ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE ENGINEERING AND TECHNOLOGY

MINI AUTOCLAVE SYSTEM FOR COMPOSITE CURING PROCESS

M.Sc. THESIS

Mehmet ÇAKICI

Department of Electrical & Electronical Engineering

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ISTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

KOMPOZİT MALZEME KÜRLEME SÜRECİ İÇİN MİNİ OTOKLAV SİSTEMİ

YÜKSEK LİSANS TEZİ

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Kontrol ve Otomasyon Mühendisliği Anabilim Dalı

Kontrol ve Otomasyon Mühendisliği Programı

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Mehmet ÇAKICI, a M.Sc. student of İTU Graduate School of Control & Automation Engineering student ID 504141119, successfully defended the thesis entitled "MINI AUTOCLAVE SYSTEM FOR COMPOSITE CURING PROCESS", which he prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

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To my curious younger self,

“A person who never made a mistake never tried anything new!”

Albert EINSTEIN

FOREWORD

First and foremost, I would like to thank my supervisors Assist. Prof. Dr. Ali Fuat ERGENÇ and Assist. Prof. Dr. Hülya CEBECİ who have shown plenty of encouragement and support to me at every point of my studies. I would like to special thanks Assist. Prof. Dr. Hülya CEBECİ for making it possible for me to benefit from the ITU Aerospace Research Center opportunities and giving inspirational ideas at every part of my master degree. Additionally, I would like to thank my life partner Nurdan İrem ÜNAL for not giving up her priceless and endless mental support at every point of my master degree. I also would like to thank my family and friends for their supports.

June 2017 Mehmet ÇAKICI

(Physics Engineer)

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ABBREVIATIONS

AC : Alternative Current
AISI : American Iron and Steel Institute
A_S : Area of Surface
atm : Atmospheric Pressure
CAD : Computer Aided Design
CNC : Computerized Numerically Controlled
Cp : Heat Capacity
DC : Direct Current
g : Gravitational Constant
h : Heat Transfer Coefficient
Hz : Frequency
J : Joule
K : Kelvin
k :Thermal Conductivity
K_D : Derivative Constant
kg : Kilogram
K_I : Integrator Constant
K_P : Proportional Constant
L_C : Characteristic Length
LED : Light Emitted Diode
mA : Milliamper
min : Minutes
mm : Millimeter
MOSFET : Metal Oxide Semiconductor Field Effect Transistor
mPa : Millipascal
mV : Millivolt
N : Newton
Nu : Nusselt Number
P : Proportional Controller
PD : Proportional – Derivative Controller
PI : Proportional – Integral Controller
PIC : Peripheral Interface Controller
PID : Proportional – Integrator – Derivative
PLC : Programmable Logical Controller
Pr : Prandtl Number
PWM : Pulse Width Modulation
Ra : Rayleigh number
RT : Real Time Communication
SISO : Single Input – Single Output
SSR : Solid State Relay
T_∞ : Infinity Time
TC : Thermocouple

T_F : Final Time
T_S : Initial Time
UV : Ultraviolet
V : Volt
W : Watt
α : Thermal Diffusivity
ρ : Density

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MINI AUTOCLAVE SYSTEM FOR COMPOSITE CURING PROCESS

SUMMARY

Composite materials which are formed of at least two constituents are highly dependable to manufacturing quality. Since the composites are two-phased, custom methods are preferred to fabricate them even in coupon test specimen level. One very widely used method is the autoclave curing of them under high temperature and pressure which helps to achieve minimum voids and very good quality.

Today, autoclave systems are frequently used in systems requiring the application of temperature, pressure and vacuum processes together. Autoclaves are produced in desired sizes according to the physical dimensions of the processes and they are generally used in sterilization, vulcanization and curing based production. The application areas are wide ranging from tool sterilization in the medical sector to production of much larger structures in the aerospace sector.

As a system for different purpose uses, sterilization autoclaves considers the temperature control as a priority however for advanced composite manufacturing autoclaves, temperature, pressure and vacuum control are the major controlling mechanisms, respectively.

The design and fabrication parameters of autoclaves depends on the field of use for the instrument but generally, temperature, pressure and the amount of vacuum are the most important aspects. Automatic control devices provide a complex control of the system for the requirements of user/process by adjusting important parameters within time. Proportional, integral and derivative (PID) control algorithm is generally used in autoclave systems where the temperature, pressure and vacuum must be changed stepwise depending on the time. In order to use this algorithm, the mathematical model of the system needs to be extracted first. After the mathematical model of each part of the system is derived as differential equations, it is converted to Laplace domain and the transfer function of the system is obtained, respectively. This transfer function is used to design the PI controller of the autoclave system mathematically and/or in a simulation environment. The PI control algorithm, designed for the autoclave system, controls the system by implementing an electronic controller. The controllers for the required algorithm might be programmable logic controllers (PLC), microprocessors and micro-controllers, as well. The temperature required by autoclaves is provided by electricity and/or flammable gases. The heater source is selected according to the size of the autoclave and the maximum temperature it must reach. While sterilization autoclaves are generally heated by electricity, the autoclaves which are vulcanizing and producing composite are heated by electricity and/or flammable gases.

The purpose of the autoclave systems in aerospace industry is to meet the requirement of very high quality production of large-sized composite parts. These parts are produced by curing the composite fabrics prepared as layers, under temperature, pressure and vacuum, to give strength. In order to increase the mechanical

performances of thermoset-based composite materials, it is necessary to increase the fiber/resin ratio or completely eliminate the air gaps formed in the material during production. The remaining voids during production reduce the strength of the part. In standard composite manufacturing methods, the part is produced under 1 atm of air pressure. During composite production by vacuum bagging, the air gaps in the material can be reduced to minimum if the part is under a high air pressure which is regular, controllable and more than 1 atm. Autoclave systems are devices with temperature and vacuum control that can be applied to the external pressures needed to increase the physical properties of the composite material produced. The amount of external pressure varies according to the type of composite to be produced and is designed and manufactured according to this requirement in the autoclave system. Since the parts that need to be produced in the aviation sector are large in size, autoclave systems can be also designed and manufactured within these requirements.

The purpose of the autoclave systems used in the aerospace industry is the requirement of very high quality production of large-sized parts, especially those used in the construction of air vehicles. These parts are produced by curing the composite fabrics prepared as layers, under temperature, pressure and vacuum, to give strength. In order to increase the mechanical performances of thermoset-based composite materials, it is necessary to increase the fiber/resin ratio or completely eliminate the air gaps formed in the material during production. The remaining voids during production reduce the strength of the part. In standard composite manufacturing methods, the part is produced under 1 atm of air pressure. At the moment of composite production by vacuum bagging, the air gaps in the material can be reduced to minimum if the part is under a high air pressure which is regular, controllable and more than 1 atm. Autoclave systems are devices with temperature and vacuum control that can be applied to the external pressures needed to increase the physical properties of the composite material produced. The amount of external pressure varies according to the type of composite to be produced and is designed and manufactured according to this requirement in the autoclave system. Since the parts that need to be produced in the aviation sector are large in size, autoclave systems can be also designed and manufactured within these requirements.

In this thesis, the designed and produced autoclave system aims to cure composite materials in the temperature, pressure and vacuum environment. The autoclave produced is much smaller (30x30x5 cm) than the size of the autoclaves used in the industry. The main reason for this small work area is the ability to produce composite materials that have been researched and developed for use in the aerospace industry in coupon specimen dimensions for the mechanical testing defined through American Society of Testing Materials (ASTM) standards. The ability to produce composite materials with different structural components in the research and development phase is of great importance for the mechanical characterization of prototype materials. The purpose of producing composites is to have more flexible, lighter, higher strength, and more flexible materials than the previous material type for aerospace applications. The autoclave to be designed and implemented in this thesis will serve to achieve high quality composite specimens for mechanical testing indicated within the standards mentioned.

The autoclave designed and produced in this thesis has a working temperature of maximum 250°C, 7 bar pressure and -1 bar vacuum environments and the system should not undergo plastic deformation at the relevant temperature and pressure. The autoclave has to withstand the forces generated at the inner surface of the chamber at a high temperature and pressure. During these forces applied to the inner surface, the autoclave system must deformed in elastic region. When the forces in the inner surface are removed, the material can be fully restored to its original shape. The wall thickness analysis of autoclaves was made on the basis of this criterion.

While the system is operating at maximum operating conditions, the amount of flexure of the material from which the autoclave produced must remain in the elastic deformation zone. For this reason, stainless steel metal is preferred for the autoclave produced in this thesis. The wall thickness of the autoclave is optimized by Von Misses analysis and the diameter of the bolts, which connecting the housing that cover the device to the main body, are again calculated by Von Misses analysis. The amount of heat required for the device to perform the relevant production processes is calculated and the plate heater is preferred to provide this heat.

After the autoclave design and production were completed, composite material production tests were carried out and the instrument was calibrated. This special autoclave is used at Istanbul Technical University, Aerospace Research Center (ITUARC), under the development of several research and development project that focuses on the mechanical property investigation of nano-engineered and polymer nanocomposites.

KOMPOZİT MALZEME KÜRLEME SÜRECİ İÇİN MİNİ OTOKLAV SİSTEMİ

ÖZET

En az iki bileşenden oluşan kompozit malzemeler, imalat kalitesine oldukça bağımlıdır. Kompozitlerin iki fazlı olması nedeniyle, kupon test örneği seviyesinde bile onları imal etmek için özel yöntemler tercih edilmektedir. Çok yaygın olarak kullanılan bir yöntem yüksek sıcaklık ve basınç altında otoklavlar aracılığıyla kürlemedir; bu da boşlukların minimum olmasını ve kalitenin çok iyi elde edilmesine yardımcı olur.

Günümüzde otoklav sistemleri, sıcaklık, basınç ve vakum süreçlerinin birlikte uygulanmasını gerektiren sistemlerde sıklıkla kullanılmaktadır. Otoklavlar işlemlerin fiziksel boyutlarına göre istenilen boyutlarda üretilir ve genellikle sterilizasyon, vulkanizasyon ve kür esaslı üretimde kullanılırlar. Uygulama alanları, tıbbi alanda donanım sterilizasyonu ile havacılık sektöründe çok daha büyük yapıların üretilmesi arasında geniş bir yelpazede bulunmaktadır.

Birçok farklı amaçla kullanılmak üzere üretilmiş olan otoklav sistemlerinin yanı sıra, sterilizasyon otoklavları için öncelikli olan sıcaklık kontrolüyken; ileri kompozit üretimi için tasarlanan otoklavlarda başlıca kontrol mekanizmaları sıcaklık, basınç ve vakum kontrolüdür.

Otoklavların tasarım ve imalat parametreleri cihazın kullanım alanına bağlıdır, ancak genel olarak sıcaklık, basınç ve vakum miktarı en önemli hususlardır. Otomatik kontrol cihazları, önemli parametreleri zaman içinde ayarlayarak sistemin kullanıcı / süreç gereksinimleri için karmaşık bir kontrol sağlar. Oransal, integral ve türev (PID) kontrol algoritması, sıcaklık, basınç ve vakumun zamana bağlı ve kademeli olarak değiştirilmesi gereken otoklav sistemlerinde genellikle kullanılır. Bu algoritmayı kullanmak için önce sistemin matematiksel modelinin çıkarılması gerekir. Sistemin her bir bölümünün matematiksel modeli diferansiyel denklemler olarak türetildikten sonra sırasıyla Laplace uzayına dönüştürülür ve sistemin transfer fonksiyonu elde edilir. Bu aktarım işlevi, otoklav sisteminin PI kontrolörünü matematiksel olarak ve / veya simülasyon ortamında tasarlamak için kullanılır. Otoklav sistemi için tasarlanmış PI kontrol algoritması, bir elektronik kontrol cihazına yüklenerek sistemi kontrol eder. Gerekli algoritmanın denetleyicileri, programlanabilir mantıksal denetleyiciler (PLC), mikroişlemciler ve mikro denetleyiciler olabilir.

Otoklavların gerektirdiği sıcaklık; elektrik ve / veya yanıcı gazlar tarafından sağlanır. Isıtıcı kaynağı, otoklavın boyutuna ve ulaşması gereken maksimum sıcaklığa göre seçilir. Sterilizasyon otoklavları genelde elektrikle ısıtılırken, vulkanize olan ve kompozit üreten otoklavlar elektrik ve / veya yanıcı gazlar tarafından ısıtılır. Yanıcı gazların kullanıldığı büyük otoklav sistemleri genelde yüksek sıcaklıkları, uzun süreler boyunca gerektirir. Bu sebeple ısıl kapasitesi yüksek gazlar, ebatça büyük otoklavlarda

tercih edilir. Elektriğin ve yanıcı gazların aynı anda kullanıldığı otoklav sistemlerinde genelde elektrik ön ısıtıcı olarak kullanılmakta, yanıcı gazlar ise sıcaklıkta devamlılık sağlamaktadır. Sadece elektriğin kullanıldığı otoklavlar ise genellikle küçük ebatlarda üretilirler.

Havacılık endüstrisinde kullanılan otoklav sistemlerinin amacı, özellikle hava taşıtlarının yapımında kullanılan büyük boyutlu parçaların çok kaliteli üretilme gereksinimidir. Bu parçalar, sıcaklığın, basıncın ve vakumun altında katman olarak hazırlanan kompozit kumaşların sertleştirilmesi ile üretilir. Termoset tabanlı kompozit malzemelerin, mekanik performanslarını arttırmak amacıyla elyaf /reçine oranını arttırmak veya malzeme içerisinde üretim anında oluşan hava boşluklarının tamamen giderilmesi gerekmektedir. Üretim sırasında kalan boşluklar, parçanın mukavemetini düşürmektedir. Standart kompozit üretim metodlarında, parça 1 atm lik hava basıncı altında üretilmektedir. Vakum torbasıyla kompozit üretimi anında parça düzenli, kontrol edilebilir ve 1 atm den yüksek bir basınç altına alınırsa, malzeme içerisindeki hava boşlukları minimuma indirilebilir. Otoklav sistemleri üretilen kompozit malzemenin fiziksel özelliklerini arttırmak için ihtiyacı duyulan dış basıncı uygulayabilen, sıcaklık ve vakum kontrolü bulunan cihazlardır. Dış basıncın miktarı, üretilecek olan kompozitin türüne göre değişmekte ve otoklav sistemide bu ihtiyaca göre tasarlanıp üretilmektedir. Havacılık sektöründe üretilmesi gereken parçaların boyutları büyük olduğundan, otoklav sistemleri de bu gereksinimler doğrultusunda tasarlanıp üretilebilmektedir.

Bu tezde tasarlanan ve üretilen otoklav sistemi sıcaklık, basınç ve vakum ortamında kompozit malzemeleri iyileştirmeyi amaçlıyor. Üretilen otoklav, endüstride kullanılan otoklavların boyutundan çok daha küçüktür (30x30x5 cm). Bu küçük çalışma alanının başlıca nedeni, ASTM (American Society of Testing Materials) standartlarına göre mekanik test için kupon örnek boyutlarında havacılık endüstrisinde kullanılmak üzere araştırılmış ve geliştirilmiş olan kompozit malzemeler üretme kabiliyetidir. Araştırma ve geliştirme aşamasında farklı yapısal bileşenlere sahip kompozit malzemeler üretme kabiliyeti, prototip materyallerin mekanik karakterizasyonu için büyük önem taşır. Kompozit malzemelerin üretilmesi amacı, havacılık uygulamaları için önceki malzeme türüne göre daha esnek, daha hafif, daha yüksek mukavemetli ve daha esnek malzemeler sunmaktır. Bu tezde tasarlanacak ve uygulanacak olan otoklav, bahsedilen standartlarda belirtilen mekanik test için yüksek kaliteli kompozit numuneler elde etmeye hizmet edecektir.

Bu tezde tasarlanan ve üretilen otoklav çalışma sıcaklığı maksimum 250^{o}C, 7 bar basınç ve -1 bar vakum ortamlarına sahiptir ve sistem ilgili sıcaklık ve basınçta plastik deformasyona uğramamalıdır. Otoklav, odanın iç yüzeyinde yüksek sıcaklık ve basınçta üretilen kuvvetlere dayanmak zorundadır. İç yüzeye uygulanan bu kuvvetler sırasında otoklav sisteminin plastik bozulma yaşamadan esnemesi gerekmektedir. İç yüzeydeki kuvvetler ortadan kalktığında malzeme eski haline tam manasıyla geri dönebilmelidir. Otoklavın cidar kalınlığı tasarımı bu kritere bağlı olarak yapılmıştır.

