on surface has resulted in an increase in flexural strength. The 3-point bending test results showed that in the samples with surface roughness, the fracture surfaces were filled with plastic material, thus improving the interface, which also led to a positive decrease in the deflection results in this study.

Zal et al. (2016) studied on effect of surface roughness of aluminium for fiber metal laminates (FML) which includes fiber glass, PVC film(0.2 mm) and aluminium. Four different surface treatments (pickled with HCl, cold rolled, holes, grinding and mechanically roughened) were applied on three-point bending samples to measure the flexural strength. The least flexural strength has been measured in etched aluminum layered sample, because of pickling removed contaminant substances from aluminium surface and tend to form chemical bond. But, degrading of these bonds were resulted as forming hydrated-oxides which displace the chemical bonds and make them weak. On the other hand, the samples with piercing of aluminium allows connection of PVC material from both sides. In this case, PVC polymer tolerates delaminating shear stress which also provides improvement of flexural strength. The overview of the results showed that mechanical treatment and roughening of the aluminum surface was found as the good treatment method to obtain a high strength PVC matrix/aluminum layer interface bonding in the produced FMLs [101]. In their study, some processes as surface treatments and having some holes similar to S2 and S3 specimens look close

to principally. The creating holes improved the interlock mechanism in both studies were resulted with higher values. Piercing on aluminium allowed the PVC filling which is similar S2-side punched and S3- perforated specimens, resulted as better outcomes.

“Effects of surface roughness and bond enhancing techniques on flexural performance of CFRP/concrete composites” subject has been studied by Ariyachandra et al. (2017). The surface preparation and bonding alternative techniques on hybrid performance were performed with three-point bending test method. The purpose was to define the effects of dissimilar surface roughness class on flexural performance. Polymer anchorages effects that delaying the end debonding were studied. The results show that, the surface roughness of concrete substrates has significant effects on bond strength of CFRP-concrete composites. Samples with shorter leg – anchorages results were not big improvements, which means longer leg-anchorage geometry were provided better outputs in three-points bending results [102]. The impact of surface preparation and roughness on the joining performance of hybrid structure was clearly demonstrated in present study. In a study similar to ours, surface roughness and treatment were developed to enhance the bonding.

Interface assessment has been studied by Karakaya (2020) as “Overmolded hybrid composites of polyamide-6 on continuous carbon and glass fiber/epoxy composites” . They have investigated an alternative joining method on overmolding of thermoplastics on thermosets composites. Three-point bending based on surface roughness was one of the test method they have used. The peel ply treated overmolding on surface indicates better adhesion performance. The tests reveal that the peel ply application increases the roughness with providing good effect on flexural strength and modulus. This effect was more evident on the sample which was prepared with 80°C temperature mold [103]. In the case of one element of joining is polymer material, the temperature of process is critical. In that study higher temperature as 80 °C provided the softening phase for polymers to improve the joining. Similar to that, the temperature in hot press was 190°C in current study, which allowed the softened PVC higher than Vicat temperature. The roughness on aluminium surface was filled with soft-polymer that enabled good adhesion.

3.2.2.1. Surface roughness simulations-Siemens Simcenter 3D

Different surface roughnesses on aluminium surface have been created as 3D models in CAD software. The linear statics and dynamic models were constructed in **Siemens Simcenter 3D - Nastran** to analyse the displacements and stresses of resistance to bending on these models. The main purpose was to simulate 3P bending process on different roughness model to see the behavior of them before handling.

According to the results obtained in analysis software, the displacement of S4 sample has minimum deflection value (1.72 mm) and the biggest resistance to bending (262.07 MPa) in Figure 3.8. The larger surface area provided higher interlocking mechanism, which was resulted as higher flexural strength. The stresses and displacements of the notch effect for specimens were given in Table 3.3.

Table 3.3: Results of stresses (MPa) and displacements (mm) in structural analysis

Results	S1	S2	S3	S4	S5	S6
Stresses (MPa)	40.55	54.92	261.83	262.07	205.76	208.05
Displacement (mm)	1.88	1.76	1.75	1.72	1.78	1.79

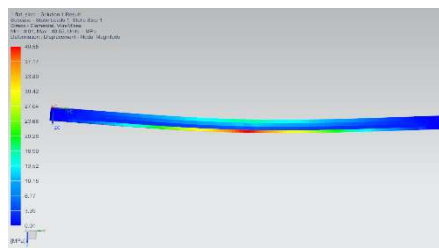
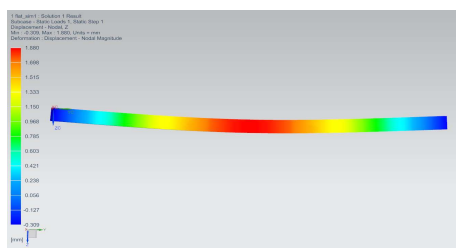
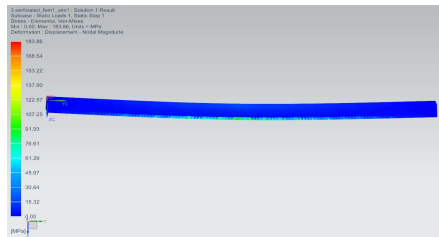
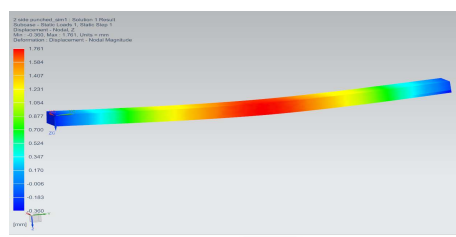
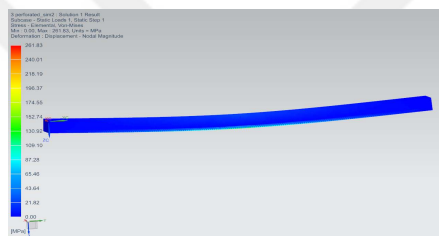
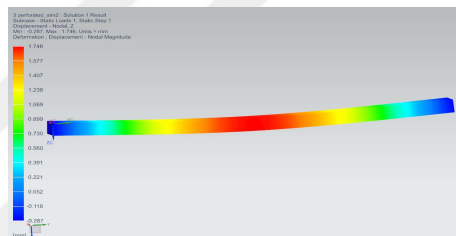
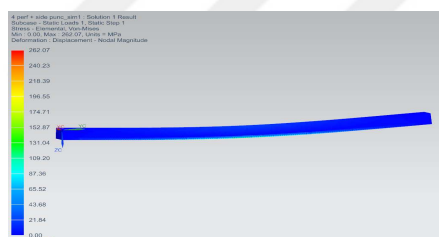
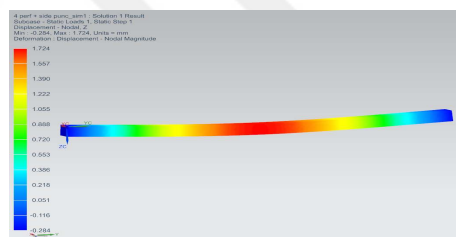
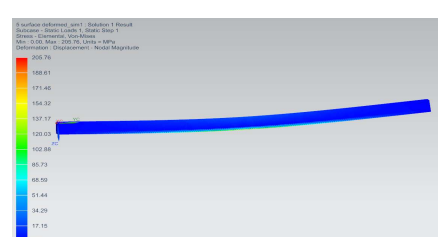
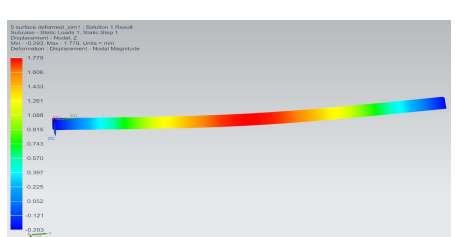
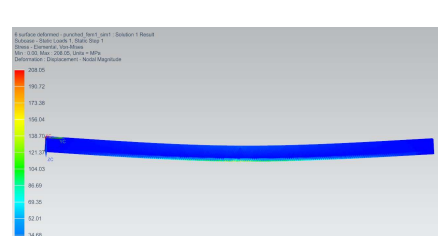
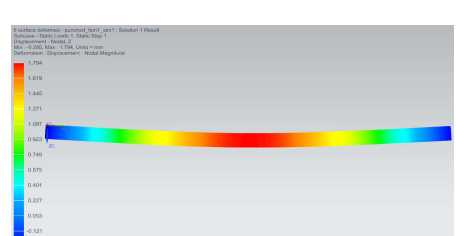
	Stresses (MPa)		Displacement (mm)	
S1		40,6		1,88
S2		54,9		1,76
S3		261,8		1,75
S4		262,1		1,72
S5		205,8		1,78
S6		208,1		1,79

Figure 3.8: Deflection analysis for different surface roughness

3.2.2.2. 3P Bending measurement results

3P bending test have been examined for S and P hybrid specimens. Testing and delaminated samples were shown in Figure 3.9. The one of important result of this test is that the geometry of aluminum did not recover after testing, implying that the alloy type in the hybrid structure must be thoughtfully determined.



