Study on Mechanical and Fracture Behaviour of Linear Low Density Polyethylene for Rotational Moulding

THESIS

Submitted in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

by

RAMKUMAR PL

Under the supervision of

Prof. D. M. KULKARNI

BIRLA INSTITUTE OF TECHNOLOGY AND SCIENCE, PILANI

2015

BIRLA INSTITUTE OF TECHNOLOGY AND SCIENCE, PILANI

CERTIFICATE

This is to certify that the thesis entitled **"Study on Mechanical and Fracture Behaviour of Linear Low Density Polyethylene for Rotational Moulding"** and submitted by **RAMKUMAR PL** ID No **2009PHXF423G** for award of Ph. D. Degree of the Institute embodies original work done by him under my supervision.

Signature in full of the Supervisor: ______________________

Name in capital block letters: **PROF. D. M. KULKARNI**

Designation: **Professor**

Mechanical Engineering Department

Dean, Administration

BITS Pilani- KK Birla Goa Campus

Date: 30-Sep-2015

ACKNOWLEDGEMENTS

I would like to record my deepest gratitude to my PhD supervisor, Prof. D. M. Kulkarni for his role in shaping my thought process. His knowledge, keen interest, constant encouragement, help, constructive criticism and endless patience have contributed greatly to making this doctoral study a successful and enjoyable journey. This work would not have reached at the conclusive end without his guidance. Besides technical support for including practical validation of various research results, his humanly support in challenging times is gratefully recorded. It has been a privilege for me to work under his guidance.

I am grateful to V.S Rao, Vice Chancellor, BITS Pilani and Prof. B.N. Jain Former Vice Chancellor, Prof. Sasikumar Punnekkat, Director, and Prof. K. E. Raman, Former Director, BITS, Pilani- K.K. Birla Goa Campus, Prof. Sanjay Kumar Verma, Dean, Academic Research, BITS, Pilani- for not only giving me an opportunity to carry research studies at the institute but also by providing necessary infrastructure and facilities to carry out my work.

Special thanks are due to Dr. Shibu Clement Head, Department of Mechanical Engineering and Dr. P.M. Singru, former Head, Department of Mechanical Engineering, BITS-Pilani, K. K. Birla Goa Campus, who allowed me to use laboratory facilities for the present study. My special thanks to Prof. N.N. Gosh and Dr. Shibu Clement for their valuable comments and role in shaping this work. Their valuable suggestions have helped in greatly enhancing the quality of the thesis. I am grateful to Dr. Prasantha Kumar Das, Associate Dean Academic Research Division and Dr. S.D. Manjare, Former Faculty-Incharge, Research and Consultancy and Education Development Division, BITS PIlani- K. K. Birla Goa Campus for his constant encouragement throughout the course of this study.

I wish to owe my special thanks to Dr. Sachin D. Waigoankar and Vikas Vinayak Chaudhari for the much needed technical support as well as encouragement at different times in the course of my study. I am thankful to all the senior Professors as well as my fellow colleagues in Department of Mechanical Engineering for providing me moral support and encouragement.

I wish to thank Mr. G. J. Desai and Mr.Vijay Suryavamshi, workshop superintendent, for their immense and continuous help in words and deeds, especially during course of experimental work. My sincere thanks also go to Mr. Vilas, Mr. Elappa, Mr. Haider Khan, and all the workshop staff of our institute who helped me during the work.

My parents deserve special mention for their unstinting moral support and prayers for completion of this work. Without their encouragement, this endeavour would not have been possible.

I also express my appreciation to my wife Vallimayil for her understanding, patience and active co-operation throughout the course of my work.

Finally, I sincerely thank all those, who have helped me either directly or indirectly at different stages of my research work.

Ramkumar PL

ABSTRACT

Plastics play a central role in today's life. It offers variety of benefits compared to other materials in various sectors like automobile, construction and packaging etc. There are numerous methods for processing plastics. These include: blow moulding, injection moulding, rotational moulding, transfer moulding and thermoforming. Selection of a suitable manufacturing process is one of the key decisions that are faced while designing plastic products. There are some crucial applications of plastics products such as automobile fuel tanks, body armor and riot shields for military, large solvent drums etc. where mechanical and fracture characterization is of great importance. Also there is a wide range of polymeric materials available. Various applications demand typical processing method and typical polymeric material in order to enhance the quality. Therefore, it is required to investigate optimum process conditions of plastic manufacturing process, yielding favourable mechanical and fracture properties. Though lightness and simplicity in processing methods of plastics attracted the designers and manufacturers, a complete understanding of process, process parameters and effect of process parameters on mechanical and fracture characterization is required to attain high standard products and its performance.

Objective based multiple attribute decision making method is suggested for the selection of plastic manufacturing process. Through this exercise, rotational moulding process is suggested as a process based on important attributes like cycle time, wall thickness, material availability etc. Rotational moulding is a high temperature, low pressure thermoplastic processing method for producing hollow parts. Among the different polyethylenes, linear low density polyethylene (LLDPE) is used in rotational moulding process because of its unique melt flow properties and favourable processing window.

Many products made by rotational moulding process using LLDPE are widely used in outdoor applications such as boats, over head tanks, and car body parts etc. In such applications, mechanical and fracture properties are considered to be critical from the quality characterization point of view. Therefore, there is a need to investigate the mechanical and fracture properties of rotomoulded products, which in turn depends upon the process parameters.

Present study aims to investigate the effect of process parameters on mechanical and fracture properties of rotational moulding products made using LLDPE. Simulation studies and experimental procedures are adopted to characterize mechanical and fracture behaviour of rotomoulded products. Simulation studies are conducted using ROTOSIM software. In order to ensure that all the factors and their interactions are systematically investigated, two approaches of design of experiment are used to plan and analyze the experiments. These are 2^2 factorial design and Box-Behenken design of response surface method (RSM). Experiments are conducted using rotomoulding machine and universal testing machine.

In rotational moulding of plastics, improving the mechanical properties like tensile strength and impact strength without sacrificing the processability is the biggest challenge. Therefore, an attempt has been made to investigate the effect of oven temperature, oven residence time and cooling media on the mechanical properties of the rotationally moulded products using simulation and experiments. A regime of optimal processing window is identified where the superior tensile, flexural and impact properties are noticed. In order to obtain high stiffness to weight ratio and good thermal insulation, polymer foams are added to base resin in rotational moulding process. The present attempt also aims to assess the rotomouldability of foamed polyethylene products and its properties. From the preliminary experiments it is predicted that 6% of foam is the optimum level that needs to be mixed with the base resin LLDPE to obtain sufficient melt flow for ease of processing and better impact strength. Experiments are planed and analyzed to determine the effect of process parameters on the impact property of the foamed rotomoulded products. Experimental results confirm that oven temperature, oven residence time and cooling medium are the principal process parameters affecting impact property of foamed rotomoulded products. Optimum process parameters yielding desired impact strength are determined.

In rotational moulding process, moulders have to depend heavily upon trial and error methods as well as experience of the operator to predict the thickness for a particular speed ratios and oven residence time. Efforts have been made to investigate the thickness of the rotational moulded parts for different speed ratios and oven residence time using experiments. Experimental results confirmed that both the process parameters ie., Oven residence time and speed ratio have significant effect on thickness of the rotomoulded products. Equation has been derived from the statistical regression model to conveniently predict the thickness for any combination of these process parameters within the experimental regime.

Investigations of fracture characteristics of rotomoulded products are carried out using universal testing machine. Since, components produced using rotationally moulding process are used in outdoor applications like oil /chemical storage tanks, automobile components, machine housing, ducts etc. fracture toughness of such products is considered as one of the essential quality feature. Oven temperature, oven residence time and cooling medium are selected as principal parameters. *R*–curve method is used to characterize the fracture behaviour of rotomoulded products made using LLDPE for various process parameters. Experiments are planed and analyzed to determine the optimum values of process parameters yielding maximum fracture toughness. Confirmatory experiments are performed to validate the predicted results.

It is concluded from the extensive experimentation that the mechanical and fracture characterization are important quality measures for rotomoulded products which in turn depends on the process parameters. From the experimental finding it is found that oven residence time, oven temperature and cooling medium are the principal process parameters which affect mechanical and fracture properties. Therefore, optimal fixations of these process parameters are required for obtaining desired mechanical and fracture properties of rotomoulded products.

TABLE OF CONTENTS

CHAPTER- 7

CHAPTER- 8

LIST OF FIGURES

LIST OF TABLES

LIST OF SYMBOLS

Y_{ij}	quantitative/qualitative value associated with each attribute
r_{ij}	normalized value of each attribute
V_j	statistical variance of the data corresponding to the j^{th} attribute
Q_j^o	objective weight of the j^{th} attribute
X_i^o	product of Q_j^o and r_{ij}^{**}
P_i^o	preference index
P	probability of accepting null hypothesis in ANOVA
t	‘t’ test value in ANOVA
R^2	Squared multiple correlation coefficients or the coefficient of determination
Δa	crack growth
W_f	total work of fracture
W_e	essential work required to fracture the polymer
W_p	non essential work required to fracture the polymer
J	*J*- integral, a fracture parameter
J_{IC}	*J*- integral, at crack initiation
m	plastic constraint factor
$б_y$	yield stress
T_m	melting temperature
E'	storage Modulus

E''	loss modulus
T_g	glass transition temperature

LIST OF ABBRIVATIONS

LLDPE	linear low density polyethylene
MFI	melt flow index
ASTM	American society of testing and materials
CT	compact tension
DOE	design of experiments
RSM	response surface method
MADM	multi attribute decision making method
PIAT	peak internal air temperature
DSC	differential scanning calorimetry
ANOVA	analysis of variance
LEFM	linear elastic fracture mechanics
EWF	essential work of fracture
COD	crack opening displacement

CHAPTER 1

INTRODUCTION

Traditional structural metals such as steel and aluminum are being replaced with plastics in numerous applications. Today it would be hard to imagine a modern society without plastics. Plastics have found a myriad of uses in fields as diverse as household appliances, packaging, construction, medicine, electronics, and automotive and aerospace components. As can be seen from this list, plastic technology can be applied with great success in a variety of ways. They have great versatility and offers properties like durability, cost effectiveness, low maintenance and corrosion resistance, etc. Reason for its success in replacing other materials in different applications is the ability to modify its properties and ease of processing. Though plastics are attractive to manufacturers, it has certain limitations regarding recycling of the same and its properties. Their properties can be essentially improved by adding additives, fillers and foams. Selection of a suitable manufacturing process is one of the key decisions that are faced while designing plastic products. Rotational moulding is a competitive alternative to other plastic manufacturing process, since it offers designers an opportunity to achieve the economic production of stress free products. Majority of rotational moulding products are made from polyethylene and out of all polyethylene linear low density polyethylene (LLDPE) is preferred. Numerous efforts have been taken to know the mechanical characterization of rotomoulded products, however as rotomoulded products are used in critical applications it is necessary to know the effect of process parameters on mechanical and fracture behaviour of rotomoulded products.

1.1 Manufacturing Process Selection of Plastics

Plastic parts can be manufactured by employing a wide variety of manufacturing process such as blow moulding, injection moulding, rotational moulding, transfer moulding, and thermoforming. Each plastic manufacturing process has some distinct merits and demerits. For producing any plastic products, one of the crucial decisions made during the design stage is process selection. The manufacturing process affects productivity, cost, and quality of the part. Traditionally, the decision to select an appropriate manufacturing process is delegated to an expert who employs a complex reasoning process based on empirical knowledge and past experience. This selection procedure may result in inconsistent or poor choices if the decision is handled by a novice who fails to map correctly the product characteristics with the manufacturing efficacy of various manufacturing processes.

During manufacturing process selection, design and manufacturing teams have to consider various sets of attributes to get optimal results. Attributes can be either quantitative or qualitative in nature. Attributes that influence selection of a process for a given application may include: operational parameters, environmental subsystems, human subsystems, manufacturing subsystems, final product quality, material properties, mould parameters, performance characteristics, availability, etc. Decision making process like selection of a manufacturing process for a particular application based on the combination of these attributes is a complex job. Therefore, there is a need for simple, systematic, and logical methods or mathematical tools to guide decision makers in considering a number of selection attributes and their interrelations. Thus, this study involves the application of objective based multi attribute decision making method (MADM) to address the issue of the process selection.

1.2 Process Taken for Study: (Rotational Moulding Process)

After going through multi attribute decision making method for manufacturing process selection of plastics, the decision is arrived to propose rotational moulding process out of many. Rotational moulding is a polymer processing technology used for producing hollow seamless articles by heating, melting and subsequent sintering of polymer powders (LLDPE) in bi-axially rotating moulds, followed by cooling the melt for solidification. It is used to manufacture hollow parts at low cost. The process is economical as one piece plastic parts can be made, thereby discarding the need of costly assembly. As this process is stress free, components produced using this process have better mechanical properties. The main advantages of this process are:

1. Hollow parts can be made in one piece.
2. Moulds used are inexpensive.
3. Multilayered products can be made.
4. In mould graphics is possible.

5. Large parts can be produced economically using this process.

To manufacture desired end product in rotational moulding process, different varieties of powdered resins are used which include Linear Low Density Polyethylene (LLDPE), Polypropylene, High Density Polyethylene (HDPE), Polyvinyl Chloride (PVC), Polycarbonate, Polyester, Acronitrile Butadine Styrene (ABS), Nylon, etc.

Some common products produced using rotational moulding process are industrial and agricultural storage tanks, industrial equipments, automobile parts, traffic barriers and battery cases. Fig 1.1 shows different products out of rotational moulding process.

Fig 1.1 Products out of rotational moulding

1.3 Process Simulation and Process Modelling of Plastics

Before last one decade, the demands on rotomoulded products were not too many. Therefore, rotomoulders or researchers have not concentrated on achieving optimum process conditions or product performance. Also, the apparent simplicity of the process meant that it attracted little attention from the research community. However, in recent years the demands of rotomoulded products have been increasing steadily with higher level of customer expectations. Thus, there is well defined need to have better understanding of technology of the process and its parameters. The rotational moulding process has been extensively reviewed by many researchers. Crawford, (1996) realized the importance of study of process parameters. According to him the influential factors in descending orders are: mould design and construction to achieve faster cooling, oven temperature, shrinkage, oven residence time, new mould materials, recycling, and internal mould release agents. It is highlighted that moulders use a rule of thumb to decide the various process parameters and have to depend heavily upon trial and error methods as well as experience of the operator. It is found that there are number of studies that successfully depict the mechanical properties like tensile, impact and flexural strength of rotomoulded products however, there is lack of insight on the effect of process parameters on those properties. Therefore, there is a crucial need to know the effect of these process parameters on the mechanical properties of final product quality.

Rotational moulding process is characterized by the compound nature of heat transfer involving several phase and density changes of polymer. Moreover, the phase changes occur over a range of temperature. Further, due to the bi-axial nature of mould rotation, there is a dearth of accurate thermal data measurement techniques and equipment at lesser cost except ROTOLOG (Crawford & Nugent, 1992). This necessitates reliable prediction of critical timings of the process like switching off the oven, de-moulding time, etc. The prediction can also economize the energy inputs to the processes and save significant energy as well as time. To the best of our knowledge, ROTOSIM software takes into account all the possibilities that exist in a typical rotational moulding process. It is a general practice in a rotational moulding industry to use trial and error methods to find the right moulding conditions. Sometimes what is considered as right moulding condition may not be the most optimum conditions in terms of degree of curing or cycle time efficiency. Therefore, in the present study the use of computer simulation software called ROTOSIM is used to assist in predicting the moulding conditions of rotomoulded parts.

Though polymeric material can be easily processed, in depth understanding of the process is required to achieve desired product quality. Process simulation and experimental investigations have assisted in this regard.

Process simulation helps in understanding, analyzing and optimizing the process. In order to depict the process and derive required characteristics, experiments are accomplished. Increasing productivity and improving quality are the key aspects of any process. As the experimenter cannot rely on costly and time consuming trial and error method to know the effect of various process parameters on the process, it is required to plan the experiments to yield a meaningful conclusion. Design of experiment (DOE) is an efficient procedure to reduce the number of experiments. In this study, two approaches of design of experiment are used to plan and analyze the experiments that are full factorial design and Box-Behenken design of response surface method (RSM). These methods are used to model and optimize the rotational moulding process resulting better quality of rotomoulded products. The experimental investigation mainly concentrates on applying the above methodology to know the effect of process parameters on thickness, impact strength and fracture toughness of rotomoulded products.

Many investigations are available which characterizes rotational moulding process of pure thermoplastics materials, however, very few investigations are found on characterizing foamed rotomoulded products. In order to improve the mechanical properties the hollow cavity of rotomoulded products can be filled or mixed with foam. Rotational moulding of foamed polyethylene has increasingly become an important process in industry. It has been used to produce parts in various applications such as fuel tanks, furniture, toys and novelties, flotation and drink containers etc. Foamed structures provide several advantages in thermoplastic products, including: a lightweight, excellent strength–weight ratio, superior insulation abilities; and energy absorbing performance (shock, vibration, and sound) (Shih- Jung Liu and Ching-Hsiung Yang, 2001). The present study is based on application of experimental techniques like DOE involving, response surface method to model and optimize the process parameters of rotational moulding process resulting a better quality foamed rotomoulded product.

1.4 Fracture Behaviour of Plastics

Although mechanical properties are given due attention, fracture behaviour of polymeric materials has recently become a major concern as engineering plastics have begun to appear in critical structural applications. One of the major polymers used in rotational moulding industry is polyethylene which is a tough, semi-crystalline thermoplastic that has found increasing use in several key engineering applications such as a pipeline material for water and gas transportation, fuel tanks, large solvent drums. However, there are few critical applications which demand resistance to fracture e.g. light weight body armor and riot shields for military and law enforcement personnel. Since the components fabricated from polyethylene are more often prone to failure and this tendency of failure is promoted by the presence of sharp flaws, especially at room temperatures (Carla et al., 2007). There are three ways in which these sharp flaws can appear in a structure. First, they can exist in a material due to its composition; second, generation of flaw in a structure during fabrication, as in welds; and third, flaws may generate during the service life of a component, like fatigue cracks, environment assisted or creep cracks. Fracture mechanics study can anticipate the load-bearing capacity of structures in the presence of initial defects.

In order to increase the quality and reliability of the rotomoulded products, researchers handled different methods, processes and polymers. This ultimately improved some of the mechanical properties influenced by the process parameters like temperature inside the mould, surface tension, vacuum and pressure etc. Even though different methods and processes are adopted in rotational moulding process, optimized parameters which in turn give optimized product quality are not clearly defined. Rotational moulding products are used in some critical applications like oil tanks, chemical tanks and industrial equipment etc. In view of criticality of the application of the rotational moulding products, it is necessary to know the fracture behaviour of the rotational moulding products which is also considered as a major grey area to address in this study. So, fracture behaviour of a LLDPE is one of the important characteristics required to be evaluated.

1.5 Scope of Present Work

Although the rotational moulding technique has been developed for more than three decades, the research efforts in rotational moulding process are still limited. The present characterization of rotational moulding process clearly emphasizes that process parameters plays an important role in producing the effective and reliable products in rotational moulding. However, no attempt is made to address the correlation between the process parameters in rotational moulding process and mechanical and fracture behaviour of rotomoulded products. Even though different methods are adopted in rotational moulding process, optimized process parameters which in turn give optimized product quality are not clearly defined.

1.6 Objectives of Present Work

Based on the existing industrial demand, following objectives are framed in order to ensure the quality of the rotomoulded products.

1. To explore an objective based multi attribute decision making (MADM) criterion for the selection of manufacturing process of plastics.
2. To investigate the effect of oven residence time on the mechanical properties of the rotationally moulded products.
3. To examine the effect of process parameters on thickness of the rotomoulded products.
4. To investigate the effect of foam percentage on the melt flow index and impact strength of LLDPE products using rotational moulding process and to determine significant process parameters affecting impact strength of foamed rotomoulded products.
5. To study the Effect of process parameters on fracture toughness of LLDPE product using rotational moulding process.

1.7 Organization of Thesis

To present the subject matter in a logical order, the thesis work is described in different chapters as follows:

Chapter-1 deals with introduction to plastics and selection of appropriate manufacturing process for plastics. A few details about rotational moulding process have been emphasized along with the applications of the same. Further, the need of process simulation and experimentation for the above processes has been specified and scopes as well as objectives of the present study have been defined.

In Chapter-2 the scope and objectives are identified through a review of literature. The content of this chapter is grouped into three major sections. The first section demonstrates the selection of suitable plastic manufacturing process for specific application using multi attribute decision making method. Second section covers the present characterization of rotational moulding process. Same section also covers different process parameters and influence of those on mechanical properties of the rotomoulded products. This section also emphasizes on process simulation and application of different additives in rotational moulding process. Third section discusses the present status and characterization of fracture behaviour of polymers. Finally, all the important findings of different researchers have been assessed and gap areas are identified.

Chapter-3 discusses about the process selection methodology and process simulation using ROTOSIM software. This chapter also discusses about experimental procedures adopted to characterize mechanical and fracture behaviour of rotomoulded products. Subsequent topics focus on different standards and testing methods to fulfill the proposed methodology.

Chapter-4 explores an objective based MADM method for plastic manufacturing process selection. This criterion considers both qualitative as well as quantitative attributes to evaluate, compare and address optimal selection of plastic manufacturing process. The method uses fuzzy logic to convert qualitative attributes into the quantitative attributes. The procedure has been adapted to rank different processes, to quantitatively assist a designer to select a suitable process from a long list for a specific application. A case study of process selection for a plastic moulded automotive fuel tank is used to explain the intricacies involved.

Chapter-5 discusses the investigation on effect of oven residence time on the mechanical properties of the rotationally moulded products. In this chapter, simulation studies using

ROTOSIM software is proposed to assess the thermal transitions, phase changes that occur in the process and determine the degree of curing of the polymers. Experiments are further conducted to verify the simulation predictions and to suggest the optimal oven residence time to get the highest possible mechanical properties of the products.

Chapter-6 examines the effect of process parameters on thickness of the rotomoulded product. Experiments based on full factorial method of design of experiments are planned and conducted to determine significant process parameters affecting the thickness of rotomoulded products.

Chapter-7 discusses the investigation on effect of foam percentage on the melt flow index and impact strength of LLDPE product using rotational moulding process. Also, it determines the effect of process parameters on impact strength of foamed rotomoulded products using response surface method (RSM) of design of experiment. Experimental results suggest the optimum level of foam percentage which yields better melt characteristics and impact strength. A statistical process optimization is further carried out to predict optimum process parameters yielding maximum impact strength.

Chapter-8 presents the experimental investigations of effect of process parameters on fracture toughness of LLDPE product using rotational moulding process. Experiments are planned based on Box-Behenken design of RSM to determine significant process parameters affecting fracture toughness of rotomoulded products. *R- curve* method is used to determine the fracture toughness of specimens for each combination of process parameters. Statistical process optimization is further carried out to predict optimum process parameters yielding maximum fracture toughness of the rotomoulded products.

Summary, outcome of present research work, specific contributions and recommendation for the rotomoulders are discussed in Chapter-9. A few suggestions for the future scope of work are also mentioned at the end of Chapter-9.

The list of tables, list of figures, list of symbols, list of abbreviations and list of subscripts are presented after contents. The references are cited in the text by author(s) name(s) with year of publication in parenthesis. In reference section, the references are listed alphabetically by author's names, followed by initials, year of publication, title of the article, name of the journal, volume number, and numbers of first and last pages. The list of publications is shown after the reference section. Appendices are labeled as A, B, C … etc. Various results of simulations, details of estimation of regression coefficients and plots pertaining to tensile tests, are included in Appendices. The brief biography of the supervisor and the student is given in the last two pages.

CHAPTER 2

LITERATURE SURVEY

In this chapter, the state of the art on process selection and present characterization of the process using simulation and experimental methods has been reviewed. The literature survey is divided into three main categories. These are: Process selection using multi attribute decision making (madm) approach, rotational moulding process and literature on fracture behaviour of polymers.

2.1 Manufacturing Process Selection Using MADM approach

For producing any plastic products, one of the crucial decisions made during the design stage is process selection. Manufacturing process affects productivity, cost, and quality of the part. In many industries, selection of the manufacturing process is primarily based on the empirical knowledge and past experience of the design and manufacturing experts. This selection procedure would be failure if the decision is made by an inexperienced person. Plastic parts can be manufactured by employing a wide variety of manufacturing process such as blow moulding, injection moulding, rotational moulding, transfer moulding, and thermoforming. Each plastic manufacturing process has some distinct merits and demerits (Tool and Manufacturing Engineers Handbook, 1996). The handbook by Bralla (1986) provides an excellent review of the various manufacturing processes and their suitability under various attributes such as material type, overall shape and size, and production volume. Various approaches were proposed in the past to help address the issue of process selection. Yu *et al.,* (1993) has proposed an expert system that helps designers select a manufacturing process in the early stage of product design. The proposed system used the concept of design compatibility analysis to represent the suitability of candidate processes with respect to the given product specifications. Raviwongse *et al.,* (2000) has developed a plastic manufacturing process selection methodology using self-organizing map (SOM) /fuzzy analysis.

Selecting manufacturing process for any specified application, various important criteria or attributes are needed to be considered. Process selection attributes are defined as attributes that influence selection of a process for a given application. These attributes include: operational parameters, environmental subsystems, human subsystems, manufacturing subsystems, final product quality, material property, mould parameters, material impact on environment, performance characteristics, availability, market trends, cultural aspects, aesthetics, recycling, target group, etc. Optimal selection of manufacturing process for particular application keeping those attributes in mind has been a complex job in any industry. The selection of an optimal process for a specific application among two or more alternative processes on the basis of two or more attributes is a multiple attribute decision making problem. The selection decision is complex as process selection is more challenging today. There is a need for simple, systematic, and logical methods or mathematical tools to guide decision makers in considering a number of selection attributes and their interrelations (Rao, 2007 & 2010). The main focus of any process selection method is to obtain suitable selection attributes and to identify proper combination of attributes in association with the actual requirement. Therefore, attempts has to be made to determine those attributes that effects the process selection for a desired application as well as to remove inappropriate alternatives, and to choose most suitable alternative using simple and logical methods. For the above mentioned situations MADM or multi-criteria decision making (MCDM) methodology is suitable for selection of a product or process based on a complex set of attributes. MADM methodology is applied in a wide range of areas like selection of electroplating system (Abhishek and Agrawal, 2009), selection of a supplier (Li *et al.,* 2007), selection of mechatronic systems (Kiran *et al.*, 2011). Yang and Hung (2007) identified techniques for order of preference by similarity to ideal solutions (TOPSIS) as a viable method for the selection of plant layout and is found suitable for the use of precise performance ratings. MCDM based on TOPSIS methodology is proposed by Thirumalai and Senthilkumaar (2013) for selecting optimum machining parameters. Sayed and Shahnaz (2013) discussed a hybrid fuzzy multi-criteria decision making approach for desalination process selection. Darji and Rao (2014), Anojkumar *et al.,* (2014) presented MCDM method for material selection of pipes in sugar industries. Caliskan *et al.,* (2013) used MCDM method for material selection of tool holder in milling process. The new approach of MADM is introduced by Quan Zhang *et al.*, (2003) which integrate subjective and objective information.

MADM along with TOPSIS approach is used for evaluation and optimal selection of robots (Bhangale *et al.*, 2004). Authors have developed a quantitative model using above methodology and compared the results with other graphical methods like line and spider graph.

MADM is also used as a methodology for optimum selection of manufacturing process for a composite product system by Durai Prabhakaran *et al.* (2006). With the help of this approach, authors ranked chopped carbon fibers as the best choice for resin transfer moulding process. Waigaonkar *et al.,* (2008) used MADM and TOSIS approach for resin selection in rotational moulding process. The procedure has been adopted to rank different resins, to quantitatively assist a rotomoulder to select a proper resin from a long list for a specific application.

2.2 Rotational Moulding Process

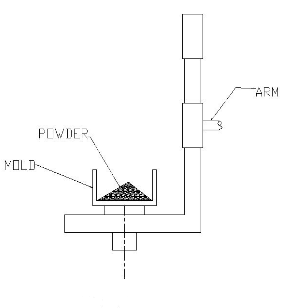
Even though a lot of manufacturing processes are available to produce hollow plastic parts like injection moulding, thermoforming, blow moulding etc., currently rotational moulding is a competitive alternative to all of those moulding processes. Rotational moulding (also called as rotomoulding or rotational casting) is a thermoplastic processing method for producing hollow parts, ranging from most simple to complex geometries (Amara Ait Aissa 2012, Crawford 1996, 1992). The products obtained from rotational moulding find wide applications in various fields like agriculture, storage tanks, industrial equipments, medical devices, material handling, road/highways, automobiles, etc. (Brent strong 2006, Crawford and Kearns 2003)

The advantages of rotational moulding process are: (Lopez Banuelos 2012, Brent strong 2006, Crawford and Kearns 2003)

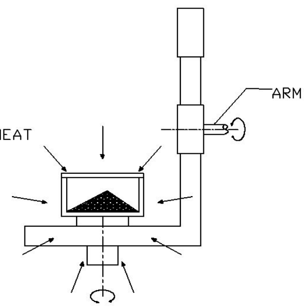
a) The parts produced by rotational moluding process are relatively stress free as compared to other moulding process like injection moulding or blow moulding, since in rotational moulding the plastic is not forced to take up a shape that is not natural for it.
b) With proper design, parts assembled from several pieces can be moulded as one single part (with no weld lines or joints), eliminating high fabrication costs.
c) Large parts can be produced economically using this process.

Different thermoplastic powders are used for rotational moulding like low density polyethylene (LDPE), linear low density polyethylene (LLDPE), Metallocene catalyzed linear low-density polyethylene (m-LLDPE), cross linked polyethylene (X-LPE), polypropylene (PP), acrylonitrile-butadiene-styrene (ABS), nylon (PA11), polyvinyl chloride (PVC) etc. Out of these polymers, LLDPE is mostly preferred for rotational moulding process (Amara Ait Aissa 2012, Brent strong 2006, Crawford and Kearns 2003). LLDPE is a linear polymer (polyethylene), with significant numbers of short branches, commonly made by copolymerization of ethylene with longer chain olefins. LLDPE has unique melt flow property which makes it suitable for rotational moulding process. Another important property which fits this to the process is its less shear sensitivity. The lower shear sensitivity of LLDPE allows for a faster stress relaxation of the polymer chains. The rheological properties of LLDPE are summarized as ‘stiff in shear’ and ‘soft in extension’. Few critical applications of rotomoulding process using LLDPE (along with other materials and additives) material are automobile fuel tanks, oil tanks, chemical tanks, traffic barriers, boats and material handling trolleys. These applications demand superior mechanical properties like impact strength, tensile strength, viscosity, flexural strength and density.

Fig 2.1(a-d) shows four different stages in a rotational moulding process. Fig 2.1(a) shows charging of the mould with polymer powder typically LLDPE of melt flow index 4.5 – 5.5g/10 min. After charging, biaxial rotation (rotator speed ratio is typically 4:1) in a convective heated environment is achieved by supplying heat externally as shown in Fig 2.1(b). Cooling with mould rotation (Fig 2.1 (c)) is done where the melted polymer powder gets settled to the walls of the mould. Finally, demoulding (Fig 2.1 (d)) is done to get the product.



(a) Mould Charging



(b) Heating and Rotation

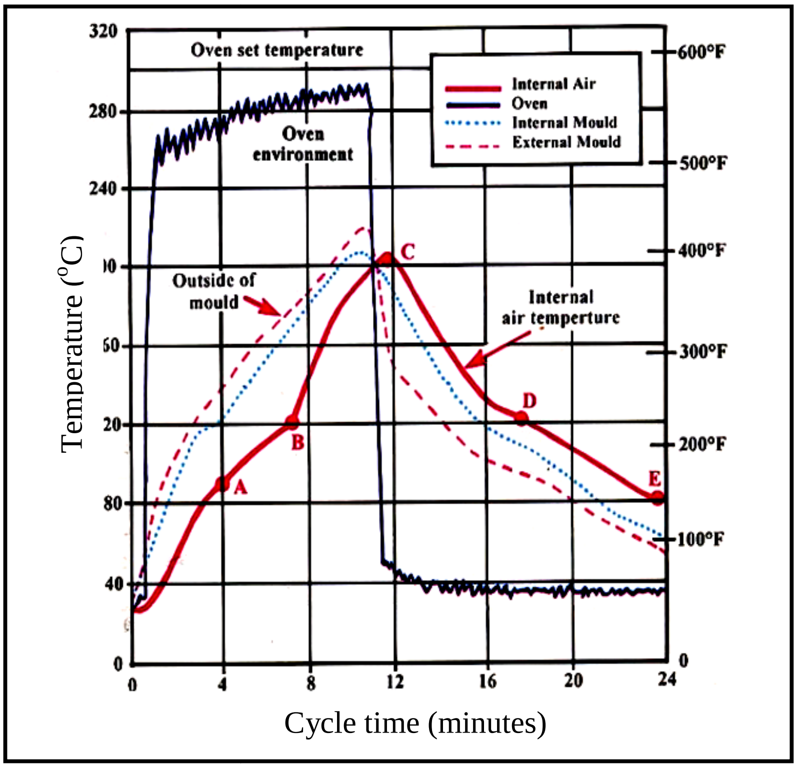
(c) Cooling and Rotation **(d)** Demoulding

Fig 2.1 Line diagrams showing the stages of rotational moulding process

2.2.1 Process Parameters in Rotational Moulding

The quality of a product obtained by rotational moulding process is governed by several process parameters like mould material, rotational speed of mould, oven temperature, cycle time, cooling medium, powder size, pigments, etc. The above mentioned process parameters plays an important role in deciding the superior mechanical properties as observed by Crawford (1996).

In order to control the rotational moulding process, the temperature inside the mould is identified as critical parameter. Crawford and Nugent, 1992[a] determined the peak internal air temperature (PIAT) of LLDPE as 200-220^{o}C which is critical parameter to obtain optimized product quality. In order to understand the temperature variation during different stages of rotational moulding process, a temperature measuring device called ROTOLOG is proposed by Crawford and Nugent, (1992[b]). The variation of mould internal air temperature during the rotational moulding process using LLDPE as a processing material has been depicted by Nugent and Crawford, (1992[b]), Nugent *et al.,* 1992, Kontopoulou, (1997) as shown in Fig 2.2.



AB: Powder Adherence
BC: Sintering
CD: Fusion.
DE: Cooling

Fig 2.2 Variation of internal air temperature in rotational moulding process (Nugent and Crawford, 1992)

Heating is the first stage where heat penetrates in to the mould and internal air temperature starts to increase at a steady rate up to point A. In powder adherence phase (AB), the powder particle (LLDPE) starts melting and it absorbs large amount of heat to form a thin film of melt on inner surface of the mould. In sintering phase (BC), individual particles stick together to the surface of the mould and form a loose porous mass. As the temperature increases, the powder takes the shape of the mould. During this stage pockets of air are still trapped in the melt, forming bubbles. In fusion stage (CD), the density of the melt pool increases and the bubbles that are formed in the previous stage decrease in size. The inner surface of the part becomes smooth. Even though the temperature at the inner surface of the mould is lesser than the temperature near the surface of metal mould, the rotational moulded products begin to degrade at the inner free surface. The presence of oxygen at the inner surface initiates the degradation process. Because of the degradation process, crack initiates at the inner surface and propagates rapidly through the rest of the unaffected structure of the plastic as proposed by Crawford (1994). In Cooling (DE) phase, the plastic gets solidified as the temperature decreases inside the mould. The air temperature inside the mould gets reduced due to the release of thermal energy during cooling. After cooling, the plastic part is allowed to be taken out from the mould as the mould temperature reaches around 70^{o}C.

The rotational moulding process has been extensively reviewed by many researchers. The importance of study of process parameters in descending orders according to Crawford, (1996) are: methods to reduce cycle times, cooling rate, oven temperature, mould materials, recycling, stress analysis and internal mould release agents. It is highlighted that moulders use a rule of thumb to decide the various processing parameters and have to depend heavily upon trial and error methods as well as experience of the operator.

Spence & Crawford (1996[a]) have shown that a low viscosity material **(**MFI 25 g/ 10 min) produces products in which bubbles or surface pores are not observed. Also overheating can reduce the bubbles, but it may tend to have adverse affect on the mechanical strength of the product. These significantly reduced because of thermal degradation as well as overheating resulting in longer cycle times reducing the efficiency of the process**.** As per Crawford (2003) different varieties of polymers that can be used in rotational moulding process are decided by some specific characteristics of the polymers which are in terms of conductivity, paintability, processability etc. This helps in precise control over the process and reduced cycle time. Various internal cooling methodologies have been studied by Tan et al., (2012) to reduce the cycle time. This includes pressurized air, cryogenic liquid nitrogen, chilled water coils and water spray cooling. Out of all stated methods, water spray cooling is chosen as a viable and effective method for internal cooling in rotational moulding.

Polypropylene is rotomoulded under variety of processing conditions. It is observed by Van Hooijdonk *et al.,* (2001) that the impact property of polypropylene is significantly influenced by difference in heating and cooling rate. Also there is very little difference in flexural modulus of samples produced using various processing methods. The highest result is achieved for the larger burner conventional machine (1013MPa), followed by injection moulded sample (941MPa). There is 10% difference between the flexural modulus of the injection moulded samples and samples made of small burner machine. During the studies on importance of vent (in the mould), it is found that, the ineffective venting of moulds could cause part deformation or warpage due to differences in cooling rates and adverse pressure gradients (Jones, 2003). Tan et al., (2011) investigated the effect of cooling rate on the morphology of rotomoulded polyolefin's. His result highlighted that rapid and symmetrical cooling across the mould generally results in smaller spherulite sizes which yields better mechanical properties. In a comparative study of rotational moulding, injection moulding and compression moulding processes, two indices viz. a thermal index and a mechanical index are derived (Godinho *et al.*, 2002). To correlate the processing conditions with the developed microstructure and subsequent mechanical properties, thermo mechanical indices are defined. The low values of thermal indices indicate slower cooling rates and higher crystallization. It is found that the low values of thermal index characterized the rotational moulding (0.5-2) and compression moulding (2-2.5) processes (indicating higher degree of crystallization), while the high values (2-11) are achieved in the injection moulding process (indicating lower degree of crystallization). The mechanical indices denote the maximum shear rate reached in moulding process. Those are zero for the rotational moulding and compression moulding process while it is found to be $7x10^3$ to $9x10^3$ in injection moulding process, signifying that high shear rates are common in that process. The enhancement of tensile properties is seen in injection-moulded parts due to combined effect of crystallinity, spherulite size and orientation induced in the material.

In the rotomoulding process, the temperature must be sufficiently high to achieve the correct viscous and elastic rheological characteristics in the polymer to promote the coalescence of granular particles, and for removal of trapped bubbles. The processibility relationship for rotational moulding process between the initial powder temperature (T_1) and polymer degradation temperature (T_d) are reported by Abbas Tcharkhtchi & Jacques Verdu (2004). Authors compared the processability characteristics of polyethylene with other polymers like polyethylene terephthalate (PET), polyvinyl chloride, polypropylene and polyamides. It is observed that polyethylene poses broader processing window compared to other polymers due to its higher thermal stability. It is also observed that in rotational moulding process, polymer spends longer time in liquid state compared to other process like injection and below moulding.

It has been estimated that one third of the failures of moulded plastic parts arises due to residual stress that develops during the moulding process.

However, the major advantage in rotational moulding process is that the residual stress is relatively less compared to other moulding methods. The best approach proposed by Crawford (2000) to reduce the residual stress in rotational moulding process is faster internal cooling and exercising the control over the release point. Late release from the mould causes lower level of residual stress. Also the use of pressure inside the mould is the best way to control the release point.

Numerical simulations performed by Bellehumeur and Tiang, (2002) described the formation and evaluation of bubbles in rotational moulding process. It is observed from the simulation that initial size of the bubble is controlled by powder particle size and packaging arrangement. It is also noted that rheological and thermal characteristics have little effect on bubble formation process where as processing conditions play a major role.

An experimental study is reported by Liu and Ho, (1999) to know the effect of different processing factors like oven temperature, part thickness, cooling condition, mould material and mould release etc. on warpage of rotational moulded parts. Experiments are carried out and profile meter is used to measure the warpage. From the detailed investigation it is observed that warpage of rotational moulded part increased with increase in oven temperature, part cooling rate, thermal diffusivity of mould material and use of mould release agent. Whereas the wrapage got decreased with thickness of part, diameter of venting pipe and mould pressurization. Also, the warpage of foamed parts is found to be less than that of non-foamed parts.

Among all the process parameters that are discussed above, cooling, heating rate and cycle time are the three parameters which play an important role in deciding the mechanical properties of the rotomoulded product. Slow cooling may result in lower impact strength and increased cycle time. Even improper heating or cooling may lead to thermal stress cracking.

2.2.2 Influence of Process Parameters on the Mechanical Properties of LLDPE Products

The roto moulded product quality is greatly influenced by its process variables which in turn, influence mechanical properties of the rotational moulded products. Depending upon the criticality of the application the desired mechanical properties of the rotational moulded products are impact strength, tensile strength, flexural strength and density. Antonio Greco and Alfonso

Maffezzoli (2004) carried out powder analysis of recycled high-density polyethylene (RHDPE). Different shape factors capable of characterizing powders are obtained at three different temperatures (T<40^{o}C; 60^{o}C<T<70^{o}C; 90^{o}C<T<100^{o}C). It is found that larger and more regularly shaped powder particles are obtained by increasing the milling temperature. At higher temperatures, isotropic configuration of polymer chains is obtained which allows cleaner cuts and smoother surfaces. A further increase in temperature lead to premature melting and adhesion of powders and consequently to formation of clusters that are not desirable for rotational moulding. The influence of shape and size of powder particle is studied by thermo-mechanical analysis. The mathematical model (Antonio Greco & Alfonso Maffezzoli, 2004) followed by experimental investigations confirmed that there exists an optimum sintering temperature at which the density of the sintered product is higher and a good quality rotationally moulded product can be fabricated.

Effect of particle characteristics and operating conditions on particle deposition in rotational moulding process is examined by Olinek et al., (2005). From the experiment it is observed that size segregation of powder particle occurred when particles had a smoother surface and regular shape. The result also showed that powder deposition pattern is largely affected by heating rate and rotational speed. The experimental analysis also yielded a better understanding on flow characteristics of rotational moulding process. Relation between the dry flow time and bulk density of rotomoulding resin powders are experimentally studied by Laws (2004). It is found that dry flow time increases with increasing material temperature.

The density changes of different thermoplastic powders during rotational moulding process are measured by McNally *et al.,* (2002). Thermo mechanical analysis on polyethylene powder revealed an increase in density between the temperature range of 130^{o}C to 150^{o}C. However, reduction in density is observed when the temperature is further increased from 150^{o}C to 200^{o}C. Identical trends are observed for other polymers like polypropylene and polycarbonate.

An experimental study to establish the relationship between the impact performances of rotationally moulded polyethylene over a range of temperatures from -60^{o}C to 20^{o}C is reported by Pick and Eileen Harkin-Jones (2003[a]). Three samples of conventional LLDPE and metallocene catalyzed LLDPE is tested for every 10^{o}C rise in temperatures between -60^{o}C and

20^{o}C. The result showed that the metallocene catalyzed LLDPE has better impact properties than conventional LLDPE. The results also indicated that although high-density polymers shows higher impact resistance, the other factors like crystalline morphology play an important role than density in the impact performance. Further, the work has been extended by Pick & Harkin-Jones (2003[b]) to find a quantitative relationship between the impact performance and thermal transition of rotomoulded linear low-density polyethylene. It is found by Spence and Crawford (1996[b]) that increased number of fine particles produces more bubbles and reduces the impact strength. They have also revealed that viscosity of melt has significant influence on the flow of powder inside the mould which in turn will affect uniformity in thickness. The property of rotational moulded and injection moulded polypropylene parts are compared by Van Hooijdonk *et al.,* (2001). It is found that the impact property of injection moulded sample showed improved crystallinity and impact strength.

In real time application, the impact performance of the plastic is very important. The impact strength of LLDPE is compared by Shih-Jung Liu (2000) for five different process parameters, four levels of oven temperatures, four different oven times, four different mould materials and four different particle sizes of polyethylene powder. The experimental result shows the impact strength in decreasing order: aluminum > brass > mild steel > stainless steel. Experimental design based on Taguchi method are used to prove that the oven temperature and oven time are the principal factors affecting the impact properties of rotationally moulded thermoplastics. According to Guobin *et al.,* (2004) in order to improve the mechanical properties like impact strength of hollow cavity made by the rotomoulded products, the product has to be filled with the polyethylene foam materials. Tan (2010) investigated the effect of cooling rate on impact strength of rotomoulded products. His result confirms that higher cooling rate reduces the level of crystallinity and improves the impact strength. Recent investigation on influence of cooling rate on thickness by Salah Sarrabi *et al.,*(2013) proved that as the cooling rate is decreased in rotational moulding process mould thickness is increased.

Shih Jung Liu and Kang Ming Peng (2010) assessed the rotomouldability of polycarbonate reinforced polyethylene composites and found that higher cooling rates yielded higher impact strength with lower tensile strength of moulded composites. Spence and Crawford , 1996 introduced internal pressure inside the mould to reduce bubbles and pores in rotational moulding process which in turn increased the impact strength up to 25 % and tensile strength up to 5 %.

It has been years that the rotational moulding industries have recognized longer cycle time creating a significant obstacle for the development of the process. Conventionally, cycle time has been reduced by using polyethylene of reduced molecular weight or melt viscosity. However, those can result in inferior impact strength (Bharat *et al.*, 2001). Sufficient progress has been shown in reducing cycle time by other methods also. Changing the thermal characteristics of the shell material (Yan *et al.*, 2003), introducing internal pressure (Crawford *et al.*, 2004), and employing internal cooling (Khouri, 2004) to mention a few.

2.2.3 Analytical Work to Simulate the Process

Considering the nature of complex heat transfer phenomena in this process, several researchers have simulated the process analytically and supported their results by experimental investigations. Some researchers adopted mathematical models and simulations to understand the characteristics of the rotational moulding process. Simulation of thermal phenomena in rotational moulding is very important to follow the evolution of the temperature in various zones of this process (Said Lotfi et al., 2012). Simulation allows for systematic and quantitative studies on the effect of moulding condition and material property on the moulding cycle. The effect of crystallization kinetics on warpage of polypropylene has been studied by Glomsaker *et al.*, (2009). A multimode crystallization kinetic model has been proposed by the author to relate the effect of crystallization on warpage of rotomoulded products. On the basis of his empirical model, they found that crystallization temperature, crystallization halftime and heat of fusion are most significant parameters influencing warpage. Recently, computer simulation based prediction of internal air temperature and degree of curing in multilayer rotational moulded parts is investigated by Alongkom (2009) using ROTOSIM simulation software.

