



MULTI OBJECTIVES PERFORMANCE OPTIMISATION OF NR/EPDM FOR COMPRESSION MOLDING PROCESS: PARAMETERS INVESTIGATION

This report is submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

MUHAMMAD RAFIQ BIN ROSMAN

B051510176

960625-04-5275

FACULTY OF MANUFACTURING ENGINEERING

2019

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

Tajuk: **MULTI OBJECTIVES PERFORMANCE OPTIMISATION OF NR/EPDM FOR COMPRESSION MOLDING PROCESS: PARAMETERS INVESTIGATION**

Sesi Pengajian: **2018/2019 Semester 2**

Saya **MUHAMMAD RAFIQ BIN ROSMAN (960625-04-5275)**

mengaku membenarkan Laporan Projek Sarjana Muda (PSM) ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. *Sila tandakan (√)

SULIT (Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysiasebagaimana yang termaktub dalam AKTA RAHSIA RASMI 1972)

TERHAD (Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/ badan di mana penyelidikan dijalankan)

TIDAK TERHAD

Disahkan oleh:

Alamat Tetap:

Cop Rasmi:

Tarikh:

Tarikh:

*Jika Laporan PSM ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh laporan PSM ini perlu dikelaskan sebagai SULIT atau TERHAD.

DECLARATION

I hereby, declared this report entitled “Multi Objectives Performance Optimisation of NR/EPDM for Compression Molding Process: Parameters Investigation” is the result of my own research except as cited in references.

Signature :
Author's Name : MUHAMMAD RAFIQ BIN ROSMAN
Date : 26 June 2019

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of University Teknikal Malaysia Melaka as partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Hons.). The members of the supervisory committee are as follow:

.....

(ASSOCIATE PROFESSOR DR RAJA IZAMSHAH BIN RAJA ABDULLAH)

ABSTRAK

NR/EPDM elastomer semakin popular dalam industri automotif yang semakin meningkat. Dengan pertambahan dan kepelbagaian aplikasi serta kajian untuk kemajuan masa akan datang, keperluan untuk mempunyai panduan yang tepat dalam pemrosesan elastomer ini telah meningkat dengan mendadak. Objektif utama dalam proses ini adalah untuk mencapai sifat mekanikal yang baik. Kajian ini telah dilakukan dengan mengkaji parameter pemrosesan (iaitu, suhu, tekanan, kadar masa pemanasan dan kadar masa tekanan) dan kesannya terhadap sifat mekanikal (iaitu kekuatan tegangan dan ketumpatan sambung) untuk menghasilkan kombinasi parameter yang optima dalam mencapai prestasi pemrosesan yang terbaik berdasarkan pelbagai objektif. Melalui Response Surface Methodology (RSM), reka bentuk eksperimen dipilih berasaskan pendekatan Box-Behnken dan seterusnya 29 susunan spesimen telah dirancang. Seterusnya, model matematik bagi setiap tindak balas dibangunkan. Kecukupan model dianalisis secara statistic menggunakan ANOVA dalam menentukan input pembolehubah (faktor) yang penting dan kemungkinan adanya interaksi di antara pembolehubah. Pelbagai faktor diagnostik dinilai untuk memeriksa keberkesanan model. Pengoptimuman dengan pelbagai objektif dilakukan melalui pengoptimum berangka dan keputusan yang dijangka akan disahkan. Pemerhatian dan analisis melalui eksperimen mencadangkan bahawa suhu dan kadar masa pemanasan merupakan faktor utama yang mempengaruhi kekuatan tegangan spesimen, manakala bagi tindak balas ketumpatan sambungan pula hanya dipengaruhi oleh satu parameter sahaja iaitu suhu. Kekuatan tegangan dan ketumpatan sambungan akan menurun sekiranya suhu dan kadar masa pemanasan meningkat disebabkan degradasi (suhu terlalu tinggi untuk suhu kerja NR/EPDM). Oleh itu, proses dibawah suhu titik degradasi NR/EPDM amat digalakkan bagi mengelakkan berlakunya pemotongan rangkaian di dalam getah tersebut dan seterusnya bagi meningkatkan keupayaan sifat mekanikal NR/EPDM.