Figure 3.9: Pictures from 3P bending samples

Two different test methods was examined. First, increasing force and deflection results were plotted for P2 sampling (Figure 3.10). Second, the deflection values were compared for 30 N for each samples which were shown Figure 3.11 and Figure 3.12.

Deflection values indicated that the roughness on aluminium surface is a critical on the displacements. Due to roughness, sample S1 had the largest negative deflection value, while sample S5 had the minimum value.

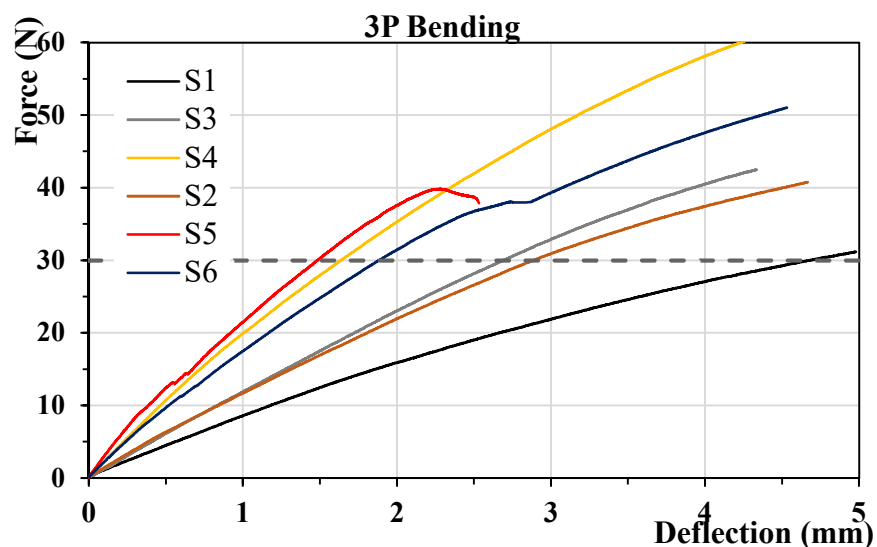


Figure 3.10: Measured deflection

3P bending test has been performed for all hybrid specimens at 30 N. The measured deflections were indicated in Figure 3.11 and 3.12 which are classified according to surface roughness and process types. Due to the test results which are shown Figure 3.11, the samples of flat aluminium surface have maximum deflection as 4.3 mm. Figure 3.11 presents any application on an aluminium surface that decreases the deflection displacement. The best results have been obtained in primered adhesive application as lower than 1 mm.

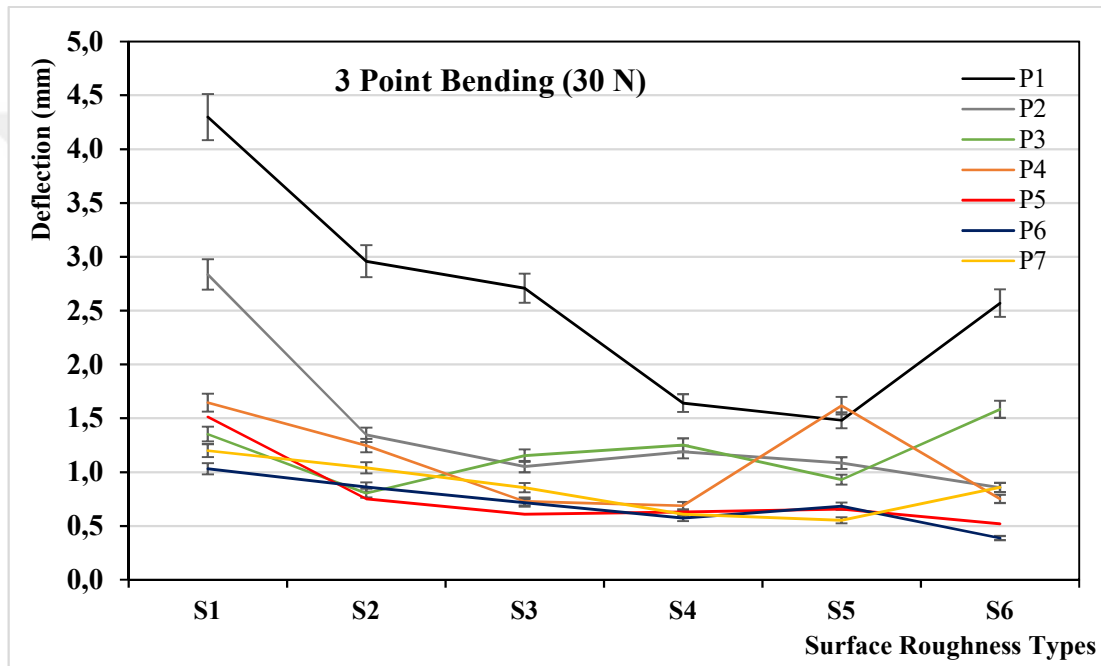


Figure 3.11: Measured deflection of 3P bending test based on surface roughness

In the case of evaluation according to process types, similar approach in decrease can be observed with additional processes (Figure 3.12). Primered adhesive and primered plasma hybrid samples have the lowest deflection as lower than 1 mm at 30 N.

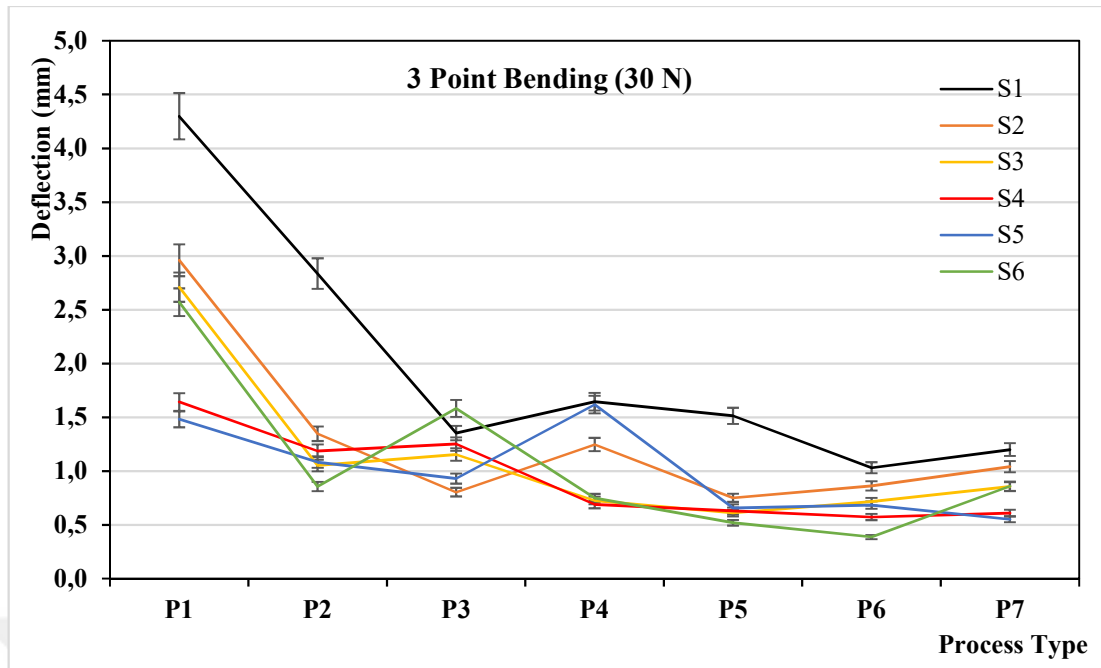


Figure 3.12: Measured deflection of 3P Bending test based on process type

The detailed overall data of 3P bending test results are available in Table 3.4.