Modified heat transfer model for the rotational moulding process has been proposed by Baneerji et al., (2008). The model is based on heat transfer to powder at the mould powder interface is through convection and the powder particles get heated up by conduction. The author has successfully modeled layer-by-layer non isothermal polymer melting and crystallization using a source-based method

Time required for complete powder deposition in rotational moulding process has been depicted in terms of theoretical and lumped numerical model by Gogos *et al.,* (1998). They developed a numerical model for three phases of rotational moulding process, namely mould heating with biaxial rotation, powder deposition on the walls of the mould and cooling of the mould. It is concluded from the numerical study that plastic melting temperature, energy required for phase change and plastic conductance affects the powder end time in rotational moulding process.

The simulation of fusion and crystallization stage of polyamide 11 in rotational moulding process using ozawa model coupled with enthalphy method is proposed by Said Lotfi *et al.*, (2012). The proposed model is to used the heat release during crystallization process.

2.2.4 Additives and Foams in Rotational Moulding Process

Yan *et al.,* (2003) investigated the effect of reinforcing the glass beads with medium density polyethylene in rotational moulding process. From their experimental observations it is noted that addition of glass beads in medium density polyethylene, increases the impact strength with reduction in tensile strength and total cycle time of the process. It is reported by Mark Kearns & Neil Collan (2000) that the use of two-layer moulding gives significant improvements in flexural modulus and considerable reduction in shrinkage of mouldings, however no benefit has been gained in terms of tensile strength and impact properties of composite mouldings. According to Bharat Indu Chaudhary *et al.,* (2001), addition of low molecular additives like mineral oil, glycerol monostearate added with polyethylene as sintering enhancers results in decreased melt viscosity and elasticity at low shear rate. Due to the addition of sintering enhancer, part thickness obtained is uniform and the cycle time is reduced without adversely affecting the impact strength on uniaxial and biaxial rotational moulding. An investigation of the effect of Talc and Mica on the properties of rotational moulded LLDPE inferred that the introduction of finer grades of Talc and Mica improved the impact strength. It is also found by Robert *et al.,* (2000) that when moulding LLDPE+10% MICA-MU85 + 3% of maleic anhydride modified high-density polyethylene PB3009, a reduction of nearly 15% of overall cycle time is achieved and for the same composition the shrinkage is reduced up to 56%.

Nano scale Calcium Carbonates are used to improve the impact strength of rotationally moulded nano composites with polypropylene as a matrix material. It is found by Alongkorn Kanokboriboon and Harkin-Jones (2005) that Calcium Carbonate could be a effective impact modifier for rotomoulded polypropylene with small decrease in strength and strain to break. Many polymers used in rotomoulded parts (such as outdoor storage tanks) contain photostabiliser additives. However, it is observed by Mark Kearns *et al.,* (2001) that those resulted in marginal decrease of impact strength. A design and cost analysis of mould made of FRP filled with copper particles [FRP/copper] has been suggested to reduce the cycle time in rotational moulding (Seibi and Sawaqed, 2002). Using that mould they have claimed 64% reduction in manufacturing time.

One drawback of rotomoulded products is its low insulative, and shock mitigation properties due to the hollow structure. In order to improve the mechanical properties, the hollow cavity of rotomoulded products can be filled or mixed with foam. Rotational moulding of foamed polyethylene has increasingly become an important process in industry. It has been used to produce parts in various applications such as furniture, toys and novelties, and flotation and drink containers (Klempener, 1991). Foamed structures provide several advantages in thermoplastic products, including: a lightweight, excellent strength–weight ratio, superior insulation abilities; and energy absorbing performance (shock, vibration, and sound).

Although the rotational moulding technique has been developed for more than three decades, the research efforts in rotational moulding of foamed parts are still limited. Guobin *et al.,* (2004) has studied the mechanisms of foaming using LLDPE foams in rotational moulding. A similar study has been conducted on the foaming mechanism of polyethylene blown by chemical blowing agent under ambient pressure (Remon *et al.,* 2008). Archer *et al.,* (2002) has investigated the foaming behaviour and flexural property of rotomoulded foamed metallocene polyethylene. He observed that metallocene based polyethylene produces foam with a lower density than conventional polyethylene and metallocene based polyethylene foam exhibits lower flexural property than conventional polyethylene. The rotomouldability of LLDPE foams is examined by Shih-Jung Liu & Chja-Hsun Tsai (1999) adding citric acid based blowing agents. The presence of blowing agent increased the impact strength of the product whereas the tensile strength was found to be decreasing with increase in the blowing agent content. The warpage of the part was also found less with the increase in foaming agent. Experimental investigation performed by Maryam et al., (2013) to know the influence of processing time and temperature on foam structure revealed that these parameters have a significant effect on the foam cellular structure. Processing time and temperature are found to be effective tools for controlling the cellular structure and physical properties of developed foam structure.

2.3 Fracture Behaviour of Polyethylene

Generally, polyethylene is a tough, semi-crystalline thermoplastic that has found increasing use in several key engineering applications, such as a pipeline material for water and gas transportation, fuel tanks, large solvent drums. However, applications demanding resistance to crack is found to be utmost importance, e.g. light weight body armor and riot shields for military and law enforcement personnel. Since the components fabricated from polyethylene are more often prone to failure under impact loading and this tendency of failure is promoted by the presence of sharp flaws, especially at room temperature (Carla *et al.,* 2007). Therefore, fracture behaviour of a polymer is one of the important characteristics required to be evaluated. Fracture of polymer materials can be brittle or ductile or mixture of two. Thermoplastics may fracture primarily by brittle or ductile manner. The fracture mode is considered to be brittle if the fracture of the thermoplastic takes place below its glass transition temperature else it was considered as ductile fracture, whereas the thermosetting plastics are considered to fracture primarily in brittle mode. Fracture toughness generally depends on temperature, environment, loading rate, the composition of the material and its microstructure, together with geometric effects (Argon, 2013, Crawford, 2006, Anderson, 2005).

Essential work of fracture under tensile and impact loading is used to evaluate the fracture toughness of high density polyethylene-organ clay with montmorillonite nano-composites with / without elastomers. It is found by Tjong & Bao (2007) that the model cannot be used to describe the fracture behaviour of pure HDPE and its nano-composites, as necking and cross yielding of the composites takes place at 70^{o}C. The dynamic fracture behaviour of linear medium density polyethylene under impact loading condition are assessed by three point bend impact experiment with modified split Hopkinson pressure bar (MHPB). It is found by Carla *et al.,* (2007) that the time duration of the stable crack blunting and growth for 6 m/s of impact velocity is much shorter than those obtained for lower impact velocities (i.e., 5.4 m/s and

2.7 m/s). It is also observed that as impact velocity is increased the time required for dynamic fracture initiation, length of stable crack growth and dynamic energy release rate gets decreased. According to Francis *et al.,* (1988) morphological analysis of the extraordinary fracture toughness of LLDPE resins compared to low density polyethylene and high density polyethylene resins. It is proved that the presence of second soft phase with a weak solvent in a hard semi crystalline matrix resulted in extraordinary fracture toughness in LLDPE. Essential work of fracture concept is used by Tamas Barany *et al.,* (2003) to find the plane stress fracture toughness of amorphous co-polyester sheets of different composition and molecular mass. It is observed that amorphous co-polyesters are ideal polymer for essential work of fracture tests, since they undergo full yielding prior to the onset of crack growth. Essential work of fracture approach is used to assess the effect of strain rate on the plane stress fracture toughness of various ductile polymeric films (LLDPE-CO-BUT, PA6, and PET). It is found by Alesseandro Pegoretti *et al.,* (2005) that specific essential work of fracture component related to crack initiation is increasing with strain rate and whereas the crack propagation is decreasing with strain rate. For the prediction of crack width and crack spacing in fiber reinforced polymer (FRP) concrete beams an analytical procedure based on the slip and bond stresses is adopted. It is predicted by Aiello & Ombres (2000) that increase in bond strength of FRP bars reduced the width of crack. Increase in reinforced ratio and cover thickness decreases the crack width.

Past literature greatly emphasizes on the fracture behaviour of different polymeric materials like HDPE, LLDPE etc and their composites. Concentration of research work is towards knowing the material characteristics rather than product characteristics. Since plastic products are used in critical applications, it is important to know the product characteristics also.

2.4 Assessment of Literature

Plastic parts can be manufactured by employing a wide variety of manufacturing process such as blow moulding, injection moulding, rotational moulding, transfer moulding, and thermoforming. Each plastic manufacturing process has some distinct merits and demerits. The aspect of appropriate process selection for plastic manufacturing is not emphasized adequately in the literature. In other material selection and/or design problems, literature survey reveals methodologies like fuzzy MADM, TOPSIS are used for selecting right process for a particular product application. As the selection decisions are complex, there is a need for simple, systematic, and logical methods or mathematical tools to guide decision makers in considering a number of selection attributes and their interrelations in making right decisions. As observed from the literature, MADM methodology is being widely applied for the above said situations. Since the problem of process selection for plastic manufacturing is a serious concern for the moulders considering the availability of number of processes with different advantages, a need exists to address this problem with suitable and available mathematical models like MADM.

A critical review of the literature on rotational moulding process highlights that many researchers have concentrated on reduction in cycle time of the process and there are very limited studies performed in knowing the effect of oven residence time on the mechanical properties. Also, there are limited simulation studies (mainly due to apparent simplicity but complex nature of heat transfer during the process), that take into account actual process conditions and their effect on mechanical properties of the product in their prediction. As a consequence, many times the predictions differ from the shop floor situations. The review on rotational moulding process also shows that the optimization of oven residence time with respect to the mechanical properties has not been emphasized, which greatly helps the moulders to the decide the appropriate oven residence time, yielding better mechanical properties like tensile, impact and flexural strength.

Although rotational moulding technique has been developed for more than three decades, the research efforts in rotational moulding of foamed parts are still limited. From the literature on rotational moulding, though lot of research centers on knowing the product and process characteristics, to the best of our knowledge, statistical methods like DOE have not been sufficiently discussed which can improve the quality of the product. The above mentioned method can also assist the experimenter in reducing the variability in the process and product. DOE has been successfully implemented in various other fields especially in manufacturing sectors for process optimization which yields better quality product. For rotational moulding process, no such attempt of application of DOE to know the effect of process parameters on the impact strength of foamed polyethylene has been carried out.

Even though different methods are adopted in rotational moulding process, optimized parameters which in turn give optimized product quality are not clearly defined. Rotational moulding products are used in some critical applications like oil tanks, chemical tanks and industrial equipments etc. In view of criticality of the application of the rotational moulding products, it is necessary to know the fracture behaviour of the rotational moulding products which is not clearly investigated yet. As it is evident from the literature that the process variables play an important role in producing the effective and reliable products in rotational moulding, however, no attempt is made to address the correlation among the process parameters in rotational moulding process with mechanical and fracture behaviour.

CHAPTER 3

METHODOLOGY

Introduction

This chapter discusses about the process selection, simulation and experimental procedure adopted to characterize mechanical and fracture behaviour of rotomoulded products made using LLDPE. Subsequent topics provides complete details of selecting a suitable manufacturing process for specific application, equipments, material and procedures used to predict the optimum process parameters yielding maximum mechanical strength and fracture toughness of rotomoulded products. Methodology is shown in the form of block diagram in Fig 3.1 for better understanding at a glance.

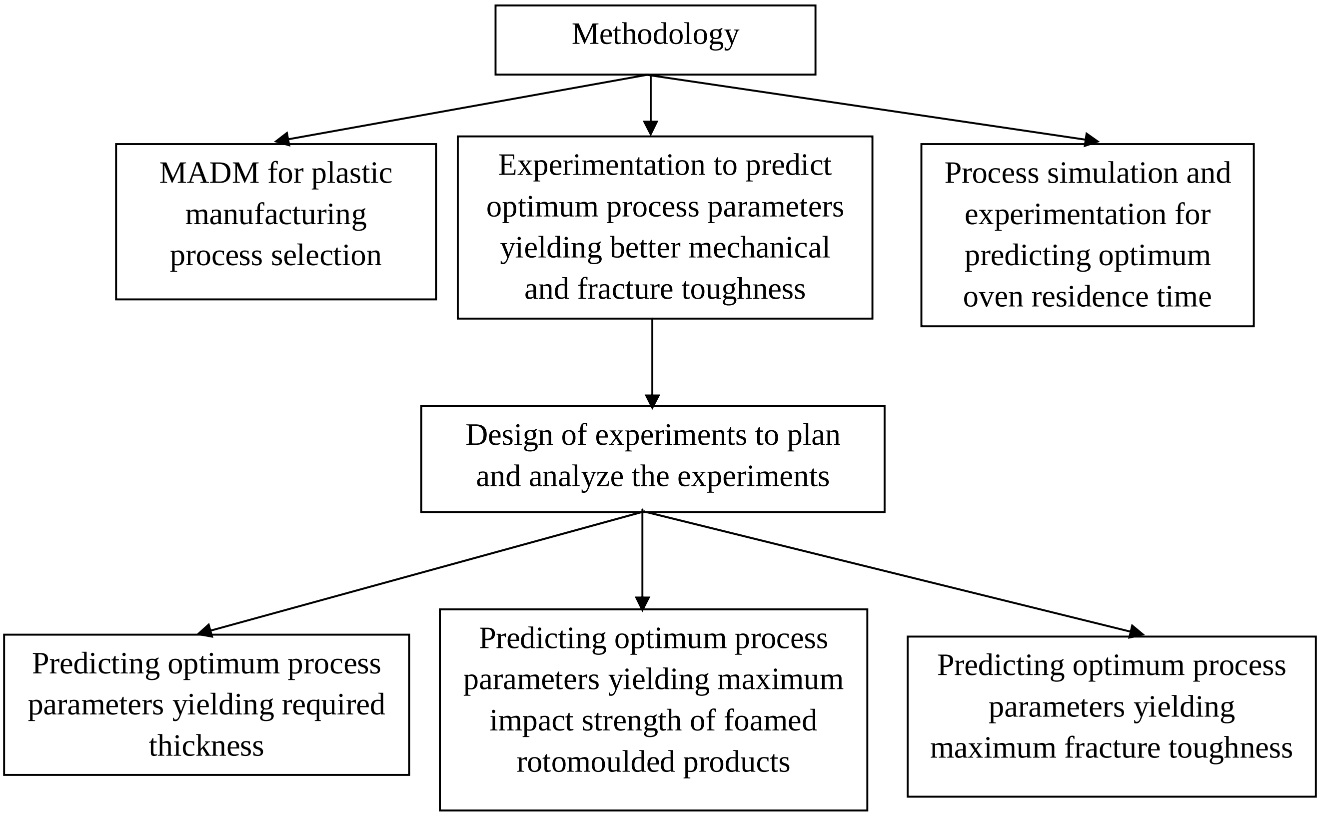


Fig 3.1 Block diagram showing the methodology

3.1 Manufacturing Process Selection

Manufacturing technology plays a vital role for the development of a country's industrial growth and largely dictates the trend of the economy. Decision making in the manufacturing environment is a strategic topic, especially in connection with the complexity of driving forces and factors influencing manufacturing systems dynamics.

Fast-changing technologies on the product front cautioned the need for an equally fast response from the manufacturing industries. The old traditional model of 'unfocused, short-term views and non-holistic vision' is being replaced by the enlightened approach of 'focused, holistic and strategic vision'. To meet the challenges, manufacturing industries have to select appropriate manufacturing strategies, product designs, manufacturing processes, work piece and tool materials, machinery and equipment, etc. The selection decisions are complex as decision making is more challenging today (Venkata Rao, 2007). Necessary conditions for achieving efficient decision making consist in understanding the factors influencing the whole manufacturing environment including the issues related to manufacturing systems design, planning, and management. Out of all the decisions, one of the crucial decisions that is made during the design stage is process selection.

Coming section presents objective based multi attribute decision making methodology for plastic manufacturing process selection which is a simple, systematic, and logical method considering number of selection attributes and their interrelations. Multiple attribute decision making is employed to solve problems involving selection from among a finite number of alternatives. In this work, the method is adapted to rank different plastic manufacturing processes namely blow moulding (BM), thermoforming (TF), rotational moulding (RM), injection moulding (IM), and contact moulding (CM) to quantitatively assist a designer to select a suitable process from a long list for a specific application. The methodology consists of four simple steps (Rao and Patel, 2010, Rao *et al.*, 2011).

The four steps of objective based MADM methodology are described below: (Example for the same are discussed in Chapter- 4)

3.1.1 Decision Table

Identify the pertinent attributes and different alternatives for the process selection problem. The attributes are of two types, beneficial (i.e. higher values are desired) and non-beneficial (i.e. lower values are desired). A quantitative or qualitative value can be assigned to each identified attribute as a limiting value for its acceptance for the problem under consideration. After listing the alternatives and the values associated with the attributes (*Yij*), a decision table, similar to that given in Table 3.1 including the values of all attributes for the listed alternative process, can be prepared.

Table 3.1 Decision table with values associated with attributes

Process Alternative	Attributes					
	B_1	B_2	B_3			B_m
A_1	Y_{11}	Y_{12}	Y_{13}	-	-	Y_{1M}
A_2	Y_{21}	Y_{22}	Y_{23}	-	-	Y_{2M}
A_3	Y_{31}	Y_{32}	Y_{33}	-	-	Y_{3M}
-	-	-	-	-	-	-
-	-	-	-	-	-	-
A_n	Y_{n1}	Y_{n2}	Y_{n3}	-	-	Y_{nM}

To convert the elements of decision matrix into scale and unit independent quantity, it is necessary to normalize their values. As the values associated with the attributes (Y_{ij}) may be in different units (e.g., part thickness is expressed in mm & product cost is expressed in rupees). Thus, normalized decision making matrix R, is obtained from the decision matrix and each element of this matrix r_{ij} is expressed as

$$r_{ij} = Y_{ij} / \sum_{i=1}^{n} Y_{ij} \tag{3.1}$$

Where r_{ij} is the normalized value of Y_{ij} and $\sum_{i=1}^{n} Y_{ij}$ is the total of the value of *jth* attribute for n number of alternatives.

Eq. (3.1) can deal with quantitative attributes; In case of qualitative attributes there should be some method to be followed to convert the qualitative value to the quantitative value.

In this work, ranked value judgment on a fuzzy conversion scale using fuzzy set theory approach, is used to convert qualitative value to quantitative value. This approach is based on the work of (Chen and Hwang, 1992). This numerical approximation system converts linguistic terms to their corresponding fuzzy numbers. An 11 point scale is proposed for better understanding. Table 3.2 represents the selection attribute on a qualitative scale using fuzzy logic, corresponding to the fuzzy conversion scale.

Table 3.2 Conversion of linguistic terms into fuzzy scores (Chen and Hwang, 1992)

Qualitative measures of selection attribute	Fuzzy number	Assigned crisp score
Exceptionally low (EXL)	M1	0.0455
Extremely low (EL)	M2	0.1364
Very low (VL)	M3	0.2273
Low (L)	M4	0.3182
Below average (BA)	M5	0.4091
Average (A)	M6	0.5
Above average (AA)	M7	0.5909
High (H)	M8	0.6818
Very high (VH)	M9	0.7727
Extremely high (EH)	M10	0.8636
Exceptionally high (EXH)	M11	0.9545

3.1.2 Objective Weights of Importance of the Attributes

The objective weights of importance of the attributes are found out using statistical variance (V_j). Eq. (3.2) shows determining the weights of importance of the attributes

$$V_j = \left(\frac{1}{n}\right) \sum_{i=1}^{n} (r_{ij} - r_{ijmean})^2 \quad (3.2)$$

where V_j is the statistical variance of the data corresponding to the j^{th} attribute and $(r_{ij})_{mean}$ is the is the mean value of r_{ij}. Statistical variance is a measure of the dispersion of a set of data points around their mean value. The objective weight of the j^{th} attribute Q_j^o can be computed by dividing the statistical variance of j^{th} attribute with the total value of the statistical variances of 'm' number of attributes. Following equation is used for calculating the objective weights of importance

$$Q_j^o = {V_j}\Big/{\sum_{i=1}^{m} V_j} \tag{3.3}$$

3.1.3 Determination of Preference Index

Each alternative is assessed with regard to its weights associated to every attribute. The overall performance score of an alternative is the weighted sum called preference index. Preference index value gives the rank of the particular alternative with respect to other alternatives. Preference index P_i^o can be calculated by summing the values of X_i^o for different alternatives. X_i^o is the product of objective weight of importance (Q_j^o) and r_{ij}^{**} for different attributes. Eqs. (3.4) and (3.5) are used to calculate the preference index

$$X_i^o = Q_j^o \mathrm{r_{ij}}^{**} \tag{3.4}$$

$$P_i^o = \sum_{j=1}^{m} X_i^o \tag{3.5}$$

where $r_{ij}^{**} = \left[r_{ij}^b = \left(r_{ij}^b\right)max\right]$ for beneficial attributes and $\left[\left(r_{ij}^{nb}\right)min = \left(r_{ij}^{nb}\right)\right]$ for non beneficial attributes. Where, r_{ij}^b and r_{ij}^{nb} indicates the normalized value of beneficial and non-beneficial attributes respectively. $\left(r_{ij}^b\right)max$ indicates the maximum value of j^{th} beneficial attribute and $\left(r_{ij}^{nb}\right)min$ indicates the minimum value of j^{th} non beneficial attributes.

All the alternatives can be arranged in a descending order of P_i^o to obtain the preference order of alternatives. It is clear that alternative with highest value of P_i^o is the best choice for the considered decision making problem.

3.1.4 Final Decision Making

Selection of plastic manufacturing process suitable to a particular application can be done on the basis of preference order prepared in the earlier section in the presence of other attributes and business manufacturing strategies. A final decision may be taken keeping in view of all practical considerations such as financial constraint, equipment constraint, material availability constraints, and labour constraints etc. Proposed methodology is user and computational friendly in optimal selection of a manufacturing process for plastics. The only inputs required for this procedure are decision table.

3.2 Simulation Using ROTOSIM

Rotational moulding involves heating and melting of powder particles to form homogenous polymer melt as well as cooling and solidification. The densification of loose powder into melt occurs over a wide range of conditions as the material passes from solid state into molten state. Moreover, the phase change occurs over a range of temperature. Further, due to bi-axial nature of mould rotation, there is a dearth of accurate thermal data measurement techniques. This necessitates the use of simulation which allows for systematic and quantitative prediction of critical timings of the process like switching off the oven, de-moulding time, etc. To the best of our knowledge, ROTOSIM software takes into account all the possibilities that exist in a typical rotational moulding process. Hence, the simulation studies are conducted using ROTOSIM.

ROTOSIM is a computer program for simulating the processes that occur in a rotational moulding cycle for polymers. It is based on a complex mathematical model of the major physical processes in the cycle. It enables the user to experiment with a variety of different operating conditions and observe the effects that these have on the resulting cycle conditions and the moulded product. During the simulation run, building up of a melt layer on the mould surface and development of solidified polymer layer can be observed. Temperature and phase change with respect to time are also generated by the simulation. The different aspects of the simulation are given below.

3.2.1 Solid Model of the Mould and Meshing

For this study, we have chosen a hollow product used as a plant vase which can be easily produced on a lab scale rotational moulding machine. An aluminum (Al) mould of 3 mm thickness has been considered, the shape of which resembles an inverted frustum of a cone. In order to have sufficient stiffness of the product, a minimal wall thickness of around 2 mm is designed. The part was initially modeled using Pro-E Wildfire software and corresponding .stl file is obtained. This file is exported to ROTOSIM software and a mould file (.mld) is created for further meshing and analysis. The outer surface of the mould is meshed using triangular elements with 3041 nodes whereas the inner surface of the mould is separated from the outside surface by the polymer layer thickness and is created during the moulding cycle. Fig 3.2 shows the detail configuration of the mould along with the sectional view, while the meshed model is shown in Fig 3.3.

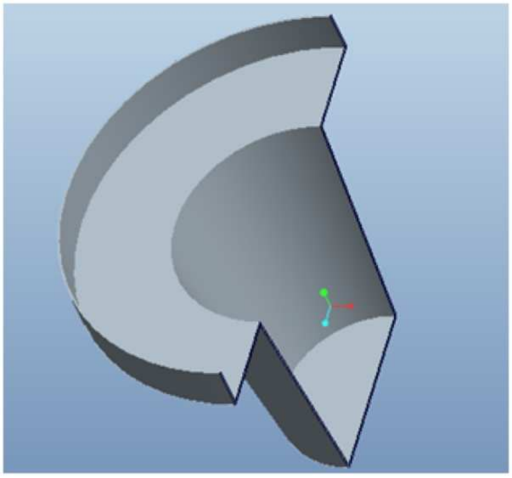


Fig 3.2 Sectional view of the mould

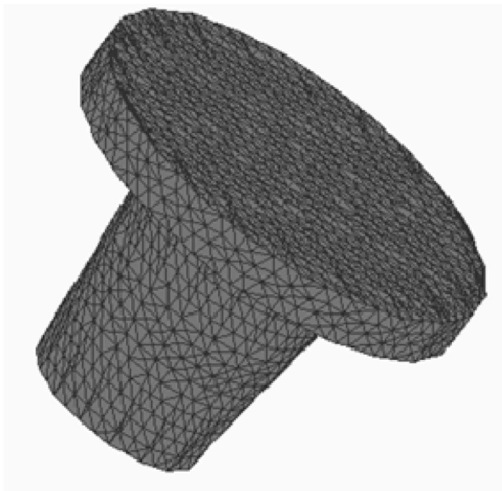


Fig 3.3 View of the meshed mould

3.2.2 Material Properties and Process Conditions

In this study, LLDPE has been used for the purpose of simulation as well as experimental work. The mould and material properties are enlisted in Table 3.3. The other process conditions used in the simulation and experimentation are summarized in Table 3.4.

Table 3.3 Moulding conditions

Moulding condition	Mould	LLDPE	Air
Thermal conductivity (W/mK)	204	0.25	0.025
Specific heat of (J/kgK)	896	2600	1006
Density (kg/m^3)	2707	749.6 (melt)	1.205
Internal convective heat transfer	-	-	5
External air convective heat (W/m^{2}K)	-	-	20

Table 3.4 Process Conditions used in the simulation and experimentation

S.No.	Process parameter	Value/ Condition
1	Temperature at the start of cycle	30^{o}C
2	Room temperature	30^{o}C
3	Initial internal air and powder temperature	30^{o}C
4	De-moulding internal air temperature	50^{o}C
5	Mould external cooling condition	Still air

3.3 Experimental Details

3.3.1 Materials

For this work, LLDPE of grade R350 A 42, which has a melt flow index of 4.2 g/10 min and density of 935 kg/m^3 manufactured by Gas Authority India Limted (GAIL) India limited is used. The average particle size of the powder is 500 microns (around 35 mesh number). This grade is normally recommended for manufacturing of water storage tanks, automobile parts, boats, etc.

3.3.2 Melt Flow Index

Melt flow index of the thermoplastic material has been checked with dynisco melt flow indexer as per ASTM D 1238 (2004). Melt flow index is the output rate (flow) in grams that occurs in 10 minutes through a standard die of 2.0955 ± 0.0051 mm diameter and 8.000 ± 0.025mm in length when a fixed pressure is applied to the melt via a piston with a load of total mass of 2.16 kg at a temperature of 190°C. Melt flow index is an assessment of average molecular mass and is an inverse measure of the melt viscosity; in other words, higher the MFI, more the polymer flow under test conditions. Knowing the MFI of a polymer is vital for anticipating and controlling its processing. Dynisco melt flow indexer is shown in Fig 3.4.Information of the model is provided in Appendix D.

Fig 3.4 Dynisco melt flow indexer

3.3.3 Machine and Mould

A lab scale electrically heated bi-axial rotational moulding machine (refer Appendix D for model information) is used as shown in Fig 3.5 (a) and 3.5 (b). An Aluminum hollow mould having the shape of an inverted frustum of cone as shown in Fig 3.5 (c) and stainless steel hollow mould having the square cross-section with polished internal surface as shown in Fig 3.5 (d) is used for getting the desired product.

(a) Rotational moulding machine

(b) Major and minor axis rotation

(c) Aluminum (Al) hollow mould

(d) Stainless steel hallow mould

Fig 3.5 Machine and Mould

Based on the volume of the mould, powder shot weight is used in order to produce moulding with a required wall thickness. The internal mould surface of the aluminum mould and stainless steel mould is coated with a silicone oil based mould release agent manually. During these experiments, biaxial mould rotation is used with the arm (major axis) to plate (minor axis) speed ratio maintained at 4:1 to get consistent wall thickness. Table 3.5 gives details of the experimental set-up.

Table 3.5 Experimental setup used for the work

Rotational moulding machine specifications	Model: Clamshell type single arm biaxial machine
	Method of heating: electrical
	Control voltage: 220V
	Power rating of oven: 8 kW
	Power rating of blower: 0.37
	Speed ratio: (major axis:minor axis) 4:1
	Drives: 0.5 HP Variable frequency drive
	Temperature range: 30 – 250^{o}C
	Maximum mould dimension: 300 mm X 300 mm X 300 mm
Mould 1 specifications	Material : aluminum(Al)
	Shape: inverted frustum of a cone
	Release agent: Metrork silicone 17 compound Dimensions: Top diameter: 300 mm Bottom diameter: 150 mm Height: 220 mm
Mould 2 specifications	Material : Stainless steel
	Shape: Square cross section
	Release agent: Metrork silicone 17 compound Dimensions: Length: 100 mm Breath: 100 mm Height: 100 mm
Raw material specifications	Material: R350 A 42 LLDPE supplied by GAIL India
	MFI: 4.2 g/10 min
	Density: 935 kg/m^3
	Shot weight: 0.6 kg
Testing Equipment	Tensile and flexural testing:

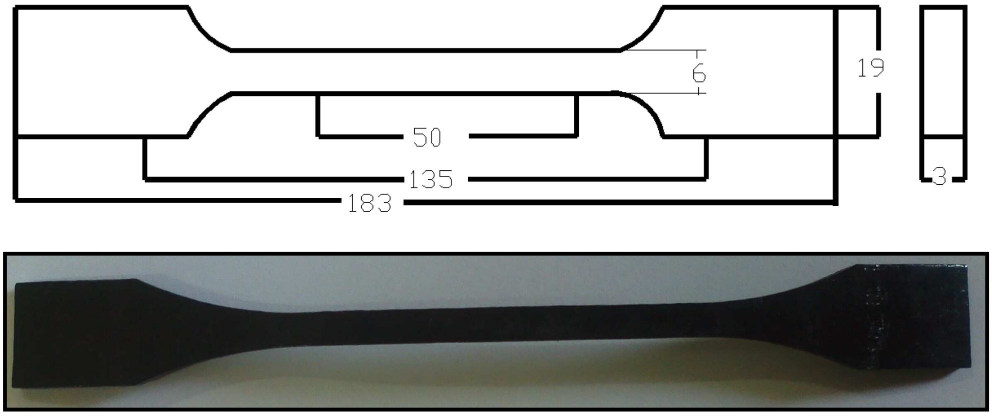
	Machine: universal testing machine cross head speed of 5 mm/min
	Impact testing Izod impact tester with 10 Joule pendulum.

3.3.4 Experimental Procedure

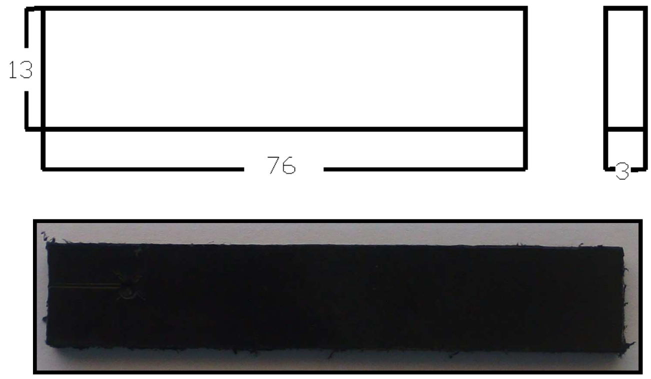
The internal mould surface of the mild steel mould / aluminium mould is coated with a silicon oil based mould release agent. The different process variables are set as per the requirement of the experiment. Preweighted quantity of the LLDPE is then placed inside the mould. The mould is then bolted into the oven where it underwent a biaxial rotation which makes the powder to spread in the internal surface of the mould. After certain temperature the thermoplastic powder melts and sticks to the wall of the mould. The process is continued till the required thickness of the plastic part is obtained. After getting the required thickness, mould is cooled and demoulded. Specimens are prepared out of the rotomoulded products to carry out mechanical and fracture characterization.

3.3.5 Testing of Mechanical Properties

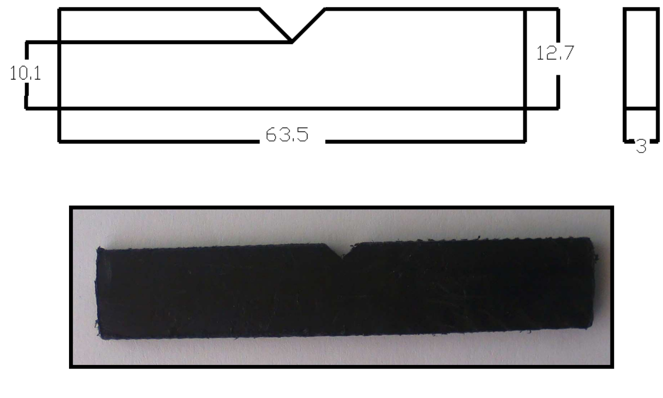
Rotationally moulded products are obtained from rotational moulding process and test specimens are prepared according to ASTM D 638 (2010) for tensile, ASTM D 790 (2010) for flexural & ASTM D 256 (2010) for impact testing as shown in Fig 3.6 (a, b and c) respectively. Bench universal testing machine as shown in Fig 3.7 (refer Appendix D for model information) is used for tensile and flexural testing. For tensile testing, Load cell of 5 kN is used along with a cross head speed of 5 mm/min. For the flexural testing, the load cell of 0.5 kN is used and flexural strength at yielding is obtained. Impact tests are performed in typical Izod impact testing machine as shown in Fig 3.8. A pendulum of maximum energy capacity of 10 Joules is used to evaluate the energy absorption ability of the material. To account for process variability during experimentation, three replications are taken for each experimental run. Thus three samples of each tensile, flexural and impact specimens are used for testing.



(a) Tensile Specimen



(b) Flexural Specimen



(c) Impact Specimen

All units are in mm

Fig 3.6 (a) Tensile, **(b)** Flexural and **(c)** Impact specimens as per ASTM standards

Fig 3.7 Universal testing machine

Fig 3.8 Izod Impact Testing Machine

3.3.6 Testing of Fracture Properties

The resin used in this study is linear low density polyethylene of grade R350 A 42 having melt flow index of 4.2 g/10 min and density is 935 kg/m^3 supplied by GAIL India limited. Rotationally moulded products are produced on a lab scale electrically heated biaxial rotational moulding machine using a stainless steel hollow mould having a square cross-section with polished internal surface. Powder weight of 160 g is used to produce rotational moulding product. The compact tension specimen is prepared from the moulded product as per the ASTM standard D6068 (2010) as shown in Fig 3.9. The pre crack is introduced by carefully cutting the specimen perpendicular to the samples edge (1.2 *W*) using a sharp steel blade of thickness 0.1 mm.

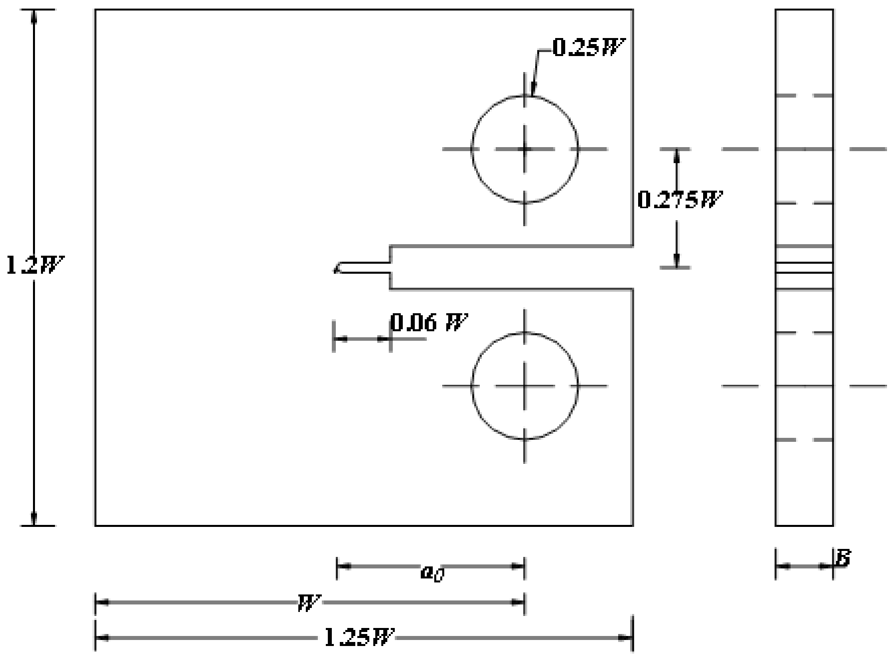


Fig 3.9 Compact tension test specimen

The tests are performed on a universal testing machine at a constant cross head speed of 1 mm/min at room temperature and a load cell of 5 kN is used for testing. Load versus load line displacement are recorded for 15 specimens.

Anti–buckling plates are fabricated to avoid out of plane buckling. A small window of 30 mm X 15 mm is prepared in the plates to observe region ahead the crack tip. Anti–buckling plates as shown on Fig 3.10 are prepared on shaper machine. Specimen with anti-buckling plates is also shown in Fig 3.11.

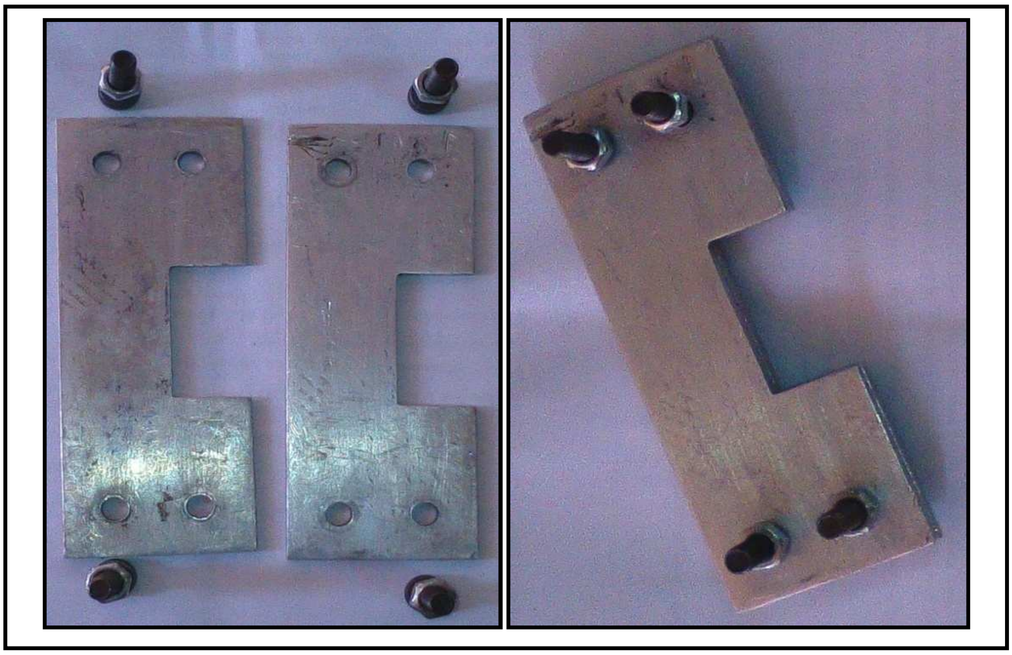


Fig 3.10 Anti–buckling plates

Fig 3.11 Specimen with anti-buckling plates

3.4 Design of Experiments

Experimental design is an important tool in the engineering community for improving the performance of manufacturing process. Engineers no longer can afford to experiment in a trial-and-error manner, changing one factor at a time. A far more effective method is to apply a computer-enhanced systematic approach to experimentation that considers all factors simultaneously. The approach is called design of experiments (DOE). Design of experiments (DOE) is a systematic, rigorous approach to engineering problem-solving that applies principles and techniques at the data collection stage so as to ensure the generation of valid, defensible, and supportable engineering conclusions. In addition, all of this is carried out under the constraints of minimal expenditure of engineering runs, time, and money.

In this study, two approaches of design of experiment are used to plan and analyze the experiments that are 2^2 factorial design and Box-Behenken design (Box and Behnken, 1960) of response surface method (RSM).

3.4.1 2^2 Factorial Design

In 2^2 factorial design, there are two factors (A & B) each at two levels. Each factor will have two levels, a "high" and "low" level. Table 3.6 shows the 2^2 factorial design in a standard order matrix.

Table 3.6 2^2 Factorial design in a standard order matrix.

Factors		Treatment combinations
A	B	
+	-	A high B low
-	-	A low B low
+	+	A high B high
-	+	A low B high

Fig 3.12 shows the treatment combination in the 2^2 design. 2^2 factorial design is used to know the effect of two factors on the response. 2^2 design considers all possible combination of the two factors considered. Therefore, 2^2 design with replications yields better results.

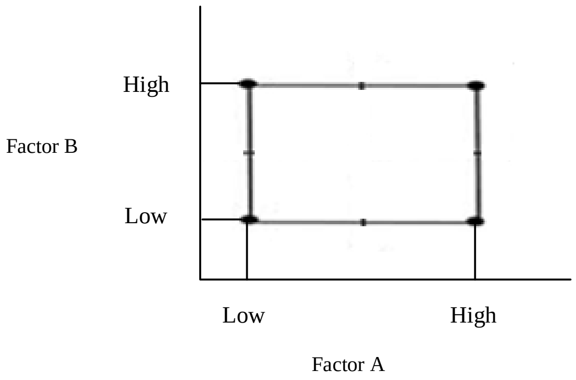


Fig 3.12 Treatment combination in the 2^2 design

3.4.2 Response Surface Method

Response surface method (RSM) is a collection of statistical and mathematical methods that are useful for modeling engineering problems. The main objective of this technique is to optimize the response surface that is influenced by various process parameters. RSM quantifies the relation between the controllable parameters and the response (Phaneendra Kiran and Shibu, 2013 and Montgomery, 2012). In this work, three parameters with three levels are considered, a full factorial scheme would have resulted in 27(3^3) experiments. Since performing experiments based on full factorial design requires substantial amount of resources and time, the experiments are planed using Box- Behenken design which is subcategory of RSM (Box- Behenken, 1960). In this design the coded variables (-1, +1) are used to develop a first order model where as the center point (0, 0) estimate the second order model with curvature effect. The considered design scheme has only 15 experimental trails, yet provides a good assessment of the response. The design is geometrically shown in Fig 3.13.

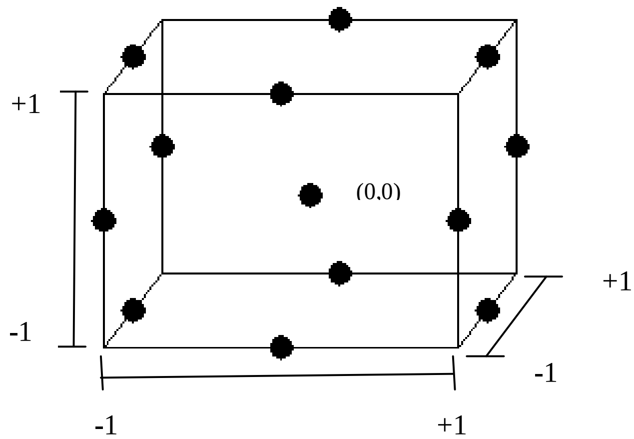


Fig 3.13 Box- Behnken design for three factors

The above stated methodologies will be used in proceeding Chapters to plan, conduct, analyze and optimize the experiments based on the objectives defined in Chapter 1.

CHAPTER 4

SELECTION OF PLASTIC MANUFACTURING PROCESS

Introduction

This chapter describes an objective based multi attribute decision making (MADM) method to deal with the process selection problem considering both qualitative as well as quantitative attributes applied in evaluation and selection of the plastic manufacturing processes. MADM based selection procedure is explored to rank the different processes to quantitatively assist a manufacturer to select a suitable process from a long list for a specific application. A ranked value judgment on a fuzzy conversion scale using fuzzy set theory approach is used to convert qualitative value to quantitative value. The proposed method helps the decision maker to arrive at a decision, based on the objective weights of the attributes. As an emerging application, the process selection procedure based on MADM is carried out for an automobile fuel tank to explain the intricacies involved. The objective based MADM methodology is sufficiently discussed in Chapter 3. The following section depicts the application of the methodology for the process selection of an automobile fuel tank.

4.1 Identification of Attributes

Selection of a plastic manufacturing process for a particular application is going to affect the overall efficiency of the product. Values pertaining to each attributes are going to decide the performance of an alternative process. It shows that identification of attributes is crucial in selection of manufacturing process from number of alternatives available.

Plastic manufacturing process consists of various numbers of sub systems such as product quality, mould parameters, operational parameters, manufacturability, human subsystems and environmental subsystems etc. All these sub systems are interdependent and inter related to each other. The performance, cost, behaviour, etc of plastic product or process depends upon the performance of each sub or sub–sub system. The various attributes which comes under these subsystems are shown in Table 4.1.

Table 4.1 Subsystems and corresponding Attributes

Subsystems	Attributes
Mould parameter	Mould material
	Mould pressure
	Mould cost
	Inside mould graphics
Final Product Quality	Wall thickness distribution
	Part detailing
	Product volume range
	Residual stress entrapped
Operational Parameters	Temperature
	Pressure
	Cycle time
Material Property	Melt flow index (MFI)
	Melting temperature
	Glass transition temperature
Manufacturability	Cost of production
	Availability of raw material
	Accuracy
	Tolerance
Human	Safety
	Instructiveness
	Training required
	Ergonomics
Environmental	Cleanliness of surrounding
	Surrounding space
	Humidity

Here, 25 attributes are found, but the user, designer or manufacturer can add or delete some of the attributes depending upon their requirement. Because it is evident that the importance of an attribute is changing depending upon application, use or industries. Even all the attributes may not be known to user, manufacturer or designer, but they may be important to them. Therefore, sensitive/critical attribute should be considered by them.