ABSTRACT

Natural Rubber/Ethylene Propylene Diene Monomer (NR/EPDM) elastomeric is gaining popularity in the growing automotive industry owing to the fact in terms of sustainability. With extensive studies and an increasing number of applications for future advancement, the need for an accurate and reliable guide in processing this type of elastomer has increased enormously. Good mechanical properties are always the ultimate objectives. The present work deals with the study of compression molding parameters (i.e. temperature, pressure, heating time and pressure time) and its effects against NR/EPDM elastomeric mechanical properties (i.e. ultimate tensile strength (UTS), crosslink density and rubber fatigue behavior) aim on establishing optimized setup of processing parameters. The optimizations are achieved through the Response Surface Methodology (RSM), Box-Behnken approach as the design of the experiment. A mathematical model for each response is developed to access the relationship between the parameters. Adequacy of models is analyzed statistically using analysis of variance (ANOVA) in the determination of significant input variables and possible interactions. Various diagnostic plots are evaluated to check the model effectiveness. Multi objectives optimization is performed through numerical optimization and predicted results are validated. The agreement between experimental and selected solution are found to be strong in between 93% and 96%, thus validating the solution as an optimal run condition. The findings suggest that temperature and heating time is the main factor affecting ultimate tensile strength, whereas for crosslink density there is only one significant parameter which is temperature. UTS and crosslink density decrease with the increases of the temperature and heating time due to the degradation (temperature too high for NR/EPDM working temperature). Therefore, it is recommended to start the process below the NR/EPDM degradation point to avoid the scissoring rubber take place and subsequently improving the mechanical properties.

DEDICATION

Only

my beloved father, Rosman bin Mat Sahad

my appreciated mother, Ramnah binti Abu Samah

my adored brother, Muhamad Ridhwan bin Rosman

my friends, Amieza, Fazzuan, Arif, Fatihin, Farah, Wei Zuan

for giving me moral support, financial support, encouragement, and understandings

Thank You So Much & Love You All Forever

ACKNOWLEDGEMENT

First and foremost, in the name of Allah, the Almighty, the most merciful, the most gracious, with the highest praise to Allah that I manage to complete this final year project successfully.

Second, my sincere and deepest appreciation goes to my respected supervisor, Associate Professor Dr. Raja Izamshah Bin Raja Abdullah for the great mentoring and guidance that was given to me throughout the project. Besides that, I also would like to express my gratitude to all technicians for guidance and advise in handling the machine and equipment that I used throughout the project.

Last but not least, I would like to give special thanks to my parents for the continuous advice and motivation given to me to complete this project.

Finally, I would like to thank all my friends and who were directly or indirectly involved in this report, as well as expressing my apology that I could not mention personally each one of you.

TABLE OF CONTENTS

Abstrak	i
Abstract	ii
Dedication	iii
Acknowledgement	iv
Table of Contents	v
List of Tables	viii
List of Figures	ix
List of Abbreviations	xi
List of Symbols	xii

CHAPTER 1: INTRODUCTION

1.1	Background	1
1.2	Problem Statement	2
1.3	Objectives	3
1.4	Scope of the Research	3
1.5	Important of the Study	4
1.6	Organization of Final Year Project	5
1.7	Summary	5

CHAPTER 2: LITERATURE REVIEW

2.1	Compression Molding	6
	2.1.1 Technique	7
	2.1.2 Mold	8
2.2	Parameters of the Compression Molding	9
2.3	Polymer	11
	2.3.1 Elastomers	11
	2.3.2 Thermosets	12
	2.3.3 Thermoplastics	13

2.4	Rubber	14
2.4.1	Natural Rubber (NR)	14
2.4.2	Synthetic Rubber	16
2.4.2.1	Ethylene Propene Diene Monomer (EPDM)	18
2.4.3	Natural Rubber / Ethylene Propene Diene Monomer (NR/EPDM)	20
2.5	Design of Experiment	22
2.5.1	Response Surface Methodology (RSM)	22

CHAPTER 3: METHODOLOGY

3.1	Introduction	25
3.2	Research Flowchart	26
3.3	Apparatus and Experimental Procedures	27
3.3.1	Raw Materials	27
3.3.2	Mold	27
3.3.3	Machines and Tools	28
3.4	Data Analysis	30
3.4.1	Tensile Test	30
3.4.2	Swelling Test	30
3.4.3	Eccentricity Test	31
3.5	Experimental Parameters	33
3.6	Design of Experiment	34
3.6.1	Response Surface Methodology (Box Behnken Approach)	34
3.7	Summary	36