Sistem maksimum çalışma koşullarında çalışırken, otoklavın ürettiği malzemenin bükülme miktarı elastik deformasyon bölgesinde kalmalıdır. Bu nedenle, bu tezde üretilen otoklav için paslanmaz çelik metal tercih edilir. Otoklavın duvar kalınlığı Von Misses analizi ile optimize edilmiş ve cıvataların çapı, cihazı ana gövdeye bağlayan mahfazayı birleştirerek Von Misses analizi ile tekrar hesaplanmıştır. Cihazın ilgili üretim proseslerini gerçekleştirmesi için gerekli ısı miktarı hesaplanmış ve bu ısıyı sağlamak için plakalı ısıtıcı tercih edilmiştir.

Otoklav tasarımı ve üretimi tamamlandıktan sonra, kompozit malzeme üretim testleri gerçekleştirilmiş ve cihaz kalibre edilmiştir. Bu özel otoklav, nano-mühendislik ve polimer nanokompozitlerin mekanik özellik araştırmalarına odaklanmış araştırma ve geliştirme projelerinin geliştirilmesi amacıyla İstanbul Teknik Üniversitesi Havacılık ve Uzay Araştırmaları Merkezi'nde (ITUARC) kullanılmaktadır.

1. THE LITERATURE OF AUTOCLAVE

Autoclaves are containers that allow the application of temperature pressure and vacuum environment at the same time to inside of vessel. These devices provide for step by step adjustment of temperature, pressure and vacuum. Autoclave systems are mainly used in sterilization, curing production and vulcanization processes, as well as a number of industrial areas. Autoclaves are produced in different sizes and mechanisms according to the area to be used. There are autoclaves from the desktop dimensions that are the production area to the size that can produce airplane wings.

1.1 What Is Autoclave?

The autoclave is a high temperature and pressure resistant device and was invented by Charles Chamberland in 1879 as shown in Figure 1.1 [1]. The autoclave is basically composed of two main parts, tank and cover. Closing the lid correctly and safely is very important as it is operated under high pressure. In addition to these two parts, the autoclave also includes a thermometer and a barometer to control the temperature and pressure values, which are important parameters for the process involved.

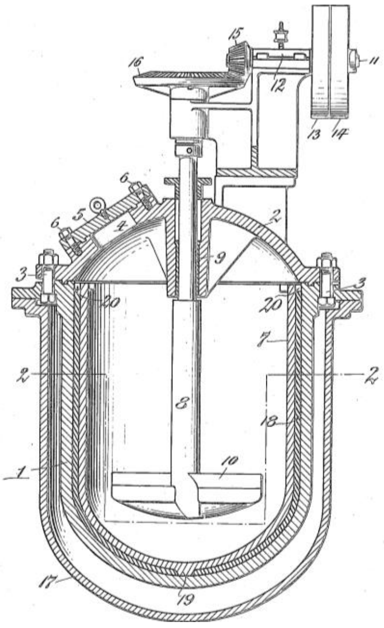


Figure 1.1: Autoclave [2].

To pressurize the autoclave tank, inert gases, usually nitrogen, are used for the purpose to eliminate the possibility of explosion of the cab by reacting gas with any component in the autoclave. The heating process is applied to the tank where different heaters such as electric heater, oil bath or natural gas can be utilized. The heater type is determined according to the temperature value required for the process [3].

Autoclave is a device that has a wide range of uses, from the pressure cooker to the advanced technology and industry. The cooker used in almost every kitchen every day is the simplest and smallest volume autoclave mechanism. On the other hand, autoclave systems are used in the medical field sterilization process and in the chemical production industries. The operating parameters such as temperature, pressure and vacuum can be adjusted depending on the requirements of the process and material [3].

1.2 The Usage Areas of Autoclave Systems

Ease of use, and process quality are the main reasons of autoclave use in several industries. Depending on the process and sector needs autoclaves may be either low cost or very high operational cost. Autoclave systems are generally used in the sterilization process in the medical sector while in the chemical industry they are used in composite curing and vulcanization processes [4].

1.2.1 The Composite Curing Process

Composite materials are manufactured using two or more materials to create new and superior materials in different dimensions. In today's world, the composite materials are generally used in construction industry and air, space, sea and land vehicles. In addition, composite materials are used as an alternative to traditional materials such as iron, steel and aluminium with their specific strength and modulus, rigidity, high impact resistance, high corrosion resistance and infinite fatigue life when it compared to conventional materials [4].

Especially in the aerospace sector, vehicles require optimization of safety, speed, economy criteria to meet the needs of the field to be used. As an example, aircraft must have the high strength and lowest weight possible. From aerodynamic point of view, the most optimum shape and weight of the aircraft is the most important requirement especially for commercial aircrafts. Today, the fuel consumption of passenger aircraft is targeted to be reduced and the most important point here is the weight of the aircraft. Composite materials can alleviate the weight of aircraft without compromising of strength similar to metal alloys. For this reason, composite materials in the aviation sector find a wide range of applications. Composite material usage rate for Boeing and Airbus aircrafts over the years is shown in Figure 1.2 [5].

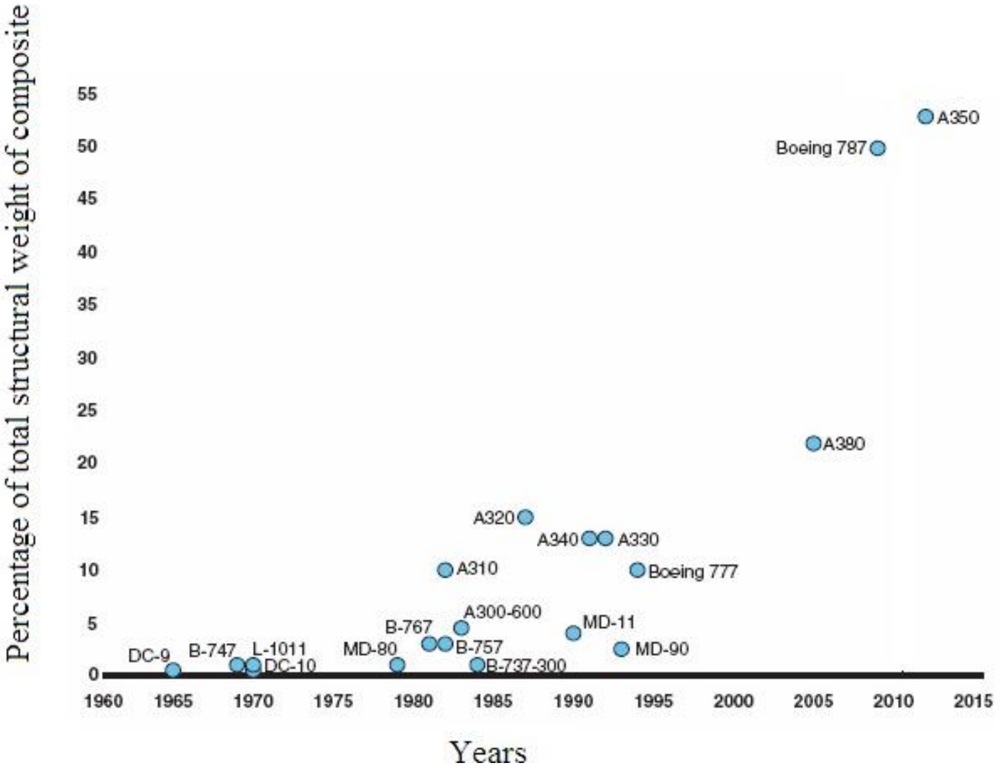


Figure 1.2: Composite material usage rate relative to the total mass of aircrafts [6].

Void-free or minimum void composite manufacturing is one of the key points necessary for high mechanical performance for thermoset based polymer reinforced composite structures. The high temperature and pressure requirements are critical factors in the manufacturing process and within this scope the inner of autoclave is pressurized at higher atmospheric pressures typically at 7-8 bars for aerospace applications [4].

The most important factors affecting the mechanical properties of composite materials are the various production parameters at the production stage. These parameters are materials brought together, temperature, pressure, humidity and production process. Each parameter directly affects the mechanical properties of the composite material. Composite materials are used in a variety of fields, from medical to aviation industry, by changing production parameters. For example, strength and durability are more important in some industries, while elasticity is more important in others. In short, composite production depends entirely on design. The desired material is produced by changing the production parameters according to the requirements of the design. For example, the proportion of composite material used on the Boeing 787 aircraft is shown in Figure 1.3 [7, 8].

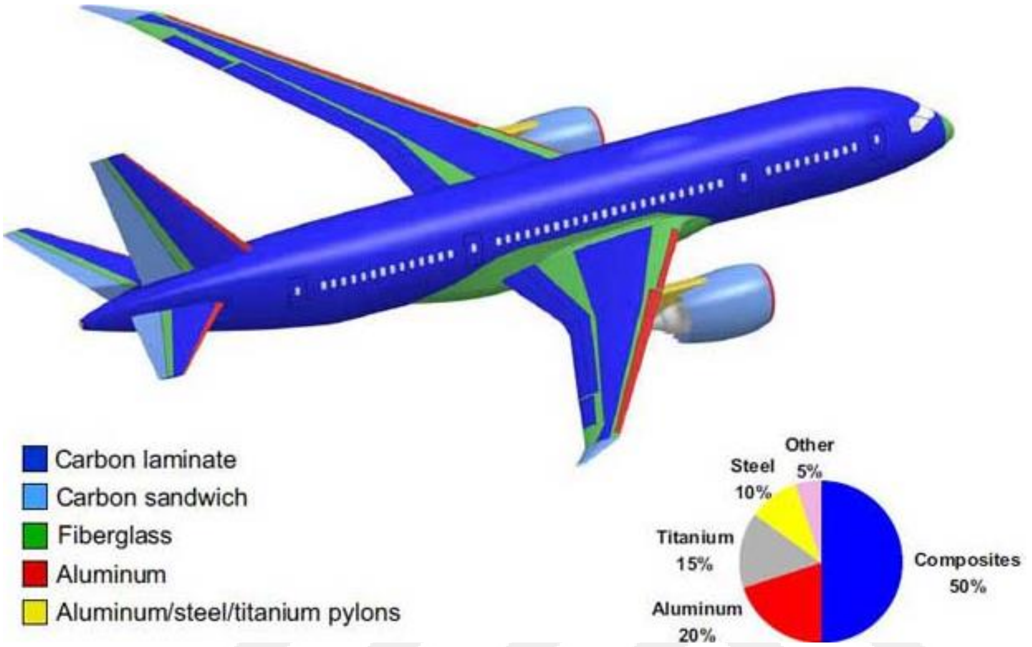


Figure 1.3: The Boeing 787 composite materials using rate.

The curing method should be used to produce composite materials. The curing method is a process of solidifying composite material components under vacuum, temperature or pressure. To achieve this curing process of composite materials, there are two methods: thermal and ultraviolet curing (UV). Since the scope of the thesis is thermoset based polymer composite structures, temperature curing process is the main focus for this study.

1.2.1.1 Thermal Curing

There are a various types of composite processing methods in todays. Composite processing methods are developed for projected mechanical properties of composite materials in time. The general classification of composite processing techniques are shown in **Error! Reference source not found.**. Each type of composite processing t echniques are used in another field of science and industry.

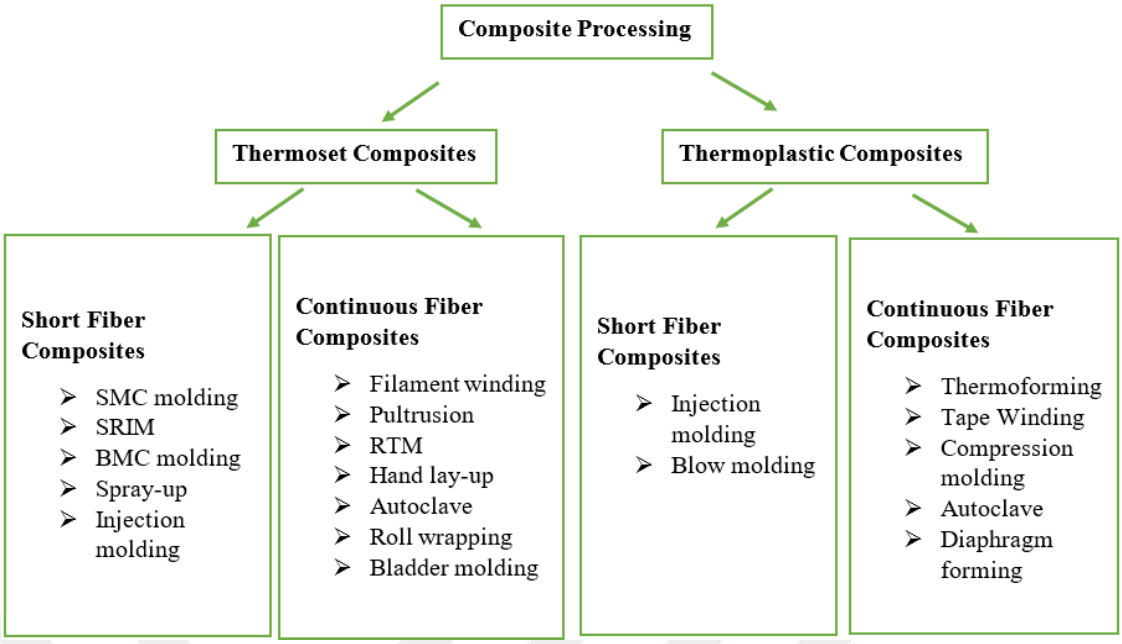


Figure 1.4: Classification of composite processing techniques [1]

The most effective method in terms of research and development of mechanical property investigation of nano-engineered and polymer nanocomposites is an autoclave method which is used for manufacturing of continuous fiber thermoset composite materials. Autoclaving method creates high volume fraction of reinforcement in composite material. This method gives high degree of uniformity in part consolidation and creates better connection between fabric layers. In terms of void content, the autoclaving method generates least voids in composite part than the other manufacturing methods. On the other hand at this method, the fibres are getting wet completely with resin and this situation creates better composition in layers [9]. Composite materials used as structural materials in aviation are traditionally produced by autoclaving [6].

The general production process start with the selection of the desired composite material. Due to the fact that every composite material have different chemical composition, the production stages and needed composite fabrics can be different from each other. In the aerospace sector, generally first step, the needed epoxy resin and composite fabrics are located inside of a vacuum bag layer by layer and this vacuum bag is placed into the autoclave. Second phase is that the autoclave is started working with related temperature, pressure, time and vacuum parameters which is based on required composite material. Finally, the production process is end up and the composite material can be used in related field or research and development studies

[4]. An example for positioning of composite fabrics, vacuum bag and epoxy resin is shown in Figure 1.5.

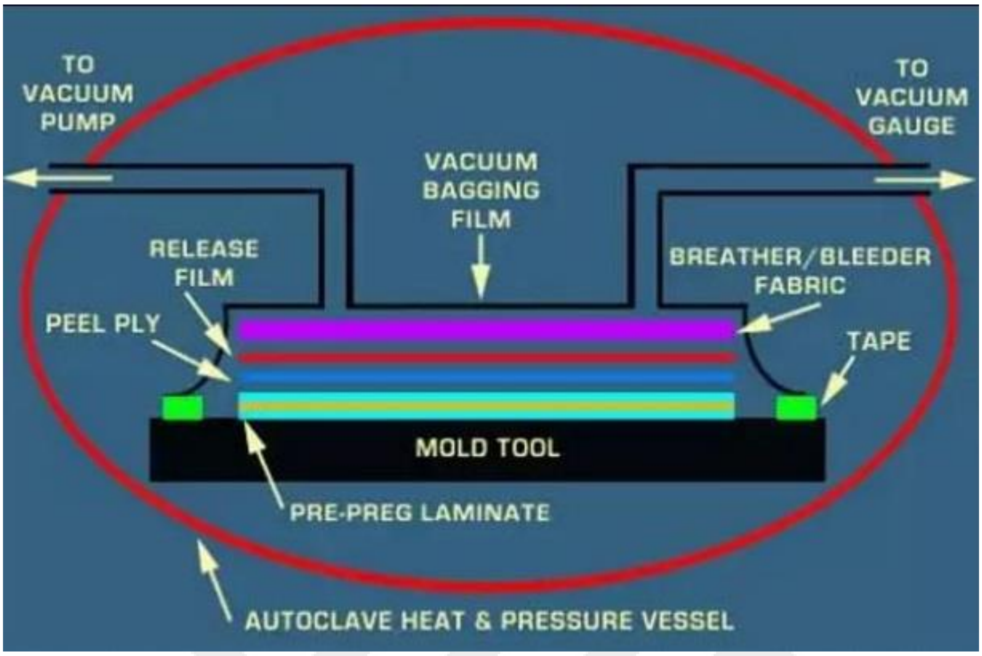


Figure 1.5: The example of positioning of materials

The autoclaving processing of composite materials is type of a thermal curing method. Autoclave curing is performed by keeping the material under vacuum under pressure during the process. For the production of a composite in autoclave, the related composite fabrics are located inside of a vacuum bag with the required epoxy resin. After that, this vacuum bag is located inside of the autoclave vessel and the curing process starts with the related production parameters such as needed temperature and vacuum.