Table 3.4: Results of 3 point bending deflection (mm)

	P1	P2	P3	P4	P5	P6	P7
S1	4.3	2.8	1.4	1.6	1.5	1.0	1.2
S2	3.0	1.3	0.8	1.2	0.8	0.9	1.0
S3	2.7	1.1	1.2	0.7	0.6	0.7	0.9
S4	1.6	1.2	1.3	0.7	0.6	0.6	0.6
S5	1.5	1.1	0.9	1.6	0.7	0.7	0.6
S6	2.6	0.9	1.6	0.8	0.5	0.4	0.9

3.2.3. Inter Laminar Shear Strength (ILSS) Results

Choi et al. 2010, have a study on “Effects of surface pre-treatment and void content on GLARE laminate process characteristics”. Surface morphology and voids produced at the metal sheet–prepreg interface of laminates were investigated. The studies were carried out on aluminum sheets with different roughness levels of surface textures (sanding and nylon-pad abrasion) and chemical etches systematically changing the surface morphology. The ILSS was measured as a function of surface roughness. Roughness and surface energy variations effectively improve laminate bonding strength [104]. In our study, similar to their outputs, the measured ILSS results were higher for the specimens which having pretreated processes on surfaces. Roughness has improved the results identically for the samples S4,S5 and S6 (perforated, punched,deformed surfaces).

Wu et al.2014, studied the impacts of various surface treatments on fiber metal laminate interlaminar shear strength (ILSS). For surface treatment of aluminum, they utilized solvent degreasing, mechanical abrasion, alkaline cleaning, and plasma treatment. Sandpaper and alkaline cleaning with NaOH had the greatest ILSS among the various surface treatments. The reason for the greater ILSS value is that when metal is abraded with a lower grit sandpaper, it achieves a higher roughness than when metal is abraded with a higher grit sandpaper. With alkaline washing, an interface layer was created, causing a bridging effect between aluminum and composite, resulting in greater adhesion. As the concentration of NaOH rises, the thickness of this interface layer increases [105]. As lower grit sand paper provided higher roughness in their study, ILSS measurements of S5 surface deformed samples were resulted more than others which is identical to outcomes of higher roughness.

Inter Laminar Shear Strength (ILSS) test has been performed for the hybrid samples. The samples after the tests are shown in Figure 3.13.

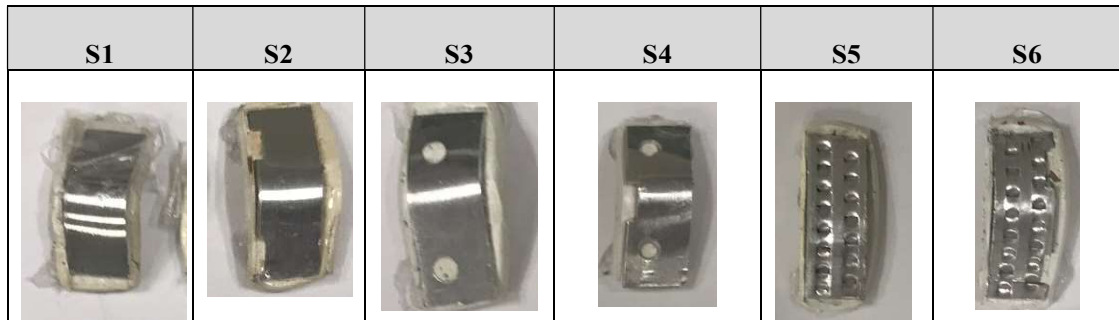


Figure 3.13: Hybrid samples after ILSS test

The experimental results of the short beam strength (SBS) test in terms of the maximum inter-laminar shear and stresses load-displacement curves are shown in Figure 3.14 and 3.15.

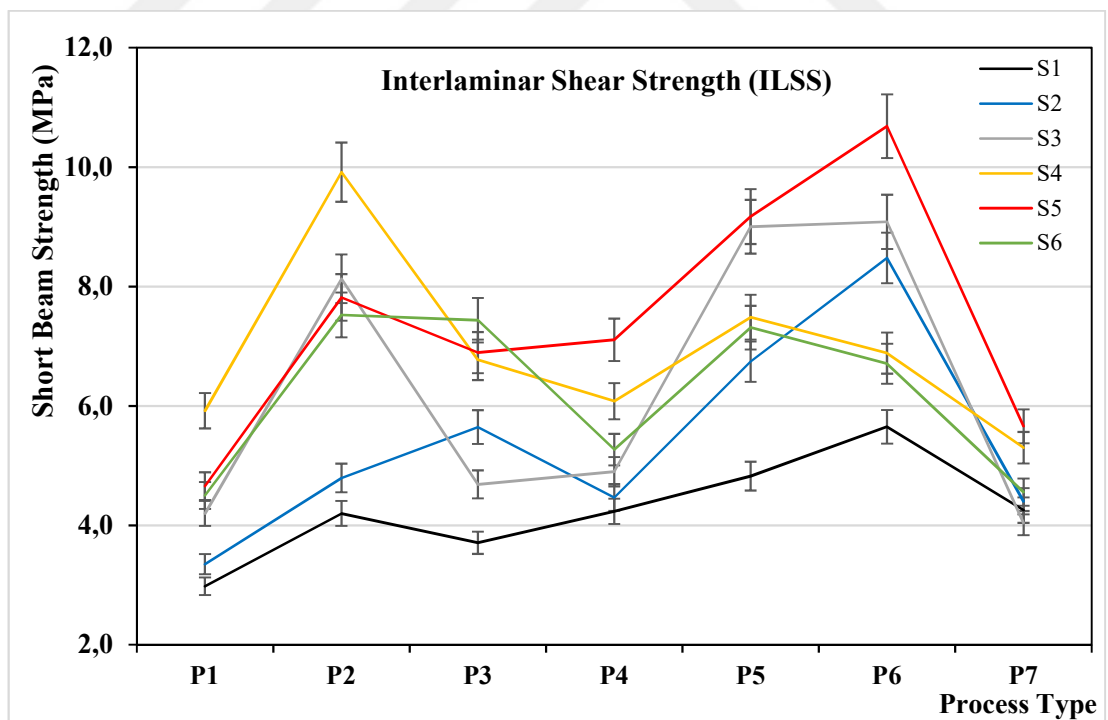


Figure 3.14: Short beam strength results according to process type

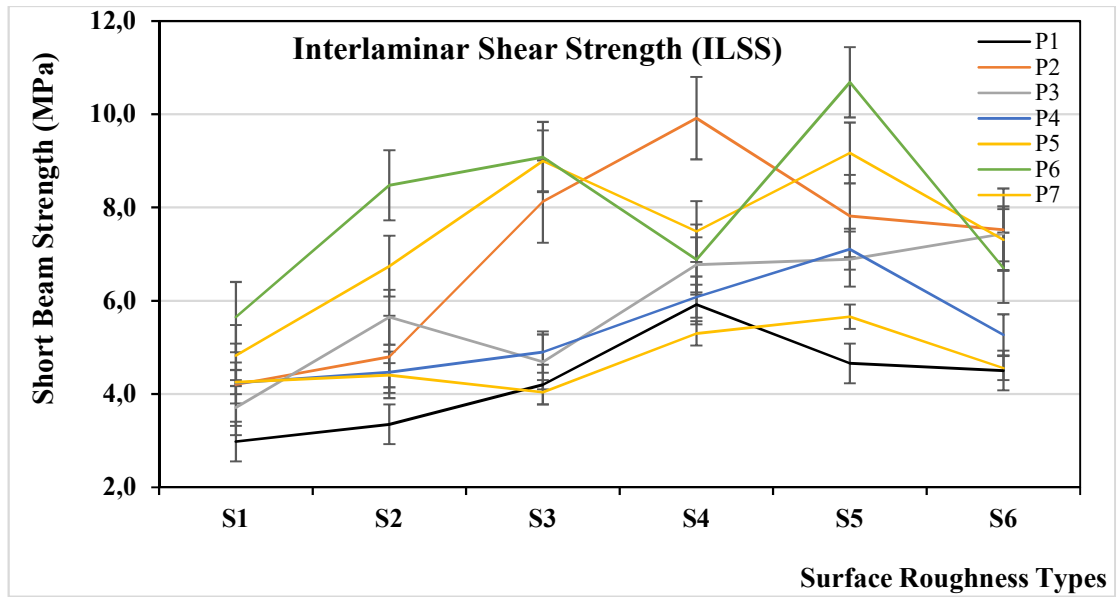


Figure 3.15: Short beam strength results according to surface roughness

Similar to other test results, surface roughness on aluminium samples and addition of chemical process for joining have a positive effect on ILSS test results.

The minimum value of ILSS results of hybrid samples were obtained on flat aluminium surface.

The detailed overall data of ILSS results are given in Table 3.5.