CHAPTER 4: RESULTS AND DISCUSSION

4.1	Experimental Results	37
4.2	Ultimate Tensile Strength	39
4.2.1	ANOVA Analysis of Ultimate Tensile Strength	41
4.2.2	Parametric Influence of UTS	42
4.3	Crosslink Density	48
4.3.1	ANOVA Analysis of Crosslink Density	49
4.3.2	Parametric Influence of Crosslink Density	50

4.4	Eccentricity	56
	4.4.1 ANOVA Analysis of Eccentricity	57
	4.4.2 Parametric Influence of Eccentricity	58
4.5	Optimization	62
4.6	Model Validation	64
4.7	Summary	66

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1	Conclusion	67
5.2	Recommendation	68
5.3	Sustainable Development	68

REFERENCES	69
-------------------	-----------

APPENDICES

A	Gantt chart of FYP 1	73
B	Gantt chart of FYP 2	74

LIST OF TABLE

2.1	Parameters condition and its effects	9
2.2	Summary of the parameters compression molding process for NR/EPDM	10
2.3	Classification of elastomers according to ASTM D 2000	12
2.4	The properties of NR	15
2.5	Types and applications of synthetics rubbers	18
2.6	The properties of ethylene propylene diene monomer	19
2.7	Environmental resistance of NR and EPDM	20
2.8	Summary of characteristics for natural, synthetic and combination elastomer	21
2.9	List of published studies on the polymer that using RSM	24
3.1	Parameters Data	32
3.2	Processing parameters in RSM using Box-Behnken approach	34
4.1	Experimental result of ultimate tensile strength	39
4.2	R-square analysis of ultimate tensile strength	41
4.3	ANOVA of ultimate tensile strength	42
4.4	Experimental result of crosslink density	48
4.5	R-square analysis of crosslink density	50
4.6	ANOVA of crosslink density	50
4.7	Experimental result of eccentricity	56
4.8	R-square analysis of eccentricity	57
4.9	ANOVA of eccentricity	57
4.10	Optical combination of input factors for single and multi-response optimization	63
4.11	Result for confirmation research for NR/EPDM elastomeric responses	65

LIST OF FIGURES

2.1	Compression molding part	7
2.2	Side view of a positive mold and flash mold	8
2.3	Compression molding parameters	9
2.4	Polymer chains in amorphous and crystalline regions	13
2.5	Natural rubber structure 2-methyl-1,3- butadiene	14
2.6	Polymeric chain of rubber	15
2.7	Ozone cracking that occurs on the side wall of the tire	16
2.8	Monomers chains that used to produce synthetic rubbers of EPDM	17
2.9	Structure of ethylene propylene monomer	19
2.10	Chemical structure of ethylene propylene diene monomer	19
2.11	Summary of RSM analysis flow	23
3.1	Several pieces of NR/EPDM elastomeric before being a process	27
3.2	Drawing of the flash mold using SolidWorks2013	27
3.3	Compression molding machine	28
3.4	Laser cut	28
3.5	Lever-controlled sample cutter	29
3.6	Analytical Balance	29
3.7	Press Machine	30
3.8	CNC roundness machine	31
4.1	Maximum average UTS	40
4.2	Minimum average UTS	40
4.3	Diagnostic plots for UTS	44
4.4	Perturbation graph of parameters towards UTS	45
4.5	3D surface graph of UTS vs temperature (A) vs heating time (B)	47
4.6	Maximum crosslink density	49
4.7	Minimum crosslink density	49
4.8	The diagnostic plots for crosslink density	52

4.9	Perturbation graph of parameters towards crosslink density	53
4.10	3D surface graph of crosslink density vs temperature (A) vs heating time (B)	55
4.11	Diagnostic plots for eccentricity	59
4.12	Perturbation graph of parameters towards eccentricity	60
4.13	3D surface graph of eccentricity vs temperature (A) vs pressure (B)	61

LIST OF ABBREVIATIONS

ANOVA	-	Analysis of Variance
ASTM	-	American Society for Testing and Materials
CM	-	Compression Molding
DOE	-	Design of Experiment
DRC	-	Dry Rubber Content
EPDM	-	Ethylene Propylene Diene Monomer
EPM	-	Ethylene Propylene Monomer
HT	-	Heating Time
IR	-	Isoprene Rubber
ISO	-	International Standard Organization
Max	-	Maximum
Mc	-	Crosslink Molecular Weight
NR	-	Natural Rubber
NR/EPDM	-	Natural Rubber/Ethylene Propylene Diene Monomer
Press	-	Pressure
PT	-	Pressure Time
RSM	-	Response Surface Methodology
SBR	-	Styrene Butadiene Rubber
Temp	-	Temperature
UTS	-	Ultimate Tensile Strength
Vc	-	Crosslink density