The polymer composite material production is an irreversible chemical reaction between epoxy and hardener. The polymer epoxy resin and hardener creates crosslinks between molecules with using heat energy and pressure [10]. The example crosslinking procedure and curing stages between the molecules resin are shown in Figure 1.6.

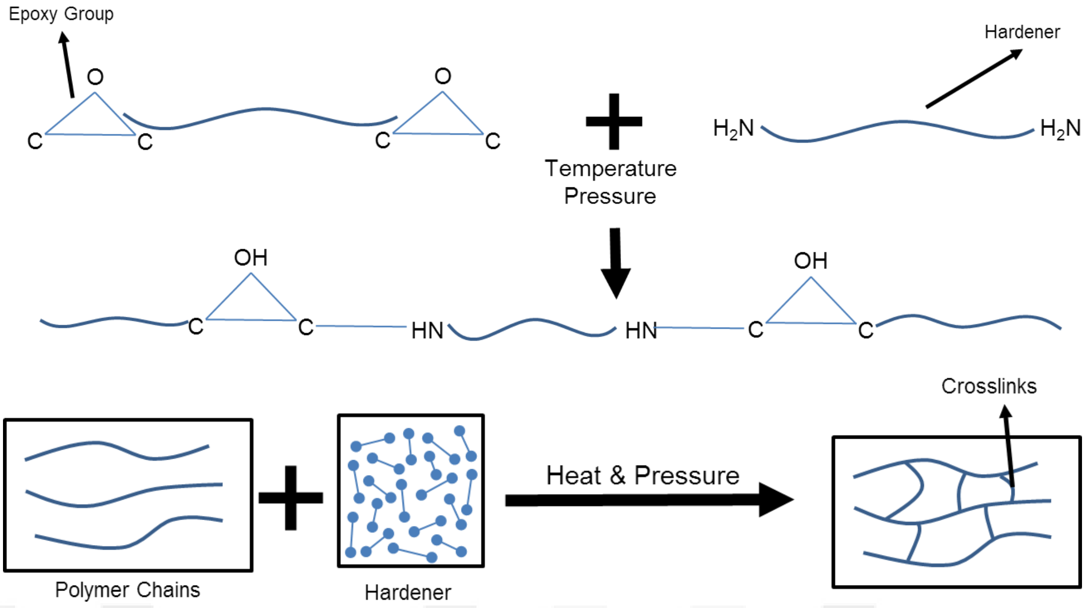


Figure 1.6: The crosslinking stages [11]

The pressure effect in autoclave systems is that the outside pressure on vacuum bag is key point in terms of decreasing of voids in fabrics and epoxy. Because the voids in material after production can creates failure cracks inside of composite under physical stress [12]. These cracks can break the material completely. In short, the voids directly affect the mechanical properties of desired composite material. For example, Judd and Wright reported that a void content as low as 1% results in a decrease in strength up to 30% in bending, 3% tension, 9% torsional shear and 8% in impact [12]. The high pressure importance for void content in autoclave systems is shown in Figure 1.7. [8]. In addition, it is an advantageous method because it enables real time control of the curing process performed in the autoclave and enables the curing of the material in various geometries and sizes [5].

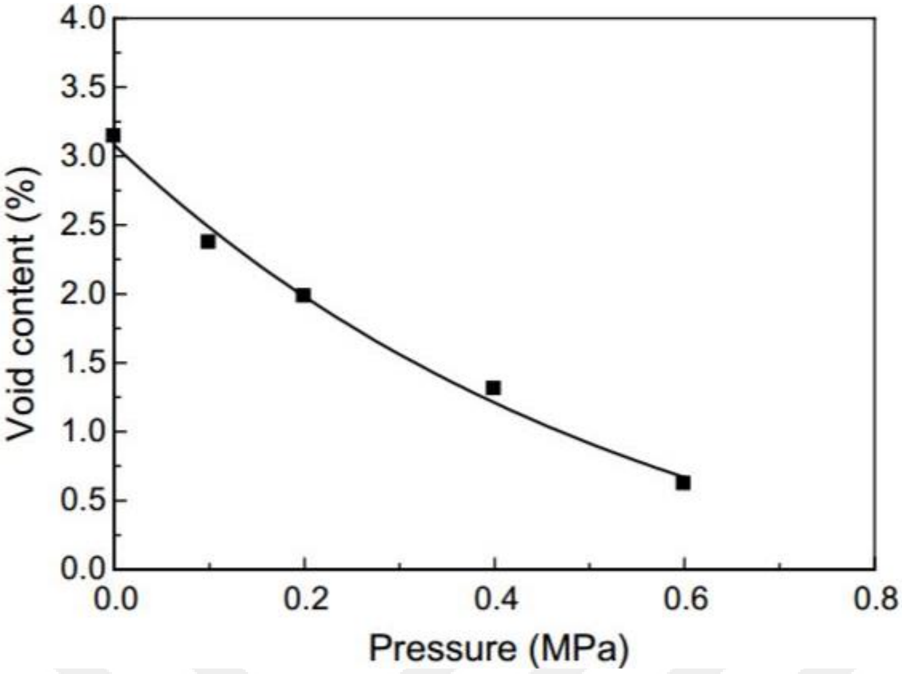


Figure 1.7: Void content rate with respect to pressure [13].

The temperature and time effect is that the process of curing temperature and time in autoclave systems is related with the chemical composition of desired composite material. For example, the autoclaves that cure the composites used in the aerospace industry have normal operating conditions of 120-230 ° C at a nitrogen atmosphere of 7 bar pressure. Aerospace composite structures are mainly focused on carbon fiber and epoxy resins to be processed in autoclaves [8]. The hardening time of the composite extends from 90 minutes to 12 hours depending on the curing kinetics, ratio and as well as chemical composition of the polymer. The viscosity of T700/TDE85 carbon fiber reinforced epoxy prepreg, which have an initial fiber volume fraction is 60 vol%, versus curing time graph is shown in Figure 1.8. The temperature is increasing 2°C per minutes and the viscosity is decreasing step by step. In the A point on graph, the viscosity reached to almost steady state value. Because of that, for this type of example composite, the 2°C per minutes increasing is most effective curing cycle [8, 13]. In short, all of the composite materials have different curing temperature and time which depends on the chemical composition and required mechanical properties.

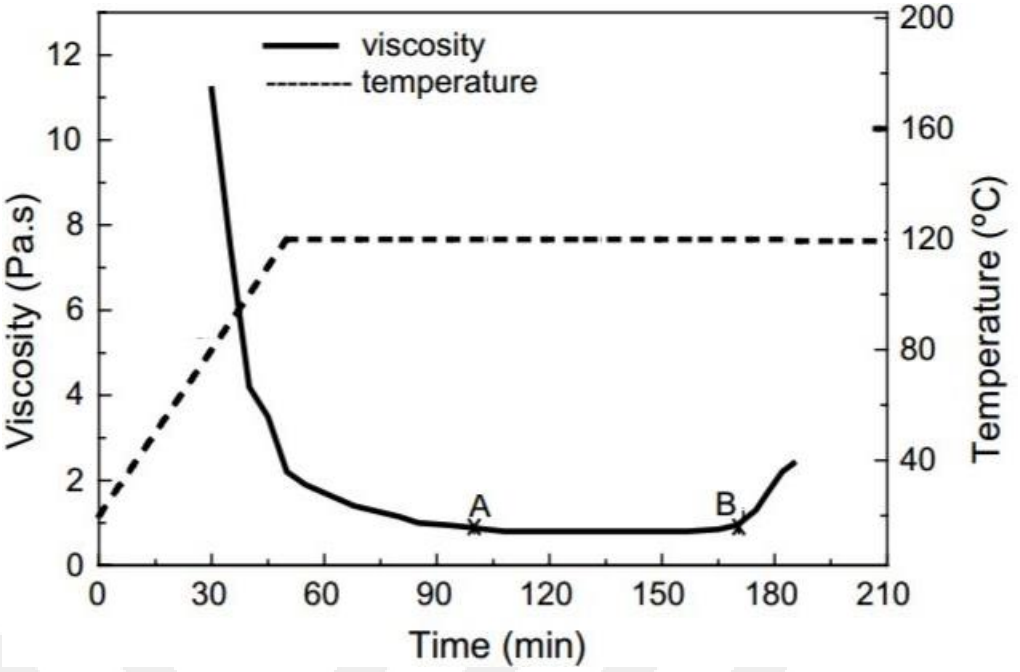


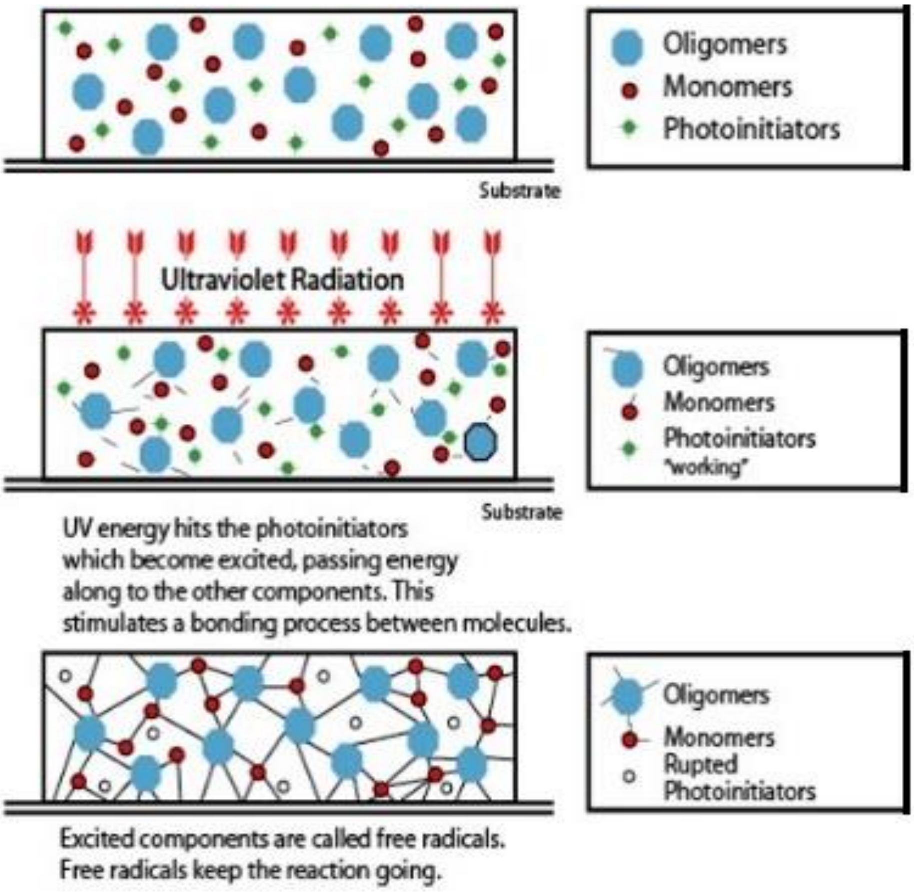
Figure 1.8: The viscosity change in time [13].

The example of large autoclaves for structural components to be used in aerospace, automotive applications is shown in Figure 1.9.

Figure 1.9: Autoclave with high working area [1].

1.2.1.2 Ultraviolet Curing

UV curing has been used in recent years as building material and for dental fillings. In the UV curing method, UV light with a wavelength ranging from 200 to 400 nm is used instead of heat or pressure to harden the composite material. The process is shown in Figure 1.10 [14]. In this process, mercury lamp, UV fluorescence or light emitted diode (LED) is used as UV source. The selection of the UV lamp should be done in consideration of the type of composite to be cured. However, the most preferred UV source is the mercury lamp. The process time is very short with UV curing, but the lamp arrangement is very important for this process to be efficient [14].



UV energy hits the photoinitiators which become excited, passing energy along to the other components. This stimulates a bonding process between molecules.

Excited components are called free radicals. Free radicals keep the reaction going.

Figure 1.10: Ultraviolet Curing Process [14].

1.2.2 The Sterilizing Processes with Autoclaves

Sterilization is the process of destroying microorganisms, such as bacteria, viruses and fungi, and their spores. One of the simplest, most effective and reliable devices that can be used for sterilization is the autoclave. Sterilization autoclaves are used for equipment sterilization in many different sectors such as microbiology laboratories, operating theatres, pharmacy laboratories, veterinary clinics, dental clinics and prosthetic manufacturing sites [15]. The dimensions and operating conditions (temperature and pressure) of the autoclaves vary according to the area used and the size of the equipment to be sterilized. The example of sterilizer autoclave is shown in Figure 1.11.

Figure 1.11: Sterilizer autoclave in medicine [2].

Autoclaves used in the medical sector provide steam sterilization. In this autoclave type, the equipment to be sterilized is exposed to a saturated steam at high temperature. When the saturated steam comes into contact with the environment to be sterilized, the liquid is condensed. Condensation transfers the heat to the material to be sterilized and to any microorganism on its surface. The protein structure in microorganisms exposed to the high temperatures and thus the organism is destroyed [16].

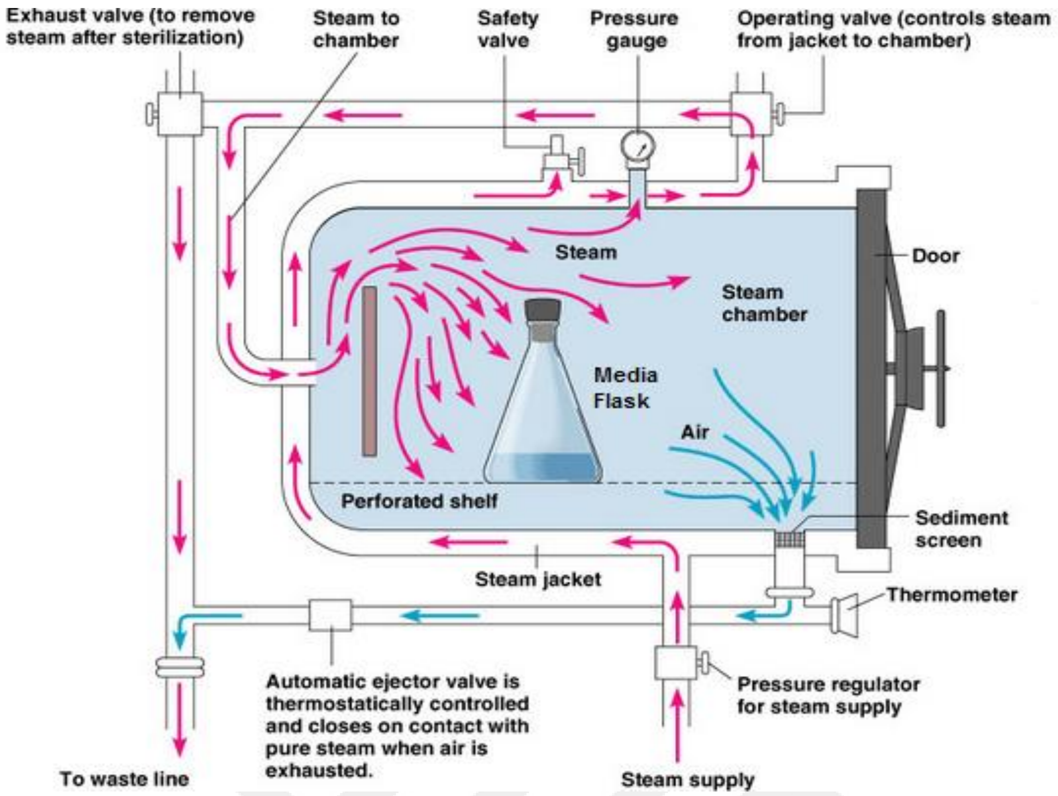


Figure 1.12: Sterilizing mechanism of autoclaves [17].

The factor that causes microorganisms to die in sterilization with autoclave is temperature, not pressure. The temperature required to kill each microorganism species is variational depending on the type of microorganism so a well-defined temperature controlling is necessary. The working mechanism of sterilizer autoclaves is shown in Figure 1.12. For example; the extinction temperature required for vegetative bacteria cells is 75-80 ^{o}C, and the extinction temperature for the same microorganism spores is 100 ^{o}C. Equipment to be sterilized in medical equipment sterilization should be stored in autoclave at 134 ^{o}C for 5 minutes under 2 atm pressure or for 15-20 minutes under 1.5 atm pressure at 121 ^{o}C [17].

1.2.3 The Vulcanization Processes with Autoclaves

Vulcanization is a chemical-technical method discovered in 1839 by Charles Goodyear, one of today's largest tire manufacturers [1]. This is a process in which rubber or similar polymers are converted into materials of higher strength by the addition of sulfur or other equivalent sulfur compounds. Crude rubber, sulfur or compounds containing sulfur in the composition are used for the vulcanization process. In addition to this, various catalysts are used to increase the speed of the reaction [18]. The chemical process of vulcanization is shown in Figure 1.13.