Table 3.5: Results of short beam strength (MPa)

	P1	P2	P3	P4	P5	P6	P7
S1	3.0	3.7	4.8	4.3	4.2	5.7	4.2
S2	3.4	5.6	6.7	4.4	4.8	8.5	4.5
S3	4.2	4.7	9.0	4.0	8.1	9.1	4.9
S4	5.9	6.8	7.5	5.3	9.9	6.9	6.1
S5	4.7	6.9	9.2	5.7	7.8	10.7	7.1
S6	4.5	7.4	7.3	4.6	7.5	6.7	5.3

3.3. Thermal Tests

3.3.1. Coefficient of Linear Thermal Expansion (CTE)

Two physical characteristics (displacement and temperature) were measured on samples that were going through a thermal cycle to estimate the coefficient of linear thermal expansion (CTE) for hybrid test samples.

Figure 3.16 and Figure 3.17 indicated that the test specimens expand upon heating and contract when cooled. The displacements on surface of each test samples have been measured after 50°C temperature difference. Depending on joining structure, the displacements were measured and CTE values were calculated.

Due to the these results, the mechanical effects on surface and chemical processes provide lower CTE value.

When these results are evaluated according to surface roughness, S4 and S5; for process side P3 and P6 samples have lower values in average as shown in Figure 3.16.

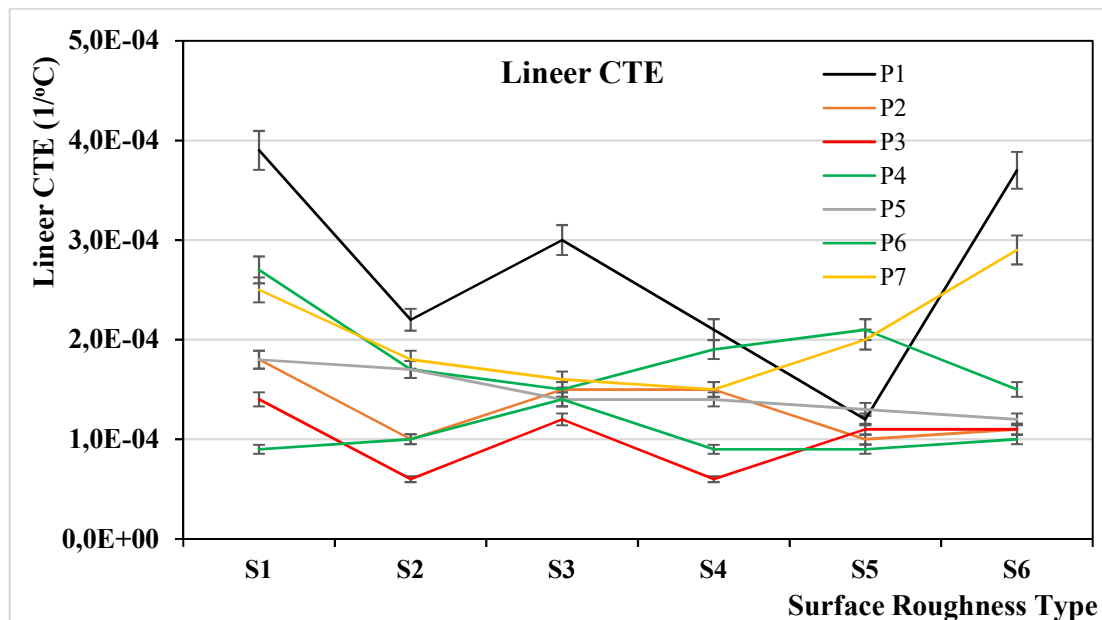


Figure 3.16: Results of linear CTE from 20°C to 70 °C based on surface roughness

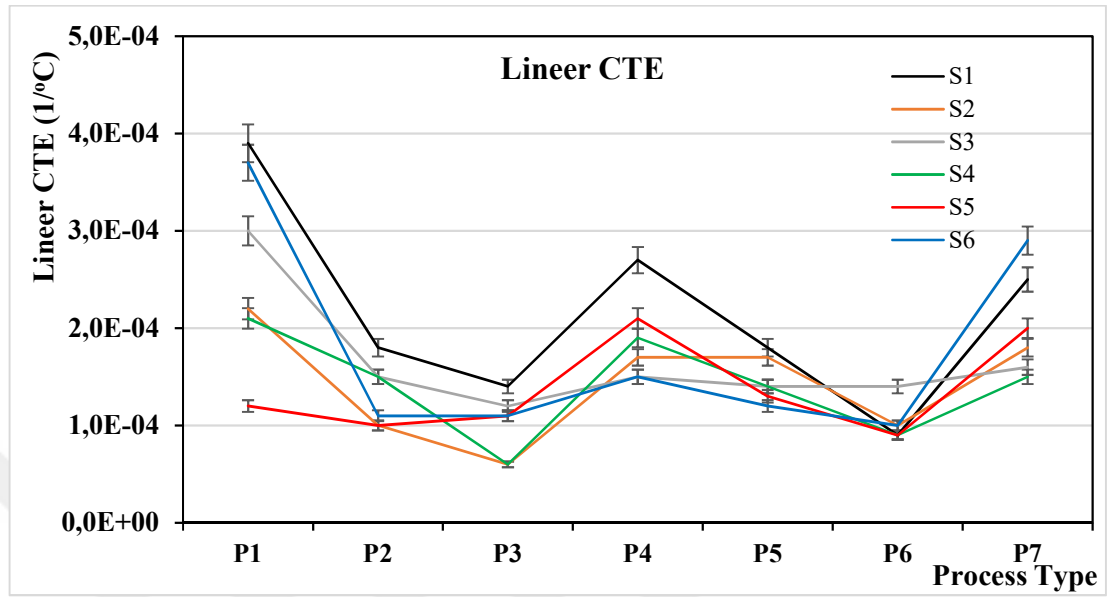


Figure 3.17: Results of linear CTE from 20°C to 70 °C based on process type

The overall data of CTE results are given in Table 3.6.

Table 3.6: Overall results of mean CTE($10^{-4}/^{\circ}\text{C}$)

	P1	P2	P3	P4	P5	P6	P7
S1	3.9	1.8	1.4	2.7	1.8	0.9	2.5
S2	2.2	1.3	0.6	1.7	1.7	1.0	1.8
S3	3.0	1.5	1.2	1.5	1.4	1.4	1.6
S4	2.1	1.5	0.6	1.9	1.4	0.9	1.5
S5	1.2	1.0	1.1	2.1	1.3	0.9	2.0
S6	3.7	1.1	1.1	1.5	1.2	1.0	2.9

CTE of each specimens were calculated by measuring length from 20 °C (T_o) to 70°C (T_f) with 10°C increasing temperature intervals. The results were analyzed in two different categories with roughness and process approaches.

S2 - non-roughness specimen was resulted as the largest in length changes according to temperature increases, and the best result with less variation was measured in S5 sample. The overall graph and data of length variation results based on roughness were given in Figure 3.18 and Table 3.7.

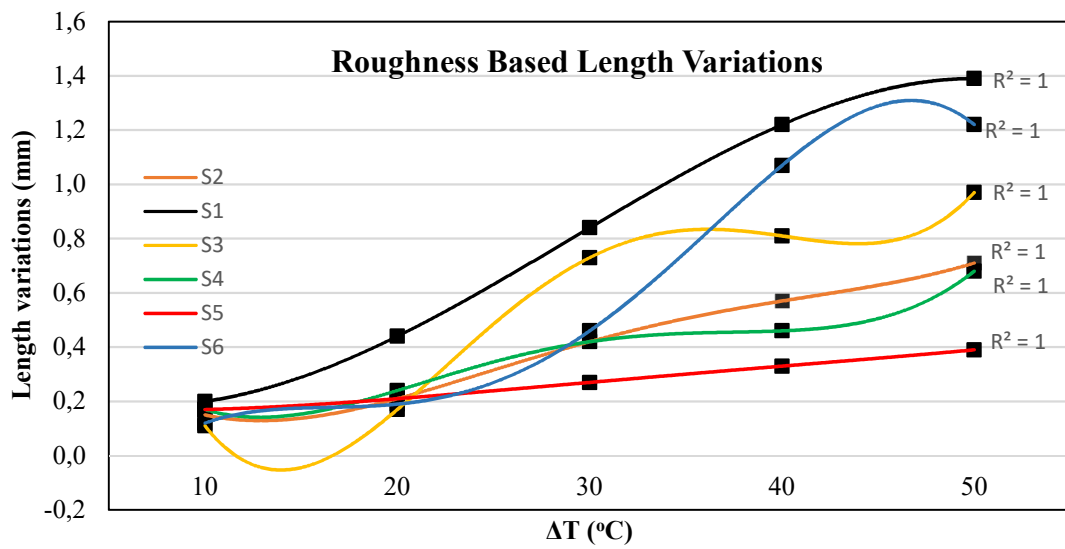


Figure 3.18: Polynomial approach (5th degree) for roughness based length variations

Table 3.7: Length variations according to process types at different ΔT (mm)

	10 °C	20 °C	30 °C	40 °C	50 °C
S1	0.20	0.44	0.84	1.22	1.39
S2	0.15	0.21	0.42	0.57	0.71
S3	0.11	0.17	0.73	0.81	0.97
S4	0.17	0.24	0.42	0.46	0.68
S5	0.17	0.21	0.27	0.33	0.39
S6	0.12	0.19	0.46	1.07	1.22

Length changes were evaluated for each process specimens at temperature differences.

