CREEP AND THERMAL RATCHETING OF SOFT MATERIALS UNDER COMPRESSION

by

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MANUSCRIPT-BASED THESIS PRESENTED TO ÉCOLE DE TECHNOLOGIE SUPÉRIEURE IN PARTIAL FULFILLMENT FOR THE DEGREE OF DOCTOR OF PHILOSOPHY
Ph.D.

MONTREAL, OCTOBER 03, 2018

ÉCOLE DE TECHNOLOGIE SUPÉRIEURE
UNIVERSITÉ DU QUÉBEC

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ACKNOWLEDGMENTS

This PhD thesis would not be possible without the guidance and expertise of my research director, Dr. Abel-Hakim Bouzid. It has been a pleasure pursing my doctoral thesis under his supervision and I wish to convey my utmost gratitude to him. His knowledge and appetite for research in pressure vessel and piping domain along with his mentorship helped me in attaining my PhD.

With immense pleasure, I wish to express my thanks to my parents, Kanthabhabha and Jeya, for their outstanding support, care, encouragement and guidance throughout my life and specifically during my PhD. A big part of my reason to pursue a PhD belongs to my parents, to whom I am forever indebted. Also, I am thankful for the support of my beloved sister, Sathya and my extended family.

My colleagues, in Static and Dynamic Sealing Laboratory, have been an excellent fellow researchers and friends. I would like to appreciate all the help and advice I received from my friends – Linbo Zhu, Ali Oweimer, Valentin Fort, Zijian Zhao, Ali Vafadar, Amir Hasrak, Mohammad Esouilem and Mehdi Kazeminia.

Finally, my special thanks to all the technicians of the department of Mechanical Engineering at ÉTS with special mentions Serge Plamondon and Michel Drouin. I would extend my appreciation to Eric Marcoux and his colleagues at the machine shop for their collaboration and technical support throughout the course of the experimental work.

Desire, Dedication and Endurance will lead to Success.

FLUAGE ET CUMUL DE DEFORMATION PAR CYCLAGE DE TEMPERATURE DES MATÉRIAUX MOUX

Rahul Palaniappan KANTHABHABHA JEYA

RÉSUMÉ

Une accroissement rapide de l'utilisation des matériaux polymères et PRFV par rapport aux matériaux métalliques conventionnels dans la production de composants de réservoirs sous pression et de tuyauterie est devenue une tendance mondiale. Cependant, les normes de conception de l'ASME ainsi que leurs équivalentes européennes pour les matériaux non métalliques ne sont pas spécifiques aux matériaux et, dans l'ensemble, suivent vaguement les normes des matériaux métalliques. Contrairement aux composants PVP métalliques, les composants polymères sont d'âge récent, ce qui limite les données statistiques disponibles sur ce type de matériaux. Parmi les composants PVP polymères, le polychlorure de vinyle (PVC) et le polyéthylène haute densité (PEHD) en constituent la majorité. L'excellente résistance à la corrosion, la légèreté et la facilité de fabrication font de ces deux matériaux polymères le remplacement idéal aux structures métalliques corrosives et lourdes. L'objectif de cette recherche est de caractériser le fluage à long terme et le rochet thermique des matériaux mous.

L'étude s'articule autour de l'analyse du comportement de rochet thermique des matériaux polymériques PVP sélectionnés, y compris les matériaux de joints à base de PTFE et à base de fibres. Le but principal de cette thèse est de caractériser les matériaux polymériques utilisés dans les assemblages à brides boulonnées. Pour l'étude sur les performances des matériaux sélectionnés à haute température, des expérimentations méticuleuses ont été réalisées à l'aide de bancs d'essais équipés de capteurs de haute précision. Comme la plage de température de fonctionnement des matériaux est très différente de celle des joints considérés, les évaluations thermiques des matériaux de brides et des joints ont été effectuées séparément. Tous les matériaux sélectionnés ont été soumis à différentes charges de compression, différentes températures et étaient dans certains cas pré-exposés au fluage pour évaluer l'interaction avec le phénomène de rochet thermique. Les tests de caractérisation des polymères ont été réalisés avec des échantillons en forme d'anneau. De plus, des essais grandeur nature des brides en PVC et PEHD de classe 150 de NPS 3 ont été effectués pour évaluer la relaxation à court terme et les résultats sont comparés à ceux obtenus avec des modèles numériques utilisant la méthode des éléments finis.

Les résultats ont permis de mieux comprendre la vulnérabilité des polymères et des matériaux mous en général au phénomène de rochet thermique. L'étude sur le comportement des matériaux sélectionnés aux cycles thermiques a mis en évidence l'intensification des dommages par fluage sur les matériaux, dont l'ampleur varie en fonction de chaque matériau. De plus, le rochet thermique modifie d'autres propriétés fondamentales des matériaux des brides et des joints, comme le module de fluage et le coefficient de dilatation thermique.

Mots-clef: Assemblage de bride boulonné, Rochet thermique, PEHD, PVC, PTFE et joints à base de fibres, Fluage.

CREEP AND THERMAL RATCHETING OF SOFT MATERIALS UNDER COMPRESSION

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ABSTRACT

A rapid increase in the utilization of polymer and FRP materials over conventional metallic materials in the production of pressure vessel and piping components has become a global trend. However, factually, the design standards of ASME and its European counterpart for non-metallic materials are not material specific and as a whole vaguely follow the standards of metallic materials. Contrary to metallic PVP components, polymer components are of recent ages and this limits the statistical data available on the materials. Among polymer PVP components, polyvinylchloride (PVC) and high-density polyethylene (HDPE) constitute the majority. The inherent excellent corrosion resistance, lightweight and ease of manufacturing make these two polymer materials the ideal replacement over corrosive and heavy metallic structures. The objective of this research is to characterize the long-term creep and thermal ratcheting of soft materials.

The research revolves around the analysis of thermal ratcheting behavior of the selected PVP polymer materials including PTFE and fiber based gasket materials. The core intent of this thesis is to characterize polymer materials used in bolted flange connections. For the investigation of thermal ratcheting performance of the selected materials, meticulous experimentations were carried out using test rigs equipped with high accuracy sensors. As the operating temperature range of selected flange materials are much different from the considered gaskets, the thermal ratcheting evaluation of flanges and gaskets were performed, separately. All the selected materials were subjected to different compressive loads, various ratcheting temperature and few pre-exposure creep to evaluate the thermal ratcheting phenomenon. The characterization tests of polymers were performed with ring shaped samples. Furthermore, full-scale tests of NPS 3 Class 150 PVC and HDPE flanges were conducted to evaluate the short-term relaxation and the results are compared to the finite element counterpart.

The results provided significant insight on the vulnerability of polymer and soft materials to thermal ratcheting phenomenon. The study on the behavior of selected materials to thermal cycling highlighted the intensification of creep damage on the materials, the magnitude of which varied depending on each material. In addition, thermal ratcheting alters other fundamental properties of flange and gasket materials such as creep modulus and coefficient of thermal expansion.

Keywords: Bolted flange joint, Thermal ratcheting, HDPE, PVC, PTFE and Fiber based gaskets, Creep.

TABLE OF CONTENTS

LIST OF TABLES

Page

LIST OF FIGURES

Page

LIST OF ABREVIATIONS

ASME	American Society of Mechanical Engineering
CNA	Compressed Non-Asbestos
ePTFE	Expanded Polytetrafluoroethylene
FEM	Finite Element Method
HOBT	Hot Blowout Test
LVDT	Linear Variable Differential Transformer
PPI	Plastic Pipe Institute
PTFE	Polytetrafluoroethylene
PVP	Pressure Vessel and Piping
PVT	Pressure Vessel Technology
UGR	Universal Gasket Ring
vPTFE	Virgin Polytetrafluoroethylene

LIST OF SYMBOLS AND UNITS OF MEASUREMENTS

MASS

mg	Milligram
Kg	Kilogram

ANGL

Deg	Degree
Rad	Radian

Length/Displacement

m	Meter
mm	milimeters
μs	Micro strain
in	Inches

TEMPERATURE

C	Centigrade
F	Fahrenheit

TIME

S	Second
H	Hours

Pressure/Stress

MPa	Mega Pascal
GPa	Giga Pascal
Psi	Pounds per Square Inches

INTRODUCTION

Modern engineering is striving towards cost effective, environmental friendly and durable products, and the domain of the pressure vessel and piping is no exception. This drive lead to the development of polymeric material components, most notably polyvinylchloride and high-density polyethylene. In little over half a decade from its first commercial production, these two materials have taken total dominance in regards to the polymeric piping industry. The growth of these materials is so humungous that it became an integral part of the urban infrastructure. More than 90% of sewage and domestic water networks in developed countries has been replaced by either of these two plastic materials. The two materials were developed around the same time, in the early 1950's and had not seen a huge up raise in commercial application until the last three decades. British chemists Eric Fawcett and Reginald Gibson developed the first stable solid form of polyethylene in 1935. In 1953, German scientists Karl Ziegler (belonging to renowned Max Planck Institute) and Erhard Holzkamp invented the High-density polyethylene (HDPE), for which Ziegler won the Noble for chemistry in 1963. The first commercial HDPE pipe was manufactured in 1965.

The high-density polyethylene is a versatile material, the utilization of which ranges from simple plastic bolt to nuclear power plant service water lines. In our Static and Dynamic Sealing Laboratory, the researches are extensive focused on sealing products with particular focus to pressure vessel and piping applications. Hence, this research is narrowed down to understand the behavior of PVC and HDPE materials used in bolted flange joint application.

Figure 0. 1 HDPE piping products

In general, the bolted flanges joints are subjected to compression and so an excellent compressive resistance property is necessary. Bolted flange joint is one of the most commonly used method of connecting pipes and pressure vessel components. In simple terms, the purpose of a bolted flange joint is to provide continuity for the flow of fluid through a piping system. In case of the HDPE, two adjoining stub flanges are held together by applying a compressive force through a pair of metallic rings and a set of bolts and nuts as shown in figure 0.2. Even though there are few alternate methods to connect mating pipe sections together, bolted flanges are the preferred solution because they can facilitate for easy inspection and evaluation of pipes in a large network of connections. Unlike HDPE, PVC flanges can be directly thread and bolted together without any additional metallic rings.

Figure 0. 2 HDPE Bolted Flange joint

Figure 0. 3 On-field HDPE flange installation

Bolted flange joint is one of a key source of leakage failure in the pressure vessel and piping system. All bolted flanges are subjected to a design compressive force to hold the pipe sections together and to avoid any loss of the confined fluid as leakage to the surrounding. Bolted flange joints are the most vulnerable point in any pressurised system and hence one of the most critical link between structures.

Generally, HPDE pipe flanges does not require the use of gasket between their mating surfaces, however, in some special cases soft gasket is used to ensure a tighter seal. The use of soft gasket is a must in metallic and metal to plastic pipe flange connections. Consequently, three commonly used gasket material were tested namely, expanded and virgin polytetrafluoroethylene and compressed non-asbestos fiber gasket. All the test samples, including gasket and flange materials, are commercially available for purchase. The characterisation of flange and gasket materials are performed separately to analysis the behavior of each material under test conditions.

Before discussing the problem with polymeric bolted flanges, some of the most important attributes are briefly presented. The standout of features of the selected polymer materials are durability, ease of manufacturing and fitting, cost effectiveness and non-hazardous. Both HDPE and PVC are well known for their excellent resistance to chemical and corrosion attacks. As a result of their extensive protection property, these pipes are employed in transportation of highly corrosive and slurry fluids. Consequently, a greater service life for piping systems is achieved with same or lower environmental impact than their metallic counterparts. In case of concrete pipes, low pH environments causes shrinkage and chemical attacks on the piping system, which lower their strength. This problem can be overcome with the use of HDPE and PVC pipes.

Another feature of the selected plastic materials is the ease of manufacture, which is very attractive for the PVP industry especially for making pipes and piping components. The preferred laying lengths for conventional metallic pipes are 6 or 12 meters, limiting the production to manufacture hollow cylinders of short lengths. This causes an increase in transportation cost, in number of pipe fitting on field, in human labour with increased risk of handling errors. Due to the advancement in modern manufacturing techniques, HDPE and PVC pipes of small diameters are produced in rolls of 50 m or more and therefore, they are called flexible pipes. The feasibility in production of a single pipe for longer lengths provides for easy field installation and limits the possibility of leakage failure.

Cost effectiveness is one of the key players in modern industrialization. Most industries are keen on expanding the difference between profit and operational cost margins. The mentioned plastic materials provide for exceptional corrosion protection, thereby reducing the cost of maintenance. Less maintenance means increased product life, which directly decreases the replacement cost. In addition, these plastic pipes are relatively light weighted, which can be manufactured in greater lengths. This particular feature helps in easier ground installation and reduces the number of labours need for the installation. Overall, as a consequence of low maintenance, higher product life and easier installations, these flexible pipes are highly cost effective.

Nowadays, health and environmental impact are of major concern and hence many restrictions, in accordance to various international government and regulatory authorities, are imposed in the selection of materials for use in PVP domain. Primarily, both HDPE and PVC are non-toxic materials, which makes them ideal for domestic water transportation. In addition, both of these materials meet the required international standards for operational safety. Furthermore, these two polymers are best for high sensitive environment as they limit requirements for multiple joint connections thereby decreasing the chances of leakage failure.

Problem Statement

As mentioned earlier, this thesis deals with the characterisation of soft materials, which includes two types of polymeric flange materials (HDPE and PVC) and three kinds of gasket materials (ePTFE, vPTFE and CNA). Even though, all of these materials are selected because of their use in bolted flange joint application, each material is evaluated separately. The reason behind individual analysis of these flange and gasket materials is to show the importance of the thermal ratcheting of each material under specific test conditions for extrapolation to other applications.

A bolted flange joint is one of the many methods of connection between two pipes or between a pipe and a vessel. In pressurised systems, this type of joint is critical, as relaxation due to creep and thermal ratcheting could lead to leakage failure, which can be fatal and hazardous.

Creep is defined as the deformation caused under constant load over a time-period. This phenomenon is extremely common in bolted flanges and it is highly unpreventable. The severity of creep damage is directly proportional to the material temperature. Since this is a time dependent property, it is mandatory to comprehend the creep behavior of the materials, particularly in case of PVP applications. The difference in tensile and compressive creep behaviour is distinct, which is typical of polymeric materials. In addition, the creep resistance of HDPE and PVC are highly important because of their limits in operational temperatures, which are rather low. Gaskets are no exception to creep and in particular Teflon-based PTFE materials. In metallic bolted flange joints, gaskets are established as the primary contributor to creep-relaxation causing loss of bolt tightness and resulting in leakage failure. Although, qualitative and quantitative studies on creep resistance under the influence of tension, compression and temperature of various polymer materials has already been partially treated by fellow researches, none have scrutinised the combined interaction of creep and thermal ratcheting phenomenon.

Thermal ratcheting is defined as the cumulative deformation induced in the material as a result of cycling of temperature. The thermal ratcheting phenomenon is different from mechanical ratcheting where the cumulative damage is caused by load cycling. Thermal ratcheting is usually overlooked in PVP applications because conventional metallic materials have higher temperature resistance. However, this is not the case for polymers and specifically HDPE and PVC, which have low operational temperature limits. As specified previously, the characterisation of flange and gasket materials are performed, separately. This facilitates identification of the test conditions of each material and establish the vulnerability of each material.

Finally, the objective of this PhD thesis is the creep-thermal ratcheting characterisation of polymeric and fiber materials utilized in bolted flange joint application.

Methodology

The characterization of creep, thermal ratcheting and combined creep-thermal ratcheting behavior of all five selected materials were performed using Universal Gasket Rig. For this purpose, extensive experimental tests were carried out in order to obtain quantitative data on the behavior of each material under different imposed test conditions. Schedule 80 HDPE and PVC pipes were precisely cut into ring shaped samples with identical thicknesses by using CMC machine. The research was keenly focused on the compressive creep analysis, which is the primary contributor for bolt load relaxation and consequently initiating failure by leakage.

A combined experimental and numerical approach is employed to analysis the long-term creep behavior of the two flange materials. Since performing a yearlong creep test to grasp the perennial behavior of the material is tiresome and unproductive way of analyzing, a short-term creep evaluation up to several days after the creep transcend to secondary stage is performed at optimal load and temperature conditions. These results provide for defining the creep model, which is required to run numerical simulation and study the relaxation behavior of HDPE and PVC bolted flange joints.

Additionally, experimental investigation on consequence of thermal ratcheting on creep, creep modulus, co-efficient of thermal expansion, percentage of thickness reduction and thermal ratcheting strain were conducted. This approach was adapted mainly due to the fact that the results on the thermal cycling phenomenon is bare minimum and experimental study is the ideal starting point.

To evaluate the numerical simulation results, four full-scale flange creep tests on NPS 3 Class 150 HDPE and PVC materials flanges were conducted. A comparison of experimental and numerical analysis was done and these results acts as the measure of accuracy of the adapted creep model.

Thesis Overview

The contents of this PhD thesis is segregated into seven chapters and a conclusion. The research data are presented in constructive manner for ease of understanding of the reader. The thesis is presented in the “thesis by paper” style of writing.

The first chapter is dedicated to the literature review, which is one of the source of instigation of this research objective. An extensive and qualitative review of existing scientific articles were performed to develop a fundamental and effective problem statement. The literature review helped in understanding the current trend and the state of art conditions of research in PVP domain.

Chapter two elaborates the operational mechanisms of the test rigs and test procedure involved in this characterization research. Since experimentation is the essence of this research, a detailed explanation on the intrigue mechanisms of the machines facilitating the complex creep-thermal ratcheting analysis with highest accuracy is vital.

Third chapter presents the first published journal article on the thermal ratcheting behavior of Teflon based PTFE gaskets. The results of this paper concentrates on the response of expanded and virgin PTFE gaskets to thermal ratcheting phenomenon. The influence of thermal cycling on the percentage of thickness reduction and co-efficient of thermal expansion are also presented.

Fourth chapter revolves around the characterization of high-density polyethylene material subjected to creep and thermal ratcheting. The paper is published in Polymers MDPI journal. This article details the fundamental behavior of HDPE under thermal ratcheting, as no scientific article was ever published in elaborating this phenomenon of the material.

The subsequent chapters five and six present the papers on the combined creep-thermal ratcheting interaction for three types of gasket and HDPE materials, respectively. Both papers are under review in their respective journals. The amplification of creep damage due to thermal ratcheting is thoroughly presented in them.

Chapter seven compares the numerical and experimental approach on creep-relaxation of HDPE and PVC bolted flange joints. The article describes the effectiveness of experimental test results in constructing the creep models, which are in turn used for predicting creep over a longer period. A full-scale bolted flange tests were conducted to validate the numerical results.

Finally, a broad conclusion highlighting the best of results and elaborating the necessity for improved or updated plastic bolted flange design standard is presented.

CHAPTER 1

LITERATURE REVIEW

1.1 Introduction

This chapter is dedicated to consolidate the existing research work on five selected materials and to explain how the current trend of research assisted in optimizing the objectives of this PhD thesis. Literature review is essential for any type of research, which acts as a pool of information that can assist in developing and understanding a problem statement. In the context of this research work, the literature review helped from narrowing down the research goals to characterising the test conditions for each selected materials. In the modern digital era, the quest for the search of published scientific information has been made easy, which helps in moulding the thesis goals.

A substantial amount of research has been carried out on understanding the mechanical behavior of HDPE, PVC, ePTFE, vPTFE and CNA materials. The research have predominantly focused on the tensile, fatigue, flexure strength and manufacturing techniques as these properties are of high importance in designing structures subjected to different operational conditions. For the sake of simpler writing, the article title, journal name and details are mentioned in the reference section only. In the main text, the first and second author name with their corresponding year of publication is used for one or two authors, and only the first author is used in case of multiple authors.

From a proper scrutinizing of scientific articles, the thesis objective went down from a broader typifying of polymer bolted flange materials to thermal ratcheting characterisation of HPDE, PVC and Teflon materials. Since the latter is primarily used in gasket application, a fiber-based material was considered to have comparative evaluation. An in-depth review of research articles and the desire to focus on the applications related to bolted joints that uses these materials facilitated the decision to characterise their cited properties under compression. Since all five materials are distinct from each other, a devoted assessment of the prevailing research publications is necessary and hence the following sub-sections briefly detail the state of the art research data published for the chosen materials.

1.2 **Review of publications on HDPE material**

This subsection gives some short and precise information on the list of publications that are focusing on the characterisation of the high-density polyethylene material. As mentioned in the introduction chapter, the research on HDPE material is more than half century old and the characterisation research has varied from general material characterisation to application specific characterisation. Since the review was done on a large volume of articles, only the most appropriate research findings of the scientific articles were concisely provided in the following paragraphs. The research publications on tensile, compressive and flexure strength, creep, HDPE composite, slow crack growth and mechanical properties of recycled HDPE polymers are presented.

The present day researchers focus on the tensile property of different HDPE composite materials, where the thermoplastic HDPE is added with a fiber material to develop a new composite material. Facca et al. (2007) investigated the tensile property of short natural fibre reinforced thermoplastics (NFRT). The research keenly studied the interaction between HDPE thermoplastic and the natural fibre under tensile loading and characterised the load sharing between the two. The scanning electron microscopic image of HDPE/hemp composite is shown in Figure 1.1.

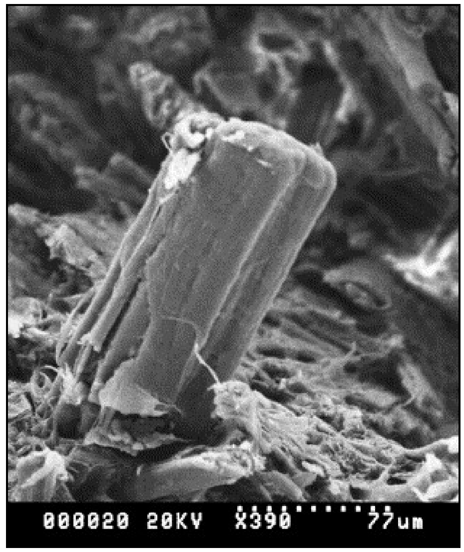

Figure 1. 1 SEM image of HDPE reinforced
with hemp fibre (Facca et al., 2007)

A detailed review on the traction behavior of polymer composites reinforced with natural fibers was done by Ku et al. (2011). The results highlighted that the tensile strength of different HDPE composites found from experimentation and rule of mixture are close to each other. Also, for HDPE composites containing different types of natural fibers, the Halpin-Tsai equation is the most effective method to predict the Young's modulus of composite materials. The authors show how the tensile strength (Figure1.2) of a composite varies with the percentage by weight of fibers in the mixture.

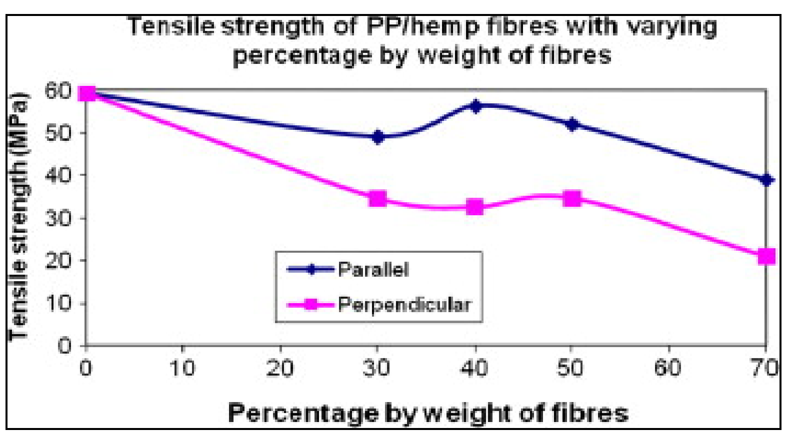

Figure 1. 2 Impact of weight of fibers in the tensile strength
of PP/hemp composite (Ku et al., 2011)

A review of kenaf reinforced polymer composites was carried out by Saba et al. (2015). The research focuses on the evaluation of mechanical property of different polymer materials reinforced by kenaf fibers. Among the various composites combinations reviewed, the article emphasised on 1:1 ratio of kenaf and HPDE resin, which did not show considerable improvement in flexure and tensile properties. Moreover, this composite material demonstrated a reduction in the tensile property at low temperature and an increase when subjected to high temperature.

Bhattacharya and Brown (1985) measured the microstructural changes that initiates slow crack growth in linear polyethylene material. The results show that an instantaneous deformation zone is formed, which grows with constant speed until the beginning of fracture. The researchers discovered that the initial velocity of deformation zone has an activation energy of 100KJ mol^{-1}, which depends on the stress applied. The fibril thinning is the process that controls crack initiation and growth. Some of the SEM images of the damage polyethylene material is presented in Figure 1.3

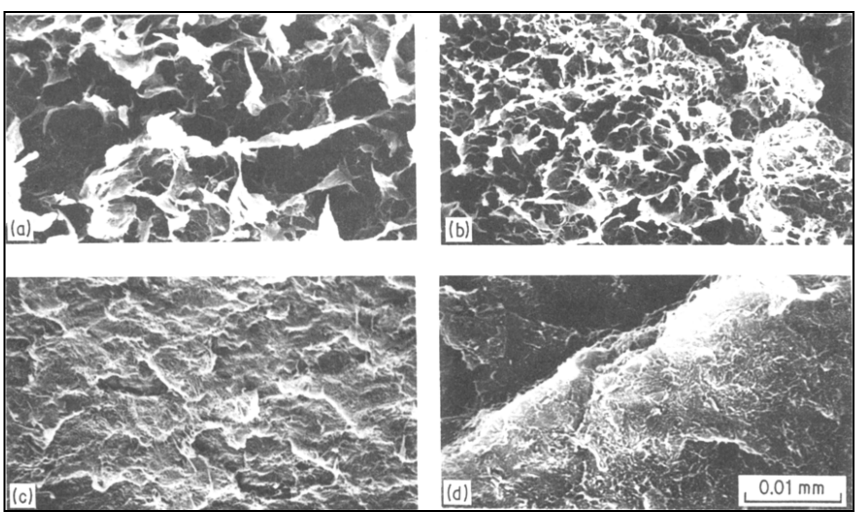

Figure 1. 3 Morphologies of damaged surface under SEM (Bhattacharya and Brown, 1985)

It has been found that the crack initiation rate of polyethylene material increases with a decrease of material density. Lu and Brown (1987) did a study on the effect of thermal history on slow crack growth of linear polyethylene. It was found that the rate of slow crack growth is affected by the variation of the yield point of the material under study.

Brown and Wang (1988) developed a new technique to measure the strain field near the boundary of craze. Based on this method, they established that the strain distribution near the crazes of homopolymers is weaker than the strain distribution in copolymers. Also, they found a way to interpret the stress field associated with the strain field based on the stress-strain curve of the tested polymers.

The researchers of the University of Pennsylvania (Lu et al. 1988) compared the rate of crack growth of HDPE and copolymer. The results from this publication shows a reduction in the rate of SCG of copolymer in comparison to the SCG rate of HDPE. The rate of slow crack growth of copolymer was about 100 to1000 slower than HDPE. The butyl branches of the copolymer was identified as the possible cause of the reduction in the rate of disentanglement and thereby slowing the SCG rate. The authors analysed the microstructural variation during SCG by using a SEM (Figure 1.4).

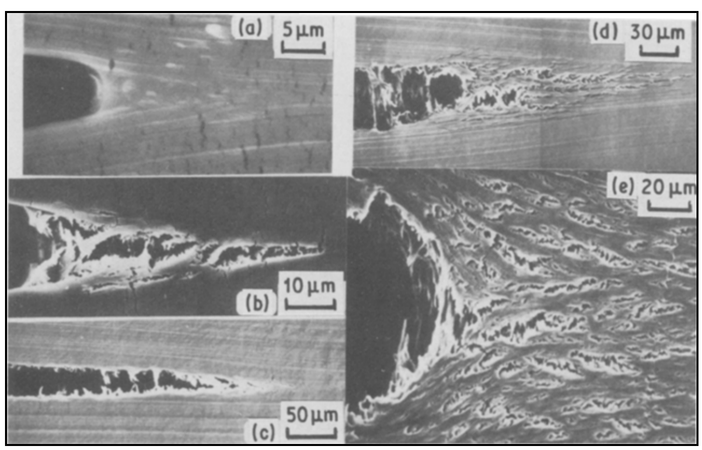

Figure 1. 4 SEM picture of HDPE under different test condition (Lu et al., 1988)

The failure of a single notched copolymer specimen under a constant tensile load was investigated by Lu and Brown (1990). It was observed that the material experienced three-failure mode: ductile, brittle and transitional. The ultimate mode of failure was predicted from microstructural changes in the notched region. The results showed clearly that the ductile failure was controlled by macroscopic creep behavior while the brittle damage was due to SCG that begins from craze. In addition, the paper elaborated the growth of notch opening over time at surface, root and border of craze during ductile (Figure 1.5), brittle and transitional failure modes.

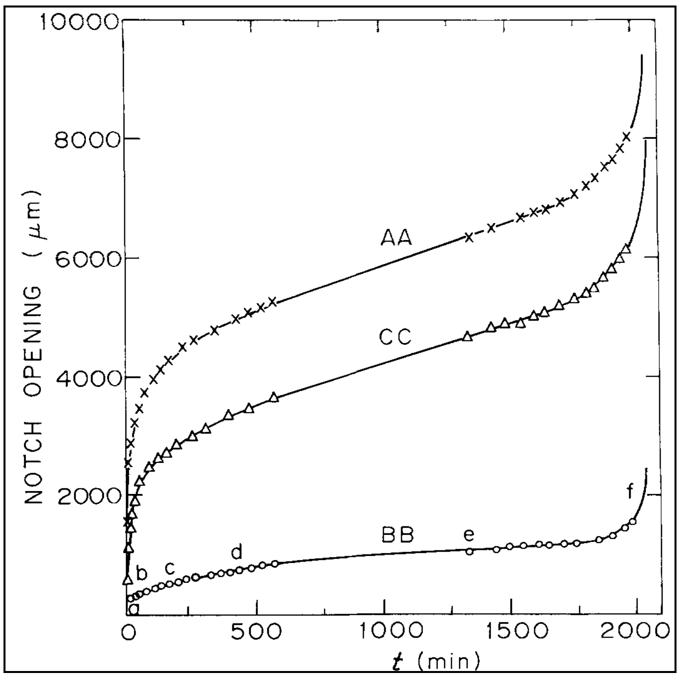

Figure 1. 5 Notch opening at AA (surface), CC (root) and BB (border of craze) against time in ductile region (Lu and Brown, 1990)

Another study by Lu et al. (1991), on the discontinuous crack growth under constant load of polyethylene material, measured the kinematics of slow crack growth. It was highlighted that for a temperature decrease, the jump distance decreases as the applied stress increases. The rate of disentanglement of fibrils at the craze dictates the initiation time of fracture. The kinematics of SCG depends on the rate of disentanglement, fibre strength, stress intensity and yield point of the resin. The material clearly exhibits different magnitude of fracture damage under different applied stress (Figure 1.6).

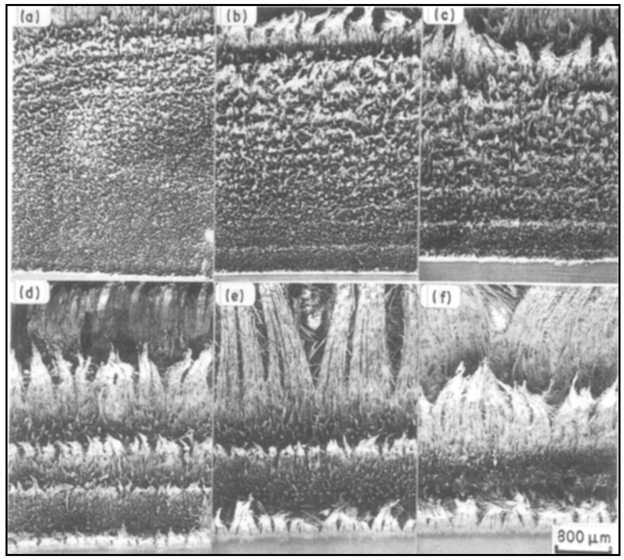

Figure 1. 6 Fractured surfaces of the specimen
at different stresses (Lu et al., 1991)

Findley and Tracy (1974) examined the creep behavior of polyethylene and PVC materials under few different tensile stress, temperature and humidity. The creep tests were performed roughly for 132,000 hrs or approximately 16 years. The researchers developed a strain equation, which accurately predicted the creep strain of PVC for the first 2000 hrs. However, the estimated strain of polyethylene was of low accuracy in comparison to the experimental results.

Zhang and Moore (1997) explored the nonlinear behavior of high-density polyethylene samples cut from a thick walled HDPE pipe. The results led to the development of nonlinear viscoelastic (NVE) model and viscoplastic (VP) model by the authors. The predicated values from NVE were not precise for all tested conditions but the results generated from VP model were in good agreement with the experimental results up to the maximum strain reversal value.

The modeling of short and long-term tensile creep of high-density polyethylene was performed by Lai and Bakker (1994). The effect of stress and physical ageing on the creep compliance at an ambient temperature were studied. It was observed that, at larger stress, the rate of creeping accelerates while at low stress, the effect of ageing was independent of the applied stress and the material exhibits strong nonlinearity. The researchers developed a non-linear creep equation that includes effect of ageing. There is good correlation between the experimental and analytical results.

A study on the creep damage mechanism of polyethylene gas pipes was published by a group of researchers from France (Hamouda et al., 2001). The research concentrated on two types of polyethylene resins, one is a ductile material while the other is brittle material. The slow crack growth clearly controls the lifetime of brittle material. SEM (Figure 1.7) was used in the study of crack initiation and propagation. Even though, catalytic residues act as an initiating factor, this is not true for all extruded resin samples. It was observed that the largest principal stress orients the micro-cracks in a direction perpendicular to it.

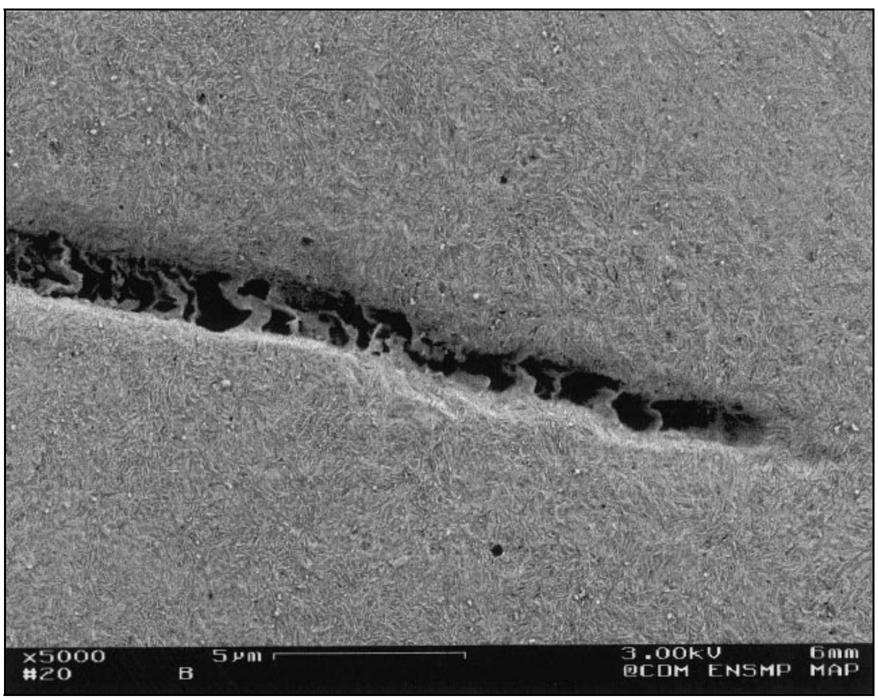

Figure 1. 7 Microstructure of secondary craze (Hamouda et al., 2001)

An investigation on the fatigue behavior of high-density polyethylene was conducted under tension-compression, pure tension and pure compression cycles. The researchers (Kaiya et al., 1989) made some morphological observations (Figure 1.8), which revealed that the type of cyclic deformation dictated the fatigue fracture induced in the material. It was observed that the fracture surface was at 45° with respect to the direction of the applied compressive load under pure compression cycles while it was almost perpendicular to the load condition under tensile fatigue.

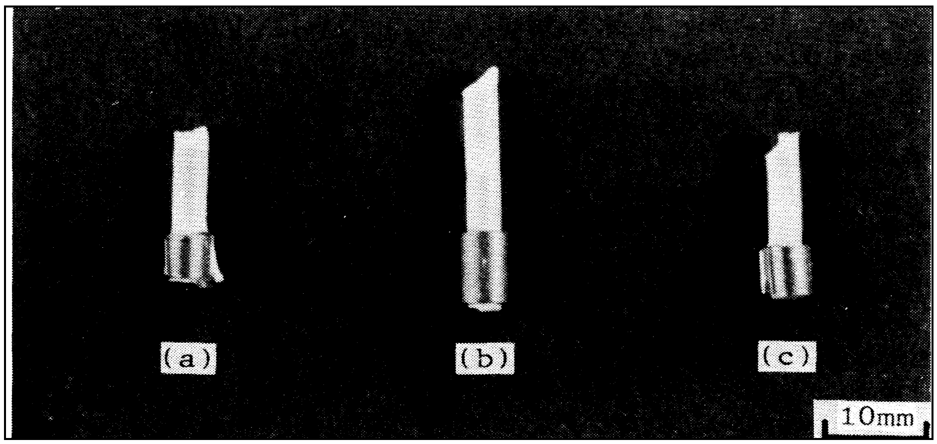

Figure 1. 8 Fractured surface of (a) pure tension, (b) tension-compression and (c) compression fatigue cycles (Kaiya et al., 1989)

Dong et al., (2011) experimentally probed into the fatigue behavior HDPE reinforced with silane modified TiO_2 composite. The results showed that the composite fatigue life improved until 30 MPa of the applied stress and at saline bath environment. However, the decline of fatigue life was evident when the applied stress magnitude increased above 30 MPa. By analysing the failure morphologies of the composite, it was understood that silane (Figure 1.9) cannot support the load and it initiates crack in the material surface.