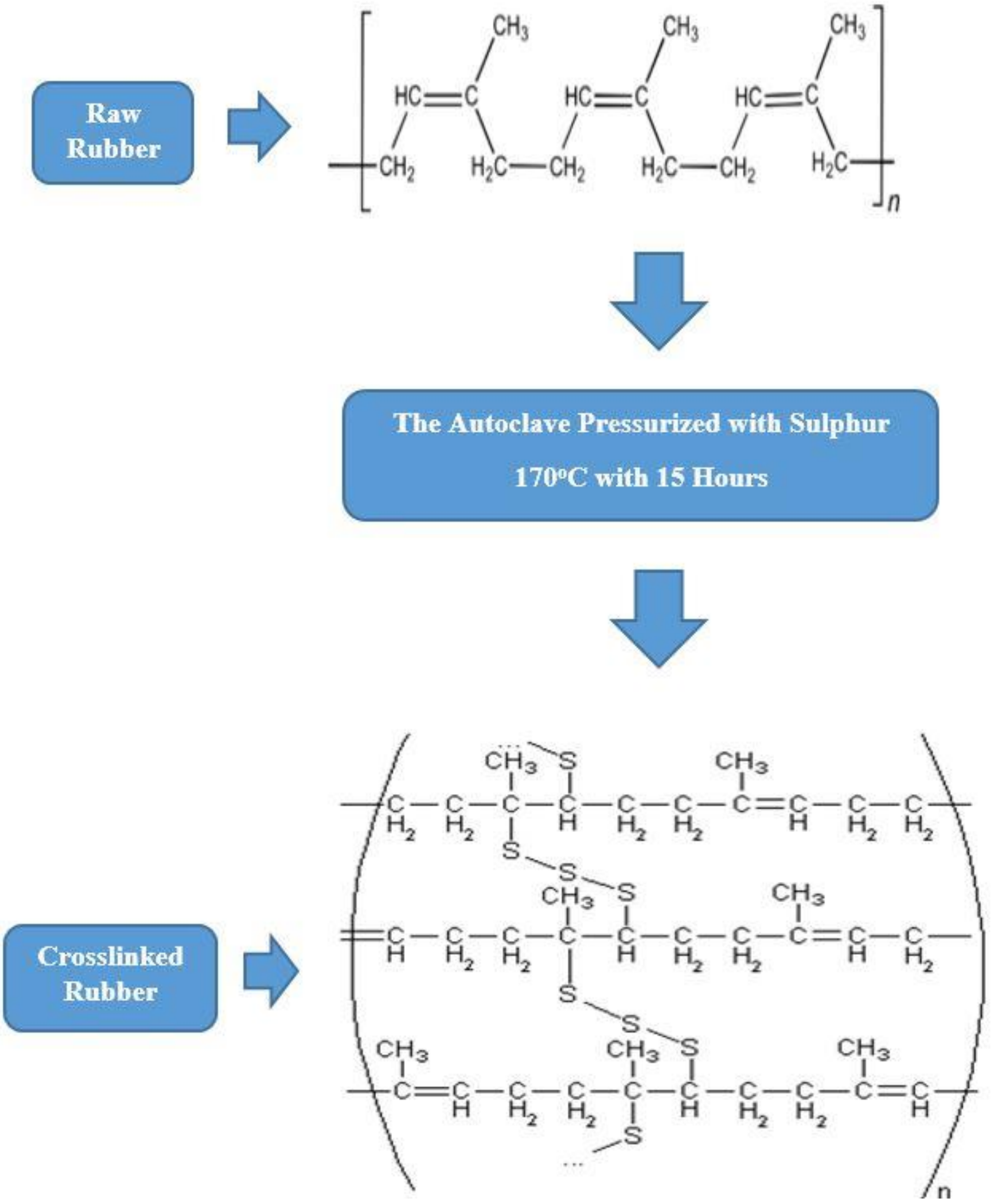


Figure 1.13: Vulcanization process [18].

As the vulcanization process requires high temperature and pressure, autoclave is used for this process. The natural rubber is cured in the autoclave and converted into a chemically cross-linked rubber product. Vulcanized rubber is less sticky and has superior mechanical properties than natural rubber. Vulcanization autoclaves are available in various sizes. The process temperatures and pressures of these devices, which are generally horizontal, vary with the rubber used. However, this process is usually carried out at temperatures of 170 ^{o}C and above. The example of vulcanization autoclave is shown in Figure 1.14 [1].

Figure 1.14: Vulcanization of car tires [1].

The most important component in the vulcanization autoclave is the cover within cost and safety. On the one hand, the operator should be able to open and close the door quickly and easily and the door production and use must meet stringent safety requirements.

Vulcanization in autoclaves is a function of temperature and time. In many conventional applications, there is no indication of when vulcanization is complete. For this reason, the cycle times of the process are usually longer than usual to ensure that all components in the autoclave are universally improved. Modern autoclaves include PLC-controlled systems and offer extended features such as variable temperature, pressure, cycle time, cycle and system alarms, and multi-loop storage.

2. THE MINI AUTOCLAVE SYSTEM FOR CURING PROCESS

In a scope of composite curing process, the special size autoclave system is designed in this chapter. The design is done with the criteria of 30x30x5 cm production volume which help us for the manufacturing of composite materials in coupon size. The related heating energy & natural convection heat loss is also calculated for system. The heater is designed with the criteria of heat calculation value also.

In addition, the wall thickness of system is optimized according to cost and feasibility. The desired autoclave system is working under 7 bar pressure and 180 Celsius degree temperature. The system is designed to withstand 10 bar pressure and 250 Celsius degree temperature because of the safety. At the end of design, the system is produced with using CNC engraving method from stainless steel.

2.1 The Mechanical Design and Analysis

The mechanical design and analyses of the mini autoclave system is performed with SolidWorks design program. The material of the main and the top components (base and cover) of the system are selected as 316 American Iron and Steel Institute (AISI) Stainless Steel, which have a density of 8238 kg/m^3 and specific heat capacity of 468 $J/kg.K^o$ [19]. The material selection is made with respect to the phases of the composite curing process and temperature variations. The composite production process includes maximum 0.7 mPa pressure and maximum 180^oC temperature. The mechanical design must be durable to these pressure and temperature values for the dimensions of 30x30x5cm in production volume. In this part of the design, the mechanical analysis must be examined to determine of the wall thickness of the system. On the other hand, the cost of the stainless steel is so high when it compared with other metal materials. The design have to be optimized in terms of the cost and process. The desired autoclave vessel is shown in Figure 2.1.

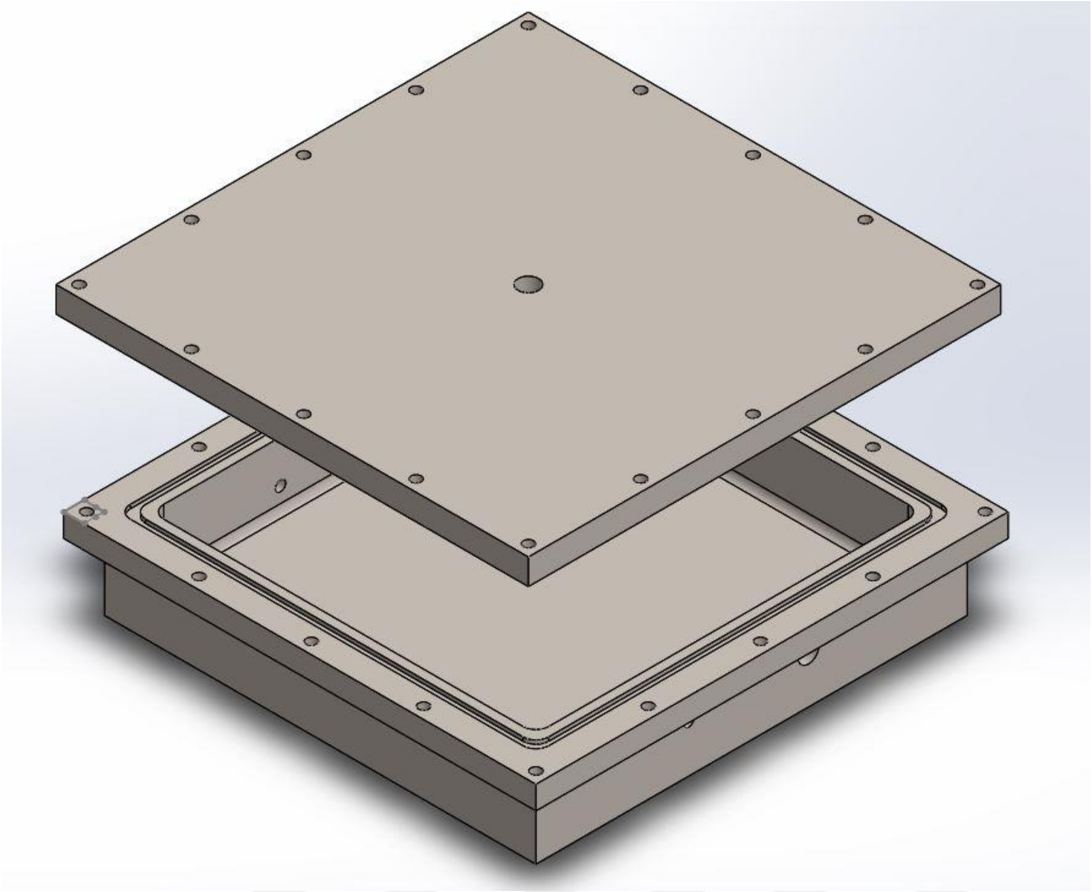


Figure 2.1: Desired autoclave system with 30x30x5 cm working area.

It is known that every solid materials have different yield strength value. This necessary value determines the border between the elastic and the plastic deformation. If the pressure is implemented to the material more than its yield strength, the material is exposed to the plastic deformation. In other words, the material has physical damage after this pressure which is more than yield strength. This deformation causes the bending, distortion or dislocation on solid materials. Because of that, the pressure is never be more that the yield strength of the material of the systems [20].

The yield strength of the materials are also changed with the temperature alteration. In the von Mises analyses, the yield strength value of the material must be taken at the maximum process temperature. The Von Misses analyse is used because the linear finite element analyse is enough and done with using yield strength parameter. When the temperature is increasing, generally the yield strength is decreasing for metals. When the autoclave is working, if the system strength changes in elastic deformation region at the maximum process pressure and temperature, the material is never take the physical damage. The wall thickness of the autoclave system have to be determined with using elastic deformation region values. Generally, if the wall thickness is increasing, the system strength is decreasing. This must be optimized with using von Mises analyse. The production of the autoclave system must be done with using this necessary physical borders [20].

For the optimization of the wall thickness of the autoclave system; the several wall thicknesses are implemented and analysed with using the von Mises stress analysis. The inner air pressure is taken at 1 mPa, and the temperature of the system is taken at 200°C due to the safety factor. If the system is working perfectly with the pressure of 1 mPa and the temperature of 200°C, the system is absolutely working in the process.

The von Mises Stress analyse is done for the wall thickness of 10 mm for main part of the system at 1 mPa pressure. The result of analyse is shown in Figure 2.2.

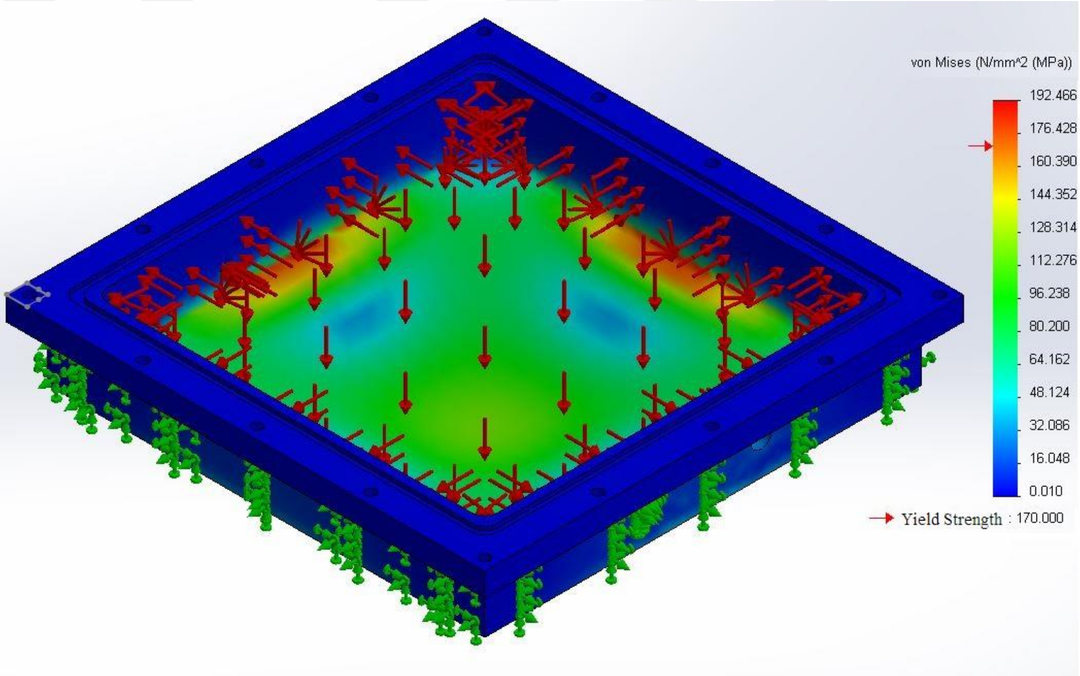


Figure 2.2: The base part stress analyse for 10 mm wall thickness.

The von Mises Stress analyse is done for the wall thickness of 15 mm for main part of the system at 1 mPa pressure. The result of analyse is shown in Figure 2.3.

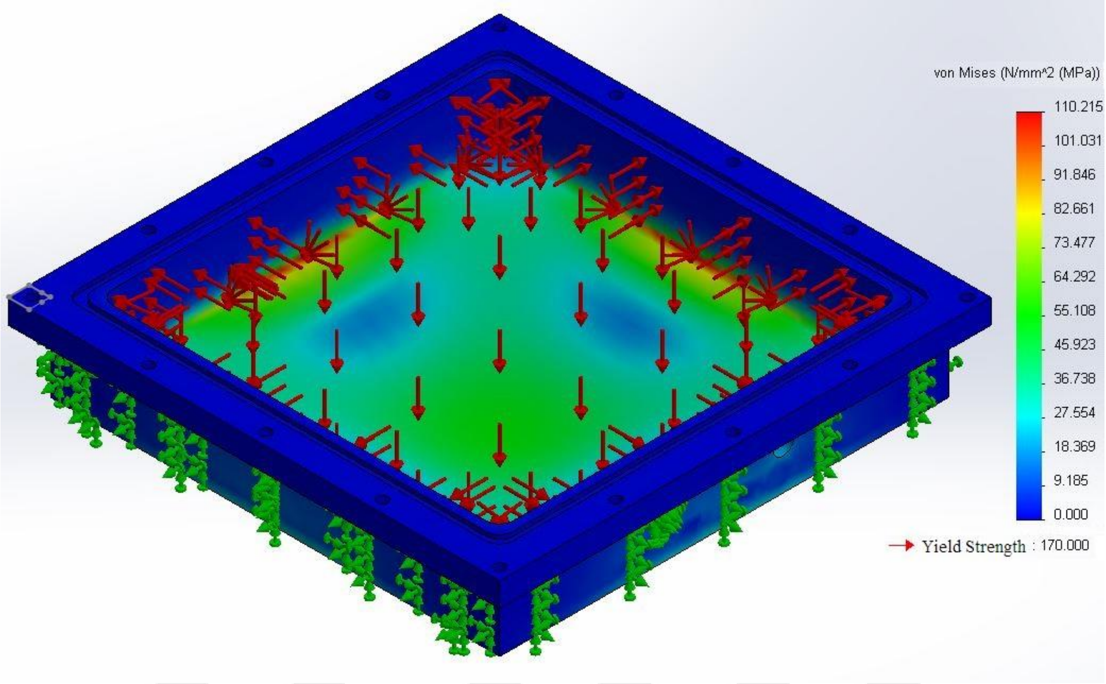


Figure 2.3: The base part stress analyse for 15 mm wall thickness.

The von Mises Stress analyse is done for the wall thickness of 20 mm for main part of the system at 1 mPa pressure. The result of analyse is shown Figure 2.4.