4.2 Decision Table

The process selection problem considered five alternative processes (PR) and fifteen attributes and the data are as shown in Table 4.2. Five alternative manufacturing processes considered for making plastic automotive fuel tank are blow moulding (BM), thermoforming (TF), rotational moulding (RM), injection moulding (IM), and contact moulding (CM). The attributes are: plastic available (PA), mould cost (MC), wall thickness uniformity (WT), residual stress (RS), part detailing (PD), inside mould graphics (MG), cycle time (CT), labour intensive (LI), typical product range (PR), automation (AM), scrap (SC), labor cost in mould (LM), cost of equipment (CE), finishing cost (FC), production cost (PC). In the stated attributes PA, WT, PD, MG, PR, AM are considered as the beneficial attributes and MC, RS, CT, LI, SC, LCIM, CE, FC, PC are considered as non-beneficial attributes for the considered application.

Table 4.2 Properties/ attributes of candidate process for plastic automotive fuel tank

sno	**PR**	**PA**	**MC**	**WT**	**RS**	**PD**	**MG**	**CT**	**LI**	**PR**	**AM**	**SC**	**LM**	**CE**	**FC**	**PC**
1	**BM**	H	L	A	A	VH	H	L	L	H	H	L	L	A	L	H
2	**TF**	H	A	A	H	H	A	L	A	VH	A	H	L	L	L	H
3	**RM**	L	L	H	L	A	H	H	H	VH	A	L	L	L	L	A
4	**IM**	H	H	A	H	H	H	H	L	H	H	H	H	H	L	VH
5	**CM**	L	L	A	A	H	H	H	VH	H	BA	H	L	L	A	L

All the qualitative attributes are converted into quantitative attributes using fuzzy conversion scale proposed in Table 4.3. Table 4.4 presents the data of all fifteen attributes after this conversion. All the attributes in Table 4.4 are then normalized for different alternatives using Eq. (4.1) and given in Table 4.5.

Table 4.3 Conversion of linguistic terms into fuzzy scores

Qualitative measures of selection attribute	Fuzzy number	Assigned crisp score
Exceptionally low (EXL)	M1	0.0455
Extremely low (EL)	M2	0.1364
Very low (VL)	M3	0.2273
Low (L)	M4	0.3182
Below average (BA)	M5	0.4091
Average (A)	M6	0.5
Above average (AA)	M7	0.5909
High (H)	M8	0.6818
Very high (VH)	M9	0.7727
Extremely high (EH)	M10	0.8636
Exceptionally high (EXH)	M11	0.9545

Table 4.4 Quantitative value using fuzzy conversion scale

sno	**PR**	**PA**	**MC**	**WT**	**RS**	**PD**	**MG**	**CT**	**LI**	**PR**	**AM**	**SC**	**LM**	**CE**	**FC**	**PC**
1	**BM**	0.6818	0.3182	0.5	0.5	0.7727	0.6818	0.3182	0.3182	0.6818	0.6818	0.3182	0.3182	0.5	0.3182	0.6818
2	**TF**	0.6818	0.5	0.5	0.6818	0.6818	0.5	0.3182	0.5	0.7727	0.5	0.6818	0.3182	0.3182	0.3182	0.6818
3	**RM**	0.3182	0.3182	0.6818	0.3182	0.5	0.6818	0.6818	0.6818	0.7727	0.5	0.3182	0.3182	0.3182	0.3182	0.5
4	**IM**	0.6818	0.6818	0.5	0.6818	0.6818	0.6818	0.6818	0.3182	0.6818	0.6818	0.6818	0.6818	0.6818	0.3182	0.7727
5	**CM**	0.3182	0.3182	0.5	0.5	0.6818	0.6818	0.6818	0.7727	0.6818	0.4091	0.6818	0.3182	0.3182	0.5	0.3182

$$r_{ij} = Y_{ij} / \sum_{i=1}^{n} Y_{ij} \tag{4.1}$$

Where r_{ij} is the normalized value of Y_{ij} and $\sum_{i=1}^{n} Y_{ij}$ is the total of the value of *jth* attribute for *n* number of alternative.

Table 4.5 Normalized data for calculating the objective weights of attributes

sno	**PR**	**PA**	**MC**	**WT**	**RS**	**PD**	**MG**	**CT**	**LI**	**PR**	**AM**	**SC**	**LM**	**CE**	**FC**	**PC**
1	**BM**	0.2542	0.1489	0.1864	0.1864	0.2329	0.2113	0.1187	0.1228	0.1899	0.2459	0.1187	0.1628	0.2340	0.1795	0.2308
2	**TF**	0.2542	0.2340	0.1864	0.2542	0.2055	0.1549	0.1187	0.1930	0.2152	0.1803	0.2542	0.1628	0.1489	0.1795	0.2308
3	**RM**	0.1187	0.1489	0.2542	0.1187	0.1507	0.2113	0.2542	0.2632	0.2152	0.1803	0.1187	0.1628	0.1489	0.1795	0.1692
4	**IM**	0.2542	0.3191	0.1864	0.2542	0.2055	0.2113	0.2542	0.1228	0.1899	0.2459	0.2542	0.3488	0.3191	0.1795	0.2615
5	**CM**	0.1187	0.1489	0.1864	0.1864	0.2055	0.2113	0.2542	0.2982	0.1899	0.1475	0.2542	0.1628	0.1489	0.2820	0.1077

4.3 Objective Weights of the Attributes

According to equation Eq. (4.2) the statistical variance for all the fifteen attributes were computed as given below in Table 4.6. Objective weights of the attributes are determined using Eq. (4.3) and are given in Table 4.7.

$$V_j = \left(\frac{1}{n}\right) \sum_{i=1}^{n} \left(r_{ij} - \left(r_{ij\,mean}\right)\right)^2 \tag{4.2}$$

Where V_j is the statistical variance of the data corresponding to the j^{th} attribute and $(r_{ij})_{mean}$ is the is the mean value of r_{ij}.

Table 4.6 Statistical variance of fifteen attributes

V_{PA}	V_{MC}	V_{WT}	V_{RS}	V_{PD}	V_{MG}	V_{CT}	V_{LI}	V_{PR}	V_{AM}	V_{SC}	V_{LM}	V_{CE}	V_{FC}	V_{PC}
0.0044	0.0046	0.0007	0.0026	0.0007	0.0005	0.0044	0.0051	0.0002	0.0015	0.0044	0.0055	0.0046	0.0017	0.003

$$Q_j^o = V_j \Big/ \sum_{i=1}^{m} V_j \qquad (4.3)$$

Where $\sum_{i=1}^{m} V_j$ is the total value of statistical variances of m number of attributes.

Table 4.7 Objective weights of fifteen attributes

Q^o_{PA}	Q^o_{MC}	Q^o_{WT}	Q^o_{RS}	Q^o_{PD}	Q^o_{MG}	Q^o_{CT}	Q^o_{LI}	Q^o_{PR}	Q^o_{AM}	Q^o_{SC}	Q^o_{LM}	Q^o_{CE}	Q^o_{FC}	Q^o_{PC}
0.1000	0.1051	0.0167	0.0583	0.0163	0.0115	0.1000	0.1161	0.0035	0.0351	0.1000	0.1255	0.1051	0.0381	0.0687

4.4 Determination of Preference Index

The preference index values are computed for different alternatives using the Eq. (4.4) and (4.5). First, X_i^o values are calculated for all the five alternatives based on Eq. (4.4) as shown in Table 4.8.

$$X_i^o = Q_j^o rij^{**} \tag{4.4}$$

Where X_i^o is the product of objective weight Q_j^o and rij^{**} for different attributes.

.

$$P_i^o = \sum_{j=1}^{m} X_i^o \tag{4.5}$$

Table 4.8 X_i^o Values for preference index

sno	**PR**	X^o_{PA}	X^o_{MC}	X^o_{WT}	X^o_{RS}	X^o_{PD}	X^o_{MG}	X^o_{CT}	X^o_{LI}	X^o_{PR}	X^o_{AM}	X^o_{SC}	X^o_{LM}	X^o_{CE}	X^o_{FC}	X^o_{PC}
1	**BM**	0.1000	0.1051	0.0122	0.0371	0.0163	0.0115	0.1000	0.1161	0.0031	0.0351	0.1000	0.1255	0.0669	0.0381	0.0320
2	**TF**	0.1000	0.0669	0.0122	0.0272	0.0144	0.0084	0.1000	0.0739	0.0035	0.0257	0.0467	0.1255	0.1051	0.0381	0.0320
3	**RM**	0.0467	0.1051	0.0167	0.0583	0.0106	0.0115	0.0467	0.0542	0.0035	0.0257	0.1000	0.1255	0.1051	0.0381	0.0437
4	**IM**	0.1000	0.0490	0.0122	0.0272	0.0144	0.0115	0.0467	0.1161	0.0031	0.0351	0.0467	0.0586	0.0490	0.0381	0.0283
5	**CM**	0.0467	0.1051	0.0122	0.0371	0.0144	0.0115	0.0467	0.0478	0.0031	0.0211	0.0467	0.1255	0.1051	0.0243	0.0687

Preference index P_i^o can be calculated by summing the values of X_i^o for each alternative process considered. Five alternative plastic manufacturing processes are ranked based on computed values of preference index and the ranks are given in Table 4.9.

Table 4.9 Ranking of five alternative plastic manufacturing process

Manufacturing Process considered	Values of objective based MADM (P_i^o)	Rank based on (P_i^o)
Blow moulding **(BM)**	0.899111208	1
Thermoforming **(TF)**	0.779731489	3
Rotational moulding **(RM)**	0.791334307	2
Injection moulding **(IM)**	0.636052682	5
Contact moulding **(CM)**	0.71582105	4

4.5 Final Selection

Ranking is done by objective based MADM. A final decision may be taken keeping in view of the practical considerations. These include constraint such as (1) Economic consideration, (2) Availability, (3) Management constraints, corporate polices, (4) International market policies, (5) material processing constraints etc. However, compromise may be made in favour of an alternative with a higher value of preference index.

4.6 Usefulness of Methodology

This methodology is user friendly even for a novice user. The only inputs required in this methodology are the values pertaining to the decision matrix and management strategies of the organization. Using this methodology the manufacturer identifies different attributes which can be stored in a database and retrieved when ever required. This will also facilitate in generation of a computerized database which will help the manufacturer to select the best manufacturing process for the given application or purpose. Manufacturing process selected by this procedure will be the appropriate process in the market according to the values of pertinent attributes specified.

4.7 Summary

1. An objective based MADM methodology is useful in evaluation, comparison and selection of a manufacturing process for particular application.
2. The method is simple, convenient and helps the decision maker to arrive at a decision based on the objective weights of the attributes.
3. Evaluation and ranking of a process based on different methods is illustrated using an example i.e. selection of a plastic manufacturing process.
4. Only by giving the decision matrix as an input, the manufacturer will directly get the ranking of each alternative.
5. The statistical variance concept of determining the objective weights of the attributes is used in the method, which is comparatively simpler than the entropy method suggested by the previous researchers (Jee and kang, 2000; Shanian and Savadogo, 2006[a]; 2006[b]; 2006[c]).
6. The method can deal with the process selection problems considering both qualitative and quantitative attributes. The ranked value judgment on a fuzzy conversion scale for the qualitative attributes is used, which will be more useful to the designers or manufacturers.
7. It is found that for the application under consideration, the highest value of index is 0.89 for blow moulding process. This is followed by rotational moulding and injection moulding.

Even though highest preference indexed process are considered as suitable process for the intended application, the final decision has to be taken based on the constrains and advantages of the process. If we consider the tradeoff between the first two ranked processes namely blow moulding and rotational moulding, blow moulding is well established process for producing various plastic products. But it has certain constraints like only thin walled parts can be made, corner radius should be generous, holes cannot be moulded in, etc. All the listed constrains for the blow moulding become advantages for the rotational moulding process and especially the parts produced by rotational moluding process are relatively stress free compared to the blow moulding process. However, having issues such as longer oven residence time, lack of prediction and optimization of process condition etc., makes this process unsuitable for some situations.

Therefore, efforts have been taken in the study to solve some of the issues related to rotational moulding process in the subsequent chapters like optimization of oven residence time, effect of process parameters on thickness, prediction and understanding of mechanical and fracture characterization.

CHAPTER 5

PREDICTION OF OVEN RESIDENCE TIME

Introduction

One of the pertinent issues of rotational moulding process i.e. oven residence time as highlighted in Chapter-4, is investigated in detail using simulation and experiments. The work mainly aims on identifying favourable process window of oven residence time for the rotomoulders which yields superior mechanical properties. In rotational moulding of plastics, improving the mechanical properties (like tensile, flexural and impact) without sacrificing the processibility is the biggest challenge. Therefore, an attempt has been made to investigate the effect of oven residence time on the mechanical properties of the rotationally moulded products. Simulation studies are conducted using ROTOSIM software to analyze different thermal transitions and phase changes that occur in the process. Degree of curing of the polymers is also assessed from the simulation study to correlate with mechanical properties. Experiments are further conducted to correlate the simulation studies and obtain conditions for optimal oven residence time. Experimental investigation revealed that there exist regions where the part is 'under-cured' and mechanical properties are found to be inferior. It is also found that when parts are 'over-cured', the mechanical properties are severely affected. A regime of optimal processing window is identified where the optimum tensile, flexural and impact properties are noticed.

5.1 Simulation and Experimental Methods

Experimental and simulation details are summarized in the Table 5.1 as shown below. The details of the same have been sufficiently discussed in Chapter-3.

Table 5.1 Experimental set up and simulation details

Rotational moulding machine specifications	Model: Clamshell type single arm biaxial machine
Mould specifications	Material : aluminum(Al)
	Shape: inverted frustum of a cone
	Release agent: Metrork silicone 17 compound

	Dimensions: Top diameter: 300 mm Bottom diameter: 150 mm Height: 220 mm
Raw material specifications	Material: R350 A 42 LLDPE supplied by GAIL India
	MFI: 4.2 g/10 min
	Density: 935 kg/m^3
	Shot weight: 0.6 kg
Testing Equipment	Tensile and flexural testing: Machine: universal testing machine load cell : 0-5 kN cross head speed of 5 mm/minutes
	Impact testing Izod impact tester with 4 J pendulum.
Simulation Studies	Part modeling: Using Pro-E wild fire Meshing details: Triangular elements with 3041 nodes. Simulation studies: Using ROTOSIM software

5.2 Preliminary Experiments for Choosing the Regime of Oven Residence Time

Preliminary experiments are initially conducted to decide the regime of oven residence time and assess the internal air temperature of the mould. In all the trials, oven temperature is set at 240^{o}C. From these experiments, it is found that oven residence time below 26 minutes is not sufficient for all the polymer powder to melt and fuse though some powder particles adhere to the inner surface of the mould forming a thin layer over the mould wall. This can be seen as un-melted powder in the flange region of the product as shown in Fig 5.1. It is also observed that at least 32 minutes are required for complete powder conversion into the melt (thus 26-32 minutes are required for powder to melt conversion). Beyond 44 minutes, we notice the burning and degradation of the polymer.

This has enabled us to choose the regime of oven residence time from 32 to 44 minutes. The internal air temperature is measured using a K type thermocouple considering uni-axial rotation of the mould as with this setup it is not possible to measure the same bi-axially. It is confirmed that this temperature is sufficient to get a peek internal air temperature of 220^{o}C in the above time regime. With the chosen regime, experiments are performed by varying the oven residence times and mechanical properties of the product in terms of tensile, flexural and impact strengths of the product are determined.

Fig 5.1 Moulding obtained for oven residence time below 26 minutes

5.3 Preliminary Experiments for Simulation Studies

For conducting the simulation studies, the temperature profile around the mould is an important input and that must be same as actual temperature around the mould. Thus to correlate the findings with the experimental conditions, the mould temperature is measured (details given in section 3.2) and the mould enviornmental profile is generated as shown in Fig 5.2. The profile is given as input to ROTOSIM. The profile shows a time period of around 1680 seconds (almost 28 minutes) to reach to peak set temperature of 220^{o}C. The temperature is held constant at 220^{o}C from 1680 to 1780 seconds using ON-OFF relays as shown by the points B-C in Fig 5.2. The oven residence time from 32 minutes to 44 minutes have been considered in this study. This can be realized as increase in the length BC in Fig 5.2. The other features like speed reversal of the mould, internal cooling of the mould, etc. have not been taken into account in this study. The outcomes of this simulation studies are compared with those obtained from experimental studies and discussed in the results and discussion (section 5.4).

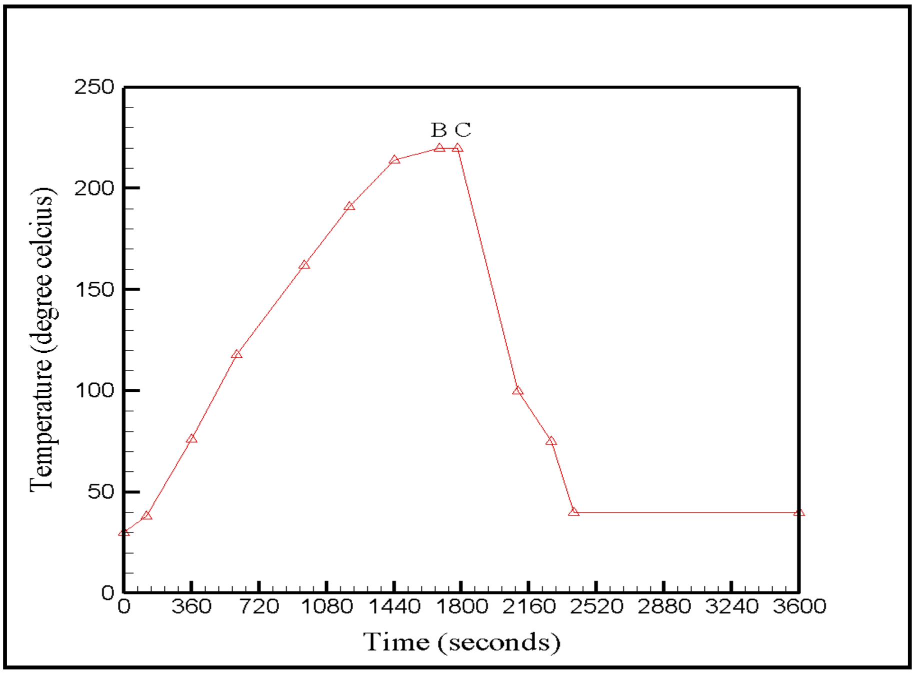


Fig 5.2 Typical mould enviornmental profile

5.4 Results and Discussions

5.4.1 Testing of Mechanical Properties

Rotationally moulded products are obtained from rotational moulding process and test specimens are prepared and tested according to ASTM D 638 (2010) for tensile, ASTM D 790 (2010) for flexural & ASTM D 256 (2010) for impact testing respectively. In order to account for process variability during experimentation, three replications are taken for each experimental run. Thus three samples of each tensile, flexural and impact specimens are cut and subjected to testing.

Table 5.2 shows the plan of experiments, the value of tensile, impact and flexural strength obtained by changing the oven residence time.

Table 5.2 Experimental run and corresponding output

Run order	Oven residence Time	Tensile strength (MPa)	Average tensile strength (MPa)	Impact strength (J)	Average impact strength (J)	Flexural strength at yield (MPa)	Average flexural strength (MPa)
1	32	16	16	0.58	0.52	16.2	18.2
2		16.5		0.46		20.2	
3		15.5		0.52		18.18	
4	34	16.4	16.2	0.72	0.69	20	18
5		16		0.68		18	
6		16.2		0.67		16	
7	36	17.2	17.2	0.96	0.92	19.7	17.6
8		17.4		0.9		15.5	
9		17		0.9		17.6	
10	38	17.6	17.4	0.95	0.97	20	17
11		17.4		0.99		16	
12		17.2		0.97		15	
13	40	17.8	17.4	0.8	1	17	17.2
14		17.4		1.2		17.6	
15		17		1		17	
16	42	18	16	1	0.8	20.8	16.7
17		14		0.6		14.6	
18		16		0.8		14.7	
19	44	15.5	15.3	0.66	0.6	20	16.0
20		15.4		0.54		14	
21		15		0.6		14	

5.4.2 Tensile Strength

The results of tensile testing are as shown in Fig 5.3. These results reveal that tensile strength gradually increases as the oven residence time increases from 32 to 40 minutes and falls after 40 minutes. A closer look into the process gives more insight of this phenomenon. As the temperature increases from room temperature (30^{o}C), the powder inside the mould tumbles and when the temperature exceeds 120^{o}C the powder starts sticking to the mould surface. With further increase in temperature, coalescence between the powder particles takes place and it becomes loose porous mass. In order to get the optimal mechanical properties, the PIAT of the mould should reach to 220^{o}C as specified earlier. In the present case, though a PIAT of 220^{o}C is achieved, the molten mass spends a short duration of time in this condition. This resulted in an incomplete fusion of the polymer particles resulting in a non homogeneous structure including entrapment of irregularly shaped air pockets. Therefore, it is obvious that the tensile strength is considerably less between the regions 32 to 35 minutes. This is referred as ‘under-curing’ and resulted in a weak product.

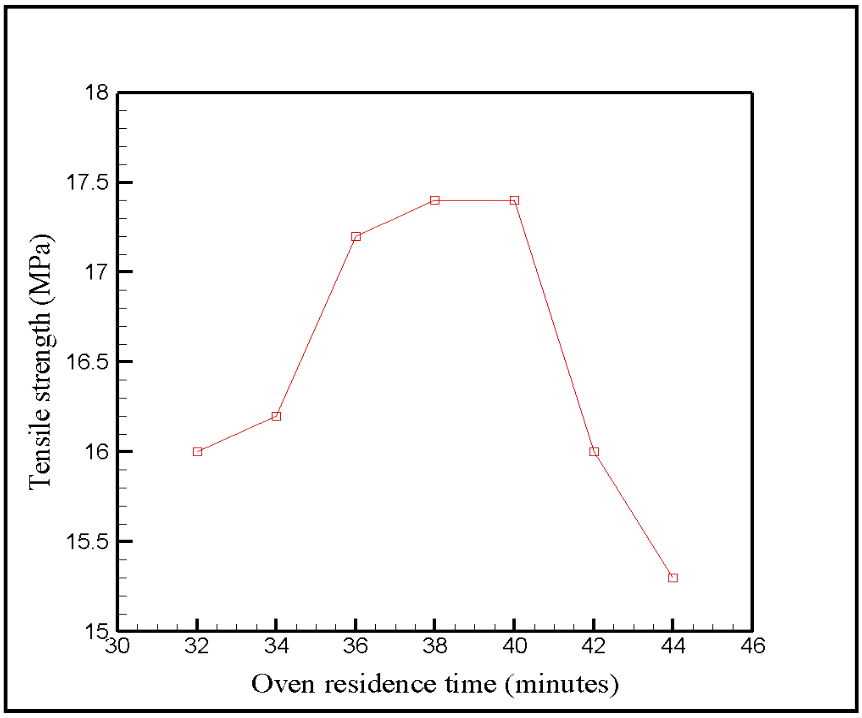


Fig 5.3 Variation of tensile strength with respect to Oven residence time

As the oven residence time is from 36 to 40 minutes, the polymer melt spent sufficient time in the oven. Thus, the irregular pockets of trapped air transform into spheres. Most of the trapped spherical bubbles disappear and a complete coalescence with homogeneous structure is achieved. This region can be referred as a 'completely-cured' region and results in a good tensile strength of the product.

Further, if the oven residence time is increased above 40 minutes the tensile strength of the product is decreased, as the degradation of the polymer started at this stage. This is referred as 'over-cured' and results in a partially burnt product with suboptimal tensile properties.

In order to correlate the mechanical behaviour of LLDPE with that of oven residence time, differential scanning calorimetry (DSC) is performed to characterize the material with a steady temperature rate of 10^{o}C/minutes. The DSC graph is as shown in Fig 5.4. It shows an endothermic peak at 124^{o}C revealing melting of LLDPE. The material shows excellent thermal stability after melting till a temperature of 249^{o}C. The exothermic peak at 249^{o}C shows the commencement of degradation of the polymer. The peaks after 400^{o}C indicates complete burning of LLDPE including additives. Since the oven heating rate in the rotational moulding set up is around 5^{o}C/minutes, the decomposition temperature of polymer is realized after 40 minutes. This resulted in a decrease in tensile strength of the product.

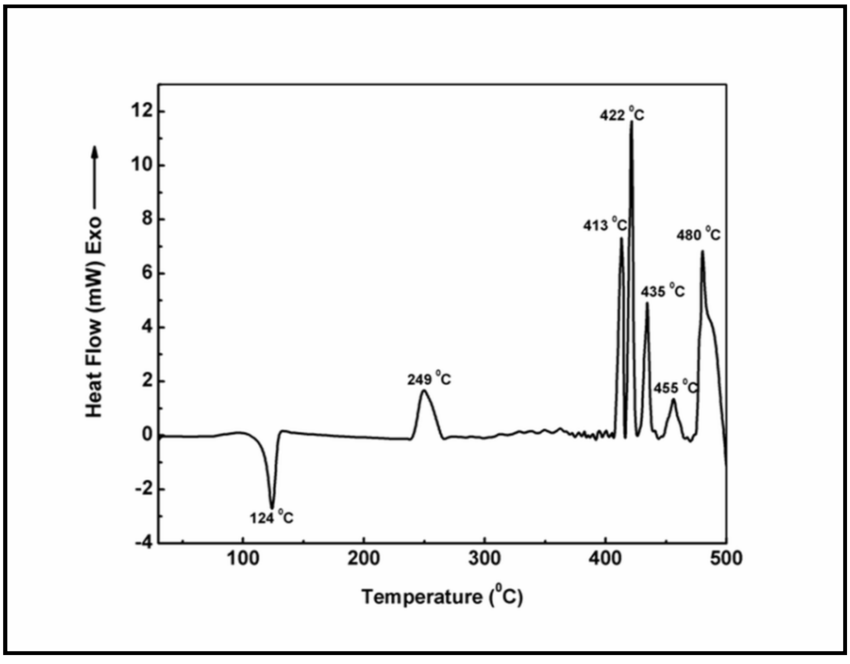


Fig 5.4 Differential scanning calorimeter (DSC) trace for LLDPE

The simulation studies conducted in section 5.3, provide further insight on the transient temperature distribution inside the mould which can be correlated with the mechanical properties of the product. The transient temperature distribution of the process is shown in Fig 5.5, while the polymer phase change plot is depicted in Fig 5.6 for 32 minutes of oven residence time.

From these plots, the time lag due to convective heating from oven (environment) to the mould could be clearly seen. The internal air temperature plot (IAT) follows the phase changes that take place in the LLDPE. For example, we can notice a horizontal straight line at around 124^{o}C (900 seconds), indicating the phase change of the polymer from the powder to the melt. This also can be seen from the phase change plots of the polymer as shown in Fig 5.6. At 900 seconds the polymer starts converting into melt thereby increasing its mass fraction. At the same time, since the powder is being converted into melt, its mass fraction decreases. It can be noticed from Fig 5.6 that at around 1200 seconds all the powder gets converted into melt and thereafter polymer remains in the molten state. From Fig 5.5, we notice that even after switching off the oven at 1800 seconds, the IAT keeps on rising and it reaches to 220^{o}C which is a PIAT of the polymer processed. At 2700 seconds the molten LLDPE again starts losing the latent heat of fusion and gets converted into solid phase. Thus complete solidification occurs at around 3200 seconds (100% solid, 0% melt) as evident from Fig 5.6. Thus we could notice from this simulation that PIAT of 220^{o}C could be reached in this setting, which is generally considered as healthy sign to get better mechanical properties of rotaionaly moulded products. Similar type of observations are recorded by Shih- Jung and Kang-Ming Peng (2010) for Polycarbonate reinforced polyethylene composites which suggested an optimum PIAT of 220^{o}C yeilding better mechanical properties. However, here the polymer has spent around 3.5 minutes (1750 to 1950 seconds) at this temperature, which has caused undercuring of the product, resulting in lower tensile properties.

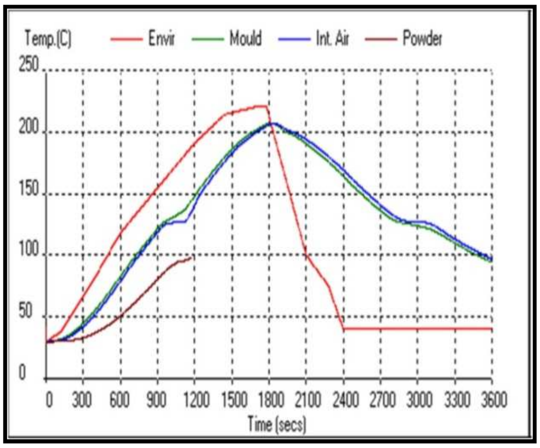


Fig 5.5 Temperature plot for 32 minutes

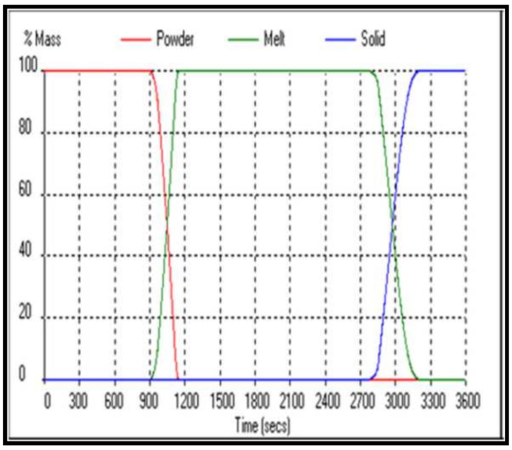


Fig 5.6 Polymer phase transitions plot for 32 minutes

Similarly, all 6 simulations are performed by varying the oven residence times starting from 32 minutes to 44 minutes keeping other parameters constant. The temperature and polymer phase transitions plots of these are as shown in Apendix A; (Figs A1to A10) . We can observe that for 34 minutes, the molten mass is above PIAT of 220°C for 360 seconds, for 36 minutes it is 480 seconds, for 38 minutes it is 600 seconds, for 40 minutes it is 720 seconds, for 42 minutes it is 840 seconds and so on. In order to reperesnt the extent of time which LLDPE has spent above the melting temperature, time-temperature curves are plotted. These are referred as the degree of curing plots and are shown in Appendix A; (Figs A 11 to A16) for different oven residence times. It can be seen that, a higher degree of curing is achieved when the oven residence time is increased. Theoretically, though the higher degree of curing means better mechanical properties, polymer degradation starts beyond a specific temperature (249°C, as confirmed by DSC), which results in reduction in mechanical properties. The results are summarized in Table 5.3 clearly indicates the influence of oven residence time, degree of curing and time for which the material is above 220°C on the tensile and flexural strength of LLDPE. Thus it can be said that it is not only PIAT but also the time that the polymer spends above the PIAT governs the mechanical properties of the rotationally moulded products.

Table 5.3 Influence of oven residence time and degree of curing on tensile &flexural strength

S.no.	Oven residence time (minutes)	Degree of curing in ^{o}C-minutes	Time in sec above 220 ^{o}C	Tensile strength (MPa)	Flexural strength in (MPa)
1	32	1340.56	300	16	18.2
2	34	1600.85	360	16.2	18
3	36	1761.95	480	17.2	17.6
4	38	2088.51	600	17.4	17
5	40	2414.14	720	17.4	17.2
6	42	2494.90	840	16	16.7

5.4.3 Flexural Strength

The results of flexural testing are shown in Fig 5.7. These results reveal that the flexural strength of the product decreases as the oven residence time increases from 32 to 38 minutes. It can be attributed to the incomplete coalescence of powder particle. Such a part can have entrapped bubbles, leading to reduction in flexural strength. With the further increase in oven residence time from 38 to 40 minutes, the entrapped bubbles may escape owing to reduction in polymer melt viscosity there by increasing the flexural strength. Beyond 40 minutes, the product is subjected to oxidation and it becomes brittle. Therefore, the flexural strength is further reduced. Similar observation of oxidation in rotational moulding process for longer oven residence time is notified by Shih- Jung and Kang-Ming Peng (2010) in the past which also resulted in decrease in strength of the parts.

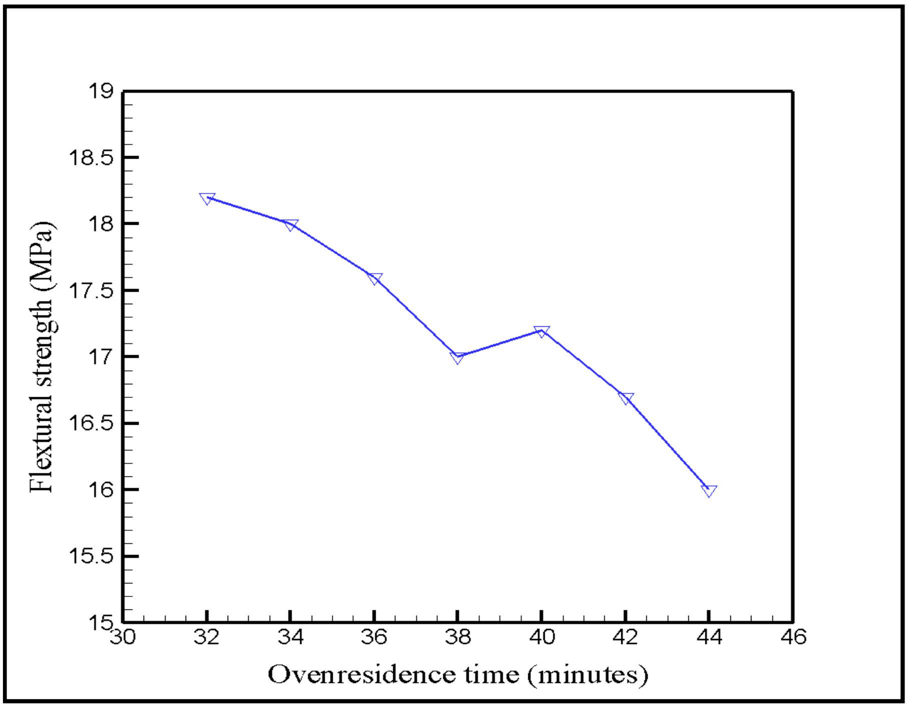


Fig 5.7 Variation of flexural strength with respect to oven residence time

5.4.4 Impact Strength

Variation of impact strength with respect to the oven residence time is as shown in Fig 5.8. These results reveal that impact strength gradually increases as the oven residence time increases from 32 to 40 minutes and falls after 40 minutes. The increase in impact strength from 32 to 40 minutes of oven residence time can be attributed to the formation of small spherulites that grows sufficiently larger and get bonded with neighboring particles. During this stage of polymer sintering, the patches of air are trapped in the polymer melt because of high viscosity. Further rise in temperature reduces the bubble diameter due to increase in pressure inside the air bubbles. As a consequence, total number of bubbles get reduced which reflects as increase in part density and improved impact strength. Identical observation is evident in the past literature by S.B Tan *et al* (2011), M.C cramez *et al* (2003) and M.J Oliveira et al (1996), confirming the increase in impact strength when all bubbles disappear. When the oven residence time is increased above 40 minutes, the product is not only overcooked and degraded but also contains course structure in which there are few large crystalline regions produced**.** Because of large crystalline regions, it is easier for the crack to propagate through the structure that leads to a reduction in impact strength. Similar trend of increase and decrease of impact strength with change in oven temperature has been observed by Shih Jung (2000) and Crawford (1996) at the past.

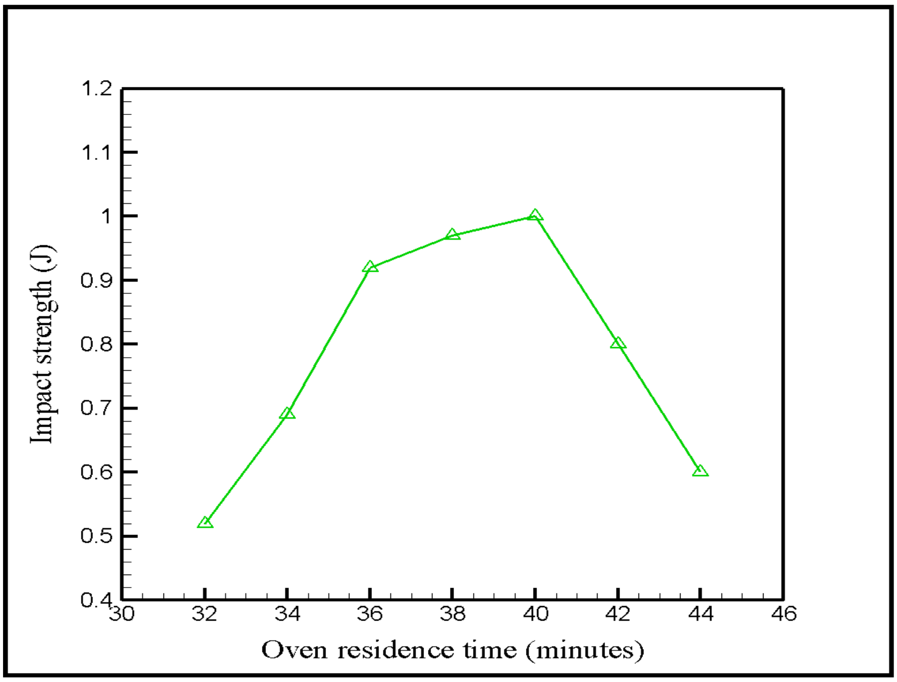


Fig 5.8 Variation of impact strength with respect to oven residence time

With the above findings, it is possible to summarize the variation of mechanical properties with respect to the oven residence time as shown in Fig 5.9. Based on the values of the mechanical properties an optimal processing window can be suggested that lies between 36 to 40 minutes (region between two vertical lines) where highest mechanical properties are obtained. Below and above these values, the quality of products obtained will be inferior.

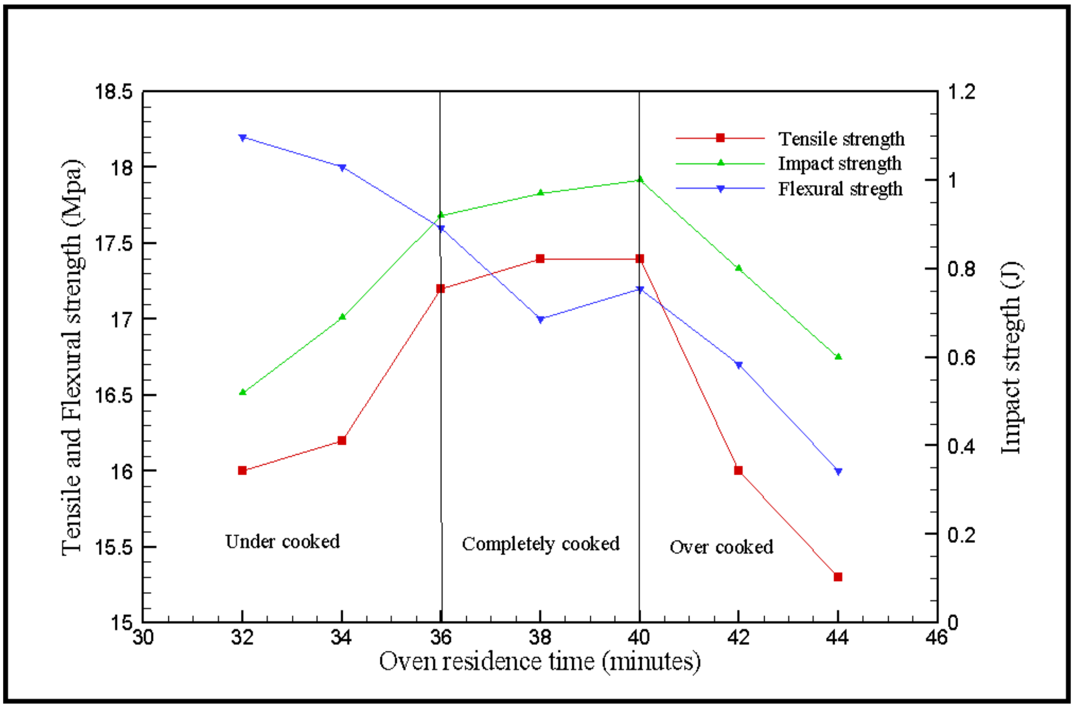


Fig 5.9 Processing windows for LLDPE product

5.5 Confirmatory Experiments

In order to verify the mechanical properties within the regime of optimal processing time window, confirmatory experiments are performed. This is done by considering the oven residence time between 36 to 40 minutes where optimum mechanical properties are predicted. Three replications are carried out for each of these three oven residence times. The results of the confirmatory experiments are shown in Table 5.4. It could be seen that values of tensile, impact and flexural strengths are in close agreement with the optimized process conditions as predicted from Fig 5.9.

Table 5.4 Confirmatory experiments

Trial no	Oven residence time (minutes)	Tensile strength(MPa)	Average tensile strength (MPa)	Impact strength (J)	Average impact strength (J)	Flexural strength (Mpa)	Average flexural strength (MPa)
1	36	17.4	17.4	0.92	0.94	17.2	17.1
2		17.4		0.94		16.9	
3		17.6		0.96		17.4	
4	38	17.6	17.6	0.94	0.96	17	16.9
5		17.6		1		15.5	
6		17.8		0.96		18.2	
7	40	17.6	17.4	1.1	1	17.2	17
8		17.4		0.92		17.2	
9		17.4		1		16.8	

5.6 Summary

In this study, the effect of oven residence time on the mechanical properties of rotationally moulded LLDPE products has been investigated. Experiments are conducted on a laboratory scale rotational moulding machine by varying the oven residence time from 32 to 44 minutes. Tests are conducted on the rotomoulded products for tensile, flexural and impact strengths according to ASTM standards.

It is observed that tensile and impact strength shows an increasing trend whereas flexural strength falls when oven residence time is increased from 32-38 minutes. This can be attributed to the formation of a homogeneous melt due to escapement of entrapped air pockets with increase in temperature. One important observation is that PIAT of 220^{o}C is not only important factor but also the time the polymer spends beyond this temperature is crucial in governing the mechanical properties. Simulation studies are conducted to analyze the different thermal transitions along with phase changes and the degree of curing of the polymers is assessed. This is correlated with the mechanical properties.

It is also observed that the mechanical properties are reduced beyond 40 minutes of oven residence time, though theoretically high degree of curing is obtained. This can be attributed to the degradation of the polymer which is confirmed by conducting DSC studies. Thus, a regime of optimal processing window is obtained between 36-40 minutes where the maximum tensile strength of 17.4 MPa, flexural Strength of 17.1 MPa and Impact strength of 1 J is noticed. These are verified by conducting confirmatory experiments.

It should be emphasized here that on an industrial scale, the rotational moulding machines take very less time to reach the set temperature (around $220^{o}C$) due to the presence of high capacity gas burners.

As in our case an electrically heated oven is used, more time is needed for the oven to reach the preset temperature of $220^{o}C$. This can be regarded as one of the practical limitations of this study. The correlation of simulation and experimental studies provide new insight of process control that can be extended to polymers like Polypropylene, ABS, etc. having a narrow processing time window but excellent mechanical properties.

CHAPTER 6

THICKNESS VARIATION IN ROTOMOULDED PRODUCTS

Introduction

One of the advantages of rotational moulding is that once the mould has been built, it can be used to produce parts with thicker and thinner walls without mould changes by simply charging the mould with more or less material. In rotational moulding process, the distribution of polymer melt is controlled by two main factors. One is speed ratio and the other is oven residence time. Predicting optimum value of these factors for required thickness is a challenge for any rotomoulder. Moulders have to depend heavily upon trial and error methods as well as experience of the operator to predict the thickness for a particular speed ratio and oven residence time. In this chapter, an attempt has been made to investigate the thickness of the rotational moulded parts for different speed ratios and oven residence time using experiments and statistical techniques. Experimental runs and analysis based on design of experiments (DOE) revealed that thickness of the part is severely affected by both of these process parameters. The procedure will quantitatively assist the rotomoulders to select a proper speed ratio and cycle time for required thickness of the rotational moulded product.

6.1 Experimental Methods

Experimental methods are summarized in the Table 6.1 shown below. The details of the same have been sufficiently discussed in Chapter 3 (Methodology).

Table 6.1 Details of experimental set up

Rotational moulding machine specifications	Model: Clamshell type single arm biaxial machine
Mould specifications	Material : Stainless steel
	Shape: Square cross section
	Release agent: Metrork silicone 17 compound
	Dimensions: 100 X 100 X 100 mm

Raw material specifications	Material: R350 A 42 LLDPE supplied by GAIL India
	MFI: 4.2 g/10 min
	Density: 935 kg/m^3
	Shot weight: 0.160 kg

6.2 Plan of Experiments

In order to fix the range (minimum and maximum level) of parameters for experimental investigations, preliminary experiments are conducted. From the preliminary experiments it is noted that for oven residence time less than 32 minutes, the product obtained are undercooked because of incomplete fusion of powder particles. On the other side for oven residence time higher than 42 minutes, polymer degradation is observed. It is also observed that speed ratio below 3:1 resulted in non uniformity in thickness of the products. Very high speed (more than 5:1), on the other hand produced large number of internal bubbles, which in turn reduces the strength of the product. Therefore, the experimental regime is set as 32 to 42 for oven residence time and 3:1 to 5:1 for speed ratio. Thus two levels of each process parameter, coded as (-1, +1) are identified and are shown in Table 6.2.

Table 6.2 Process parameters and their levels for rotational moulding

Process Parameter	Unit	Designation	Lowest level	Highest level
Speed ratio	Rpm	X1	3:1	5:1
Oven residence time	Minutes	X2	32	42

As there are two parameters, each at two levels with three replications of each and three center points, full factorial design yields 15 (2^2 x 3 + 3) experiments. In full factorial design, the coded variables (–1, +1) are used to develop a first order model while centre points (0, 0) estimates the curvature effect with the help of second order model. The considered design scheme has only 15 experimental trails, yet provides a good assessment of the response (thickness).

6.3 Results and Discussions

6.3.1 Statistical Analysis

Thickness values (in mm) obtained from the experiments as per the design scheme considered is shown in Table 6.3. Statistical analysis using analysis of variance (ANOVA) is carried out to know the factors or interactions which are significantly affecting the response. The stated analysis is executed by considering 5% level of significance (p = 0.05). A 'p' value less than 0.05 can be deemed as significant. This is performed on the grounds that at 95% level of confidence, it rejects the null hypotheses that the factors have no effect on thickness (against an alternative hypothesis that the factors have significant influence on thickness).