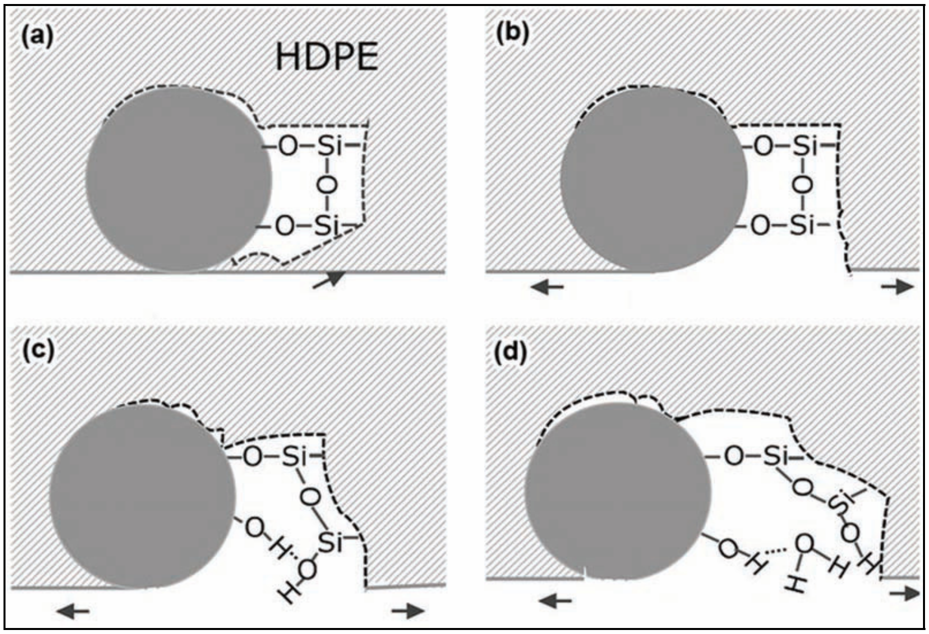

Figure 1. 9 Surface Crack formation (a) no load, (b) mechanical stress, (c) in air - broken TiO_2 and (d) in saline solution – broken TiO_2 (Dong et al., 2011)

Chen et al., (1997), evaluated the effect of joining mechanism on the bending fatigue and fracture behavior of high-density polyethylene. The study compared three types of joining methods namely butt fusion, electrofusion and plain part. It was found that the butt fusion (Figure 1.10) specimen failed at the fusion zone while the electrofusion specimen failed at fusion joint. The butt fusion joint had a superior resistance to fatigue bending than electrofusion sample; however, the plain unwelded sample exhibited the highest resistance.

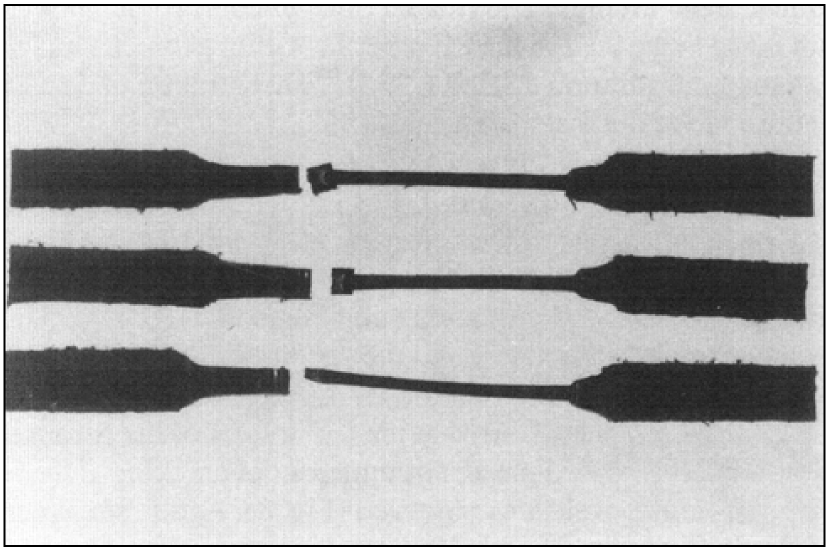

Figure 1. 10 Failure of butt fusion samples (Chen et al., 1997)

1.3 Scientific evaluation of Polyvinyl Chloride

Polyvinyl chloride or PVC, as commonly known, is the most valuable and the largest used polymeric material of all time. The utility of PVC is extraordinary, which ranges from electric cables to pipes, from clothing to healthcare, from furniture to construction etc… It has been more than a century since the first commercial production of PVC by Fritz Klatte. Since then the growth of PVC is undoubtedly significant. With growth and popularity comes the assessment and evaluation, which has led to characterisation of the material in general and to specific application. Since 1912, a humungous amount of research were conducted on characterising PVC. Reviewing and summarizing all the scientific articles would be impossible and tedious; therefore, this section of the chapter will broadly discuss the recent publications in the characterisation of PVC material.

In the last three decades, the tensile, compression and flexure characterisation researches are mainly fixated on PVC thermoplastic composites. The study on characterising the impact and tensile strength of PVC composite filled with hollow glass beads by Liang 2002 demonstrated that the tensile yield strength (Figure 1.11) of the composite decreased with increase in volume fraction of the hollow glass bead (HGB). However, the tensile break strength increases a little with increase of HGB. The impact strength decreased significantly with the increase of fiber volume fraction but when the fiber volume fraction is higher than 5% of the total, then the rate of decrease of the impact strength is not significant.

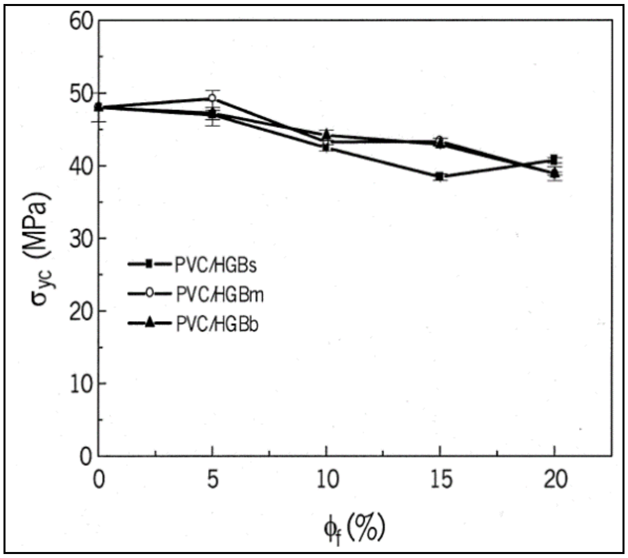

Figure 1. 11 Tensile yield strength dependence on volume fraction of HGB (Liang, 2002)

Ge et al., (2004) performed a comparative study on the tensile and thermal properties of two PVC composites reinforced with bamboo and pine flour, respectively. It was observed that the PVC composite with pine flour showed better tensile strength then the other composite under the same loading and particle size conditions. In comparison to bamboo fibers, short pine fiber inside PVC resin showed greater alignment and dispersion under scanning microscope evaluation (Figure 1.12).

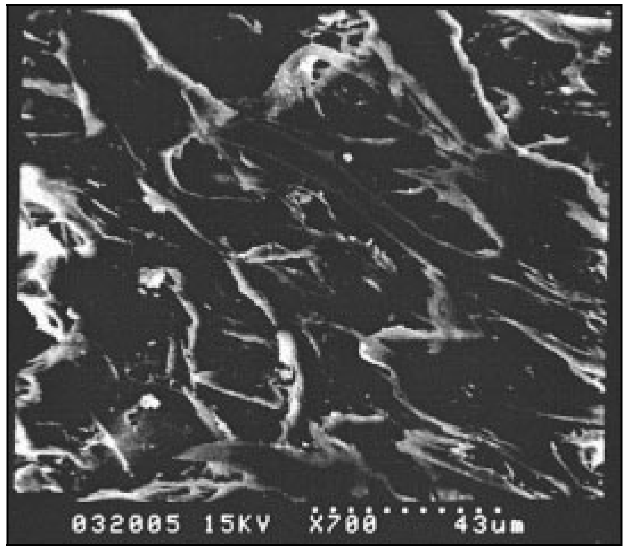

Figure 1. 12 SEM image of pine fiber dispersion
in PVC resin (Ge et al., 2004)

The tensile strength and fracture toughness of PVC foam material was examined. The authors (Kabir et al., 2006) found that the level of cross-linking bonds dominates the fracture toughness of the material; however, the cracks in rise and flow direction and loading rate does not produce a noteworthy effect on fracture toughness. Experimental tests revealed that the foam density (Figure 1.13) dictates the tensile strength, modulus and fracture toughness of the material. It was observed that all foam samples failed in a brittle manner.

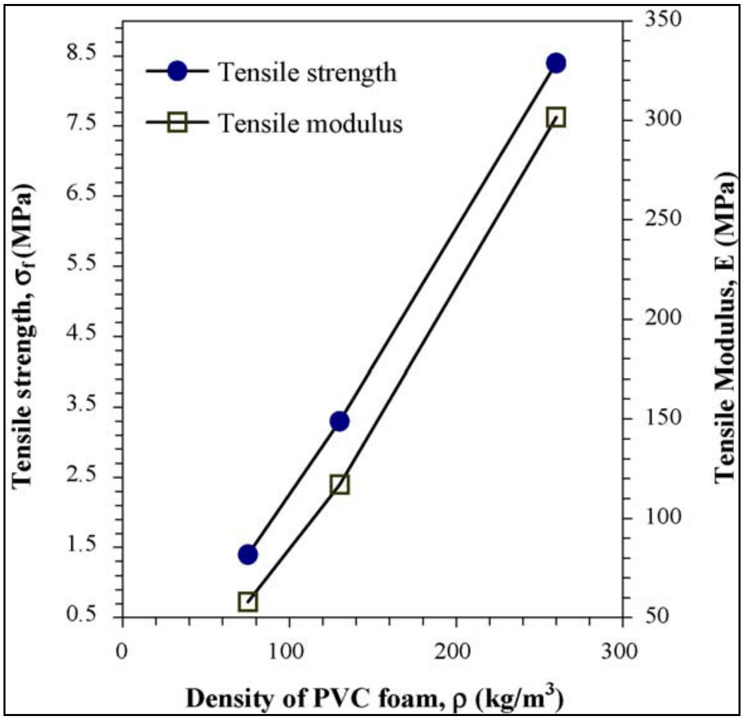

Figure 1. 13 Impact of foam density on the tensile strength and modulus (Kabir et al., 2006)

The work by Sabuncuoglu et al., (2011) examined the viscoelastic properties of polypropylene material using a series of creep tests. They limited the creep tests to very short time in order to limit the sudden drop of stress during the initial few seconds. The results were verified with the viscoelastic model was in good concordance with experimental tests, where the samples were subjected to tensile strain rates lower than $0.01 s^{-1}$.

The primary creep behavior of polypropylene was studied experimentally and numerically to simulate long-term behavior of the material (Dropik et al., 2002). These researchers performed experimental creep tests and utilized a procedure developed by Dougherty, 1996 to determine the creep constants for non-linear Maxwell model. The established constants were used in ANSYS creep formulas and the creep behavior of the material was simulated. On comparing experimental results with ANSYS results, there was a 11.1% and a 16.6% difference for creep and relaxation (Figure 1.14), respectively.

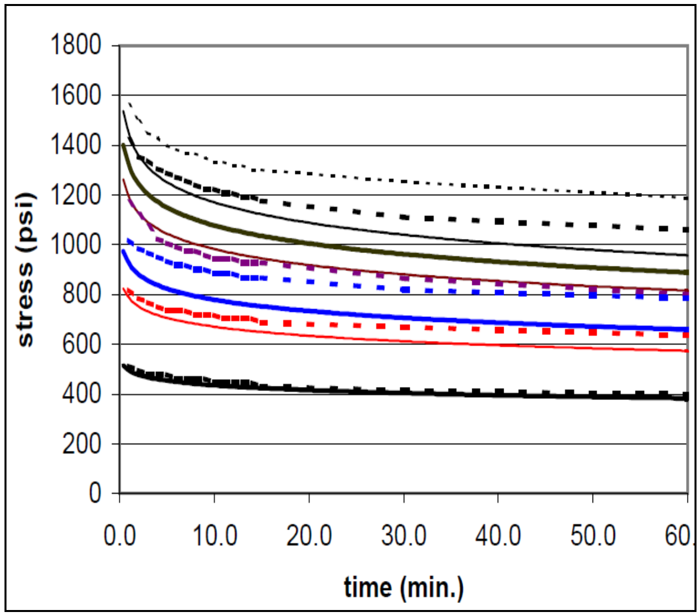

Figure 1. 14 Comparison experimental and ANSYS results for relaxation (Dropik et al., 2002)

Barbero and Ford (2004) modeled the effect of physical ageing and temperature on the creep and relaxation behavior of polymers. The developed equivalent time temperature model (ETT) is an extension of the existing equivalent time method. The time-temperature superposition method used in the article is applicable only to unaged data. The procedure to shift creep data to time-temperature superposition master curve was detailed.

Laiarinandrasana et al., (2011) investigated the creep behavior of PVC on round bar samples. For experimentation, the researchers utilized virgin and aged pipes (22 to 35 years of service). Fracture mechanics for the creep of solid tools were used to study the creep failure of PVC pipes under internal pressure. From the experimental data, it was found that the creep strain rate was higher at the external surface than at the internal surface of smooth PVC pipes. The effect of ageing (Figure1.15) was cited as the reason for this decrease in creep strain.

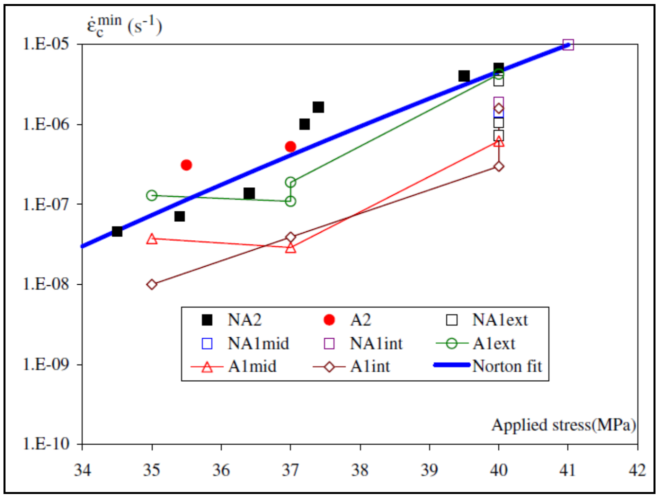

Figure 1. 15 Effect of ageing in creep strain
(Laiarinandrasana et al., 2011)

A group of engineers (Pulngern et al., 2013) worked on finite element simulation of strengthened wood/PVC composite to predict the creep response of the material. The research detailed the effect of strengthening high carbon steel bars on the performance of the composite. The power creep law was used to determine the constants and the results from the ABAQUS software showed a good correlation (Figure 1.16) with the experimental data.

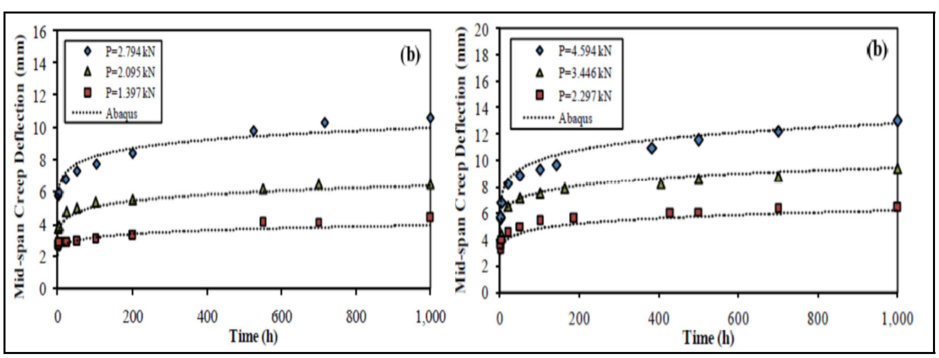

Figure 1. 16 Numerical vs experimental results before (left)
and after (right) strengthening (Pulngren et al., 2013)

An experimental study on the bending fatigue of PVC pipe and joints was performed by two professors from university of Akron (Scavuzzo and Srivatsan, 2006). The PVC samples were subjected to an internal pressure (varying from 0 to 280 psig) and a four point bending to evaluate its response to bending fatigue. It was deduced that the internal pressure acting on the pipe plays a significant role in determine the fatigue life of the PVC pipe. The samples tested under four point bending without internal pressure were found to have weaker fatigue life than the samples tested with internal pressure. It was suggested that the fatigue cycling rate and the hold times at maximum load might affect the fatigue strength of the PVC pipe.

The permeation of organic solvents into the PVC pipe was extensively studied by Mao et al., (2011). The researchers found that the external chemical activity controls the propagation rate, where the rate of propagation increases with the increase in number of organic solvents in the medium of transfer. It was also determined that the permeability of contaminants depends on the time of contact between the pipe and fluid (Figure 1.17). After two years of test, it was observed that PVC pipe showed highest resistance to permeation commercial gasoline among other tested fluids.

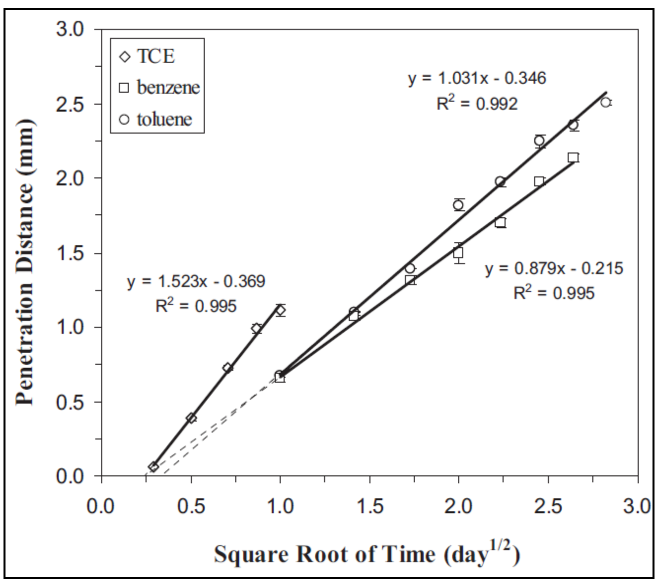

Figure 1. 17 Penetration distance vs Sq. root of time
(Mao et al., 2011)

The characterization of oriented polyvinyl chloride (PVCO) to ground scale deformation was studied by a group of civil engineers from Cornell University (Wham et al., 2016). They evaluated the capacity of PVCO pipe with bell and spigot joints to resist large deformations. The assessment varied from full-scale fault rupture test to fundamental material property evaluations. The axial pullout and compressive load capacity of the joints control the performance of the pipe. The longitudinal and circumferential (Figure 1.18) elastic modulus and Poisson ratios were obtained from an uniaxial tensile coupon test and an internal pressurization test, respectively. The results highlighted that a significant amount of fault movement can be accommodated by PVCO pipeline.

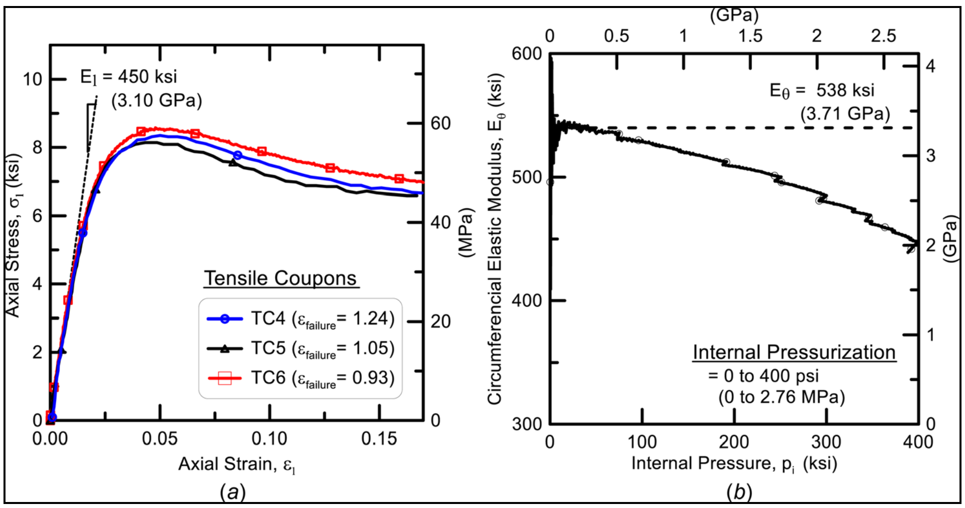

Figure 1. 18 Longitudinal and circumferential property determination from tensile coupon and internal pressurization tests (Wham et al., 2016)

Bouzid and Chaaban (1997) developed a method to measure accurately the relaxation in bolted flange joints. This article details the working of inbuilt computer program called SuperFlange ©, which can predict relaxation of bolted joints and the results act as a realistic evaluation of the leak over time. The proposed model effectively predicted the creep relaxation displacement of different gaskets at different thicknesses, as shown in Figure 1.19.

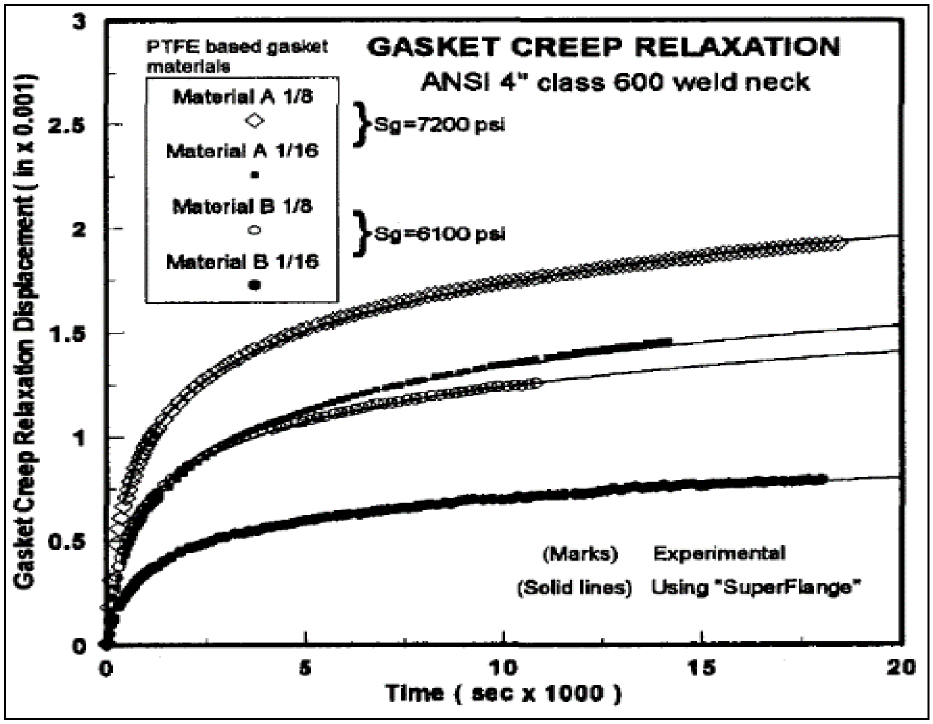

Figure 1. 19 Experimental vs super flange evaluation of creep-relaxation displacement over time (Bouzid and Chaaban, 1997)

The impact of shell and hub creep in the relaxation analysis of bolted flange joints was assessed by Nechache and Bouzid (2008). The paper highlights that the shell and hub were accountable for 60% loss of bolt load under the evaluated conditions. Also, the article compared the analytical model (Figure 1.20) with the results from the FEM simulation, which were found to be in close agreement with each other.

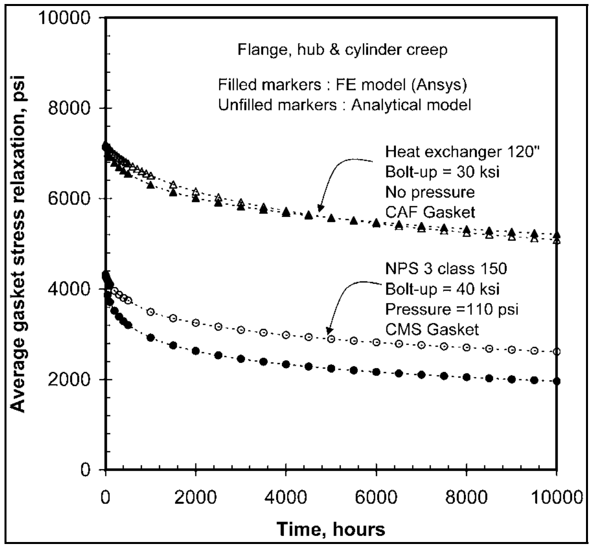

Figure 1. 20 Analytical model vs FE model
(Nechache and Bouzid, 2008)

1.4 Gasket literature review

The service temperature assessment of PTFE based gasket by Bouzid et al. (2001) provided new insights to the HOBT characterisation of PTFE gaskets. The paper emphasised the effect of thermal cycling on the creep-relaxation behavior of gasket materials. The authors looked into the effect of cumulative damage and discussed several methods to determine the coefficient of thermal expansion (Figure 1.21) of gasket materials.

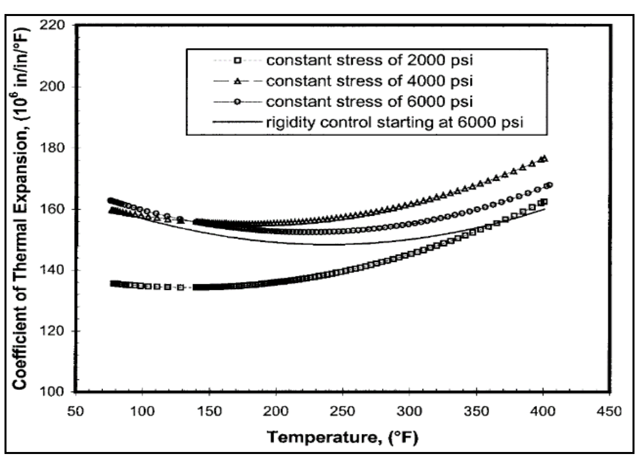

Figure 1. 21 CTE vs ratcheting temperature
(Bouzid et al., 2001)

Grine and Bouzid (2013) examined the leak rates through porous gaskets at high temperature. The authors utilized the analytical model of slip flow regime (Grine and Bouzid, 2011) to validate the model with experimental data. The obtained results showed the effectiveness of the developed model (Figure 1.22).

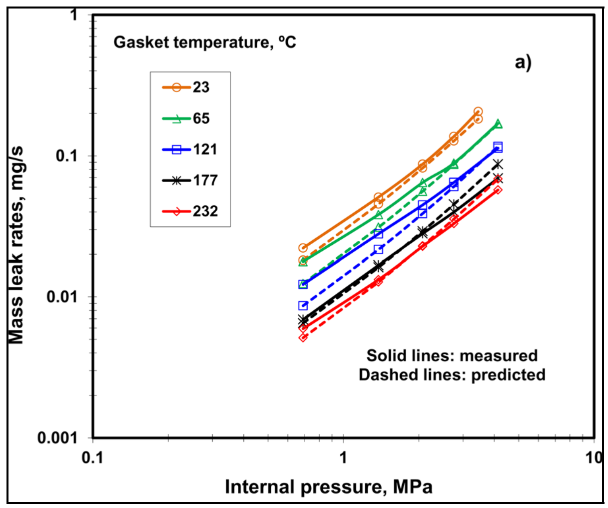

Figure 1. 22 Measured vs predicted leak rates
(Grine and Bouzid, 2013)

The compressive ratcheting behavior and the effect of loading rate on the stress-strain response of PTFE material were probed by Chen and Hui (2005). It was observed that the material was sensitive to the loading rate up to 40 N/s after which the loading rate (Figure 1.23) becomes insignificant to ratcheting strain induced in the material. The magnitude of ratcheting strain was the highest at the lowest tested loading rate; however, the ratcheting strain increased with an increase in the mean stress applied to the test samples.

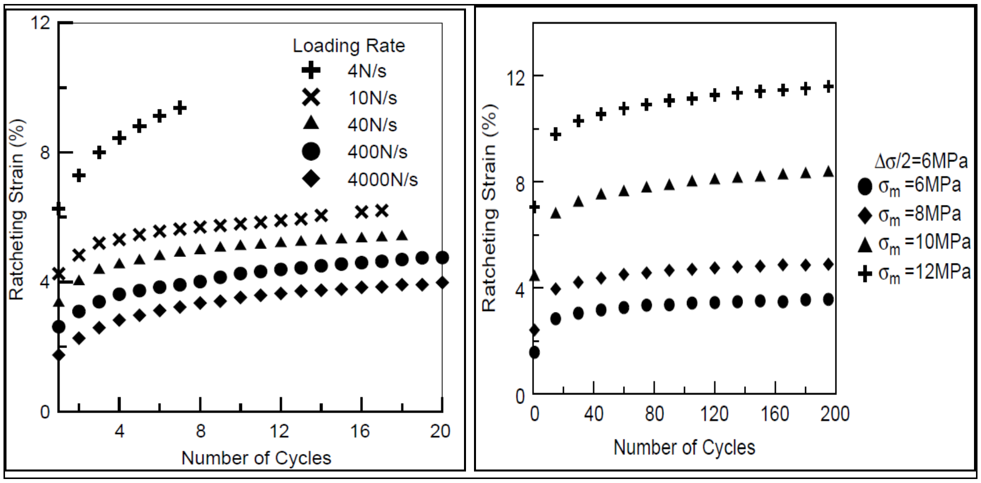

Figure 1. 23 Behavior of ratcheting strain with loading rate (left) and applied mean stress (right) (Chen and Hui, 2005)

Kletschkowski et al., (2002) developed a model to predict the nonlinear behavior of polytetrafluoroethylene material. The developed model predicted the short and long-term behavior (Figure 1.24) of filled PTFE accurately. The model works well under small and finite strains.

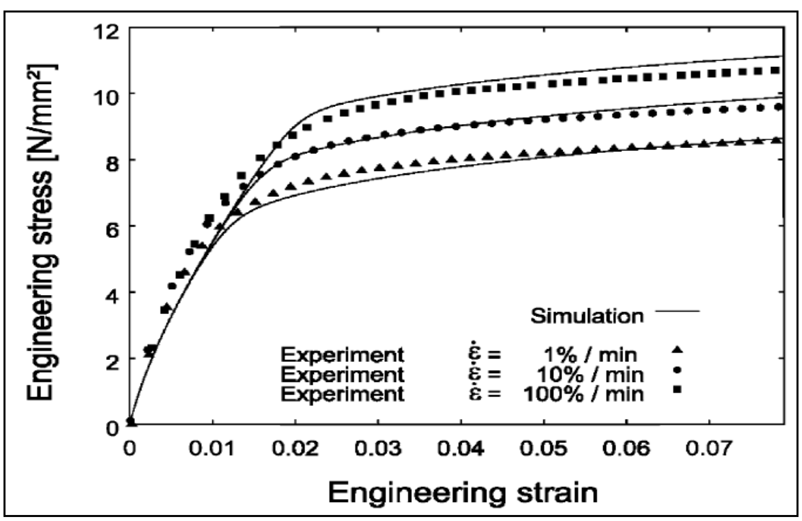

Figure 1. 24 Comparison of experimental vs analytical results (Kletschkowski et al., 2002)

The compression property of Teflon based PTFE materials samples from DuPont, 7A and 7C, were experimental studied (Rae and Dattelbaum, 2004). The research probed into the effect of strain rates and temperature on the mechanical properties of the materials. The strain rate and temperature varied from 10^{-4} to 10^{-1} s^{-1} and from -198 to 200°C, respectively. The two material variants demonstrated similar true strain relaxation upon unloading (Figure 1.25) under different loading and unloading strain rates. The impact of temperature was also evident. Lowering of temperature causes an increase in true stress under the same loading rate.

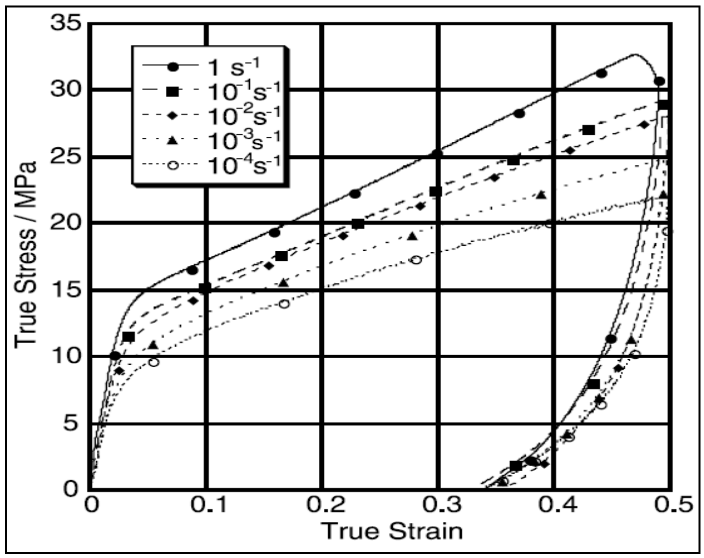

Figure 1. 25 Effect of strain rate on DuPont 7A material
(Rae and Dattelbaum, 2004)

Bergström and Hilbert (2005) worked on establishing a constitutive model to predict temperature and time dependent mechanical properties of fluoropolymers. The researchers conducted a variety of experimental tests to validate the capabilities of this new model. The results demonstrated the accuracy of the constitutive model in predicting the material response. Figure 1.26 shows the effectiveness of the model in predicting the uniaxial tension and compression behavior of glass filled PTFE samples. In addition, the model can predict cyclic uniaxial loading.

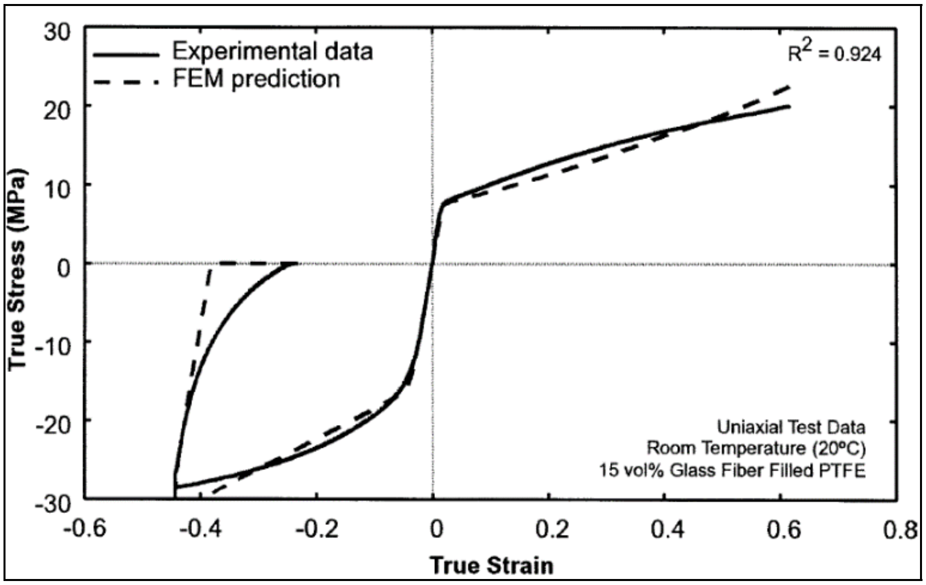

Figure 1. 26 Experimental vs FEM prediction of uniaxial test data (Bergström and Hilbert, 2005)

Solid cylindrical polytetrafluoroethylene specimens were subjected to experimental mechanical ratcheting tests (Zhang and Chen, 2009). The results demonstrated that the ratcheting strain was influenced by the applied axial stress (Figure 1.27), cyclic shear strain range and shear strain rate. While both axial stress and shear strain range proportionally influenced the ratcheting strain, the decrease in shear strain rate amplified the ratcheting strain. The impact of loading history on the progress of ratcheting strains was evident and this effect was due to the hardening effect under previous loading.

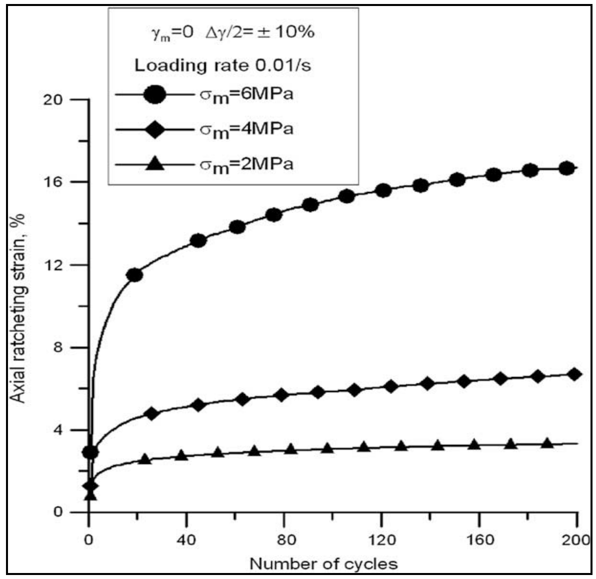

Figure 1. 27 Effect of constant axial load
(Zhang and Chen, 2009)

The paper by Nunes et al., (2011) focused on developing a new model to predict the mechanical behavior of polytetrafluoroethylene material under tensile loading with different strain rates. Visual strain measurements using a non-contact video extensometer were taken in this experimental evaluation. The values of constants used in the developed model were determined from experimental test results obtained under different constant strain rates. On comparing the experimental data with the analytical results, a good agreement is observed as seen in Figure 1.28.