The results indicated that the biggest length change according to temperature increases was without additional treatment specimen, and the best result was measured in P3 sample, where the change was less. The polynomial approaches and data of length changes results based on process were given in Figure 3.19 and Table 3.8.

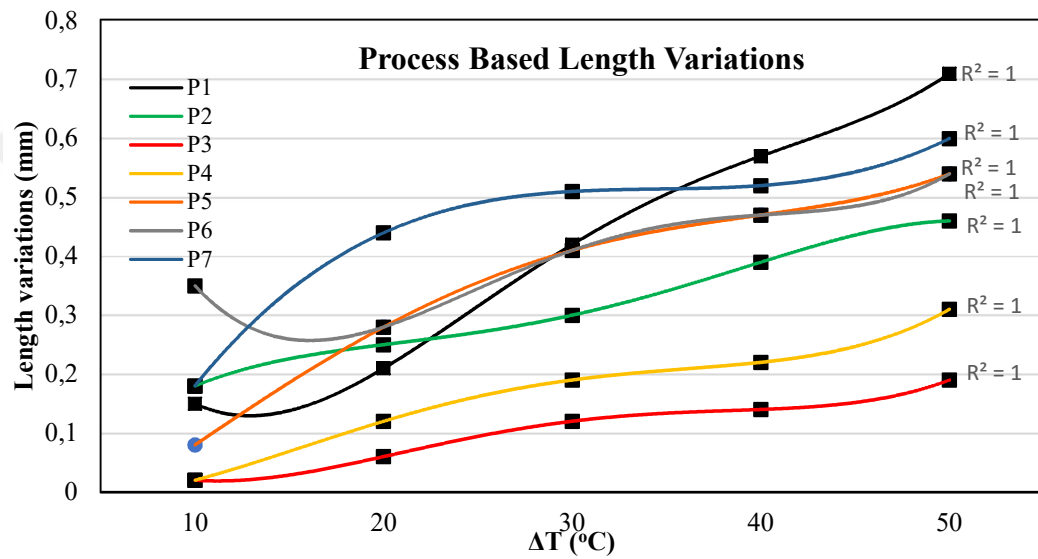


Figure 3.19: Polynomial approach (5th degree) for process based length variations

Table 3.8: Length changes (S2) according to process types at different ΔT (mm)

	10 °C	20 °C	30 °C	40 °C	50 °C
P1	0.15	0.21	0.42	0.57	0.71
P2	0.02	0.12	0.19	0.22	0.31
P3	0.02	0.06	0.12	0.14	0.19
P4	0.18	0.25	0.3	0.39	0.46
P5	0.35	0.28	0.41	0.47	0.54
P6	0.08	0.28	0.41	0.47	0.54
P7	0.18	0.44	0.51	0.52	0.60

3.4. Microscopic Analysis

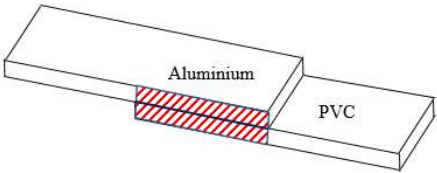
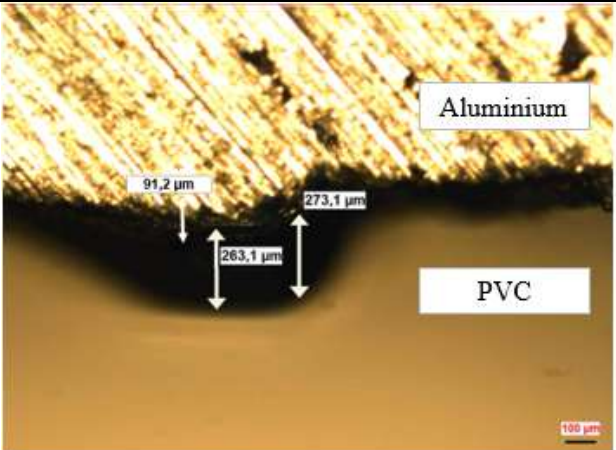
3.4.1. Optical Microscope

The surface microstructure characteristics of the samples were measured using an optical microscope technic. Some pictures from interface section of hybrid samples are dimensioned in Optical Microscope.

S1- Flat aluminium surface;

On flat samples, Figure 3.20 illustrates the gaps between the PVC and aluminium interface regions. In some local areas of the interface, there is a gap of nearly 263 μm , which has a significant impact on the result of the sample, which has no surface roughness.

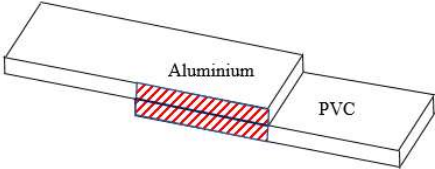
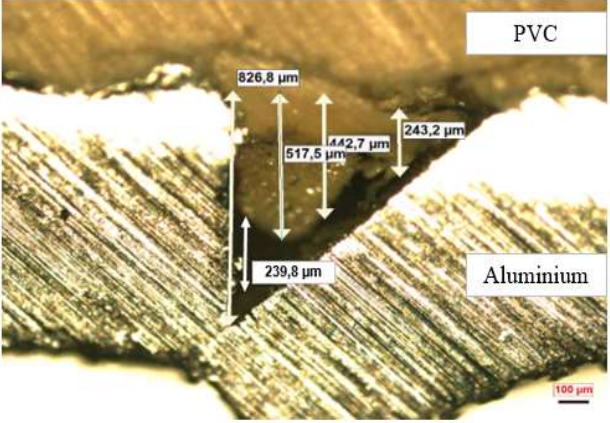
Table 3.9: Optical microscope result of interface on S1

Surface Type & Sampling Section	Result
 S1  Section of sampling	

S5 - Surface deformed aluminium;

In Figure 3.21, optical microscope result of S5 hybrid sample is shown. OM picture indicates that PVC material penetrated through the cavity on aluminium surface. The dimension of PVC penetration was measured as 517.5 μm which provides an advantage as interlocking on test results. But, there is still void (239.8 μm) on this joining structure where PVC can not fully fill since it is closed gap with air. Air in these gaps could not be replaced with PVC.

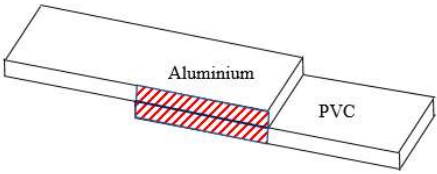
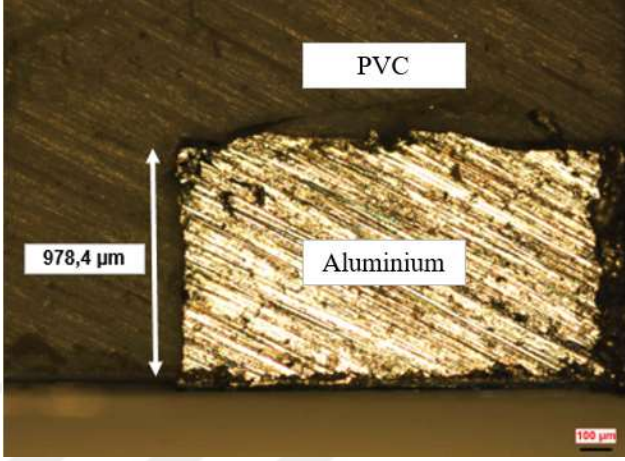
Table 3.10: Optical microscope result of interface on S5

Surface Type & Sampling Section	Result
 S5  Section of sampling	

S2 Side punched aluminium surface;

The optical microscope output for sidepunched aluminium samples is shown in Figure 3.22. OM result presents on cross section that removed part of aluminium were filled by PVC material. The depth of penetrated PVC was measured to be 978.4 mm, which is the best interlocking mechanism among the other surface roughness.