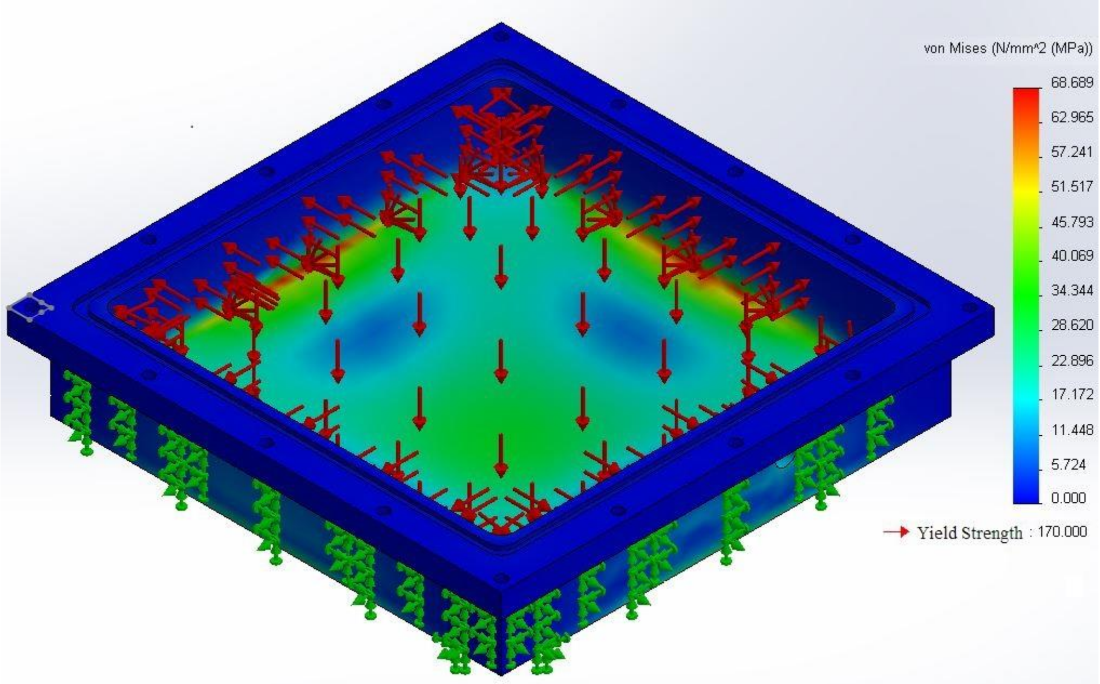


Figure 2.4: The base part stress analyse for 20 mm wall thickness.

The von Mises Stress analyse is done for the wall thickness of 15 mm for top part of the system at 1 mPa pressure. The result of analyse is shown in Figure 2.5.

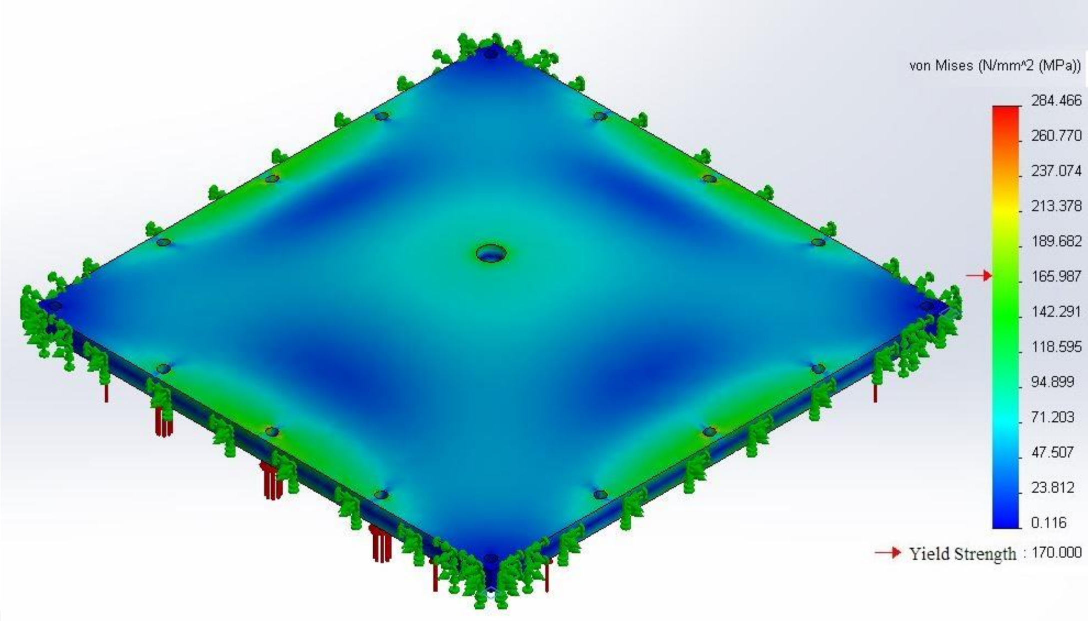


Figure 2.5: The cover part stress analyse for 15 mm wall thickness.

The von Mises Stress analyse is done for the wall thickness of 20 mm for top part of the system at 1 mPa pressure. The result of analyse is shown in Figure 2.6.

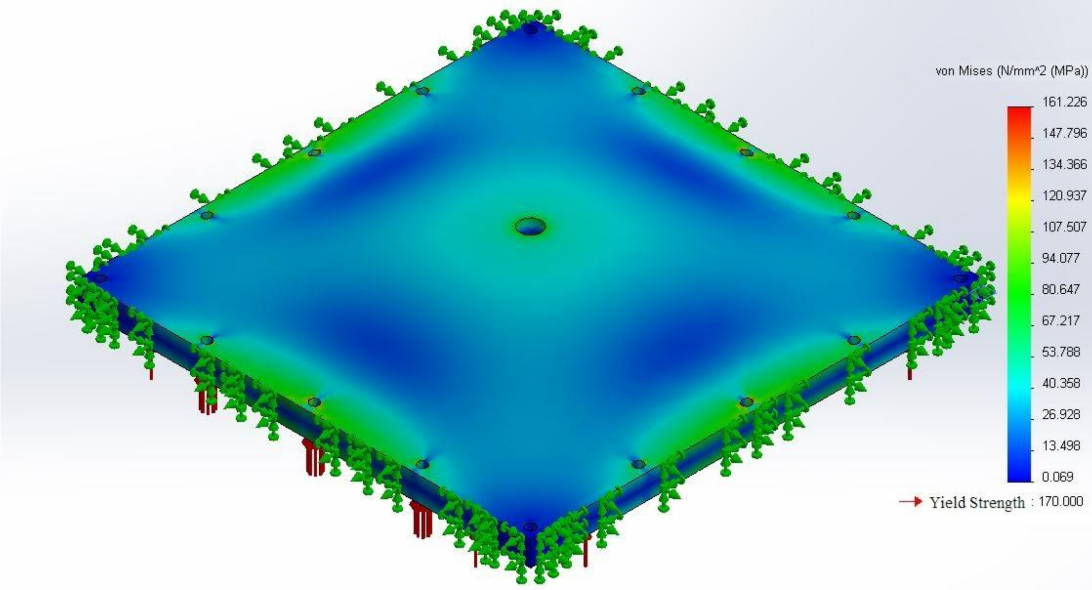


Figure 2.6: The cover part stress analyse for 20 mm wall thickness.

After analysing of the main and the cover part of the autoclave for different wall thickness, the optimized value can be discoverable. The wall thicknesses of 15 mm and 20 mm for main and cover part respectively, are useful because there are any point which have a stress more than the yield strength of stainless steel. This means that the 15 mm and 20 mm wall thickness can be used under the temperature of 200 and the pressure of 1 mPa.

However, the stainless steel is manufactured with the multiples of 10 mm wall thickness in Turkey. The cost of the engraving of the stainless steel to the 15 mm from 20 mm is additional expense. This is the necessary point of the optimization in the sense of wall thickness. Due to the fact that the engraving is an additional expenses for all structure, the system is designed with the 20 mm wall thickness for the main and the cover parts.

Another mechanical design point is that the tie rods calculation between the main and the cover part. In the temperature of 200°C and the pressure of 1 mPa, there are stress on tie rods which connects the main and the cover part. The tie rods must be working perfectly under these process conditions. The total force on the cover part in the maximum process condition is 90000 N. There are 16 tie rods in the design, the force on one tie rod is 5625 N. The strains of tie rods is calculated as 447.6 N per meter square. This value is under the tensile force of the stainless steel tie rods. Thus, the number of tie rods with the diameter of 4 millimetre is highly enough for the composite curing process.

2.2 The Heat Calculations and Analysis

2.2.1 The Heat Energy for Warming Process

The major phase of the production process is warming up of the whole Stainless Steel based parts from room temperature to 180°C with the 2°C/min steps. The temperature of the system must be increase two degree in one minutes because of the composite curing process. The computer aided design (CAD) design of whole autoclave system is shown in Figure 2.7.

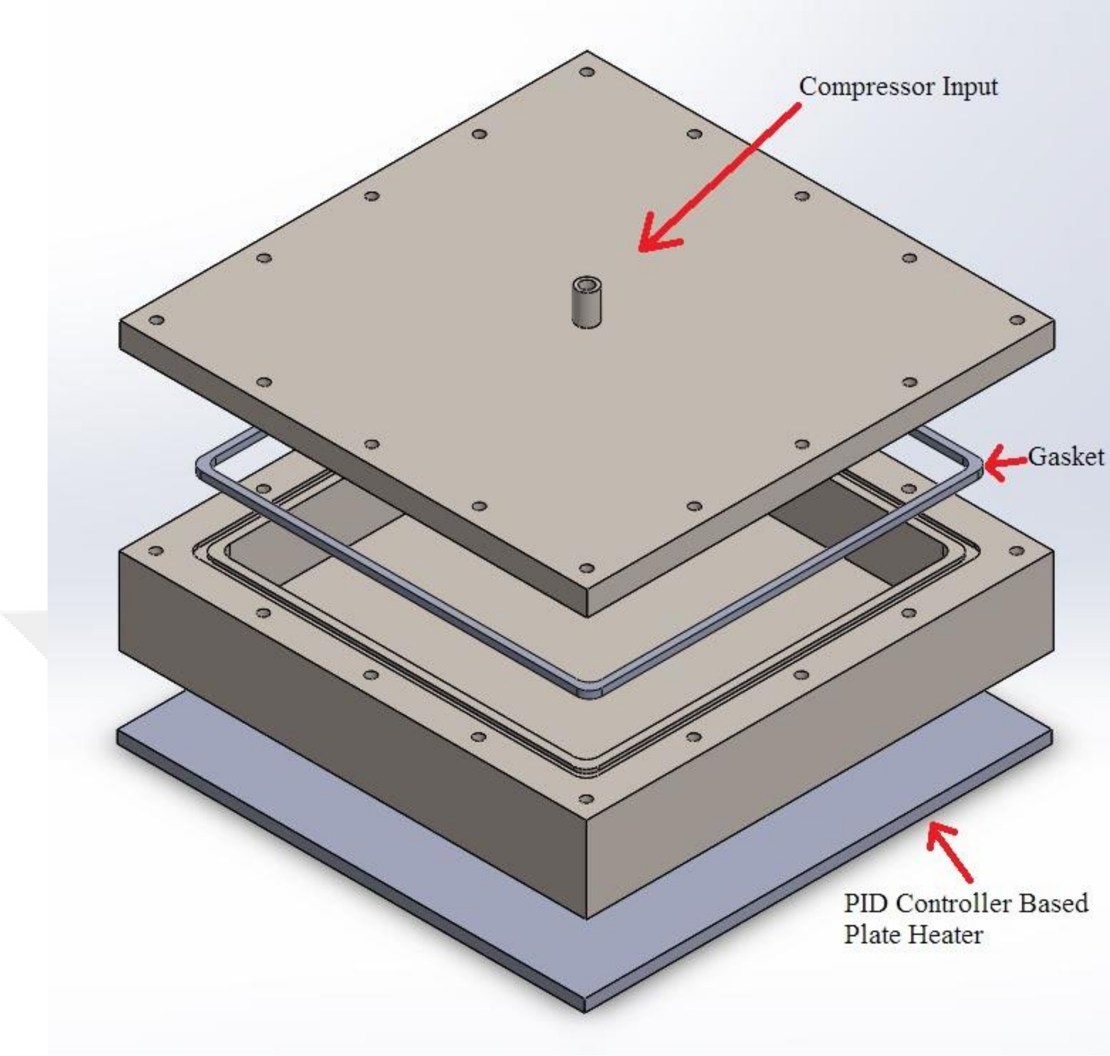


Figure 2.7: All parts of the mini autoclave system.

The heat calculations is that the stainless steel have 468 J/kg.C of heat capacity (Cp), 75 kg of mass. The temperature increasing of autoclave is 2°C per minutes.

In order to increase 2°C; $Q = m*Cp*T$

$Q = 75*468*2$

$Q = 70200$ Joule Energy in minutes [21]

The energy needs in second; $W = Q/60$

$W = 1170$ W Energy in second

The total weight of the autoclave system is 67.477 kg, because of the safety factor; the calculations are made as the system weight is 75 kg. The calculation shows that the system needs 70200 J energy for heating up to the temperature of 2°C per minutes. If the system needs that energy per minutes, the system needs 1170 W energy per seconds for warming. The plate heater of the system must be give this energy in ideal conditions such as perfect heat transfer with conduction between heater and the autoclave, no heat loss with natural convection.

2.2.2 The Heat Loss with Natural Convection

The plate heater have to be designed in order to giving energy at least 1170 W to the system under the heat loss conditions. The composite curing process temperature ranges from almost 25ºC to 180ºC. The maximum temperature is 180ºC. It is known that when the system is warming up, the heat loss of the system is increasing step by step because of the natural convection. The plate heater must be compensate this heat loss to the environment in order to heating up with the temperature of 2ºC per minutes. The natural convection heat loss is shown in Figure 2.8.

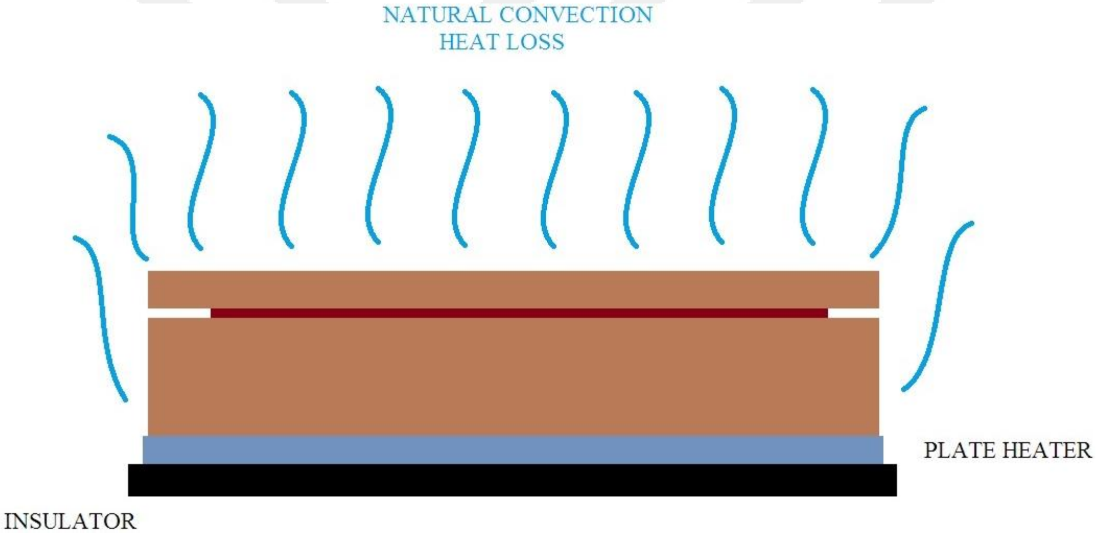


Figure 2.8: The heat loss with natural convection of mini autoclave.

The heat loss per second at 180ºc with Natural Convection is calculated below [21].

$L_c = A_s / p$ $A_s = (38*9)*4 + (38*38) = 2812\ cm^2 = 0.2812\ m^2$

$P = 38*4 = 152\ cm = 1.52\ m$

$L_c = 0.185\ m$

Upper surface hot plate; $Ra_L : 10^7 - 10^{11} \rightarrow Nu = 0.1*(Ra_L)^{(1/3)}$

Stainless Steel AISI 316: at 300K $\rho = 8238\ kg/m^3$

$k = 13.4\ W/mK$

$Cp = 468\ J/kgK$

$\alpha = 3.48*10^{-6}\ m^2/s$

$T_f = (Ts + T_\infty) / 2 = 102.5\ ^oC = 375.5\ K$ $\quad$ $Ts = 180\ ^oC$ and $T_\infty = 25\ ^oC$

At 102.5 °C and 1 atm Air ; $k = 0.031125\ W/mK$

$\upsilon = 2.333*10^{-5}\ m^2/s$

$Pr = 0.7106$ $\quad$ $\beta = 1 / T_f$

$Ra_L = [g* \beta*(Ts - T_\infty)*(L_c)^3*Pr] / \upsilon^2 = 3.34734*10^7$

$Nu = 32.2279$

$h = k*Nu / L_c = 5.422\ W/m^2K$

$Q = h*As*(Ts - T_\infty)$ = **236.323 W** → Heat loss at 180°C

It is easy to state that if the heater can compensate this natural convection heat loss at 180°C, the system can works at every temperature below 180°C. Because, the heat lost at 180°C is every time much more than below temperatures. The heat loss of the system with the natural convection is 237 W at 180°C. The plate heater have to give 1170 + 237 = 1407 W energy to the system in order to increase temperature with 2°C/min.