Table 6.3 Plan of experiments and corresponding value of thickness

Run order	X_1 (rpm)	X_2 (mins)	Thickness (mm)
1	3	42	2.98
2	5	32	2.90
3	5	42	3.00
4	3	42	2.96
5	3	32	2.40
6	3	32	2.20
7	5	32	2.90
8	3	32	2.12
9	4	37	3.00
10	5	42	3.00
11	4	37	2.70
12	5	32	3.00

13	4	37	2.80
14	5	42	2.98
15	3	42	3.12

The results obtained from statistical analysis based on ANOVA along with‘t’ test is shown in Table 6.4. It is observed from the result that the process parameters have significant linear effect on the thickness. The coefficients of the regression model |β|, which in general can be written as shown in Eq. (6.1).

Table 6.4 Results of ANOVA for Thickness

Term	Coefficient	Standard error Coefficient	T= Coefficient/ Standard error	*p*
Constant	2.7967	0.03033	92.20	0.000*
X_1	0.1667	0.03033	5.49	0.000*
X_2	0.2100	0.03033	6.92	0.000*
X_1*X_2	-0.1800	0.03033	-5.93	0.000*
Ctpt	0.0367	0.06782	0.54	0.601

Note: * Significant at 5% level of significance

T is the test value used in hypothesis testing

$$y = X\beta + \epsilon \quad (6.1)$$

where,

$$[y] = \begin{bmatrix} y_1 \\ y_1 \\ . \\ . \\ y_n \end{bmatrix} \quad [X] = \begin{bmatrix} 1 & x_{11} & x_{12} & .. & x_{1k} \\ 1 & x_{21} & x_{22} & & x_{2k} \\ . & . & . & .. & . \\ . & . & . & .. & . \\ . & . & . & .. & . \\ 1 & x_{n1} & x_{n2} & .. & x_{nk} \end{bmatrix} \quad [\beta] = \begin{bmatrix} \beta_1 \\ \beta_2 \\ . \\ . \\ . \\ \beta_n \end{bmatrix} \quad [\epsilon] = \begin{bmatrix} \varepsilon_1 \\ \varepsilon_2 \\ . \\ . \\ . \\ \varepsilon_n \end{bmatrix}$$

Here, [*y*] is a ($n \times 1$) vector of the observations, [*X*] is a ($n \times j$) matrix of the levels of the independent variables, [β] is a ($n \times 1$) vector of the regression coefficients and [ϵ] is a ($n \times 1$) vector of random errors. The procedures of obtaining these constants have been discussed at sufficient length by Montgomery (2012). Appendix B gives further details and illustrative example (Waigaonkar, 2010 and Waigaonkar *et al.*, 2011). The empirical model for thickness in terms of coded units considering only significant terms from ANOVA is obtained as:

$$\text{Thickness} = 2.7967+0.1667X_1+ 0.2100\ X_2 - 0.1800\ X_1\ X_2 +0.0367 \qquad (6.2)$$

Eq. (6.2) can be comfortably used to estimate thickness for any combination of process parameters within the regime of experimentation (i.e., –1 to +1).

6.3.2 Effect of Speed Ratio and Oven Residence Time on Thickness

To identify the interaction effect of all the parameters on thickness, the interaction plot and surface graph is generated using Minitab 14.0. The interaction plot and surface graph is as shown in Fig 6.1 & 6.2. In these plots, the variation of thickness with respect to the combination of process parameters at different levels is represented. The intersecting lines in the interaction plot show that, when both the parameters are varied simultaneously, the combined effect will be evident on the response.

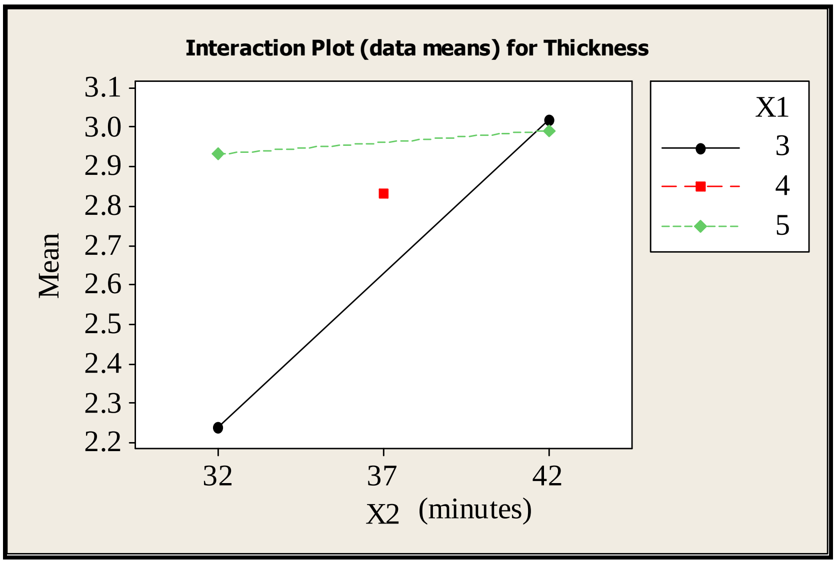


Fig 6.1 Interaction plot for thickness

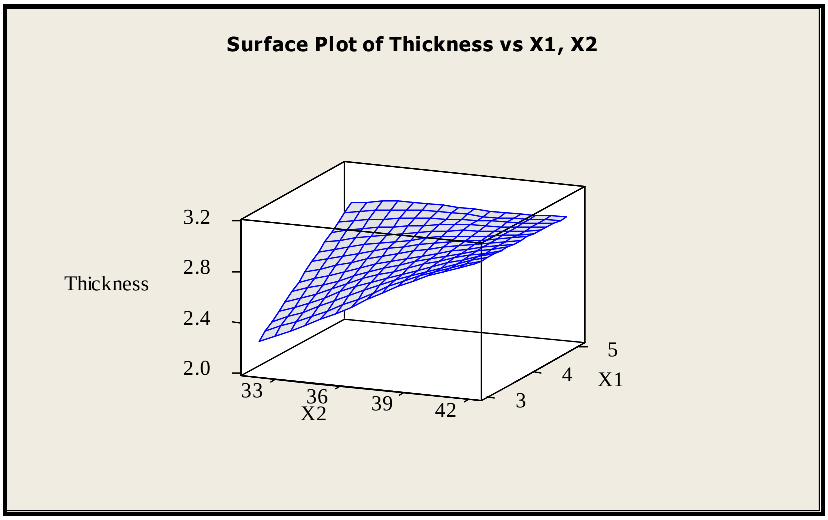


Fig 6.2 3-D Surface plot for cycle time and speed versus thickness

As observed from the interaction plot and surface graph, thickness increases when oven residence time and speed are increased simultaneously. This can be pertained to the fact that the buildup of the plastic on the mould acts as an insulating barrier separating the external heating from the resin that is still tumbling in interior of the mould. This can also be due to the variation in powder particle size, difference in heat absorption rate and there may be time lag between the melting of powders, this time lag may ultimately leads to the thickness variation in the final product.

Similar trend of increase in thickness with respect to increase in oven residence time is observed for nylon (Brent Strong, 2006). The critical importance of powder particle shape and size which affects the flow of powder and ultimately thickness of the mould in rotational moulding is also highlighted by Myer Kutz in (2011).

6.3.3 Statistical Optimization

Optimization is performed using ‘D’ (determinant) optimal design. It is customary to use ‘D’ optimal criterion (Appendix B gives further details and illustrative examples about ‘D’ optimal design) in regression analysis as it is the appropriate and efficient optimization method which rapidly moves towards the optimal solution. This optimality criterion results in minimizing the generalized variance of the parameters for a fitted model. MINITAB 15.0 is used for this optimization. Fig 6.3 shows MINITAB output, the number shown at the top of the window refers to the highest (Hi) and lowest (Lo) level of the process parameters considered for the experimentation. The values shown at the middle in red colour are the current optimized values (Cur) of the process parameters. The values of process parameters, X_1 and X_2 are found as 4:1 and 37 minutes, respectively yielding required thickness of 2.83 mm which is closer to the target value of thickness 2.85 mm indicating a desirability of 0.95238. ‘D’ optimal value of 0.95238 (very close to 1) indicates a proper convergence to optimal solution. Each cell of the graph in Fig 6.3 shows how the response changes as a function of one of the factor while other factor remain fixed and horizontal blue line represent the level of optimized value.

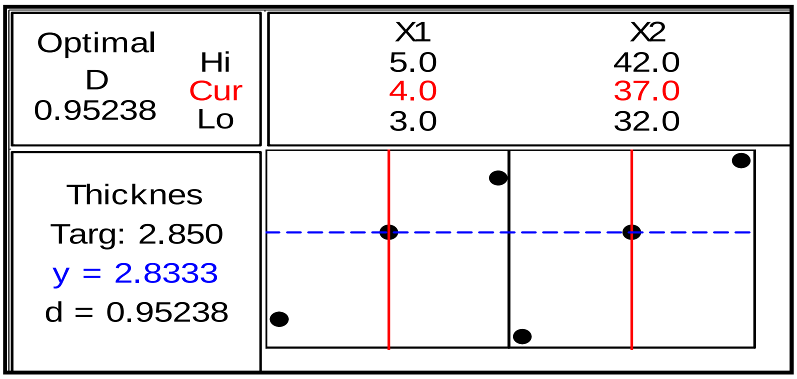


Fig 6.3 Results of D optimality test

6.3.4 Confirmatory Experiments

In order to verify the above results, new set of process parameters are used to carry out the confirmatory experiments. Predicted and experimental results are shown in Table 6.5. The values pertaining to predicted and experimental results are closer to each other. A minor variation in them could be because of the error [Є in 'equation (6.1)'], prompted by the aspects like change in atmospheric temperatures, humidity, etc., while performing the experiments. The mean value of thickness is obtained as 2.76 mm. Thus, above process parameter setting can be suggested to get a required thickness.

Table 6.5 Confirmatory experiments

Run order	X1	X2	Thickness (mm)			% Error with average
			Predicted	Experimental	Average	
1	4:1	37	2.83	2.76	2.76	2.35
2	4:1	37	2.83	2.78		
3	4:1	37	2.83	2.75		

6.4 Summary

In this chapter, thickness variation of the products for different combination of process parameters in rotational moulding process is presented. The full factorial method is used for the design of experiments and fifteen experiments are designed and conducted. Experimental result using ANOVA confirmed that both the process parameters ie. oven residence time and speed ratio have significant effect on thickness of the rotomoulded product. The result obtained confirms that there is a linear relation between the process parameters and response (thickness). Using equation (6.2), the thickness can be predicted for any combination of process parameters within the regime of experimentation (i.e., –1 to +1).

CHAPTER 7

IMPACT STRENGTH OF FOAMED ROTATIONAL MOULDED PRODUCT

Introduction

For the last three decades, rotational moulding has received a great attention because of its low machinery cost, simple tooling, and little waste. One stumbling block of rotomoulded products is its insulative, and shock mitigation properties due to the hollow structure. In order to improve the impact strength, LLDPE foam is mixed with the base resin LLDPE to produce rotomoulded products. Rotational moulding of foamed polyethylene has increasingly become an important process in industry. It has been used to produce parts in various applications such as furniture, toys, and flotation drink containers. Foamed structures provide several advantages in thermoplastic products, which includes lightweight, excellent strength–weight ratio, superior insulation abilities; and energy absorbing performance (shock, vibration, and sound) (Shih- Jung Liu and Ching- Hsiung Yang, 2001).

The main focus of this chapter is to examine the rotomouldability of a foamed polyethylene and their impact strength. Since components produced using rotational moulding process are used in outdoor applications (like overhead chemical storage tanks, water storage tanks, automobile fuel tanks, car bodies etc), impact strength of such products are considered as one of the prime quality feature. This work centers on the use of statistical technique to analyze the impact strength of foamed rotationally moulded products. In this chapter, an attempt has been made to investigate the effect of process parameters and to identify the optimum process parameter yielding maximum impact strength of the foamed rotationally moulded products. Experimental investigations are carried out by planning and performing trials based on design of experiments (DOE). DOE approach, called response surface method (RSM) is used for data analysis.

7.1 Experimental Details

Experimental details are summarized in the Table 7.1 shown below. The details of the same have been sufficiently discussed in Chapter 3, methodology.

Table 7.1 Experimental details

Rotational moulding machine specifications	Model: Clamshell type single arm biaxial machine
Mould specifications	Material : Stainless steel
	Shape: Square cross section
	Release agent: Metrork silicone 17 compound Dimensions: Length: 100 mm Breath: 100 mm Height: 100 mm
Raw material specifications	Material: R350 A 42 LLDPE supplied by GAIL India
	MFI: 4.2 g/10 min
	Density: 935 kg/m^3
	Shot weight: 0.6 kg
Testing Equipment	MFI: Dynisco melt flow indexer MFI specimens: Made as per ASTM D 1238 Impact testing: Izod impact tester with 4 J pendulum Impact specimens: Made as per ASTM D256

7.2 Plan of Experiments

Proper control of the process parameters is required to obtain a quality product. The important process parameters that govern the impact strength of foamed rotomoulded product are:

1. Oven temperature (X_1)
2. Oven time (X_2)
3. Cooling medium (X_3)

In order to plan the experiments, the response surface method (RSM) is used. RSM is a collection of statistical and mathematical methods that are useful for modeling engineering problems. The main objective of this technique is to optimize the response surface that is influenced by various process parameters. RSM quantifies the relation between the controllable parameters and the response (Phaneendra Kiran and Shibu, 2013 and Montgomery, 2012). In this work, three process parameters with three levels (-1, 0, +1) are considered. For a combination of three process parameter with three levels, a full factorial scheme will result in 27 (3^3) experiments. Since performing experiments based on full factorial design requires substantial amount of resources and time, the experiments are planed using Box- Behenken design which is subcategory of RSM (Box- Behenken, 1960). In this design the coded variables (-1, +1) are used to develop a first order model where as the center points (0, 0) estimates the second order model with curvature effect. The considered design scheme has only 15 experimental trails, yet provides a good assessment of the response (impact strength). Thus three levels of each process parameter coded as (-1, 0, +1) are identified and shown in Table 7.2.

Table 7.2 Process parameters and their levels for rotational moulding

Process Parameter	Unit	Designation	Lowest level (-1)	Middle level (0)	Highest level (+1)
Oven	^oC	X_1	210	220	230
Oven residence	Minutes	X_2	32	37	42
Cooling		X_3	Still air	Fan	Water

7.3 Preliminary Experiments

Initially some preliminary experiments are conducted to decide the percentage foam that needs to be mixed with base resin LLDPE and to understand the rotomouldability of LLDPE foam mixer. In order to know the processing characteristics of the LLDPE foam mixer, three samples of LLDPE foam mixer (2, 4, 6, 8 & 10 in percentage) is tested and its melt flow property is recorded. During the experimental runs it is observed that the maximum melt flow index is obtained for the foam percentage of 6 when mixed with the base resin LLDPE. The above situation is clearly evident from the Fig 7.1.

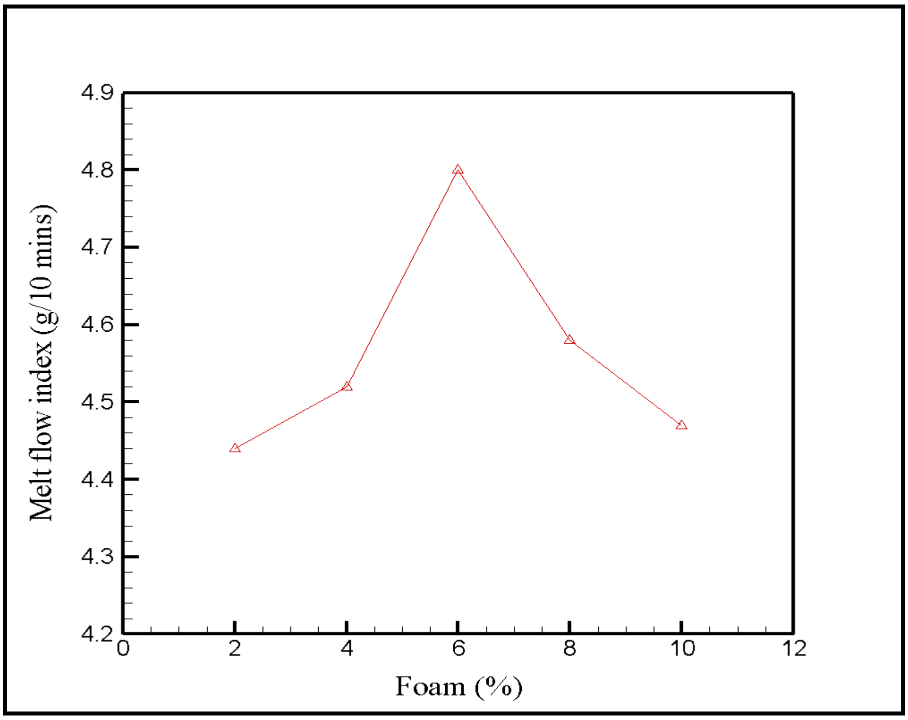


Fig 7.1 Variation of melt flow index with respect to percentage of foam in LLDPE

Further to decide the percentage that needs to be mixed with base resin LLDPE, three samples of LLDPE foam mixer (2, 4, 6, 8 & 10%) is tested for its impact strength. From the experiments it is noticed that maximum impact strength is obtained for the foam percentage of 6 when mixed with the base resin LLDPE. The above situation is clearly evident from the Fig 7.2.

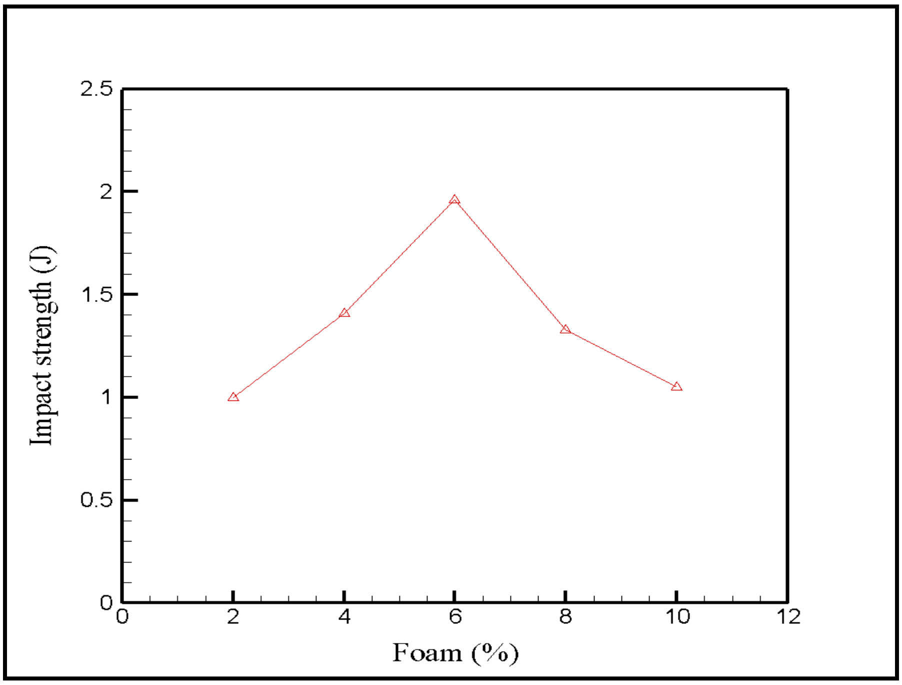


Fig 7.2 Variation of impact strength with respect to percentage of foam in LLDPE

Few preliminary expriments are also conducted for fixing up the range of process parameters which influences the impact strength. From the preliminary experiments it is noted that for oven temperature lower than 210^{o}C, the products obtained are undercooked because of incomplete fusion of powder particles. On the other side for oven temperature higher than 230^{o}C polymer degradation is observed. Identical observations are noted for the oven residence time when it is below 32 minutes and above 42 minutes.

From the above preliminary experiments it is predicted that 6% of foam is the optimum level that needs to be mixed with the base resin LLDPE to obtain sufficient melt flow for ease of processing and better impact strength. Also minimum and maximum level of temperature and oven residence time can be considered as 210, 230°C and 32, 42 minutes.

7.4 Results and Discussions

7.4.1 Statistical Inference

The impact strength values (in Joules) obtained from the experiments as per the design scheme considered is shown in Table 7.3. From the experimental value the mean impact strength is found as 2.19 J with a standard deviation of 0.151 J indicating wide process variability.

Table 7.3 Plan of experiments and corresponding value of impact strength

Run Order	X1	X2	X3	Impact strength (J)
1	1	0	-1	2.1
2	-1	1	0	2.11
3	-1	-1	0	2
4	0	0	0	2.34
5	0	-1	-1	1.968
6	0	1	-1	2.13
7	1	0	1	2.42
8	-1	0	1	2.22
9	0	0	0	2.4
10	0	0	0	2.32
11	1	-1	0	2.11
12	0	-1	1	2.34
13	0	1	1	2.32
14	1	1	0	2.21
15	-1	0	-1	1.996

Statistical analysis using analysis of variance (ANOVA) is carried out to know the factors or interactions which are significantly affecting the response. The stated analysis is executed by considering 5% level of significance (p = 0.05). A 'p' value less than 0.05 can be deemed as significant. This is performed on the grounds that at 95% level of confidence, it rejects the null hypotheses that the factors have no effect on impact strength (against an alternative hypothesis that the factors have significant influence on impact strength).

The results obtained from statistical analysis based on ANOVA along with 't' test is shown in Table 7.4, it is observed from the result that the process parameters have significant linear as well as non-linear (quadratic) effects of process parameters on the impact strength.

Table 7.4 Results of ANOVA for impact strength

Term	Coefficient	Standard error Coefficient	T= Coefficient/ Standard error Coefficient	*p*
Constant	2.35333	0.01860	126.520	0.000
X_1	0.06425	0.01139	5.641	0.002*
X_2	0.04400	0.01139	3.863	0.012*
X_3	0.13825	0.01139	12.137	0.000*
X_1^2	-0.12567	0.01677	-7.495	0.001*
X_2^2	-0.12017	0.01677	-7.167	0.001*
X_3^2	-0.04367	0.01677	-2.604	0.04*
X_1X_2	-0.00250	0.01611	-0.155	0.883
X_1X_3	0.02400	0.01611	1.490	0.196
X_2X_3	-0.04550	0.01611	-2.825	0.037
*Significant at 5% level of significance.				

The coefficients of the regression model |β|**,** which in general can be written as shown in Eq. (7.1).

$$y = X\,\beta + \epsilon \qquad (7.1)$$

where

$$[y] = \begin{bmatrix} y_1 \\ y_1 \\ \cdot \\ \cdot \\ y_n \end{bmatrix} \quad [x] = \begin{bmatrix} 1 & x_{11} & x_{12} & .. & x_{1k} \\ 1 & x_{21} & x_{22} & & x_{2k} \\ . & . & . & .. & . \\ . & . & . & .. & . \\ . & . & . & .. & . \\ 1 & x_{n1} & x_{n2} & .. & x_{nk} \end{bmatrix} \quad [\beta] = \begin{bmatrix} \beta_1 \\ \beta_2 \\ . \\ . \\ . \\ \beta_n \end{bmatrix} \quad [\epsilon] = \begin{bmatrix} \varepsilon_1 \\ \varepsilon_2 \\ . \\ . \\ . \\ \varepsilon_n \end{bmatrix}$$

Here, $[y]$ is a $(n \times 1)$ vector of the observations, $[X]$ is a $(n \times j)$ matrix of the levels of the independent variables, $[\beta]$ is a $(n \times 1)$ vector of the regression coefficients and $[\epsilon]$ is a $(n \times 1)$ vector of random errors. The procedures of obtaining these constants have been discussed at sufficient length in Montgomery (2012). Refer Appendix B for further details and illustrative example (Waigaonkar, 2010 and Waigaonkar et al., 2011). The empirical model for impact strength in terms of coded units considering only significant terms from ANOVA was obtained as:

$$\text{Impact strength} = 2.353 + 0.064\, X_1 + 0.044\, X_2 + 0.138\, X_3 - 0.125\, X_1^2 - 0.120\, X_2^2 - 0.043\, X_3^2 - 0.00250\, X_1 X_2 + 0.02400\, X_1 X_3 - 0.04550\, X_2 X_3 \quad (7.2)$$

Eq. (7.2) can be comfortably used to estimate the impact strength for any combination of process parameters within the regime of experimentation (i.e., –1 to +1).

7.4.2 Effect of Oven Temperature and Oven Residence Time on Impact Strength

Using Minitab 15.0 software, contour plots and 3D graphs for the effect of process parameters on impact strength are generated in order to find the responsible parameters or combinations of these. A contour plot is a graphical technique used for representing a three dimensional surface by plotting constant z slices called contours on a 2-dimensional format. A surface graph provides a three dimensional view, which gives a clear picture of the response surface (Phaneendra Kiran and Shibu, 2013).

The contour and surface plots for combinations of process parameters are as shown in Fig 7.3 & 7.4. Fig 7.3 & 7.4 shows the variation of impact strength with respect to change in oven residence time and temperature. As it is evident from the Fig 7.3 & 7.4 that the impact strength gradually increases as the oven temperature and time is increased from their least value. This can be attributed to two different facts. First, higher oven temperature reduces the viscosity of polymer making it easier for bubbles to diffuse from the material. With increase in oven residence time and temperature, the polymer will be in molten state for a long period, allowing more bubbles to diffuse. This reduces the bubble diameter thereby increasing the relevant part density and improving the impact strength. Identical observation of improved impact strength with increase in part density is evident in the past literature by S.B Tan et al., (2011).

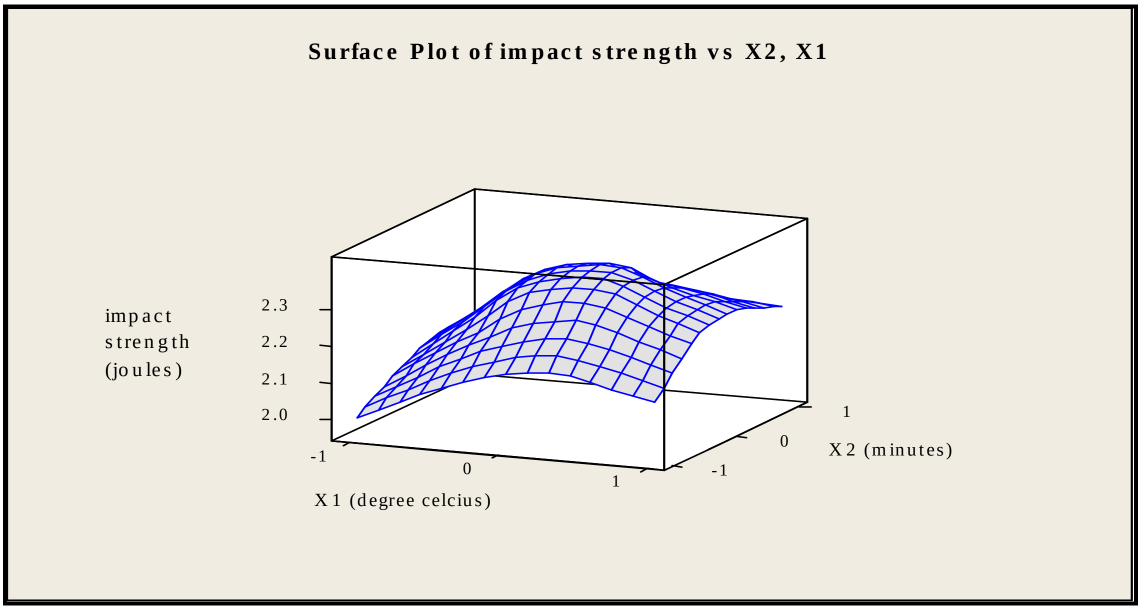


Fig 7.3 Surface plot of impact strength versus oven residence time and temperature

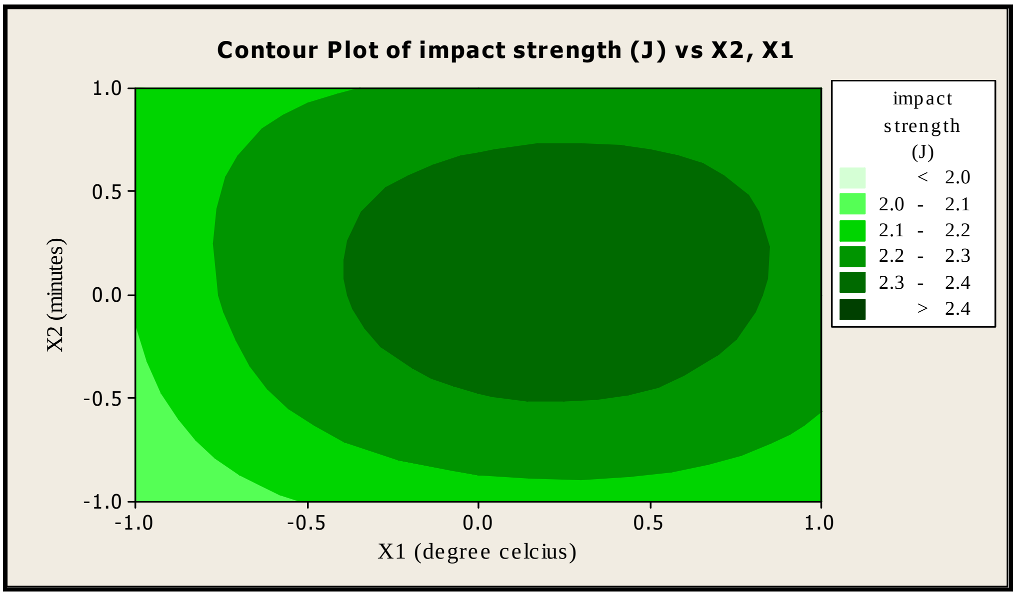


Fig 7.4 Contour plot of impact strength versus oven residence time and temperature

Second, improvement of impact strength can also be due to the thicker walls of foamed samples which are concentrated by a longer oven residence time.

Further increase in temperature and oven residence time leads to cell coarsening. When two cells of different size are adjacent, the gas will diffuse from the smaller cell to the larger one because of the pressure difference in the cells (Vahid and Klempner, 1991, Remon et al., 2008). As a consequence, larger cell will get larger, and finally the two cells will become one large cell. When this cell coarsening occurs, the cell-population density is deteriorated which is accompanied with degradation of polymer foam mixture and leads to impact strength reduction. Therefore, highest strength is perceived when the material is homogenous half way between these two parameter levels. The characteristics of the lump in the curve can be interpreted with this aspect.

7.4.3 Effect of Cooling Medium and Oven Temperature on Impact Strength

As it can be observed from Fig 7.5 and 7.6 that for a given set of oven temperature rapid cooling (water cooling) results in better impact strength than slow (still air) and medium cooling (forced air).

In faster cooling, polymer melt passes through the maximum crystalline temperature very quickly, leaving most of the molecular chains in amorphous form. This produces finer cells with uniform structure. As amorphous polymers are found to be highly irregular and have entangled pattern of polymer chains, it is tough for the crack to propagate after impact. In contrast, slow and medium cooling produces crystalline structure with larger and denser spherulites and the polymer chains are exposed near the maximum crystalline temperature for a long period. Thus, slower cooling rate assist the development of crystalline regions with regular structure of molecular chains and hence reduced impact strength. Identical observation of formation of crystalline structure and larger spherulites caused by slow cooling is evident in the past literature by S.B Tan et al., (2012). Similar trend of increase in impact strength with rapid cooling and reduction in impact strength with low and medium cooling is also observed in the past by Crawford (2000) for rotomoulded parts.

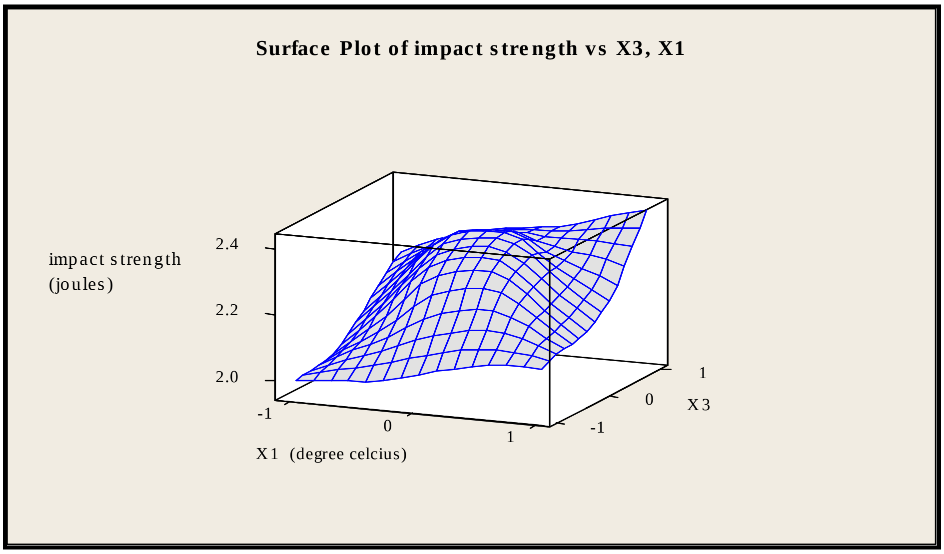


Fig 7.5 Surface plot of impact strength vs. cooling medium, temperature

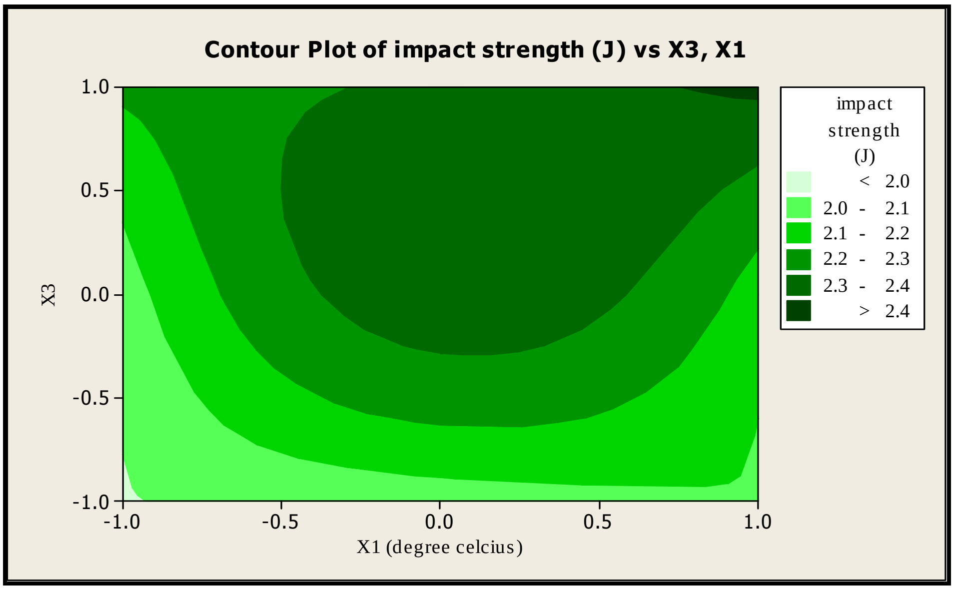


Fig 7.6 Contour plot of impact strength vs. cooling medium, temperature

7.4.4 Statistical Optimization

Optimization is performed using 'D' (determinant) optimal design. MINITAB 15.0 is used for this optimization. Fig 7.7 shows MINITAB output, the number shown at the top of the window refers to the highest (Hi) and lowest (Lo) level of the process parameters considered for the experimentation. The values shown at the middle in red colour are the current optimized value (Cur) of the process parameters. The optimum values of process parameters, X_1, X_2 and X_3 in coded form are found as 0.3458, –0.0763 and 1.0000, respectively. Predicted response Y for the optimized (Cur) factor setting is 2.46 J which is closer to target of 2.50 J indicating a desirability of 0.96289. 'D' optimal values of 0.96289 (very close to 1) indicating a proper convergence to optimal solution. Each cell of the graph in Fig 7.7 shows how the response changes as a function of one of the factor while other factors remain fixed and horizontal blue line represents the level of optimized value.

The relationship between the coded (X_c) and a real variable (X_r) is given in Eq. (7.3):

$$X_c = \frac{X_r - \left(\frac{X_{max} + X_{min}}{2}\right)}{\left(\frac{X_{max} - X_{min}}{2}\right)} \tag{7.3}$$

Where X_{max} & X_{min} are lowest and highest values of the corresponding process parameter respectively. Using Eq. (7.3), the actual values are interpreted as: X_1 = 223.45°C, X_2 = 36.6 minutes, and X_3 = water.

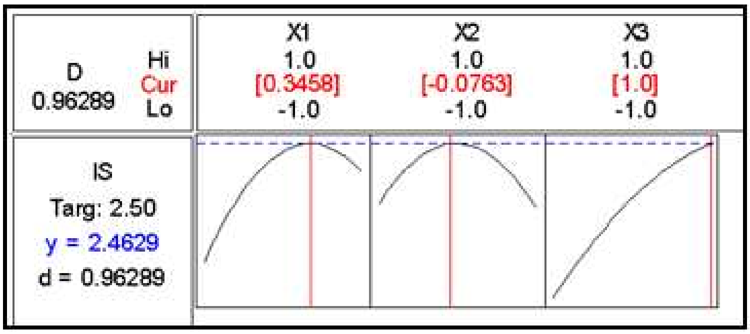


Fig 7.7 Results of D optimality test

7.5 Confirmatory Experiments

In order to verify the above results, new set of process parameters are used to carry out the confirmatory experiments. Predicted and experimental results are shown in Table 7.5. The values pertaining to predicted and experimental results are closer to each other. A minor variation in them could be because of the error [Є in 'equation (7.1)'], prompted by the aspects like change in atmospheric temperatures, humidity, etc., while performing the experiments. The mean value of impact strength is obtained as 2.36 J. Thus, above process parameter setting can be suggested to have improved impact strength.

Table 7.5 Confirmatory experiments to validate optimization of rotational moulding process

Run order	X1	X2	X3	Impact strength (J)			% Error with average
				Predicted	Experimental	Average	
1	0.3458	-0.0768	1	2.46	2.39	2.39	2.5
2	0.3458	-0.0768	1	2.46	2.46		
3	0.3458	-0.0768	1	2.46	2.34		

7.6 Summary

In this work, a detailed investigation is performed to identify the optimum process parameters yielding maximum impact strength of foamed rotationally moulded LLDPE product. Box-Behenken designs of RSM are applied to plan and analyze the experiments. The findings can be compiled as follows:

1. It is observed that impact strength is enhanced with increase in oven temperature and time in rotational moulding process. The improvement is due to the diffusion of bubbles in polymer melt. However, further increase in these parameters decreased the impact strength due to the initiation of polymer degradation and cell coarsening.

2. Impact strength of foamed rotational moulding products are found to be improved with faster cooling aids. Faster cooling develops highly irregular and complex pattern of polymer chains in amorphous region which arrests the crack propagation.

3. A statistical optimization is performed using 'D' optimal design criterion and the optimal process parameters are identified to attain adequate impact strength. These are: Oven temperature = 223.45°C, Oven time = 36.61 minutes, and water shower as cooling medium. The above set of process parameters yielded a theoretical estimated value of impact strength as 2.45 J.

4. New set of process parameters (*X*1, *X*2, and *X*3) are used to perform the Confirmatory experiments. The values of impact strength are found to be closer to the target (2.39 J).

It should be stated that the above results are acceptable for existing experimental facilities. It is common to have thickness up to 10 mm in rotational moulding process in an industrial environment. Therefore, aspects like residual stress, warpage and shrinkage needs to be equally considered for experimental investigations.

CHAPTER 8

FRACTURE CHARACTERIZATION OF ROTOMOULDED PRODUCTS

Introduction

Before the development of fracture mechanics, yield strength or ultimate tensile strength with a suitable safety factor were the conventional design criteria. Fracture mechanics addresses the situation where the presence of a flaw in the material causes fracture or failure when the conventional design criteria would deem the component as safe (Ming Luen Lu *et al.,* 2004). Since parts made of LLDPE using rotational moulding process find applications like overhead chemical storage tanks, automobile components, oil tanks etc., Fracture toughness of such products is considered as one of the essential quality feature. In this chapter, an attempt has been made to investigate the effect of process parameters and to identify the optimum process parameter yielding superior fracture toughness of the rotationally moulded products. Experimental investigations are carried out by planning and performing trials based on design of experiments (DOE). DOE approach, called response surface method (RSM) is used for data analysis.

8.1 Fracture Characteristics of Polymers

Linear elastic fracture mechanics originally developed for brittle materials has been used to determine fracture in many polymers. Polymer as engineering materials, is gaining importance in high demanding structural applications (Ljungberg, 2003). Polyethylene in particular used in many applications like water and natural gas pipes (Fabiano *et al.*, 2013, Jannson 2003 and Mills, 1993), fuel, oil and chemical tanks which require strength, toughness and wear resistance. Failure in such situations may lead to severe accidents, hence a deep understanding and evaluation of fracture process in this material is advisable.

The assessment of fracture toughness of ductile polymers by concepts of fracture mechanics is a great challenge. Linear elastic fracture mechanics (LEFM) fails to provide us with proper fracture toughness values for ductile polymers due to plasticity ahead of crack tip.

In order to overcome this difficulty, methods of the non-linear fracture mechanics (denoted also as ductile, elastoplastic or post-yield fracture mechanics) have gained considerable attention. Although several approaches of the latter are proposed to consider the large-scale plastic deformation during loading, the widely used method is *J*-integral (Kolednik *et al* 2014, Ochensberger and Kolednik 2014, Tamas *et al* 2003, Wang *et al* 1992, Paton and Hashemi 1992, Mai and Powell, 1991 and Williams 1987). *J* integral approach is proposed by Rice, (1968) as a two dimensional energy line integral that can be used as an analytical tool to characterize the crack tip stress and strain field under both elastic and plastic stress and strain. In *J*-integral technique, the critical value under mode-I deformation, J_{Ic}, is defined by the intercept of the blunting line or it's offset with the *J* versus Δa (*J*-*R*) curve, where Δa designates the crack growth (Prashant kumar, 2011).

8.2 Fracture Characteristics of Rotomoulded Products

8.2.1 Plan of Experiments

Proper control of the process parameters are required to obtain a quality product. The important process parameters that govern the fracture properties of rotomoulded products are:

1. Oven temperature (X_1)
2. Oven time (X_2)
3. Cooling media (X_3)

To plan the experiments, the response surface method (RSM) is used. RSM is a collection of statistical and mathematical methods that are useful for modeling engineering problems. The main objective of this technique is to optimize the response surface that is influenced by various process parameters. RSM quantifies the relation between the controllable parameters and the response (Phaneendra Kiran and Shibu, 2013 and Montgomery, 2012). In this work, three parameters with three levels are considered, a full factorial scheme would have resulted in 27 (3^3) experiments. Since, performing experiments based on full factorial design requires substantial amount of resources and time, the experiments are planed using Box- Behenken design which is subcategory of RSM (Box- Behenken, 1960). In this design the coded variables (-1, +1) are used to develop a first order model where as the center points (0, 0) estimates the second order model with curvature effect. The considered design scheme has only 15 experimental trails, yet provides a good assessment of the response (fracture toughness). Thus three levels of each process parameter coded as (-1, 0, +1) are identified and shown in Table 8.1.

Table 8.1 Process parameters and their levels for rotational moulding

Process parameter	**Unit**	**Designation**	**Lowest level (-1)**	**Middle level (0)**	**Highest level (+1)**
Oven temperature	^{o}C	X_1	210	220	230
Cycle time	Minutes	X_2	32	37	42
Cooling medium		X_3	Still air	Fan	Water shower

8.2.2 Experimental Details

Experimental details are summarized in the Table 8.2 shown below. The details of the same have been discussed in Chapter-3.

Table 8.2 Experimental details

Rotational moulding machine specifications	Model: Clamshell type single arm biaxial machine
Mould specifications	Material : Stainless steel
	Shape: Square cross section
	Release agent: Metrork silicone 17 compound Dimensions: Length: 100 mm Breath: 100 mm Height: 100 mm
Raw material specifications	Material: R350 A 42 LLDPE supplied by GAIL India
	MFI: 4.2 g/10 min

	Density: 935 kg/m^3
	Shot weight: 0.6 kg
Testing Equipment	Tensile Machine: universal testing machine load cell : 5 kN cross head speed of 1 mm/min

8.2.3 Fracture Test

The procedure involves measurement of area under the load versus load–line displacement curve to determine the fracture toughness value (J_{IC}). Fracture tests are carried out on a compact tension test specimens prepared as per the ASTM standard D6068 as shown in Fig 8.1. Specimens are precracked using sharp steel blade of thickness 0.1 mm. The tests are performed on a universal testing machine at a constant cross head speed of 1 mm/min at room temperature. The crack initiation is closely observed with the help of a magnification lens and the load versus load-line displacement are measured and recorded for each specimen.

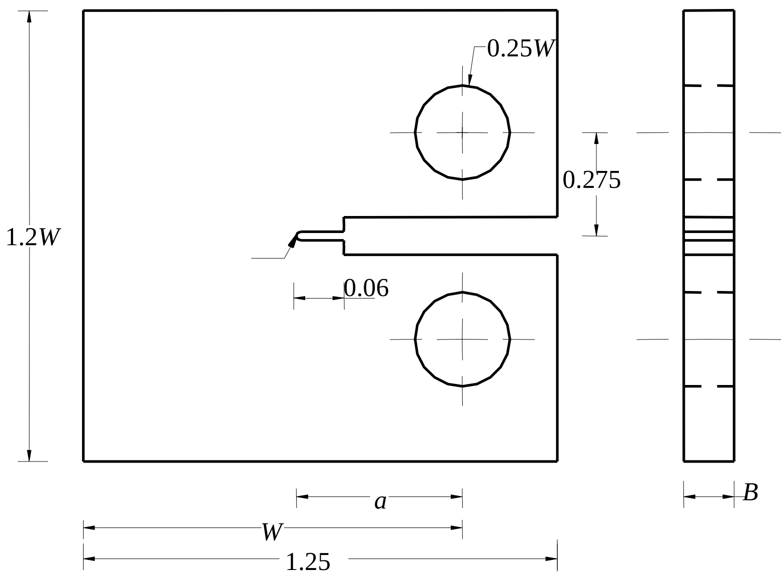


Fig 8.1 Compact tension test specimen as per ASTM D 6068 (2010)

8.2.4 Evaluation of Fracture Toughness

J integral is originally defined as a path independent line integral for two dimensional problems. ASTM D6068 (2010) outlines a test method for estimating *J* integral for a compact tension test specimen. According to ASTM D6068 (2010), *J* integral is given by Eq. (8.1)

$$J = J_e + J_p \tag{8.1}$$

Here J_e & J_p are the elastic and plastic components of total *J* value given by the Eq. (8.2)

$$J_e = \eta_e \, U_e / \, B(W - a)$$

$$J_p = \eta_p \, U_p / \, B(W - a) \tag{8.2}$$

Were U_e & U_p are the elastic and plastic components of the total energy. η_e & η_p are their corresponding work factors. $(W - a)$ is the unbroken ligament length. η_e & η_p is equal to $(2 + 0.522(W - a/\, W))$ and $U_e + U_p = U$. (where *U* is the total area under the load vs load line displacement curve). Therefore, equation is reduced to (Hashemi & Williams, 1986 & ASTM D6068, 2010)

$$J = \eta \, U \, / \, B(W - a) \tag{8.3}$$

In accordance with the recommended procedure for establishing J_{IC}, *J-R* curve method is proposed to characterize the fracture toughness behaviour of rotomoulded product made using LLDPE for various process parameters. The value of *J* for six crack increment is determined using Eq. (8.3) and plotted against crack extension. A straight line known as R line is best fitted through the *J* points. A blunt line $J = (\sigma_{ys} + \sigma_{us})\Delta a$ is drawn whose intersection with R line gives J_{IC}. During the initial loading of a precracked specimen, because of the intense plastic deformation the crack tip blunts before the stable crack growth initiates. Blunting can be considered as a small crack growth as shown in Fig 8.2. For estimating small crack growth, the blunt crack can be modeled as a semicircle of radius, half of the crack tip opening displacement ie., (*CTOD*)/2. The *CTOD* is then related to *J* by the following relationship.