Table 3.11 : Optical microscope result of interface on S2

Surface Type & Sampling Section	Result
 Aluminium Punched Surface  Section of sampling	

3.4.2. SEM Micrograph

Hashemia et al.2020 has studied on “Investigation on the mechanical behavior of fiber-metal laminates based on polyvinyl chloride reinforced by 3D glass fibers”. PVC is utilized to fill the interior space of 3D glass fibers, and PVC films are used to adhere the layers together. FMLs were prepared at a variety of temperatures, pressures, and processing times. The mechanical properties of FMLs were examined and analyzed, including their tensile and flexural strength. The best impregnation and bonding were observed on the fracture surfaces of the tensile and bending areas of the flexural test, according to scanning electron microscopy (SEM) imaging. SEM (Scanning Electron Microscope) pictures of the specimens were obtained to look into the fracture mechanism, deformation process, and layer sliding movement [106]. Similar to this aim, SEM micrographs in our study were used to have an identical idea on improvement on interface layer structures. The fractured state in cross section indicates weaker bonding between the PVC and the aluminium, resulting in non-uniform force distribution between the fibers and local fracture, which leads to failure.

The SEM micrographs have been prepared on two different samplings. First micrograph was done on crosscut section of interface layer before delamination. Second one was done on delamination surface of PVC and aluminium which are shown in Table 3.12.

Table 3.12: Description of SEM samplings

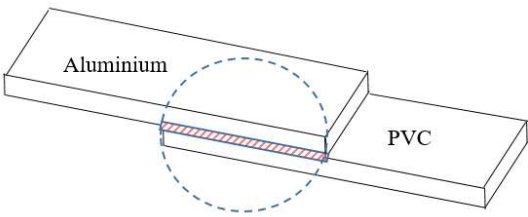
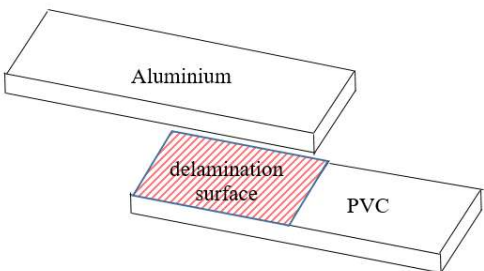
SEM 1 – Crosscut section of interface layer before delamination	SEM 2 -Delamination surface
	

Table 3.13 presents 2 pieces P3 crosscut sections in SEM micrographs. P3 which includes only priming process has been selected to observe roughness on interface. Cross sections were clearly showed in Table 3.13 that the primer has good effect on increasing roughnes. These micrographs on crosscut specimens were recorded X150, X500 and X1000.

Table 3.13: SEM micrographs for P3 specimen crosscut section

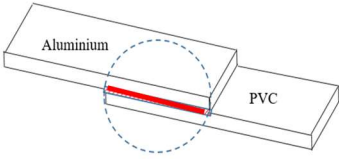
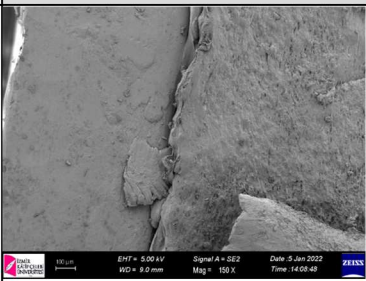
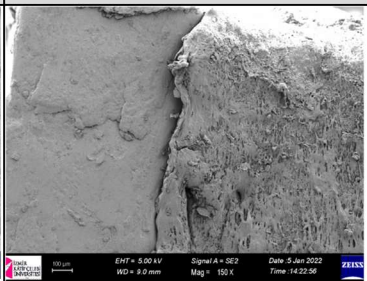
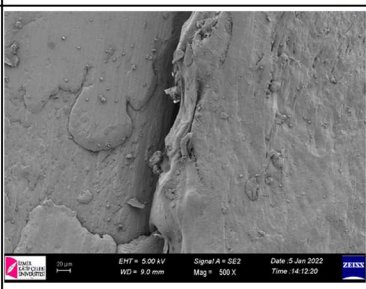
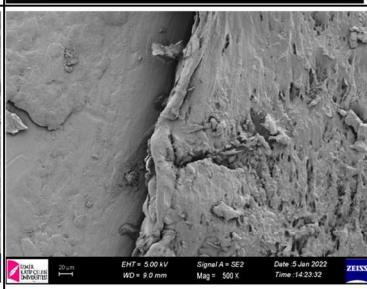
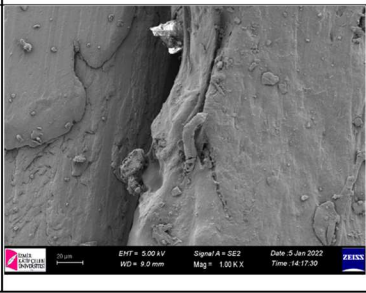
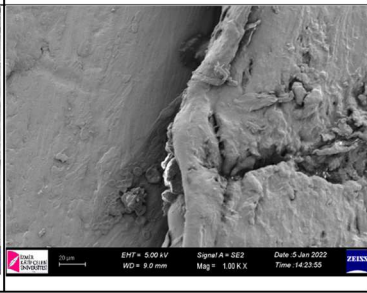
	Crosscut section 1	Crosscut section 2
X 150		
X 500		
X 1000		

Table 3.14 presents the cross section of P7 sampling which is primed adhesive process. Used adhesive showed an important impact with filling joining layer. These micrograph on crosscut specimens were recorded X150, X500, X1000, X2000 and X3000. Although X3000 magnifying was provided, there was no any void or gap observed on interface layer. This SEM analysis has confirmed the good test results of adhesive application which caused the interlock mechanism to be strengthened on joining structure.

Table 3.14: SEM micrographs for P7 specimen crosscut section

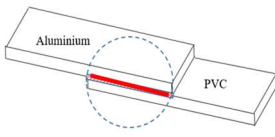
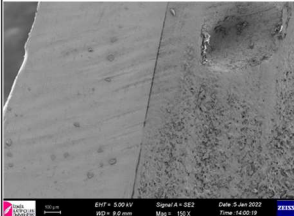
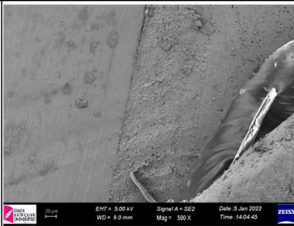
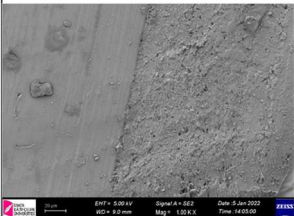
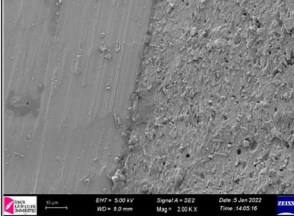
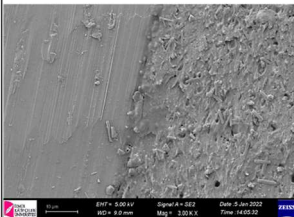
	Crosscut section
X 150	
X 500	
X 1000	
X 2000	
X 3000	

Table 3.15 present SEM micrographs for delaminated surfaces of PVC and aluminium specimens were recorded X100 and X250 respectively. Process descriptions have been mentioned as P2, P3, P5, P6 and P7. Table 3.16 magnifies part of Table 3.15.