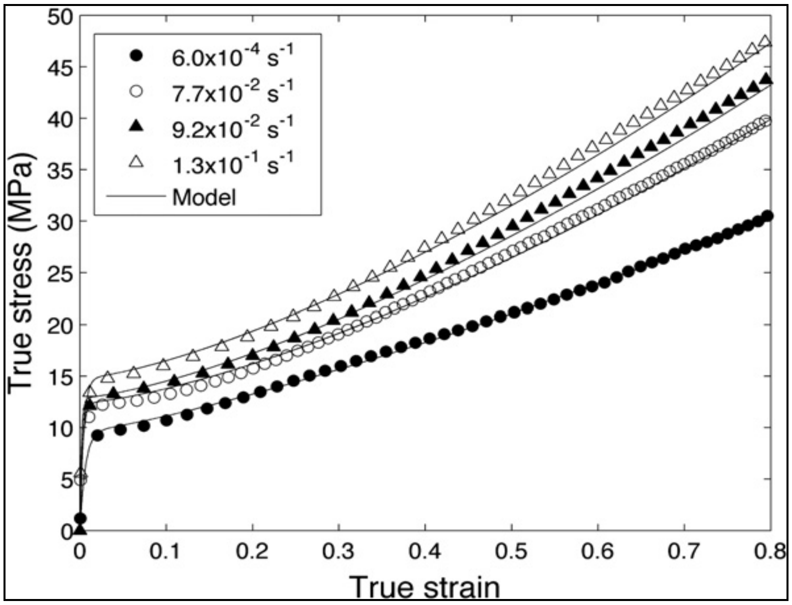

Figure 1. 28 Model vs experimental results under different strain rates (Nunes et al., 2011)

Existing standards (ASTM E 228-1, E 831-14, D 696-16) were reviewed to adopt a test procedure to measure linear thermal expansion coefficient of the tested materials. In addition, the ASTM standard for Hot blowout test for gasket material was studied. Significant amount knowledge was obtained by studying the research publications on the thermal characterisation and creep resistance of Teflon based PTFE gasket material (Marchand et al. 1992, Derenne et al. 1999, Payne and Bazergui 1990, Payne et al. 1987).

Bouzid and Benabdullah (2015) worked on analysing the HOBT test procedure and suggested to increase the number thermal cycling for improvement of the test standard. The authors performed up to twenty thermal cycles and looked into the effect of thermal cycles on bolt load relaxation of bolted flange joint. Furthermore, the researchers showed the influence of applied stress on the coefficient of thermal expansion (Figure 1.29) of PTFE material.

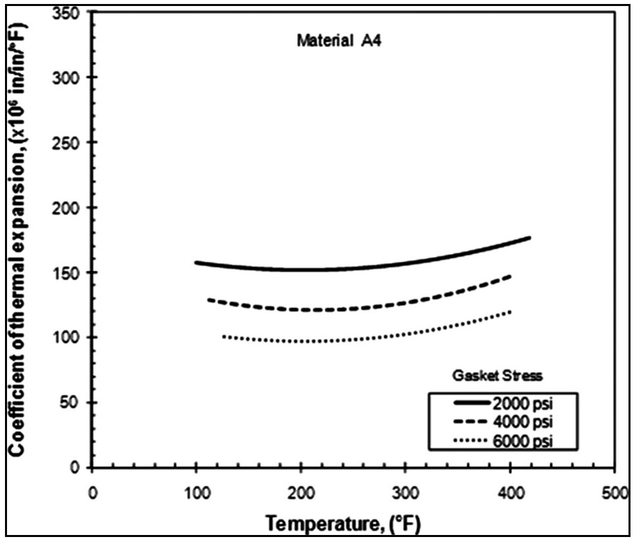

Figure 1. 29 Influence of applied load on the CTE
(Bouzid and Benabdullah, 2015)

1.5 Research objective

From the extensive study and review of scientific articles and from an analysis of the current trend of research on characterisation of soft materials, it is clear that the amount of research on thermal ratcheting behavior of the selected materials is rare and the information on the adverse effect of this phenomenon is very limited. Furthermore, there is no published research of the interaction of thermal ratcheting and the creep response of these chosen materials.

The current state of the art in the characterisation researches focus on improving the properties of HDPE and PVC materials by developing a new composite material out thermoplastic resins; thereby widening the application range of these flexible plastic materials. However, the application of HDPE and PVC in pressure vessel and piping domain is out pacing the volume of application-oriented research done on these materials. As explained earlier, bolted flange connection is one of the most critical component of the PVP system. With the ever-increasing operational and fugitive emission restrictions, it is important to characterise the behavior of HDPE and PVC bolted flange joints. The current ASME and European polymeric bolted flange joint standards are directly derived from standards of metallic bolted flange joint. However, not only the thermal and creep properties of polymers and soft materials are significantly different from those of its metallic counterparts, their high temperature data under compressive load is simply not available. Because of these factors, the research is focused to follow the objectives or goals described below:

- Study thermal ratcheting phenomena of few Teflon and fiber based gasket materials
- Study the cumulative damage of HDPE and PVC materials produced by thermal ratcheting under compressive load
- Investigate the vulnerability of gasket materials to creep and thermal ratcheting
- Study the relationship between thermal ratcheting on the creep behavior of HDPE and PVC materials
- Conduct an analytical and numerical modeling of load relaxation of HDPE and PVC bolted flange joints subjected to short-term creep.

CHAPTER 2

EXPERIMENTAL SET-UP

2.1 Introduction

This chapter provides a detailed description of the working principal of Universal Gasket Rig and Hot Blowout Test bench. The sophistication of these two home-built test equipment paved the way for successful testing and characterisation of the selected test materials. Both test rigs were designed and built by students of the Static and Dynamic Sealing Laboratory with the assistance of department technicians. The primary purpose of the HOBT rig is to perform hot blowout tests on PTFE-based gaskets to determine their safe operating temperature limits and the UGR was designed to characterise gasket materials for leak and relaxation performances. Both test equipment were modified over time to accommodate for the analysis of different types of gasket and flange materials, fluid media and operating conditions.

The intricacy of the UGR and HOBT are presented in the following sections of this chapter. In the framework of this research, the creep and thermal ratcheting characterisation of the selected materials are achieved through the use of universal gasket rig and the full scale relaxation tests of HDPE and PVC bolted flange joints are conducted using the HOBT rig.

2.2 Universal Gasket Rig

The speciality of the universal gasket rig (Figure 2.1) is its capability to execute compressive creep, relaxation, leak and high temperature analysis of any material as along it satisfies the dimensional constraints of the machine. Since the initial intention of UGR is to investigate the behavior of gaskets, the machine was design to facilitate a gasket shaped or in general ring shaped samples. The working principal of the machine can be sub-divided into sections with respected to the type of load applied, namely mechanical, thermal, pressurisation & leak.

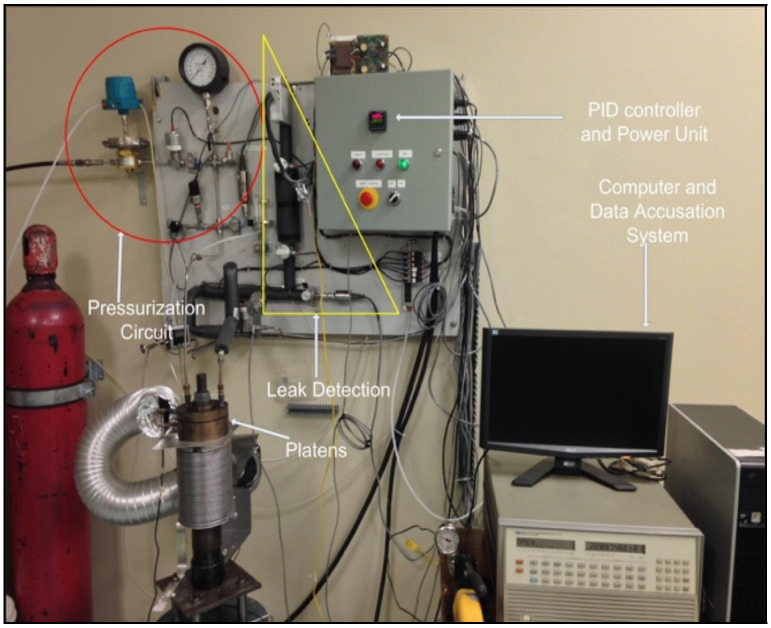

Figure 2. 1 Universal Gasket Rig

2.2.1 UGR mechanical system

In simplest terms, the uniaxial compressive load on the ring shaped specimen is exerted through hydraulic pump. The sample is compressed between two enclosing platens, which prevents the movement of the test piece under the application of the load. The upper and lower platens are circular in shape, which is held in position by means of a central stud and a nut. The schematic of the mechanical system is presented in Figure 2.2. The hydraulic fluid pumped from the manual hand pump causes extension of tensioner head, which in turn exerts the force onto the materials through the central stud and the nut. The upward movement of tensioner head pushes the platens against the nut, which is tightly secured, producing a compressive load on the specimen. High sensitive Linear Variable Differential Transformer (LVDT) keenly monitors the deformation in the axial direction of the test piece. The upper and lower platens have sideward projections from the outer perimeter, which provides for fixation of LVDT with the system to measure the axial deformation. The LVDT (Figure 2.3) is secured to the projection in the lower platen by a small screw and the head of the LVDT is compressed against the sideward extension of the upper platen. This method effectively measure the minute changes in the thickness or the axial deformation of the specimen.

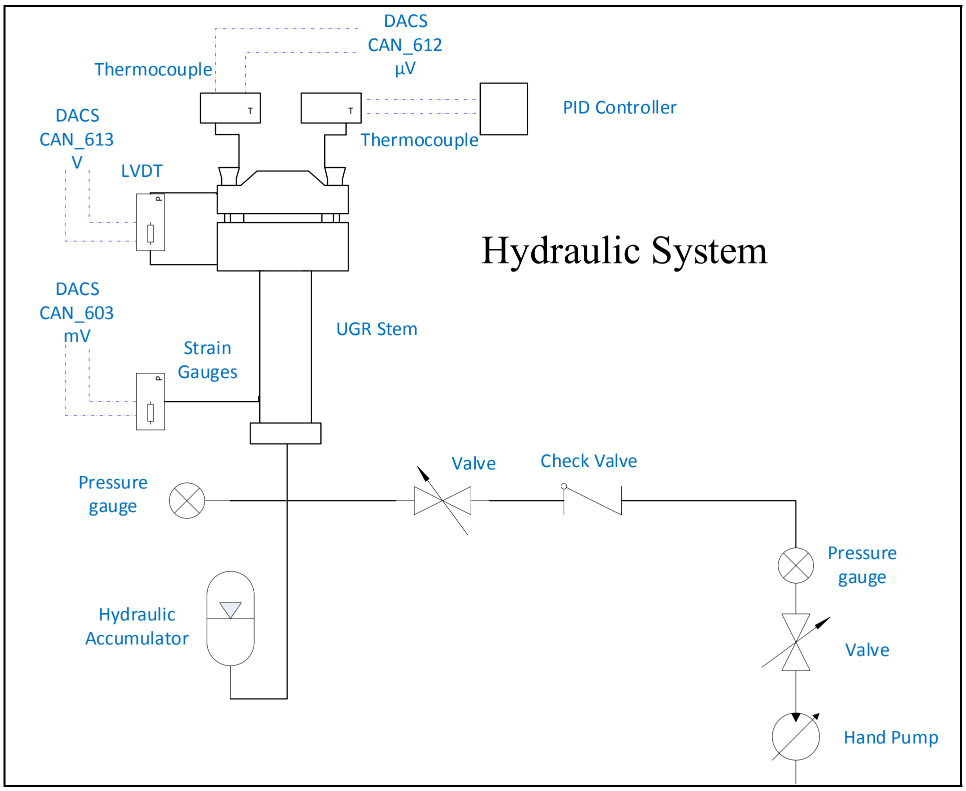

Figure 2. 2 Schematic of the Mechanical/Hydraulic System

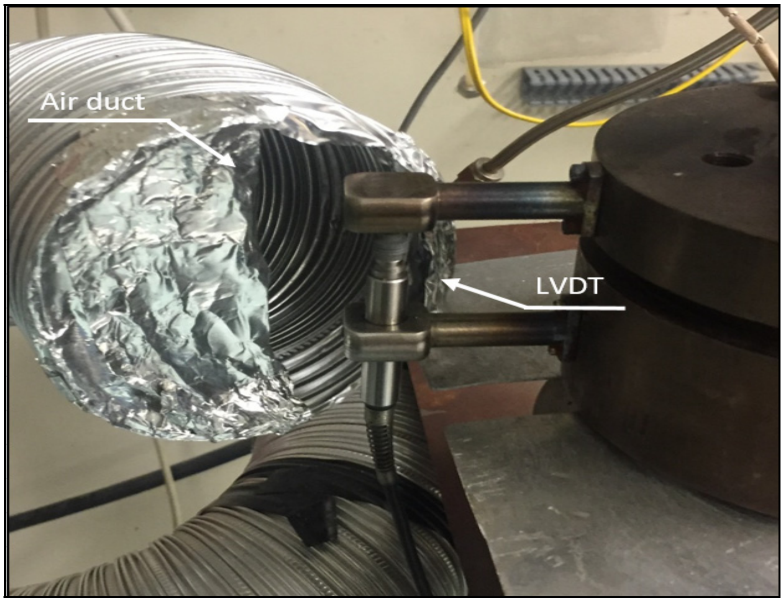

Figure 2. 3 Axial displacement measurement through LVDT

The LVDT system is provided with an air duct to maintain its temperature in the acceptable operational range. The compressive force applied to the sample is measured through a Full Bridge Wheatstone strain gauge, affixed at the bottom of the central stud. The strain gauges pick up the stretch of the central rod as a result of the movement of the tensioner head, which is later converted into a compressive force and converted into compression stress through the LabVIEW program. The central stud is fixed to supporting frame of the UGR, shown in the Figure 2.4. For experimental creep analysis, maintaining a constant load on the test material is a necessity, and this is achieved by a hydraulic accumulator added to the hydraulic system. The function of the accumulator is to maintain pressure in the system, which provides a constant load on the specimen as it deforms.

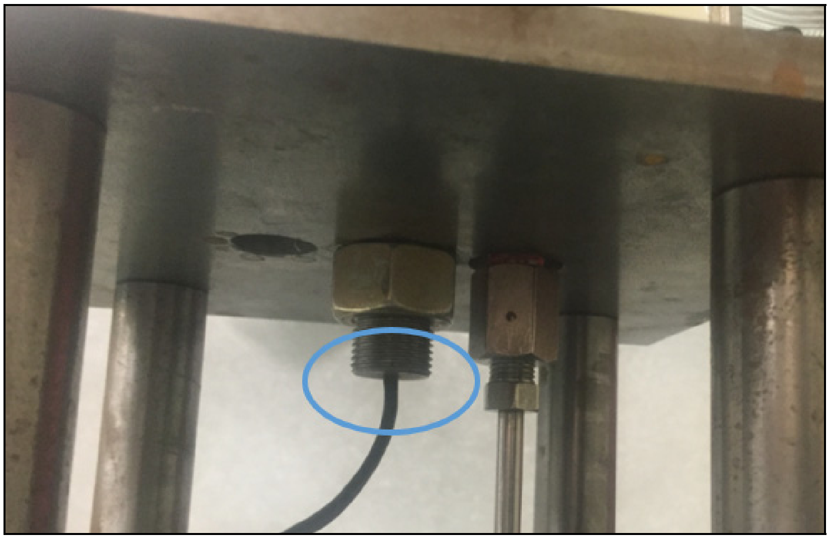

Figure 2. 4 Full Bridge strain gauge

2.2.2 UGR thermal system

Almost every material exhibit a change in its mechanical properties when subjected to change in temperature; hence, thermal characterisation is of importance. The heating of the test samples is achieved by using an electrical ceramic band heater (Figure 2.5), which is wrapped around the upper and lower platens and transfers heat through conduction and convection.

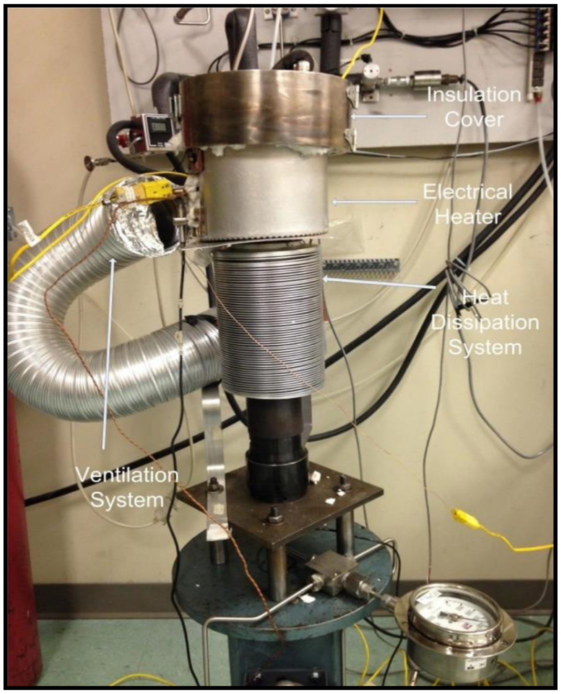

Figure 2. 5 Electrical ceramic band heater

It is to be noted that the circular band heater requires two insulating shield to prevent the loss of heat to the surroundings from its top and bottom sides. For this purpose, a special insulation cap with thermally opaque fiber materials is used on the top section of the band heater. The fiber material fills the gap on the top between the upper platen and the insulation cover. On the bottom side, the stud is extended to host a cylinder with fins in order to avoid the transfer of heat to the bolt strain gauges and the hydraulic tensioner. The heating system has capacity to apply up to 900°F of heat on the ring shaped sample.

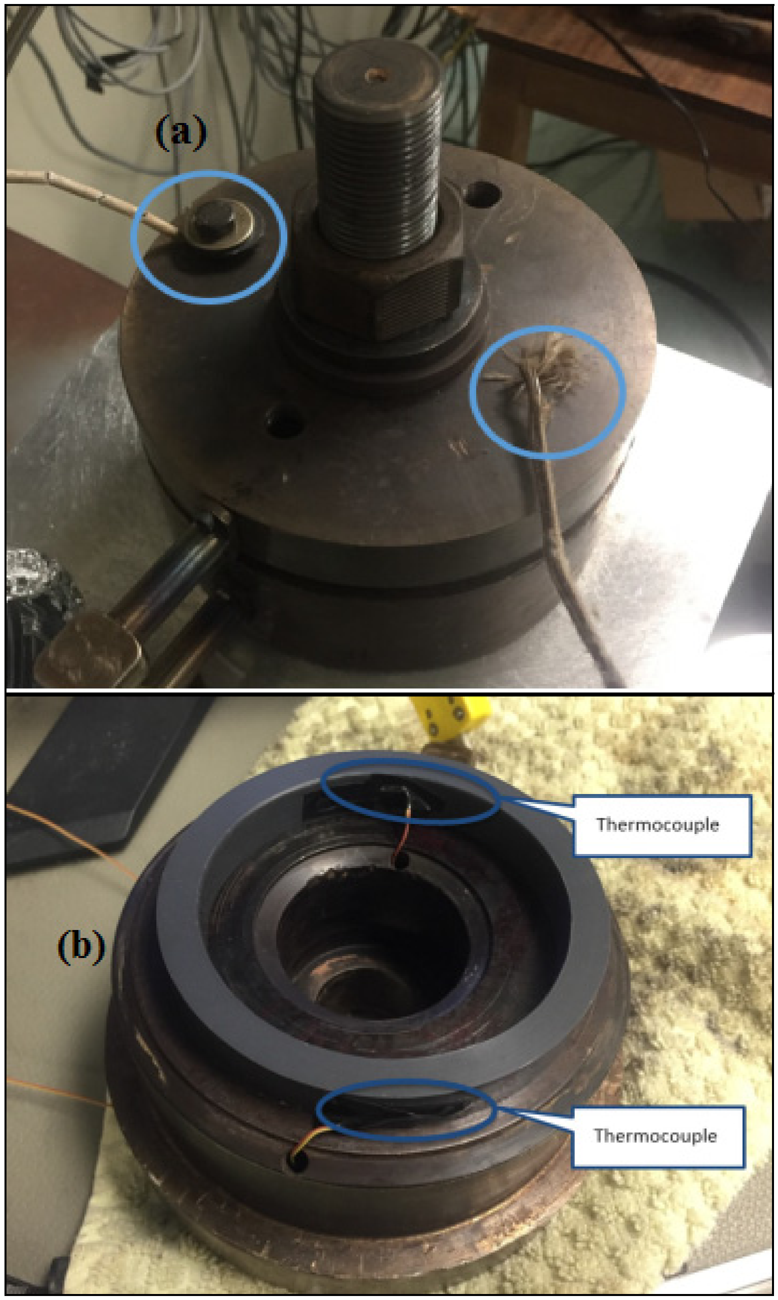

Figure 2. 6 (a) external thermocouple (b) internal thermocouple

Since the test requires either maintaining constant temperature or conducting thermal cycles, the control of temperature is very important. This is accomplished by using two external thermocouples, which are fitted to the upper platen as shown in Figure 2.6.a, and two internal thermocouples that are connected to the inner and outer diameter of the specimen. Generally, the use of two external thermocouples are enough as only 3°F of difference is noted between the external and internal thermocouples for the tested samples. One of the thermocouples is connected to a PID controller that regulates heat to obtain the desired temperature. The other three thermocouples are connected to the computer through a data acquisition and control system.

2.2.3 Leak measurement and pressurization system

Additionally, the UGR machine is capable of exerting internal pressure on the test sample to study the influence of internal pressure or to analysis the rate of leakage through the material. Since pressurisation and leak measurement studies are not done on this research, an elaborate description of these two working mechanisms are not provided. The schematic diagrams of the pressurisation and leak measurement system are shown in Figure 2.7 and 2.8 respectively.

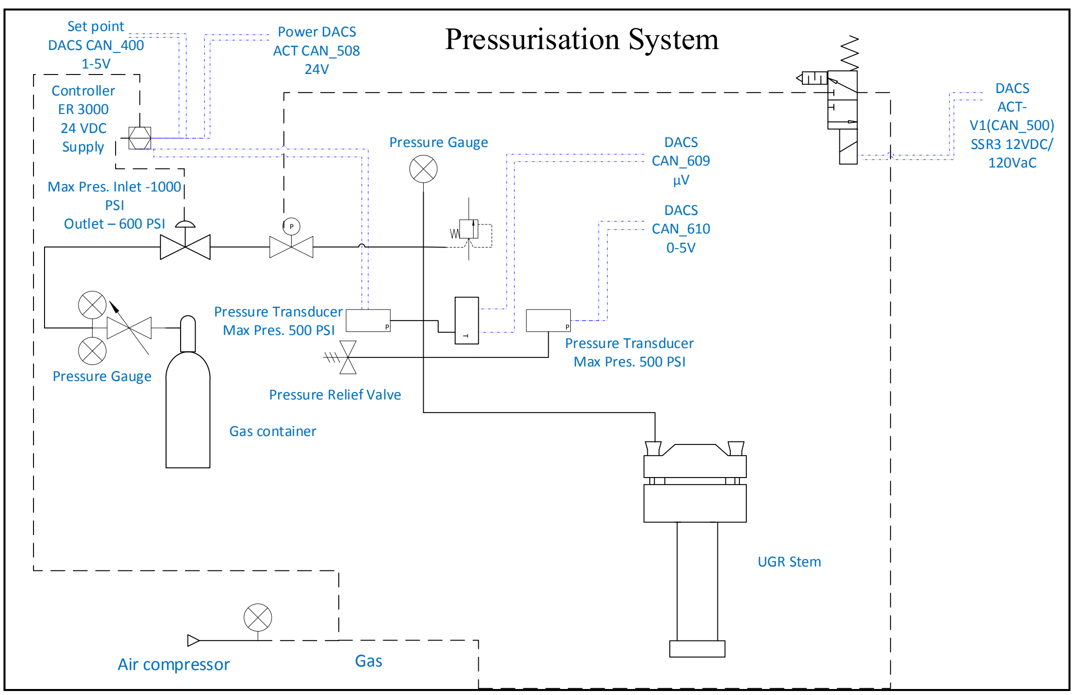

Figure 2. 7 Schematic diagram of pressurisation system

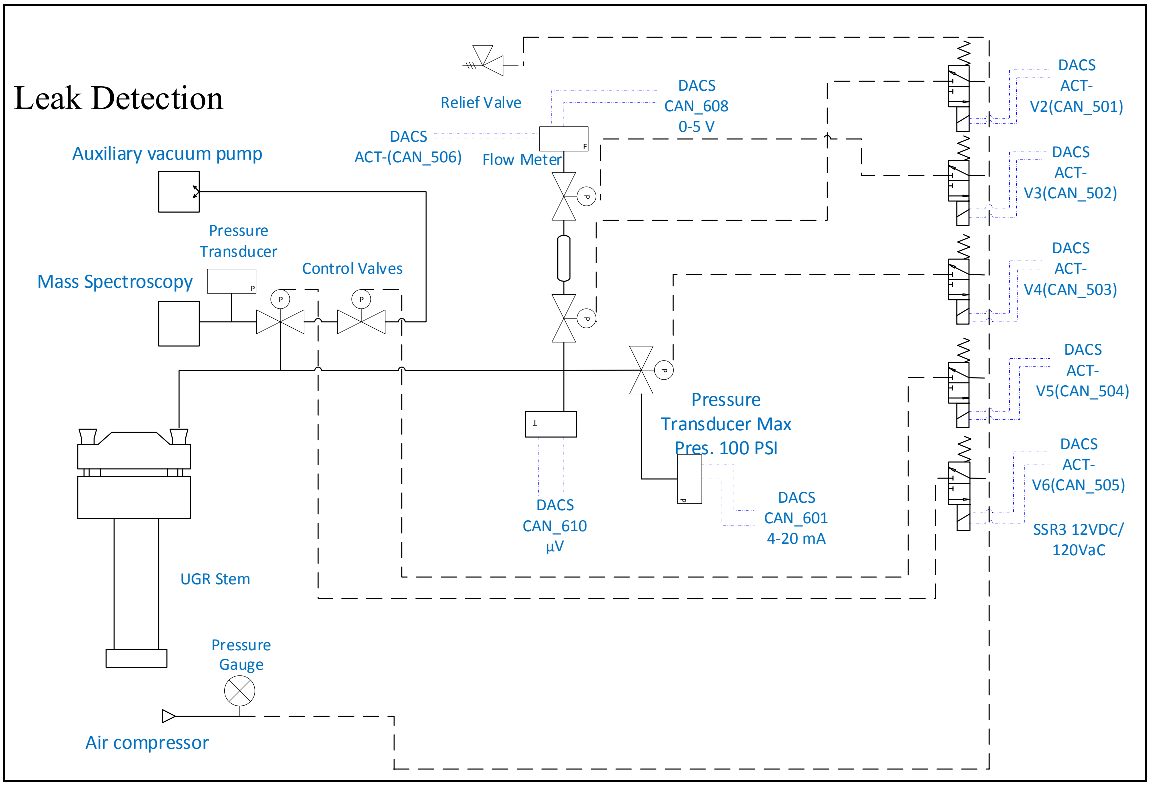

Figure 2. 8 Schematic diagram of leak detection system

2.2.4 LabVIEW data acquisition and control program

The thermal and internal pressure loading are applied using LabVIEW platform while the mechanical loading in applied manually using a hand pump. All instrumentation and control devises are connected to the DAC, which is in turn is connected to a computer. The LabVIEW program enables for easy monitoring of all the measured data from the different sensors. The platform is capable of monitoring the readings every 10 s while recordings can be timed at 10 to 300 s. The recorded values are used for the post processing of the results. The sensors are calibrated and thermally compensated prior to testing. The corresponding constants are feed to the LabVIEW program to improve the accuracy of the results. In addition to monitoring the test parameters, the LabVIEW program communicates with the ER 3000 electronic valve and PID temperature controllers to apply the target pressure and temperature, respectively. The typical graphic user interface (GUI) of LabVIEW program is shown in Figure 2.9.

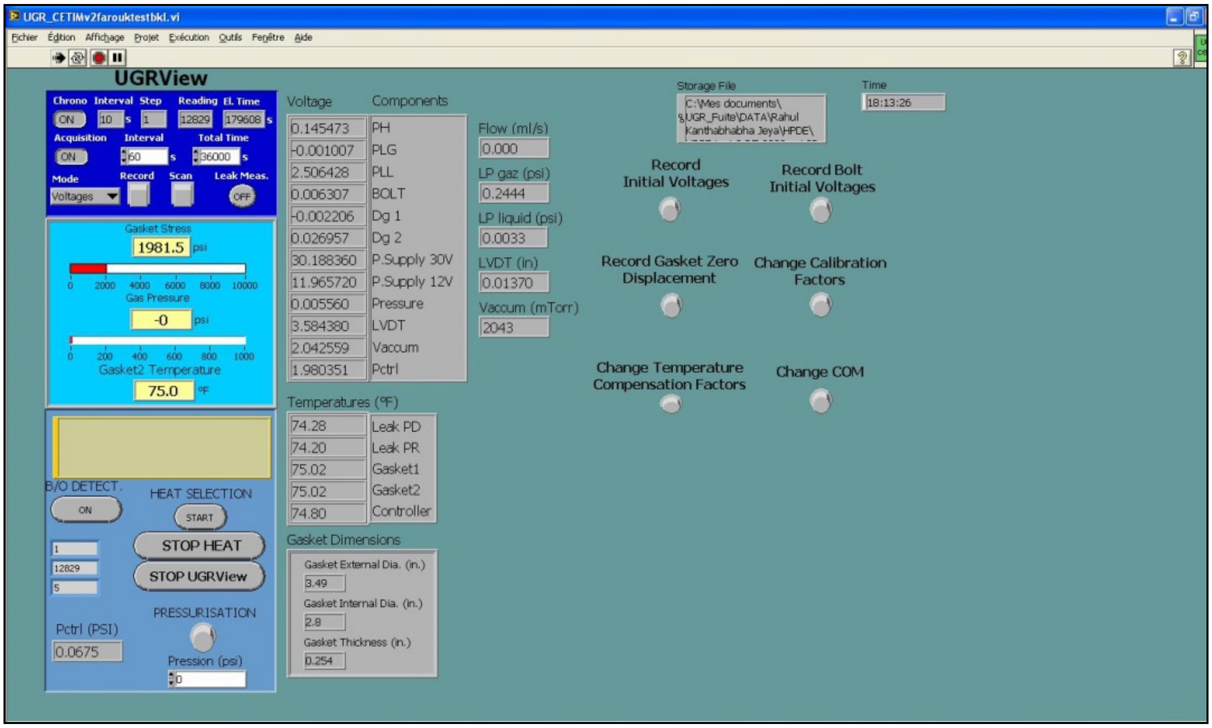

Figure 2. 9 UGR user interface

2.2.5 Test Procedure and Material Properties

As the first step, the material dimensions are measured and fed to the LabVIEW program along with the test conditions. The ring-shaped sample is then placed in between the two platens and it is secured in this position by hand tightening of the nut. Subsequently, the LVDT is adjusted to be in contact with the upper platen and this is position is set as zero or the reference value for axial displacement and the compressive load. Next, the heater and the insulation cover enclose the platens. Later, depending on the test to be performed – ambient or high temperature, compressive load or heating is applied. If it is a room temperature test, the compressive load is applied to the sample using the hydraulic system. The corresponding compressive load or compressive stress value is monitored in the LabVIEW platform. The heating is automated through the LabVIEW program, by which the target temperature for a constant or thermal cycling test condition is achieved. Also, the rate of heating is controlled through the program. In case of high temperature tests, before setting the reference position, the heating command is used to remove the thermal expansion of the material. The temperature is maintained for few hours to insure temperature stabilisation after which the reference is set. Afterwards the desired compressive load is applied to the material. The software enables automation of pressurization and leak measurement system, however, both these features are not used in this research. The time of recording the changes in the parameters under study can be set at any value between 10 and 300 s. Usually for creep study, the time of recording is set at 10 s during the initial few days and then changed to 300s after the material exhibits secondary creep phase. The thermal ratcheting or cycling is achieved through writing a simple program, which controls the heating and cooling with the upper and lower limit of mentioned ratcheting cycle. The machine has capacity of exerting 5 MPa of internal pressure to the specimen under a maximum temperature of 450°C. Nearly, 60 test were conducted using the UGR test bench, the details of which are presented in the following chapters.

Table 2. 1 Material Properties

Properties	ePTFE*	vPTFE*	CNA*	HDPE*	PVC*
Tensile Strength (MPa)	27.57	25	-	43	52
Elastic Modulus (GPa)	2.25	0.75	4.2	1.5	3.3
Density (g/cm^3)	2.15	2.3	1.76	0.96	1.38
Poisson Ratio	0.46	0.46	-	0.45	0.4
Melting Temperature (°C)	327	300	426	150	180

* Material properties are taken from their corresponding material datasheet provided by the manufacuter.

2.3 HOBT Test Bench

The bolted flange joint assembly was developed to perform Hot Blowout Test on gaskets to determine the safe operating temperature for the corresponding gasket type. This test fixture consists of fixed metallic bottom flange with four threaded bolts to accommodate an upper flange in order to perform a leak test or HOBT on gasket materials. The system facilitates for measuring the bolt load, axial displacement and leak rate. High temperature analysis of bolted flange or gasket material can be achieved with HOBT rig. The machine was developed as per the ASME standards so conduct Hot Blowout test on gaskets. The rig can accommodate NPS 3 Class 150 flange, which limits the internal and external diameter of the gasket to 3.5 and 5 in., respectively. The entire unit of HOBT test bench is shown in Fig. 2.10.

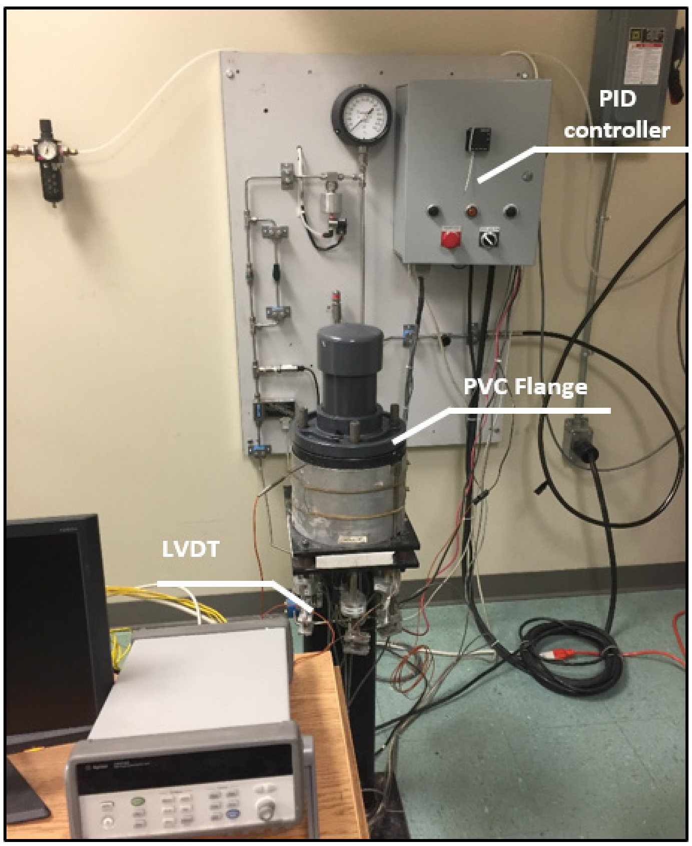

Figure 2. 10 HOBT Test bench

2.3.1 Bolt load and displacement measurement

The induced bolt load on the flange is determined by using vertically fitted strain gauges. Four thread bolts are in contact with four ceramic rods, which are connected to four individual strain gauges through a simple stretch and rest mechanism (Figure 2.11). The bottom end of all four fitted bolts are in contact with their corresponding ceramic rods while the top end is used to the tight the nuts to secure the two flanges together at the desired bolt load. By tightening the nuts on the upper flange, the bolts are stretched which in turn exerts force on the ceramic rod and this causes stretching of strain gauges at the bottom and thereby obtaining the load applied on the bolt. Under relaxation, the stretched strain gauges relaxes with the contraction of the bolts by which the loss of bolt load can be measured. The axial displacement of the gasket or the flange itself without a gasket is measured through a mechanism much similar to the way the bolt load is calculated. The stretching of displacement measuring strain gauges are achieved by the transmitting the axial force through two special screws, which are attached to the upper flange. These two screws push the ball bearings (as shown in the Figure 2.12), thereby stretching the ceramic rods and the strain gauges attached at the bottom. The ceramic rods are used to the avoid the exposure of strain gauges to the test temperatures while performing a test. All the measuring sensors are connected to an Agilent 34970a data acquisition system, which interacts with a computer through a specially developed LabVIEW program. This LabVIEW program facilitates for monitoring and application of external loads on the test sample.

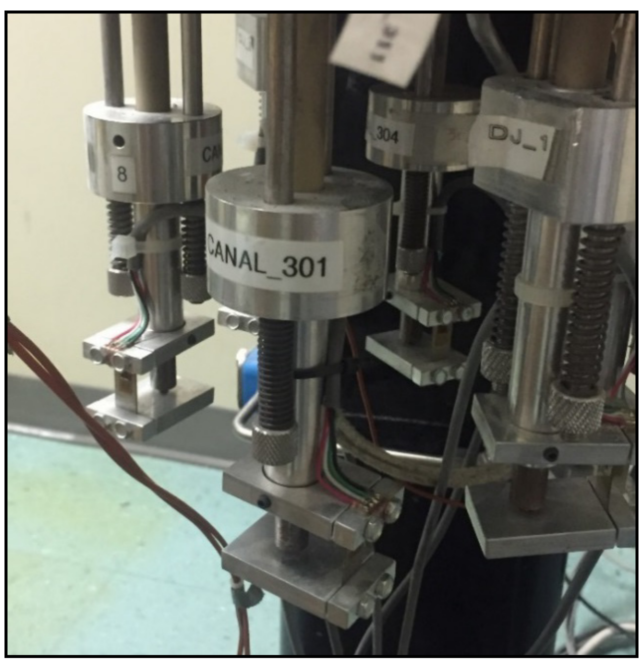

Figure 2. 11 HOBT Bolt load measurement

Figure 2. 12 HOBT displacement measurement system

2.3.2 Heat, Pressurization and Leak measurement

The HOBT test rig is sophisticated enough to apply heat and internal pressure to the bolted flange set-up. The heating is achieved through an electrical cartridge heater, which is placed inside the central stud of the fixture. The heat is transferred to the flange and gasket through convection. Pressurization and leak measurement were not done during the course of this research hence the working mechanism of them are not explained.