LIST OF SYMBOLS

°F	-	Fahrenheit
%	-	Percentage
°C	-	Degree Celcius
Cal/cc	-	Calories/cubic centimeter
cm	-	Centimeter
g	-	Gram
m	-	Meter
min	-	Minute
mm	-	Millimeter
MPa	-	Mega Pascal
N	-	Newton
psi	-	Pounds per square inch
sec	-	Seconds
β	-	Beta
ρ	-	Density

CHAPTER 1: INTRODUCTION

This introductory section tells of some of the subtopics that need to be present to give an early look at the research to be done. There are seven sub-topics that need to be included in this section which are the background of the study, problem statement, objectives, and scopes of the final year project. Furthermore, this section also describes significant of the study, organisation of the report and ends up with a summary for this chapter.

1.1 Background

NR and EPDM are the two types of rubber. These two types of rubber are mixed together to produce several mechanical properties. EPDM has better chemical resistance and weathering oxidation (Sahakaro, Naskar, Datta, & Noordermeer, 2006) while NR has high strength and good dynamic properties (Botros, 2007). The mixing between EPDM and NR has inclined upward thrust to their heat and ozone resistance (Sahakaro et al., 2006).

Various types of rubber based products can be produced using a variety of processes. One of them is the compression molding process. This process provides high production rates, accurate repeatability and produced an end product (Collyer, 2016). In general, this process has been introduced to the molding rubber compound starting from the twentieth century and it provides a vibrant development especially in the sector of automotive and appliance applications to this day. The compression molding process on this rubber is a process where the material is placed in the mold that had been heated at a certain temperature and then is pressed to form the product that looks exactly like the shape of the mold. An analogy for this process can be observed by making waffles.

The parameters play a very important role in the compression molding process primarily to produce products from rubber. Therefore, this parameter should be controlled so as not to cause damage to the product which causes the product to be rejected and will indirectly increase the high cost of making the product. From literatures it shows that there are only four parameters that influence the mechanical properties in compression molding which are temperature, pressure, heating time and pressure time. Therefore, this study is concentrate on optimization of compression parameter for NR/EPDM in order to have a

longer shelf life in term of strength and good hydrogel network in term of crosslink density.

In order to enhance the feedback that might be altered by several input variables, design of experiment (DOE) method is applied. This is carried out according box behnkkan design tools of Response Surface Methodology (RSM) using Design-Expert 8 P (DX8P) software for selected factors. With RSM method, the cost for this project can be reduced and the experiment becomes faster and effective (Kandar & Akil, 2016).

1.2 Problem Statement

In today's sophisticated technology era, there are various processes capable of producing various types of products from rubber. This matter indirectly causes various parties to seek and determine the appropriate parameters that can be applied when processing rubber to produce the best product without affecting mechanical properties and physical properties while reducing costs and extending the shelf life of the product. One of the famous processes is a compression molding which forming the rubber by means of heat and stress in the mold.

Every process has its disadvantages as well as compression molding. The compression molding process is slower and expensive (De and White, 1996). Compression molding process for NR/EPDM can be disturbed by mold curing and cause shrinkage, poor strength and crosslink. There are three factors related to compression molding process for rubber i.e. temperature, time and pressure (Campbell, 2004). In addition, the selection of the selected parameter value affects the rubber product quality. If the setting temperature is too high, the properties of fibers and matrix may additionally get modified. But if the temperature is too low, the fibers won't get nicely wetted. Same goes to pressure, if the pressure is too low, it leads to bad interfacial adhesion of fiber and matrix while if the setting pressure is too high, it will cause fiber breakage. For the heating time and pressure time, both must be set up in optimum condition or otherwise it will cause any defect

The different between the thermal linear growth of rubber and mold will affect the shrinkage of the mold cured rubber product while the poor vulcanization process will result of porosity. In addition, product that manufacture by NR/EPDM usually causes ozone cracking on the surface of the product and directly reduce the shelf life of that product. Thus, the right selection or optimum parameter selection are crucial for the rubber product quality which will be the main work that will be discovered by this project.