Table 3.15: SEM micrographs for delaminated surface- X100 of (a) P2-PVC, (b) P2-Alum, (c) P3-PVC, (d) P3-Alum, (e) P5-PVC, (f) P5-Alum, (g) P6-PVC, (h) P6-Alum, (i) P7-PVC, (j) P7-Alum

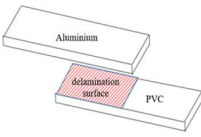
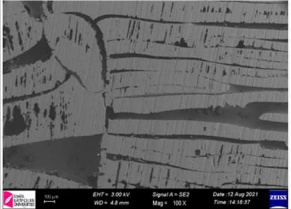
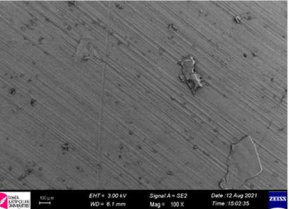
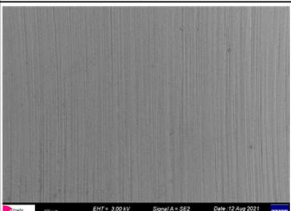
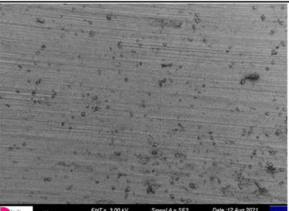
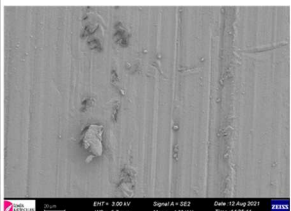
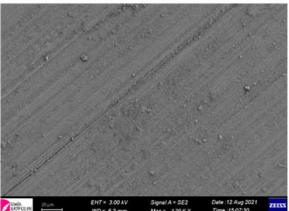
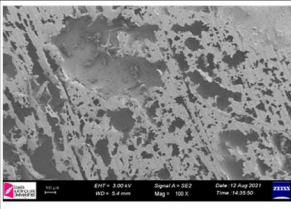
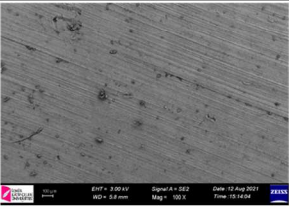
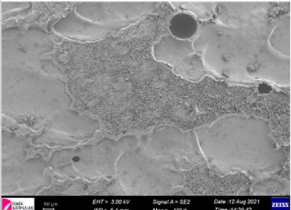
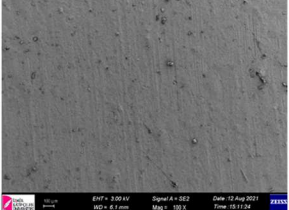
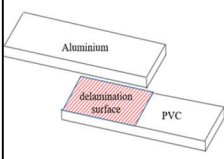
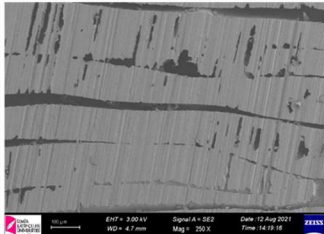
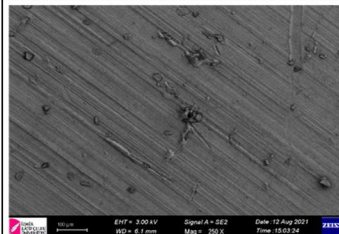
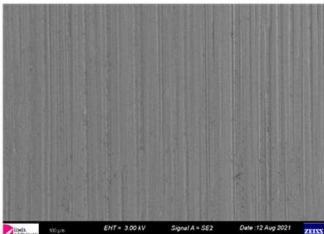
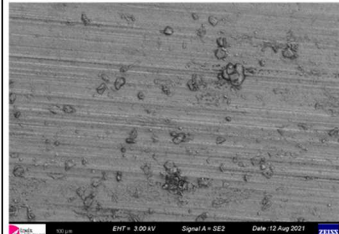
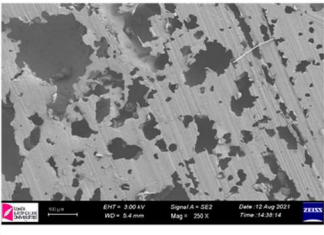
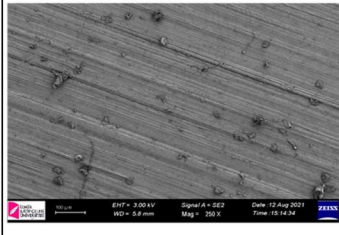
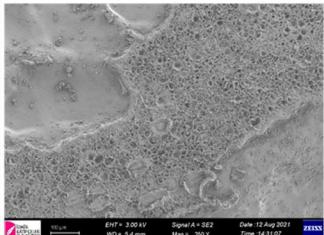
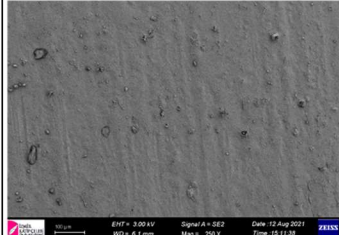
	PVC surface	Aluminium surface
	X 100	
P2	 (a)	 (b)
P3	 (c)	 (d)
P5	 (e)	 (f)
P6	 (g)	 (h)
P7	 (i)	 (j)

Table 3.16: SEM micrographs for delaminated surface- X250 of (a) P2-PVC, (b) P2-Alum, (c) P3-PVC, (d) P3-Alum, (e) P5-PVC, (f) P5-Alum, (g) P6-PVC, (h) P6-Alum, (i) P7-PVC, (j) P7-Alum

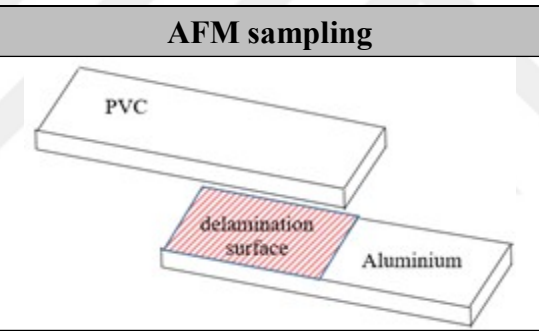
	PVC surface	Aluminium surface
	X 250	
P2	 (a)	 (b)
P3	 (c)	 (d)
P5	 (e)	 (f)
P6	 (g)	 (h)
P7	 (i)	 (j)

3.4.3. AFM

A sample's amplitude parameters are specified by parameters that provide information about statistical average values, histogram height shape, and other extreme properties. The mean height determined over the full measured length/area is the average roughness (Ra). The vertical distance between the highest and lowest points in the evaluated length/area is the maximum peak to valley height roughness (Rt), which represents the overall roughness of the surface [95, 107].

AFM micrographs for aluminium surface which has P7 primered-adhesive process were presented in Figure 3.20 and 3.21. Sampling was done on delaminated surface of aluminium.

Table 3.17: Sampling description of AFM



The morphology outcomes of this analysis shows the roughness on surface after delamination. The roughness on surface topology is supporting the obtained test results.

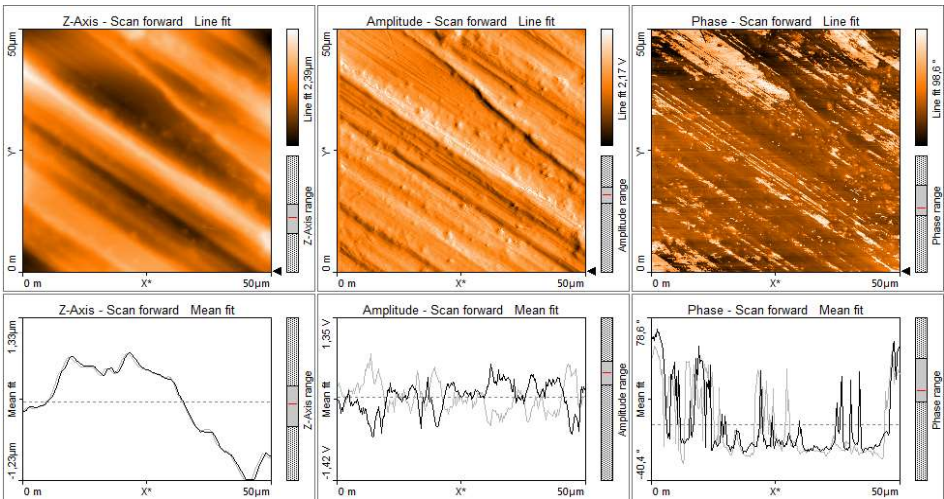


Figure 3.20: AFM micrograph of aluminium surface on primered adhesive process for scanning areas 50µm

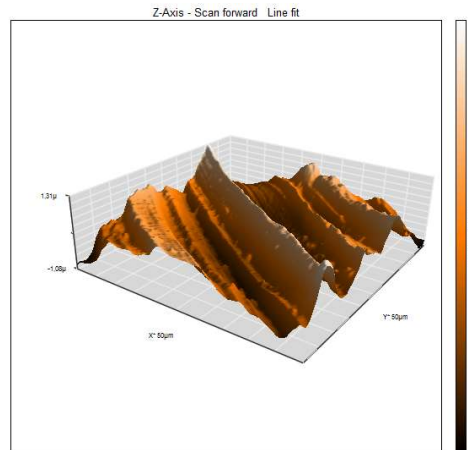


Figure 3.21: Surface topology on aluminium surfaces

The results of area and line roughnesses of AFM analysis have been given in Table 3.18.