2.3.3 Test Procedure, Applied bolt load and Torque Sequence:

Four full scale bolted flange creep-relaxation tests were performed in HOBT test bench with two on HDPE stub flanges and two on PVC flanges. The two tests performed on PVC were at high temperature while one test on high temperature and other on room temperature were conducted on the HDPE stub flanges. Both polymer flanges were investigated without the use of gasket. Since the two materials exhibit significant creep deformation, the use of gasket was avoided. The magnitude of bolt load to be applied on the two types of flange materials are calculated by using the guide provided in PVC technical manual and from the bolt torque manual of plastic pipe institute for PVC and HDPE, respectively. The details of the recommended torque for different flange sizes are given in the following two tables.

$$T = K \times P \times D \tag{2.1}$$

Where: T= tightening torque (in-lbs), K*= dynamic coefficient of friction, P= total bolt load / number of bolts (lbf), D= nominal bolt diameter (in).

Table 2. 2 HDPE bolt load

Nominal Pipe Size	Internal Diameter	External Diameter	Torque	n	Bolt Load	Number of Bolt	Adapter Stress
Inch	Inch	Inch	Ft-lbs		lbs		PSI
1 1/4	1.66	2.75	29	0.2	2784	4	2950
1 1/2	1.9	3.3	29	0.2	2784	4	1948
2	2.375	3.85	29	0.2	2784	4	1544
3	**3.5**	**5**	**41.5**	**0.2**	**3984**	**4**	**1591**
4	4.5	6.54	41.5	0.2	3984	8	1802
5	5.563	7.55	55	0.2	4400	8	1720
6	6.625	8.6	62.5	0.2	5000	8	1694

Table 2. 3 PVC Recommended Torque

Flange Size (in.)	Recommended Torque (ft. lbs.)
1/2 - 1-1/2	12
2 - 4	25
5	30
6 - 8	40
10	64
12	95
14 - 24	110

The standard torque sequence is detailed in the following figure, which is a crisscross pattern. The figure is referenced from SPEARS 2014.

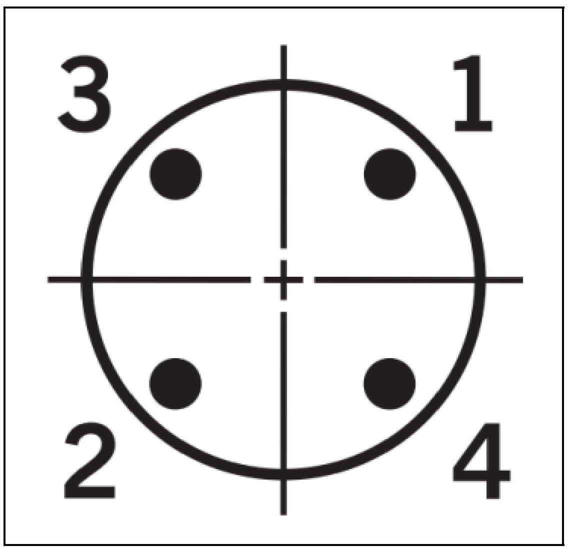

Figure 2. 13 Torque Sequence (SPEARS, 2014)

2.4 Finite Element Modeling

For the performing numerical simulation of creep-relaxation behavior of HDPE and PVC flanges, finite element model was developed and analyzed using ANSYS Workbench platform. To simulate the creep model, a good understanding of the creep behavior is essential. The term creep is defined as the deformation induced in the material over time when subject to a constant load below the yield point of the material. In general, the creep is studied in three stages or phase namely primary creep, secondary creep and tertiary creep. As the name indicates, it represents the first, second and final stages of the creep life of a material. The primary creep is the instantaneous or rapid deformation caused under constant load. The secondary creep is the longest phase, where a gradual increase in deformation occurs. This phase usually take place for several years for many materials. The tertiary stage is fast and it causes ultimate failure of the material structure. The different stages of creep are dissipated in Figure 2.14.

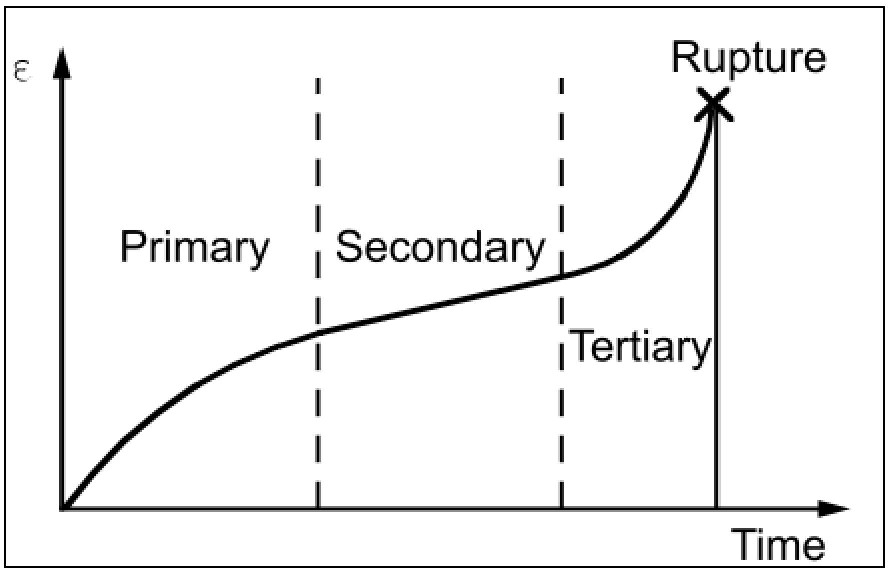

Figure 2. 14 Creep stages (ANSYS, 2016)

The creep vulnerability of HDPE and PVC are scrutinized in few scientific publications. This gradual deformation can cause catastrophic disaster when utilized in long-term applications. Typical to bolted joints, creep causes reduction in the applied force of the bolts, which causes stress relaxation and failure. Importance of creep and relaxation while designing pumps and pressure vessels are discussed (Johnson and Roth, 2002).

2.4.1 Creep Analysis using ANSYS:

ANSYS Workbench has inbuilt creep equations for creep analysis of structures. The creep equations in ANSYS are categorised into two types, first one is the implicit creep equations and the other is explicit creep equations. Out of the two variant, the most commonly used method is the implicit equations. The reasons for the popularity of implicit creep equations over explicit equation is that implicit equations are accurate, rapid and stable. This method of analysis can perform long-term simulation and it works well with larger creep strain. Since many polymer materials exhibit big creep strain, implicit creep equations are the apt method of study. Moreover, pure creep or creep with isotropic plasticity can be evaluated through this method. The superiority of implicit equations over explicit is highlighted by the fact that the implicit method can calculate creep and plasticity simultaneously, thereby making it more accurate and efficient of the two. In addition to the time-dependence considerations, the implicit method includes temperature-dependent constant. Contrary to implicit equations, the explicit method is good for small time steps. Furthermore, the explicit equations are limited by its inability to execute creep and plasticity at the same time and by lack temperature-dependent constants in the equation. However, to overcome the temperature dependency problem, the Arrhenius function can be used. Also, with the combined evaluation of alternate plasticity options and explicit creep, the software can achieve simultaneous analysis of creep and plasticity but the plastic behavior is calculated before the creep.

Some of the available implicit creep equations in ANSYS are given the following table.

Table 2. 4 Implicit Creep Equations

Creep Model (*TBOPT*)	**Name**	**Equation**		**Type**
1	Strain Hardening	$\dot{\varepsilon}_{cr} = C_1 \sigma^{C2} \varepsilon_{cr}{}^{C3} e^{-C4/T}$	$C_1>0$	Primary
2	Time Hardening	$\dot{\varepsilon}_{cr} = C_1 \sigma^{C2} t^{C3} e^{-C4/T}$	$C_1>0$	Primary
3	Generalized Exponential	$\dot{\varepsilon}_{cr} = C_1 \sigma^{C2} r e^{-rt}$, $r = C_5 \sigma^{C3} e^{-C4/T}$	$C_1>0$, $C_5>0$	Primary
4	Generalized Graham	$\dot{\varepsilon}_{cr} = C_1 \sigma^{C_2} (t^{C_3} + C_4 t^{C_5} + C_6 t^{C_7}) e^{-C_8/T}$	$C_1>0$	Primary
5	Generalized Blackburn	$\dot{\varepsilon}_{cr} = f(1 - e^{-rt}) + gt$ $f = C_1 e^{C_2 \sigma}$, $r = C_3 (\sigma / C_4)^{C_5}$, $g = C_6 e^{C_7 \sigma}$	$C_1>0$, $C_3>0$, $C_6>0$	Primary
6	Modified Time Hardening	$\dot{\varepsilon}_{cr} = C_1 \sigma^{C_2} t^{C_3+1} e^{-C_4/T} / (C_3 + 1)$	$C_1>0$	Primary
7	Modified Strain Hardening	$\dot{\varepsilon}_{cr} = \{C_1 \sigma^{C_2} [(C_3 + 1)\varepsilon_{cr}]^{C_3}\}^{1/(C_3+1)} e^{-C4/T}$	$C_1>0$	Primary
8	Generalized Garofalo	$\dot{\varepsilon}_{cr} = C_1 [\sinh(C_2 \sigma)]^{C_3} e^{-C_4/T}$	$C_1>0$	Secondary
9	Exponential form	$\dot{\varepsilon}_{cr} = C_1 e^{\sigma / C_2} e^{-C_3/T}$	$C_1>0$	Secondary
10	Norton	$\dot{\varepsilon}_{cr} = C_1 \sigma^{C_2} e^{-C_3/T}$	$C_1>0$	Secondary
11	Combined Time Hardening	$\varepsilon_{cr} = C_1 \sigma^{C_2} t^{C_3+1} e^{-C_4/T} / (C_3 + 1)$ $+ C_5 \sigma^{C_6} t e^{-C_7/T}$	$C_1>0$, $C_5>0$	Primary + Secondary
12	Rational polynomial	$\dot{\varepsilon}_{cr} = C_1 \frac{\partial \varepsilon_c}{\partial t}$, $\varepsilon_c = \frac{cpt}{1 + pt} + \dot{\varepsilon}_m t$ $\dot{\varepsilon}_m = C_2 10^{C_3 \sigma} \sigma^{C_4}$ $c = C_7 \dot{\varepsilon}_m^{C_8} \sigma^{C_9}$, $p = C_{10} \dot{\varepsilon}_m^{C_{11}} \sigma^{C_{12}}$	$C_2>0$	Primary + Secondary
13	Generalized Time Hardening	$\varepsilon_{cr} = f t^r e^{-C_6/T}$ $f = C_1 \sigma + C_2 \sigma^2 + C_3 \sigma^3$ $r = C_4 + C_5 \sigma$		Primary

where: ε_{cr} = equivalent creep strain, $\dot{\varepsilon}_{cr}$ = change in equivalent creep strain with respect to time, σ = equivalent stress, T = temperature (absolute), C_1 through C_{12} – constants, t = time at end of sub step, , e = natural logarithm base.

2.4.2 Modeling and Boundary conditions

One of the basic or fundamental building block of a good simulation is the creation of exact geometrical structure for analysis. ANSYS has inbuilt design modeller which can used directly to develop the model or it supports products from several different 3D design software. For this research, both techniques were used. The design of HDPE stub flange (Figure 2.15 a) was done in CATIA design software while the PVC flange (Figure 2.15 b) was modeled in ANSYS. Dimensions of the flanges are acquired from the data sheet provided with purchased flanges. Also, since the flanges are symmetrical in nature only 1/8th of the entire shape of the structure was modeled. This simplified model enables faster calculation and limits the requirement for use of high performing super computers.

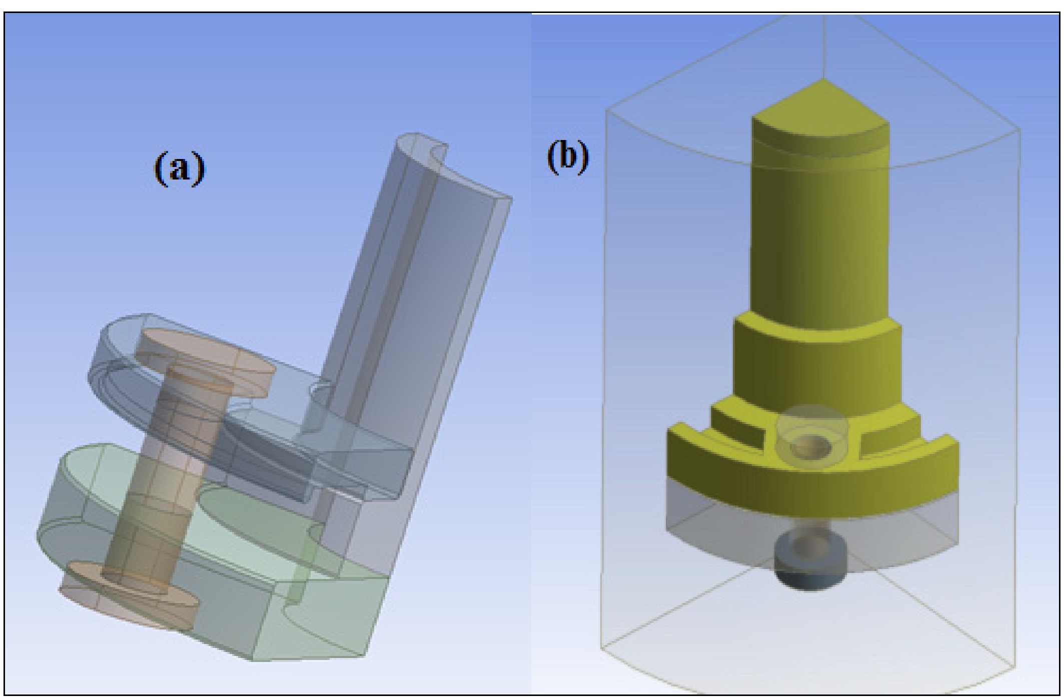

Figure 2. 15 (a) HDPE modeling in CATIA, (b) PVC model in ANSYS.

Because of the symmetry of the structure, both flanges are constrained at the two sides of the cut section and also at the bottom side of the flange. The rotations in the radial direction made to zero and the displacements are limited to their corresponding planes. The displacement constrain at the bottom of the flange is for restricting motion in the upward direction. Figure 2.16 show the boundary condition applied in PVC simulation.

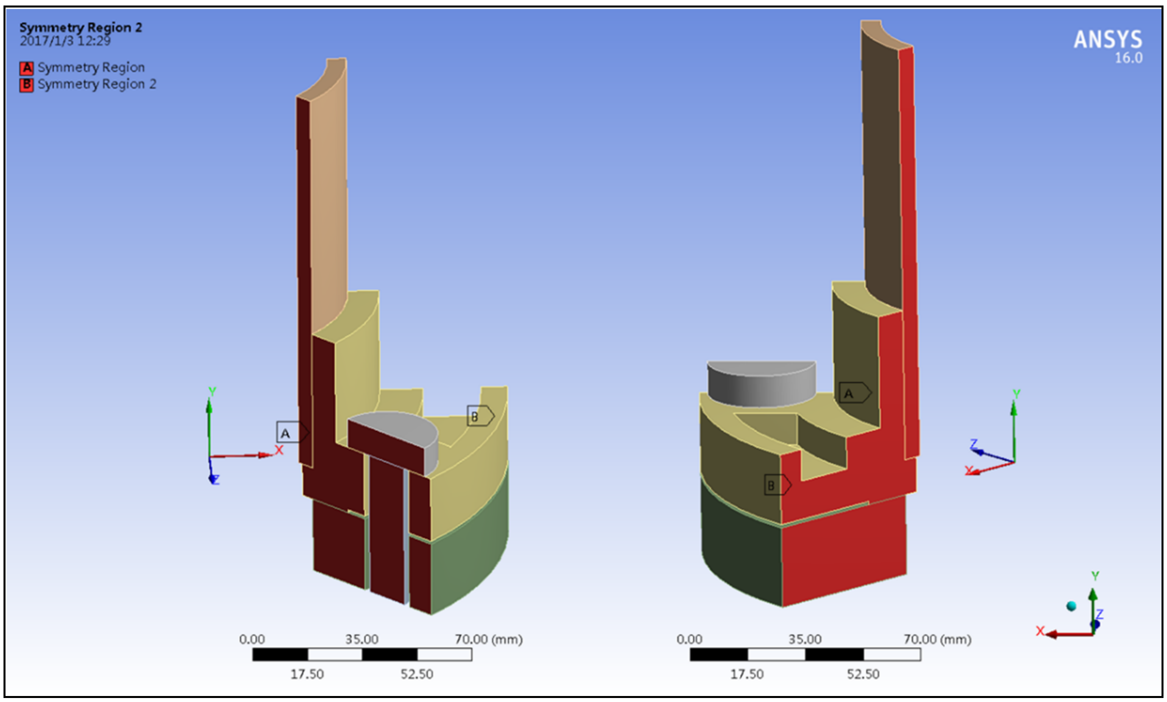

Figure 2. 16 Boundary condition in ANSYS

2.4.3 Bolt Pretension

Finally, the replica of applying torque load on the bolts of the HOBT test rig is achieved by using the pre-existing command on ANSYS called Bolt Pretension. This option supports for the application of axial bolt load. The command (Figure 2.17) requires the application of load and subsequently maintaining the load by using the lock option. The load applied for the two flanges types are given the below table. A special coordinate system was developed at the middle of the bolt to apply the pretension load along the axial direction of the bolt.

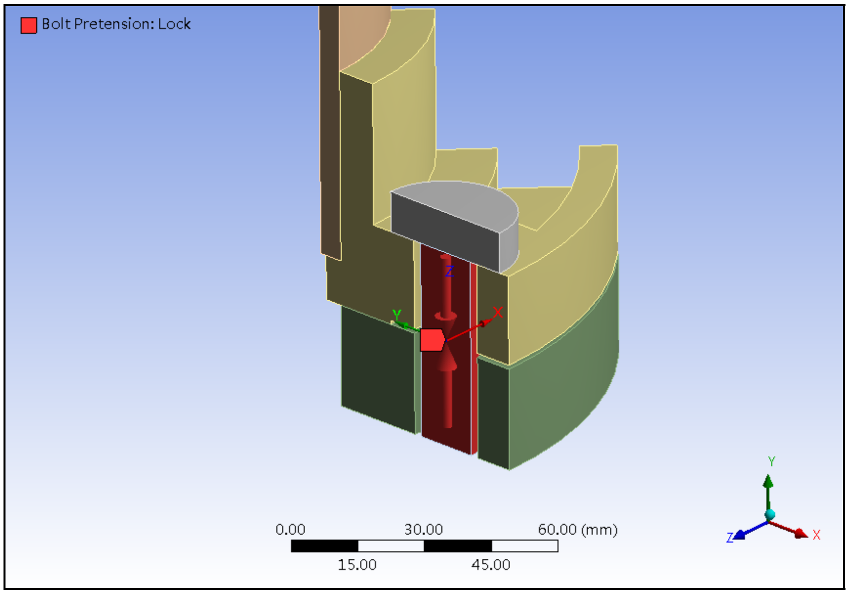

Figure 2. 17 Bolt Pretension

Table 2. 5 Bolt Pretension Load

Steps/Material	Load (N)	Lock	Lock
PVC	3426	Yes	N/A
HDPE	3984	Yes	N/A

CHAPTER 3

CREEP AND THERMAL RATCHETING CHARACTERIZATION OF POLYTETRAFLUOROETHYLENE-BASED GASKET MATERIALS

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This article is published in the Journal of Advanced Materials Proceedings, volume **2**(10), 609-614, 2017, DOI: 10.5185/amp.2017/027

3.1 Abstract

Characterization of Teflon polymer based gaskets under expedited aging is the objective of this work. Teflon gaskets are exploited frequently as a replacement to asbestos fiber gaskets because of their excellent leak tightness and nonhazardous physical degradation properties. The research focuses profoundly on the adverse influence of temperature and thermal cycles on the creep and cumulative damage phenomenon under compressive load. Virgin and expanded PolyTetraFluoroEthylene (PTFE) are tested under 28 and 41 MPa of gasket stress at different temperatures. Intricate analysis of creep under coalesces of thermal ratcheting and principal stress is achieved through Universal Gasket Rig (UGR). The instigated cumulative damage is distinguishable into upper and lower bound temperature region indicating the escalation and decrease of thickness change during cycling which saturates after 12 thermal cycles for expanded PTFE while no saturation is reached for virgin PTFE in even after 20 thermal cycles. Percentage of thickness reduction at different applied stress is nearly the same for virgin PTFE whereas expanded PTFE shows largest reduction under lower stress. Compressive creep bespeaks the impact of temperature and load, thereby dictating the magnitude of ratcheting damage and contrariwise. Finally, the creep and thermal ratcheting has a proliferating effect on value of the coefficient of thermal expansion for all chosen gaskets.

3.2 Introduction

Polytetrafluoroethylene is one of the sought-after material for gasket components. Its excellent leak tightness and chemical resistant made it stand out among conventional gaskets. In addition to high temperature applicability, PTFE are nonhazardous degradable material in contrast to the asbestos fiber gaskets making them appropriate for aggressive fluid and corrosive environment applications. In spite of the advantages, one of the major drawback for PTFE material is the creep response to compressive load while the other is the extrusion failure when utilized in class 150 and 300 pipe flanges under particular environments (Keywood, 1994; Winter and Keywood, 1996). This lead to the development of a standardized procedure on relaxation and blowout characteristics of PTFE based gaskets (Derenne et al., 1999). The standard test procedure is developed on a Nominal Pipe Size (NPS) 3 class 150 flange joint fixture with relaxation capabilities.

Researches (Payne et al., 1990; Payne et al., 1987) evaluated the test method for characterizing of non-asbestos gasketing material at elevated temperature. However, numerous gaskets are operated under cyclic temperature environment and climatic discrepancies creating the necessity to inspect the damage under ratcheting. The thermal ratcheting or cycling of temperature on the cumulative creep damage of PTFE materials is of importance in bolted gasketed joints. Thermal ratcheting induces cumulative damage on the gasketed material leading to its thinning. This generates a further loss of compressive load on the gasket projecting for radial extrusion under the internal pressure and instigating failure by blow out. Only few reported literature (Bouzid et al., 2001; Bouzid et al., 2000; Bouzid and Benabduallah, 2015; Bouzid 2011; Marchand et al., 1992) investigated on to the thermal ratcheting phenomenon of PTFE based materials but none studied the coupling of creep and thermal ratcheting.

Literatures (Bouzid and Benabdullah, 2015; Bouzid, 2011) explored into the effect of thermal ratcheting and applied load on the thermal expansion of PTFE nonetheless these researches were limited to couple of thermal cycles and a maximum ratcheting temperature of 204°C.

Coefficient of thermal expansion is an important characteristic for the modern design codes of bolted joints including finite element analysis. The prevailing test standards developed by the American Society of Testing and Materials (ASTM E 228-11, 2016; ASTM E 831-14, 2014; ASTM D 696-16, 2016) do consider the effect of load induced creep phenomenon. The work by Bhattachar (1997) on instantaneous coefficient of linear thermal expansion is independent of reference temperature in contrary to the ASTM E228 and E289. However, the effect of thermal ratcheting on polymer materials is not addressed. While independent researches by Kirby (1956) and Touloukian (1977) have reported quantitative results on coefficient of thermal expansion for PTFE and other polymer materials respectively, none scrutinized the behavior under creep and thermal ratcheting at high compressive loading.

Converging from the literature search the objective of this research is focused on comprehending the creep and thermal ratcheting response of expanded and virgin PTFE materials. In excess, elaborated information on the thickness reduction of gasketing material and coefficient of thermal expansion under compressive load, creep and thermal ratcheting behavior are attempted.

3.3 Experimentation and test procedure

The creep and thermal ratcheting of gasket materials are studied through Universal Gasket Rig (UGR) shown in Figure 3.1. The UGR has the capacity to conduct both mechanical and leakage characterization test to provide for multiple physical properties of gasket materials. The experimental test bench composed of two platens (upper and lower) between which the gasket material is compressed by means of a manual hydraulic system. The platen accommodates gasket of minimum inner diameter of 50 mm and maximum outer diameter of 100 mm with the thickness up to 10 mm. The inbuilt test rig facilitates the application of internal pressure and temperature on to the gasket as illustrated in Figure 3.1 and 3.2. The maximum controlled gasket internal pressure of 5 MPa is achievable at the limit temperature of 450^{o}C.

The highlight of Universal Gasket Rig is to simultaneously measure the creep and thermal ratcheting of materials under high compressive loads. High sensitive Linear Differential Variable Transformer (LVDT) is used to monitor the change in thickness under creep and cumulative damage phenomenon. A central stud and hydraulic bolt tensioner operated by a manual hydraulic pump is used to impose the desired compressive stress on the gasket between two platens. A full bridge strain gauge fixed to the central stud measures the load imposed on the gasket with the information of gasket dimensions (Table 3.1).

Table 3. 1 Gasket dimensions.

Material Type	Outer Diameter (mm)	Inner Diameter (mm)	Thickness (mm)
ePTFE	76.2	45.72	3.2
vPTFE	75.4	45.72	3.2

The hydraulic tensioner is connected with an accumulator to maintain a relatively constant load on the gasket while thickness of the gasket reduces. The machine is capable of applying a maximum gasket stress of 69 MPa on a gasket area of 645.16 mm2. Specially designed inlet and outlet ports in the upper platen are available to pressurize the internal gasket surface and measure the leak rate, when required.

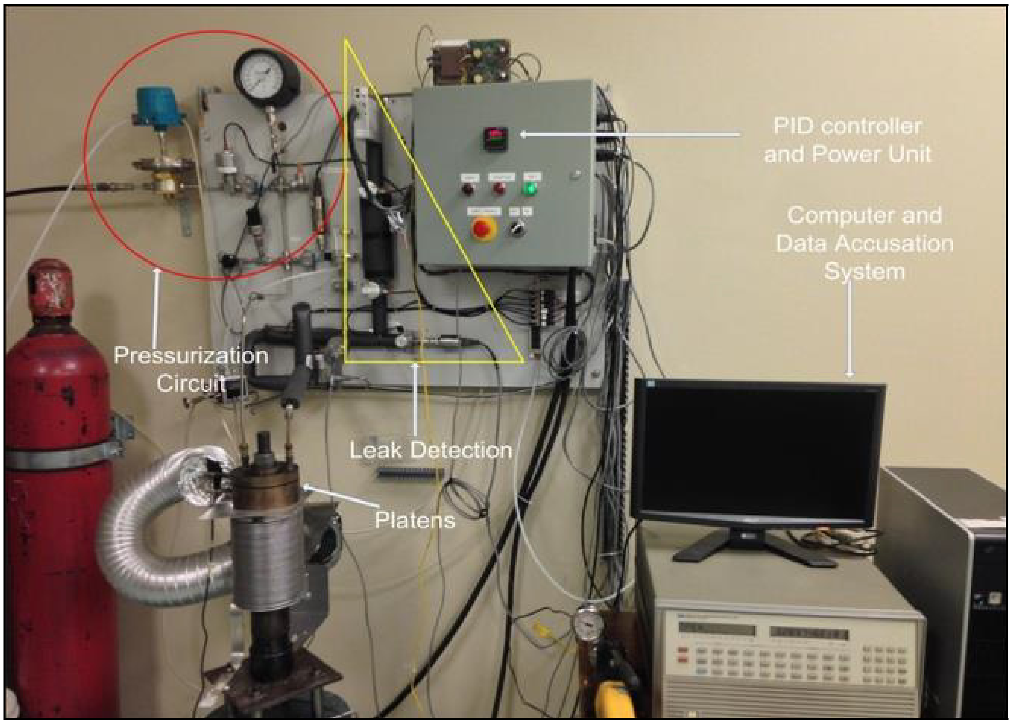

Figure 3. 1 Universal Gasket Rig

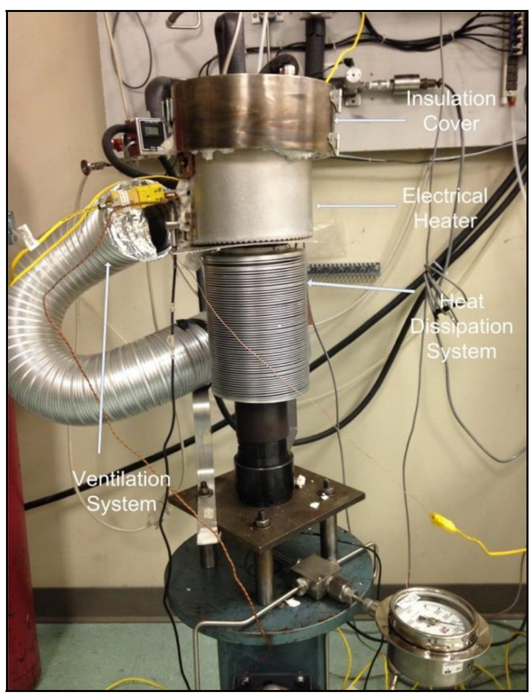

Figure 3. 2 Heating system – UGR

The heating of gasket is achieved through an electrical ceramic band heater, which is enfolded around the platens to transfer the heat by conduction to the gasket material. A Proportional Integral Derivative (PID) controller is used to control the temperature of the heater by monitoring the temperature of gasket materials through thermocouples which are connected to a computer through data acquisition and control system. The power unit consists of electrical control panel for the heater with on-off switch, emergency stop button and digital monitor for set and current temperature of the platens and heater system. The heat applied in ramp of 1.5°C/min, which is a representation of most bolted gasketed joint applications. Special insulation cap and fiber materials are used to avoid any loss of heat to the surroundings. The system is cooled through natural convection after the shut-off of heater. The rigidity is controlled through the use of Belleville washers.

The experimental procedure begins with the measurement of gasket dimensions for the determination of applied gasket load through the strain gauge. Initially, the gasket is compressed between the two platens manually by hand tightening of a nut on the central stud. This position is set to be the zero reading for gasket load and displacement. The gasket material is compressed to the desired load through the hydraulic system which is followed by heating at a rate of 1.5°C/min. Data acquisition system monitors readings every 10s while recording them every 60 seconds for post processing. Cycling of temperature to induce thermal ratcheting phenomenon is accomplished through automation with PID controller. Different gasket materials are scrutinized under different test conditions depending on their material properties, which are elaborated in Table 3.2. In general, the gaskets are compressed and subjected to 24 hours short term creep and then ratchetted with temperature to study the coupled damage and estimating the perennial property of the material. LabVIEW program is used to monitor and record various parameters of the system to characterize the gasket behavior.

The coefficient of thermal expansion is calculated with respect to reference (Bouzid et al., 2001), which elaborates the importance for measuring the property during the cool down cycle. The axial displacement or thickness variation measured by the LVDT sensor is thermally compensated to counter act the effect of heat, which is negligible in terms of gasket load.

3.4 Results and discussion

The results extracted from the compressive creep and thermal ratcheting tests on the selected PTFE based gasket materials provided valuable insights into the coupling behavior of the two-damage phenomenon. The compressive creep on 3.2 mm thickness expanded and virgin PTFE is shown in Figure 3.3a. It is clearly seen that the compressive creep for virgin PTFE is higher than the expanded PTFE under a lower gasket stress than the latter. The difference is sighted to the rigidity of the two materials where the expanded is soft while the virgin is harder. As the initial reduction of thickness under compressive load is significant for expanded, it exhibits better resistance to creep.

It is important to note that the secondary creep rate is slightly higher for virgin than expanded PTFE, which is alleged to extreme thickness loss and small growth of creep curve even after 5 days of compressive creep testing. Figure 3.3b illustrates the influence of magnitude of compressive load on the creep behavior for virgin PTFE gaskets. As suspected the compressive creep increases with increase of applied gasket stress level. As expected the primary creep rate if higher when the extent of load is higher.

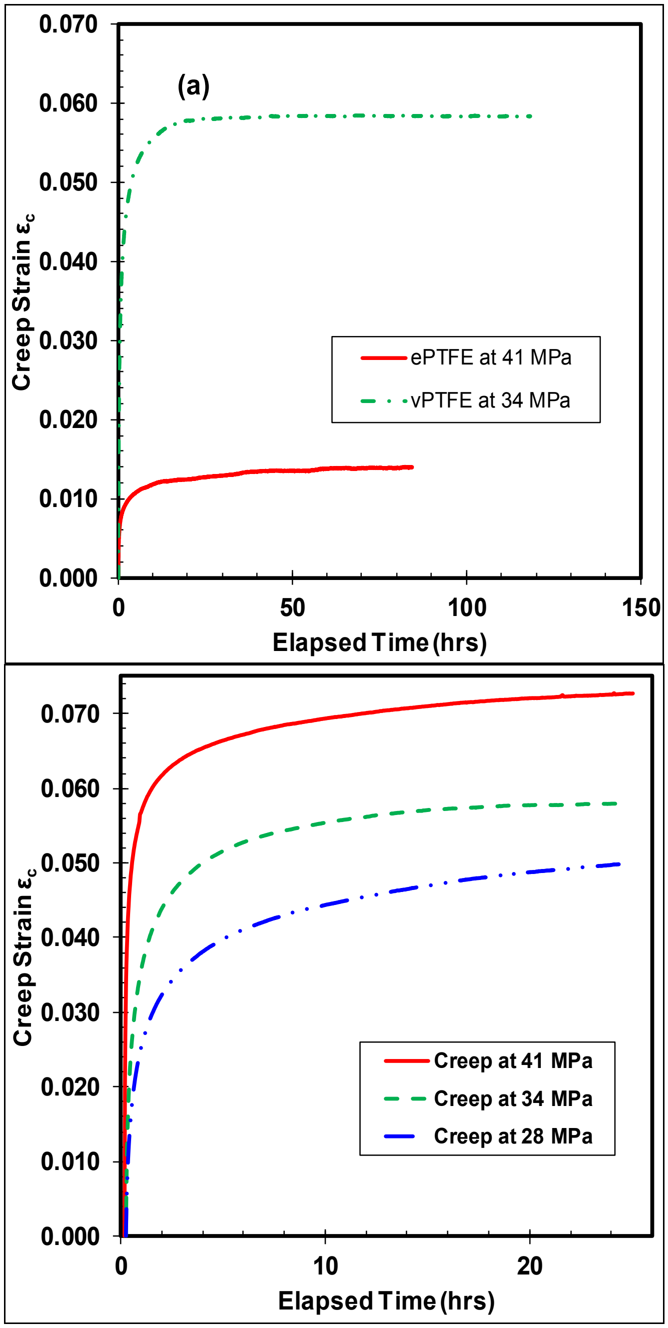

Figure 3. 3 Compressive Creep (a) comparison between expanded and virgin PTFE, (b) Compressive creep response under different loads-virgin PTFE.

The cumulative damage incurred due to the cycling of temperature or thermal ratcheting is significant in two types of selected gasket materials under different ratcheting temperature as mentioned in Table 3.2. The characterization of damage due to thermal ratcheting is visualized through decrease of gasket thickness after each cycling of escalation and decline of temperature. The originated cumulative damage under ratcheting of temperature is distinguishable into upper and lower bound region indicating the thickness change during the cycling. The expanded PTFE material under thermal ratcheting exhibits saturation of the cumulative damage around the 12th cycle as shown in Figure 3.4a. The magnitude of damage depends on the applied load and ratcheting temperature as evident from the Figure 3.4a.

Table 3. 2 Thermal ratcheting and creep test parameters.

Expanded Polytetrafluoroethylene			
Test title	Gasket Stress	Ratcheting Range	Number of cycles
Test 1	28 MPa	38-260ºC	20
Test 2	41 MPa	38-260ºC	20
Test 3	41 MPa	Ambient	1 (5 days)
Virgin Polytetrafluoroethylene			
Test title	Gasket Stress	Ratcheting Range	Number of cycles
Test 1	35 MPa	Ambient	1 (5 days)
Test 2	41 MPa	38-177ºC	20
Test 3	28 MPa	38-177ºC	20

The response of virgin PTFE to thermal ratcheting (Figure 3.4b) is quite similar to expanded PTFE but do not display saturation of damage even after 20 cycles. As compared to expanded PTFE, the virgin gasket's thickness change due to expansion-contraction under consecutive thermal cycles varies tremendously especially in the initial few cycles.

The adverse influence of thermal ratcheting is clearly shown in the graph of percentage of thickness reduction versus number of thermal ratcheting cycles. As anticipated, virgin PTFE (Figure 3.5b) exhibited higher proportion of material thickness loss in comparison to the expanded PTFE (Figure 3.5a). The reason is attributed to the inherent rigidity of the two materials or their capacity to resist load. Initial thickness reduction of 73% for expanded gaskets as compared to the 33% drop for virgin PTFE under stress of 28 MPa justifies lower loss in the case of the former. Therefore, the relatively greater loss of thickness for expanded gaskets under initial loading is proposed as the reason for lesser percentage of thickness reduction under thermal ratcheting. Virgin PTFE exhibits similar percentage of thickness reduction when subjected to same ratcheting temperature conditions with two different applied stress of 28 and 41 MPa.