1.3 Objectives

Based on the challenges faced in compression molding process for NR/EPDM, this project intends to look on the following objectives:

1. To investigate on the effects of compression molding parameters (temperature, pressure, heating time, pressure time) of NR/EPDM.
2. To optimize the compression molding parameters for NR/EPDM elastomeric material for achieving high quality NR/EPDM product.
3. To validate on the effectiveness of the proposed optimum compression molding parameter

1.4 Scopes

In order to achieve research objectives and to plan the research procedure framework, the scopes of this study have to be predetermined. The compression parameters to be studied as aforementioned in the objective will be obtained from the specimen (NR/EPDM) that through the compression molding process using the following tests:

1. Response Surface Methodology (RSM).
2. Compression test (eccentricity).
3. Swelling test (crosslink density).
4. Tensile test (ultimate tensile strength).

1.5 Important of the Study

There are some potential benefits that can be gained after the completion of this study. First and foremost, is the temperature effect on the rubber? All materials have their molecular pattern. The molecules are constantly vibrating to each other. When a material is exposed to a high temperature causes the molecule to vibrate intensely. The impact of these molecular collisions causes the material to expand a bit. This phenomenon is known as thermal expansion. As the temperature changes from very hot to very cold, the materials expand and contract over and over again, eventually leading to cracks. However, this situation is different for rubber material as rubber will expand when exposed to low temperatures and shrink when exposed to high temperatures. This occurs because of the unusual polymer structure of rubber. When the long chains get hotter and vibrate, they actually shorten, causing the material to contract. When the chains cool down, they relax and stretch out, causing the material to expand.

Moreover, the pressure effect on the rubber. The pressure in compression molding is needed to do three kind of task. First, it forces the elastomer prep into the cavity and to fill the cavity. Second, is to hold the mold closed to enable proper flash removal and since some compounds would foam, to prevent foaming. Third, certain part dimensions will be off if the mold is not fully closed possibly leading to inferior part performance. Therefore, the pressure must be controlled to get these three kind of task can be done smoothly without any issues.

1.5 Organization

- i. Chapter 1 is an introduction chapter that explains about the background of this project, the problem statement that had been identified through observation and the objectives that must be achieved throughout the study. Then, followed by the scope of this project which narrows down the area of the study, important of this study is also revealed, organization for this project and summary for this chapter.
- ii. Chapter 2 is a literature review that explains on the basic theories regarding the research topic and the previous studies from journal, book and the internet and the methods for evaluating system and proposing alternative.
- iii. Chapter 3 is a methodology is an overview of study which explain on how the project is done by discussing the process and method that to be used.
- iv. Chapter 4 is a result and discussion that explains on the result of data that has been getting from rubber compression test, tensile test and swelling test.
- v. Chapter 5 is a conclusion and recommendation is an overview of the overall of a project that has been done.

1.7 Summary

All the parameters involved in the compression molding process are very important. This is because all these parameters have their respective roles in ensuring that the process goes smoothly. Indirectly, rubber-based products that go through this process can be manufactured with full quality that is able to satisfy the consumer. Therefore, the failure to control this parameter will result in the impairment of the product. However, how about controlling all the parameters? Hence, this research is able to give real value or range value for all these parameters so that it can regulate more are effectively when running the compression molding process on the rubber.

CHAPTER 2: LITERATURE REVIEW

The literature review was carried out with the attention to attain the objectives of this project. It consists of the facts associated with natural rubber materials, compression molding machine, overall performance observations and modelling by response surface methodology (RSM). All the information and facts in this section served as a reference and guidelines for this project.

2.1 Compression Molding

Compression molding is a process whereby heat and pressure are directed to the mold that has been placed together with the resin. This process is also a fast and stable process that is appropriate for processing materials such as thermoplastics, thermosets and elastomers. It has been used extensively for these polymeric materials. According to Allen et al. (2009), the compression molding process is usually done using a certain range parameter:

- i. Pressure: 2000 – 10000 psi on part
- ii. Cure time: 30 – 300 seconds
- iii. Temperature: 300 - 400°F

CM only focuses on producing homogenous, isotropic test specimens. This thing is not done by various other processes (Focatiis, 2012). CM also relies only on accomplishing controlled and repeatable thermal transition of the polymer being shaped. There are square measure elementary variations within the method that a solid state is achieved that depends upon whether or not the polymer being molded is in a position to crystallize.