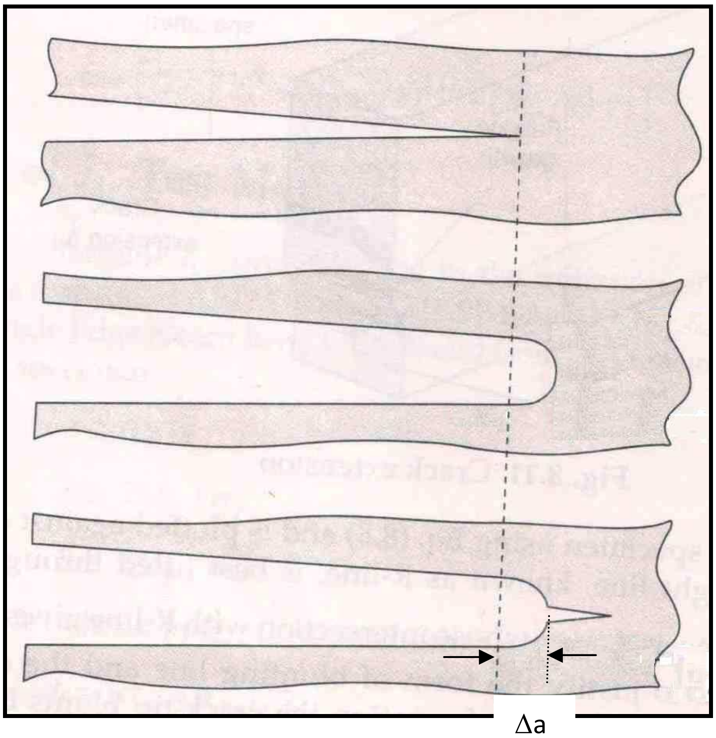
$$CTOD = J/\sigma_f \tag{8.4}$$

The small crack growth due to blunting is $J/2\sigma_f$

$$J = 2\sigma_f \Delta a \qquad (8.5)$$

where σ_f is a flow stress and is given by $(\sigma_{ys} + \sigma_{us})/2$

$$J = (\sigma_{ys} + \sigma_{us})\Delta a \qquad (8.6)$$



(a) Before loading

(b) Blunting stage

(c) Crack initiation

Fig 8.2 Finding *J* integral by *J-R* curve method (Prashant Kumar, 2011)

Series of experiments are conducted to investigate the fracture toughness characteristics of rotomoulded products made using LLDPE by changing the process parameters. Representative load verses load line displacement curve for one set of process parameters (210^{o}C temperature, 37 mins as oven residence time and Still air as cooling medium) is reported in Fig 8.3, while the curves for other process parameters are depicted in Appendix C; (Fig 1-14). The value of *J* is determined from the area under its load versus load-line displacement curve and the relationship given by Eq. (8.3). By using the above stated procedure the *J-Δa* curve as shown in

Fig 8.4 [for a set of process parameter (210-37-SA), while the curves for other process parameters are depicted in Appendix C; (Fig 15-28)] is then constructed and value of J_{IC} is determined for each specimen.

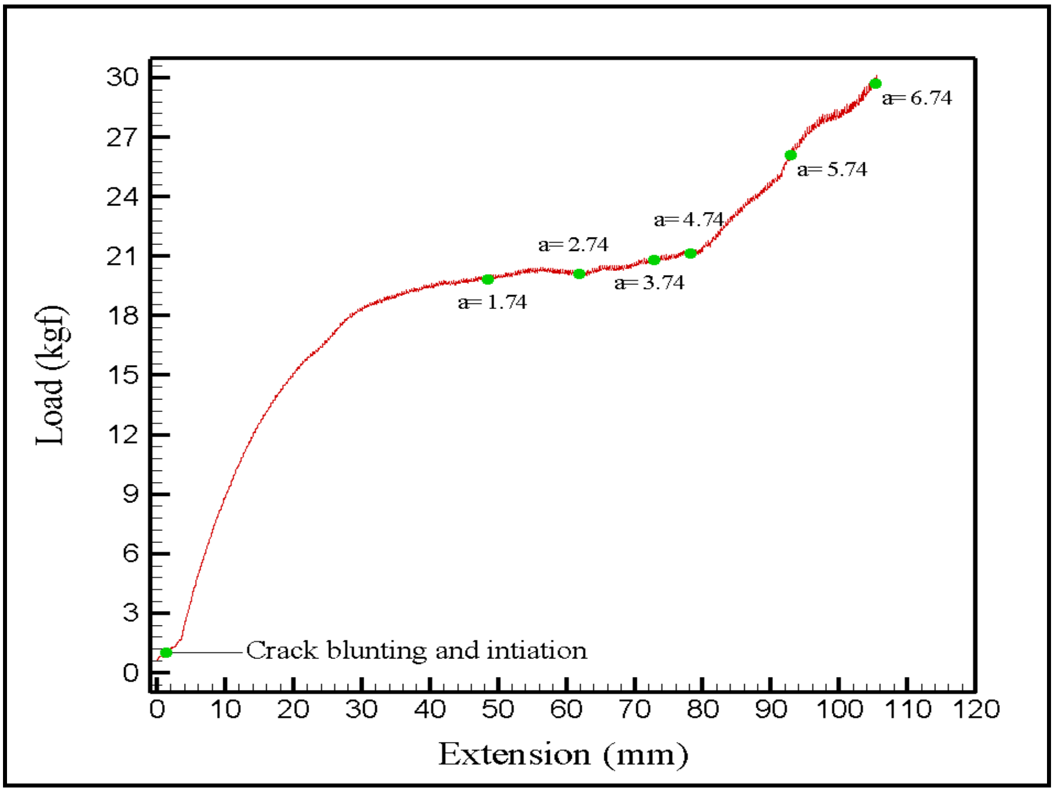


Fig 8.3 Load Verses load line displacement curve for process parameters (210-37-SA)

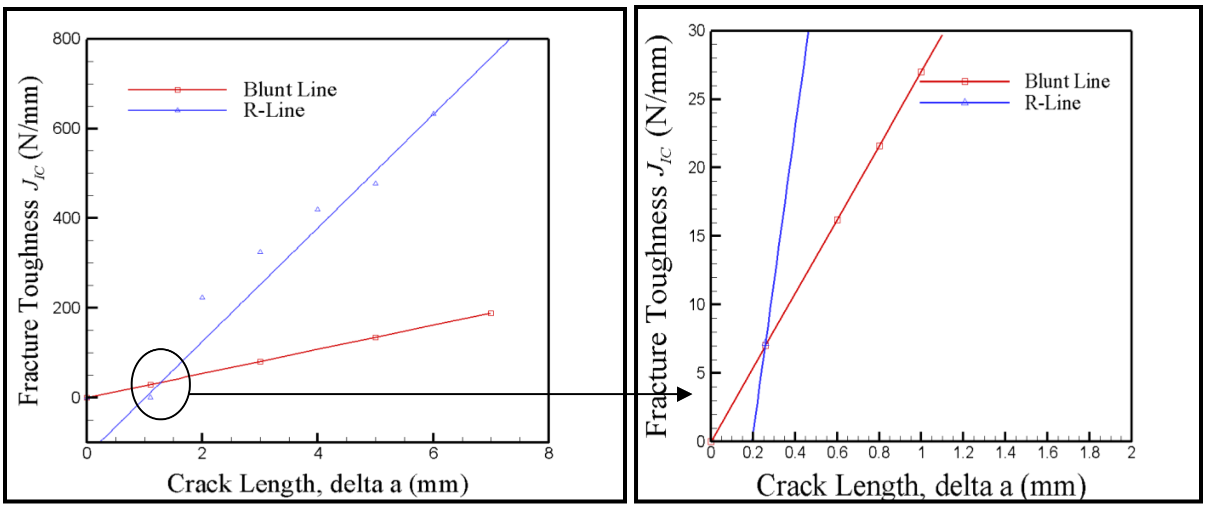


Fig 8.4 *J-R* curve for process parameter (210-37-SA)

When a tensile force is applied to a specimen, elastic deformation takes place; in that, polymer chains are elongated along the direction of the stress. From the load versus load line displacement curve it is evident that, the increase in load immediately after the test started, indicates the formation of stable crack tip blunting followed by initiation as shown in Fig 8.5.

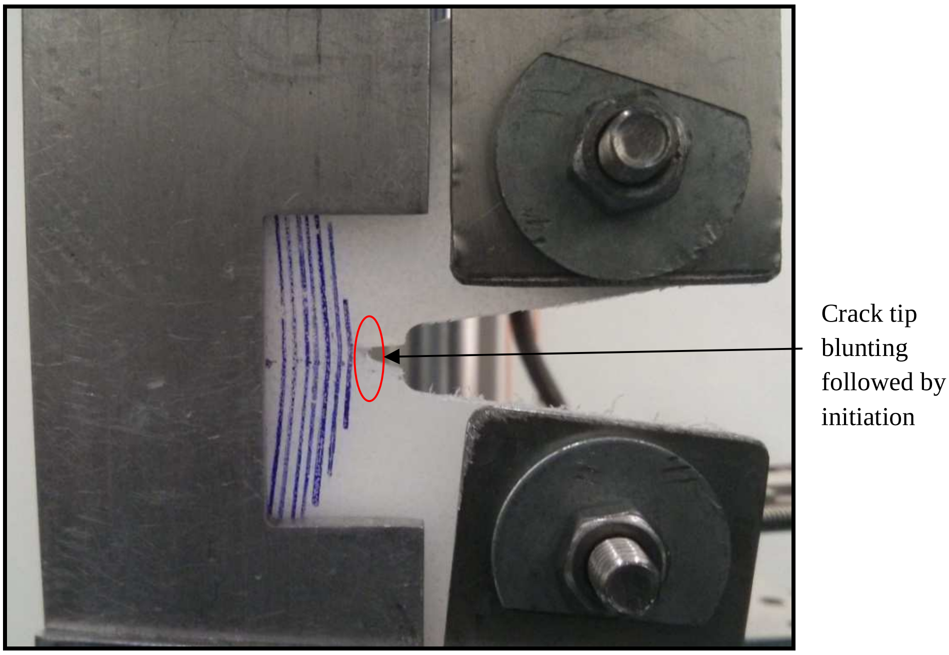


Fig 8.5 Crack tip blunting and initiation

Further increase in load, forces the polymer to release its energy. One of the important energy release mechanism in polymer deformation is crazing. A craze can be considered as a micro crack bridged by multiple, highly-oriented polymer fibrils. Crazes are typically initiated at sites with defects or molecular inhomogeneities. First, micro voids nucleate at the defects under a tensile stress. Then, the micro voids grow in a plane perpendicular to the maximum principal stress, which is a cavitations process. Next, instead of coalescing and forming a crack, these micro voids are stabilized by the surrounding highly-oriented polymer fibrils spanning the craze (Myer Kutz, 2013). The fibrils inside the craze zone are shown in Fig 8.6.

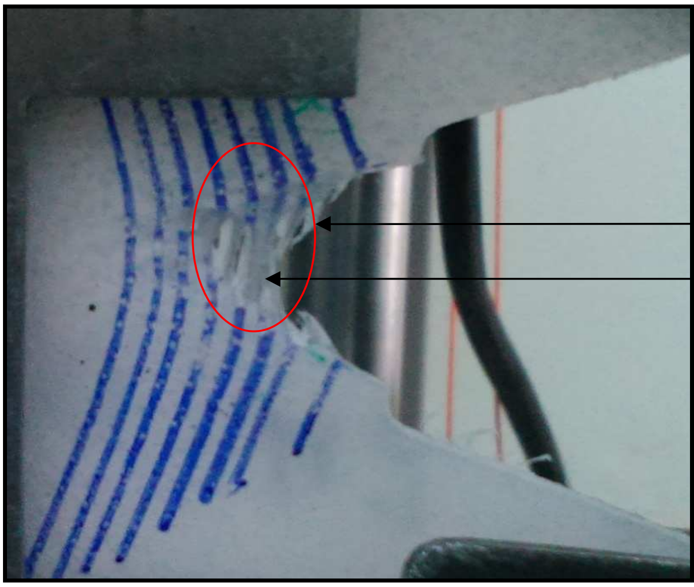


Fig 8.6 Polymer fibrils bridging in the craze zone

8.3 Statistical Inference

Fracture toughness values obtained from the experiments as per the design scheme is as shown in Table 8.3. From the experimental value the mean fracture toughness is found as 18.6 N/mm with a standard deviation of 0.140 N/mm indicating wide process variability. Statistical analysis using analysis of variance (ANOVA) is carried out to know the factors or interactions which are significantly affecting the response. The stated analysis is executed by considering 5% level of significance (p = 0.05). A 'p' value less than 0.05 can be deemed as significant. This is performed on the grounds that at 95% level of confidence, it rejects the null hypotheses that the factors have no effect on fracture toughness (against an alternative hypothesis that the factors have significant influence on fracture toughness).

Table 8.3 Plan of experiments and corresponding value of fracture toughness

Run order	Oven Temperature in ^{o}C (X_1)	Oven Residence Time in min (X_2)	Cooling Medium (X_3)	Fracture Toughness in N/mm (J_{IC})
1	230	37	1	19.09
2	210	42	0	11.07

3	230	42	0	39.20
4	220	42	-1	17.38
5	220	37	0	6.93
6	220	37	0	6.64
7	210	37	1	17.60
8	210	37	-1	7.22
9	230	32	0	13.36
10	220	32	1	26.59
11	230	37	-1	27.81
12	210	32	0	29.89
13	220	32	-1	23.32
14	220	42	1	28.67
15	220	37	0	5.52

The results obtained from statistical analysis based on ANOVA along with‘t’ test is shown in Table 8.4. It is observed from the result that the process parameters have significant linear as well as non-linear (quadratic) effects of process parameters on the impact strength. The coefficients of the regression model |β|, which in general can be written as shown in Eq. (8.7).

$$y = X\,\beta + \epsilon \qquad (8.7)$$

where

$$[y] = \begin{bmatrix} y_1 \\ y_1 \\ . \\ . \\ y_n \end{bmatrix} \quad [X] = \begin{bmatrix} 1 & x_{11} & x_{12} & .. & x_{1k} \\ 1 & x_{21} & x_{22} & & x_{2k} \\ . & . & . & .. & . \\ . & . & . & .. & . \\ . & . & . & .. & . \\ 1 & x_{n1} & x_{n2} & .. & x_{nk} \end{bmatrix} \quad [\beta] = \begin{bmatrix} \beta_1 \\ \beta_2 \\ . \\ . \\ . \\ \beta_n \end{bmatrix} \quad [\epsilon] = \begin{bmatrix} \varepsilon_1 \\ \varepsilon_2 \\ . \\ . \\ . \\ \varepsilon_n \end{bmatrix}$$

Table 8.4 Results of ANOVA for fracture toughness

Term constant	Coefficient	Standard error Coefficient	T= Coefficient/ Standard error Coefficient	*p*
Constant	6.3633	1.834	3.47	0.018*
X_1	4.21	1.123	3.749	0.013*
X_2	0.395	1.123	0.352	0.739
X_3	2.0275	1.123	1.806	0.131
$X_1 * X_1$	5.4783	1.653	3.314	0.021*
$X_2 * X_2$	11.538	1.653	6.981	0.001*
$X_3 * X_3$	6.0883	1.653	3.684	0.014*
$X_1 * X_2$	11.165	1.588	7.031	0.001*
$X_1 * X_3$	-4.775	1.588	-3.01	0.03*
$X_2 * X_3$	2.005	1.588	1.263	0.262
*Significant at 5% level of significance.				

Here, [y] is a ($n \times 1$) vector of the observations, [X] is a ($n \times j$) matrix of the levels of the independent variables, [β] is a ($n \times 1$) vector of the regression coefficients and [ϵ] is a ($n \times 1$) vector of random errors. The procedures of obtaining these constants have been discussed at sufficient length in Montgomery (2012). Refer Appendix B for further details and illustrative example (Waigoankar, 2010 and Waigaonkar *et al.*, 2011). The empirical model for fracture toughness in terms of coded units considering only significant terms from ANOVA is obtained as:

$$J_{IC} = 6.36 + 4.21\, X_1 + 0.395\, X_2 + 2.0275\, X_3 + 5.47\, X_1^2 + 11.53\, X_2^2 + 6.08\, X_3^2 + 11.16\, X_1X_2 - 4.77X_1X_3 + 2.005\, X_2X_3 \quad (8.8)$$

Eq. (8.8) can be conveniently used to predict the fracture toughness for any combination of process parameters within the regime of experimentation (i.e., –1 to +1).

8.4 Results & Discussions

8.4.1 Effect of Oven Temperature and Oven Residence Time on Fracture Toughness

Using Minitab 15.0 software, 3D graphs and interaction plots for the effect of process parameters on fracture toughness are generated in order to find the responsible parameters or combinations of these. Fig 8.7 & Fig 8.8 shows interaction plot and surface plot depicting the variation of fracture toughness with respect to change in oven residence time and temperature.

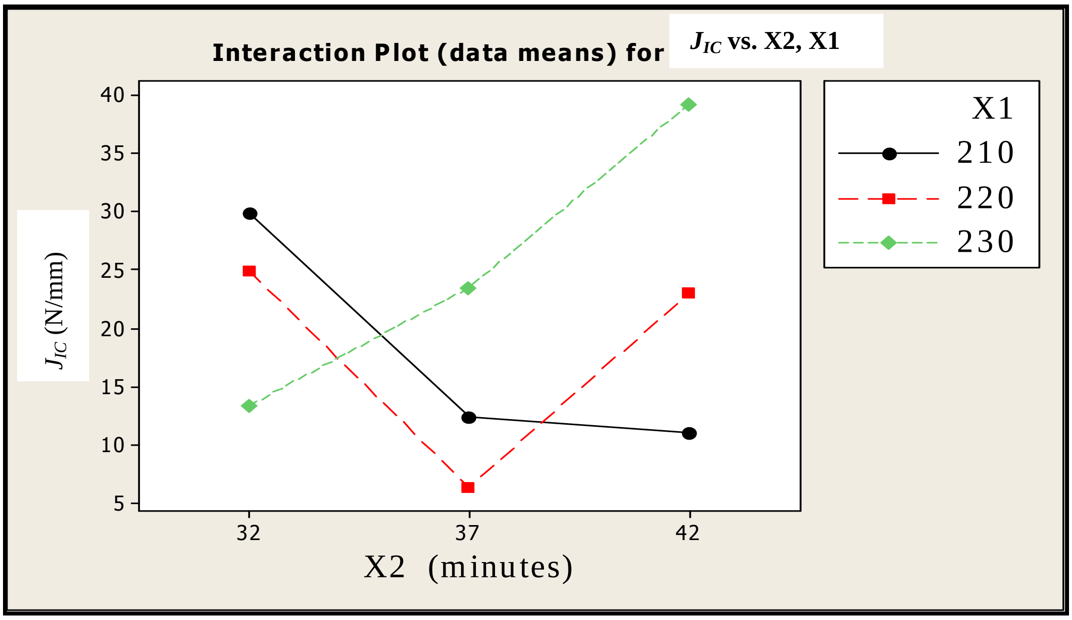


Fig 8.7 Interaction plot showing variation of fracture toughness with respect to change in oven residence time and temperature.

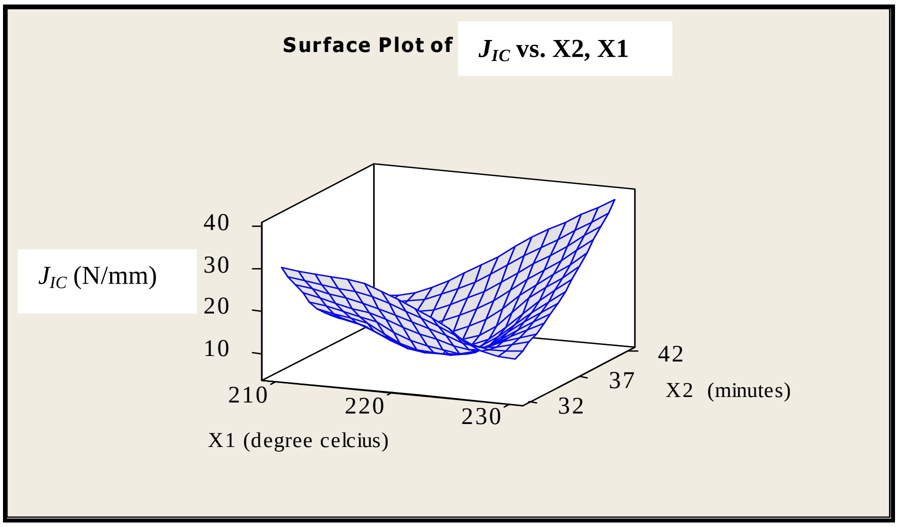


Fig 8.8 Variation of fracture toughness with respect to change in oven residence time and temperature.

It is observed from the surface graph and interaction plots that for the lower temperature of 210^{o}C, the fracture toughness (J_{IC}) of the rotomoulded product decreases as the oven residence time is increased from 32 to 42 minutes. The above situation may be attributed to two different facts; a) the mobility of element taking part in plastic deformation is low at lower temperature and the energy dissipation increases which produce instabilities in the elements. b) The rate of deformation increases drastically in micro cracks which may lead to fracture. Similarly for higher temperature of 230^{o}C, the fracture toughness value increases when oven residence time is increased from 32 to 42 minutes. This increase in fracture toughness is owing to higher mobility of elements and lower energy dissipation which produces stabilities in the elements (Michler and Balta, 2005). For mid range of 220^{o}C the fracture toughness value decreases when oven residence time is increased from 32 to 37 minutes and then it increases there by to a maximum of 23.02 N/mm at 42 minutes. The above observation shows that reduction in fracture toughness by increase in oven residence time up to 37 minutes can be balanced by increasing the temperatures.

8.4.2 Effect of Cooling Medium and Oven Residence Time on Fracture Toughness

For a given setting of oven residence time, it can be seen from the Fig 8.9 & 8.10 that there is a reduction of fracture toughness value, when it is subjected to slow and medium cooling.

However it can also be seen that for rapid cooling in all three cases yields maximum fracture toughness. One possible explanation for this is that the slow cooling gives adequate time for the chains to arrange themselves in crystalline structures. When a tensile stress is applied to a polymer, elastic deformation will take place; in that, the polymer chains will elongate along the direction of the stress. In the initial stage of deformation, the tie molecules in the amorphous regions slip past each other and become extended and aligned in the tensile direction, while the lamellae regions maintain their structures as blocks of folded ribbons. In the next stage, crystalline segments separate from the lamellae and remain attached to each other by tie molecules. Finally, the segments and tie molecules become orientated in the direction of the tensile axis (Myer Kutz, 2013 and Michler and Balta Calleja, 2005). In case of slow cooling sufficient time is available for the molecule to align themselves in crystalline format which results in inferior toughness and higher strength of the product.

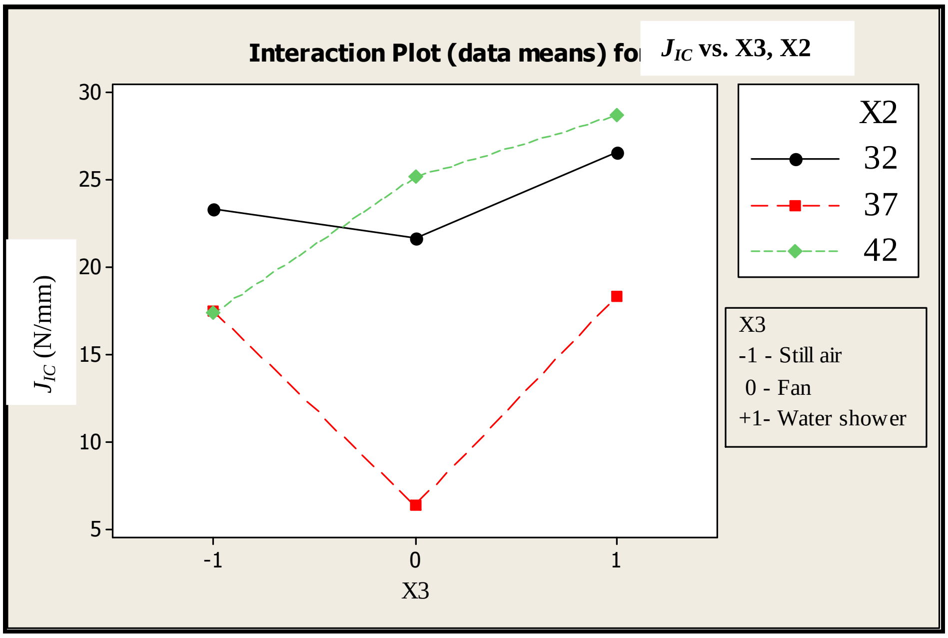


Fig 8.9 Interaction plot showing variation of fracture toughness with respect to change in oven residence time and cooling medium

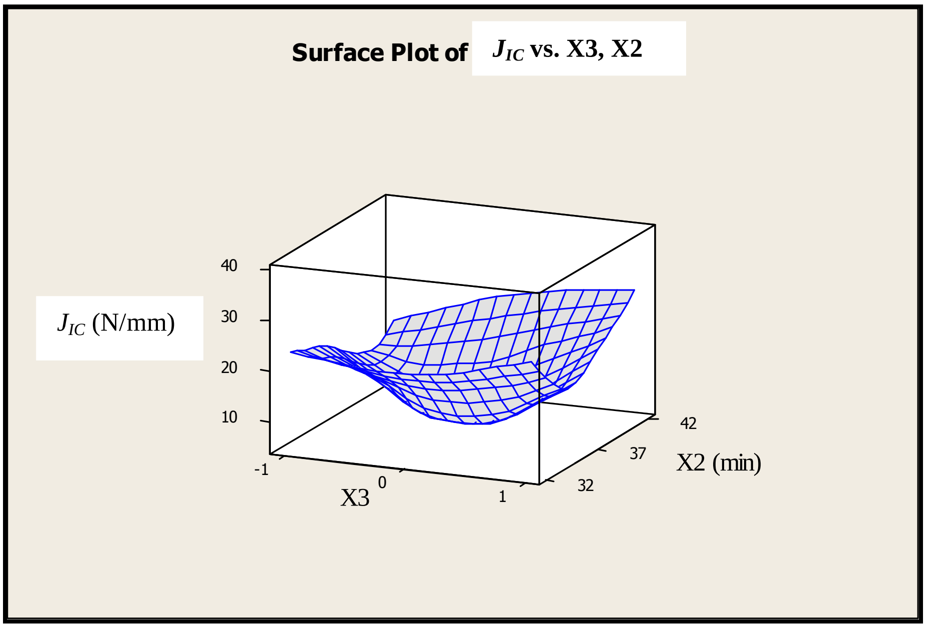


Fig 8.10 Variation of fracture toughness with respect to change in oven residence time and cooling medium

The above observation of crystalline morphology is confirmed by conducting a dynamic mechanical analysis (DMA). DMA is a technique which is widely used to characterize material property as a function of different parameters like temperature, time, atmosphere etc or combination of these parameters. To verify the formation of crystalline morphology for a particular combination of process parameters (220 ^{o}C temperature, 42 mins oven residence time and still air (SA) being slowest cooling method (220-42-SA)), the storage modulus, loss modulus and tan delta is obtained from DMA result. As the polymers are viscoelastic materials, storage modulus measures the stored energy, representing the elastic portion, loss modulus measures the energy dissipated as heat, representing the viscous portion and the ratio of loss to storage modulus is termed as tan delta. Set of process parameters considered (220^{o}C-42-SA) for DMA test yield a higher Storage Modulus (*E'*) of 5264 MPa , loss modulus(E'') of 185.8 MPa at Gama Glass Transition (*T*g) and lower tan delta value of 0.0798 at room temperature as observed from the Fig 8.11. It may be inferred that this condition leads to comparatively more crystalline and less amorphous regions. The slow cooling gives adequate time for the chains to arrange themselves in crystalline structures. In contrast, higher fracture toughness is obtained for faster cooling. This indicates that faster cooling gives less time for the chains to arrange themselves in regular (crystalline) pattern and amorphous regions are comparatively higher. In amorphous state the molecule bypasses melting temperature T_m upon cooling and relative motion of the molecules becomes restricted.

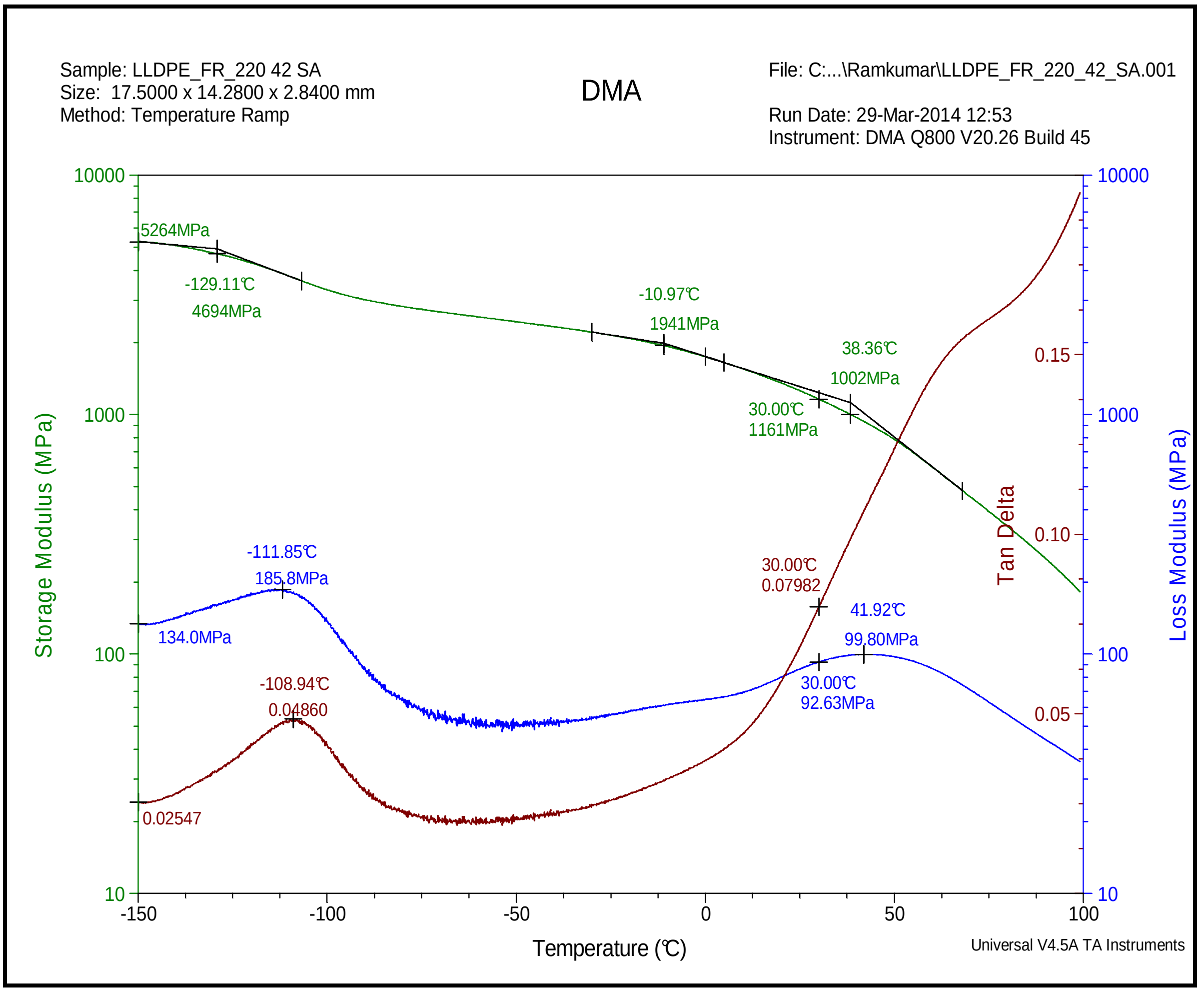


Fig 8.11 Dynamic mechanical analysis for process parameter 220 42 SA

8.4.3 Effect of Cooling Medium and Temperature on Fracture Toughness

Similar argument of formation of amorphous region can be posed for obtaining the maximum fracture toughness value when medium and faster cooling is employed for sample processed at the oven temperature of 210°C. Whereas for the same temperature, considerably lesser toughness value is obtained for slow cooling as shown in Fig 8.12 & 8.13 due the formation of crystalline region and arrangement of chains in the form of crystalline structures.

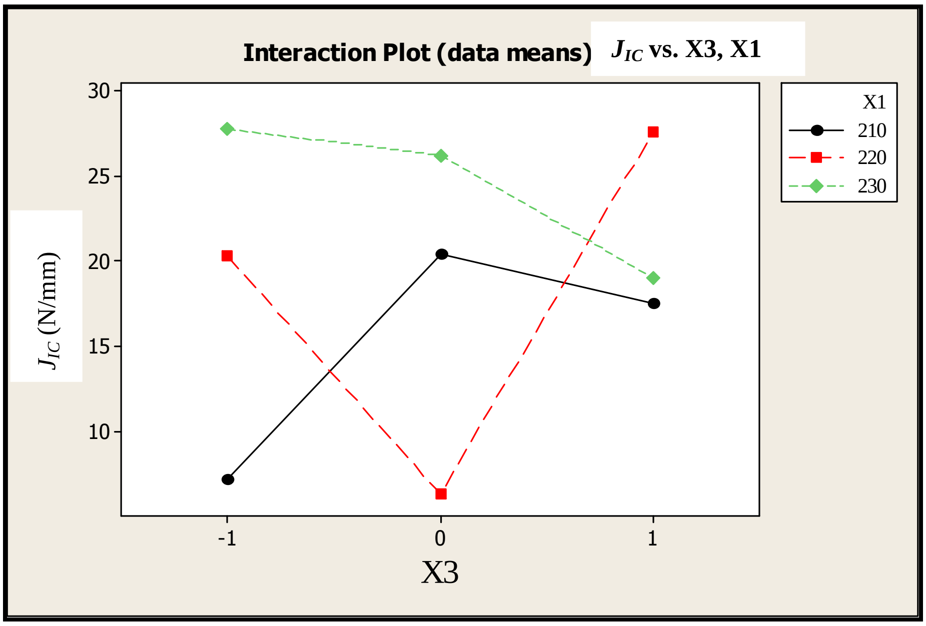


Fig 8.12 Interaction plot showing variation of fracture toughness with respect to change in temperature and cooling medium

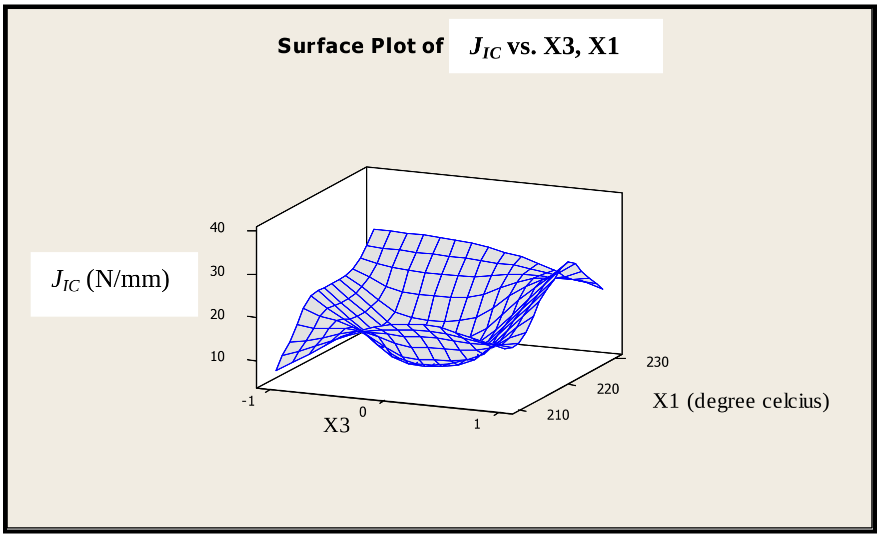


Fig 8.13 Variation of fracture toughness with respect to change in temperature and cooling medium

In contrast, at 220°C the fracture toughness value is lesser on medium (fan) cooling and considerably more at slow and faster cooling. This result clearly shows that the sample shows huge resistance to deformation, thereby reducing its toughness and increasing its strength.

At the temperature of 230°C the fracture toughness value is higher for slower cooling and its lower for the faster cooling. This may be attributed to the fact that, keeping the sample at a higher processing temperature of 230°C in the oven for appreciable amount of time leads to the polymer degradation, resulting in reduction of fracture toughness of the sample.

8.5 Statistical Optimization and Confirmatory Experiments

The optimization is performed using 'D' (determinant) optimal design. MINITAB 15.0 is used for this optimization. Fig 8.14 shows MINITAB output, the number shown at the top of the window refers to the highest (Hi) and lowest (Lo) level of the process parameters considered for the experimentation. The values shown at the middle in red colour are the current optimized value (Cur) of the process parameters. The optimum values of process parameters, X_1, X_2 and X_3 yielding maximum toughness are found as 210 °C, 32 minutes and 1 (water shower) respectively. Predicted response y for the optimized (Cur) factor setting is 39.20 N/mm which is closer to target of 39.20 N/mm indicating a desirability of 1. Each cell of the graph in Fig 8.14 shows how the response changes as a function of one of the factor while other factors remain fixed and horizontal blue line represents the level of optimized value.

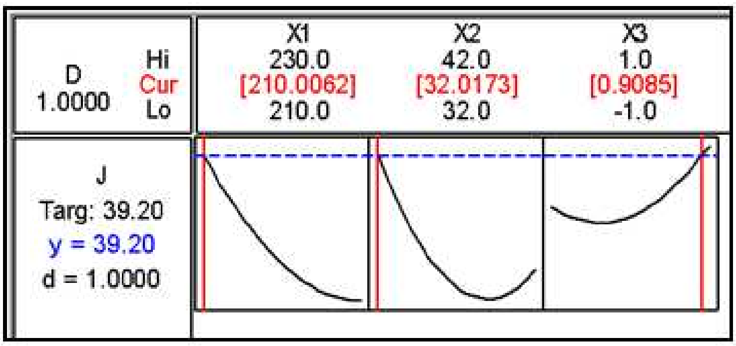


Fig 8.14 Results of D optimality test

In order to verify the above results, new set of process parameters are used to carry out the confirmatory experiments. Predicted and experimental results are shown in Table 8.5. The values pertaining to predicted and experimental results are closer to each other. A minor variation in them could be because of the error [Є in 'equation (8.1)'], prompted by the aspects like change in atmospheric temperatures, humidity, etc., while performing the experiments. The mean value of fracture toughness is obtained as 38 N/mm. Thus, above process parameter setting can be suggested to have improved fracture toughness.

Table 8.5 Confirmatory experiments

Run order	**X1**	**X2**	**X3**	**Fracture Toughness (J_{IC}) in N/mm**			**% Error with average**
				Predicted	Experimental	Average	
1	210	32	1	39.20	37.8	38	2.8
2	210	32	1	39.20	38.1		
3	210	32	1	39.20	38.3		

8.6 Summary

Statistical technique is applied for investigation of fracture toughness of rotational moulded product made using LLDPE. The present investigation yielded optimal level of process parameter for improving the fracture toughness of rotationally moulded LLDPE product. Box-Behenken designs of RSM are applied to plan and analyze the experiments. The findings can be compiled as follows:

1. For the lower temperature of 210^{o}C, the fracture toughness (J_{IC}) of the rotomoulded product decreases as the oven residence time is increased due to lower mobility of element and formation of micro cracks at the crack tip. However, the above situation is vice versa for the higher temperature of 230 ^{o}C.
2. Faster cooling aids improves the fracture toughness in rotational moulding process, as faster cooling develops highly irregular and complex pattern of polymer chains in amorphous region which arrests the crack propagation. Increase in the portion of amorphous material in the structure results in increase in fracture toughness value.
3. Employing slower cooling aids decreases the fracture toughness value in rotational moulding process due to the formation crystalline region. Crystalline morphology is confirmed by conducting a dynamic mechanical analysis for set of process parameters.

The trial 220-42-SA seems to have higher storage modulus (*E'*) of 5264 MPa, loss modulus of 185.8 MPa at gama glass transition (*T*g) and lower tan delta value of 0.0798 confirming more crystallininity and less amorphous regions.

4. A statistical optimization is performed using 'D' optimal design criterion and the optimal process parameters are identified to attain adequate fracture toughness. These are: Oven temperature = 210°C, Oven time = 32 minutes, and water as cooling medium. The above set of process parameters yielded a theoretical estimated value of fracture toughness as 39.2 N/mm.
5. Based on the statistical analysis, equation has been derived to predict the fracture toughness for any combination of process parameters (X_1, X_2, and X_3) within the regime of experimentation (i.e., –1 to +1).
6. New set of process parameters (*X*1, *X*2, and *X*3) are used to perform the Confirmatory experiments. The average value of fracture toughness is found as 38 N/mm, which is found closer to the target value.

CHAPTER 9

CONCLUSIONS

9.1 Summary

Rotational moulding is currently fastest growing sector of plastic processing industry. In order to sustain this growth it is essential that moulders have a better fundamental understanding of the manufacturing process. The research work presented in this thesis is aimed to understand the mechanical and fracture behaviour of rotational moulded products using LLDPE. In order to understand the importance of this study, a simple systematic logical method called objective based MADM for manufacturing process selection is used. This method is proposed to assist a manufacturer to select an appropriate plastic manufacturing process from a long list for a specific application. The proposed method is clearly described considering emerging application of automobile fuel tank. Based on the above mentioned method, blow moulding and rotational moulding process are the two processes which turned evident for producing plastic fuel tank. Since, blow moulding process is a well established process for producing plastic products; efforts have been taken to solve the pertinent issues related to rotational moulding process to make rotational moulding process more promising. In the present work, extensive process modelling using statistical techniques has been performed to determine the optimum process parameters yielding better mechanical and fracture properties of the rotomoulded product considering LLDPE and LLDPE foam as a material.

9.2 Critical Findings

The major outcomes of the present research work are:

Engineers and designers have wide range of manufacturing methods available with them while they are considering the use of plastics in product design. The wide choice is advantageous in providing scope of ingenuity but it means that designers must have awareness of the capabilities and limitations for the selection of a processing method. The problem of appropriate process selection for a particular application has been a concern for the moulder considering the accessibility of number of plastic manufacturing processes. To address this, objective based MADM is proposed for process selection. As an emerging application, the process selection procedure is carried out for an automobile fuel tank. It is seen that the methodology ranked blow moulding and rotational moulding as the first and second choice of process respectively. From this study it is concluded that the research focus needs to be centered towards rotational moulding process by solving some pertinent issues and make the process promising in any situation.

One of the pertinent issues in rotational moulding process is to determine the optimum oven residence time which yields superior mechanical properties. In order to achieve this, simulation study and experimental investigation is conducted on rotational moulded product made using LLDPE. Simulation studies are conducted using ROTOSIM software to analyze different thermal transitions and phase changes that occur in the process. Degree of curing of the polymers is also assessed from the simulation study to correlate with mechanical properties. Experiments are conducted on a laboratory scale rotational moulding machine by varying the oven residence timings from 32 to 44 minutes. The products are tested for tensile, flexural and impact strengths according to respective ASTM standards. Experimental investigation revealed that there exist regions where the part is 'under-cured' and mechanical properties are found to be inferior. It is also found that when parts are 'over-cured', the mechanical properties are severely affected due to degradation which is confirmed by conducting differential scanning calorimetric test of LLDPE. A regime of optimal processing window is identified between 36-40 minutes where the highest tensile strength of 17.4 MPa, flexural strength of 17 MPa and impact strength of 0.96 J are noticed.

The major potential problem of thickness variation in rotational moulding is studied. Statistical technique is used to investigate and predict the optimum value of process parameters yielding required thickness. Experiments are performed on a lab-scale bi-axial rotational moulding machine wherein full factorial design is used to plan and analyze the experiments. Since rotational moulding uses biaxial rotation, relative speed of rotation about major and minor axis and time spent by the polymer inside the oven decides the distribution of polymer inside the mould. Therefore, Oven residence time and speed ratio are selected as process parameters. Experimental results confirmed that both the process parameters i.e. oven residence time and speed ratio have significant effect on thickness of the rotomoulded product. The result obtained confirms that there is a linear relation between the process parameters and thickness of the product. The linear relation can be pertained to the difference in heat absorption rate of powder particles inside the mould and there may be time lag between the melting of powders, this time lag may ultimately leads to the thickness variation in the final product. A statistical optimization is performed to obtain the proper set of process parameters to achieve required thickness. Form the experimental investigation and statistical optimization, the optimum value of oven residence time and speed ratio is determined as 37 minutes & 4:1 which yields a required thickness.