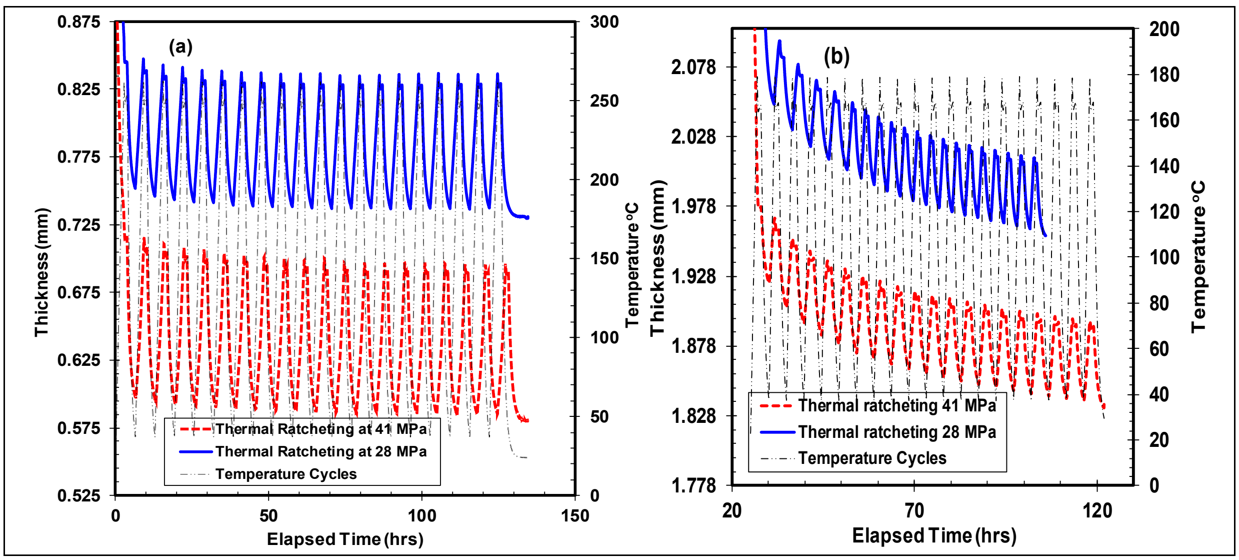

Figure 3. 4 Thermal ratcheting (a) Expanded PTFE,
(b) Virgin PTFE

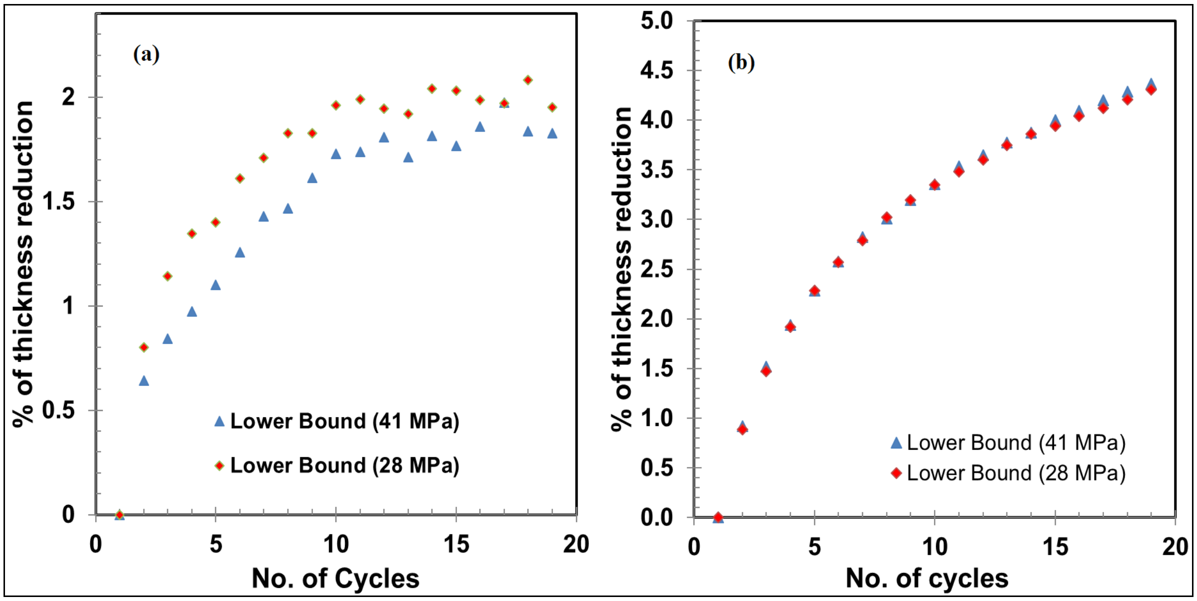

Figure 3. 5 Percentage of thickness reduction due to ratcheting (a) Expanded PTFE, (b) Virgin PTFE.

Nevertheless, a ratcheting test, in excess of 20 thermal cycles, preferably till saturation would lead to clearer understanding on the influence of stress level for virgin PTFE materials. Figure 3.5a displays the percentage of thickness reduction for same ratcheting temperature at two stress levels. Constructive effect with rise of applied load is evident due to the fact that the expansion is higher with lower applied load.

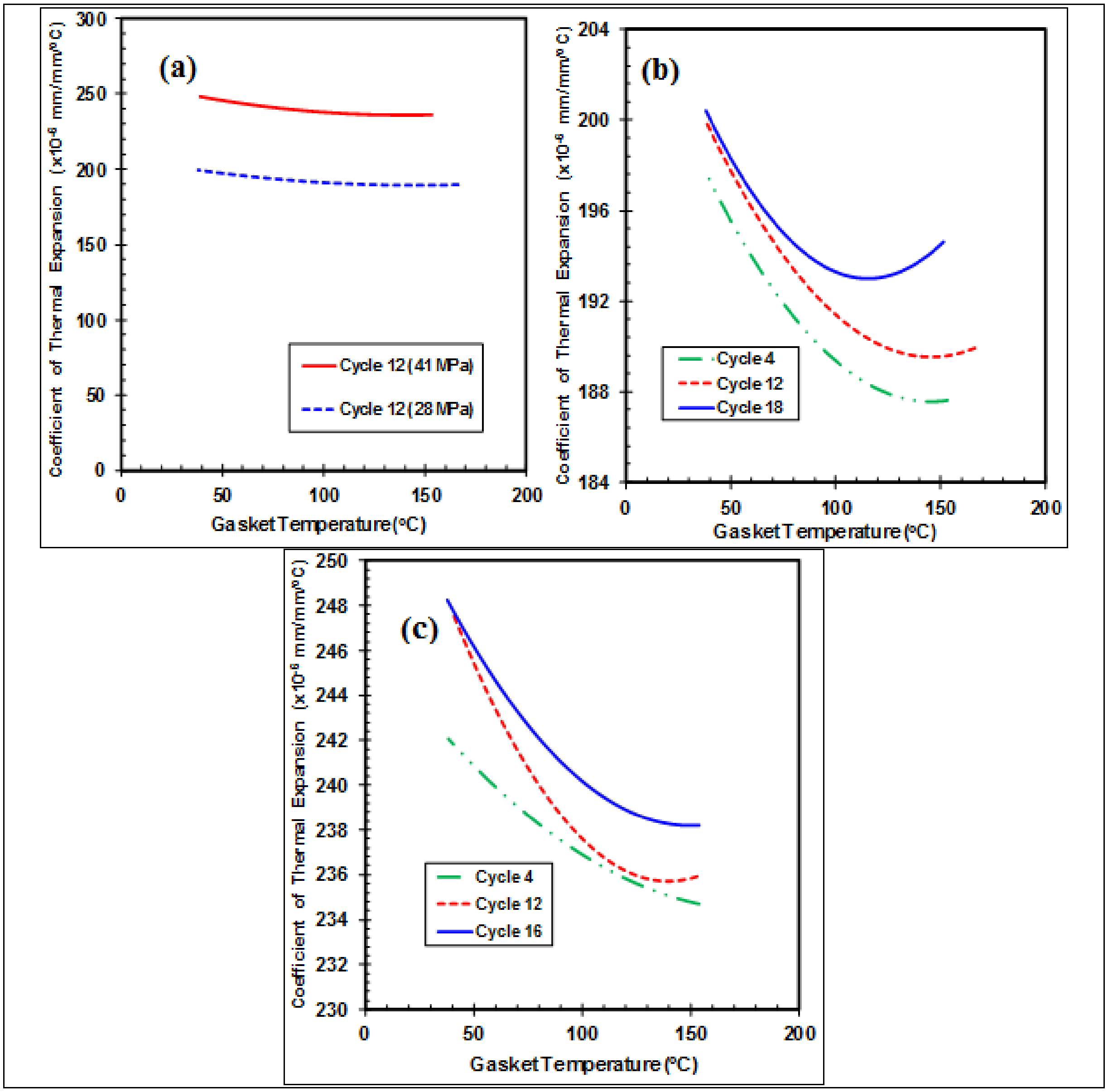

Figure 3. 6 Coefficient of thermal expansion – Virgin PTFE
(a) under applied load, (b) under different ratcheting cycles for 28 MPa,
(c) under different ratcheting cycles for 41 MPa.

The coefficient of thermal expansion (CTE) of the selected Teflon based materials are influenced by both applied stress and thermal ratcheting. From Figure 3.6a, the appreciable rise in thermal expansion coefficient for virgin PTFE material between 28 and 41 MPa of compressive stress level is evident. CTE intends to surge with respect to the rise in applied load. The thermal ratcheting effect intensifies the surge of CTE with each thermal cycling till saturation is achieved as seen in case of expanded PTFE material. Figure 3.6b and Figure 3.6c illustrates the variation of coefficient of thermal expansion with respect to thermal cycles under 28 and 41 MPa of compression, respectively. While the uniaxial test conditions are replicated as to the standard tests, the data can be used in design and finite element analysis of bolted flange connections.

3.5 **Conclusion**

Assessment of thermal ratcheting and short-term creep response of PTFE based gaskets materials are performed through Universal Gasket Rig. Both types of gasket material exhibited substantial thinning and deformation; two material properties that are of major importance in gasketing product. Expanded PTFE gasket unveiled better resistance to creep and cumulative damage due to thermal ratcheting in comparison to virgin PTFE materials because it gets much thinner under load. Thermal ratcheting damage tends to get saturated around 12th cycle for expanded PTFE material while virgin PTFE material continued to reduce in thickness throughout 20 cycles of test. The coefficient of thermal expansion of these materials varied notably with applied load and thermal ratcheting where the contribution of former is higher than the latter. Hence, test results show the need for development of comprehensive standard test procedure to determine the thermal ratcheting, creep and co-efficient of thermal ratcheting for PTFE based gasketing materials under load.

CHAPTER 4

COMPRESSION CREEP AND THERMAL RATCHETING BEHAVIOR OF HIGH DENSITY POLYETHYLENE (HDPE)

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This article is published in the Journal of Polymers, volume 10(2), 156, 2018, doi:10.3390/polym10020156

4.1 Abstract

The characterization of thermal ratcheting behavior of high-density polyethylene (HDPE) material coupled with compressive creep is presented. The research explores the adverse influence of thermal cycling on HDPE material properties under the effect of compressive load, number of thermal cycles, creep time period, and thermal ratcheting temperature range. The compressive creep analysis of HDPE shows that the magnitude of creep strain increases with increase in magnitude of applied load and temperature, respectively. The creep strain value increased by 7 and 28 times between least and maximum applied temperature and load conditions, respectively. The creep modulus decreases with increase in compressive load and temperature conditions. The cumulative deformation is evident in the HDPE material, causing a reduction in the thickness of the sample under thermal ratcheting. The loss of thickness increases with increase in the number of thermal cycles, while showing no sign of saturation. The thermal ratcheting strain (TRS) is influenced dominantly by the applied load condition. In addition, the TRS decreases with increase in creep time period, which is cited to the extended damage induced due creep. The results highlight the need for improved design standard with inclusion of thermal ratcheting phenomenon for HDPE structures particularly HDPE bolted flange joint.

4.2 Introduction

In recent times, the polymer or plastic materials have seen a rapid growth in replacing the conventional metallic piping structures, mainly due to their economical production cost and minimal dependence and impact on the environment. The typical advantages of polymers over metallic materials are extensive protection against chemical and corrosion attacks, extended service life, and that they are lightweight with high strength and modulus. Amongst the different types of polymeric materials commercially available, high density polyethylene (HDPE) polymer has the second largest share of spoils behind polyvinyl chloride (PVC). HDPE is a good candidate for application in chemical fluid and slurry transfer pipes, because of its excellent chemical resistance and near frictionless flow characteristics. The applicability of HDPE material has seen a recent boom in the piping system against PVC, due to its excellent resistance to fatigue and UV radiation. The dominance of HDPE pipe in urban service piping network for water and gas, nuclear service water, and desalination piping, is evident. Similar to most polymer materials, the research on characterizing HDPE material properties is abundant. Since the early eighties, a large number of research studies have focused on the creep property of polymers, as this phenomenon is perceived as a hindrance and a drawback of polymer materials. Quantitative data on creep, and other perennial properties of two varieties of PVC and polyethylene materials under liquid pressure at different temperatures, was published by Niklas and Eifflaender (1959). The creep behavior of thermoplastics at temperatures close to the glass transition region of polymers was studied by Bergen (1976).

The viscoelastic creep response of high-density polyethylene is explored in two creep models, a viscoplastic model and a nonlinear viscoelastic model, which on being fitted with experimental data, gave near perfect and moderate accuracy, respectively (Zhang and Moore, 1997; Zhang and Moore, 1997). The developed nonlinear creep model of high-density polyethylene has a good agreement with the experimental data, including the effect of ageing (Lai and Bakker, 1995). The recent enthusiasm towards viscoelastic property has lead into probing of the viscoelastic and viscoplastic behavior of HPDE under cyclic loading conditions (Colak and Dunsunceli, 2006). Crack initiation and propagation of ductile and brittle polyethylene resin under creep damage show that only the brittle resin exhibits a lifespan controlled by slow crack growth (SCG) (Hamouda et al., 2001).

Researchers (Ries et al., 2013; Ries et al., 2013) studied the impact of strain rate and temperature on tensile properties of the post-consumer recycled HPDE. A large quantity of research focused on the mechanical cycling or fatigue behavior of HDPE material (Dusunceli et al., 2010), HDPE geogrid (Cardile et al., 2016), solid extruded HDPE (Kaiya et al., 1989), HDPE composite (Dong et al., 2011), HDPE pipe joints (Chen et al., 1997), but almost none on the thermal ratcheting effect. The mechanical property of filled HDPE and the effect of loading and manufacturing method on the properties of HPDE are well documented in the studies (Khalaf, 2015; Dusenceli and Aydemir, 2011) respectively. Additionally, statistical analysis of HDPE fatigue life is performed (Khelif et al., 2001). The influence of cyclic loading rates under different temperature conditions on the cyclic creep behavior of polymers and polymer composites was examined by Vinogradav and Schumaker (2001). The brittle and ductile failure under creep rupture testing of high-density polyethylene pipes are thoroughly researched (Krishnaswamy, 2005). There are no typical references available on the creep data of polymeric bolted flange joint subjected to compression. In metallic bolted flange gasketed connections, the gasket component is usually blamed for relaxation, and hardly ever the flange material itself (Bouzid and Chaaban, 1997). However, such is not the case with polymeric flanges, hence, quintessential analysis of compressive creep behavior is a necessity.

Furthermore, most polymer materials have restrictive features of low operational temperature conditions, thereby making them vulnerable to any temperature fluctuations. Consequences of thermal ratcheting or cycling of temperature on polymers can be severe, yet remain a relatively rare phenomenon in reported scientific literature. The work on the hot blowout testing procedure for polytetrafluoroethylene (PTFE) based gaskets (Bouzid and Benabdullah, 2015) and the creep–thermal ratcheting analysis of PTFE based gaskets

(Kanthabhabha Jeya and Bouzid, 2017) are a few examples of the rarest research on the thermal ratcheting behavior of polymers.

In the present work, a detailed characterization of high-density polyethylene under compressive creep, thermal ratcheting phenomenon, and the coupled effect of compressive creep and thermal ratcheting at different temperature and stress conditions, is presented. Even though the compressive creep problem of the polymeric bolted flange joint is the driving factor for this research, the output can be adapted to a wider range of polymer applications.

4.3 Materials and Methods

4.3.1 Experimental Setup

The universal gasket rig (UGR) is an innovative in-house built experimental test bench for performing mechanical and leak characterization of polymeric materials, shown in Figure 4.1a. The significance of UGR is highlighted through the capacity to perform intricate compressive creep and thermal ratcheting analysis of HDPE material. Conceptually, the UGR generates a simple distributed compressive load on the specimen with hydraulic pump and two platens. A central stud, screwed to the hydraulic tensioner head, transmits the required compressive stress to the sample. The conservation of load on the material is achieved through an accumulator connected with hydraulic system. The UGR can facilitate ring shaped samples with a maximum outer and minimum inner diameter of 100 and 50 mm in between the two platens. The polymer test pieces are limited to an allowable thickness of 10 mm. This simple and sophisticated machine supports the complex analysis of material properties through the ability to apply an integrated load of internal pressure, compression, and heat. Typically, the maximum operating condition of UGR unit is restricted to 5 MPa of internal pressure on a controlled high temperature environment of 450°C.

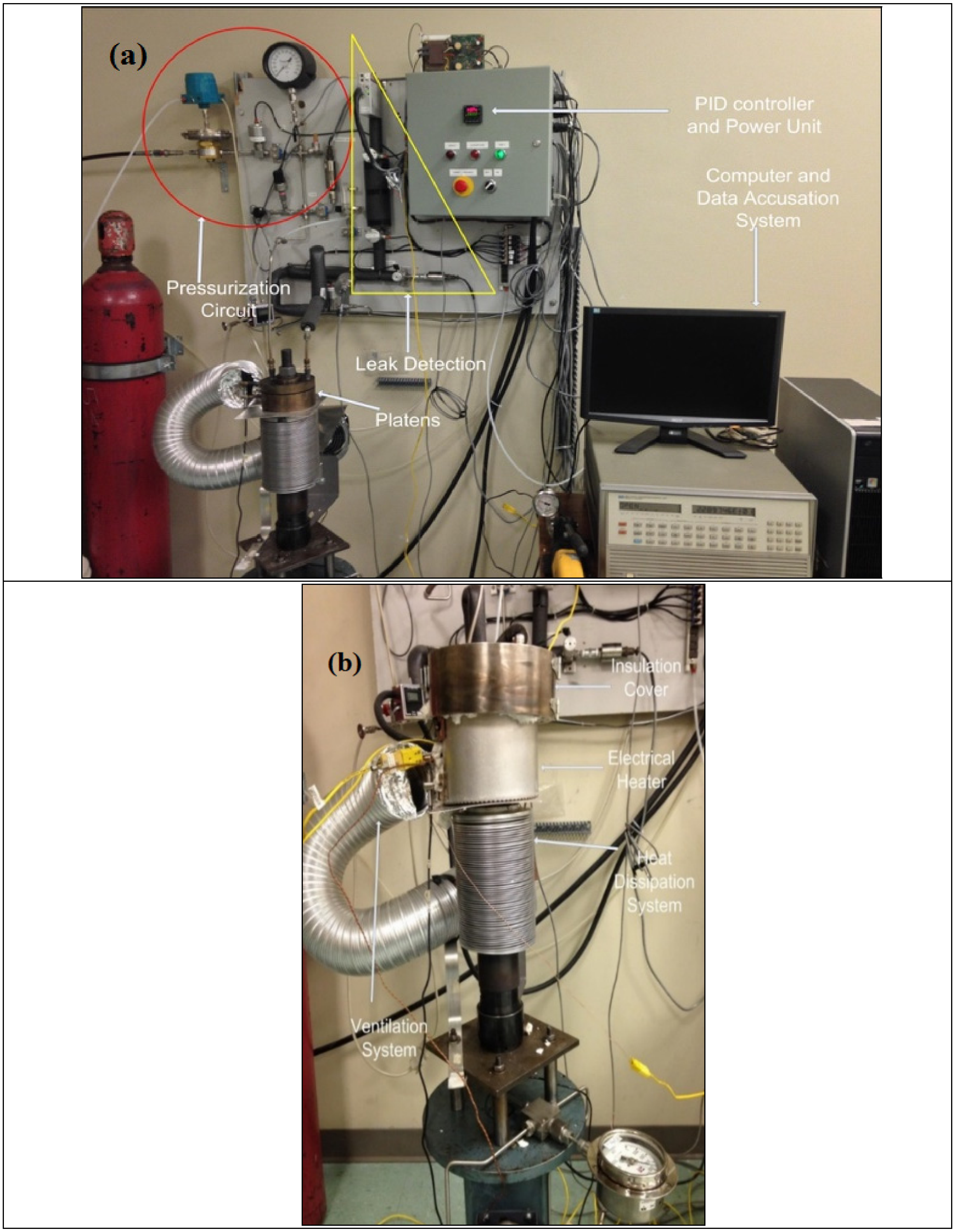

Figure 4. 1 Universal gasket rig (a) entire unit, (b) heating system

The real-time reduction in the thickness of the sample under compressive creep is measured using a high sensitive linearly variable differential transformer (LVDT). The samples are heated by means of an external ceramic band, which encloses around the platens to apply heat on the sample in form of conduction (illustrated in Figure 4.1b). A proportional integral derivative (PID) controller is used to control the temperature of the heater by monitoring the temperature of gasket through a thermocouple, which is connected to a computer by RS232 serial port. The rate of heating is set at 1.5 °C/min, while the cooling is accomplished through natural convection by shut-off of the heater once the desired temperature is attained. The rigidity is controlled using an appropriate number of Belleville washers.

A special insulation cover, comprising fiber materials and a stainless-steel cap, successfully accomplishes the prevention of heat loss to the surroundings. A Wheatstone bridge circuit strain gauge is affixed at the bottom of the central stud, to measure the strain induced on the test piece due to the application of load. The machine can apply a maximum stress of 70 MPa on a sample area of 645.16 mm^2. Specially designed inlet and outlet ports in the upper platens are handy in pressurizing the internal surface and measuring the leak rate under different test conditions. The exclusive feature of UGR is the thermal ratcheting analysis, which is accomplished by the combined use of PID controller and LabVIEW program. The system requires a complete definition of ratcheting temperature range, number of temperature cycles, and time period of hold-off between thermal cycles to achieve thermal ratcheting.

4.3.2 Test Procedure and Material Specifications

The mechanical characterization of ring shaped HDPE material is achieved through the sophistication of the UGR test bench. Typically, the physical measurement of polymer dimensions is the start point for the test procedure (Figure 4.2), followed by initialization of LabVIEW program to set up all the measuring sensors. The measured polymer ring dimensions are fed as inputs, which are used in the evaluation of applied compressive stress from the measured compressive strain by full bridge strain gauge. Initially, manual tightening of the nut is required to hold the ring specimen in position between two the contacting platen surfaces before any application of load through hydraulic pump. The zero position for LVDT sensor and sample gasket stress is defined at the instant of locking the platens manually. Subsequently, depending on the requisite of test to be performed, the chosen load level is exerted on to the polymer specimen, before or after the application of heat. In accordance with the industrial fluid process heating rate, the ceramic band electrical heater is set at a heating rate of 1.5 °C/min. Specially developed LabVIEW program provides for facile real-time monitoring of all the sensors of the test rig, while also enabling for options to modify the temperature and pressure conditions. The system has the capacity to monitor changes in the test conditions at a minimum interval of 10 s, with option to record values at a time interval between 10 to 600 s, as necessitated by behavior of the material over time. The creep and thermal ratcheting characterization of HDPE polymer is conducted in two phases. The first phase involves the short-term compressive creep analysis of the HDPE samples for 4 to 5 days under a variety of temperature and stress settings. The second phase is dedicated to the analysis of thermal ratcheting phenomenon, which is evaluated by preforming 10–20 thermal cycles between two target temperatures, with or without a day of creep. The information provided in Tables 4.1 and 4.2 elaborates, in detail, the types of tests performed. The ring sample of HDPE material respects 3 inch iron pipe size (IPS), standardized to 72 mm and 90 mm as inner and outer diameter, along with a material thickness of 6.35 mm.

Figure 4. 2 Specimen sample

Table 4. 1 Creep test parameters

High Density Polyethylene			
Test no.	**Temperature (°C)**	**Stress (MPa)**	**Test Time Period**
1	23	7, 14 & 21	5 days
2	50	7 & 14	5 days
3	60	7 & 14	5 days
4	70	7, 14 & 21	5 days

Table 4. 2 Thermal ratcheting test conditions

Test no.	Applied Stress (MPa)	Creep Temp (°C)	Ratcheting Temp (°C)	Days of Creep + No. of Thermal Cycles
High Density Polyethylene				
T1	7	23	28–55	1 + 20
T2	14	--	28–55	0 + 20
T3	14	23	28–55	1 + 20
T4	14	23	28–55	4 + 20
T5	14	--	28–60	0 + 20
T6	14	--	28–40	0 + 10

4.4 Results and discussion

4.4.1 Creep Strain

The vulnerability of polymer materials to creep and fatigue phenomenon are widely known, and high-density polyethylene is no exception. Unlike metallic materials, the variation in creep strain of polymers under tensile and compressive load is distinct. Furthermore, the HDPE material is suspected to be prone to thermal ratcheting damage, due to inherent low melting temperature. Therefore, a quantitative assessment of thermal ratcheting behavior, coupled with compressive creep of HDPE material, is essential. The experimental creep test results highlight the importance of both applied compressive load and temperature on the creep strain of HDPE. Influence of applied load on creep strain is shown in Figure 4.3, where the magnitude of induced creep strain increases with increase in the value of applied compressive stress. Importance of applied load on the transition time from primary to secondary creep stage is evident, as the specimen demonstrates different time-periods to reach the secondary phase. Yet, all HDPE samples demonstrate secondary creep, mostly within the first few hours of test. At ambient temperature, the creep strain of HDPE at 14 MPa of stress grew by six times the value of creep strain at 7 MPa of compressive stress. Whereas, on comparing the creep strain of HDPE at 14 and 21 MPa of compressive stress, the sample demonstrates a growth of 4.7 times the creep strain value at the lower load. The jump in magnitude of creep strain between 7 and 21 MPa of load is quite significant, which is nearly 28 times the former. The magnitude of the primary creep strain tends to increase with increase in applied load, causing subtle variations in the transition from primary to secondary creep for all the tested HDPE polymer samples. Alike to applied load, on varying the sample temperature under the same load (Figure 4.4), the creep strain tends to rise with intensification of temperature. Roughly, there is 20 percent increase in the creep strain with 10 °C escalation of temperature for HDPE material under the tested load. Even though the material's response to creep under the two types of loading is similar, their magnitude of deformation is straight out different. The magnitude of primary creep phase amplifies with increase in the sample temperature under the same compressive creep load. Analogous to compressive stress, the magnitude of primary creep strain is proportional to applied sample temperature. A compressive creep test at a sample temperature of 70 °C was performed to understand if there is any drastic change in the material response, as the particular temperature is above the standard maximum operating temperature. The consequent reaction of HDPE sample is consistent with rest of the test results.

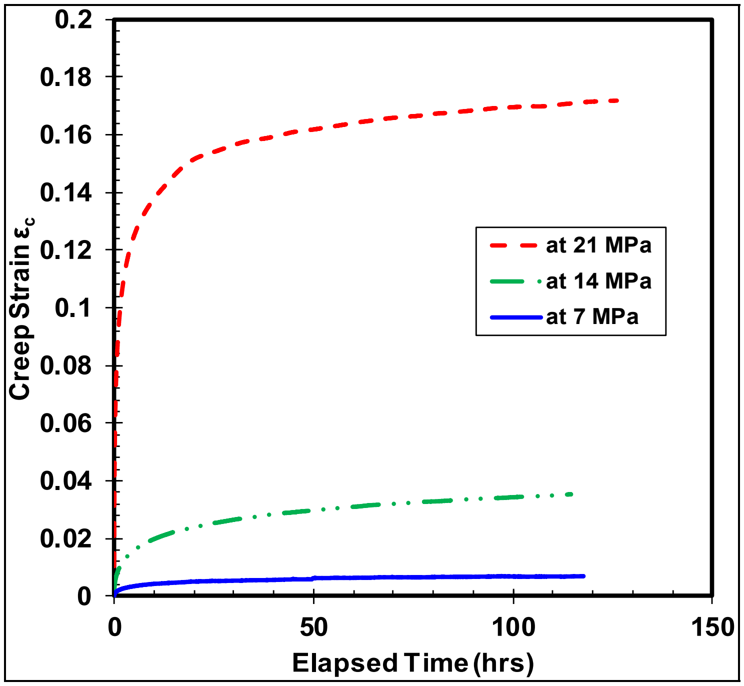

Figure 4. 3 Creep strain under different loads at ambient temperature

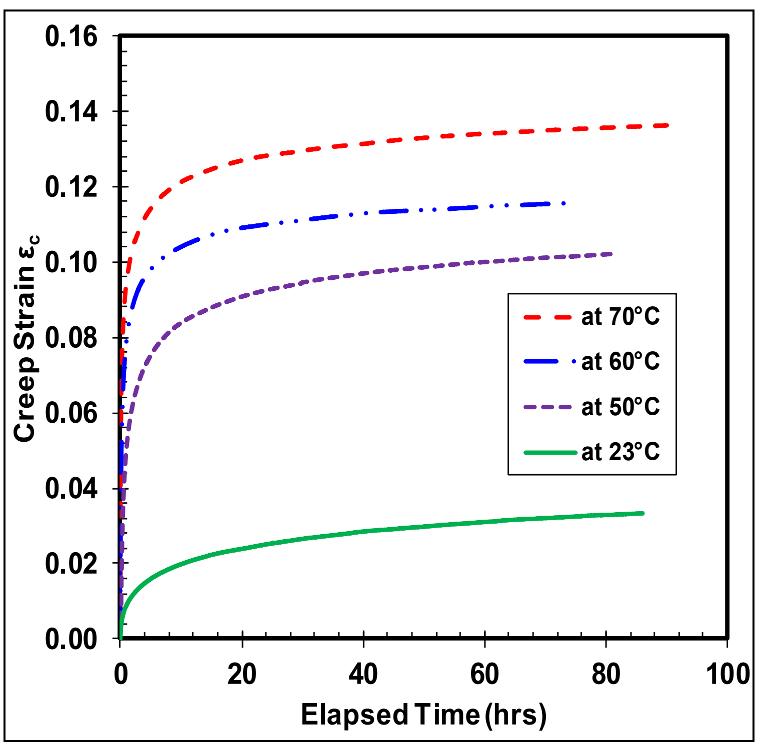

Figure 4. 4 Creep strain under different temperatures at 14 MPa

4.4.2 Creep Modulus

The Creep modulus is another important characteristic property of polymer material. The creep modulus is defined as the instantaneous elastic modulus of the material that varies with time. The ratio of creep stress over creep strain computes the creep modulus of the material. Since the creep stress is maintained constant, the creep modulus is inversely proportional to the creep strain. The creep modulus is dependent of the applied stress, where an increase of compressive load amplifies the loss in creep modulus of the material. The drop-in value of HDPE creep modulus over time under different compressive stress levels at room temperature is illustrated in Figure 4.5. The trend of creep modulus curve is similar to the creep strain curve, but in the inverse direction, where the material loses a substantial amount of creep modulus initially followed by a gradual saturation over time. The descent of creep modulus is maximum with the highest applied stress, while the magnitude of loss in creep modulus decreases as the value of applied compressive stress lowers. The saturation of creep modulus happens swiftly for the sample compressed under 21 MPa of stress, whereas the material requires a minimum of 10 and 30 h for transition to near saturation state under the compressive stress of 14 and 7 MPa, respectively. The change in creep modulus between 7 and 14 MPa of stress is higher than the difference in creep modulus between 14 and 21 MPa of stress, even though the increase in magnitude of compressive stress remains constant at 7 MPa. The probable reason cited for this behavior is the greater extent of irreversible damage induced in the specimen tested at higher compressive load, leading to earlier saturation. The influence of applied temperature on the creep modulus of HDPE is presented in Figure 4.6, which exhibits the loss of creep modulus with different chosen temperature under the applied compressive stress of 14 MPa. Consistent with the applied compressive stress condition, the trend in loss of creep modulus under different temperatures is similar where the highest reduction in creep modulus happens at the highest applied temperature under the same compressive stress. The magnitude of reduction in creep modulus between the room and high temperature samples is enormous. Besides, the saturation of creep modulus drop is evident with the highest temperature test, while the ambient temperature did not yield over the tested time period. The compressive creep test at 70 °C shows that the creep modulus drops by 8 times the initial value after saturation. By comparing the value of creep modulus at different temperatures, the consequence of temperature is apparent. At 70 °C, the material loses nearly 75% of the creep modulus value at 23 °C of temperature.

$$\text{Creep Modulus} = \sigma_c/\varepsilon_c = \frac{\text{Constant applied stress}}{\text{Creep strain at the instant}}, \tag{4.1}$$

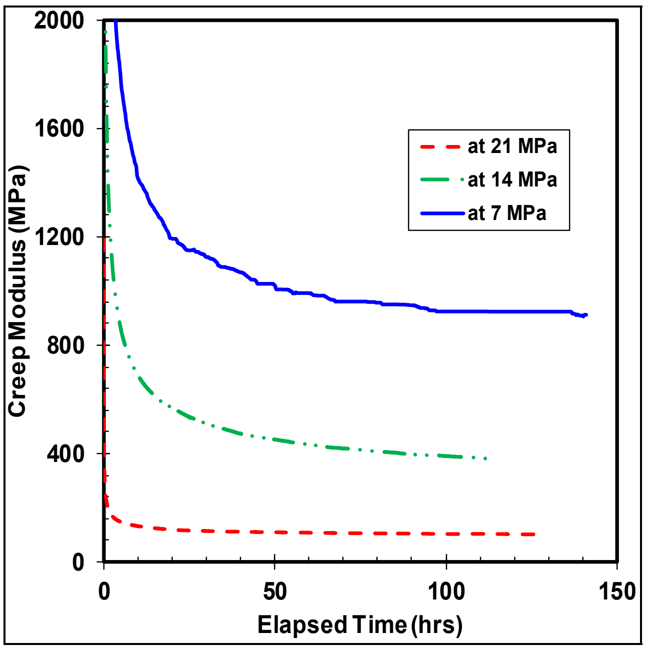

Figure 4. 5 Creep modulus under different loads at ambient temperature

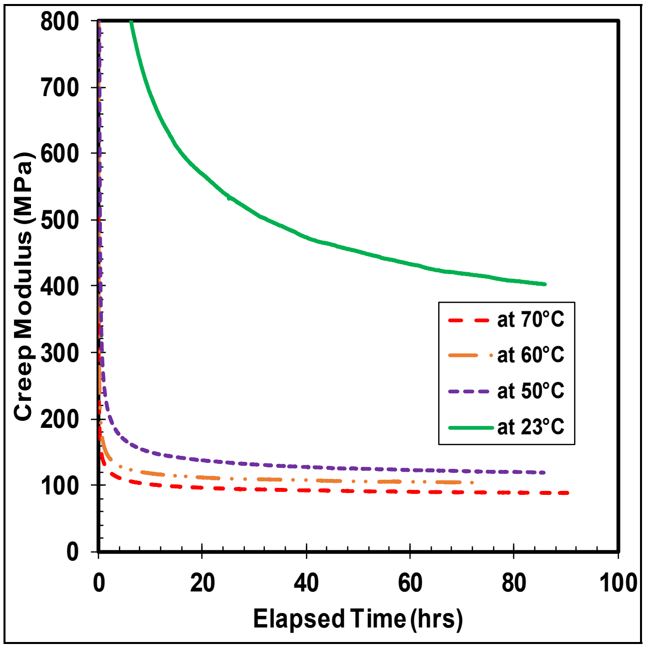

Figure 4. 6 Creep modulus under different temperatures at 14 MPa

4.4.3 Thermal Ratcheting

As mentioned earlier, the thermal ratcheting behavior is of utmost importance for polymeric materials, primarily due to their inherent property of low melting temperature. It is fair to say that a large selection of polymer materials can operate only in a moderate temperature conditions, hence, a small oscillation in temperature can cause a noteworthy change in the physical dimensions of the structure. Thermal cycling or thermal ratcheting induces a permanent cumulative deformation in the structure as a result of cycling of temperature under load. Similar to mechanical ratcheting or fatigue, thermal ratcheting induces cumulative deformation on the material, where the scientific publications on this phenomenon is near to zilch. A proper understanding of thermal ratcheting phenomenon is important for HDPE bolted flange joint application, as a small loss in flange thickness would lead to a significant reduction in the bolt load, thereby causing a leakage failure. The test bench, universal gasket rig, facilitates the thermal ratcheting analysis of high-density polyethylene material.

Quantitatively, five different combinations of thermal ratcheting tests were performed to distinguish the consequence of creep time, applied load, and temperature on the thermal ratcheting strain of high-density polyethylene. Under all thermal ratcheting tests, the cyclic escalation and reduction of thickness is apparent, with a net decrease in thickness. The wavy nature of the graphs (Figures 4.7–4.9) illustrates the change in thickness under each thermal cycle. The rise and fall of thickness corresponds to the increase and decrease of temperature during the thermal cycling, which is clearly visualized with thermal cycles and thickness change plots in Figure 4.7. The material accumulates damage with each thermal cycle, causing an overall reduction in thickness. The significance of time period of creep and applied load on the thermal ratcheting strain is presented in Figures 4.8 and 4.9, respectively. From the assessment of Figure 4.8, a substantial swift in thickness of HDPE samples tested at the same thermal ratcheting temperature, but with different initial period of creep, is noticeable. The amplification of the amount of deformation or reduction in thickness is partially cited to the initial creep of the material, while the rest is the coupled interaction of creep and thermal ratcheting, as the material simultaneously creeps and cumulates deformation, due to thermal ratcheting. On physical evaluation of the two-test specimen mentioned in Figure 4.8, the difference in thickness reduction is evident after removal of the applied load. Both samples demonstrated radial flow with increase and decrease of external and internal diameter, respectively. With the addition of one day of creep, an increase of 2% in the overall reduction of thickness is observed under similar ratcheting temperature. The impact of magnitude of applied compressive load on the thermal ratcheting deformation is demonstrated in Figure 4.9. HDPE samples tested under the same ratcheting temperatures, but with different applied stresses of 7 and 14 MPa, were evaluated to understand the importance of applied load condition. The specimen tested at 14 MPa of stress lost in excess of 7-fold of the thickness of the sample tested at 7 MPa after 20 thermal cycles. Since the two tests were performed without creep, the reduction in thickness is the coupled interaction of instantaneous compressive creep due to load and thermal ratcheting only. The cumulative deformation sustained during the initial cycles for HDPE material under 14 MPa is higher in comparison with the sample under 7 MPa of stress. This distinctive behavior is consistent with thermal cycles after 1 day of creep under 14 MPa, where the magnitude of initial damage, especially in the first four cycles, is great. A probable reason cited for this behavior is a change in the co-efficient of thermal expansion.