There are energy saving method with using rockwool insulation. The heat loss with natural convection is 237 W per second at 180°C. The five of six surfaces of autoclave is directly connected to the environment. These five surface create natural convection heat loss. If these five surface is covered with using insulation material, the natural convection will be decrease. In Figure 2.9, the rockwool insulation schema is shown.

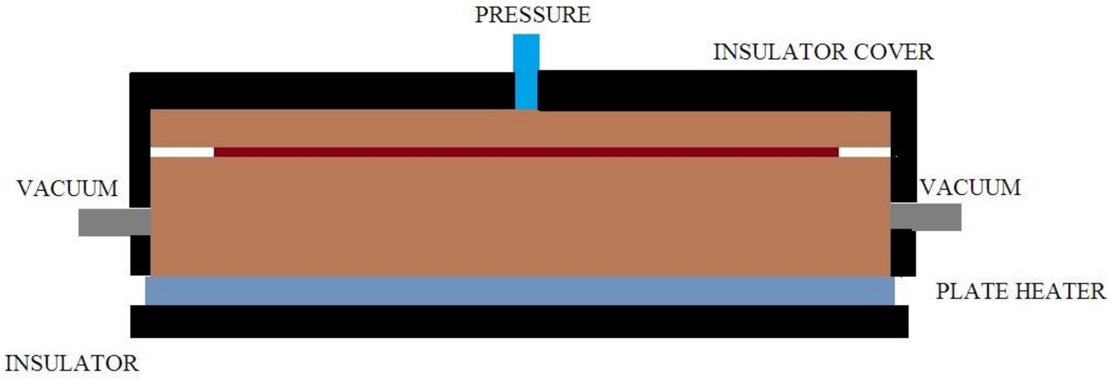


Figure 2.9: The rockwool insulation of autoclave

2.3 The Heater Design and Selection

The system have to be takes at least 1407 W energy for heating up with 2°C per minutes. The heater must be designed for generating almost 1700 W heat with the 20% of safety factor. The heat loss of the heater with the natural convection is so small because there are a successful thermal insulator around the plate heater. The thermal insulator is made from rock wool whose thermal resistivity coefficient is 1.47 m^2K/W [22]. Whole system loses the energy only with the natural convection from the autoclave surface. The insulator around heater cannot let the heat loss. In addition, the rock wool insulators are so useful in many heat based applications such as high temperature ovens, special heaters, buildings etc. [21, 22] . Because, the rock wools are non-flammable, never gets corrosion, uneatable by insects and durable.

In Turkey, the electricity is 220 volt (V) alternative current (AC) with 50 Hz frequency. The heater needs 7.7 A current for 1700 W energy generation. The resistance is 28 Ohm which is calculated with using this current value. The plate heater dimensions are 25x25x0.5cm with 28 Ohm wire resistance. In short words, when the plate heater connected to the 220 V AC without controller, it generates 1700 W heat with 7.7 A current flow. The generated heat directly transferred to the autoclave with the conduction. Finally, the autoclave system starts warming up with the temperature of 2°C per minutes which is necessary for curing the composite structures inside of it.

3. THE CLOSED LOOP CONTROL SYSTEM OF MINI AUTOCLAVE

The desired autoclave system have to be temperature controlled. The user can determine the temperature increasing and decreasing process step by step. For the scope of curing process, the heater voltage have to be controlled with designed control algorithm. In this chapter, the desired control system is designed and simulated in MatLab Simulink environment. The design results are implemented to the process controller device and tested. The curing tests is done with using the controller of system.

3.1 The Heater Heating Mechanism

The process of heating with electric is that the conversion of the electrical energy to the heat energy. The electrical heaters are using in a lots of technological areas such as: factories, laboratories, electronical devices, homes, cars etc.

The electrical energy is transferred to the heat energy with using the resistors which is called as Joule heating. The most used heating resistors are generally made from nichrome wire which have an insulator at outside of wire. The resistivity of nichrome is 1.5×10^{-6} ohm per meter. This value is calculated with using multi-meter. The resistance of nichrome wires are configurable with changing the cross section area, and the length of it. When the potential difference is constant on a resistor, the current is directly related with the resistance of the resistor because of the ohm law. The related energy for heating the systems can be adjusted with using this property [21].

For the autoclave system, the 1700 W energy is needed for the heating up with 2 ^{o}C per minutes. The calculated resistance value is 28 Ohm before. The plate heater of the system have to be includes the finally 28 ohm nichrome wires inside of it which is shown in Figure 3.1. The insulation around plate supports the system for heat transferring to the autoclave only.

Figure 3.1: Nichrome wired plate heater.

The plate heater is manufactured with 28 ohm wire resistance in the dimensions of 25x25x0.5 cm for the autoclave system. The heater includes a nichrome resistance wire and an aluminum cover around the resistance. The plate heater shape is square but the resistance is placed as a long wire inside the aluminum cover. There are two heat transfer phase inside the heater.

First phase, the electrical current is converted to the heat energy with electrons and atoms. Second phase, the produced heat is transferred to the aluminum cover with the convection and the conduction. Finally, the heater can produce the related heat with the electrical current. The plate heater transfers the energy to the system with the conduction [21].

The heater is used with SSR for PID type controlling by controller. For generating the energy for the autoclave system, the heater needs more electrical energy than the controller provides. Because of that, the SSR is used for switching of the electrical current source and the heater. The plate heater takes the energy from electric network with the switching of SSR which is controlled by PI controller.

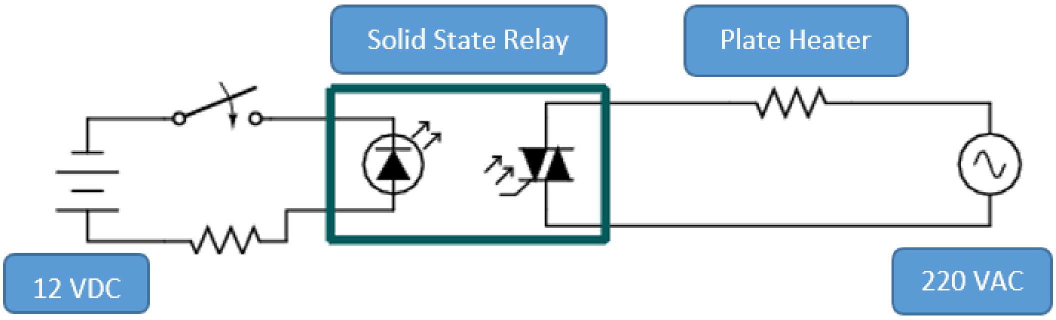


Figure 3.2: Solid state relay circuit for heating.

The SSRs, which include two metal oxide semiconductor field effect transistors (MOSFET), are type of a semi-conductor materials with is triggered by 5-12 V direct current (DC) and 7.5 mA current KAYNAK which is shown in Figure 3.2. This semi-conductor based relay helps for controlling the high currents up to 25 A with the low currents and Vs. The PI controller can switch the SSR only with short pulses. The plate heater warm up behaves like PI controlled heater with this pulses which is more than 100 times a second. This extremely quick ON/OFF process lets the controller controls the heater as PI controlling methodology. Thus, instead of changing the potential difference of AC for controlling the heat, the SSRs are more useful in order to controls like PI controller.

3.2 The Transfer Function of Mini Autoclave System

Every physical system has transfer function. The transfer functions are used for the representing of system in mathematical way. The improvements of the technology and engineering is directly related with mathematical representations of the physical systems. The transfer function converts inputs to the outputs in terms of system dynamics. In other words, the system dynamics gives reaction due to the action on the system. The action is called as input, the reaction is called as output. The transfer function transfers the input to the output [23].

Generally, in control systems, the transfer functions are represented in Laplace domain S domain. The generation of the transfer function is done with using the dynamical differential equations of related system. It’s known that every dynamical process can be written in differential equation form. The transfer functions are generated with using Laplace transformation on the differential equations. The reason of using the

Laplace domain, the calculations in this domain are so easy when it compared with differential form. In order to calculation of closed loop transfer function of whole system, the some mathematical multiplications are needed and the mathematical operations are easier in Laplace domain than differential form. Because of that in control system designs, the whole systems are taken into account in Laplace domain [23].

There are a lots of ways for generating the transfer functions of the systems. The most useful method is model matching method which is created by J.G.Ziegler and N.B.Nichols in 1942 [24]. This method states that the step response of any system gives the estimated transfer function of it. The step response of the system can be generated by running the system in open loop configuration for constant input which is shown in Figure 3.3. When the control signal takes maximum constant value from beginning to the final on the process, the output (necessary measured value) of the system called as step response. It can be also defined as the reaction of the system to the maximum input value. This reacted value, which is measured with sensors, is saved with real time. The values gives the response curve which is used for the model matching method [24].

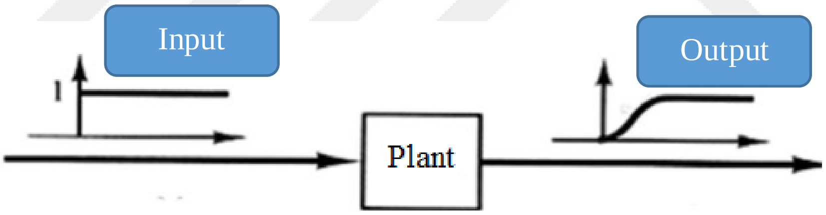


Figure 3.3: Open loop system step response.

The step response curve is generally shaped as S letter. This S shape gives lots of information about the transfer function of the system. The S shape can be stated as first order system with the L time delay. The S curve is so important in terms of determining of the L time delay and the time constant “τ” value for model matching method. The τ and L values are used for indicating of the transfer function of the system. The K is the final value of the output according to the maximum input value. When the system reached to the constant output value after enough time, this point is called as K value. The time constant value is determined with using %63.2 of the final value of system. The step response of any system is shown in Figure 3.4. The first order transfer function of system in Laplace domain, which is generated by model matching method, is shown in equation 3.1 [24].

$$Plant\ (s) = \frac{Ke^{-sL}}{\tau s + 1} \tag{3.1}$$

The plant can be modelled as shown in the formula. The PI controllers are designed with using this transfer function. The characteristics of the system can be adjusted with using the controller parameters.

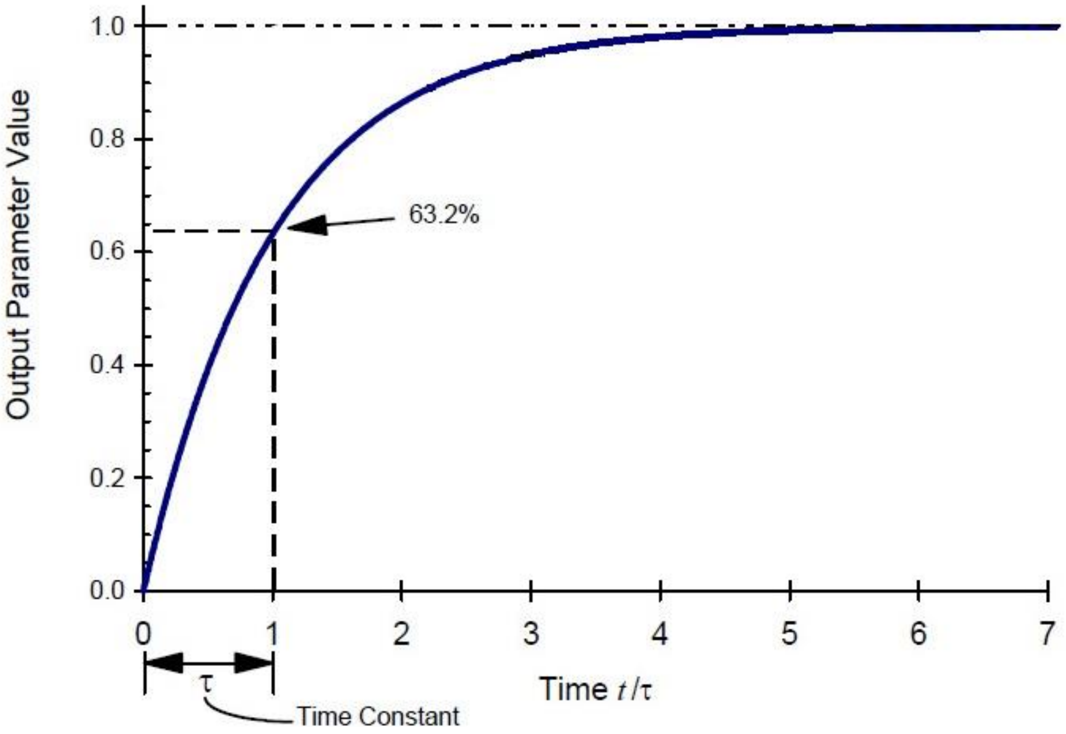


Figure 3.4: Step response of any system.

After manufacturing of the autoclave system, the step response have to be examined in order to determining of the warming process with respect to time. The temperature of system with respect to time is recorded. This process takes almost 20000 seconds which stands for 5.5 hours. The meaning is that there are 20000 temperature values until the temperature of system becomes stable. The temperature must be stable ultimately in terms of step response logic. The system is worked at full input. In other words, for the step response, there are no controller and the heater of the system is worked at maximum value. This situation creates step response of the autoclave system. The temperature values give the transfer function of system with the help of model matching method. The step response of autoclave system shown in Figure 3.5 [24, 25].

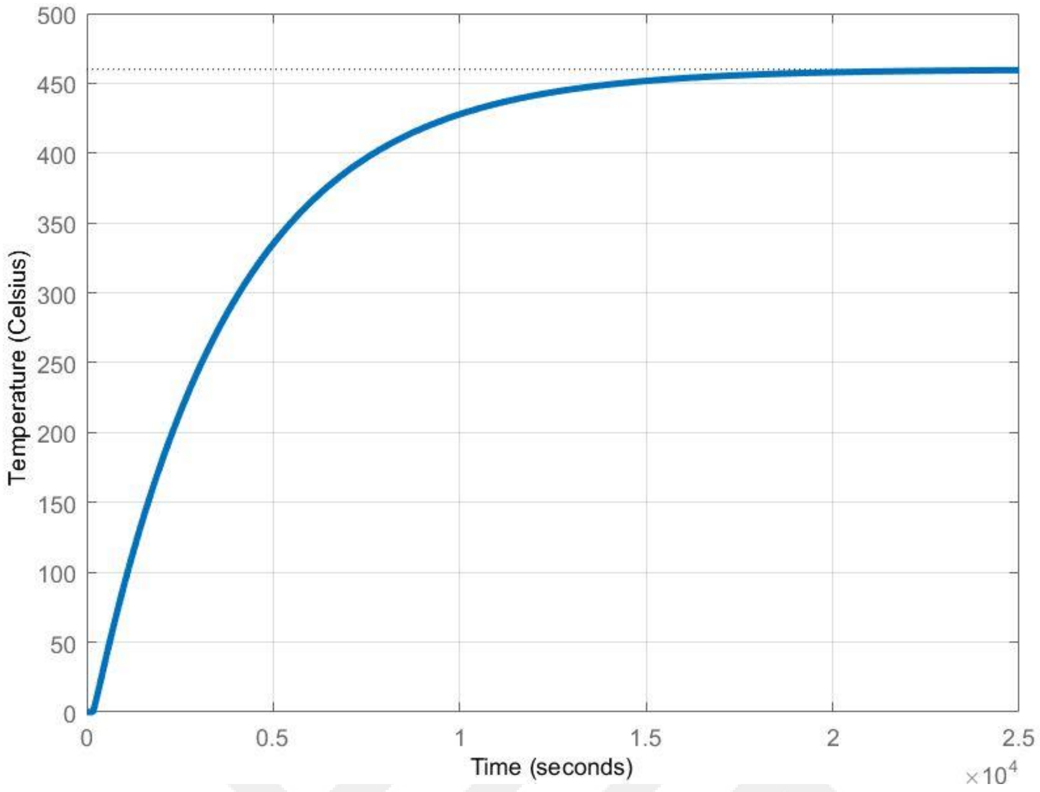


Figure 3.5: Step response of mini autoclave system.

The model matching method is implemented to the step response of system which is shown in Figure 3.6. As shown in figure below, the K value represents maximum temperature value which equals to 460 ^{o} C. The time delay of system is 89 seconds which is represented by letter “L” in figure. The time “τ” is 3688 seconds [24].