Since, components made of rotational moulding process are used in outdoor applications (like overhead chemical storage tanks, automobile fuel components etc), impact strength of such products is considered as one of the essential quality feature. Therefore, focus is to examine the rotomouldability of foamed polyethylene and the effect of process parameters on moulded product property. Experiments are performed on a lab-scale bi-axial rotational moulding machine wherein Box-Behenken design is used to plan and analyze the experiments. Experimental result confirms that oven temperature, oven residence time and cooling media are the principal process parameters affecting impact property of foamed rotomoulded products. Regression equations are used to predict the variations in impact strength within the regime of experimentation. For a foamed rotomoulded product it is found that, impact strength improves with oven temperature and time. These process parameters reduce the melt viscosity making it easier for bubbles to escape there by increasing the impact strength. However, impact strength is decreased with further increase in these parameters due to commencement of polymer degradation and cell coarsening. It is also observed that the proportion of amorphous region is increased due to faster cooling which favors improved impact strength. Optimum process parameters yielding desired impact strength are achieved using statistical optimization. Based on the experimental investigation and statistical optimization, the optimum setting of oven residence time, oven temperature and cooling medium is arrived as 37 minutes, 223^{o}C and water cooling which resulted maximum impact strength of 2.39 J.

The process modelling using statistical technique is further extended to determine the fracture toughness of rotomoulded products made using LLDPE. The oven temperature, oven residence time, and cooling media are considered as the critical process parameters affecting the fracture toughness. The experiments are performed on a lab-scale bi-axial rotational moulding machine wherein Box-Behenken design is used to plan and analyze the experiments. *R*-curve method is used to determine the fracture toughness of rotomoulded products. The procedure involves the measurement of load versus load–line displacement to determine the fracture toughness value (J_{IC}). All the fracture tests are carried out on a compact test specimen prepared as per the ASTM D6068 standard. Specimens are notched using sharp steel blade of thickness 0.1 mm. The tests are performed on a universal testing machine at a constant cross head speed of 1 mm/min at room temperature. From the experimental results it is found that for lower temperature fracture toughness value decreases as the oven residence time is increased owing to lower mobility of element and formation of micro cracks at the crack tip. It is also found that rapid cooling method favours better fracture toughness of rotomoulded products. A statistical optimization is carried out to obtain the proper set of process parameters (oven residence time: 32 minutes, oven temperature: 210^{o}C and cooling medium: water) to achieve maximum fracture toughness of 38 N/mm.

9.3 Specific Contributions

1. Objective based multi attribute decision making method is proposed for the selection of plastic manufacturing process. The proposed method is useful in evaluation, comparison and selection of a manufacturing process for particular application even for a novice user.
2. Statistical technique is used to investigate the mechanical and fracture toughness of rotomoulded product. Statistical optimization is performed to obtain the proper set of process parameters yielding better mechanical and fracture properties.
3. Optimum value of speed ratio and oven residence time is identified for required thickness of rotomoulded products.
4. Fracture toughness of rotomoulded products made using LLDPE is determined and optimum value of process parameters for desired fracture toughness is identified.

9.4 Recommendations

Following recommendations are proposed based on lab scale experimentation

1. Objective based multi attribute decision making method can be used for plastic manufacturing process selection.
2. It is recommended that the use of statistical modelling in polymer processing methods saves significant cost and time required for solving severe quality issues.
3. It is recommended to set the oven residence time between 36 to 40 minutes in rotational moulding process to achieve highest mechanical property.
4. In order to achieve the highest impact strength in foamed rotomoulded products, it is recommended to choose oven temperature, time as 223^{o}C & 37 minutes with faster cooling aids.
5. It is recommended to set the oven temperature, time as 210 ^{o}C & 32 minutes with faster cooling aids in rotational moulding process to achieve highest fracture toughness.

9.5 Future Scope of Work

1. Experimental studies can be extended to know the effect of thickness variation on fracture toughness of rotomoulded products.
2. Experimental studies can be extended on investigation of rotomoulded product produced using LLDPE blended with other additives or fillers like calcium carbonates, china clay and talc etc.
3. Present study is carried out at atmospheric temperature, however, low temperature and high temperature effect on fracture toughness of rotomoulded products can be studied for specific applications.

REFERENCES

Abhishek K. and Agrawal V.P. (2009): Attribute based specification, comparison and selection of electroplating system using MADM approach, *Expert System with Application*, Vol. 36, pp. 10815–10827.

Aiello M.A. and Ombres L. (2000): Crack analysis of FRP-reinforced concrete flexural members, *Mechanics of composite materials*, Vol. 36, pp. 645-654.

Alesseandro Pegoretti., Elisa Bertoldi and Theonis Ricco. (2005): Plane stress fracture toughness of ductile polymeric flims: effect of strain rate on the essential work of fracture parameters, *proceedings 11th International Conference on fracture*, pp. 167-175.

Ali S Argoan. (2013): *The Physics of Deformation and Fracture*, Cambridge University Press, Newyork.

Alongkorn Kanokboriboon and Harkin-Jones E. (2005): Processibility and properties of a rotomolded nano composite, *Rotation*, Vol. 6, pp. 61–64.

Alongkorn Kanokboriboon. (2009): Computer simulation & internal air temperature assist in moulding multi – layer rotationaly moulded parts. *Rotation*, Vol. 5, pp. 66-71.

Amara Ait Aissa, Carl Duchesne and Denis Rodrigue (2012): Characterization of polymer powder motion in a spherical mold in biaxial rotation. *Polymer Engineering and Science*, Vol.52, pp. 953-963.

Anderson T.L. (2005): *Fracture Mechanics Fundamentals and Applications*, CRC Press, Boca Raton, U.S.

Anojkumar L., Ilangkumaran M. and Sasirekha V. (2014): Comparative analysis of MCDM methods for pipe material selection in sugar industry. *Expert Systems with Applications*, Vol. 41, pp. 2964-2980.

Archer E., Harkin-Jones E., Kearns M.P., Crawford R.J and Fatnes A.M. (2002): An investigation of the rotational moulding of foamed metallocene polyethylenes, *Rotation*, Vol. 11, pp. 27–29.

ASTM D 638-10 (2010): Standard test method for tensile properties of plastics. American Society of Testing Materials, Philadelphia.

ASTM D1238-04 (2004): Standard test method for melt flow rate of thermoplastics. American Society of Testing Materials, Philadelphia.

ASTM D256-10 (2010): Standard test method for determining the izod pendulum impact resistance of plastics. American Society of Testing Materials, Philadelphia.

ASTM D6068-10 (2012): Standard test method for determining *J-R* curves of plastic materials. American Society of Testing Materials, Philadelphia.

ASTM D790-03 (2010): Standard test method for flexural properties of unreinforced and reinforced plastics. American Society of Testing Materials, Philadelphia.

Banerjee S., Yan W. and Bhattacharyya D. (2008): Modeling of heat transfer in rotational molding. *Polymer Engineering and Science,* Vol.48, pp. 2188-2197.

Bellehumeur C.T. and Tiang J.S. (2002): Simulation of non-isothermal melt densification of polyethylene in rotational molding, *Polymer Engineering and Science*, Vol.42 (1), pp. 215-229.

Bhangale P.P., Agrawal V.P. and Saha S.K. (2004): Attribute Based Specification, Comparison and Selection of a Robot, *Mechanism and Machine Theory*, Vol.39, pp.1345-1366.

Bharat I.C., Elizabeth T. and John V. (2001): Processing enhancers for rotational molding of polyethylene, *Polymer Engineering and Science*, Vol. 41, pp. 1731–1742.

Box G. and Behnken D. (1960): Some new three level designs for the study of quantitative variables. *Technometrics*, Vol. 2, pp. 455–475.

Bralla J.G. (1986): *Handbook of Product Design for Manufacturing*, McGraw-Hill publication, New York, USA.

Brent strong A. (2006): *Plastics Materials and Processing*, Pearson Education Inc, New jersey, United States.

Caliskan H., Kursuncu B., Kurnbanoglu C. and Guven S. Y. (2013): Material selection for the tool holder working under hard milling conditions using different multi criteria decision making methods. *Material and Design*, Vol. 45, pp. 473-479.

Carla F.M., Mohammad A.I and Vikas Prakash. (2007): Dynamic fracture of linear medium polyethylene under impact loading conditions, *Materials Science and Engineering*, Vol. 465, pp. 211-222.

Chaudhary B.I., Takacs E. and Vlachopoulos J. (2001): Processing enhancers for rotational molding of polyethylene, *Polymer Engineering and Science* Vol.41 (10), pp. 1731-1742.

Chen C.H., White J.L. and Ohta Y. (1991): A fundamental experimental study of the mechanisms of warpage and shrinkage of polyethylene in rotational molding. *International Polymer Processing*, Vol. 6(3), pp. 212-216.

Cramez M.C., Oliveira M.J. and Crawford R.J. (2003): Optimization of the rotational moulding process for polyolefin's. *Proceedings of the Institution of Mechanical Engineers, Part B: Journal of Engineering Manufacture*, Vol.217, pp. 323-334.

Crawford R.J. (1992): *Rotational Moulding of Plastics*, Research study press, Somerset.

Crawford R.J. (1994): Causes and cures of problems during rotomoulding, *Rotation*, Vol.3, pp. 10–14.

Crawford R.J. (1996): Recent advances in the manufacture of plastic products by roto molding, *Journal of Material Processing Technology*, Vol. 56 (1-4), pp.263-271.

Crawford R.J. (2000): Residual stress in rotational moulded parts, *Rotation,* Vol.11, pp. 20–24.

Crawford R.J. (2003): Vision 2020 through 2002, *Rotation*, Vol.12 (1):16-19.

Crawford R.J. (2006): *Plastic Engineering*, Elsevier India Private Ltd, New Delhi, India.

Crawford R.J. and Kearns M.P. (2003): *Practicle Guide to Rotational Moulding*, Rapra Technological Limited, Shropshire, U.K.

Crawford R.J. and Nugent P.J. (1992[a]): Impact strength of rotationally moulded polyethylene articles. *Plastics, Rubber and Composites Processing and Applications,* Vol. 17 (1), pp. 33-41.

Crawford R.J. and Nugent, P.J. (1992[b]): A new process control system for rotational moulding. *Plastics, Rubber and Composites Processing and Applications,* Vol. 17 (1), pp. 23-31.

Crawford R.J., Spence A.G., Cramez M.C. and Oliveira M.J. (2004): Mold pressure control as a tool in rotational molding, *Proceedings of the Institution of Mechanical Engineers, Part B: Journal of Engineering Manufacture*, Vol. 218 (12), pp. 1683-1693.

Darji V. P. and Rao R. V. (2014): Intelligent multi criteria decision making methods for material selection in sugar industry. *Procedia Material Science*, Vol. 5, pp.2585-2594.

Durai Prabhakaran R.T., Babu B.J.C. and Agrawal V.P. (2006): Optimum selection of Composite product system using a MADM approach, *Journal of Materials and Manufacturing Processes,* Vol. 21, pp.883 – 891.

Fabiano Moreno Peres., Jose Ricardo Tarpani and Claudio Geraldo Schon (2013): An assessment of essential work of fracture testing method applied to medium density polyethylene (MDPE). *Engineering Fracture Mechanics*, Vol.105, pp. 136-151.

Francis M., Mirabella J.R., Stanley P.W., Fernando P.L., Emory A.F. and Williams J.G. (1988): Morphological explanation of the extraordinary fracture toughness of linear low density polyethylenes, *Journal of Polymer Science Part B: Polymer physics*, Vol. 26 (9), pp. 1995-2005.

Glomsaker T., Hinrichsen E.L., Larsen A., Doshev P. and Ommundsen E. (2009): Warpage-crystallinity relations in rotational molding of polypropylene, *Polymer Engineering and Science*, Vol.49 (3), pp. 523 – 530.

Godinho J.S., Cunha A. and Crawford R.J. (2002): Influence of thermo mechanical conditions on the structure development and mechanical properties of a LMPDE grade under different molding techniques. *Proceedings of Annual Technical Conference (ANTEC), San Francisco.*

Gogos G., Olson L.G., Liu X. and Pasham V.R. (1998): New models for rotational molding of plastics, *Polymer engineering and science,* Vol.38 (9), pp.1387-1398.

Greco A. and Maffezzoli A. (2004): Powder-shape analysis and sintering behaviour of high-density polyethylene powders for rotational molding, *Journal of Applied Polymer Science*, Vol. 92, pp. 449-460.

Guobin L., Chul Park B. and John Lefas A. (2004): Rotational moulding of low-density lldpe foams, *Polymer Engineering and Science,* Vol. 38, pp. 1997–2009.

Jansson L.E. (2003): *Plastic pipes for water supply and sewage disposal*, Stockholom: VBB/SWECO.

Jee D.H. and Kang K.J. (2000): A method for optimal material selection aided with decision making theory. *Materials and Design,* Vol. 21, pp. 199–206.

Jones R. (2003): Vent design for rotational molding. *Rotation*, pp. 42- 45.

Kearns M.P. and Collan N.M. (2000): The use of hollow microspheres in rotational molding of plastic parts-Part 2: Two layer molding, *Rotation*, pp.20-24.

Kearns M.P., Wallace W., Murphy M., Armstrong C. and Crawford R.J. (2001): An introduction to the use of additional UV absorbers in the rotational molding process, *Rotation*, pp.18-22.

Khouri R.M. (2004**)**: *Reducing cycle time in rotational moulding of plastics*. Ph.D Thesis, Queens University of Belfast, U.K.

Kiran C.P., Shibu Clement and Agrawal V.P. (2011): Coding, evaluation and optimal selection of a mechatronic system, *Expert System with Application*, Vol. 38, pp. 9704-9712.

Klempener D. and Frisch K.C. (1991): *Handbook of polymeric foams and foam technology*, Hanser publication, New York, USA.

Kolednik O., Schöngrundner R. and Fischer F.D. (2014): A new view on *J*-integrals in elastic–plastic materials. *International Journal of Fracture*, Vol. 187, pp.77-107.

Kontopoulou M., Bisaria M. and Vlachopoulos J. (1997): An experimental study of rotational molding of polypropylene and polyethylene copolymers, *International Polymer Processing*, Vol.12 (2), pp.165-1 73.

Laws R.D. (2004): An in-depth look at the influential factors of the dry-flow/bulk-density test used in the rotomoulding industry, *Rotation*, Vol.10, pp. 12–14.

Li G.D., Yamaguchi D. and Nagai M.A. (2007): Grey-based decision making approach to the supplier selection problem, *Mathematical and Computer Modeling,* Vol. 46, pp. 573–581.

Liu S.J. and Ho C.Y. (1999): Factors affecting the warpage of rotationally molded parts, *Advances in Polymer Technology*, Vol.18 (3), pp. 201-207.

Ljunggberg L.Y. (2003): Materials selection and design for structural polymers. *Materials and Design*, Vol.24, pp.383-390.

Mai Y.W. and Powell P.J. (1991): Essential work of fracture and j-integral measurements for ductile polymers, *Journal of Polymer Science Part B: Polymer physics*, Vol. 29 (7), pp. 785-793.

Maryam Emami., Michael R. Thompson and John Vlachopoulos. (2013): Experimental and numerical studies on bubble dynamics in nonpressurized foaming systems. *Polymer Engineering and Science*, Vol.54, pp. 1947-1959.

McNally G.M., Kearns M.P. and Crawford R.J (2002): A new method for observing density changes of plastics during rotational molding, *Rotation*, pp.46-48.

Michler G.H. and Balta Calleja F.J. (2005): *Mechanical Properties of Polymers Based on Nanostructure and Morphology*, Taylor & Francis Group, Boca Raton.

Mills N.J. (1993): *Plastics: microstructures and engineering applications*, Edward Arnold, London.

Ming Luen Lu., Chang Bing Lee and Feng Chih Chang. (2004): Fracture toughness of acrylonitrile butadiene styrene by *J* integral method. *Polymer Engineering and Science*, Vol.35, pp. 1433-1439.

Montgomery D.C. (2012): *Introduction to Statistical Quality Control*, Wiley India, New Delhi, India.

Myer Kutz. (2011): *Applied Plastics Engineering Handbook Processing and Materials*, William Andrew Publishing, Waltham, USA.

Myer Kutz. (2013): *Handbook of Measurement Science and Engineering*, John Wiley & Sons Publishing, Hobokan, New Jercy.

Nugent P., Crawford R.J. and Xu L. (1992): Computer prediction of cycle time during rotational moulding of plastics, *Advances in Polymer Technology*, Vol.11 (3), pp.181-191.

Ochensberger W. and Kolednik O. (2014): A new basis for the application of the *J* -integral for cyclically loaded cracks in elastic–plastic materials. *International Journal of Fracture*, Vol. 189, pp.77-101.

Olinek J., Anand C. and Bellehumeur C.T. (2005). Experimental study on the flow and deposition of powder particles in rotational molding, *Polymer Engineering and Science*, Vol.45 (1), pp.62-73.

Oliveira M.J., Cramez M.C. and Crawford R.J. (1996): Structure-properties relationships in rotationally moulded polyethylene. *Journal of material science*, Vol.31, pp. 2227-2240.

Paton C.A. and Hashemi S.J (1992): Plane-stress essential work of ductile fracture for polycarbonate, *Journal of Material Science*, Vol.29, pp. 2279-2290.

Phaneendra Kiran C. and Shibu Clement (2013): Surface quality investigation of turbine blade steels for turning process, *Measurement*, Vol. 46, pp. 1875-1895.

Pick L.T and Jones E.H. (2003[b]): Understanding the impact behaviour of rotomolded linear low-density polyethylenes, *Rotation*, pp. 26-30.

Pick L.T. and Jones E.H. (2003[a]): An investigation into relationship between the impact performance of rotationally molded polyethylene products and their dynamic mechanical properties, *Polymer Engineering and Science*, Vol.43 (4), pp. 905-918.

Prashant Kumar (2011): *Elements of Fracture Mechanics*, Tata Mcgraw Hill, New Delhi, India.

Quan Zhang., Jason C.H.C., Yan-Qun He., Jian Ma and Duan-Ning Zhou. (2003): Multiple attribute decision making: approach integrating subjective and objective information, *International Journal of Manufacturing Technology and Management*, Vol.5, pp. 338-361.

Rao R.V. (2007): *Decision Making in the Manufacturing Environment Using Graph Theory and Fuzzy Multiple Attribute Decision Making Method*, Springer Verlag, London.

Rao R.V. and Patel B.K. (2010): A subjective and objective integrated multiple attribute decision making method for material selection. *Materials and Design,* Vol.31, pp. 4738- 4747.

Rao R.V., Patel B.K. and Parnichkun M. (2011): Industrial robot selection using a novel decision making method considering objective and subjective preferences, *Robotics and Autonomous Systems*, Vol. 59, pp. 367-375.

Raviwongse R., Allada V. and Sandidge Jr T. (2000): Plastic manufacturing process selection methodology using self organizing map (SOM) / fuzzy analysis, *Internal Journal of Advanced Manufacturing Technology,* Vol. 16, pp. 155-161.

Remon Pop- Iliev., Ning Dong., Donglai X.U and Chul B Park. (2008): Visualization of the foaming mechanism of polyethylene blown by chemical blowing agents under ambient pressure, *Advanced Polymeric Technology*, Vol. 26, pp. 213-232.

Rice J.R. (1968): A Path Independent Integral and the Approximate Analysis of Strain Concentration by Notches and Cracks, *Journal of Applied Mechanics*, Vol. 35(2), pp.379-386.

Robert A., Orr J.F and Crawford R.J. (2000): Influence of mica and talc fillers on the properties of rotational moulded lldpe, *Proceedings of Annual Technical Conference (ANTEC), San Francisco*, pp. 1399-1404.

Sachin D. Waigoankar (2010): *Experimental investigations involving quality modeling concepts for rotational moulding of plastics and compression moulding of composites*. Ph. D Thesis, Birla Institute of Technology and Science, Pilani.

Sachin D. Waigoankar., Babu B.J.C and Durai Prabakaran R.T. (2008): A new approach to resin selection in rotational moulding. *Journal of Reinforced Plastics and Composites*, Vol. 27, pp. 1021-1037.

Sachin D. Waigoankar., Babu B.J.C and Amit Rajput (2011): Six sigma DOE approach: a case study of rotational moulding process. *International Journal of Six Sigma and Competitive Advantage,* Vol. 6, pp. 256-277.

Said Lotfi Hafsaoui., Mokhtar Benziane and Abbas Tcharkhtchi (2012): Thermal transfer simulation regarding the rotational moulding of polyamide 11. *Journal of thermal analysis and calorimeter,* Vol. 112, pp. 285-292.

Salah Sarrabi., Boyer S.A.E., Marie France Lacrampe., Krawczak P. and Abbas Tcharkhtchi (2013): Metallocene polypropylene crystallization kinetic during cooling in rotational molding thermal condition. *Journal of Applied Polymer Science*, Vol.130, pp. 222-233.

Santosh Bawiskar and James L. White (1994): Comparative study of warpage, global shrinkage, residual stresses, and mechanical behaviour of rotationally molded parts produced from different polymers. *Polymer Engineering and Science,* Vol.34 (10), pp. 845-820.

Seibi A.C. and Sawaqed N.M. (2002): Design of copper filled fiberglass molds for manufacturing a customized product using linear programming optimization and finite element analysis, *Composites Part A: Applied Science and Manufacturing*, Vol.33 (121), pp.677-1683.

Seyed Ali Ghassemi and Shahnaz Danesh. (2013): A hybrid fuzzy multi – criteria decision making approach for desalination process selection, *Desalination*, Vol. 313, pp. 44-50.

Shanian A. and Savadogo O. (2006[a]): A material selection model based on the concept of multiple factor decision making. *Materials and Design,* Vol. 27, pp. 329–37.

Shanian A. and Savadogo O. (2006[b]): A non-compensatory compromised solution for material selection of bipolar plates for polymer electrolyte membrane fuel cell (PEMFC) using ELECTRE IV. *Electrochimica Acta*, Vol. 51, pp. 5307–5315.

Shanian A. and Savadogo O. (2006[c]): TOPSIS multiple-criteria decision support analysis for material selection of metallic bipolar plates for polymer electrolyte fuel cell. *Journal of Power Source.* Vol. 159, pp. 1094–1096.

Shih- Jung Liu and Ching- Hsiung Yang (2001): Rotational moulding of two layered polyethylene foams, *Advances in Polymer Technology*, Vol. 20, pp. 108-115

Shih-Jung Liu and Chja-Hsun Tsai. (1999): An Experimental Study of Foamed Polyethylene in Rotational Moulding, *Polymer Engineering and Science*, Vol. 39, pp. 1776–1786.

Shih-Jung Liu and Kang-Ming Peng. (2010): Rotational molding of polycarbonate reinforced polyethylene composites: processing parameters and properties. *Polymer Engineering and Science*, Vol.50, pp. 1457-1465.

Shih-Jung Liu. (2000): Optimizing the impact strength of rotational moulded parts, *Polymer. Science and Engineering*, Vol. 40, pp. 473-480.

Society of Manufacturing Engineers (SME). (1996): *Tool and Manufacturing Engineers Handbook (TMEH) Volume 8: Plastic Parts Manufacturing*, Society of Manufacturing Engineers.

Spence A.G. and Crawford R.J. (1996[a]): Removal of pin holes and bubbles in rotationaly moulded products. *Proceedings of the Institution of Mechanical Engineers, Part B: Journal of Engineering Manufacture*, Vol.210, pp. 521-533.

Spence A.G. and Crawford R.J. (1996[b]): The effect of processing variables on the formation and removal of bubbles in rotationally molded products, *Polymer Engineering and Science*, Vol.36 (7), pp. 993-1009.

Tamas Barany., Tibor Czigany and Jozsef Karger-Kocsis. (2003): Essential work of fracture concept in polymers, *Periodica Polytechnica. Ser. Mech. Eng*, Vol. 47 (2), pp. 91-102.

Tan S.B. (2010): *Accelerated Cooling of Thermoplastics in Rotational Moulding*. Ph.D. Thesis in Mechanical and Aerospace Engineering, Queen’s University Belfast, UK.

Tan S.B., Hornsby P.R., McAfee M.B., Kearns M.P. and McCourt M.P. (2011): Internal cooling in rotational moulding- a review. *Polymer Engineering and Science*, Vol.51, pp. 1683-1692.

Tan S.B., Hornsby P.R., McAfee M.B., Kearns M.P. and McCourt M.P. (2012): Water sprays cooling of polymers. *Polymer Engineering and Science,* Vol.52, pp. 1069-1080.

Tcharkhtchi A. and Verdu J. (2004): Structure-processibility relationship during rotational molding of plastics, *Advanced Engineering Materials*, Vol.6 (12), pp. 983 - 992.

Thirumalai R. and Senthilkumaar J.S. (2013): Multi-criteria decision making in the selection of maching parameters for inconel 718, *Journal of Mechanical Science and Technology*, Vol. 27, pp. 1109-1116.

Tjong S.C. and Bao S.P. (2007): Fracture toughness of high density polyethylene/sebs-g-ma/montmorillonite nanocomposites. *Composite Science and Technolology*, 67 (2), pp. 314-323.

Vahid Sendijarevic and Klempner D. (1991): *Handbook of Polymeric Foams and Foam Technology*, Hanser Verlag Publishers, Germany.

Van Hooijdonk J.P.F., Kearns M. P., Armstrong C.G. and Crawford R.J. (2001): Effect of processing conditions on the properties of rotationally molded polypropylene. *Rotation*: 28-30.

Wang M.D., Nakanishi E. and Hashizume Y. (1992): Fracture energy analysis of single-edge-cracked isotropic ductile polyolefin’s, *Polymer*, Vol. 33, pp. 3408-3414.

Williams J.G. (1987): *Fracture mechanics of polymers*, Ellis Harwood, Chichester.

Yan W., Lin R.J.T., Bickerton S. and Bhattacharya D. (2003): Rotational moulding of particle reinforced polymeric shell structures, *Material Science Forum*, pp. 235-238.

Yan W., Lin R.J.T., Bickerton S. and Bhattacharyya D. (2003): Rotational molding of particulate reinforced polymeric shell structures, *Rotation*, pp. 16-19.

Yang T and Hung C.C. (2007): Multiple-attribute decision making methods for plant layout design problem, *Robotics and Computer- Integrated Manufacturing*, Vol. 23, pp. 126–137.

Yu J.C., Krizan S. and Ishii K. (1993): Computer-aided design for manufacturing process selection, *Journal of Intelligent Manufacturing*, Vol. 4, pp. 199–208.

PUBLICATIONS BASED ON PRESENT WORK

Published / Accepted Papers in International Journals

1. **Ramkumar PL**., Kulkarni D.M. and Chaudhari V.V. (2014): Parametric and Mechanical Characterization of Linear Low Density Polyethylene (LLDPE) Using Rotational Moulding Technology. *Journal of Sadhana, Springer,* Vol: 39, issue: 3, pp 625-635. **(IF: 0.39)**.

2. **Ramkumar PL** and Kulkarni D.M. (2014): Objective Based Multiple Attribute Decision Making Method for Plastic Manufacturing Process Selection. *International Journal of Manufacturing Technology and Management, Inderscience.* Vol: 28, issue: 4/5/6, pp. 184-199.

3. **Ramkumar PL**., Kulkarni D.M., Abhijit V.V.R and Aditya Cherukumudi (2014): Investigation of Melt Flow Index and Impact Strength of Foamed LLDPE for Rotational Moulding Process. *Procedia Materials Science, Elsvier.* Vol: 6, issue: 3, pp. 361-367.

4. **Ramkumar PL**., Kulkarni D.M. and Waigonkar S.D. (2013): Effect of Cycle Time on Mechanical Properties of LLDPE during Rotational Moulding Process. *Journal of Polymer Research, Nova,* Vol: 8, issue : 1.

5. **Ramkumar PL**., Kulkarni D.M and Waigaonkar S.D.: Simulation Based Experimental Investigation of Effect of Oven Residence Time on Mechanical Properties in Rotational Moulding. *Journal of Sadhana, Springer (under review).*

6. **Ramkumar PL** and Kulkarni D.M: Impact Strength Investigation of Foamed LLDPE in Rotational Moulding Process Using Design of Experiments. *International Journal of Material Engineering and Innovation, Inderscience (under review).*

Book Chapter

1. **Ramkumar PL.**, Kulkarni D.M. and Waigaonkar S.D. (2015): Macro Level Investigation on Thickness Variation of Rotomoulded LLDPE Products. Book chapter from Advanced Polymeric Materials. *Apple Academic Press.* (Accepted).

Proceedings of International Conferences

1. **Ramkumar PL**., Kulkarni D. M. and Sachin D. Waigaonkar (2013): Effect of Speed Ratio and Cycle Time on Thickness of LLDPE Products in Rotational Moulding Process. *ICAPM-2013, organized by MG university Kerala, Beijing University China & Wroclaw University Poland.* OCT: 11-13, pp 118-119.

2. **Ramkumar PL** and Kulkarni D. M. (2012): Mechanical and Fracture Characterization of Linear Low Density Polyethylene (LLDPE) using Rotational Moulding Technology. *2nd International Conference on Nanotechnology- Innovative Materials, Processes, Products and Applications (Nanocon 012) organized by BVU*, Pune. OCT: 18-19, pp: 22-31.

APPENDICES

Appendix A

Temperature, polymer phase transition and degree of curing plots for different oven residance time

Temperature and polymer phase transition plots for different oven residance time

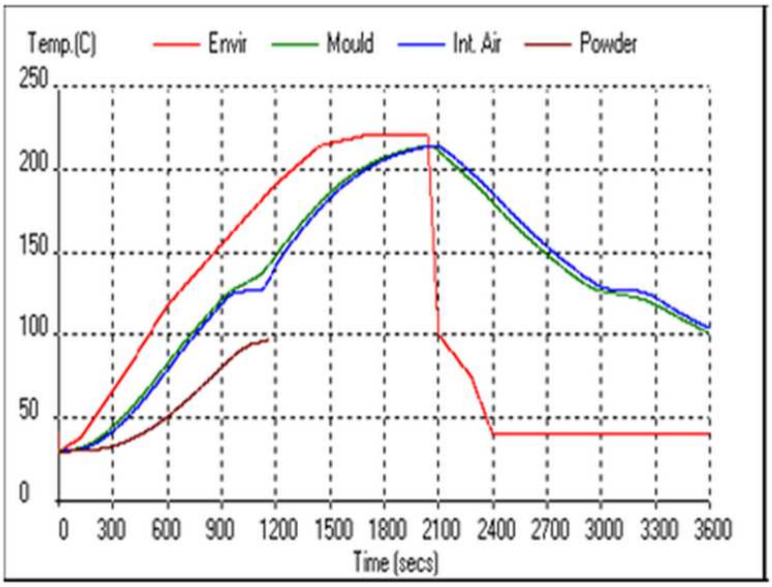


Fig A 1 Temperature Plot for 34 mins

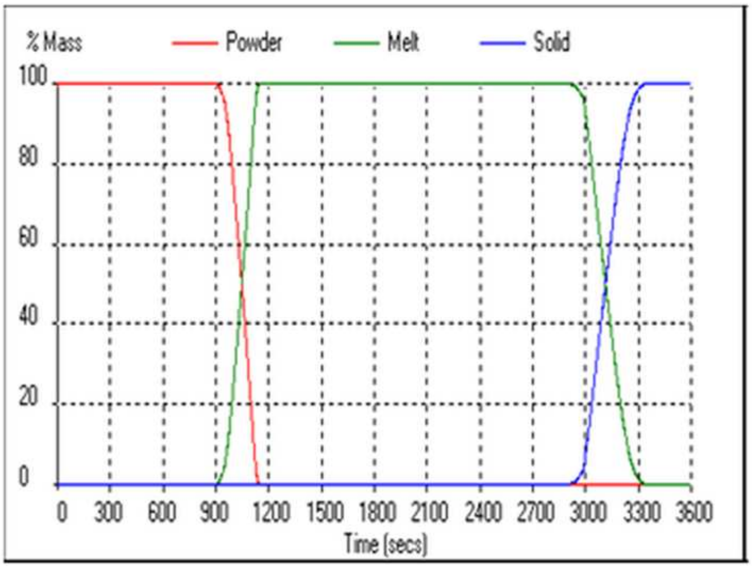


Fig A 2 Polymer Phase Transitions plot for 34 mins

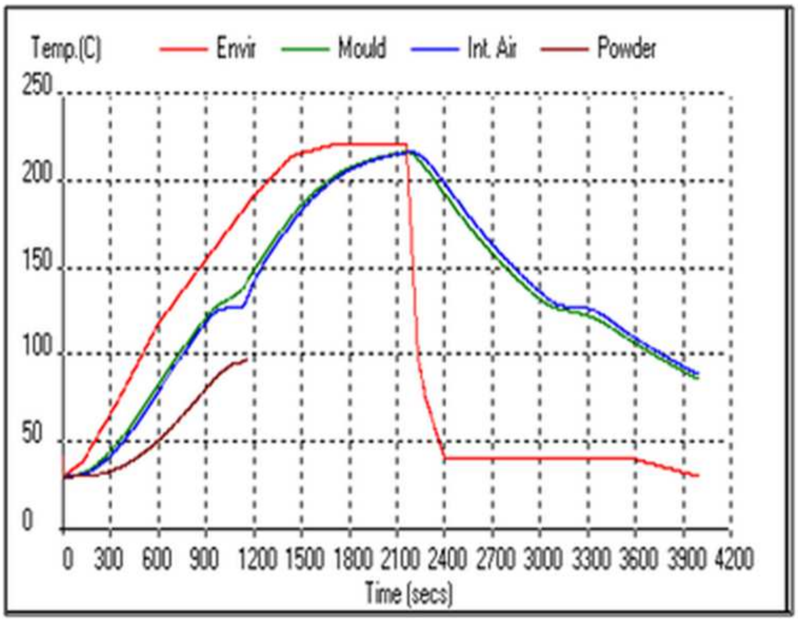


Fig A 3 Temperature Plots for 36 mins

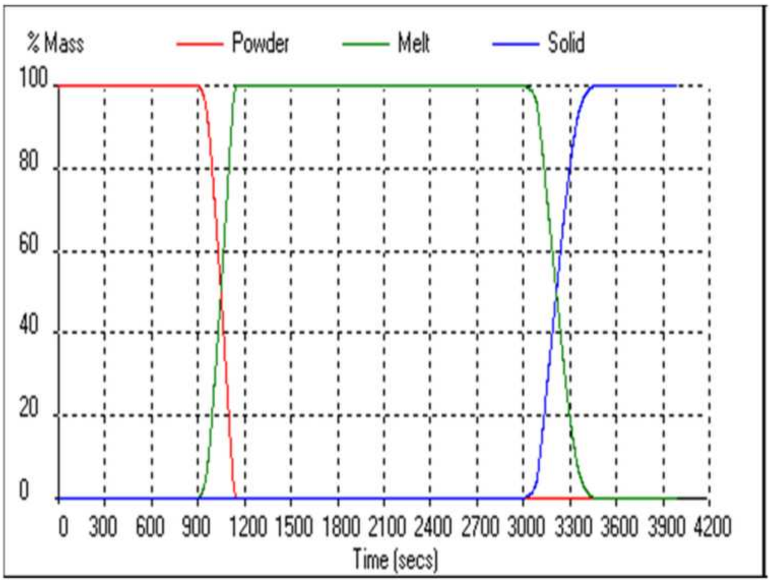


Fig A 4 Polymer Phase Transitions plot for 36 mins

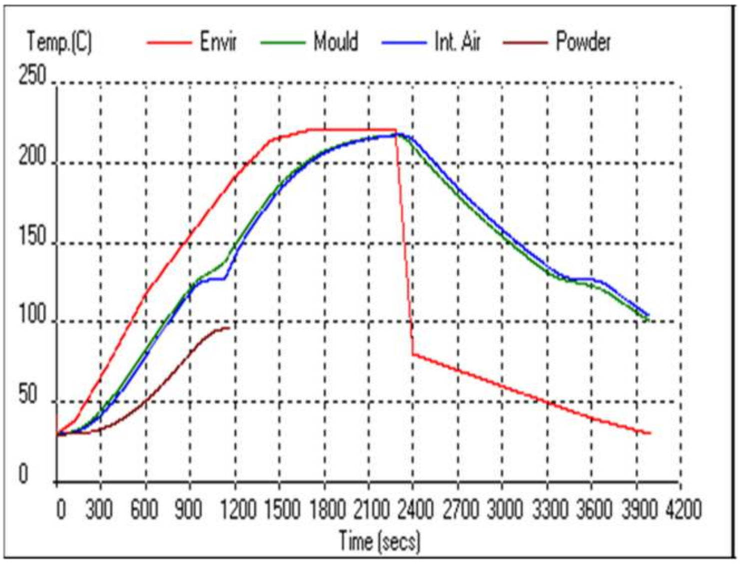


Fig A 5 Temperature Plots for 38 mins

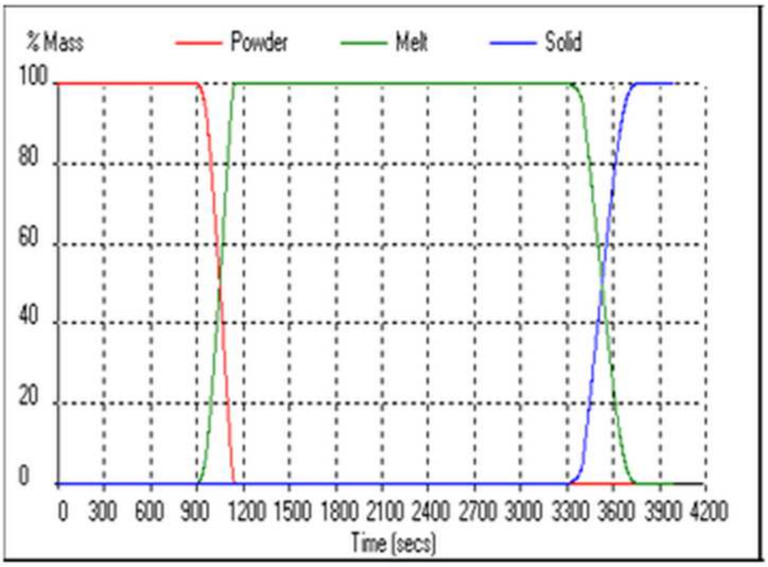


Fig A 6 Polymer Phase Transitions plot for 38 mins

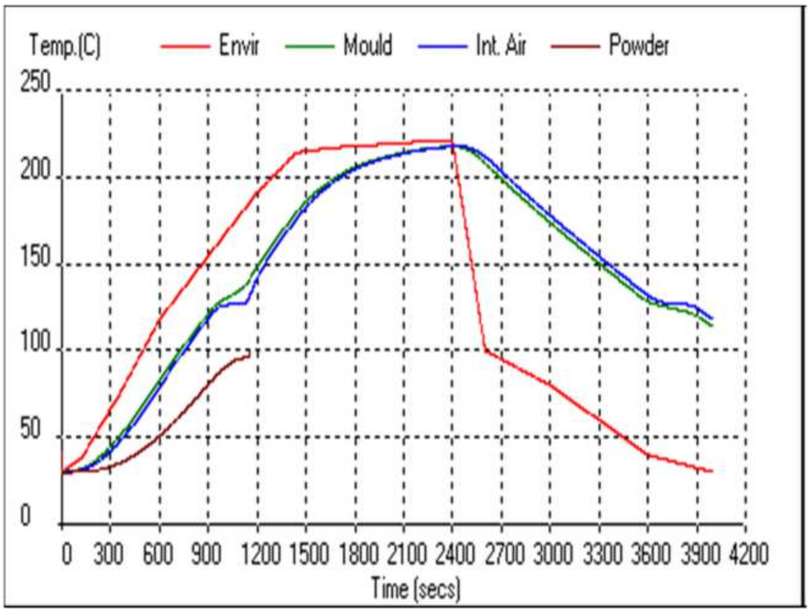


Fig A 7 Temperature Plots for 40 mins

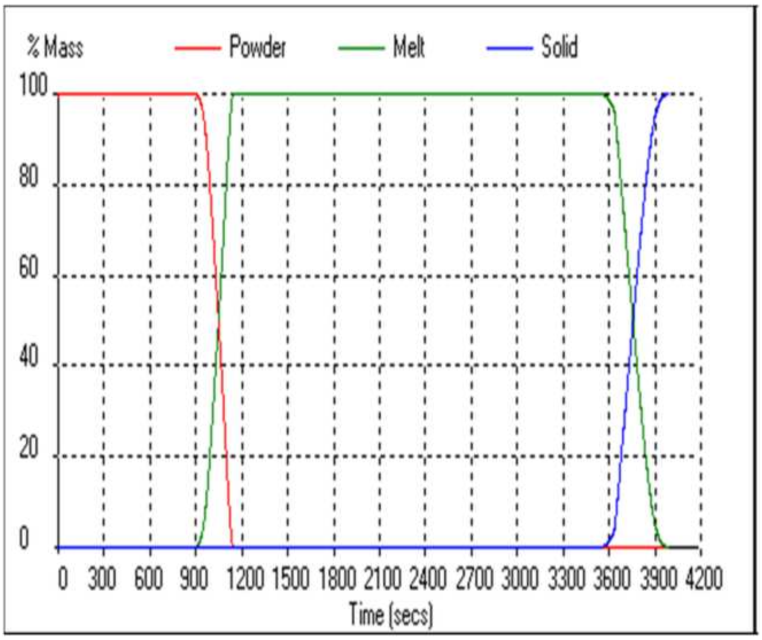


Fig A 8 Polymer Phase Transitions plot for 40 mins

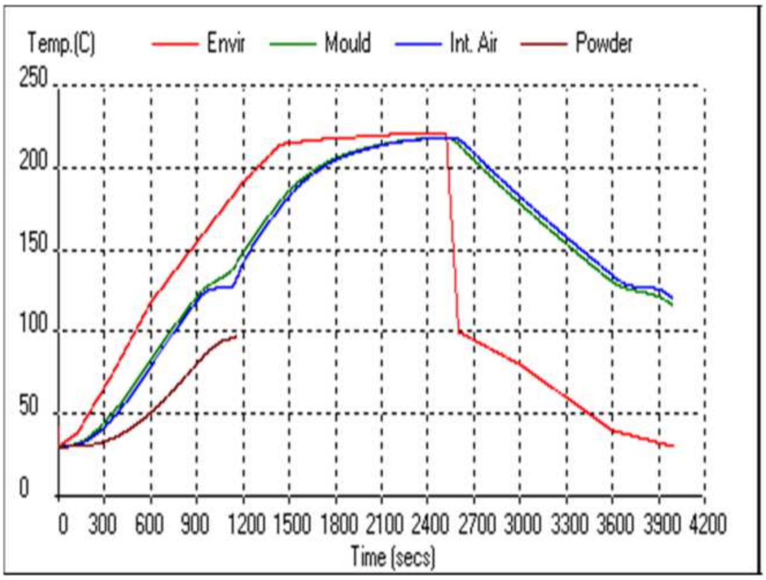


Fig A 9 Temperature Plots for 42 mins

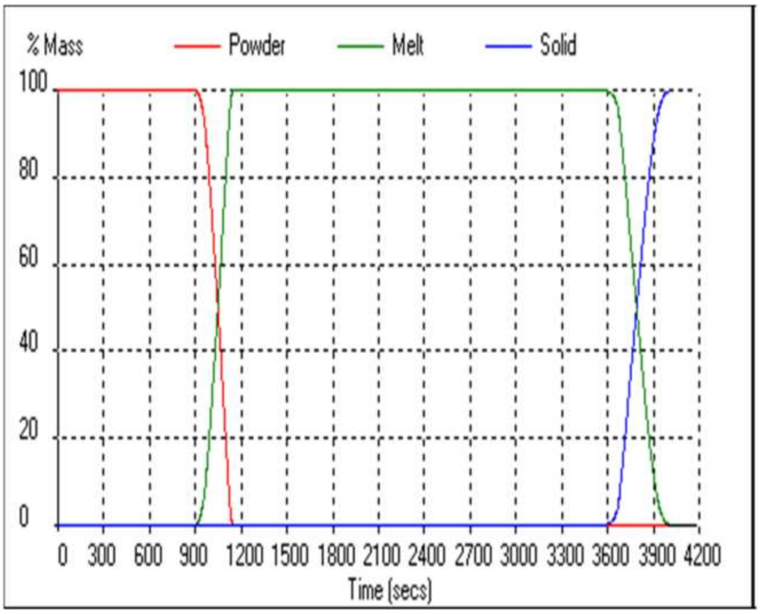


Fig A 10 Polymer Phase Transitions plot for 42 mins

Degree of Curing plot for different oven residance time

Fig A 11 Degree of curing plot for 32 mins

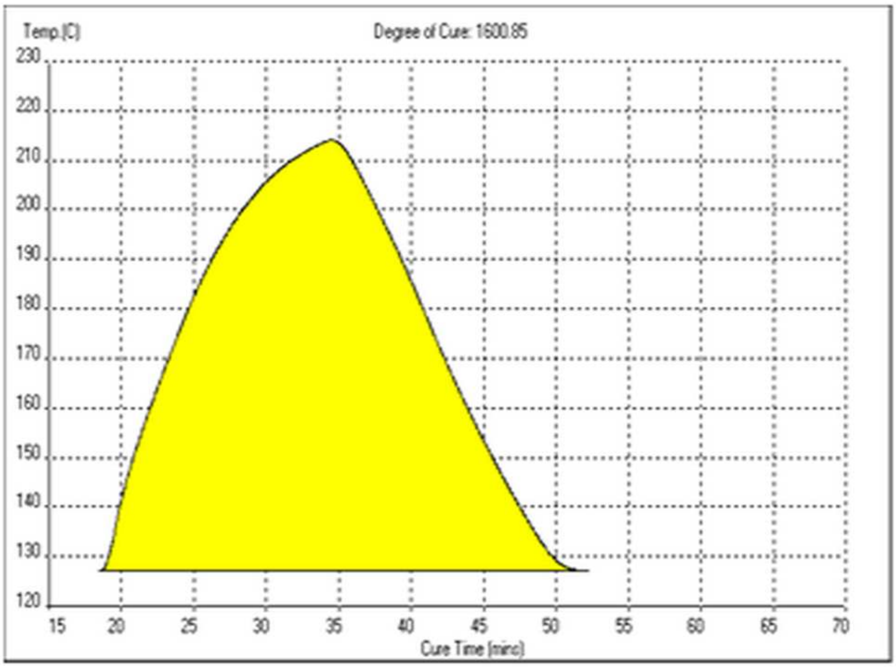


Fig A 12 Degree of curing plot for 34 mins

Fig A 13 Degree of curing plot for 36 mins

Fig A 14 Degree of curing plot for 38 mins

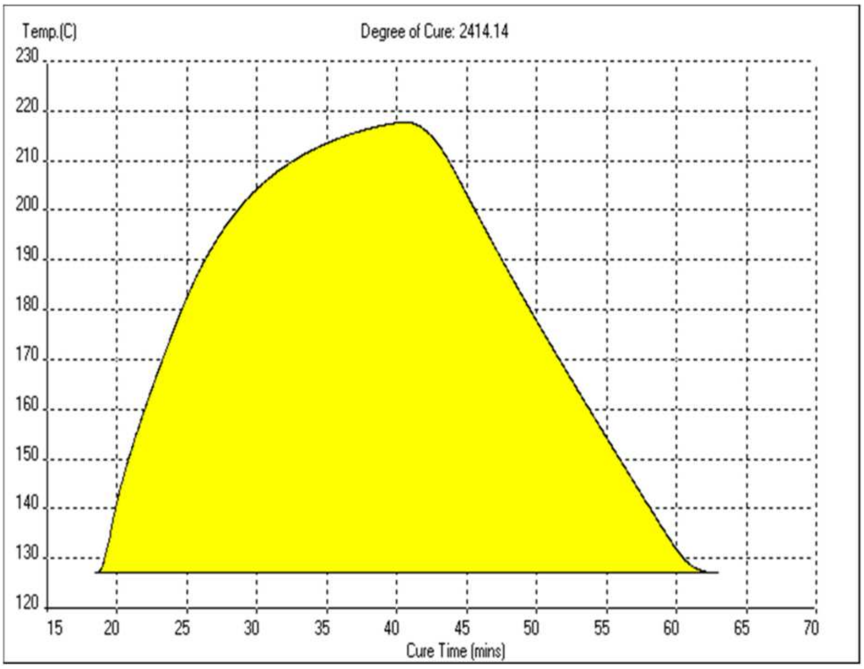


Fig A 15 Degree of curing plot for 40 mins

Fig A 16 Degree of curing plot for 42 mins

Appendix B

Estimation of parameters in regression models

Estimation of Regression Coefficients

To find the regression coefficients in a regression model, the method of least squares is adapted. This appendix discusses the method with reference to linear regression equations (Montgomery, 2004).