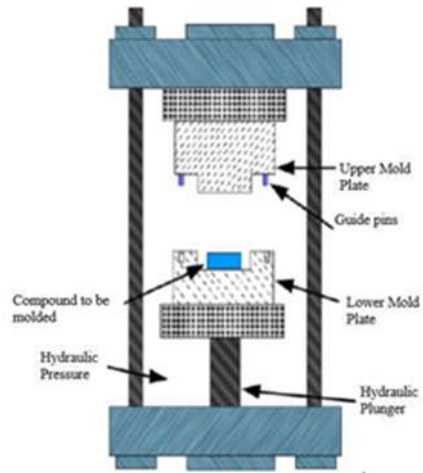


Figure 2.1: Compression molding part (Walker, 2012)

Although CM has been widely used in the manufacturing industry, it is also unlikely to run away from CM's shortcomings. One of them is that CM is struggling to not only eliminate and even reduce damage to specimens when it has been successfully removed from its molds, especially on the surface (Focatiis, 2012). This is due to the surface of the surface that goes directly to the surface of the mold.

2.1.1 Technique

The first step in starting the CM process is to heat up the mold first. Then, after the mold is heated, it is filled with suitable reference material on the mold basis. After that, the mold should be heated at certain temperature and time before being subjected to pressure on it. The pressure given will cause the mold to form the mold. However, upon completion of all these matters, the material should be hardened before the mold is opened to avoid defects in the compressed material. The hardening process is different for each substance. For example, for thermoplastic materials, it is affected by chilling and under pressure in the mold while for thermoset, further heating and under pressure in the mold that affects the hardening process. (Allen *et al.*, 2009).

2.1.2 Mold

There are some important things to know about the CM process. Among other things, the material inserted into the mold should be thicker than the latter but smaller, wide and long and located at the center of geometry. When a reference is made, the cavity will be filled by a preform scattered. However, there is one parameter that needs to be taken into account when implementing the CM process (Bickerton & Abdullah, 2003). This parameter is the separation of the platen mold at the beginning of the injection of the resin. The early separation is defined as the resistance offered by the mold cavity by filling and also the dominating charging properties.

Flash molds and positive molds are two types of molds. The first is about positive molds. This mold is designed to produce direct pressure to the polymer to be formed. Therefore, this mold is difficult to design because all cavities in solid metal need to be compressed. Furthermore, the molded specimens are difficult to remove unless it has been sufficiently shrinking to pull the specimen out of the mold. This is different from the second mold which is flash molds because it is designed for no pressure on the polymer formed so that this polymer can be pulled out of mold during molding. According to (Focatiis, 2012), flash molds are also easy to install as they can be produced from suitable sheet thickness of the sheet metal.

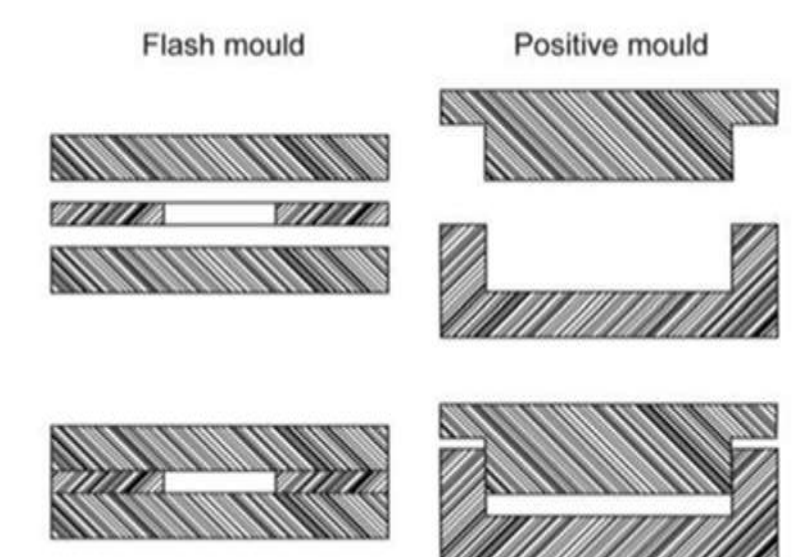


Figure 2.2: Side view of a positive mold and flash mold (Institution, 2005)