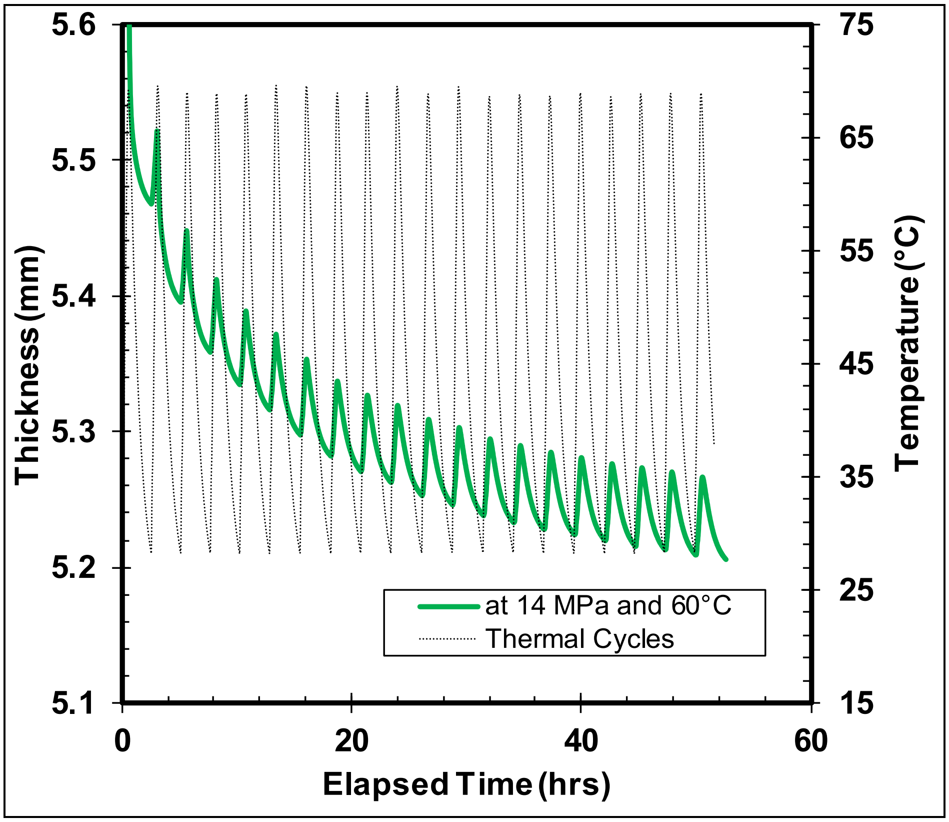

Figure 4. 7 Thickness variation of high density polyethylene (HDPE) under 14 MPa of stress and a thermal ratcheting temperature range of 28 to 60°C

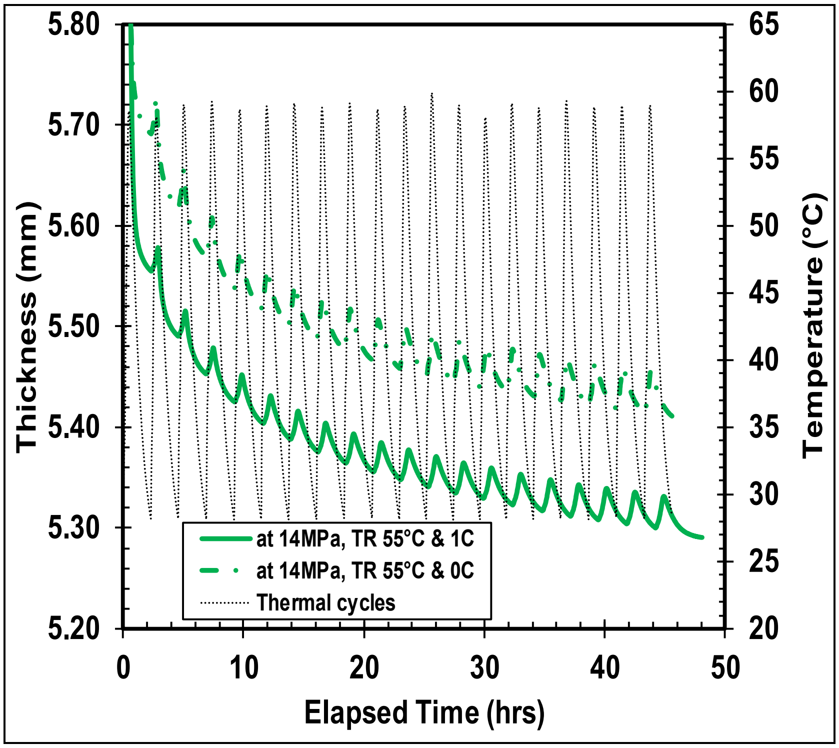

Figure 4. 8 Ratcheting of HDPE with and without 1 day creep at 14 MPa of stress

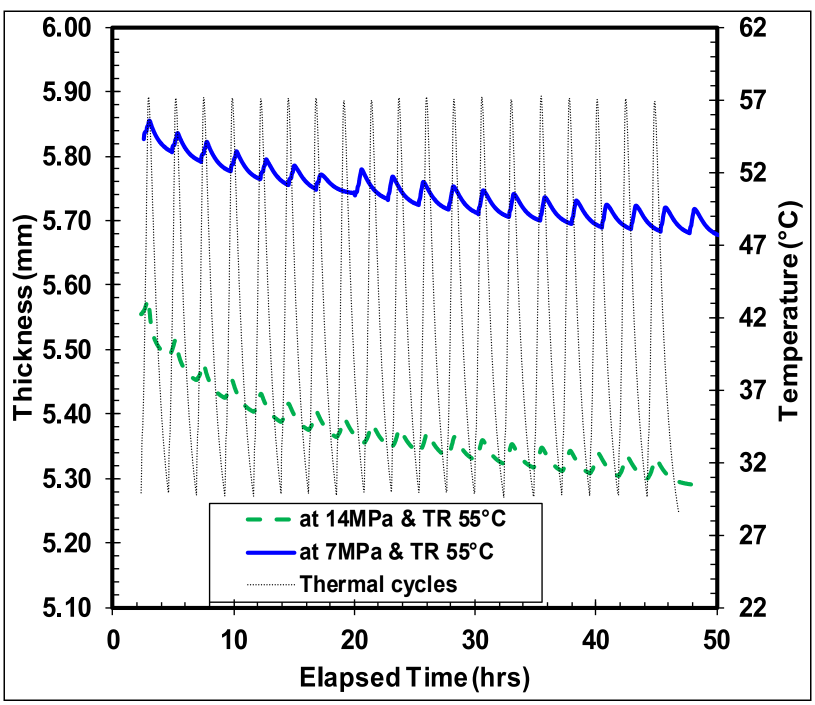

Figure 4. 9 Ratcheting of HDPE under 7 and 14 MPa of stress

4.4.3.1 Thermal Ratcheting Strain

The thermal ratcheting strain (TRS) is the cumulative strain induced in the material as a consequence of thermal ratcheting phenomenon. The thermal ratcheting strain increases with increase in the number of cycles, which is observed in Figures 4.10–4.12. The prominence of creep time period on thermal ratcheting strain is seen in the comparison between HDPE samples under the same cycling conditions, but at different initial creep time period. From the thermal ratcheting strain graph (Figure 4.10), it is noticeable that the TRS is higher for the sample tested without creep than the sample tested with one and four days of creep. This phenomenon indicates the effect of creep on the thermal ratcheting strain. Therefore, the creep time period is inversely proportional to the thermal ratcheting strain. Furthermore, the difference in thermal ratcheting strain between 1 day of creep, and without creep, is quite small, while maintaining a similar trend of thermal ratcheting strain. The thermal ratcheting strain increased by 10 percent for the test performed without 1 day of creep, in comparison to the test conducted with one day of creep. Moreover, the difference in TRS between 4 days of creep and zero days of creep is significant, where the TRS reduced by 36%, indicating that the effect of thermal ratcheting is severe without initial creep. The hardening of the material under creep is cited as the reason for increased resistance to thermal ratcheting, and consequently, reducing the magnitude of TRS with increase of creep time period. The vulnerability of HPDE to thermal cycling is undoubtedly exposed, prompting for further investigation. The change in TRS as an outcome of difference in applied compressive load is revealed in Figure 4.11. The variation in TRS between 7 and 14 MPa of stress is substantial, where the latter is higher than the former by approximately 1.9 times. It is noteworthy to mention that the TRS curve is much smoother at lower stress than at higher stress, where after the 7th thermal cycle, a change in slope of the curve is evident. Both the thermal ratcheting tests at the same ratcheting temperature range and different compressive stress, are executed after one day of creep.

$$\text{Thermal Ratcheting Strain } \varepsilon_{TRSx} = (LTR1 - LTRX)/Lo, \tag{4.2}$$

where, LTR1 refers to the thickness of the sample after the first thermal cycle, while LTRX represents the thickness of the sample with each thermal cycle, and Lo is the initial thickness of the specimen.

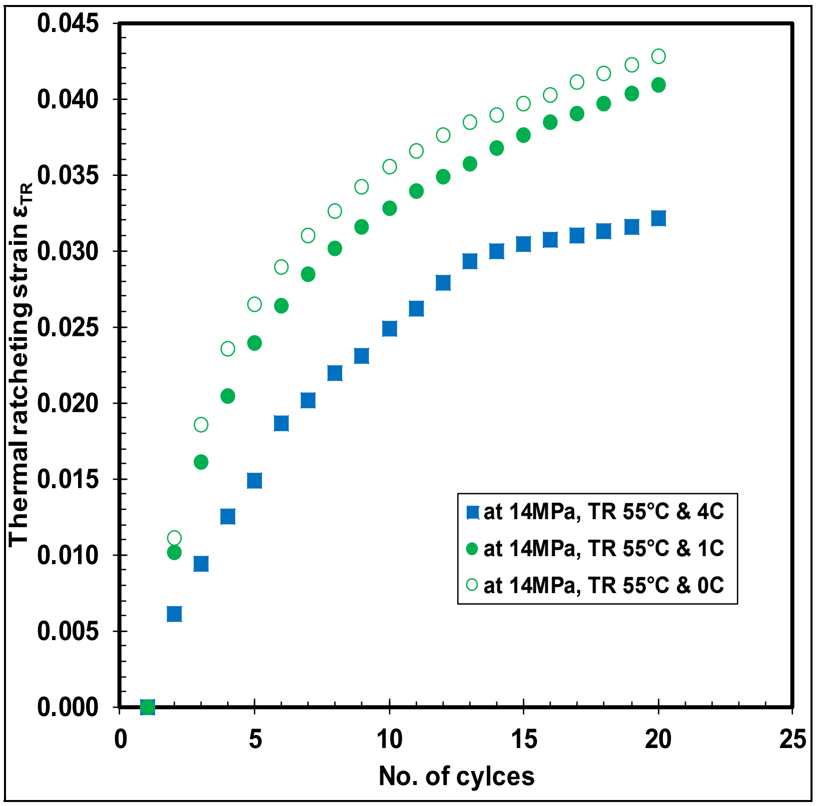

Figure 4. 10 Thermal ratcheting strain under different time periods of initial creep at 14 MPa

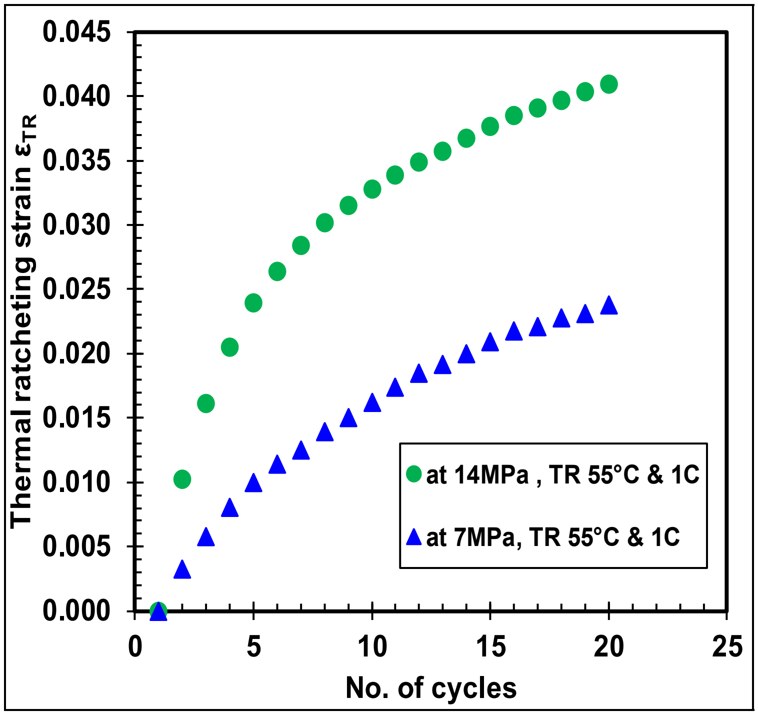

Figure 4. 11 Thermal ratcheting strain at different applied loads

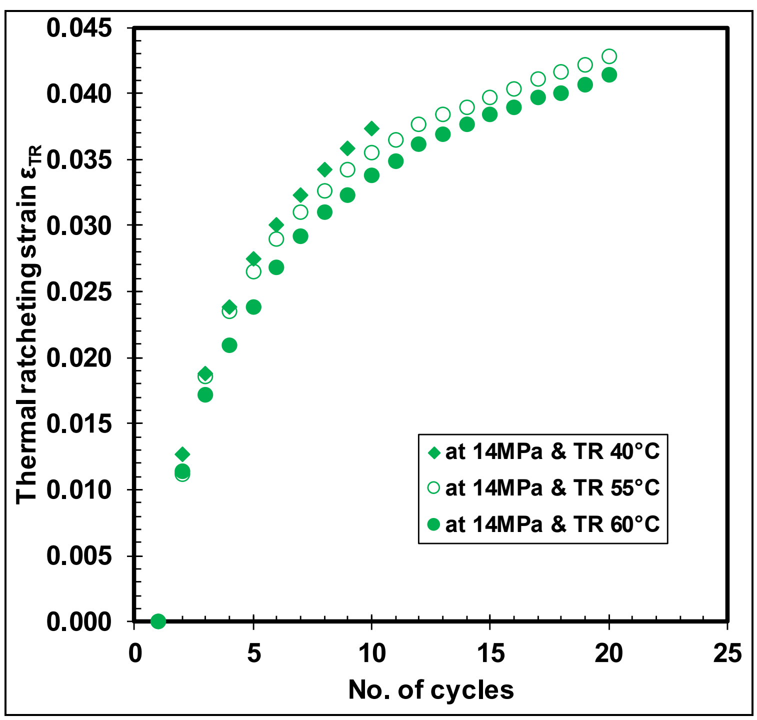

Figure 4. 12 Thermal ratcheting strain under different ratcheting temperature ranges

Finally, the importance of variation in ratcheting temperature range while maintaining a constant load is demonstrated in Figure 4.12. The trend of the three thermal ratcheting strains is similar, with a slender variation in the magnitude. The tests were performed at three different ratcheting temperature ranges (28–40 °C, 28–55 °C, and 28–60 °C). Notably, the thermal ratcheting strain of the HDPE tends to decrease with an increase in ratcheting temperature range. During the initial few cycles, the difference in TRS for all the three ratcheting temperature ranges is minimal, but a clear deviation occurs after the 7th cycle of thermal ratcheting. Nevertheless, the thermal ratcheting strain, after 10 thermal cycles, was reduced only as little as 5% between 28–40 °C and 28–55 °C TR tests, while the difference is 4.7% between 28–55 °C and 28–60 °C tests. Unlike gasket materials (Kanthabhabha Jeya and Bouzid, 2017), HDPE material did not saturate under 20 thermal cycles in the tested conditions.

4.5 **Conclusion**

The compressive creep and thermal ratcheting characterization of high density polyethylene (HDPE) material is successfully studied through universal gasket rig. The highlights of mechanical characterization tests are summarized:

- The HPDE material exhibits substantial creep damage under different compressive and thermal load conditions. The specimen shows a growth of 28 times the creep strain at 21 MPa from 7 MPa of compressive stress, while the increase in creep strain from the lowest tested temperature to highest tested temperature is 7-fold.
- The creep strain is directly proportional to the applied load and applied temperature, exposing the vulnerability of the material.
- Creep modulus is dependent on the applied stress. The magnitude of creep modulus decreases with increase in applied compressive load and material temperature. The maximum loss of creep modulus occurred at the highest applied stress and temperature, respectively.
- The impact of thermal ratcheting is evident, where the extent of cumulative deformation is dominated by the compressive load, followed by material

temperature. The thermal ratcheting is very similar to the mechanical ratcheting or fatigue, causing an accumulation of deformation with each thermal cycle.

- In addition, all HPDE specimens demonstrate thinning of structural thickness under thermal ratcheting, and none of the specimens show any sign of saturation of deformation under the 20 tested thermal cycles.
- The thermal ratcheting strain of HDPE material is influenced by applied load, temperature, time-period of creep, and number of thermal cycles. TRS is higher for the tests without creep, suggesting the deformation due to thermal ratcheting is critical during the initial period of the operation.

Finally, the coupled behavior of compressive creep and thermal ratcheting phenomenon of HDPE material raises the question on considering a common design criterion for polymeric materials, especially in bolted flange joint application. The results clearly indicate the necessity for upgraded design standards with inclusion of thermal ratcheting effect.

CHAPTER 5

EFFECT OF THERMAL RATCHETING ON THE MECHANICAL PROPERTIES OF TEFLON AND FIBER BASED GASKET MATERIALS

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This article is accepted for publication in the Journal of Applied Polymer Science on 3rd October 2018

5.1 Abstract

This paper discusses the effect of thermal ratcheting on material properties of expand polytetrafluoroethylene (ePTFE), virgin polytetrafluoroethylene (vPTFE) and compressed non-asbestos fiber (CNA) gasket materials. ePTFE and vPTFE materials demonstrate a 7.7% and 28% increase in creep strain after thermal ratcheting in comparison to creep strain at constant temperature for the same time. In addition, the thermal ratcheting produces a substantial reduction of creep modulus of ePTFE and vPTFE. The CNA material does not exhibit significant change in creep strain or in creep modulus with thermal ratcheting. However, CNA along with ePTFE and vPTFE show a momentous raise in the creep strain value if the material temperature is lessened. On declining the gasket temperature from 212°F to 100°F at the end of 20th thermal cycle, the materials – ePTFE, vPTFE and CNA exhibit 27, 48 and 15% increase in creep strain value, respectively. The percentage of thickness reduction (%TR) raises with increase of ratcheting temperature and with increase of creep time, except for CNA where only a small variation is observed. Contrary to the coefficient of thermal expansion (CTE) of CNA, the CTE of both PTFE materials show significant vulnerability to ratcheting temperature and initial creep time.

5.2 Introduction

Polytetrafluoroethylene (PTFE) and compressed non-asbestos fiber based materials are among the most sought-after material for gasket products. Their inherent properties such as excellent leak tightness, effective resistance to chemical attack and electrical surge, extensive operating pressure and temperature range make them an ideal replacement for conventional gasket materials. Further to these sealing performances, these materials are appropriate for aggressive fluid and corrosive environment applications. Despite of its widespread application, PTFE material has a few major drawbacks namely its low creep resistance and high coefficient of thermal expansion/contraction, which dictates the leak performance (Bazergui and Payne, 1984).

Even though, the research on PTFE material is more than half century old, the amount of information available on the compressive creep and thermal ratcheting properties are limited. Apart from the load deflection data, only a little information on the effect of temperature over the mechanical properties of PTFE under compressive load is provided in the research publication (Keywood, 1994). PTFE gasket creep response to compressive load and the failure by extrusion when utilized in class 150 and 300 pipe flanges under specific working conditions is reported in journal papers (Keywood, 1994; Winter and Keywood, 1996). The consequence of their low creep resistance led to the development of a standard test procedure on relaxation and blowout characteristics of PTFE based gaskets (Derenne et al, 1999). The current ASTM draft standard test procedure for hot blowout (HOBT) of PTFE gasketing products is based on a Nominal Pipe Size (NPS) 3 class 150-flange joint fixture with relaxation capabilities (ASTM EN 13555, 2013).

Researchers developed new test methods to characterize non-asbestos gasket materials at specific constant elevated temperatures (Payne and Bazergui, 1990 and Payne et al., 1987). However, typically a large constituent of gaskets encounter sudden surge or cycling temperature over their productive lifespan. The need of the hour is to evaluate the impact of the cumulative damage incurred to gasket products subjected to thermal ratcheting. Thermal ratcheting phenomenon can be defined as the accumulation of damage, which is sustained due to cyclic variation of temperature of the material under stress. This damage mechanism is of particular interest for bolted gasketed joints as these pressure vessel components are already prone to the leakage failure under the loss of bolt load due to effects such as creep, thermal expansion/contraction, aging and degradation. Thermal ratcheting generates a further loss of compressive load on the gasket projecting for radial extrusion under the operational internal pressure, instigating a failure by blow out. Only few reported scientific publications investigated thermal ratcheting phenomenon of PTFE based materials but none have provided a comprehensive analysis or a quantification of this effect (Marchand et al., 1992; Bouzid et al., 2000; Bouzid et al., 2001; Bouzid, 2011; Bozuid and Benabdullah, 2015). Unlike metallic materials, most gasket materials are susceptible to temperature with relatively lower operating temperatures. Unfortunately, the available papers on material ratcheting, specifically the ones on PTFE material, are based on mechanical fatigue or load cycling and not on thermal cycling (Chen and Hui, 2005; Tao and Xia, 2007; Zhang and Chen,2009).

Some authors have briefly investigated the influence of thermal ratcheting and applied load on the thermal expansion of PTFE nonetheless these researches were limited to few tests with few thermal cycles and with a maximum ratcheting temperature of 400°F (Bouzid et al., 2000 and Bouzid and Benabdullah, 2015). Coefficient of thermal expansion is an important characteristic for modern flange design codes including the finite element thermal analysis (EN 1591-1, 2013). The prevailing test standards developed by the American Society of Testing and Materials do not consider the effect of load or thermal ratcheting on the coefficient of thermal expansion (ASTM E 228-11, 2016; ASTM E 831-14, 2014; ASTM D 696-16, 2016). The work by Bhattachar (1997) on instantaneous coefficient of linear thermal expansion is independent of the reference temperature unlike in the ASTM E228 (2016) and E831 (2014). However, the effect of thermal ratcheting on the coefficient of thermal expansion is not addressed. Independent researchers have reported quantitative results on coefficient of thermal expansion for PTFE and other polymeric materials, none has scrutinized the behavior under compressive creep and thermal ratcheting conditions (Kirby, 1956; Touloukian et al., 1977). Furthermore, the research on the integrated effect of magnitude of applied, creep and thermal ratcheting of gasket and high-density polyethylene materials is limited to respectively (Kanthabhabha Jeya and Bouzid, 2017; Kanthabhabha Jeya and Bouzid, 2018).

This paper is a continuation of the work presented in and it focuses on comprehending the impact of thermal ratcheting on the creep behavior of ePTFE, vPTFE and CNA materials under compression (Kanthabhabha Jeya and Bouzid, 2017). In addition, the research provides valuable data on the thickness reduction of gasketing materials and coefficient of thermal expansion under the influence thermal ratcheting through experimentation.

5.3 **Materials and Methods**

5.3.1 Experimentation:

The materials under characterisation are CNA (compressed non-asbestos Fiber Gasket), ePTFE (expanded PTFE) and vPTFE (virgin PTFE) gaskets. The tests are preformed using a home built multifunctional experimental test bench known as Universal Gasket Rig (UGR), shown in Figure 5.1. The UGR adapts simple technologies to perform complex mechanical, thermal and leak characterisation testing on ring or gasket shaped material specimens. The size limitations of the ring shaped specimen are 2 by 4 in. in inside and outside diameters, respectively. The maximum allowable thickness of the samples is 0.375 in between the two enclosing platens of the experimental setup. The greatest benefit of UGR is the ability to apply an integrated mechanical and thermal loading with internal pressure conditions. The capacity of the UGR is 500 psi of internal pressure at 35000 lbs. and 650°F on the specimen sample. The details on the working mechanisms of UGR are elaborated in the work of Kanthabhabha Jeya and Bouzid (2017 and 2018).

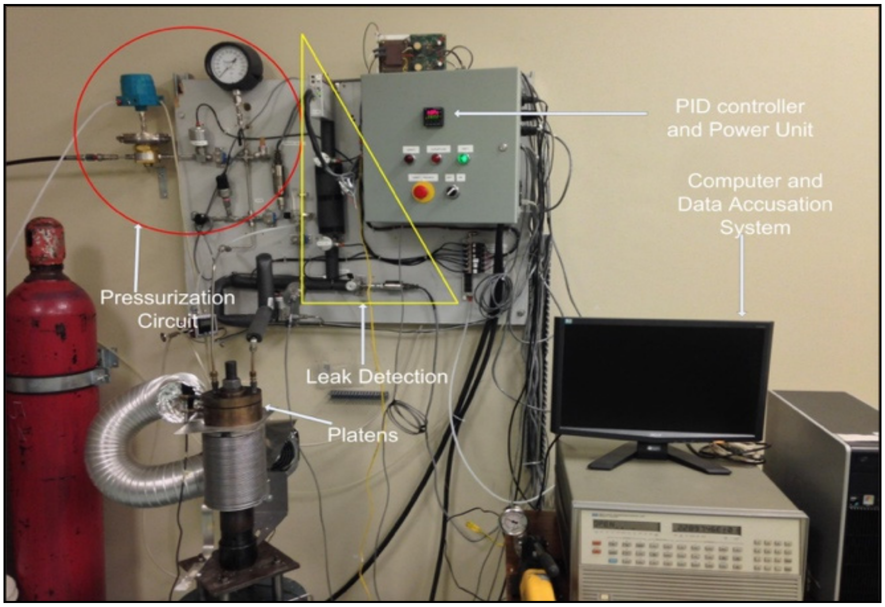

Figure 5. 1 Universal Gasket Rig.

5.3.2 Test Procedure and material dimensions:

The UGR is semi-automated system where manual operations are initial required. The dimensions of the gasket materials are measured using a digital Vernier caliper. These are fed as inputs to the LabVIEW program along with other required properties and test conditions. The gasket is placed between the two platens and secured in this position by manual tightening of the nut over the central stud. This position is defined as the zero reference for displacement measured by a linear velocity differential transformer (LVDT) sensor. The measured deformation is due to compressive load or thermal expansion or contraction of material thickness under different test conditions. This position also acts as the zero reference for the applied stress to the gasket. Subsequently, the required or desired compressive load is applied to the specimen through a hydraulic system. The application of heat is performed at the rate of 3°F/min as per a typical industrial fluid process-heating rate, using a ceramic band heater. Specially developed LabVIEW program is used to automate the application of heat during the thermal ratcheting or constant heating phase and to apply the internal pressure to the gasket sample. Through a data acquisition and control unit, the in-house LabVIEW program provides the real-time monitoring of all the instrumentation including temperature, pressure, gasket stress and displacement and time. Depending on the test requirements, these parameters are monitored and recorded at regular intervals between 10 to 600 seconds. The characterization of the tested gaskets is conducted in two phases. The first phase involves the study of creep and the influence of thermal ratcheting on the creep of all the three materials, while the second phase focuses on the effect of different test conditions on the thermal ratcheting behavior of the selected gaskets. The physical dimensions of the three test specimens are given in the Table 5.1.

Table 5. 1 Gasket dimensions

Material Type	**Outer Diameter (in.)**	**Inner Diameter (in.)**	**Thickness (in.)**
ePTFE	3	1.8	0.1
vPTFE	2.96	1.8	0.125
CNA fiber	2.80	1.94	0.120

5.4 **Results and discussions:**

The consequence of thermal ratcheting on various material properties of expanded PTFE, virgin PTFE and compressed non-asbestos fiber gasket materials are elaborated in the following sub-categorizes. While the significance of thermal ratcheting on the creep strain and the creep modulus are performed at similar temperatures for all three materials, the other effects of ratcheting are studied under different ratcheting temperatures as a result of individual material property and their vulnerabilities detailed (Kanthabhabha Jeya and Bouzid, 2017).

5.4.1 Creep strain

Unlike fiber gasket material, the Teflon based ePTFE and vPTFE gaskets are relatively vulnerable to creep and temperature effect, which affects its leakage tightness property. This susceptibility of PTFE based gasket led to the study on the coupled interaction between thermal ratcheting and creep of all three-gasket materials. The results highlight the effect of thermal ratcheting in amplifying the creep strain, in other words the thermal ratcheting accelerates thickness change of Teflon based gaskets. For both ePTFE and vPTFE, the magnitude of creep strain increases with each thermal cycle and it did not exhibit saturation after 20 thermal cycles. From the point of convenience and characterization, all three-gasket materials were tested for creep at 212°F and the ratcheting temperature cyclic range was maintained consistent between 100° and 212°F. The provided creep data are conducted at 4000 or 6000 psi of compressive stress depending on the material. The stress-temperature combination is typical of 150 class flange joint applications and therefore is representative of the thermal ratcheting study. Since all the three materials exhibits secondary creep within one day of creep, the ratcheting tests were performed after 1 day of creep at the same temperature. After the fourth cycle (Figure 5.2), the creep strain of ePTFE with ratcheting deviates from creep strain curve of ePTFE without thermal ratcheting. The difference between the two creep curves is because of cumulative damage induced with each thermal cycle. Hence, it clearly indicates that creep of ePTFE intensifies with thermal cycling at all stages of creep of the material. If the material is maintained at 212°F at the end of 20 thermal cycles, the new creep strain curve is higher than the creep strain under constant temperature at the same load by 7.7%. It is to be noted that the strain cycles are due to thermal expansion contraction. The lower bound points represent the expansion at the highest temperature and the upper bound points represent the contraction when cooling to the lowest temperature between each cycles. For the three gasket materials, the lower bound point in the creep strain curves is the point of comparison with pure creep test.

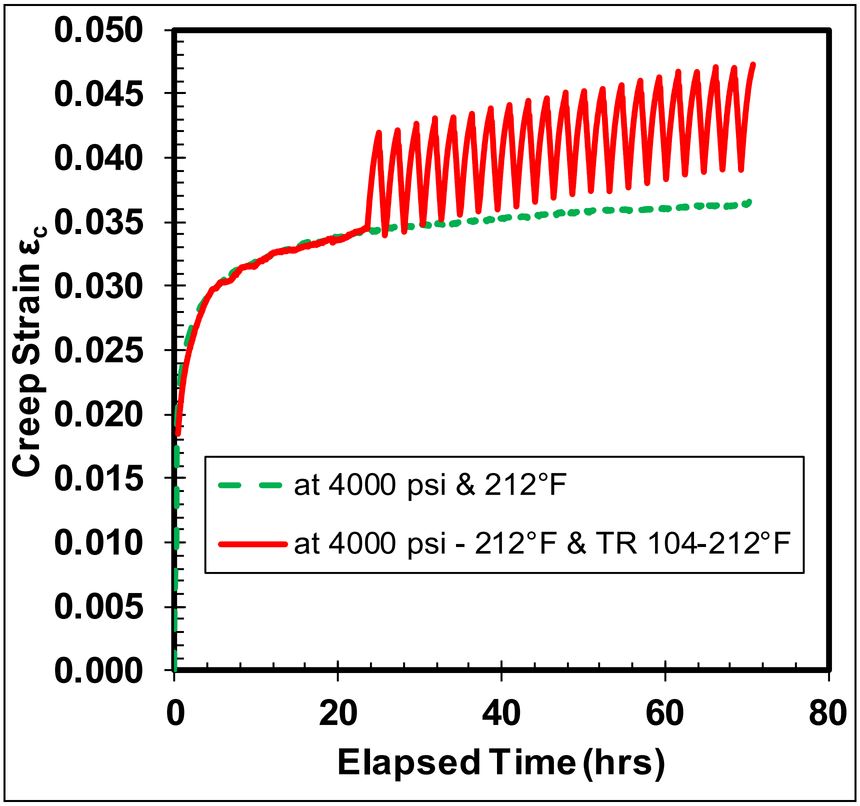

Figure 5. 2 ePTFE creep strain with and without thermal ratcheting

Out of the three tested gasket materials, virgin PTFE material is the most vulnerable in terms of creep and thermal ratcheting behavior (Kanthabhabha Jeya and Bouzid, 2017). Hence, a study on the interaction of both phenomenon would be of interest for the particularly gasket applicants. From a quick glance of Figure 5.3, it is seen that the thermal ratcheting significantly alters the creep strain of vPTFE. Even though the trend of creep strain curves under thermal ratcheting of ePTFE and vPTFE are very similar, the creep strain rate is much higher with vPTFE material. In addition, the effect of thermal ratcheting is rapid and starts as early as the first thermal cycle with a 6% increase in creep strain after the first cycle. vPTFE material records the highest growth in creep strain value under thermal ratcheting in comparison to the other two gasket materials. The creep strain is increased by 28% after 20 thermal cycles for vPTFE material. This is a major concern to be addressed in the long-term applicability of this material when exposed to thermal cycling.

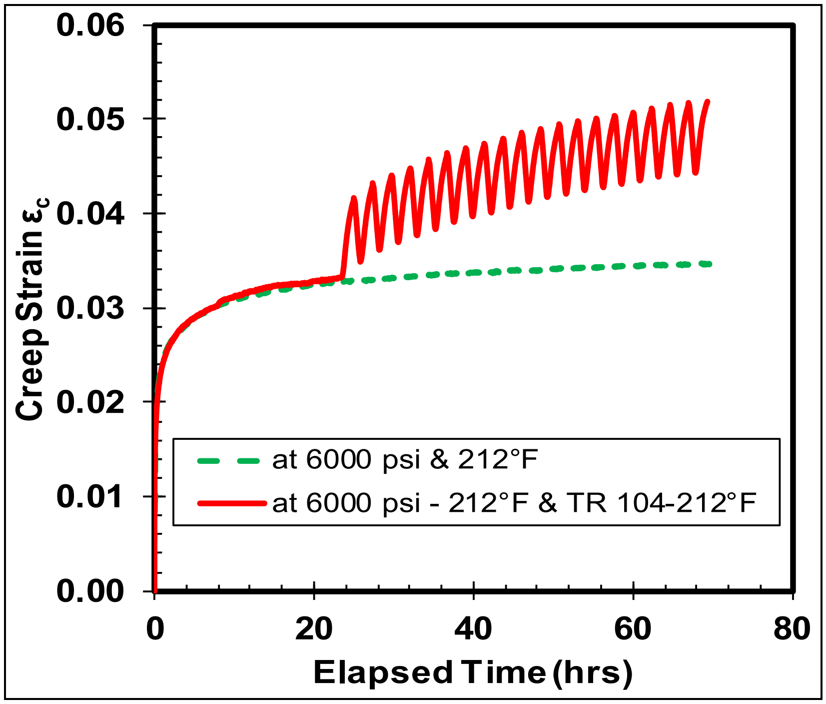

Figure 5. 3 vPTFE creep strain with and without thermal ratcheting

In contrast to Teflon based gaskets, the compressed non-asbestos fiber gasket material exhibits higher resistance to both creep and thermal ratcheting. On cycling between 100° and 212°F, the CNA (Figure 5.4) did not show significant increment in creep strain. At the end of 20 thermal cycles, the creep strain values remained in the same range as those of pure creep strain without thermal ratcheting. This indicates that the tested temperature is rather low for the study of thermal ratcheting phenomenon of CNA. Nevertheless, the CNA demonstrates little vulnerability to thermal ratcheting at this low temperature range. Another important point to mention is the amplification of strain with a decrease in temperature from a higher magnitude. This is primarily due to the reduction in thermal energy thereby causing contraction of the material thickness. In addition, this value similar to the creep strain at highest temperature point (lower bound), which increases with each thermal cycle. Hence if at the end 20 ratcheting cycles the material temperature is lowered to least value of ratcheting temperature range, then augmented creep strain value is higher from pure creep by 27%, 48% and 15% for ePTFE, vPTFE and CNA respectively. This behavior signifies the importance of temperature of the material on predicting the long-term creep properties of a gasket material.

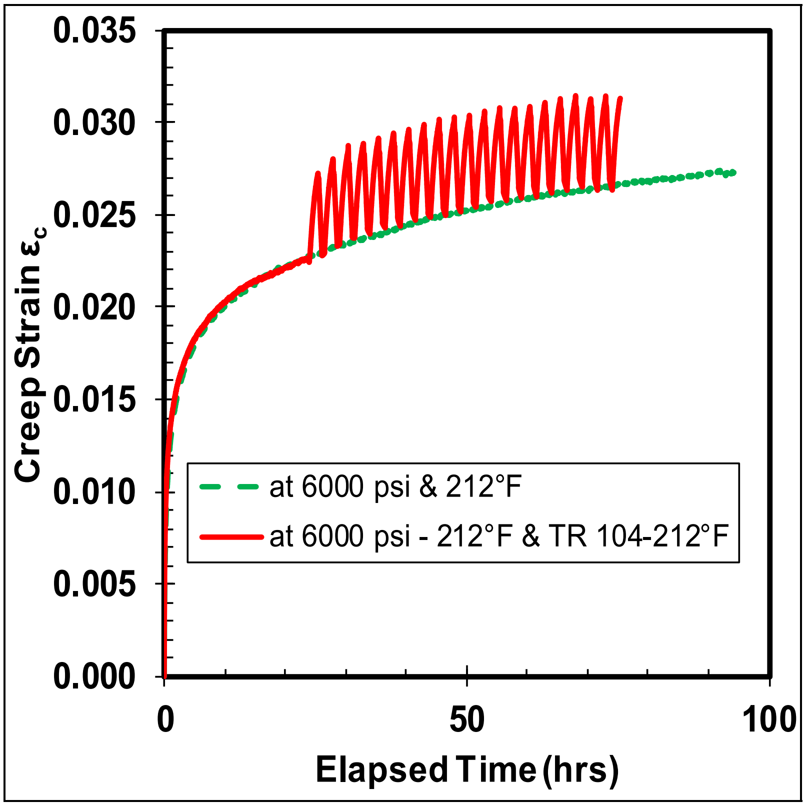

Figure 5. 4 CNA creep strain with and without thermal ratcheting

5.4.2 Creep modulus

Creep modulus is defined as the ratio of constant applied stress over time-dependent strain. Unlike creep strain, the creep modulus decreases with increase in time but evidently displays primary and secondary phases, relating to rapid decrease followed by gradual saturation over time. The consequence of thermal ratcheting on the creep modulus for the three gasket materials is shown in Figures 5.5, 5.6 & 5.7. Both the Teflon based materials demonstrate higher reduction in creep modulus with thermal ratcheting over fiber gasket material.

$$\textit{Creep Modulus} = \sigma_c/\varepsilon_c = \frac{\textit{Constant applied stress}}{\textit{Creep strain at the instant}}, \tag{5.1}$$

Where σ_c is the applied creep stress (MPa) and ε_c is the creep strain at that instant.