Let there be n (n > k) observations of the response variables, say y_1, y_2, ... y_n. For each observed response y_i, an observation will have regressor variable and let x_{ij} denote the same for i^{th} observation for level of variable x_j. The data will appear as in Table B 1. We assume that the error term Є in the model has E (Є) = 0 and V (Є) = σ^2 and that {$Є_i$} are uncorrelated random variables.

The model equation for linear regression in terms of the observations in Table B1 can be written as:

$$y_i = \beta_0 + \beta_1 x_{i1} + \beta_2 x_{i2} + \ldots + \beta_k x_{ik} + Є_i \qquad \text{(B 1.1)}$$

$$= \beta_0 + \sum_{j=1}^{k} \beta_j x_{ij} + Є_i \qquad i = 1, 2, \ldots, n$$

According to the method of least squares, β's in Equation (B 1.1) are chosen such that the sum of the squares of the errors, $Є_i$, is minimized. The least squares function is written as:

$$L = \sum_{i=1}^{n} \epsilon_i^2$$

$$= \sum_{i=1}^{n} \left((y_i - \beta_0 - \sum_{j=1}^{k} \beta_j x_{ij} \right) \qquad \text{(B 1.2)}$$

The function L is to be minimized with respect to β_0, β_1, . . . , β_k. To satisfy the condition for minimisation of error, the least squares estimators say $\hat{\beta}_0$, $\hat{\beta}_1$, . . . , $\hat{\beta}_k$, must satisfy:

$$\left.\frac{\partial L}{\partial \beta_0}\right|_{\hat{\beta}_0, \hat{\beta}_1, \ldots \hat{\beta}_k} = -2 \sum_{i=1}^{n} \left((y_i - \hat{\beta}_0 - \sum_{j=1}^{k} \hat{\beta}_j x_{ij} \right)^2 = 0 \qquad \text{(B 1.3)}$$

and

$$\left.\frac{\partial L}{\partial \beta_j}\right|_{\hat{\beta}_0, \hat{\beta}_1, \ldots \hat{\beta}_k} = -2 \sum_{i=1}^{n} \left((y_i - \hat{\beta}_0 - \sum_{j=1}^{k} \hat{\beta}_j x_{ij} \right)^2 = 0 \qquad \text{(B 1.4)}$$

where j = 1, 2, . . . , k

Table B 1 Data for multiple linear regressions

y	x_1	x_2		. . .		x_k
y_1	x_{11}	x_{12}		. . .		x_{1k}
y_2	x_{21}	x_{22}		. . .		x_{2k}
.	.	.		. . .		.
.	.	.		. . .		.
.	.	.		. . .		.
y_n	x_{n1}	x_{n2}		. . .		x_{nk}

Simplifying Equation (B 1.3) and (B1.4), we obtain:

$$n\hat{\beta}_0 + \hat{\beta}_1 \sum_{i=1}^{n} x_{i1} + \hat{\beta}_2 \sum_{i=1}^{n} x_{i2} + \ldots + \hat{\beta}_k \sum_{i=1}^{n} x_{ik} = \sum_{i=1}^{n} y_i$$

$$\hat{\beta}_0 \sum_{i=1}^{n} x_{i1} + \hat{\beta}_1 \sum_{i=1}^{n} x_{i2} + \hat{\beta}_2 \sum_{i=1}^{n} x_{i1} x_{i2} + \ldots + \hat{\beta}_k \sum_{i=1}^{n} x_{i1} x_{ik} = \sum_{i=1}^{n} x_{i1} y_i$$

. . . .

. . . .

$$\hat{\beta}_0 \sum_{i=1}^{n} x_{ik} + \hat{\beta}_1 \sum_{i=1}^{n} x_{ik} x_{i1} + \hat{\beta}_2 \sum_{i=1}^{n} x_{ik} x_{i2} + \ldots + \hat{\beta}_k \sum_{i=1}^{n} x_{ik}^2 = \sum_{i=1}^{n} x_{ik} y_i$$

(B 1.5)

These equations are referred as the least square normal equations. Note that there are p = k + 1 normal equations, one for each of the unknown regression coefficients. The solution to the normal equations will be the least square estimators of the regression coefficients $\hat{\beta}_0$, $\hat{\beta}_1$, ... $\hat{\beta}_k$.

The above normal equations can be solved easily, if expressed in matrix notation. The normal equations (B1. 4) now are written in matrix form. The model in terms of observations, Equation (B1.1), may be written in matrix notation as

$$\mathbf{y} = \mathbf{X}\boldsymbol{\beta} + \epsilon \qquad \text{(B 1.6)}$$

where;

$$\boldsymbol{y} = \begin{bmatrix} y_1 \\ y_2 \\ . \\ . \\ . \\ y_n \end{bmatrix}, \mathbf{X} = \begin{bmatrix} 1 & x_{11} & x_{12} & \dots & x_{1k} \\ 1 & x_{21} & x_{22} & \dots & x_{2k} \\ . & . & . & \dots & . \\ . & . & . & \dots & . \\ . & . & . & \dots & . \\ 1 & x_{n1} & x_{n2} & \dots & x_{nk} \end{bmatrix}, \boldsymbol{\beta} = \begin{bmatrix} \beta_0 \\ \beta_1 \\ . \\ . \\ . \\ \beta_k \end{bmatrix} \text{ and } \epsilon = \begin{bmatrix} \boldsymbol{\epsilon}_0 \\ \boldsymbol{\epsilon}_1 \\ . \\ . \\ . \\ \boldsymbol{\epsilon}_k \end{bmatrix}$$

Here, **y** is an (n X 1) vector of the observations, **X** is an (n X p) matrix of the levels of the independent variables, **β** is a (p X 1) vector of the regression coefficients and Є is an (n X 1) vector of random errors.

We wish to find the vector of least squares estimators,$\hat{\beta}$ that minimizes

$$L = \sum_{i=1}^{n} \epsilon_i^2 = \boldsymbol{\epsilon}'\boldsymbol{\epsilon} = (\boldsymbol{y} - \boldsymbol{X\beta})'(\boldsymbol{y} - \boldsymbol{X\beta}) \qquad \text{(B 1.7)}$$

Note that L may be expressed as (Mongomery, 2004)

$$L = y'y - \beta'X'y - y'X\beta + \beta'X'X\beta$$

$$= \boldsymbol{y'y} - \boldsymbol{2\beta'X'y} + \beta'X'X\beta \qquad \text{(B 1.8)}$$

Because **β'X'y** is a (1 X 1) matrix or a scalar, and its transpose (β'X'y)' = y'Xβ is the same scalar. The least squares estimators must satisfy:

$$\frac{\partial L}{\partial \boldsymbol{\beta}}_{\hat{\beta}} = -2\boldsymbol{X'y} + 2\boldsymbol{X'X\hat{\beta}} = 0$$

which simplifies to

$X'X\hat{\boldsymbol{\beta}} = X'y$ (B 1.9)

Equation (B 1.7) is the matrix form of the least squares normal equations. It is identical to equation (B 1.4). To solve the normal equations, multiply both sides of equation (B 1 .7) by the inverse of **X'X**. Thus the least squares estimator of β is

$$\hat{\boldsymbol{\beta}} = (\boldsymbol{X}'\boldsymbol{X})^{-1}\boldsymbol{X}'\boldsymbol{y} \quad \text{(B 1.10)}$$

It is easy to see that the matrix form of the normal equations is identical to the scalar form. Writing out equation (B1 .7) in detail, we obtain

$$\begin{bmatrix} n & \sum_{i=1}^{n} x_{i1} & \sum_{i=1}^{n} x_{i2} & \cdots & \sum_{i=1}^{n} x_{ik} \\ \sum_{i=1}^{n} x_{i1} & \sum_{i=1}^{n} x_{i1}^2 & \sum_{i=1}^{n} x_{i1}x_{i2} & \cdots & \sum_{i=1}^{n} x_{i1}x_{ik} \\ \vdots & \vdots & \vdots & \cdots & \vdots \\ \sum_{i=1}^{n} x_{i1} & \sum_{i=1}^{n} x_{ik}x_{i1} & \sum_{i=1}^{n} x_{ik}x_{i2} & \cdots & \sum_{i=1}^{n} x_{ik}^2 \end{bmatrix} \begin{bmatrix} \hat{\beta}_0 \\ \hat{\beta}_1 \\ \vdots \\ \hat{\beta}_k \end{bmatrix} = \begin{bmatrix} \sum_{i=1}^{n} y_{i1} \\ \sum_{i=1}^{n} x_{i1}y_i \\ \vdots \\ \sum_{i=1}^{n} x_{ik}y_i \end{bmatrix} \quad \text{(B 1.11)}$$

If the indicated matrix multiplication is performed, the scalar form of the normal equations, [i.e., Equation (B 1.4)], will result. In this form, it is easy to see that **X'X** is a (p X p) symmetric matrix and **X'y** is a (p X 1) column vector. Note the special structure of the **X'X** matrix. The diagonal elements of **X'X** are the sums of the squares of the elements in the columns of **X**, and the off-diagonal elements are the sums of cross-products of the elements of the columns of **X**. Furthermore, note that the elements of **X'y** are the sums of cross-products of the columns of **X** and the observations $\{y_i\}$.

The fitted regression model is

$$\hat{\boldsymbol{y}} = \boldsymbol{X}\hat{\boldsymbol{\beta}} \quad \text{(B 1.12)}$$

Illustration of Regression Equations

To illustrate the formation of regression equation, a 2^3experiment was considered with 4 center points. The matrix X represents the different combinations of experimental runs whereas vector y represents the outcomes (responses) of these experiments (Montgomery, 2004).

$$X = \begin{bmatrix} 1 & -1 & -1 & -1 \\ 1 & 1 & -1 & -1 \\ 1 & -1 & 1 & -1 \\ 1 & 1 & 1 & -1 \\ 1 & -1 & -1 & 1 \\ 1 & 1 & -1 & 1 \\ 1 & -1 & 1 & 1 \\ 1 & 1 & 1 & 1 \\ 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 \end{bmatrix} \qquad y = \begin{bmatrix} 32 \\ 46 \\ 57 \\ 65 \\ 36 \\ 48 \\ 57 \\ 68 \\ 50 \\ 44 \\ 53 \\ 56 \end{bmatrix} \qquad \text{(B 1.13)}$$

This gives

$$X^{'}X = \begin{bmatrix} 12 & 0 & 0 & 0 \\ 0 & 8 & 0 & 0 \\ 0 & 0 & 8 & 0 \\ 0 & 0 & 0 & 8 \end{bmatrix} \qquad X^{'}y = \begin{bmatrix} 612 \\ 45 \\ 85 \\ 9 \end{bmatrix} \qquad \text{(B 1.14)}$$

As $X^{'}X$ is a diagonal matrix, its inverse is also diagonal and the least square estimates of regression coefficients is

$$\hat{\beta} = (X^{'}X)^{-1}X^{'}y = \begin{bmatrix} 1/12 & 0 & 0 & 0 \\ 0 & 1/8 & 0 & 0 \\ 0 & 0 & 1/8 & 0 \\ 0 & 0 & 0 & 1/8 \end{bmatrix} \begin{bmatrix} 612 \\ 45 \\ 85 \\ 9 \end{bmatrix} = \begin{bmatrix} 51.00 \\ 5.62 \\ 16.62 \\ 1.12 \end{bmatrix} \qquad \text{(B 1.15)}$$

Thus the regression equation can be written as:

$y = 51.00+5.62X_1+16.62X_2+1.12X_3$ (B 1.16)

'D' Optimality Criterion of Optimisation

For design optimality, there are several criteria. The most widely used one is the D-optimality criterion. A design is said to be D-optimal if $\left| X^{'} X \right|$ is maximized. This means that the optimal design matrix (say X^{*}) contains the n experiments which maximize the determinant of (X'X). In other words, the n runs span the largest possible volume in the experimental region.

To explain this, following basic example has been considered with two factors (X_1 and X_2) at three levels (-1, 0, 1). The possible experimental runs are shown in Table B 2:

Table B 2 Experimental runs for two factors three levels experiments

Exp. No.	1	2	3	4	5	6	7	8	9
X_1	-1	-1	-1	0	0	0	1	1	1
X_2	-1	0	1	-1	0	1	-1	0	1

For the purpose of illustration, only four possible design matrices were evaluated and compared them according to 'D' criteria. The four selected subsets in the matrix notation can be written as:

$$\gamma_1 = \begin{bmatrix} -1 & -1 \\ 0 & 0 \\ 1 & 1 \end{bmatrix} \quad \gamma 2 = \begin{bmatrix} 0 & -1 \\ -1 & 0 \\ 0 & 1 \end{bmatrix} \quad \gamma_3 = \begin{bmatrix} -1 & -1 \\ 1 & 0 \\ 0 & 1 \end{bmatrix} \quad \gamma_4 = \begin{bmatrix} -1 & -1 \\ 1 & -1 \\ -1 & 1 \end{bmatrix} \qquad \text{(B 1.17)}$$

Fig B1 shows the above combinations.

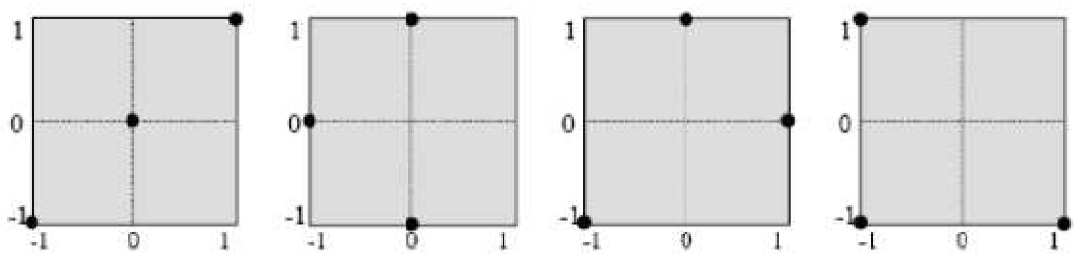


Fig B 1 Distribution of design matrices according to equation (B 1.17)

To determine the quantity $|X'X|$ for γ_2, $(X'X)$ can be determined as:

$$(X'X) = \begin{bmatrix} 1 & 1 & 1 \\ 0 & -1 & 0 \\ -1 & 0 & 1 \end{bmatrix} \begin{bmatrix} 1 & 0 & -1 \\ 1 & -1 & 0 \\ 1 & 0 & 1 \end{bmatrix} = \begin{bmatrix} 3 & -1 & 0 \\ -1 & 1 & 0 \\ 0 & 0 & 2 \end{bmatrix} \quad \text{(B 1.18)}$$

which gives

$|X'X|$=4.

Similarly the determinant for other combinations of experimental runs can be found out. Table B 3 shows the determinants obtained for the four designs considered.

Table B 3 Determinants for different experimental designs

Design	$\lvert X'X \rvert$
γ_1	0
γ_2	4
γ_3	9
γ_4	16

If we compare the outcome of this investigation, it is obvious that design γ_4 has the highest determinant and therefore is the best D-optimal design. The selected candidates of γ_4 are all located on the corners of experimental region. All designs which investigate three out of the four possible corners have a determinant of 16 that also span the biggest area over the experimental region, as described above. Hence γ_4 in this case can be regarded as the best D optimal design.

APPENDIX C

Load verses load line displacement curve and J integral R curve for Different process parameters

Load verses load line displacement curve for Different process parameters

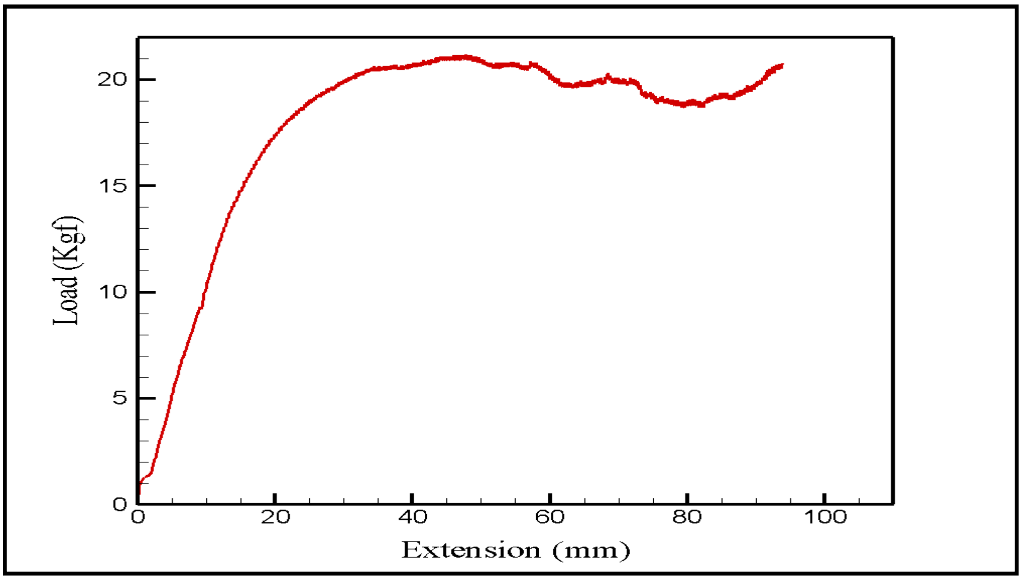


Fig C 1 Load verses load line displacement curve for process parameter 220 37 Fan

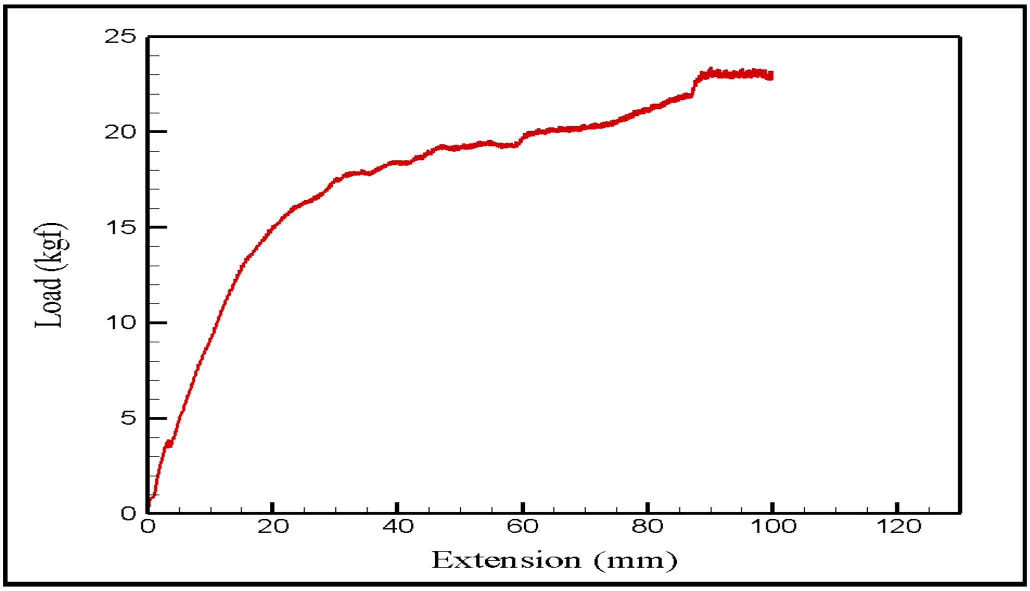


Fig C 2 Load verses load line displacement curve for process parameter 210 37 Water

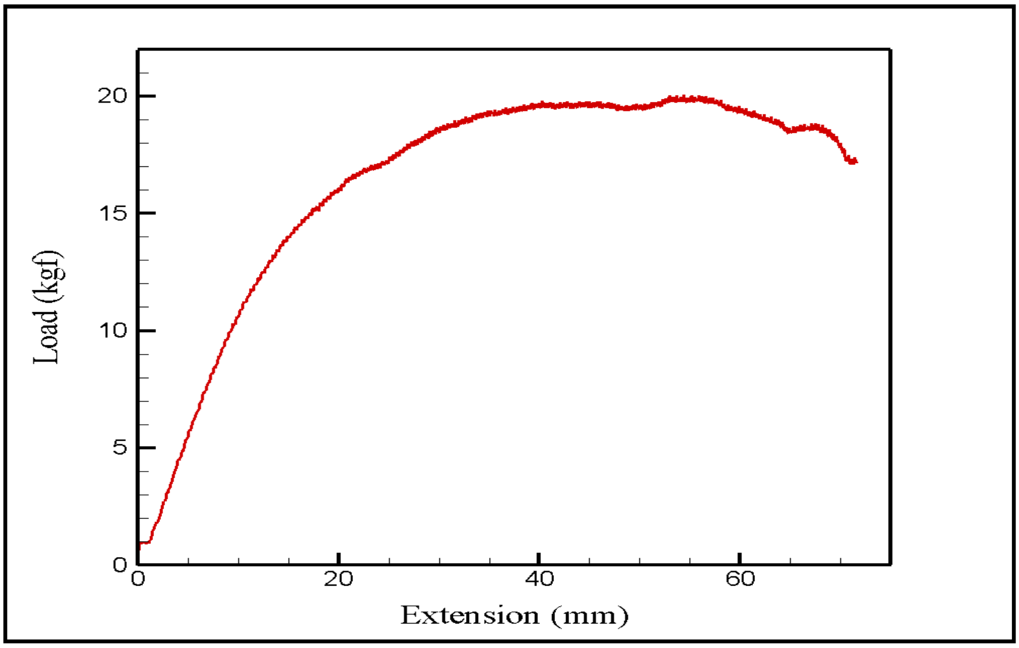


Fig C 3 Load verses load line displacement curve for process parameter 210 42 Fan

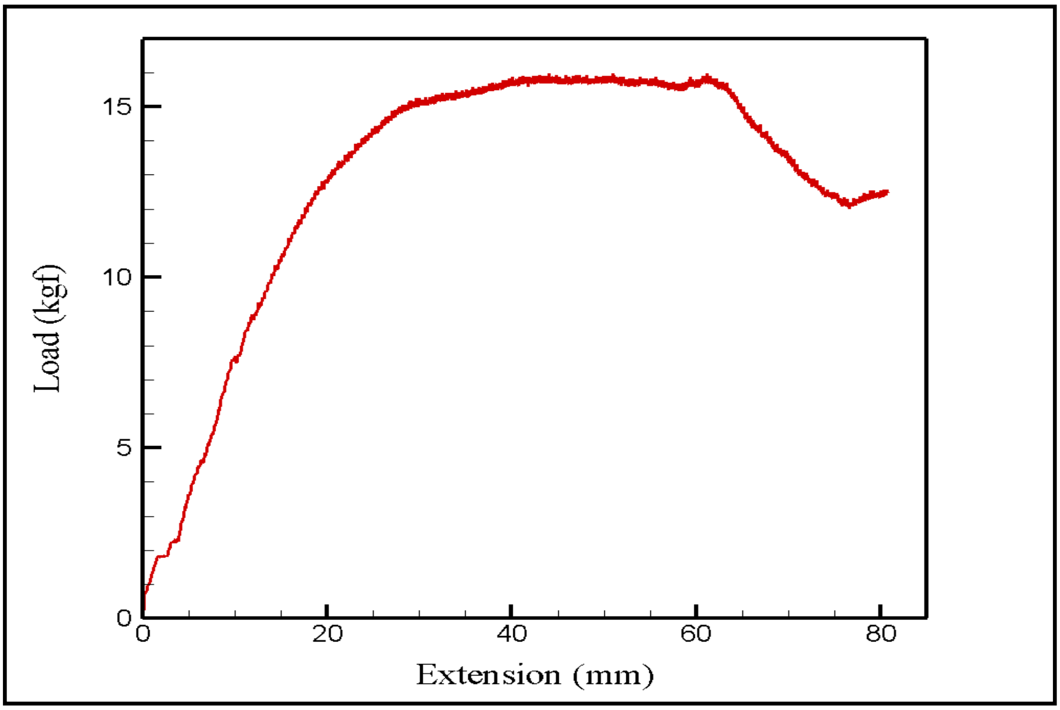


Fig C 4 Load verses load line displacement curve for process parameter 220 32 SA

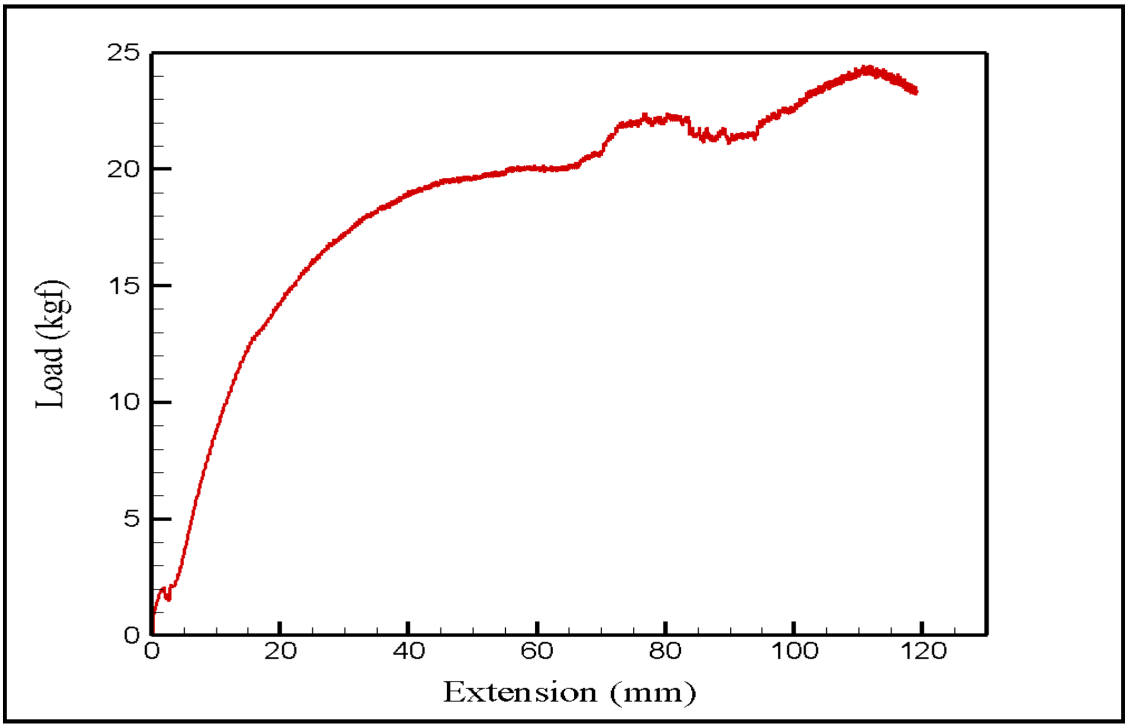


Fig C 5 Load verses load line displacement curve for process parameter 220 32 Water

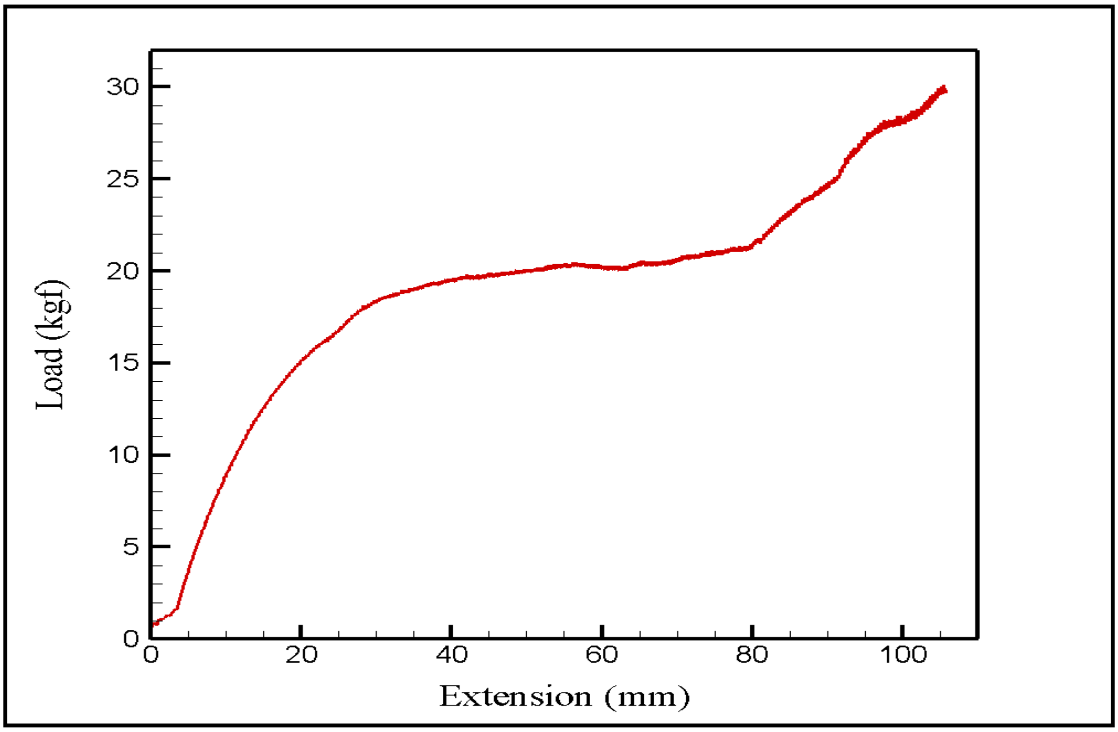


Fig C 6 Load verses load line displacement curve for process parameter 220 37 Fan

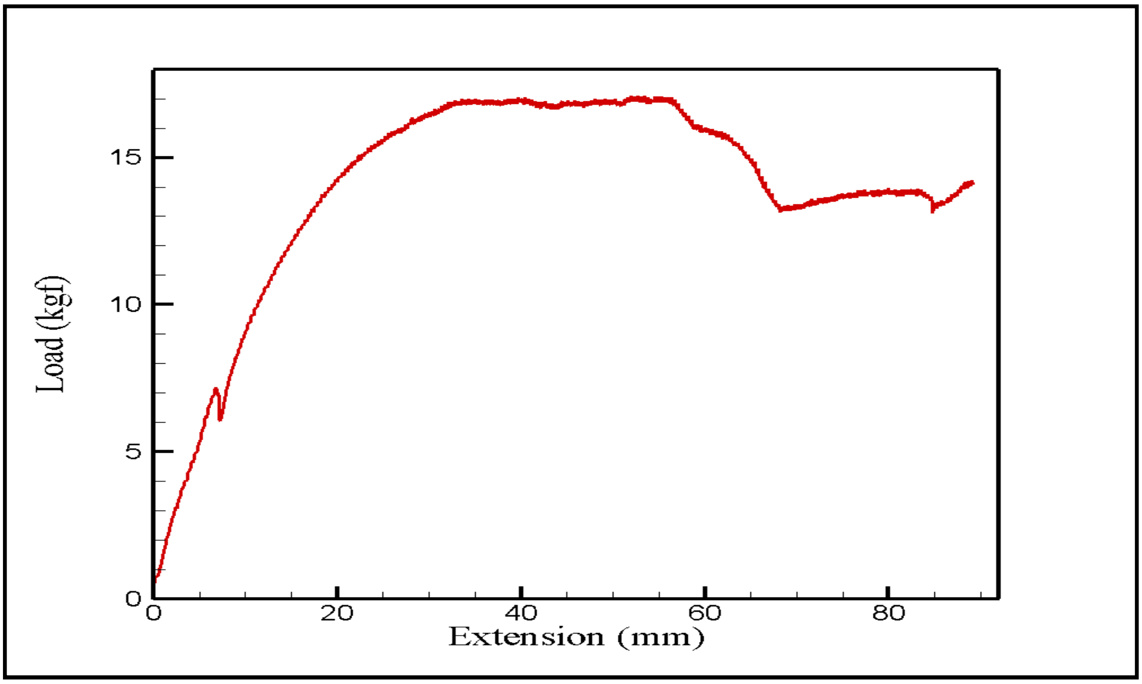


Fig C 7 Load verses load line displacement curve for process parameter 220 42 SA

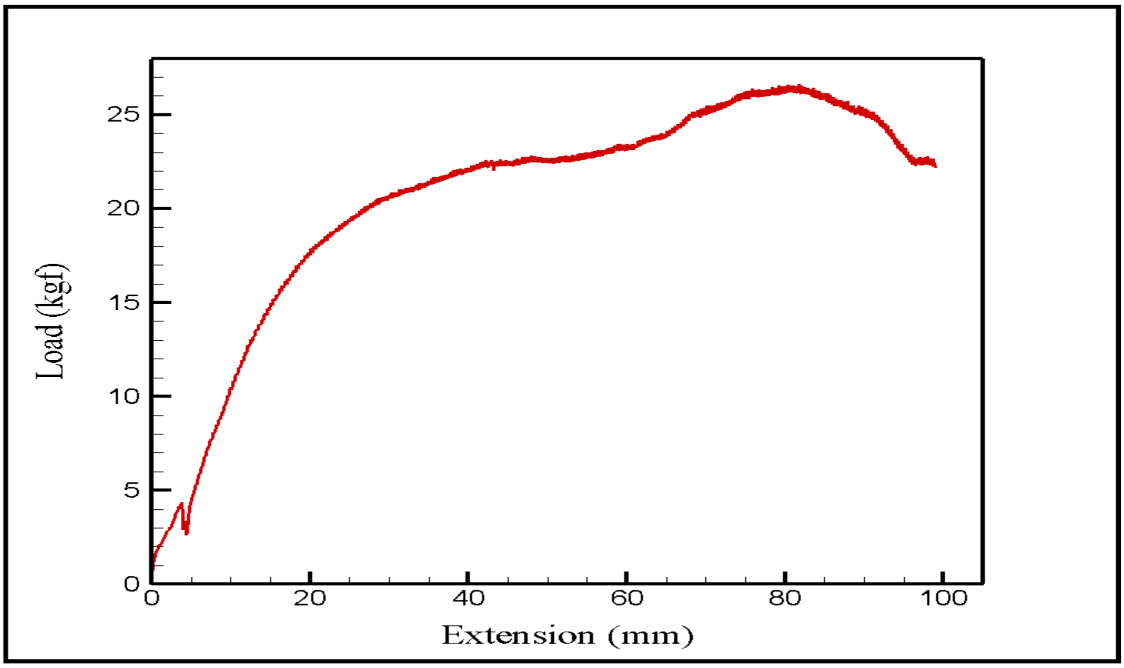


Fig C 8 Load verses load line displacement curve for process parameter 220 42 Water

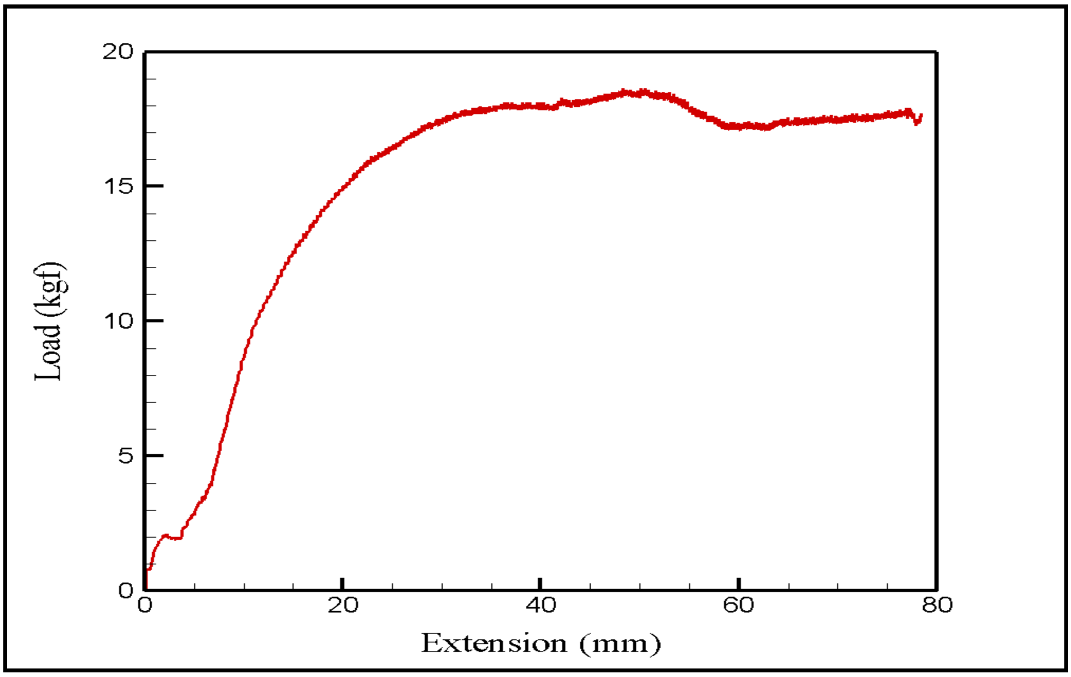


Fig C 9 Load verses load line displacement curve for process parameter 230 32 Fan

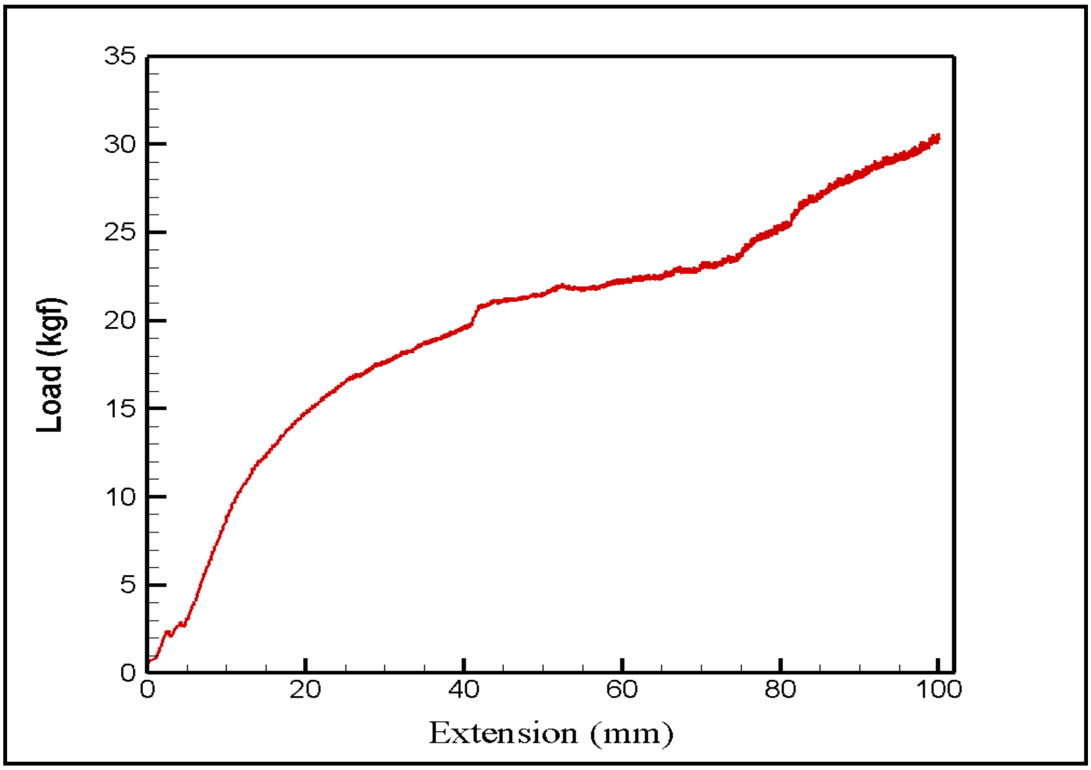


Fig C 10 Load verses load line displacement curve for process parameter 230 37 SA

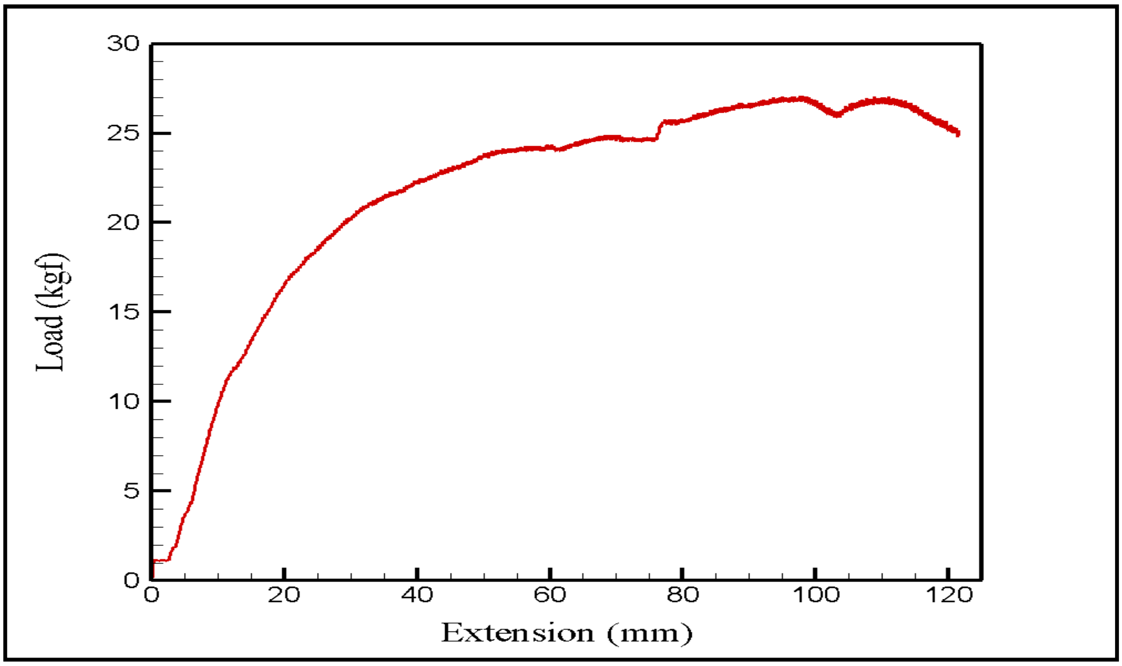


Fig C 11 Load verses load line displacement curve for process parameter 230 37 Water

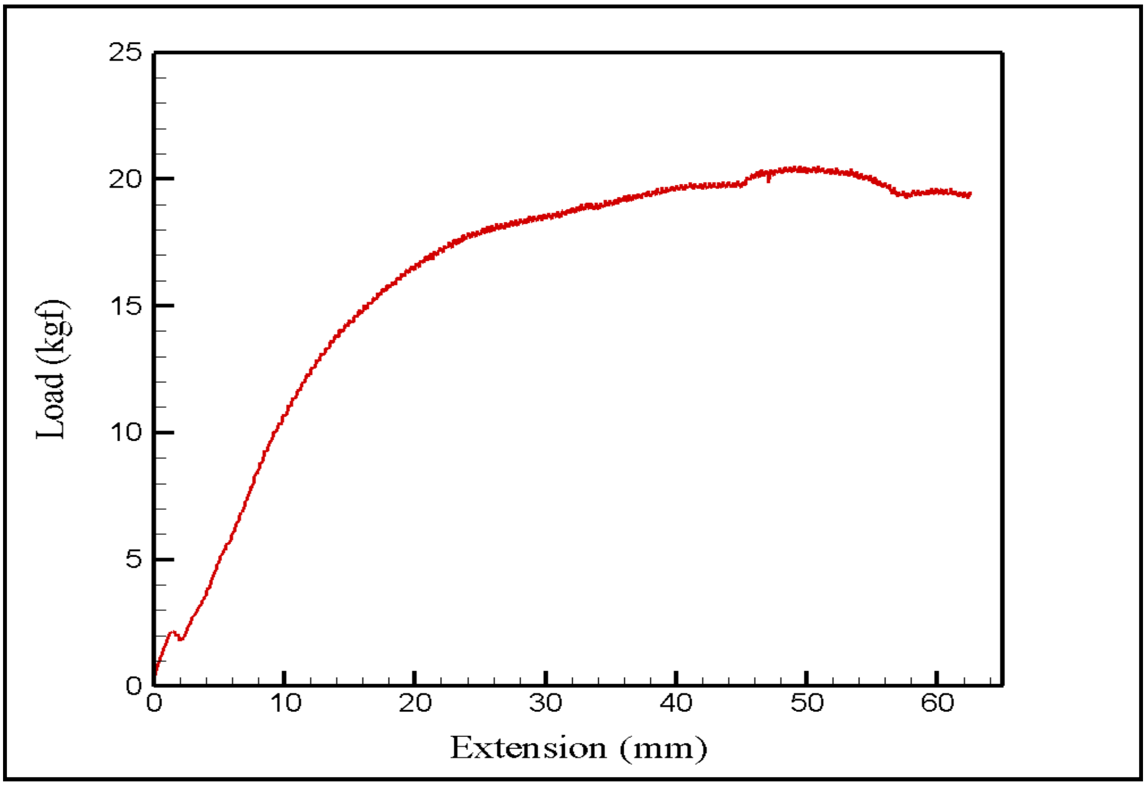


Fig C 12 Load verses load line displacement curve for process parameter 230 42 Fan