Table 3.18: Results of surface roughness in AFM analysis

-- Area Roughness --		-- Line Roughness	
Area	2,52 nm ²	-	-
Sa	239,89 nm	Ra	267,83 nm
Sq	319,06 nm	Rq	355,92 nm
Sy	1881,7 nm	Ry	1,554 Åµm
Sp	955,49 nm	Rp	751,11 nm
Sv	-926,17 nm	Rv	-802,93 nm
Sm	-14,092 fm	Rm	-13,984 fm

Chapter 4

4. Conclusion

The joining process which is considered as an important topic in many different application areas and different segments appears in many details of daily life. There are different materials used together with joining in the contents of different product groups, from computers to wood products, from paper industry to building materials.

In this study, the idea of having hybrid of PVC and aluminium a solid product within improved joining structure that can replace the steel sheet. Current commercial systems have the following drawbacks into the profile with additional labor costs, accelerate the window manufacturing time, is based on the idea of PVC windows and doors. However, the modulus of elasticity of steel is approximately 3 times higher than aluminium and this aluminium can provide a monolithic structure with PVC. It has a positive reduction on the deflection values in the moment of inertia for hybrid materials when using aluminium in multiple structures. For this reason, it was necessary to initiate a study with enhanced bonding properties at the PVC and aluminium interface.

The numerous joining procedures of two distinct materials were prepared as a consequence of this investigation and the surface roughness and extra processes that generated the optimal results were determined. Prior any experimental study, design of experiment methodology was exploited for all the parametric studies. In the study, which aims to strengthen the joining between PVC and aluminium, the good results were obtained from the samples of the metal surface, with side punched and perforated aluminium surfaces and the adhesion values of the samples subjected to the primer and adhesive process, with a positive increase of around 40%.

At the beginning of the study, the different surface morphologies performed on the aluminium surfaces to see the effect of roughness on the results. Six different geometry types as S1 (flat-no any roughness), S2 (side punched), S3 (perforated), S4 (perforated and side punched), S5 (surface deformed) and S6 (surface deformed and side punched) surface were chosen. The specimens with side cuts and perforated holes with interlock mechanisms resulted in the expected direction, where shear forces appear to be difficult due to the plastic raw material filling the emptied space. This view was also supported by cross-sectional images taken with an optical microscope. In the side punched samples, it was observed that PVC and aluminium formed an interlocked structure which provides advantage on hybrid structure. Contrary to expectations, in the surface deformed samples, the PVC material could not fill the deformed area on the surface, since the air is trapped inside. Therefore, the results of surface deformed(S5) specimens were not as successful as expected.

As a second step in improvement of interface layer, additional processes on treatment of aluminium or any materials that can support this joining have been investigated. Seven process types have been identified which are P1 as reference (no additional process), P2 (emulsion PVC), P3(primer), P4 (plasma) , P5 (primered E-PVC), P6 (primered adhesive), P7 (primered plasma).

Because the PVC and aluminum joining as hybrid structured product will be operated during the extrusion process, hotpress equipment was developed to achieve similar process conditions. Following a series of parametric testing, it was agreed to preheat PVC for 15 minutes at 70°C and aluminum for 5 minutes at 190°C. The optimum values were obtained by combining both specimens heating at 190 °C for 15 minutes and compression time for 5 minutes. All the aluminium surfaces (S1,...S6) and the process types (P1,...,P7) were joined at these conditions.

When the results are studied on a process basis, it becomes clear that whether the surface priming technique is used alone or in combination with other processes, the pretreatment of the aluminium surface has a significant impact. Cleaning and preparing the metal surface for the next procedure had a favorable impact on the results. On the treatment part for aluminium, the alternatives of primers to be utilized, as well as their suitability for the process, were investigated. The effect of the appropriate primer alone

was examined, and it was understood that to use it as a pre-treatment for other processes as emulsion PVC, adhesive and plasma.

In the interface development studies, a material close to the PVC polymer family that can also provide adhesion on metal was investigated. Since it is well known that E-PVC (%48 PVC) is employed in the impregnation of several PVC and glass fibre hybrid products, the emulsion PVC was used in our study. The results show that E-PVC coating on aluminium surface increased the values positively.

The effect of adhesive was investigated with primer treatment in the process types (P6). The PUR based hotmelt adhesived specimens had resulted well in lap-shear, 3P bending and thermal tests.

Plasma (P4), which was considered as another process, resulted below the expected values in the study. Plasma's pore-opening property on metal surfaces is well known, and its impact on interface adhesion was included in the study processes. However, in practice, the parameters such as the distance of the plasma to the surface and the amount of energy transferred should be examined and the experiments should be planned in more details in order to obtain better results in further studies.

Several tests have been performed to determine the performance of joining in hybrid products. Lapshear, ILSS and 3P bending tests were examined to focus on mechanical properties deviation. In a thermal experiment, CTE test was set to see how enhancing the joining affects the thermal expansion behavior. Microscopic analysis as optical microscope, SEM and AFM were indicated the information on interface with micrographs.

The outputs were analysed in DOE using with full factorial design techniques. Minitab software was used for DOE studies. Main effects plots were created as an average results of each conditions.

The main effect plots for lapshear test results were summarized that S2, S3 and S4 samples in surface types, P3, P6 and P7 in process types have resulted as higher than 2 MPa which was 1.08 MPa without any roughness and 0.59 MPa without treatment. Additional processes have an improvement on lap-shear results. In the second main effect plots for 3P bending indicated that decrease of the deflection after surface

modifications and treatments. S4, S5 and S6 in surface types, P5, P6 and P7 in process types have lower deflection with the result of good bonding properties.

Third main effects plot for ILSS, S3 and S5 ; P2,P5 and P7 specimens were resulted as higher values. In contrast to desired expectation, sample treated with plasma as P4 and P6 had lower results. Plasma needs to have some optimisation especially on applying time. Since optimum time which has been explained longer in literature was not provided, the results were not acceptable.

Last main effect plot was created for CTE which is a critical for the products needs to exposed to outside weathering conditions. Therefore, the thermal expansion of each material for hybrid structure must be examined if the value in limitation at certain temperature differences. The results revealed that S2,S4 and S5; P2, P3 and P4 have lower coefficients among other types.

The lap shear results obtained in the study indicated that the hybrid products based on the primer and adhesive application process, with S2 and S4 (side cut and perforated surface) shaping, highlighting the adhesion properties of aluminium and PVC materials, will be available to use in the industry of different markets.

Microanalysis have been provided on optical microscope, SEM and AFM. The datas obtained from micrographs of them allowed for a more accurate estimation of how the surface topology created at the interface impacts on adhesion. For example, when S1 and S5 graphs of optical microscope were compared as cross-section, the cavities which were noticed in S5, the fractures and delaminations occurred faster in mechanical tests since these voids were not filled with PVC material. In S1 which has certain side cuts, has been observed as fully the interlock mechanism in micrograph, resulted as higher values in experiments as desired direction.

SEM analysis had been setup on cross section of interface layer before delamination and on surface after delamination. When the interface SEM micrograph of P3(primered) sample has been examined, the voids owing to surface abrasion were noticed on cross section which means PVC did not fill these gaps well. On the other hand, for P7 (primered adhesive) specimen, it was noticed that the gaps created by the primer on the interface were completely filled with adhesive and both materials are integrated properly at SEM graph magnified to X3000.

SEM of the delamination surface specimens show that the blocks caused by adhesion were visible, particularly on the PVC surface of the samples utilizing primer and adhesive.

The other micro-analysis has been arranged for AFM in the study. The surface topology and surface roughness values have been evaluated with other test results.

Considering all of the parameters and findings of this study, it has been determined that the surface roughness has a critical effect on joining structure, and that mechanical and thermal results can be improved by adding additional processes to the specified surface.

When the findings of these studies are analyzed, new coupling techniques or parameter adjustments are still in discussion, and the potential implications of new applications on the results will be investigated further in the study's subsequent steps.

In further steps of this study, it is thought that the interface formation on the coated aluminium surfaces has a positive effect on the bonding. For this reason, the coating on aluminium surface with 60-80 μm , will be included in the next steps of the work. Cathaphoresis coating applications on the aluminum surface will be examined as an additional stage in the study. Also, the acid catalysed melamine cross linked saturated aromatic polyesters have known as good adhesives on improving interface layers. In further study, it will be examined to have commercial hybrid products for building markets.

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Improvement of Interface in PVC and Aluminium Structures

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Doctoral Thesis

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