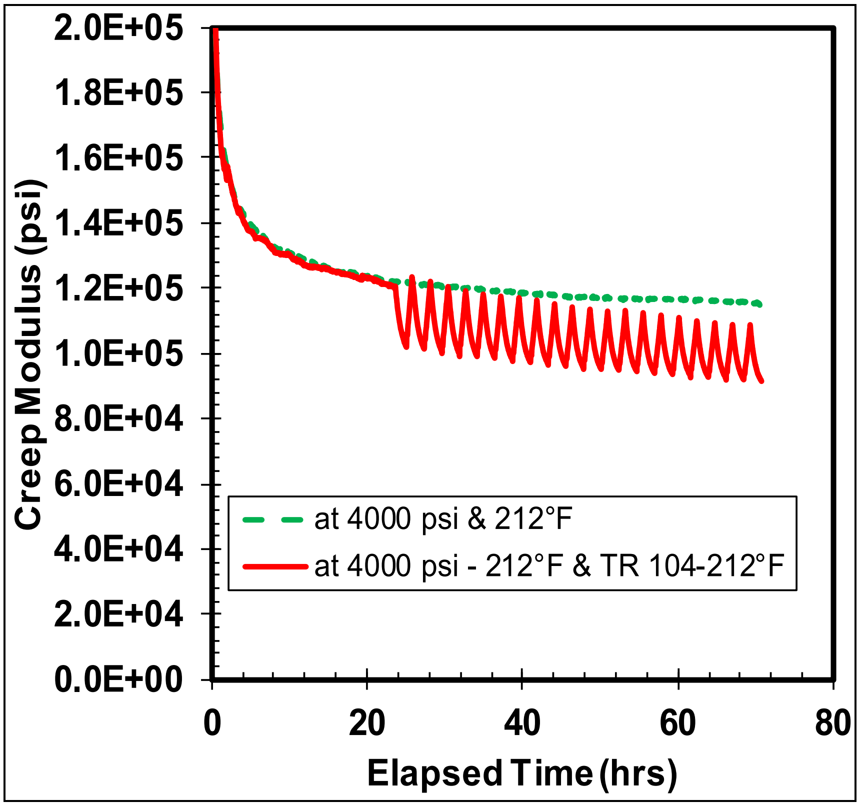

Figure 5. 5 ePTFE creep modulus with & without thermal ratcheting

On comparing creep modulus under constant temperature and under thermal cycling, the creep modulus tends to decrease with each thermal cycle. During the primary phase, ePTFE creep modulus reduced by almost 70% under the tested condition. The effect of thermal ratcheting becomes significant after the fourth cycle for ePTFE. The value of creep modulus (Figure 5.5) after 20th cycle is nearly 7% lower than the creep modulus at the same time under constant temperature. The amount of decrease in creep modulus between each thermal cycle tends to decrease which indicates that saturation may have occurred had the tested continued for few more thermal cycles. The susceptibility of vPTFE to thermal ratcheting is understandable from the study of creep modulus (Figure 5.6). The gradual decrease of creep modulus as early as from the first thermal cycle is apparent and this decrease of creep modulus after 20th cycle is roughly 21%. Similar to ePTFE creep modulus, vPTFE creep modulus decreases with each thermal cycle and this decrease is higher during the initial few thermal cycles. This suggests that the cumulative damage induced by thermal ratcheting creates a hardening effect on the material thereby decreasing the amount of damage with each cycle. In addition, the reduction of creep modulus of vPTFE during primary phase is 70%, which is similar to the ePTFE.

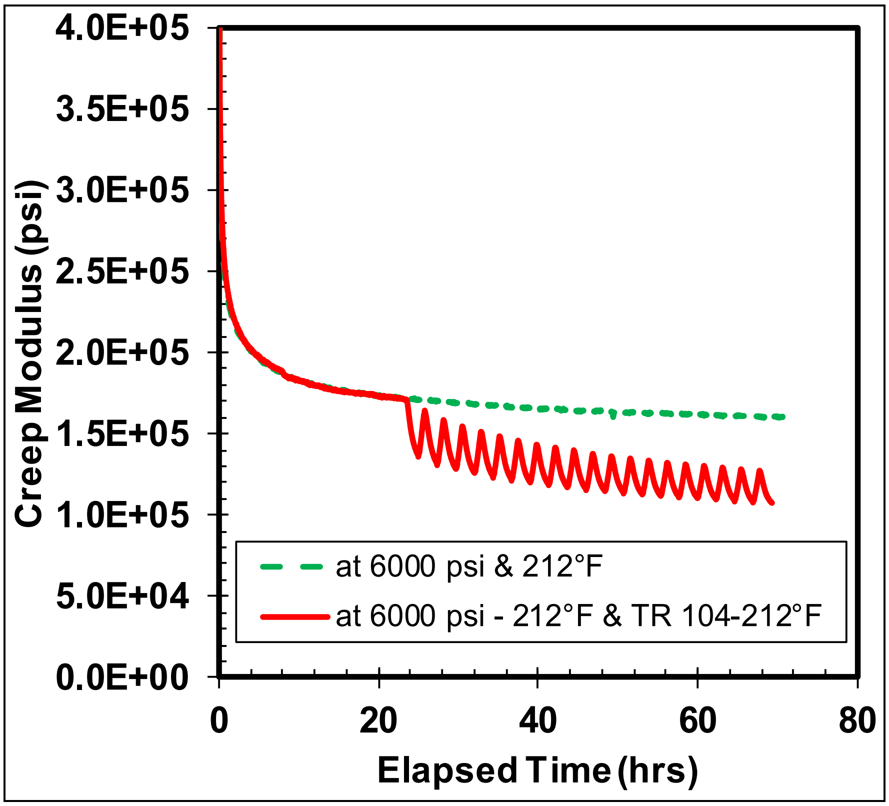

Figure 5. 6 vPTFE creep modulus with and without thermal ratcheting

The effect of thermal ratcheting on the creep modulus for CNA (Figure 5.7) is not significant at the tested ratcheting temperature. Even after 20 thermal cycles at 210F, the creep modulus superimposes with the creep modulus under constant temperature. Consistent with other gasket materials, the initial loss of creep modulus during the primary phase is extremely high. The impact of loss of thermal energy during cool down of each thermal cycle on the creep modulus is clearly visible for all three gaskets from the Figure 5.5, 5.6 & 5.7. Moreover, this contraction intensifies the loss of creep modulus with each cycle for ePTFE and vPTFE material.

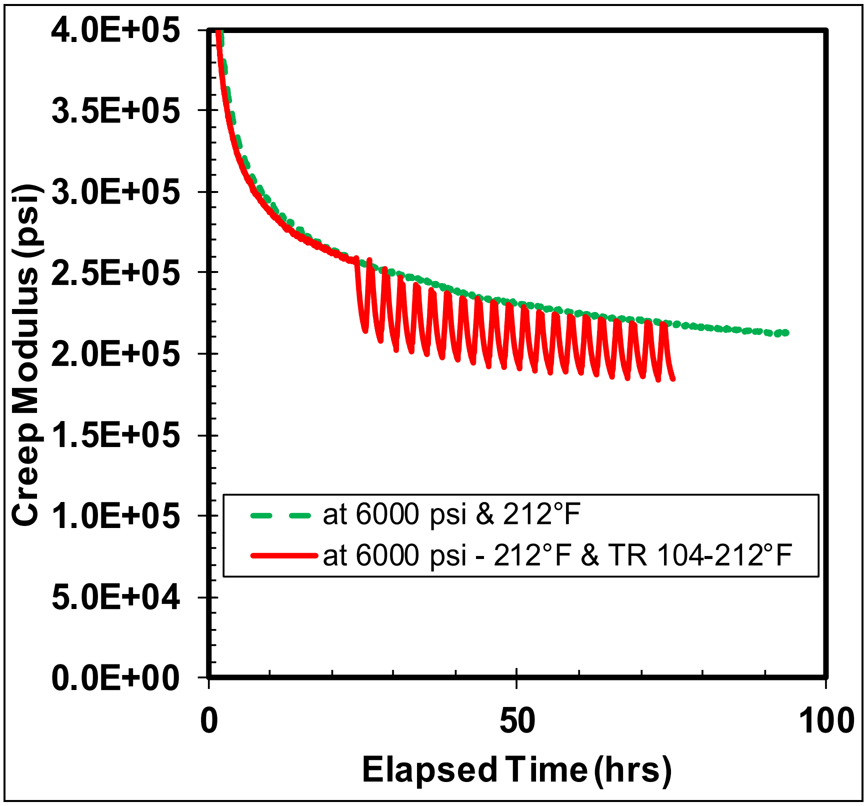

Figure 5. 7 CNA creep modulus with & without thermal ratcheting

5.4.3 Thermal ratcheting

Thermal ratcheting is defined as the cumulative damage induced in the material due to cycling of temperature. This phenomenon is similar to mechanical ratcheting or fatigue but instead of cycling of load at constant temperature, it is the consequence of temperature cycling at constant load. The impact of thermal ratcheting on the percentage of thickness reduction and coefficient of thermal expansion are presented in the following sub-section. The significance of the ratcheting temperature and initial creep on the mentioned properties of all three materials are discussed.

5.4.3.1 Percentage of thickness reduction

The evolution of loss of gasket thickness under thermal ratcheting and compressive load is presented in terms of percentage of thickness reduction (%TR). This valuable parameter is used to estimate the bolt load drop and in leakage assessment in bolted flange joints. Figures 5.8, 5.9 & 5.10, present the progressive loss of thickness of the tested gasket materials under various test conditions. As anticipated, the highest percentage of loss of thickness occurred with vPTFE material while the least of reduction under thermal ratcheting happened with fiber gaskets. The impact of ratcheting temperature range and effect of creep on the magnitude of thickness reduction is the focus of this study. As a general trend, the loss in thickness increases with increase of number of thermal cycles. Furthermore, it is observed that the magnitude of %TR increases with the increase in ratcheting temperature. The probable reason cited for this behavior is the vulnerability of the material to temperature. The ePTFE material loses nearly 73% of its initial thickness under the application of load while vPTFE only loses 24% of its initial thickness. Because of this initial loss, a saturation of damage occurs causing a reduction in the thickness lost under thermal ratcheting for ePTFE material. Unlike high-density polyethylene material, both Teflon based gaskets show an increased loss in thickness when tested under thermal ratcheting after one day of creep (Kanthabhabha Jeya and Bouzid, 2018). The results show that ePTFE, under the same applied load and ratcheting temperature conditions exhibits a 1% addition loss in thickness after one day of ambient temperature creep (Figure 5.8). This indicates that the impact of thermal ratcheting is amplified with initial creep.

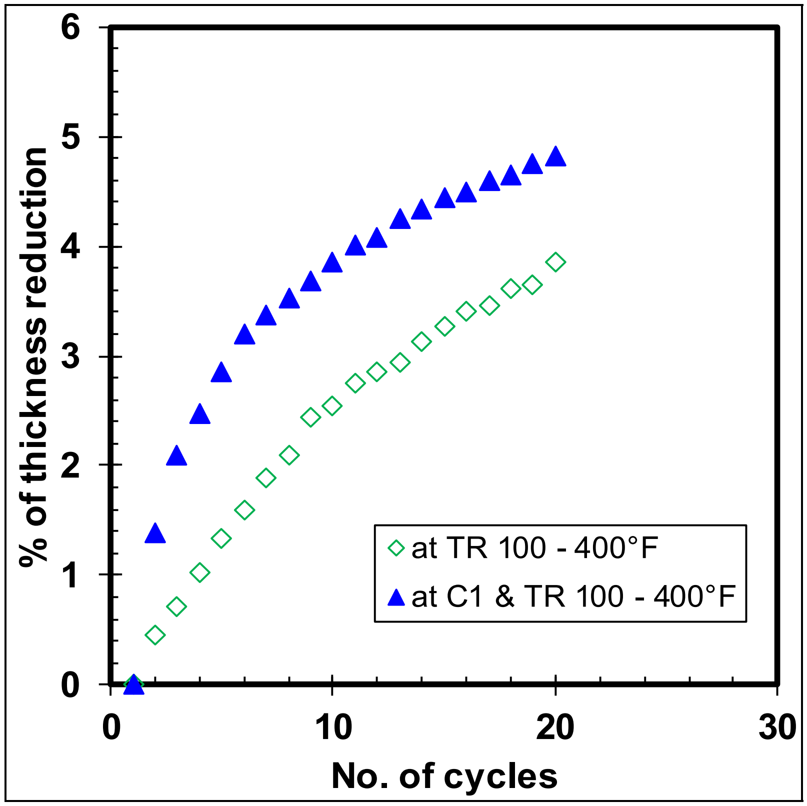

Figure 5. 8 ePTFE - % of thickness reduction under
the effect of creep time period

The percentage of thickness loss of vPTFE material doubled when the ratcheting temperature is increased from 250 to 350°F (Figure 5.9a); hence, the importance of ratcheting temperature is unmistakable. The induced cumulative damage is significant and this would influence the bolt load drop in a flange joint. In addition, the percentage of thickness loss (Figure 5.9b) is intensified by 2% with an initial one-day creep prior to cycling. The susceptibility of vPTFE to creep and thermal ratcheting is much higher than ePTFE material.

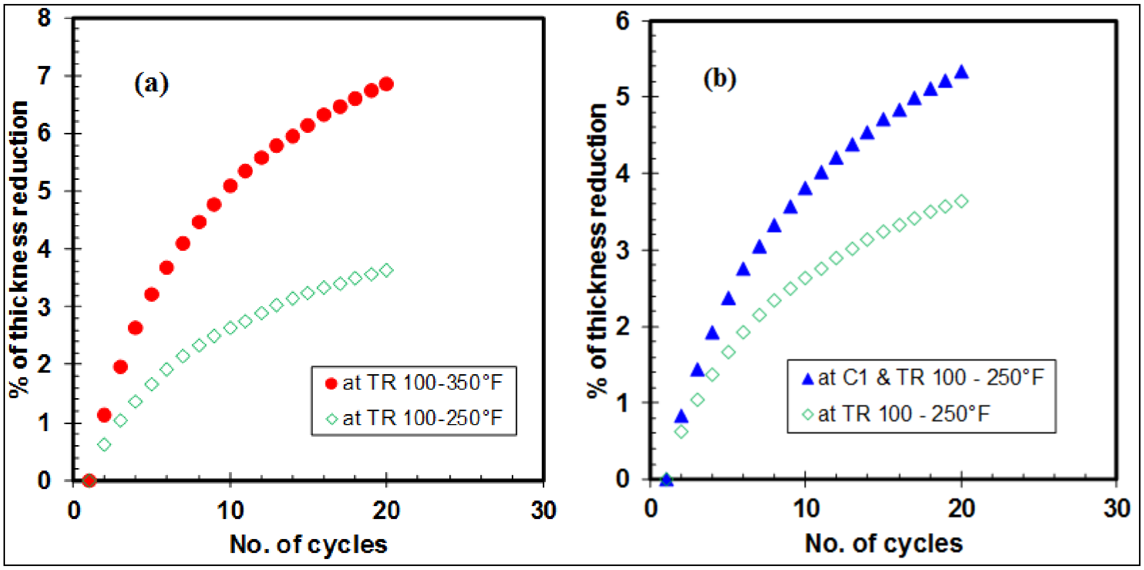

Figure 5. 9 vPTFE - % of thickness reduction
(a) under different ratcheting temperature range,
(b) under the effect of creep time period

The compressed non-asbestos fiber gasket is less affected by the variation of the ratcheting temperature and initial creep. From Figure 5.10a, CNA displays a 1% increase in thickness loss with an increase of 100°F in the ratcheting temperature. The rate of thickness reduction is higher for the CNA gasket subjected to higher ratcheting temperature. As for as the effect of creep prior to thermal ratcheting (Figure 5.10b) is concerned, there is hardly any increment in the percentage of thickness loss. The data indicate that there is virtually no subsequent effect of initial creep on the thickness loss; however, the information pertaining to a wide range of initial creep exposure prior to thermal cycling is essential before any generalization can be made.

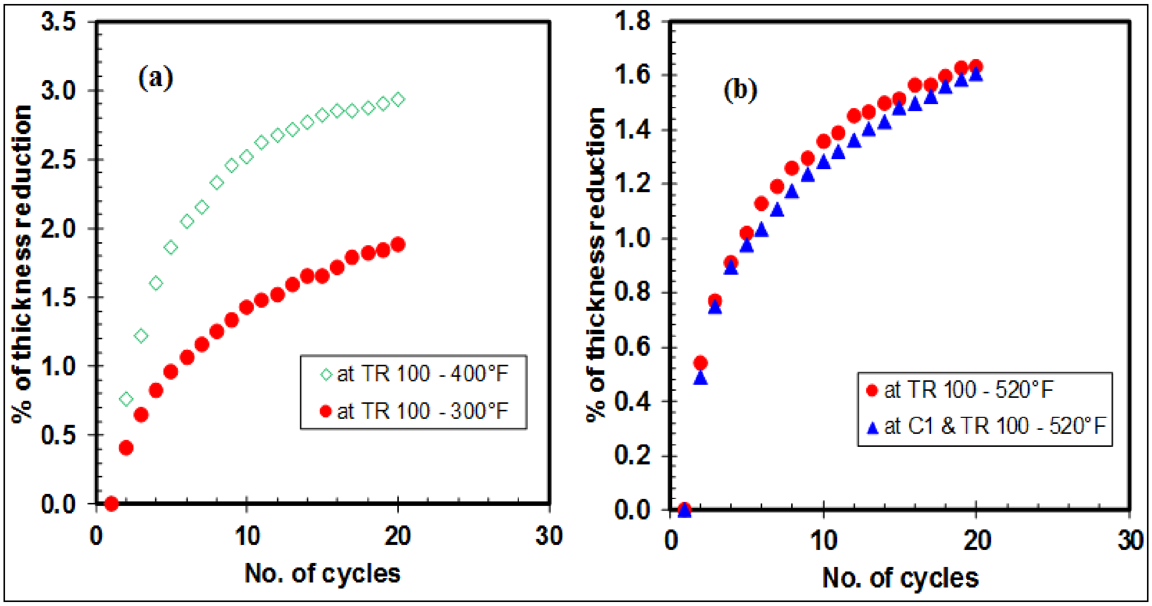

Figure 5. 10 CNA - % of thickness reduction (a) under different ratcheting temperature range, (b) effect of initial creep exposure prior to ratcheting

5.4.3.2 Coefficient of thermal expansion

Finally, the effect of thermal ratcheting (CTE) on the coefficient of thermal expansion of the three-gasket materials is presented. This work is a continuation of the previous research (Kanthabhabha Jeya and Bouzid, 2013) on thermal ratcheting behavior of Teflon based gaskets. It has been clearly established that the CTE varies with the applied load and with each thermal cycle. To further the study, consequence of creep pre-exposure and thermal ratcheting temperature is evaluated. The equation for the determination of CTE is obtained from article published by professor Bouzid et al. (2001). The ePTFE material (Figure 5.11) exhibits a decrease in CTE with an increase in creep pre-exposure under the same thermal ratcheting temperature. The trend of the curve is more convex for the specimen tested without initial creep than the specimen tested with one day of creep exposure under the same ratcheting temperature. The CTE is given by:

$$\alpha = \frac{dD_g}{dT_g}\frac{1}{t_g} \tag{5.2}$$

Where, α is the coefficient of thermal expansion in in./in.°F, D_g is the gasket displacement in inch, t_g is the gasket thickness in inch and T_g is the gasket temperature in °F.

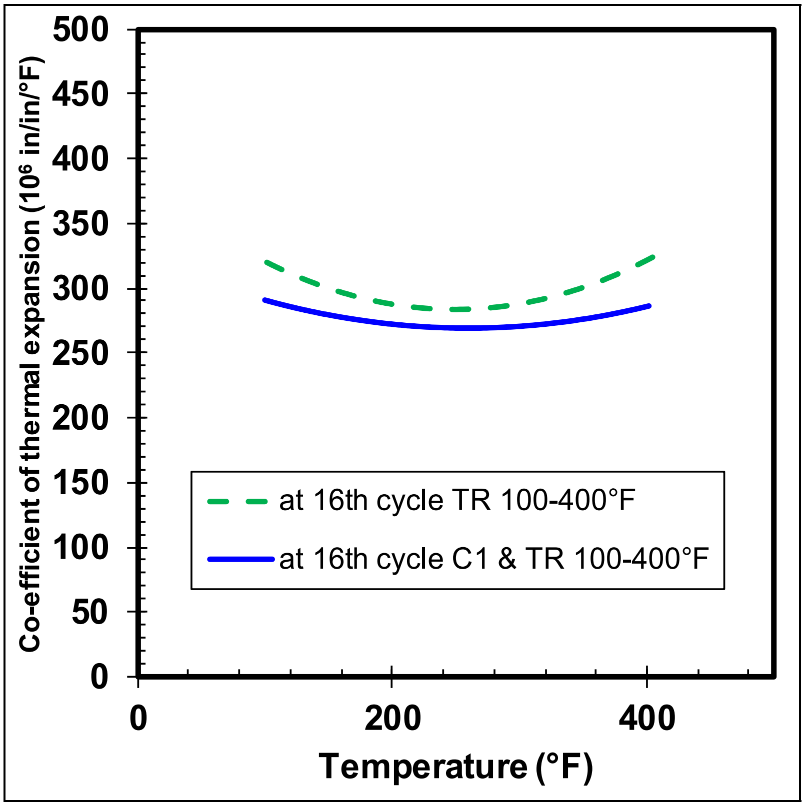

Figure 5. 11 ePTFE – Effect of creep pre-exposure on the coefficient of thermal expansion

From Figure 5.12b, the consequence of creep pre-exposure on the CTE of vPTFE is obvious, where the magnitude of CTE decreases with increase in creep time. On comparison to ePTFE, the initial creep significantly affects the CTE of vPTFE. It accounts to roughly a 30 percent decrease in CTE under same ratcheting conditions. The effect of ratcheting temperature range on CTE (Figure 5.12a) of vPTFE is not significantly different even though the CTE curve is almost linear for the lower ratcheting temperature in contrast to the parabolic shape under higher temperature.

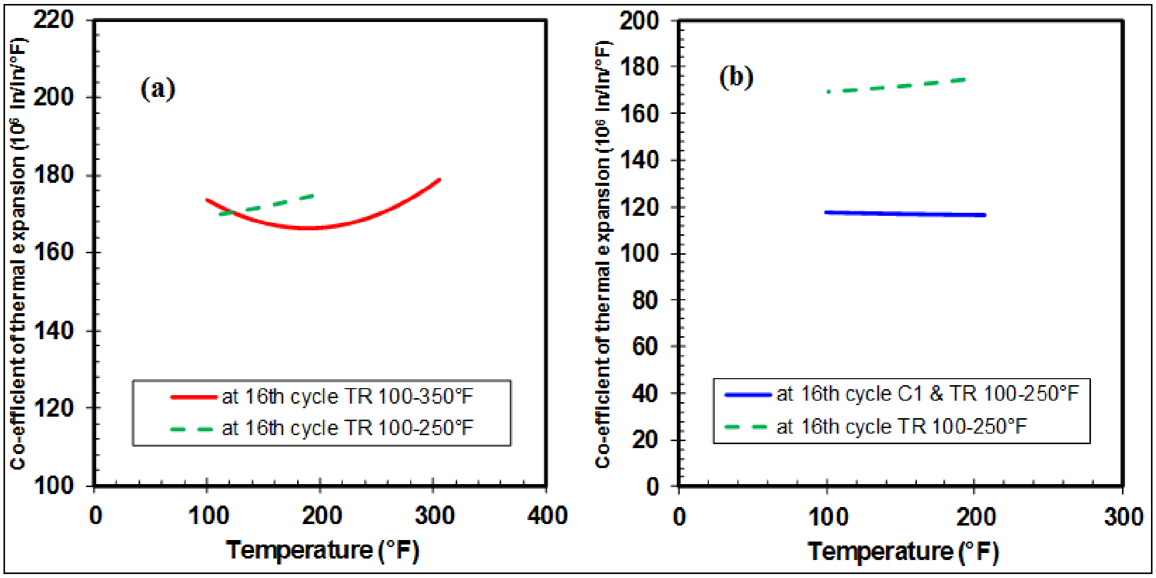

Figure 5. 12 vPTFE – co-efficient of thermal ratcheting (a) under different ratcheting temperatures, (b) with creep pre-exposure

For compressed non-asbestos fiber gasket, the variation in CTE under the same load but at different ratcheting temperatures is apparent. The magnitude of CTE decreases with the increase of ratcheting temperature as shown in Figure 5.13a. However, the creep pre-exposure does not have similar impact on the CTE of CNA. The CTEs of CNA (Figure 5.13b) with and without creep pre-exposure are intertwined with gap between the two values is higher at the highest temperature. It suggests that the coupled effect of creep and thermal ratcheting is nominal and it will tend to decrease with increase creep pre-exposure.

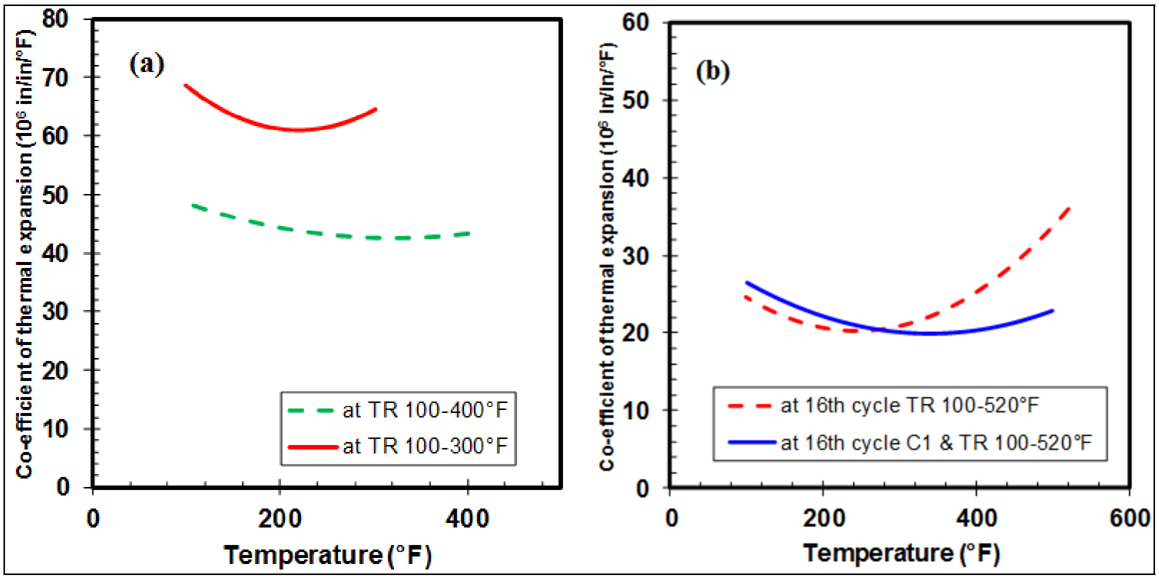

Figure 5. 13 CNA – co-efficient of thermal ratcheting (a) under different ratcheting temperature range, (b) under the effect of creep time-period

5.5 Conclusion

The creep and thermal ratcheting characterization of two Teflon based and a compressed non-asbestos fiber gasket materials are investigated experimentally using universal gasket rig. The major outcomes of the characterization tests are abridged:

- Both Teflon based materials, ePTFE and vPTFE, exhibit significant increase in creep strain with thermal ratcheting during the secondary creep phase. The magnitude of damage is higher in vPTFE than in ePTFE.
- CNA exhibits insignificant change in creep strain and creep modulus values under thermal ratcheting. Nevertheless, it exhibits up to 3% cumulative thickness change after 20 cycles at 450°F.
- The creep modulus of both PTFE gaskets decreased excessively with each thermal cycles in comparison to the creep modulus loss under constant temperature. vPTFE material demonstrated the high vulnerability to thermal ratcheting.
- All three materials show an increase in creep strain with decrease in the material temperature. It is cited to the reason that the decrease in thermal energy causes contraction of the material thickness.

- The percentage of thickness reduction amplifies with increase in thermal ratcheting temperature for vPTFE and CNA. In addition, both PTFE materials displayed intensification of thickness loss when exposed to creep prior to thermal cycling while CNA showed no significant thickness change.
- The variation of CTE with and without creep pre-exposure is evident in all three materials but the largest change occurred with the vPTFE material. The effect of ratcheting temperature on the CTE for vPTFE and CNA shows an intertwining and visible clear change in CTE for the two materials, respectively.

This data distinctively provides further justification in the necessity to include thermal ratcheting for Teflon based gasket materials in predicting their long-term behavior in different applications. The results are useful for FE simulation in order to estimate the perennial behavior of these materials. A quantitative data covering a wide range of creep and thermal ratcheting property conditions would be handy for practical bolted flange joint applications.

CHAPTER 6

INFLUENCE OF THERMAL RATCHETING ON THE CREEP AND MECHANICAL PROPERTIES OF HIGH DENSITY POLYETHYLENE (HDPE)

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This article was submitted to the Journal of Engineering Materials and Technology on 9th July 2018

6.1 Abstract

The objective of this research is to describe the consequence of thermal ratcheting on the long-term creep property of HDPE material. The thermal ratcheting phenomenon amplifies significantly the creep strain of HDPE in comparison to the steady creep strain under constant temperature. The magnitude of creep strain of HDPE increases by 8% after just 20 thermal cycles between 28 and 50°C. The creep modulus which is inversely proportional to the creep strain depletes further under thermal ratcheting. Both properties change significantly with the number of thermal cycles. The coefficient of thermal expansion (CTE) of HDPE varies with the applied compressive load, with successive thermal cycles and with the thermal ratcheting temperature. The impact of thermal ratcheting diminishes with increase in initial steady creep exposure time-period but still the magnitude cumulative damage induced is noteworthy. The magnitude of growth in creep strain drops from 8 to 2.4% when thermal ratcheting is performed after 1 and 45 days of steady creep, respectively. There is a notable change in thickness of the material with each heating and cooling cycle even after 45 days of creep however, the thermal ratcheting strain value drops by 80% in comparison to thermal ratcheting strain after 1 day of creep and under similar test conditions.

6.2 Introduction

The range of engineering applications involving the use of polymeric materials is on a rapid raise, varying from aerospace to electronic systems to domestic sewage piping network. This excessive exploitation led to intensive research on comprehending the mechanical properties along with the viscoelastic and viscoplastic behavior of the polymers under a variety of loading conditions. In this framework, an extensive knowledge on the creep response of polymeric materials subjected to thermal ratcheting is of high value in order to predict the long-term performances. A proper assessment of the perennial properties is the need of the hour as it aides in overcoming the premature failures due to creep and other time dependent failures. Nowadays, PVC and HDPE materials constitute for a major share of polymeric pipes in use with 75% and 20% of the total, respectively (Mruk et al., 1988; Stewart, 2005). Primarily the pressure pipes are susceptible to failure due to SCG slow crack growth, hence, a quantitative number of experimental test to understand the SCG/creep failure of various polyethylene are performed (Brown and Bhatacharya, 1985; Lu and Brown, 1987; Brown and Wand 1988; Lu et al., 1988; Wang and Brown, 1989; Lu and Brown, 1990; Lu et al., 1991, Ward et al., 1990). Some of the early data on the creep property of PVC and other polyethylene materials under liquid pressure at different temperatures are published by Niklas and Eifflaender (159). The author (Bergen, 1967) worked on the creep response of the polymers at temperatures near the glass temperatures Tg of the materials.

A large influx of research on the creep property of HDPE material led to development of equations to describe this time dependent property (Zhang and Moore, 1997; Zhang and Moore, 1997; Lai and Bakker, 1995; Colak and Dunsunceli, 2006). Out of the two models presented in the articles (Zhang and Moore, 1997 and Zhang and Moore, 1997), the viscoplastic model has a higher accuracy with the experimental results. The authors (Lai and Bakker, 1995) studied the effect of ageing on the nonlinear creep model. The work presented by Hamouda et al., (2001) characterizes the crack initiation and propagation of ductile and brittle polyethylene under creep damage.

In addition, a considerable amount of research work focused on the fatigue or mechanical ratcheting of HDPE (Dusunceli et al., 2010), HDPE geogrid (Cardile et al., 2016), solid extrude HDPE (Kaiya et al., 1989), HDPE composite (Dong et al, 2011), HDPE pipe joint (Chen et al., 1997), however, the research on the impact of thermal ratcheting is still in the nascent stages. Thermal ratcheting is the cumulative damage induced in the material due to the cycling of temperature. Unlike metallic materials, the vulnerability of polymers to temperature is well documented; thereby a quantitative analysis of the thermal ratcheting behavior of HDPE for predicting perennial properties is essential. The resistance to thermal ratcheting is of particular interest for HDPE material especially in bolted flange joint application, as fluctuation of thickness with temperature cycles would amplify the loss of bolt load. The authors (Kanthabhabha Jeya and Bouzid, 2017; Kanthabhabha Jeya and Bouzid, 2018) presented extensive information on the thermal ratcheting behavior of polymer and fiber-based materials. The work on HDPE (Kanthabhabha Jeya and Bouzid, 2018) elaborates the effect of thermal ratcheting and creep under compression on the overall deformation induced in this material.

In this research paper, the influence of thermal ratcheting in intensifying the deformation incurred in the material subjected to a subsequent steady compressive creep is presented. In addition, the effect of thermal ratcheting on the coefficient of thermal expansion (CTE) and the influence of thermal ratcheting at different creep exposure time-period are evaluated.

6.3 Materials and Methods

6.3.1 Experimental Setup

The thermal ratcheting behavior of high-density polyethylene is successfully evaluated by the use of sophisticated Universal Gasket Rig (UGR) experimental setup shown in Figure 6.1. The cumulative damage induced on the circular sample is assessed precisely by measuring the changes in the axial thickness of the specimen under compressive load. The test bench facilitates for coupled analysis of mechanical, thermal and leak testing of the material sample using simple technologies. The system restricts the samples to be in ring or gasket shape as initially this machine was designed with objective to characterise gasket materials. The maximum and minimum diameter along with maximum thickness of the sample that can be accommodated are 100, 50 and 10 mm, respectively. The standout feature of UGR is the capability to exerted combination of loads – mechanical, thermal and internal pressure. The UGR can function accurately under extreme conditions, where a maximum internal pressure of 5 MPa could be applied on the specimen at 450°C under a compressive stress of 100 MPa. An elaborate operation of the UGR is presented in scientific articles (Kanthabhabha Jeya and Bouzid, 2017; Kanthabhabha Jeya and Bouzid, 2018).

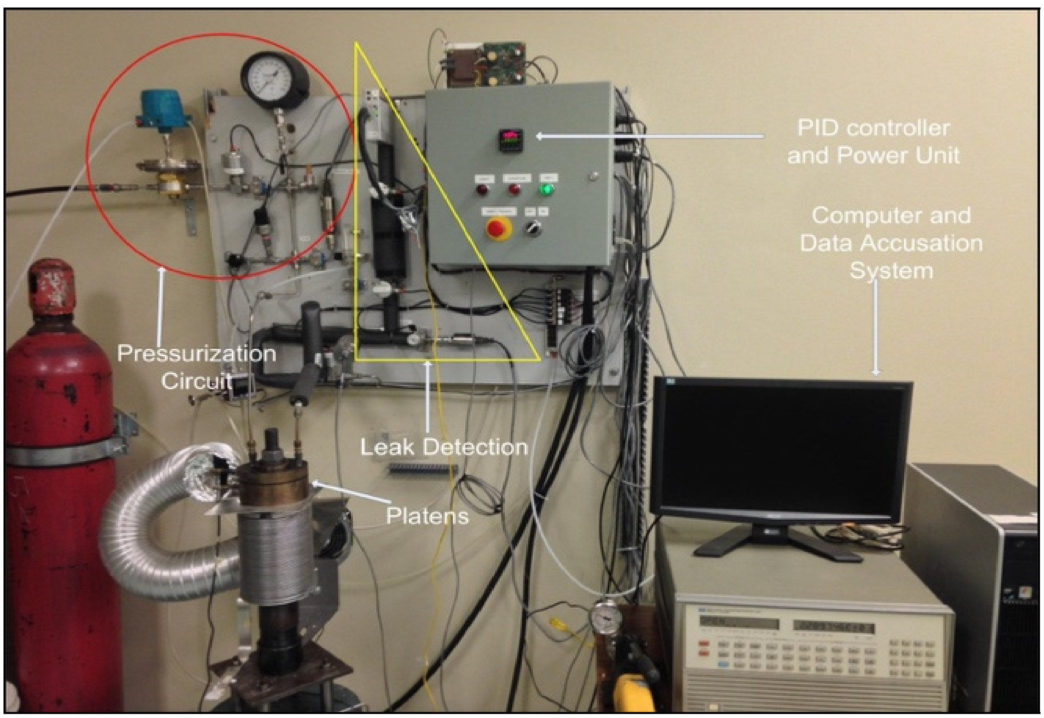

Figure 6. 1 Universal Gasket Rig test bench

6.3.2 Test procedure

The greatest advantage of UGR is that the system is semi-automated with few initial manual maneuvers. To start-off, the dimensions of the test samples are physically measured using a Vernier caliper. Subsequently, these values are given as inputs to the LabVIEW program, which is used in the evaluation of applied stress on the material. The UGR consist of two enclosing platens resting on a central stud, between which the specimen is compressed. The sample is held in position between the two platens by means of manual locking of a nut on the central stud. Later, the desired amount of compressive stress is exerted on the ring sample by the use of a hydraulic system with real-time monitoring of stress through a Full-bridge strain and a program written in LabVIEW software. Before the application of the compressive load by the hydraulic system, the zero-reference position for the LVDT sensors is obtained by applying a minimum stress on the specimen by manual hand tightening. The heating is performed at a rate of 1.5°C/min while the cooling is achieved through dissipation or loss of heat to the surroundings after shut-off heater at the desired temperature. The system is equipped with high sensitive instrumentations with a high range of precision namely LVDTs, strain, temperatures and time gages. All measurements are monitored in real-time with a refreshment rate of 10 seconds and recorded at regular intervals depending on the test through the LabVIEW program. The physical dimensions of the HDPE samples are in accordance with an NPS 3 schedule 80 pipe and a cut sample is provided in figure 6.2. Further details and explanation on the test procedure are provided in journal publications (Kanthabhabha Jeya and Bouzid, 2017; Kanthabhabha Jeya and Bouzid, 2018).