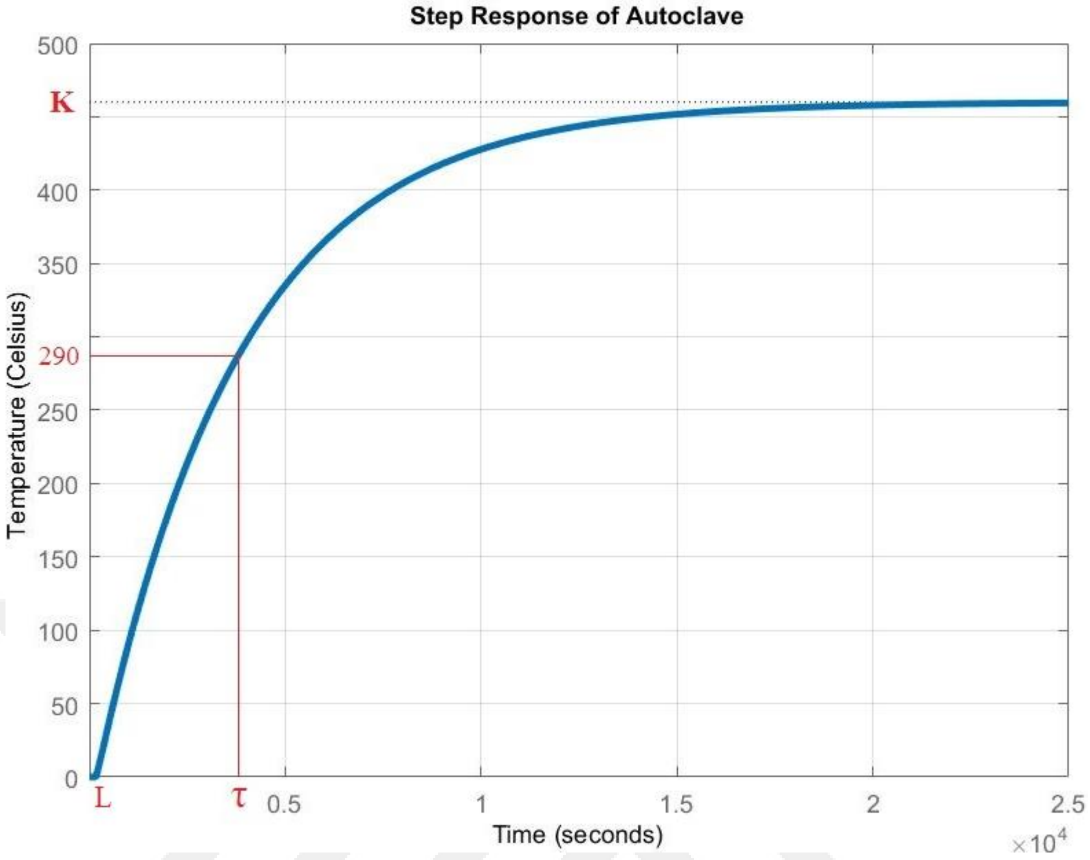


Figure 3.6: Determining of time constant

After all these significant values, the model matching transfer function of the system according to the step response is shown in equation 3..;

$$G(s) = Plant\ (s) = \frac{460e^{-s89}}{3688s + 1} \tag{3.2}$$

3.3 The Temperature Controller: ORDEL AC491

The designed autoclave system is type of a process which includes temperature, vacuum and air pressure control. The system have to be controlled with using an electronical control device. The selection of the device is done with the criteria of temperature controlling with PI algorithm. In the process, the user have to be observe the process values such as heater temperature, control signal and system temperature. In addition, the user can be adjust the final temperature value and the heating process ramp such as 1°C, 2°C or 3°C per minutes.

There are a lots of process control devices in market. This system can be controlled with using PLCs or Microcontrollers such as Arduino and Peripheral Interface

Controller (PIC), temperature controller or any other processor. When the cost of all devices taken into account, the basic temperature control devices are more useful for this system. The temperature process control device is selected as ORDEL Advance Process Control AC491 model which is shown in Figure 3.7 [26].

Figure 3.7: Ordel PI temperature controller AC491

The AC491 type process control device have a lots of properties which are listed below [26].

- 4 digits numeric display
- 6 LED indicators
- 1 transmitter supply output (24VDC)
- 1 universal sensor input (Thermocouple (TC), Real time communication (RT), potential difference (V))
- 1 auxiliary analog input (0-4 / 20mA)
- 1 potentiometer input (100-1500W)
- 2 digital inputs (15V)
- 1 RS485 communication unit
- 1 analog output (0 / 4-20mA, 0 / 2-10V)
- 4 relay or logic output (24V)

- 100-240V AC universal or 24V AC / DC feeding
- Insulation between input and output modules
- PID heating / cooling
- Auto-tuning (automatic adjustment of PID parameters)
- Automatic / manual operation modes
- Bumpless transfer feature
- Sensor fault detection
- Remote set point (remote set point setting)
- 4 selective set point
- Ramp function
- Retransmission (for process and set values)
- 15 different relay functions
- ON / OFF, P, PI, PD, PID control
- Linear and time-proportional control output
- 100ms sampling and control cycle

The PID controllers for AC491 control device are designed following chapters. The main aim at this part, the PID controller parameters are calculated theoretically in MatLab environment. The system transfer function is directly used at this MatLab simulation. Thus, the designed PI parameters are implemented to the ORDEL process control device. The expected result is heating with 2^{o}C per minutes. In other words, the system must be heating to the 120^{o}C in 3600 seconds or 3000 seconds from 20^{o}C ambient condition.

The ORDEL process controller can control the process temperature with using PI controller algorithm. The PI controller algorithm is adjust the potential difference of the heater. By this way, the heat energy can controllable in order to increasing the system temperature to the desired point. However, the PI controller is never change the potential difference of the heater. Every time the 220 AC potential difference remains constant. The AC491 controller controls the system with using the pulse width modulation (PWM) [26].

Firstly, the system calculates the needed potential difference for rising system temperature to the set value with the measuring of process temperature. This potential difference calculation is done with using the PI control algorithm.

Secondly, the PI control signal is converted to the PWM signal in order to controlling of SSR for warming. The heater of system is controlled with SSR because the 220 AC potential difference conversion is more and more difficult than switching. When the SSR takes 5 V DC signal with low current, it lets the 220 V AC goes to the heater and also this situation called as ON position for SSR. Otherwise, when the SSR takes 0 V DC signal, the 220 V AC does not go to the heater and this is called as OFF position for SSR. The useful point of this system, the high potential difference is controlled with low DC signal [27].

Finally, the turning ON and OFF position can change up to 100 times per second with using SSR. When the PI signal is converted to the PWM signal, which have an amplitude of 5 or 0 DC V, the SSR can turning ON and OFF how much system needs. For example, when the heater takes %50 PWM signal in second, the heater connects to the 220 AC potential difference only 0.5 second; the other 0.5 seconds 220 AC potential difference does not connect to the heater. In one second, the heater turning ON and OFF position equal time. This process creates half heating on system, seems like the heater takes 110 AC potential difference in one second continuously. These types of PWM variations create adjustable heat energy on autoclave system. The PI controller algorithm controls effectively the heater system with small currents and potential differences. This methods are more useful in today’s industry. The follow chart of the heating process is shown in Figure 3.8 [27].

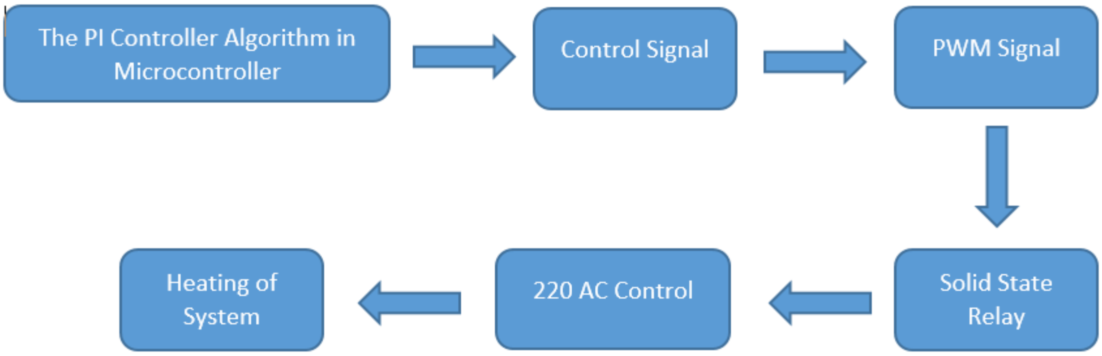


Figure 3.8: Flow diagram of system

3.4 The PI Controller

The temperature of the autoclave system have to be controlled due to the fact that the process of composite curing. The temperature must be increased 2 Celsius in 1 minute. The chemical content of composite materials are need curing slowly and according to the literature, the temperature acceleration is so important in terms of the composite curing process [8]. Because of that, the heater must be controlled sensitively for increasing temperature.

The controller algorithm is used as PI controller which is very popular in today’s engineering world. The PI controllers are also called as “second order controllers”. The PI controllers help us for determining the significant characteristics such as settling time, rising time, steady state error and etc. of any system. These features give an opportunity to controlling of systems whatever it is require. The process controlling of the systems are also done with using this controller algorithms. The PI controllers give an excessive response to systems with the help of the feedback [23, 24]. The classical closed loop controller algorithm is shown in Figure 3.9 with the feedback.

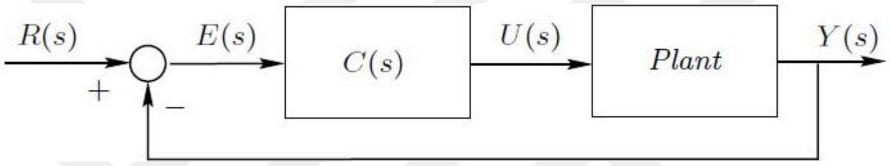


Figure 3.9: The basic closed loop feedback control diagram

The feedback in control systems have an important role for controlling. The feedback information, which comes from the output Y(s), are compared with reference R(s) and this creates the error E(s) which is shown in equation 3.3.

$$E(s) = R(s) - Y(s) \tag{3.3}$$

The error E(s) uses for compensation at PI controller C(s) for creation of new output U(s). Then controller output U(s) is implemented to the plant G(s). Thus the plant takes input and gives the output according to its dynamic. This situation is repeated until the error E(s) is minimum as possible. The controller always tries to minimize the error E(s) in system. This loop is called as closed loop control system [24]. The PI controller parameters adjust the behaviour of plant. In short words, the major aim of the PI controllers are using for control the system in terms of the related process. The reference R(s) changes in process time according to the process requirements. The PI controller tries to keep error E(s) zero as possible especially autoclaves like temperature control systems [23].

In autoclave system, the main aim is controlling of the inner temperature of the pressurized vessel. There are a PI heat controller on system which controls power of electricity. This control is done with using the SSR which is stated as before. The controller parameters have to tune for composite curing process. The related vessel system have to be warming up with 2°C per minute. Therefore, the PI controller is tuned for this process with using MatLab environment, especially single input – single output (SISO) control system tool. The transfer function of the autoclave system is used for the SISO design.

3.4.1 The PI Controller Design

The PI controller is selected for controller algorithm. The PI controller algorithm is enough for the first order heater system. The integral factor of controller provides to eliminate steady-state error of system. Normally the derivative factor in PID controllers reduce the overshoot and increases the stability of system. However, in autoclave system there is no any high overshoot, because of that the derivative factor is not using here. In other words, the system response is so slow and this slowness not causes to overshoot [24].

The design of PI controller is done with using the desired closed loop transfer function. The settling time and overshoot criteria are using for the determination of the damping ratio and natural frequency of system. With using damping ratio and natural frequency parameters, the characteristic polynomial of system is generated. Finally, the closed loop transfer function of system, which includes PI controller parameters, equalizes to desired characteristic polynomial. Thus the PI controller parameters Kp and Ki is indicated with using the polynomial equality [23].

The controller design criteria is that the design is done with using the temperature increasing logic, the system temperature must be increased with 2 celcius degree in minute, the maximum over shoot is 5% percent is shown in equation 3.4 and the last criteria is that the settling time of system is 60 seconds is shown in equation 3.5.

$$\zeta = \frac{-\ln(0.1)}{\sqrt{\pi^2 + \ln^2(0.1)}} \tag{3.4}$$

$$\zeta = 0.5911$$

$$Ts = \frac{4}{\zeta\ \omega} \tag{3.5}$$

$$\omega = 0.112$$

The characteristic polynomial Pc(s) of desired closed loop transfer function is shown in equation 3.6.

$$Pc(s) = s^2 + 2\omega\zeta s + \omega^2 \qquad (3.6)$$

$$Pc(s) = s^2 + 0.13216s + 0.012544$$

In order to determining of the PI controller parameters, the closed loop transfer function of system is used. The polynomial equality gives the necessary controller parameters. Firstly, the closed loop transfer function is calculated as shown in equation 3.7.

$$\frac{460(Ki + Kps)}{460Ki + s(1 + 460Kp + 3688s)} \qquad (3.7)$$

The closed loop characteristic polynomial is shown in equation 3.8.

$$\frac{115Ki}{922} + \frac{s}{3688} + \frac{115Kps}{922} + s^2 \qquad (3.8)$$

With using the polynomial equality, the Kp = 1.0574 and Ki = 0.10057. The first order autoclave system PI controller is shown in equation 3.9.

$$C(s) = 1.0574 + \frac{0.10057}{s} \qquad (3.9)$$

3.4.2 The Simulink Simulation

The PI controller of the autoclave system is design with using the MatLab Environment. The Simulink is much useful for these type of control system simulation. If the transfer function of the system is known, the controller design and simulations can be done in Simulink.

The simulation requires to load all system to the Simulink as real-like. The Simulink simulation includes a transfer function of the autoclave system, a converter that converts the PI control signal to the PWM, the PI controller, the SSR, reference temperature and delay time. The 220 AC potential difference controlling with SSR as mentioned before chapters. The main simulation Simulink diagram is shown in Figure 3.10.

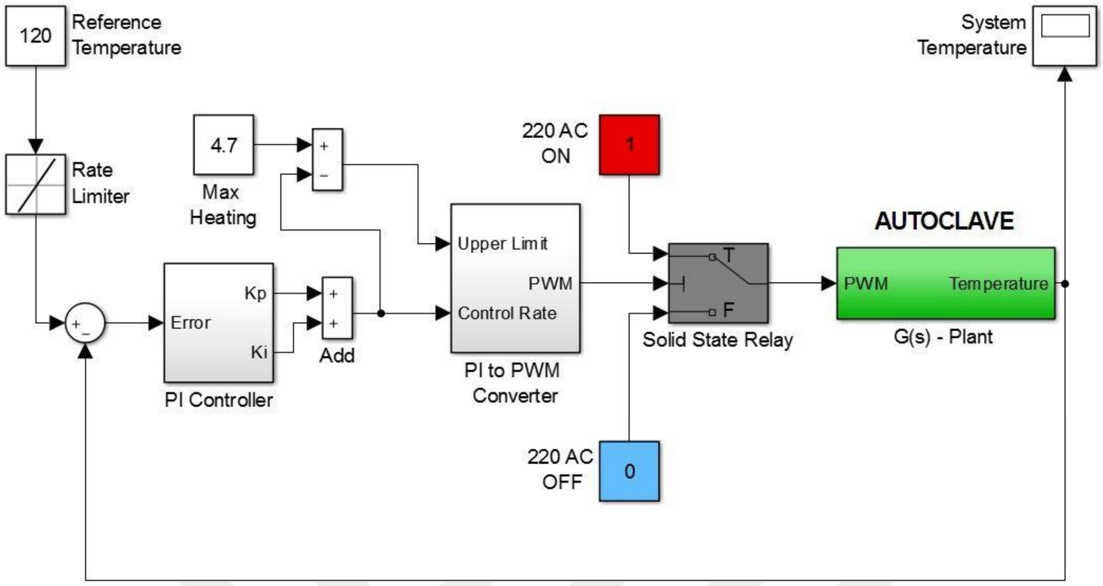


Figure 3.10: The Simulink simulation diagram of system.

The flow of simulation is start with the reference temperature. After that the reference temperature is limited with rate limiter in order to increasing with 2ºC, 3ºC or 4ºC degree per minutes. The rate limiter increases the temperature step by step. Then, the reference temperature is compared with system temperature and this creates an error for PI controller.

The error is using in PI controller for creation of needed control signal. The calculated control signal is must converted to the PWM signal in order to switching of 220 AC potential difference. This switching controls the generated heat energy in plate heater. Consequently, the autoclave system is warming up to the related point. The system temperature gives feedback to the controller which is shown in **Error! Reference source not found.**.