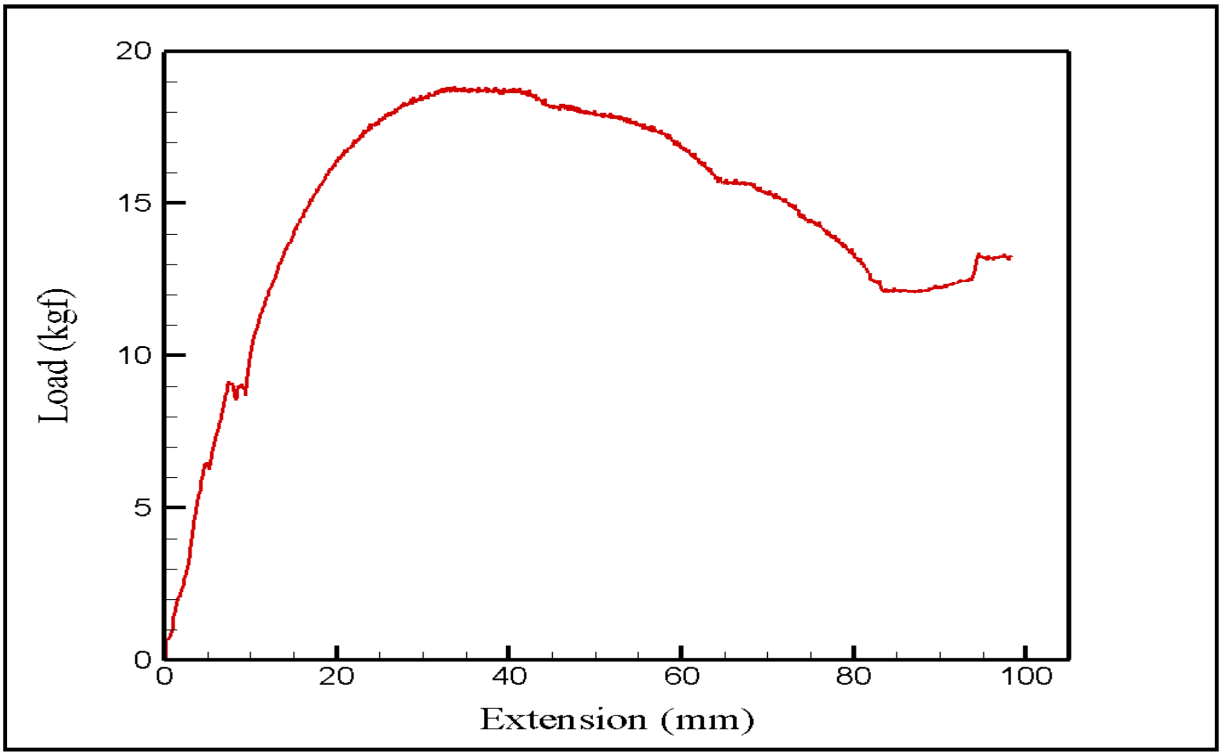


Fig C 13 Load verses load line displacement curve for process parameter 220 37 Fan

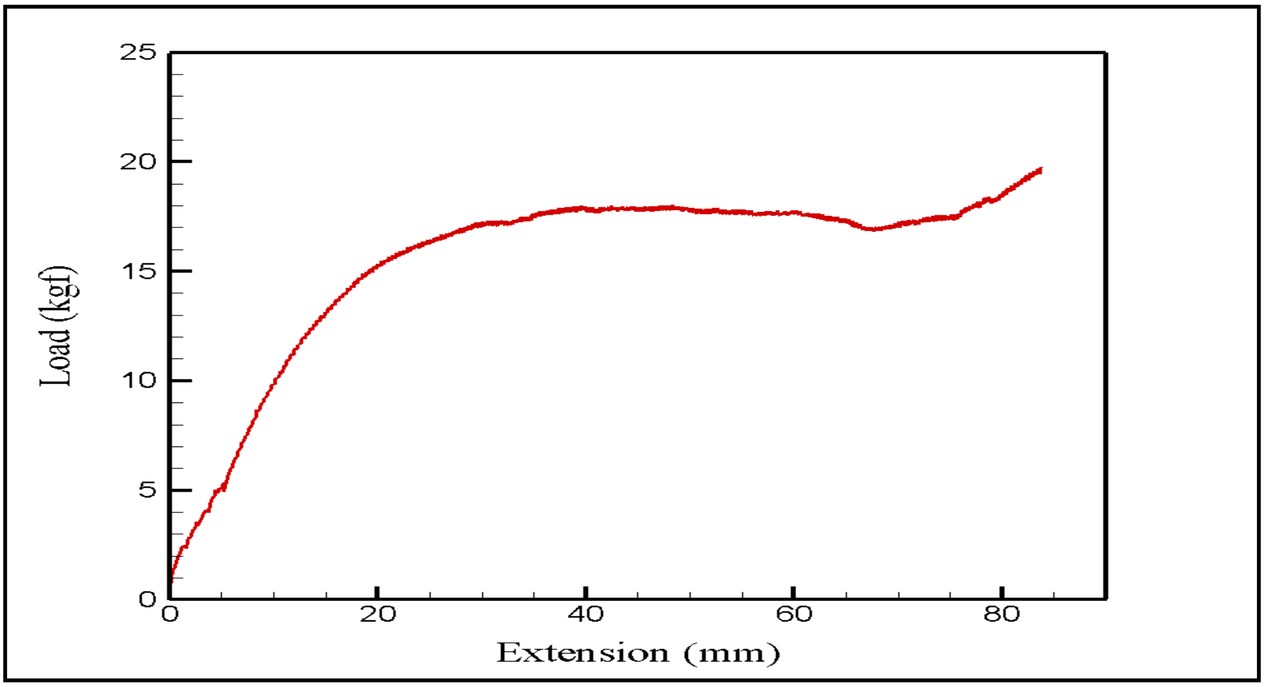


Fig C 14 Load verses load line displacement curve for process parameter 210 32 Fan

J integral R curve for different process parameter

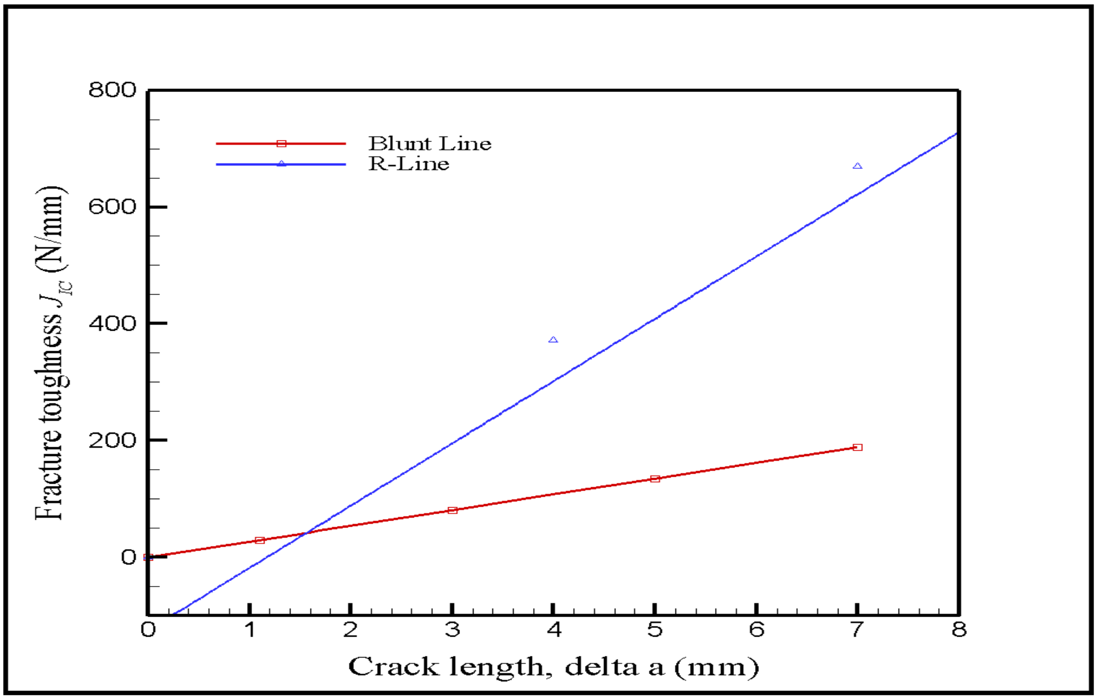


Fig C 15 *J* integral *R* curve for process parameter 220 37 Fan

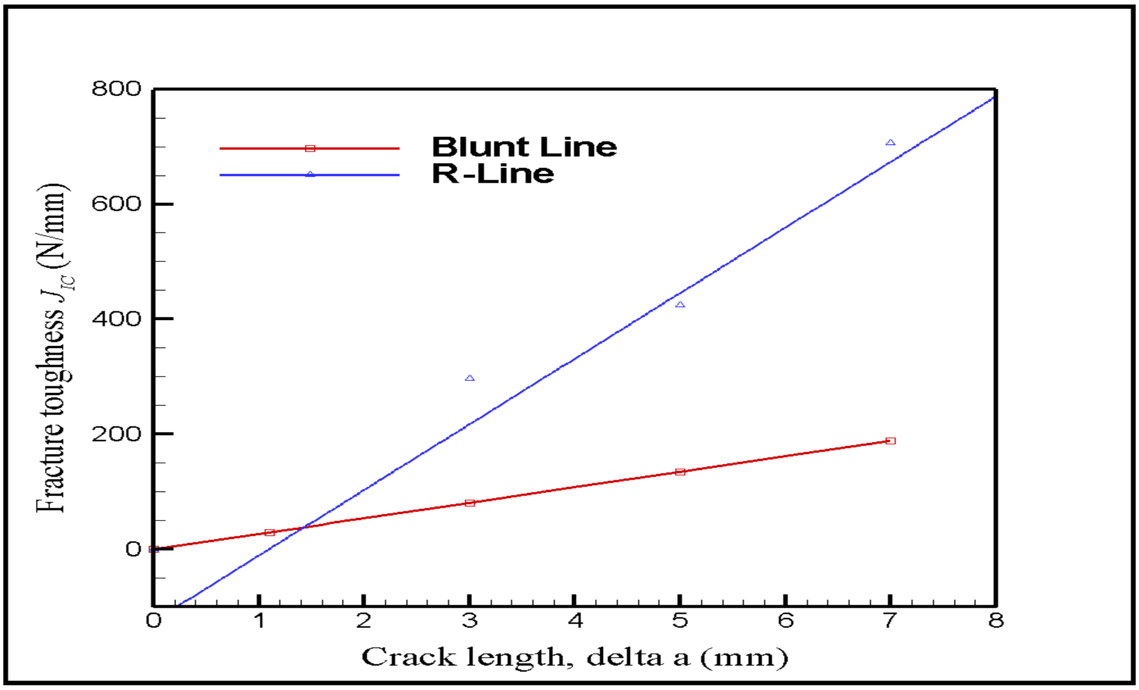


Fig C 16 *J* integral *R* curve for process parameter 210 37 Water

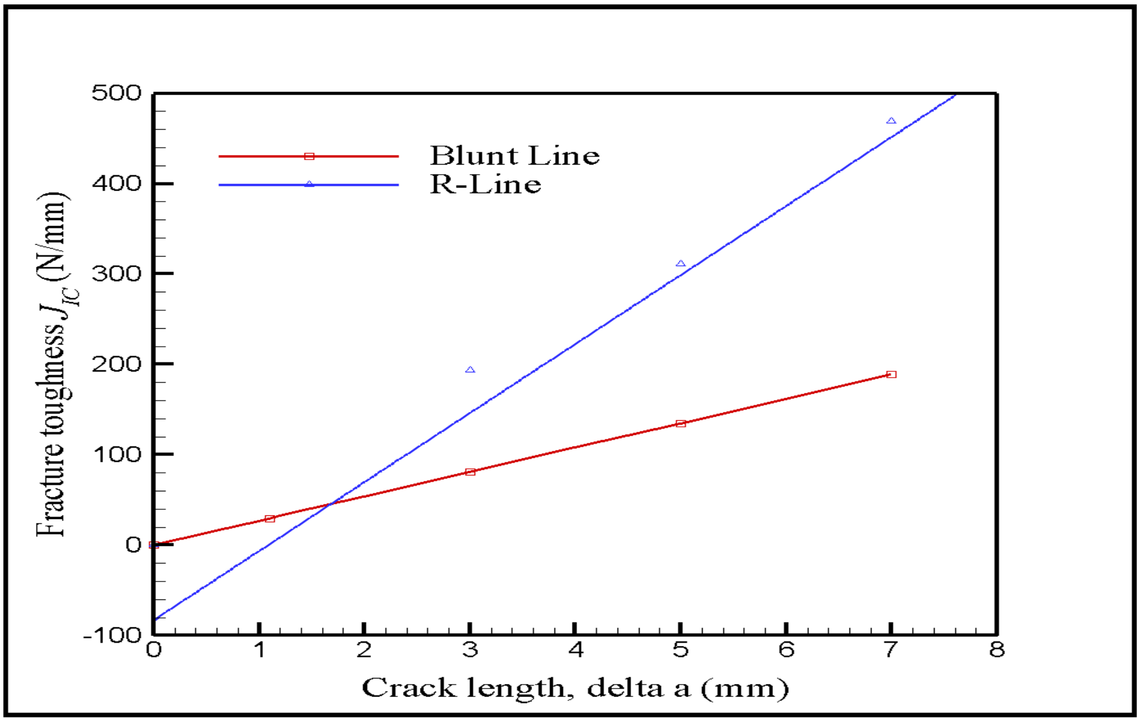


Fig C 17 *J* integral *R* curve for process parameter 210 42 Fan

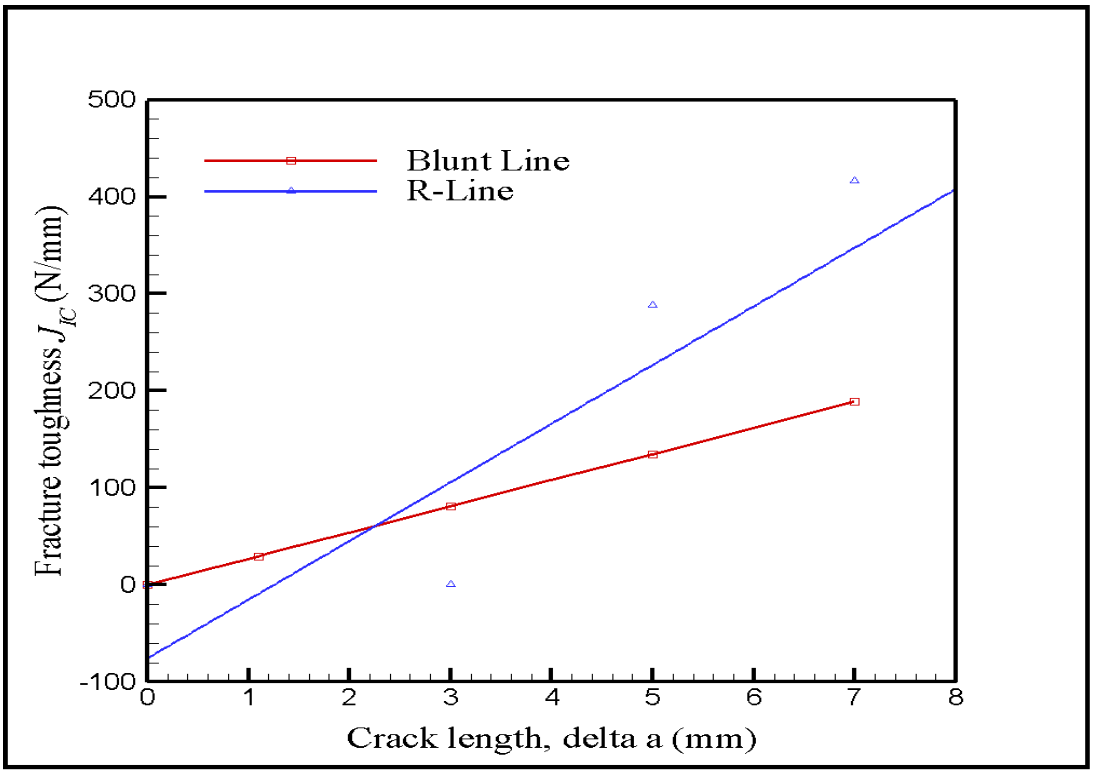


Fig C 18 *J* integral *R* curve for process parameter 220 32 SA

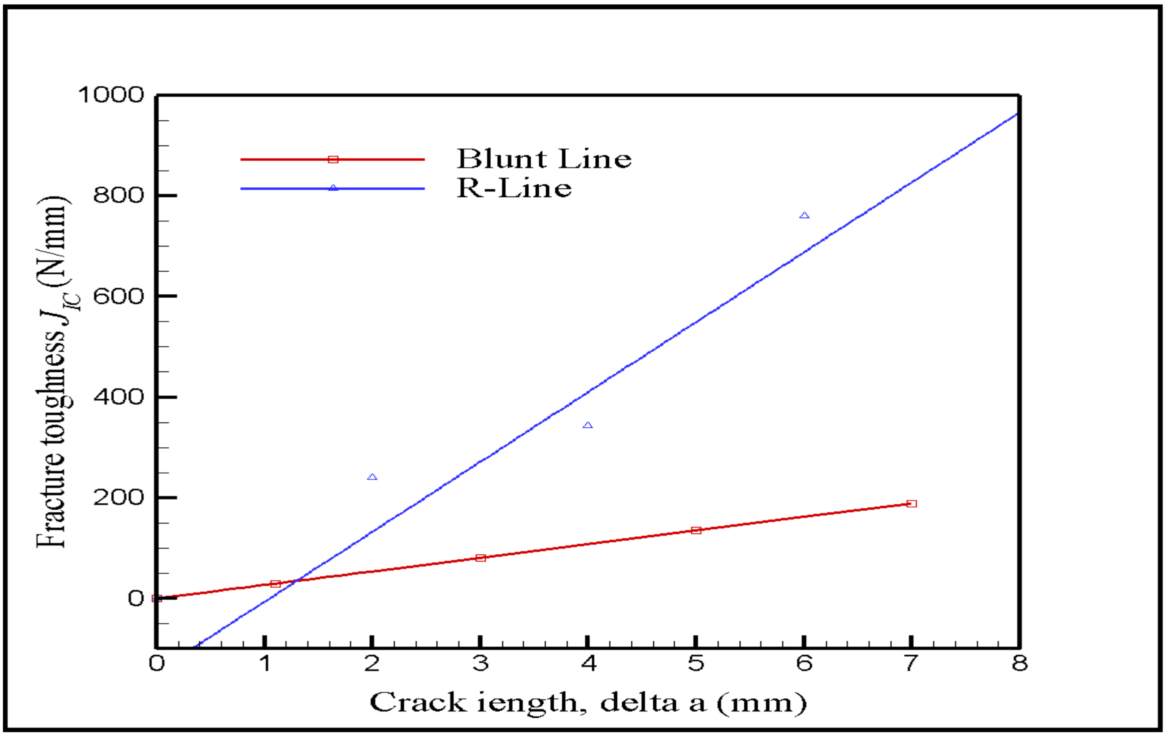


Fig C 19 *J* integral *R* curve for process parameter 220 32 Water

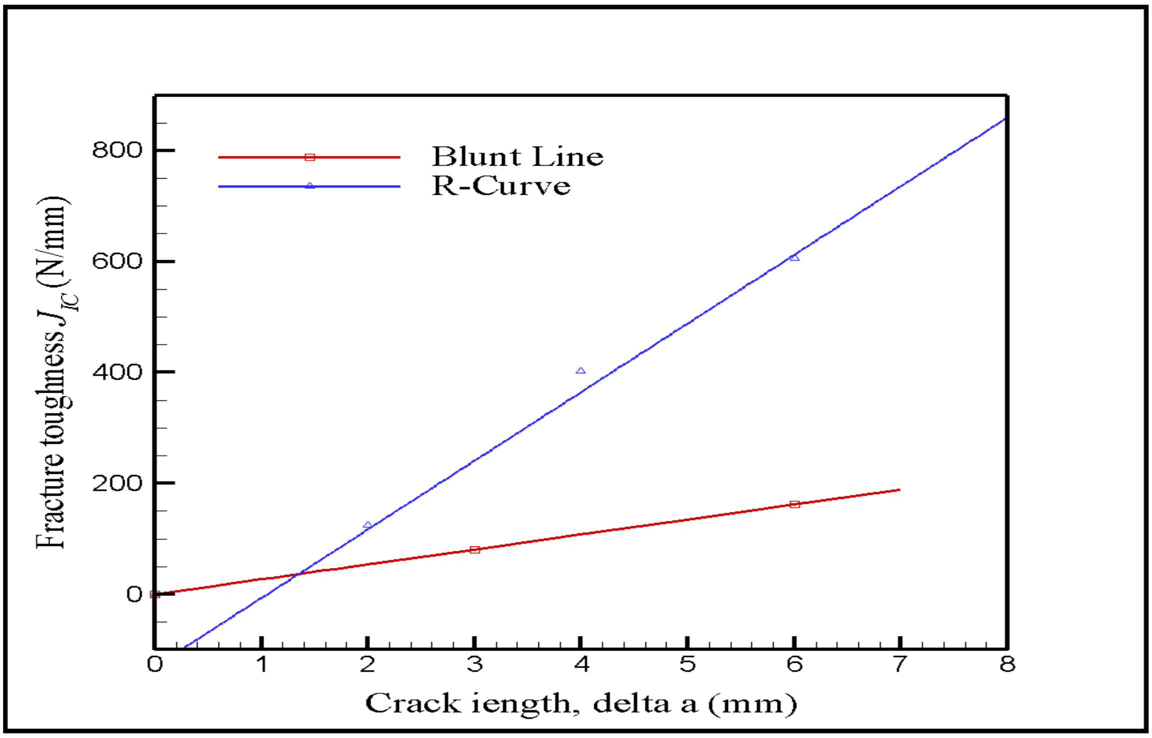


Fig C 20 *J* integral *R* curve for process parameter 220 37 Fan

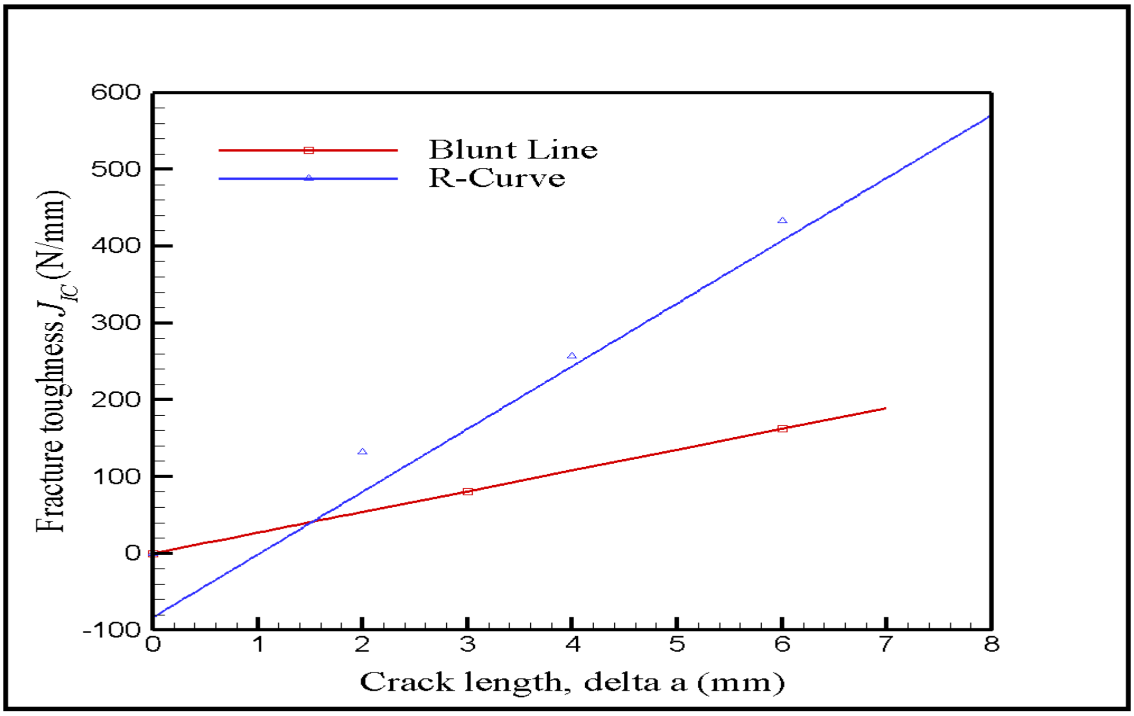


Fig C 21 *J* integral *R* curve for process parameter 220 42 SA

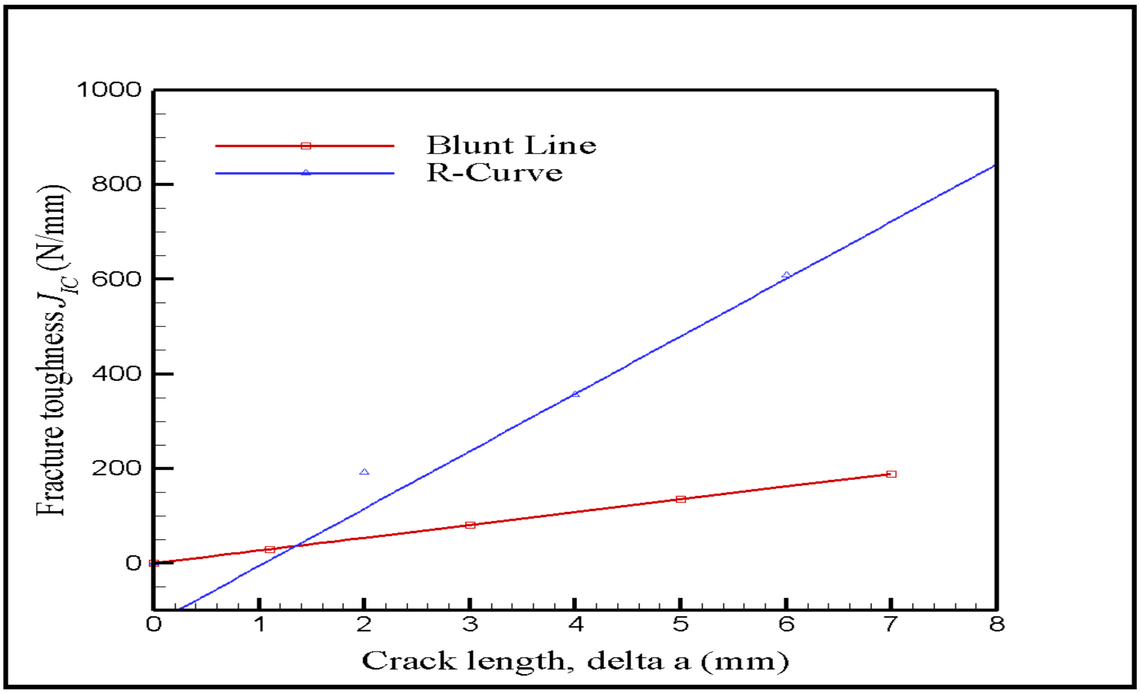


Fig C 22 *J* integral *R* curve for process parameter 220 42 Water

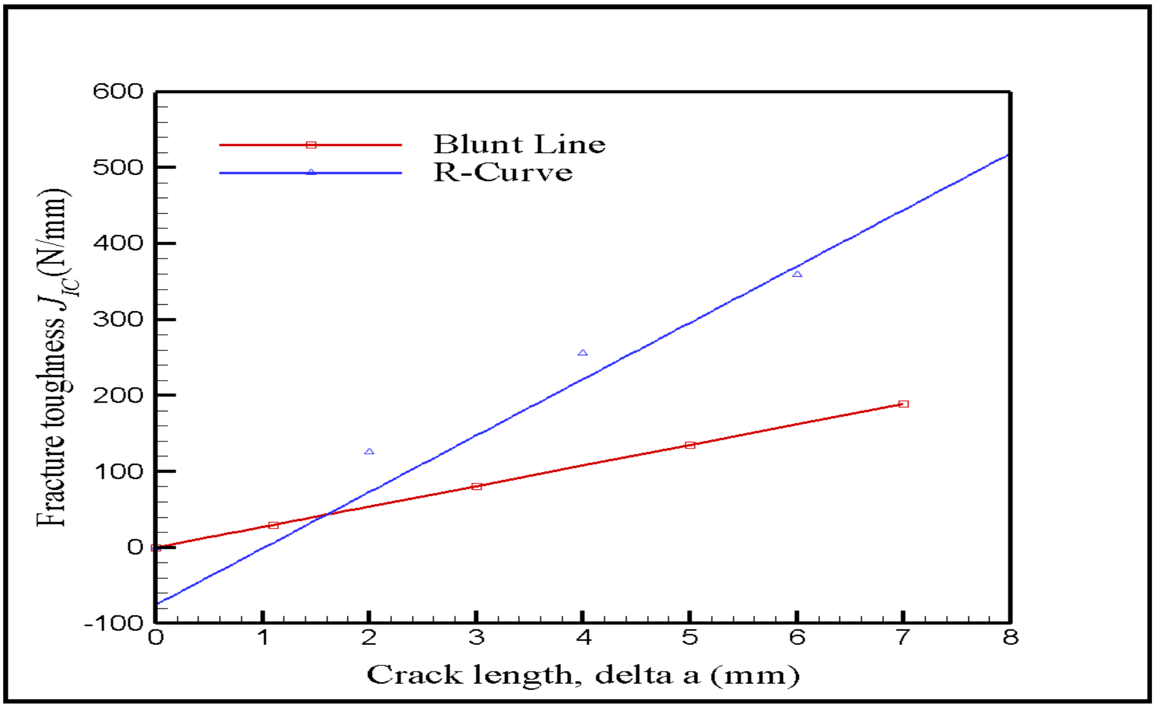


Fig C 23 *J* integral *R* curve for process parameter 230 32 Fan

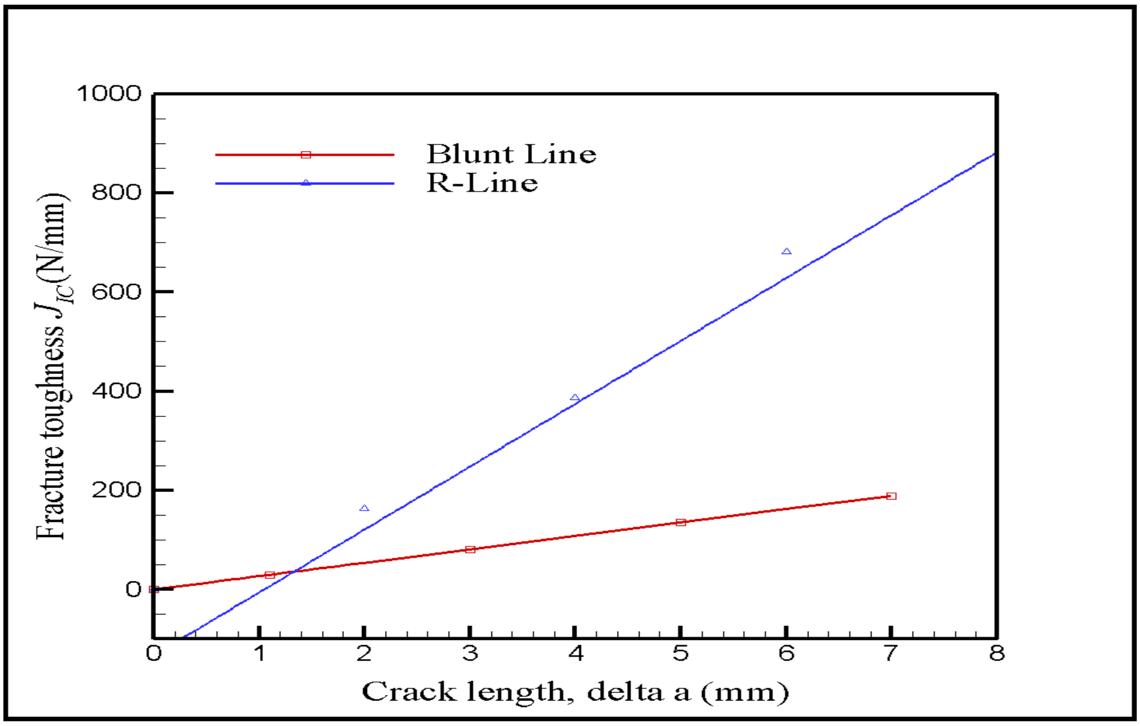


Fig C 24 *J* integral *R* curve for process parameter 230 37 SA

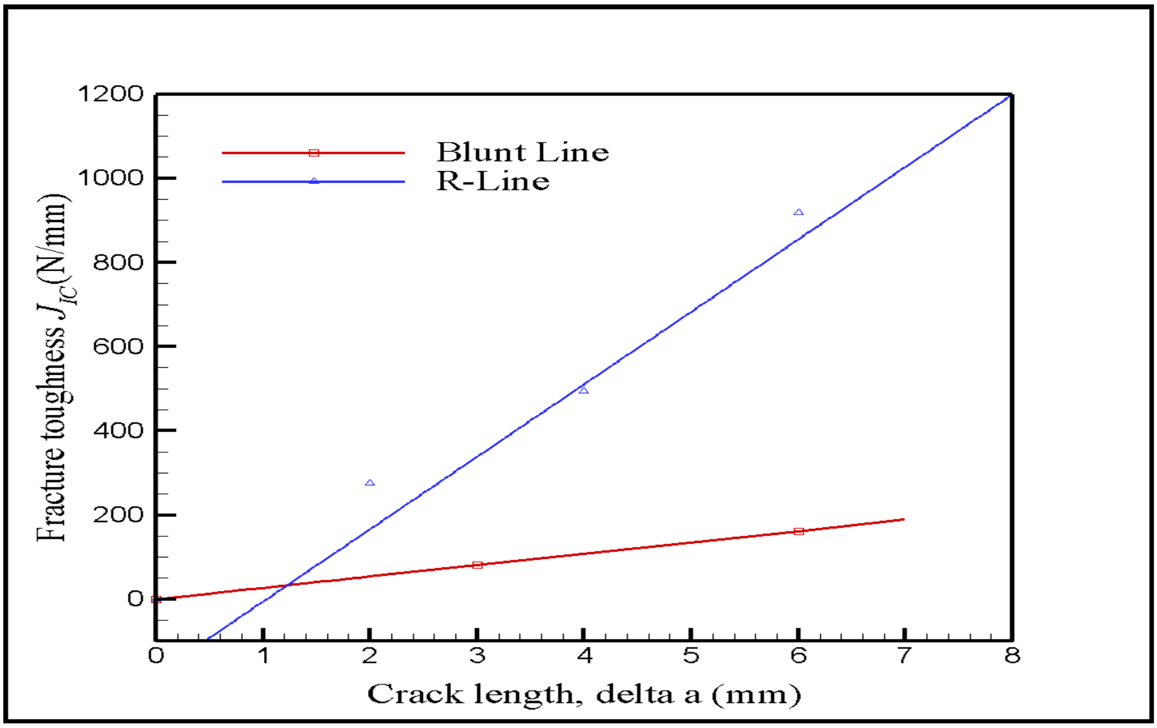


Fig C 25 *J* integral *R* curve for process parameter 230 37 Water

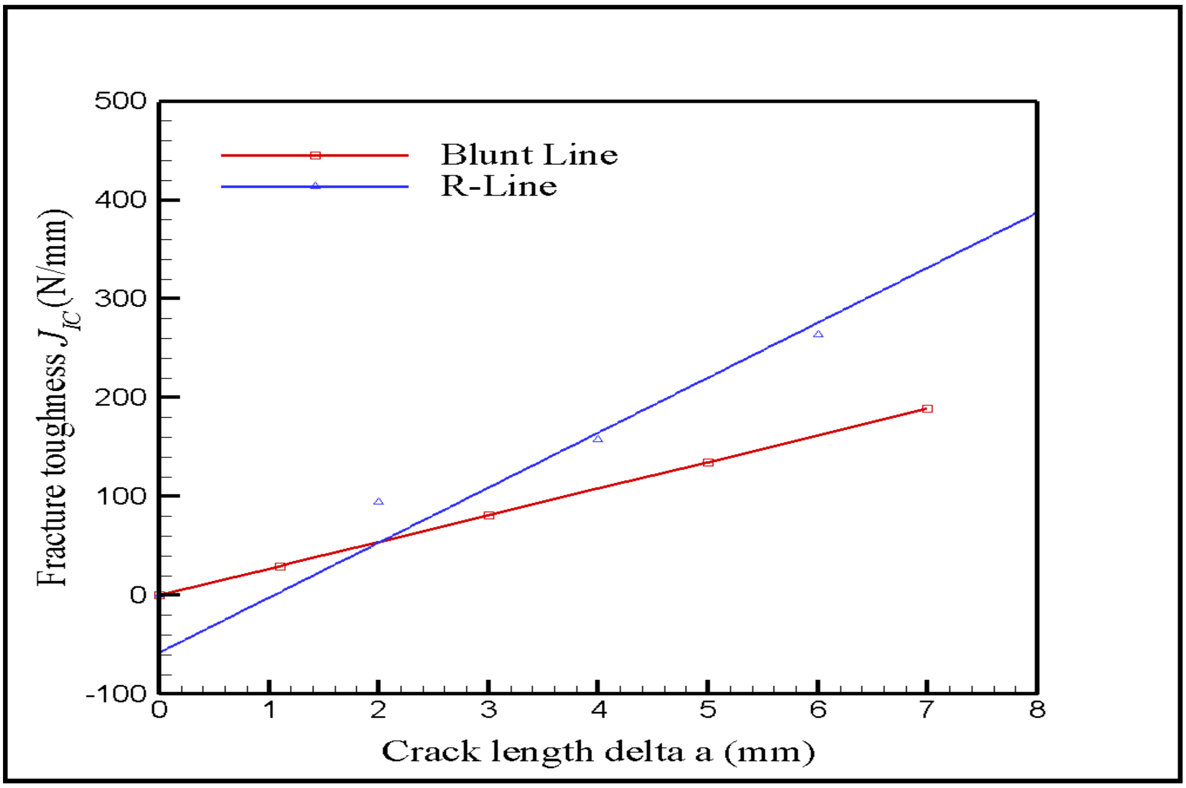


Fig C 26 *J* integral *R* curve for process parameter 230 42 Fan

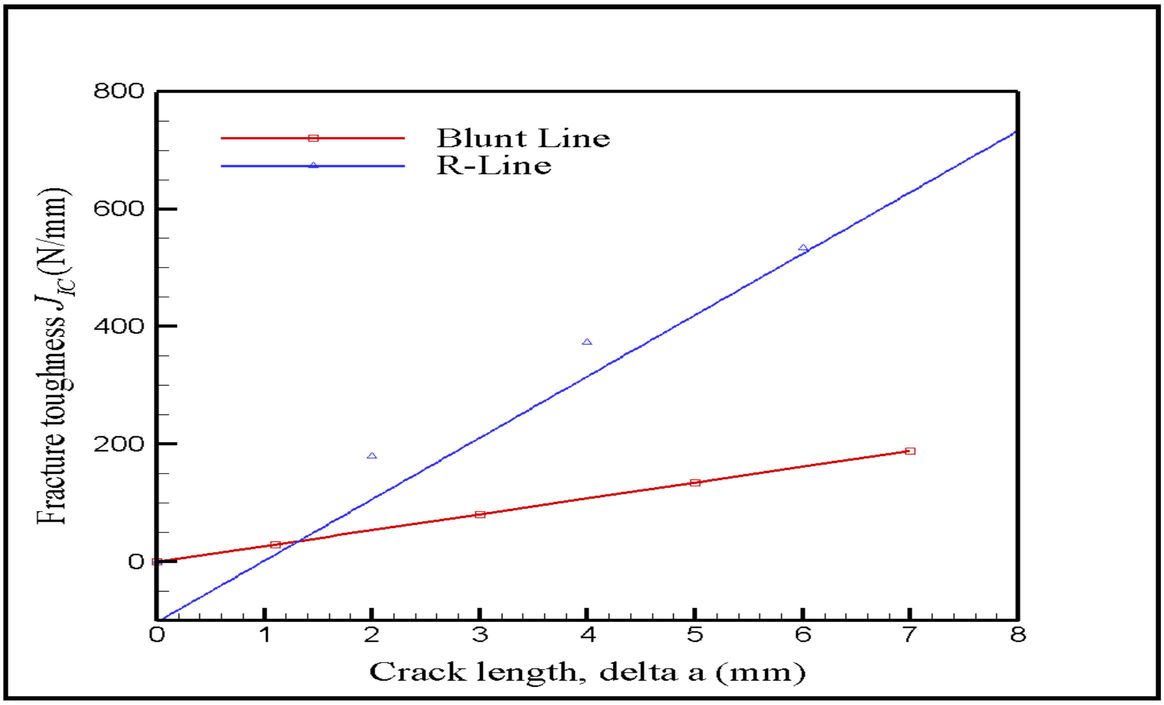


Fig C 27 *J* integral *R* curve for process parameter 220 37 Fan

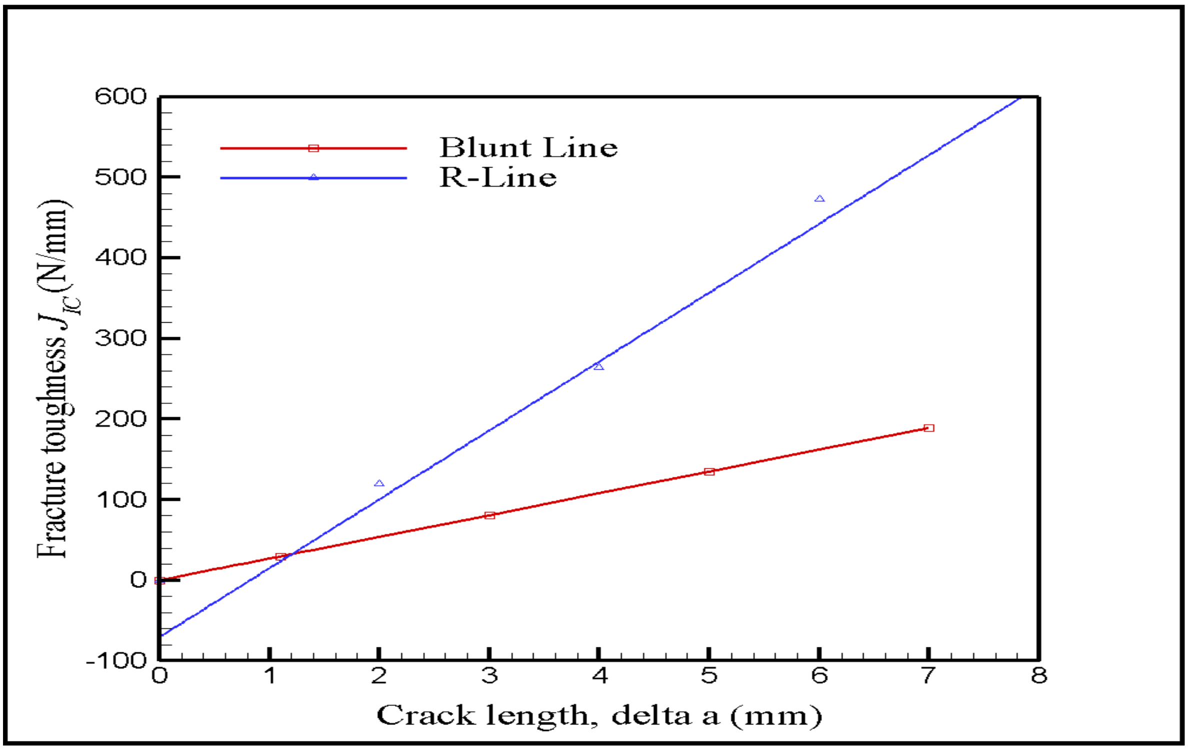


Fig C 28 *J* integral *R* curve for process parameter 210 32 Fan

Appendix D

Specifications of machines and equipments used for the thesis work

Rotational moulding machine

Make	Vinodrai Inc, Jalna (INDIA)
Model	Clamshell type single arm biaxial machine
Model no	EN- Lab model

Computerized universal testing machine

Make	Deepak poly-plast private limited, Gujarat (INDIA)
Capacity	1000 kg

Izod impact testing machine

Make	Deepak poly-plast private limited, Gujarat (INDIA)
Capacity	10 J test with only hammer

Melt flow indexer

Make	Dynisco polymer test, Franklin (US)
Model	LMI 4000

Differential scanning calorimeter

Make	Shimadzu (JAPAN)
Model	DSC-60

Dynamic mechanical analyzer

Make	T A Instruments, Delaware (USA)
Model	DMA Q800

BRIEF BIOGRAPHY OF THE SUPERVISOR

Prof. D M Kulkarni is Professor in Mechanical Engineering Department and Dean Administration in BITS Pilani, K K Birla Goa Campus. He has his Masters from IIT Kharagpur and PhD from BITS Pilani. He has 16 years of teaching and 2 years of industrial experience. His research area is Fracture Mechanics, Biomechanics and Advanced Finite Element Analysis. He has research grants from Industry and DST in the area of Biomechanics. He has published over 40 research papers in the international journal / conferences of repute. He has also published a book on Engineering Graphics with AutoCAD, which is implemented by many autonomous Universities. He is certified Instructor of American Society of Mechanical Engineering (ASME) to design the courses and train the industrial professionals in India. He has conducted hands-on training sessions in Advanced Finite Element Analysis at industrial sites in Maharashtra and Gujarat. He has been awarded 2 times (2011 & 2012) for Faculty Excellence for his contribution in Teaching, Research and Administration. He was also awarded by BITS alumni for 'Distinguished Faculty Award (2010).

BRIEF BIOGRAPHY OF THE CANDIDATE

PL. Ramkumar is a lecturer in the Mechanical Engineering department at the Birla Institute of Technology and Science, Pilani, KK Birla Goa campus, Goa, India. He has over 9 years of teaching experience. He has been teaching core mechanical courses like Computer aided design, Mechanics of solids, Computer aided analysis and design, Product design etc. He completed his graduation in Mechanical Engineering from Madurai Kamaraj University in 2003. He obtained M. Tech degree in Computer Aided Design from Anna University, Tamilnadu, India in 2008 securing **University rank** with **Gold medal**. His area of research includes experimental analysis of manufacturing processes, process modeling, computer aided analysis and optimization. He has published over 6 research papers in the international journal / conferences of repute.