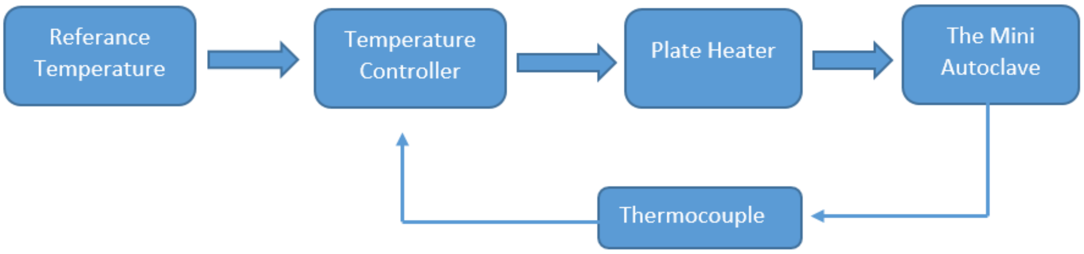


Figure 3.11: The closed loop control diagram of system

3.4.3 The Sub-Blocks in Simulink

The G(s) plant part of the simulation represents the transfer function of autoclave. This green block includes the transfer function of autoclave in S domain.

The PI controller block of simulation includes the PI controller algorithm with Simulink blocks which is shown in Figure 3.12.The input of this block is error of temperature, the output of this block is three different calculated control signal such as proportional, integral and derivative parts.

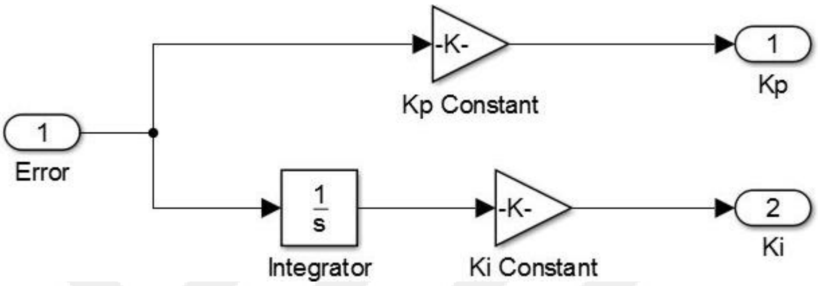


Figure 3.12: The PI controller sub-block content.

The three PI parameter takes the error and calculates the related control signal. The design meaning is that the proportional (Kp), integral (Ki) and derivative constant (Kd) values must be adjusted for system. In the design part, all of the major aim is properly adjusting of these three parameters.

The converter block converts the PI controller signal to the PWM signal for switching of electricity power. The PWM signal frequency have significant value at this point. The frequency of it depends on the inputs of this block. There are two inputs for this sub-block: one of them is upper limit and the other one is control rate, which comes from PI controller. These two value must be converted to the switching ON and OFF rates. This sub-block takes the ON and OFF rates and generates the pulses with the unit amplitude and related frequency width. The main work of this block, the comparing of these two inputs and creates the PWM signal. The inside of converter sub-block is shown in Figure 3.13.

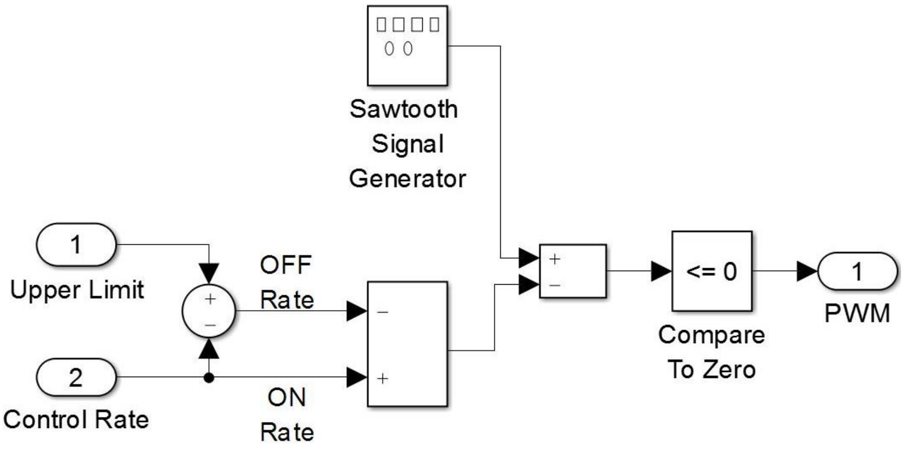


Figure 3.13: PI to PWM converter sub-block.

The most important thing is that the PWM frequency is generated with using the PI control signal with the way of this converter block. The duty cycle of signal makes switching on SSR. Thus, the heater takes energy and gives heat to the autoclave system.

Normally, this ON and OFF position can be thinking about this is Bang Bang control. However, the ON and OFF switching number is 100 times per second. This high amount of switching rate controls the system like PI controller algorithm. Because of that, the PI to PWM converter have significant role at that point [27].

The differences between ON and OFF rate generates a specific number. The Saw tooth wave generator is generates triangular periodic waves which have 1 Hz frequency and 1 unit amplitude. The differences between saw tooths and mentioned specific number is using for the comparing with zero. If the differences is more than zero, the “compare to zero” block gives 0, else the differences is less than zero, the block gives 1. The rate of 1 or 0 values simultaneously change the duty cycle of PWM. This changing is triggered the SSR in order to let the heat goes to autoclave.

The results of the simulation are generated with running under the curing procedure. The temperature versus time graph of the function is shown in Figure 3.14. The system temperature is increased according to the production procedure.

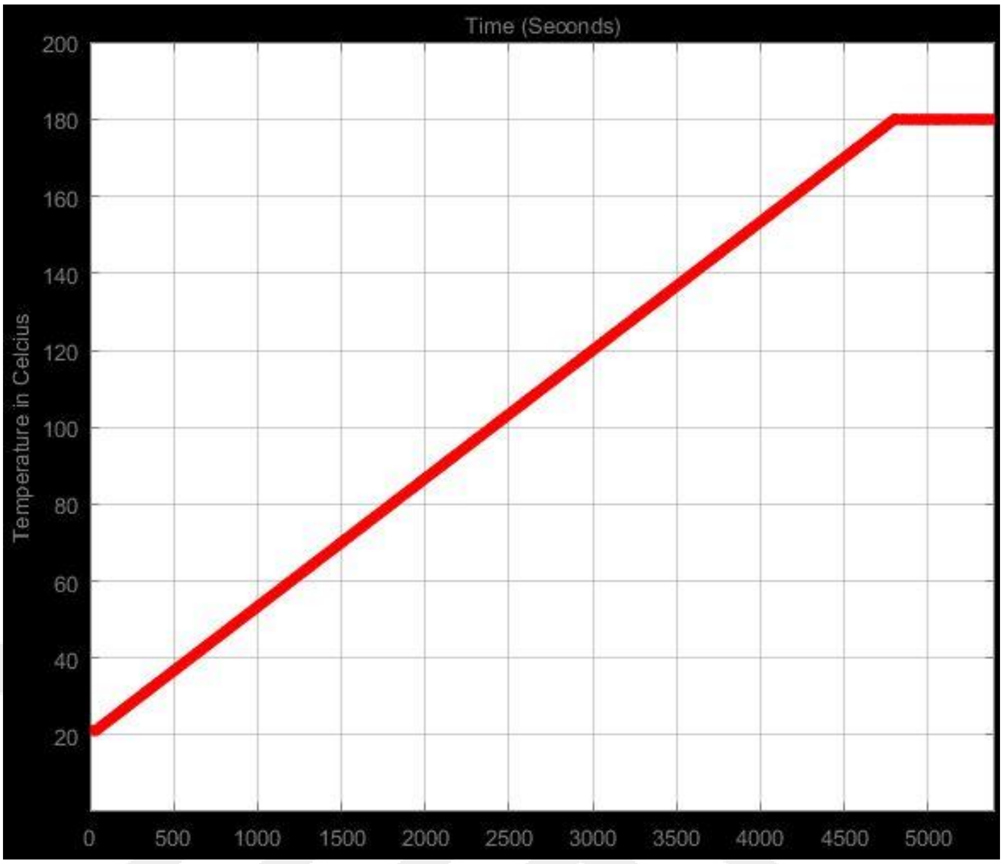


Figure 3.14: Curing procedure simulation result

4. CONCLUSION

After the mechanical, electronical and controller design of autoclave, the system can be produced into the real world. The top and the base parts of autoclave system are analysed as mentioned before and the wall thicknesses of parts are determined optimally. The mechanical production of autoclave bodies such as base and top cover, are done with using computerized numerically controlled (CNC) engraving. The engraving of parts are done from stainless steel block. After engraving, the top and base parts are shown in Figure 4.1 and Figure 4.2.

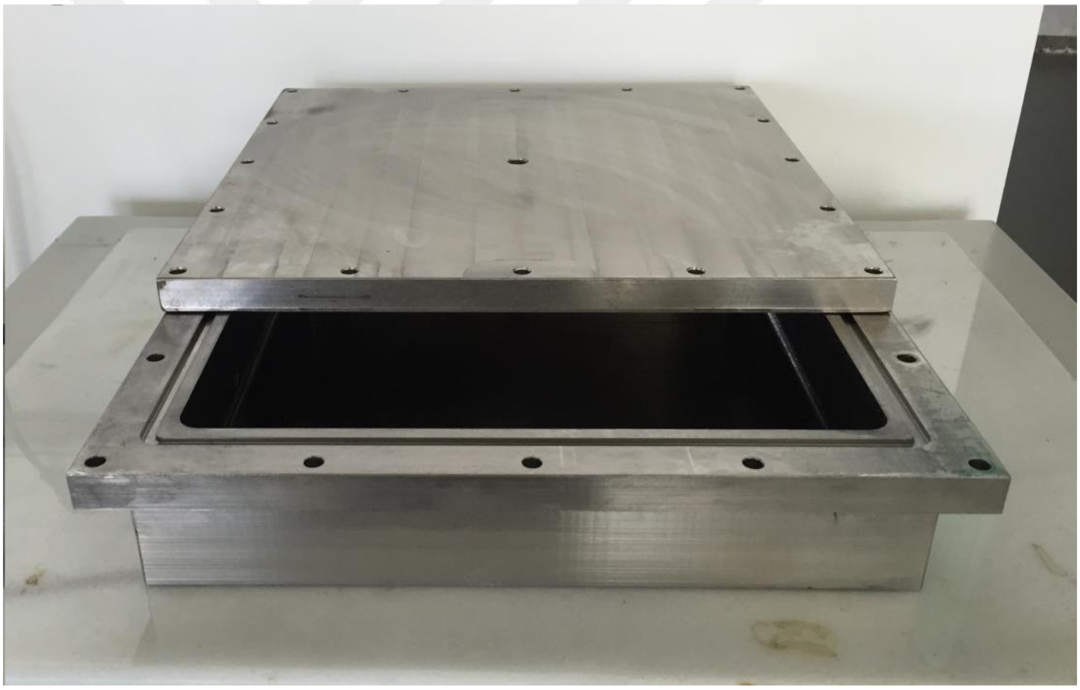


Figure 4.1: The front view of autoclave after engraving

The corner view of autoclave is shown in Figure 4.2. The system shape is square from top side, the shape is rectangular from other sides.

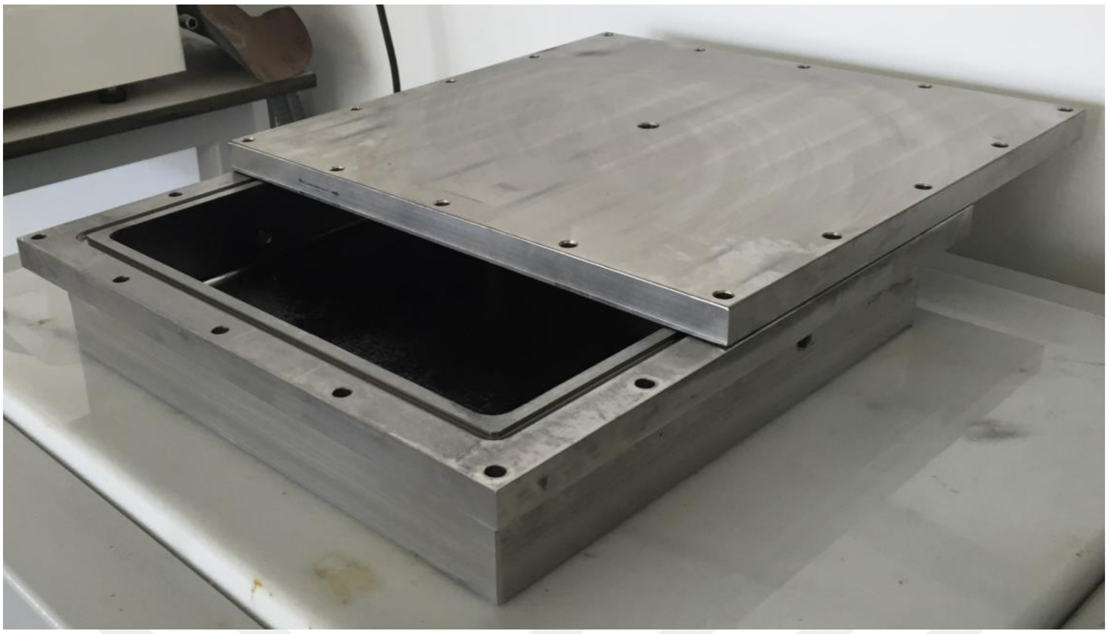


Figure 4.2: The corner view of autoclave after engraving

It’s known that the composite curing process includes vacuum and air pressure. The system must be have three regulated type valve which connects outside environment to inside of vessel. The locations of vacuum valves are placed right and left sides and the pressure valve is placed top cover of vessel which is shown in Figure 4.3.

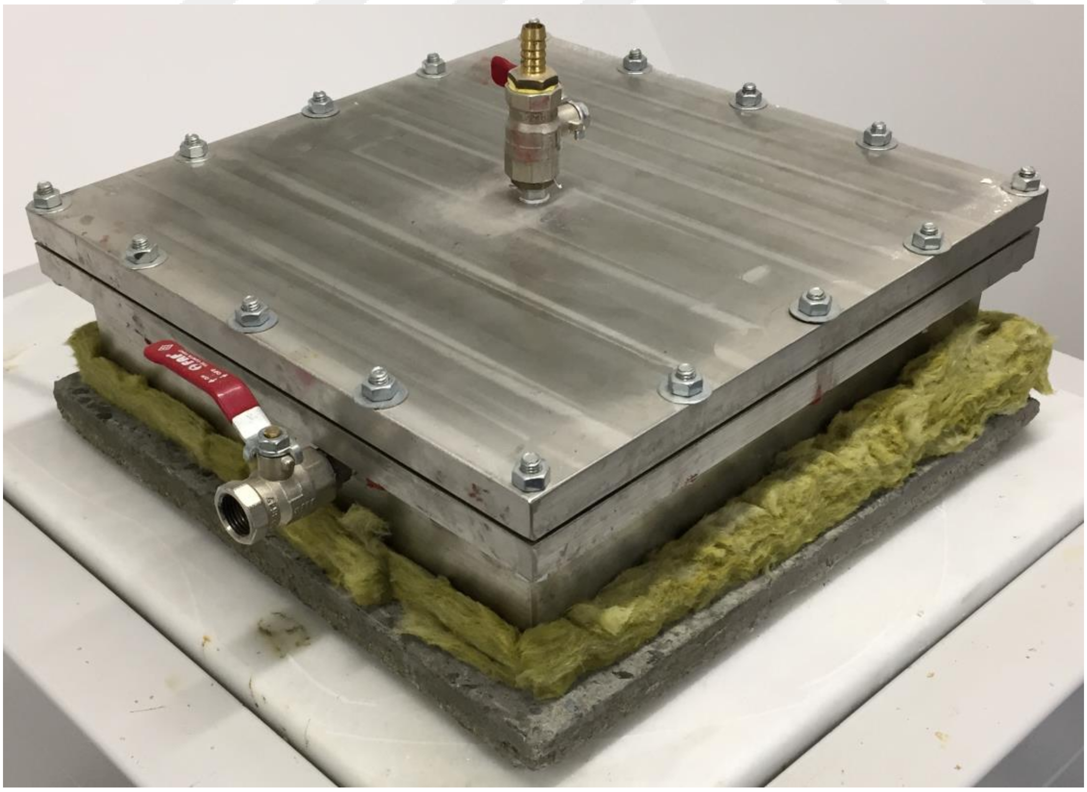


Figure 4.3: The Autoclave in special sizes.

This autoclave is successfully used for the research and development of prototype of composite materials in Istanbul Technical University, Aerospace Research Center. Working temperature is maximum 7 bar pressure, 220 degree temperature. The temperature increase can be set to 1 ^{o}C or 2 ^{o}C per minute.

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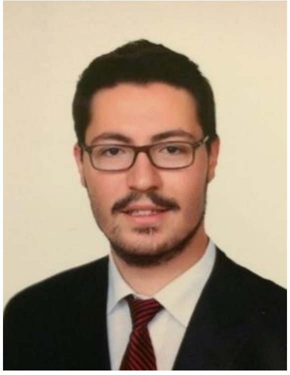

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