Figure 6. 2 HDPE test sample

6.4 RESULTS AND DISCUSSIONS

The objective of this work is to understand the influence of thermal ratcheting on the creep strain of high-density polyethylene material. Thermal ratcheting is defined as the cumulative damage induced in the materials as result of cycling of temperature. The creep vulnerability of HDPE under compression is highlighted in paper (Kanthabhabha Jeya and Bouzid, 2018). The results on the consequence of thermal ratcheting and creep of HDPE are analyzed and presented under the sections - creep strain, creep modulus, co-efficient of thermal expansion and coupled creep thermal ratcheting analysis. The results signify the importance of considering the effect of thermal ratcheting in the estimation of the long-term performance of HDPE. The present research work is of particular interest to piping industry that uses HDPE bolted flange joints, for which creep is cited to be the primary reason for leakage failure.

6.4.1 Creep Strain

Simplistically a comparative study on the creep behavior of HDPE at high temperature with HDPE creep at high temperature and thermal ratcheting would be the point of interest to comprehend the consequence of thermal ratcheting on the HDPE creep response. It has been clearly understood that the raise in temperature of the HDPE material under compression would accelerate the creep rate (Kanthabhabha Jeya and Bouzid, 2018). Hence, any further amplification of creep would be critical, especially in bolted flange applications where HDPE flange creep strain causes bolt load drop, which would lead to leakage failure. Also, from the paper of Kanthabhabha Jeya and Bouzid (2018), the creep phase transition from primary phase to secondary phase occurs within 24 hours of start of HDPE creep test. Therefore, a thermal ratcheting study after 1 day of creep would be enough to justify the effect, as the secondary creep growth persist for months to years depending on the material. From Figure 6.3, it is obvious that the thermal ratcheting phenomenon intensifies the creep growth of HDPE. Interestingly the growth of creep strain did not saturate under the 20 thermal cycles, hence a subsequent ratcheting would further augment the creep strain under compressive load. The comparative study was performed on two ring samples cut from the same HDPE pipe, which were tested under 14 MPa of compressive stress at 50°C with or without thermal ratcheting. The ratcheting temperature range was chosen as 28 to 50°C, so that at the end of each cycle the new creep strain with the same load and temperature of the former sample is obtained. The cumulative damage induced due to thermal ratcheting is the primary attributor for the extensive damage on the HDPE material. The impact of thermal ratcheting is evident as early as the third thermal cycle as the creep strain curve of the sample tested with thermal ratcheting starts to deviate from that of the creep strain of the sample under constant temperature. The magnitude of growth of creep strain aggregates with each thermal cycle and its amount is close to 17% after 20 thermal cycles.

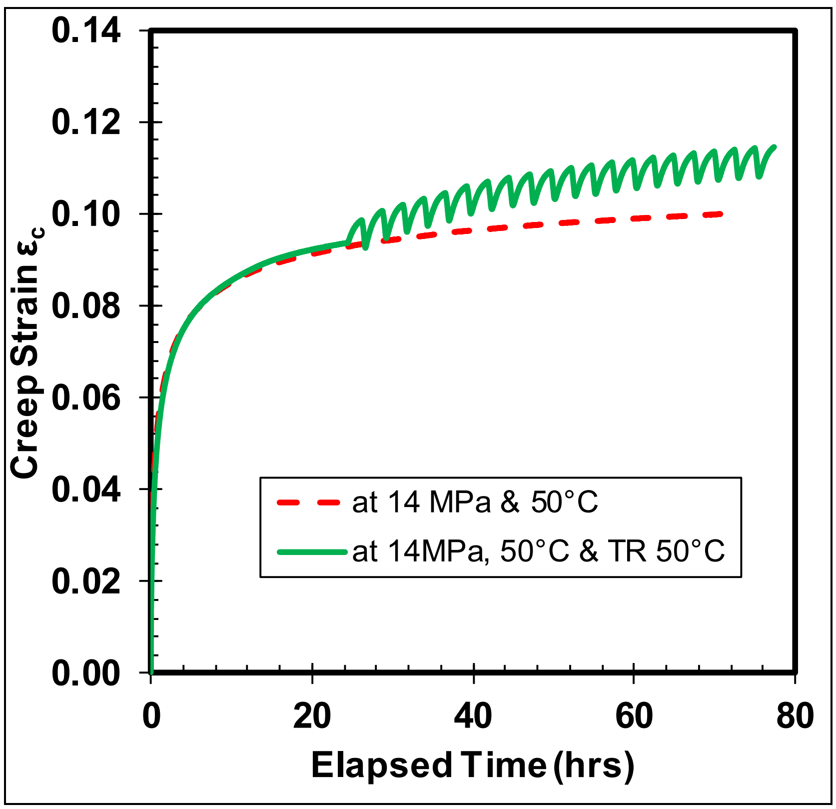

Figure 6. 3 HDPE creep strain with and without thermal ratcheting

As shown from Figure 6.4, the projected value of creep strain at 100 hours of test after thermal ratcheting is significantly higher than secondary creep strain tested under constant temperature. Therefore, the effect thermal ratcheting should be taken into consideration when designing structures subjected to thermal cycling, as an 17% increase in creep strain means a substantial loss in bolt load thereby a rapid raise in the chances of failure by leakage. It has been established that the temperature of the materials plays a major role in creep behavior of HDPE. With the raise in temperature the magnitude of creep strain increases; however, on lowering the core temperature of the material during the secondary creep stage intensifies the damage due to creep under the same load rather than decreasing the creep strain. The material exhibits a rapid increase in creep strain as the lowering of temperature continues and then continues to creep in a new secondary creep stage, once the temperature of the material saturates. The loss of thermal energy due to diminution of temperature is cited as the reason from this characteristic growth of creep strain.

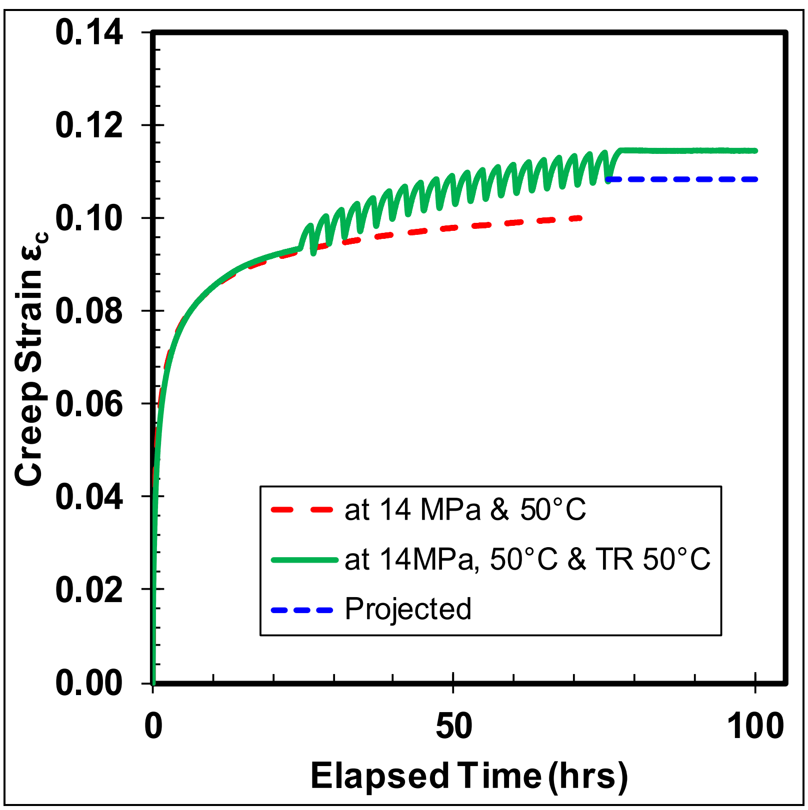

Figure 6. 4 HDPE creep strain projected at same constant temperature after 20 thermal cycles

6.4.2 Creep Modulus

The result evidently indicates that the drop-in creep modulus intensifies with thermal ratcheting phenomenon. The assessment of impact of thermal ratcheting on the creep modulus is achieved from the same samples used in the creep strain section. The creep modulus is the ratio of creep stress over creep strain, which decreases with time. Since the creep stress remains constant and it has been already established (Figure 6.3) that the creep strain gains in magnitude with each thermal cycle, a further decrease of creep modulus is expected with thermal ratcheting. As seen from Figure 6.5, the decrease of creep modulus of HPDE sample under thermal ratcheting intensifies with each thermal cycle in comparison to the creep modulus under the same load but at a constant temperature. The analysis of the influence of thermal ratcheting on the creep modulus is performed during the beginning of the secondary creep phase to comprehend the adverse influence at a later stage of this creep phase. As discussed in the article by Kanthabhabha Jeya and Bouzid (2018), the strain hardening of the material to thermal ratcheting is evident but there is no absolute saturation of the cumulative damage. The magnitude of decrease in creep modulus with each thermal cycle depletes, however, even at the end of 20th thermal cycle the reduction of the creep modulus did not stop. The decrease in creep modulus due to thermal ratcheting at the end of 20th thermal cycle as compared to the one under steady creep is nearly 17%. This amount is obviously significant thereby, highlighting the adverse influence of thermal ratcheting on this HDPE material property. On evaluating the creep modulus at the same temperature between the 19th and 20th thermal cycle, there is a 1% decrease while between the 5th and 6th there is a 2.1% decrease between the two cycles. This behavior validates the reduction of the impact of thermal ratcheting with the increase of the number of thermal cycles. Similar to creep strain, the initial reduction of creep modulus is significantly higher characterizing the primary phase and the material loses 85% of the initial creep modulus value during this phase.

$$\textit{Creep Modulus} = \sigma_c/\varepsilon_c = \frac{\textit{Applied stress}}{\textit{Creep strain at the instant}}, \tag{6.1}$$

Where σ_c is the applied creep stress (MPa) and ε_c is the creep strain at that instant.

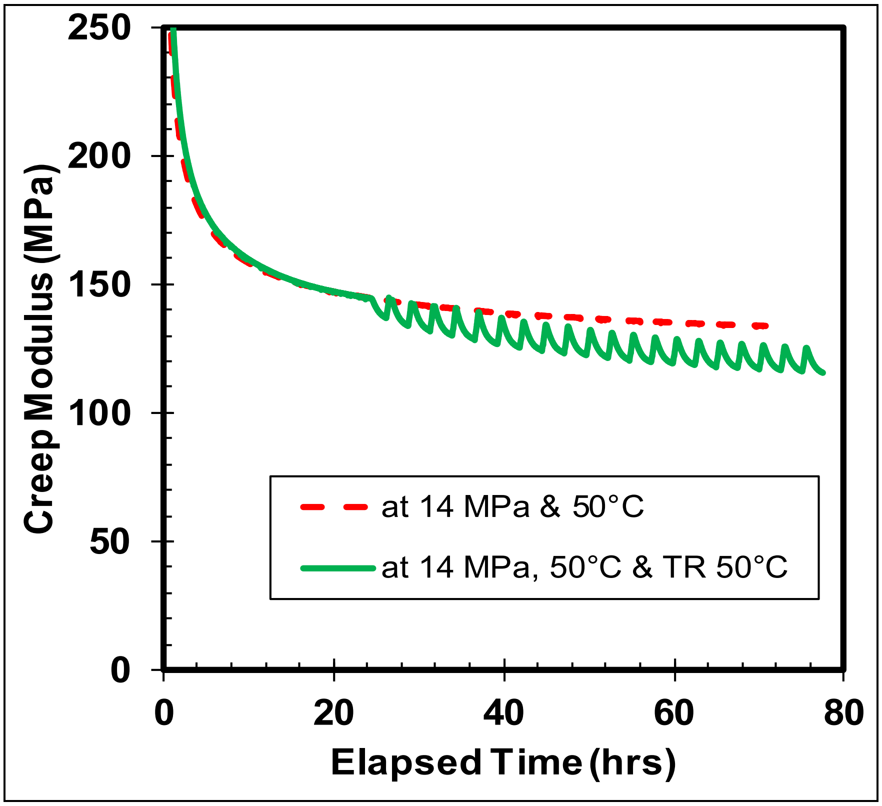

Figure 6. 5 HDPE creep modulus with and without thermal ratcheting

As observed from Figure 6.5, there is a gradual decrease in the magnitude of creep modulus with each cycle when comparing the upper and lower bound of the thermal ratcheting curve. The upper and lower bound refers to the highest and lowest temperature of the thermal ratcheting cycle, respectively. The reason for this particular behavior is due to the rate of heating and cooling of the specimen during the ratcheting test. While the heat is administered at 1.5°C/min, respecting industrial fluid process standard, the cooling is achieved through natural convention. This indicates that the cooling process is longer than the heating stage, which provides for higher creep time thereby increasing the magnitude of cumulative damage incurred during the cooling stage than in the heating stage. Even though the magnitude of loss in creep modulus increases with increase in temperature of the material, the effect of cooling from a higher temperature is of a major concern. As explained earlier the loss of thermal energy causes a significant increase in the damage induced by thermal ratcheting.

6.4.3 Coefficient of thermal

A comprehensive study on the thermal ratcheting behavior of HDPE material is published in the journal paper (Kanthabhabha Jeya and Bouzid, 2018). This section focuses on the coefficient of thermal expansion (CTE) of HDPE under the influence of creep and thermal ratcheting. CTE is defined as the measure of expansion or contraction of a material under varying temperature. This value is of importance for performing long-term finite element simulation of structures subject to cyclic temperature. Furthermore, with the possibility of measuring the radial displacement of the sample with each thermal cycle would lead to analyzing the link between CTE and radial flow of the material.

The results on the CTE of HDPE give insight on the changes occurring in the magnitude of the property under different test conditions. Principally, the influence of applied load, the number of thermal cycles and thermal ratcheting temperature range on the CTE of the HDPE are studied. The paper (Kanthabhabha Jeya and Bouzid, 2018) presented the effects of these test conditions on the thermal ratcheting strain of the material. The assessment of CTE is performed with the formula provided by Bouzid et al. (2001), which is standardized by ASTM. Overall, all three-test conditions cause a significant change in the value of coefficient of thermal expansion. A single NPS 3 schedule 80 HDPE pipe was precisely cut into multiple samples of similar thickness with the use of CNC machine. Dimensionally, all the test samples are the same thereby limiting the discrepancy due to physical shape. As shown in Figure 6.6, the effect of the compressive load on the CTE is evident, where the CTE increase with the applied load under the same thermal ratcheting temperature range and initial creep pre-exposure time-period. In addition, it can be observed that the CTE is not linear over the ratcheting temperature range under the compressive load. Although, the variation in the amount of CTE between the upper and lower ratcheting limit is meagre, the change is significant with the compressive load. The maximum difference in the CTE value with the applied load condition occurs at the lowest and the lower and upper bound ratcheting temperatures as these bounds relates to the sudden change in the cooling and heating phases. The value of CTE increases up to 120 µmm/mm/°C with a 7 MPa increase of compressive stress at the lowest ratcheting temperature bound. However, on an average, doubling the load would produce an 80 μmm/mm/°C increase in the magnitude of CTE of HDPE material.

$$\alpha = \frac{dD_g}{dT_g}\frac{1}{t_g} \tag{6.2}$$

Where α – coefficient of thermal expansion, D_g – material displacement (mm), T_g - material temperature (°C), t_g – material thickness (mm).

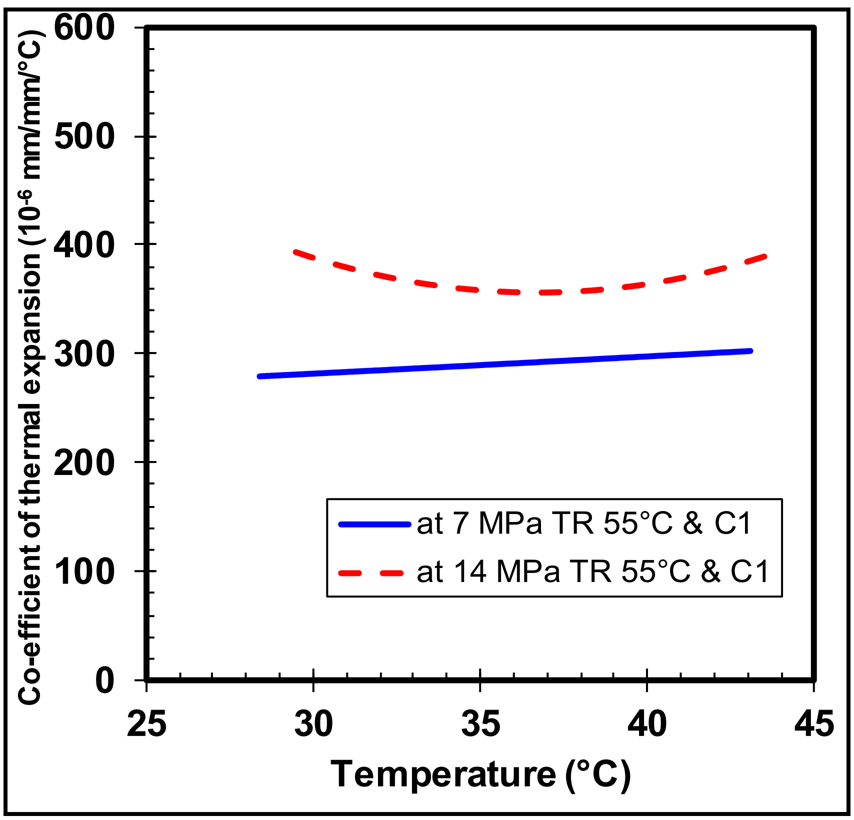

Figure 6. 6 HDPE - CTE under change in applied compressive load

The influence of number of thermal cycles on the CTE of HDPE is illustrated in Figure 6.7. The impact of thermal ratcheting is strongly evident as the CTE of HDPE decreases with each thermal cycle. The HDPE test specimen was subject to a 7 MPa of compressive load and ratcheted thermally between 28 and 55°C after one day of initial creep. Figure 6.7 shows a comparison of the CTE at three different thermal cycles – 5^{th}, 9^{th} and 17^{th} cycles. The decrease in the CTE is due to the induced cumulative damage with each cycle. The amount of reduction with each cycle diminishes, which suggests that a hardening effect takes place. Comparing to compressive load, the thermal cycles have lesser effect on CTE.

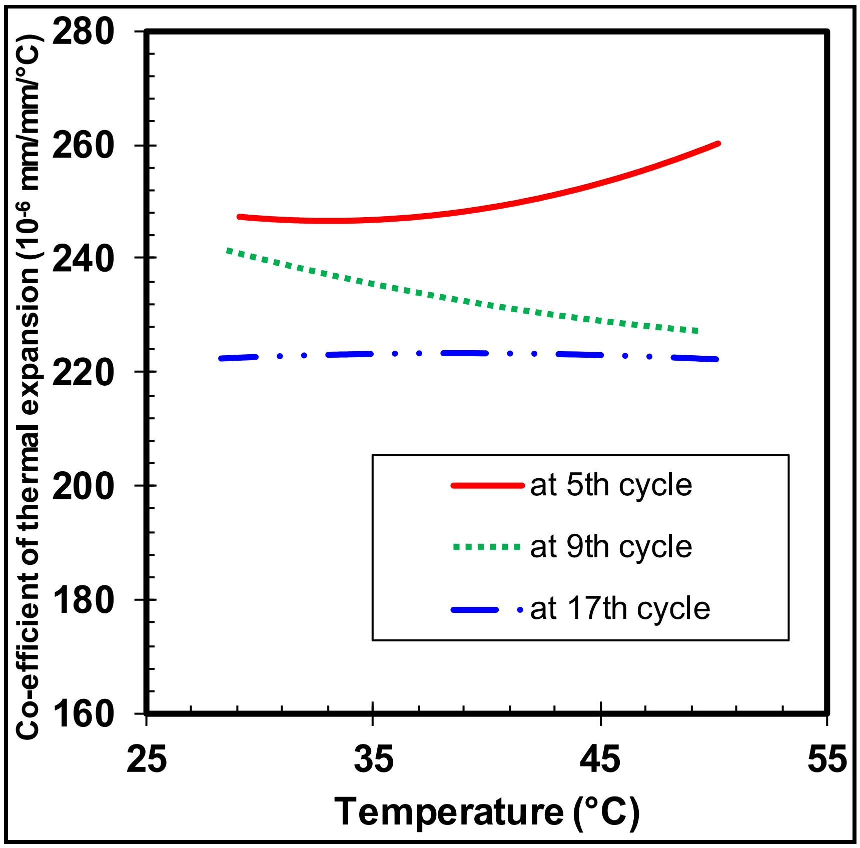

Figure 6. 7 HDPE - CTE under change in number of thermal cycles

Figure 6.8 is an example of the influence of the third test condition on the CTE with similar creep-time and compressive load. Relatable to other two test conditions, the CTE varies with changes in the thermal ratcheting temperature range. It is interesting to note that the magnitude of CTE shrinks with raise in the thermal ratcheting temperature range. The increase of the cumulative damage with ratcheting temperature range is cited as the reason. The difference in the CTE value of HDPE between two different thermal ratcheting temperature samples is around 10 μmm/mm/°C at the lower bound.

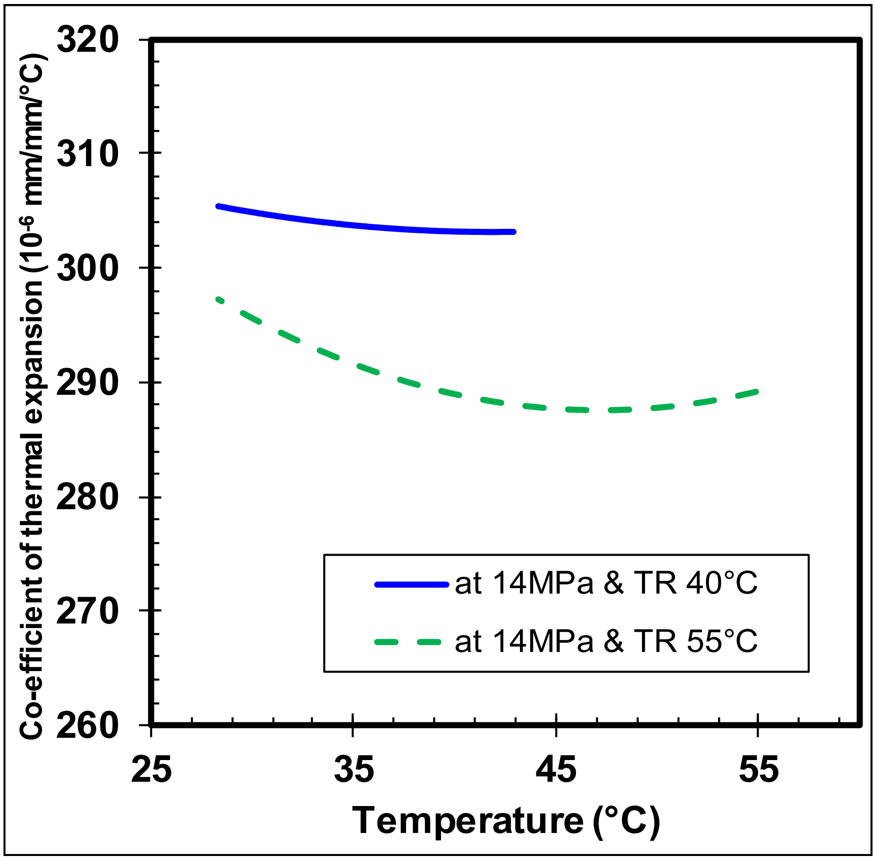

Figure 6. 8 HDPE – CTE at different thermal ratcheting temperature

6.4.4 Coupled Creep Thermal Ratcheting Analysis

The significance of creep pre-exposure time on the thermal ratcheting behavior of HDPE, was presented in (Kanthabhabha Jeya and Bozuid 2018), in terms of the level of thermal ratcheting strain. The magnitude of the subsequent thermal ratcheting strain decreases with an increase in creep pre-exposure time. Hence, it is suspected that the consequence of thermal ratcheting would weakens post long secondary creep period. Therefore, a thermal ratcheting test after 45 days of initial steady creep at 60°C is performed to evaluate the influence of thermal ratcheting on the creep strain. Figure 6.9 shows the creep strain curve of HDPE test at 60°C and 14 MPa of compressive stress. On comparing the secondary creep growth between day 10 and day 45, a 6% increase in creep strain is observed. A 30 thermal cycles between 28 and 60°C after 45 days of creep, produces a 2.4% increase in creep strain value. With reference to time exposure, thermal ratcheting intensifies the creep strain by 2.4% in three days while creep at constant temperature produces only 0.17% for the same period. Even though, the level of growth of creep strain decreases with an increase in creep pre-exposure time, the effect of thermal ratcheting cannot be neglected.

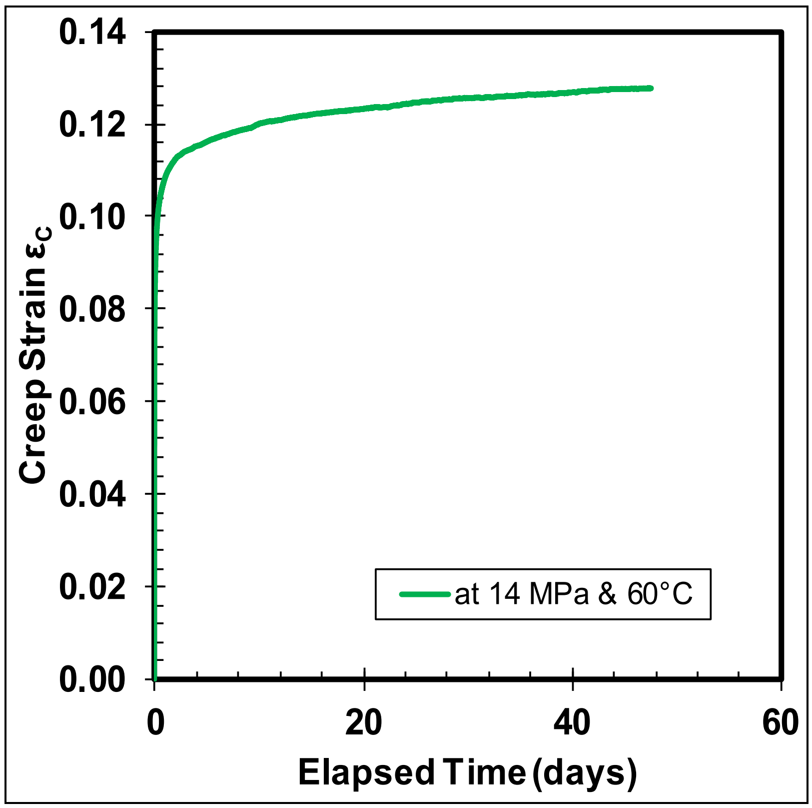

Figure 6. 9 HDPE creep strain at high temperature (45days)

Figure 6.10 illustrates the thickness variation during the thermal ratcheting test and in particular the reduction of thickness with thermal cycling, emphasizing the cumulative damage produced in the material. The thickness of the sample varies within 0.06 mm representing 1% strain. This can result in a significant bolt load change in flange joint application.

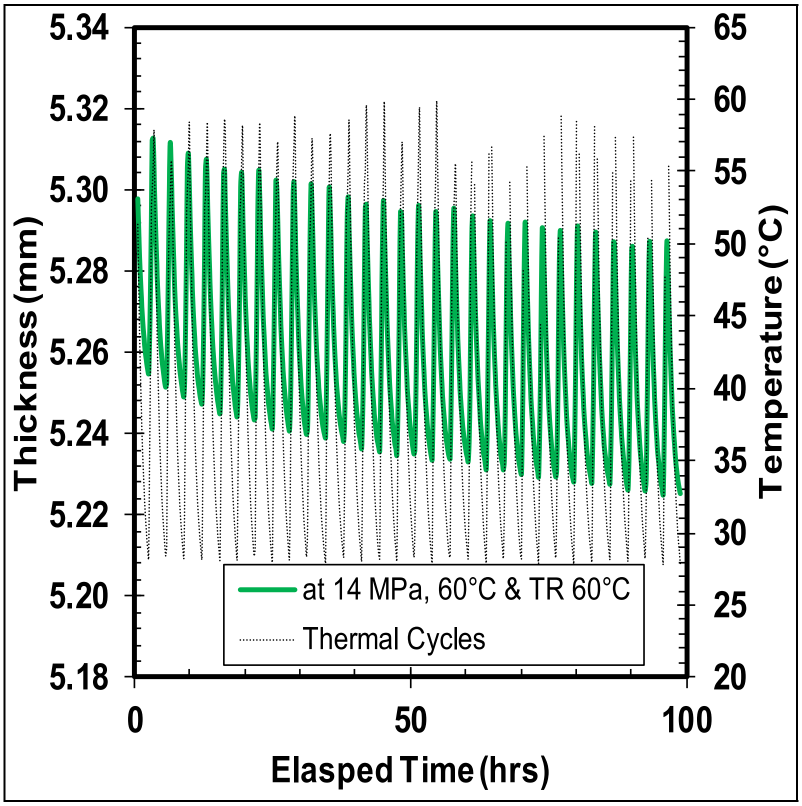

Figure 6. 10 Thickness variation with thermal cycling after 45 days of creep pre-exposure

Finally, a comparative study on the impact of creep pre-exposure on the thermal ratcheting strain of HDPE material is presented in Figure 6.11. The thermal ratcheting strain is calculated by the formula given below (Eq. 3). The HDPE material exhibits a hardening effect, which increases with increase in creep pre-exposure time. This causes a decrease in magnitude of cumulative damage induced due to thermal ratcheting thereby reducing the thermal ratcheting strain value. In fact, thermal ratcheting strain decreases by 80% after 45 days of steady creep in comparison to the TRS with zero days of steady pre-exposure creep. Nevertheless, thermal ratcheting strain (TRS) increases with each cycle but the magnitude of growth with each cycle is lesser as compared to that of smaller initial creep pre-exposure time. The result demonstrates the significance of thermal ratcheting when taking place early in the life time of polymeric piping and structures in general.

$$\text{Thermal Ratcheting Strain } \varepsilon_{TRSx} = (LTR1 - LTRX)/Lo, \qquad (6.3)$$

Where, LTR1 refers to the thickness of the sample after the first thermal cycle, while LTRX represents the thickness of the sample with each thermal cycle, and Lo is the initial thickness of the specimen.

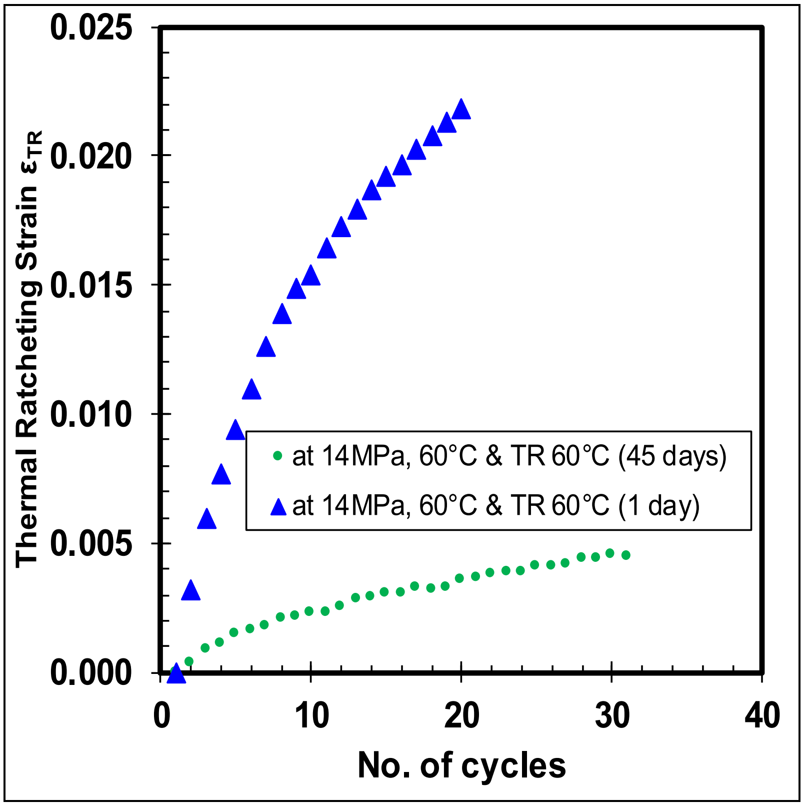

Figure 6. 11 HDPE - thermal ratcheting strain after 1 and 45 days of pre-exposure creep

6.5 Conclusion

The importance of thermal ratcheting characterization of HDPE is demonstrated through meaningful experimental testing. The standout outcomes of the influence of thermal ratcheting on HDPE material behavior are concise as follows,

- The effect of thermal ratcheting on deterioration of the creep resistance of HDPE is evident. The magnitude of the overall creep strain increases by 17% when HDPE is subjected to 30 thermal cycles under 14 MPa and 60°C.

- The creep modulus of HDPE is also affected by thermal ratcheting. The phenomenon causes a decrease in creep modulus of the material. Since the creep modulus is inversely proportional to the creep strain, the intensification of creep strain implies the receding of creep modulus.
- CTE of HDPE varies with applied load, thermal ratcheting temperature range and number of thermal cycles. The CTE decreases with an increase in number of thermal cycles and an increase in thermal ratcheting temperature while the CTE increases with an increase in the applied load.
- The impact of thermal ratcheting is significantly reduced with an increase of creep pre-exposure time; Nevertheless, even after 45 days of creep, the thermal ratcheting produces a growth of 2.4% of the creep strain, which is still significant in application such as bolted joints.

These findings further validate the need for inclusion of thermal ratcheting behavior of polymeric materials in design standards. This research was launched to cover polymeric piping and flange connections especially after initiatives and concerns raised by the ASME Committee for Non-metallic Piping Systems and the NESCC Polymer Piping Task Group (PPTG).

CHAPTER 7

CREEP-RELAXATION MODELING OF HDPE AND PVC BOLTED FLANGE JOINTS

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This article was submitted for publication in the Journal of Pressure Vessel Technology on 12th August 2018

7.1 Abstract

Similar to many polymer materials, High-Density PolyEthylene (HDPE) and PolyVinyl Chloride (PVC) show a clear creep behavior, the rate of which is influenced by temperature, load and time. Most bolted flange joints undergo relaxation under compression, which is caused by the creep of the material. However, the creep property of the two polymers are different under tension and compression loading. Since the sealing capacity of a flanged gasketed joint is impacted by the amount of relaxation that takes place, it is important to properly address and predict the relaxation behavior due to flange creep under compression and thereby reducing the chances of leakage failure of HDPE and PVC bolted flange joints.

The main objective of this study is to analyze the compressive creep behavior of HDPE and PVC flanges under normal operating conditions. This is achieved by developing a respective creep model for the two materials, based on their short-term experimental creep test data. Both numerical and experimental simulations of the polymeric flange relaxation behavior are conducted on an NPS 3 class 150 bolted flange joint of dissimilar materials, where one of the flange is made of HDPE or PVC material and the other one is made of steel SA105. The study also provides a clear picture on how the compression creep data of ring specimen may be utilized for predicating the flange bolt load relaxation over time at the operating temperatures.

7.2 Introduction

Within half century from its time of invention, both high-density polyethylene and polyvinyl chloride materials have invaded most of the applications of traditional materials, such as clay, iron, steel and other metallic materials. The popularity of the two polymer materials in Pressure Vessel and Piping (PVP) domain is due to its advantages of immunity to corrosion and resistance to chemical attacks. In addition, convenient installation and easier maintenance are the other important factors that have contributed to its wide range of applications.

HDPE and PVC pipes have been widely used for fluids conveyance and transportation in industrial and domestic household sites. Bolted flange joints are an important PVP component that are mainly used as a dismountable connection to ensure continuity of fluid confinement and circulation in pressurized equipment. Therefore, bolted flange joints is one of the critical point of the PVP system to safeguard structural integrity and leakage tightness. Furthermore, bolted flange joints are recommended assembly type to connect polymeric pipes and process equipment together. The creep behavior of HDPE and PVC materials have raised attention since the earlier times. Faupel, J.H (1958) has studied PVC pipe creep and stress rupture behaviors under different conditions of static stress and time. Niklas and Eifflaender (1959) tested the long-term behavior of two material types; PVC and polyethylene pipes loaded with liquid under pressure at various temperatures. After a decade, research works on high temperature creep behavior of PVC and HDPE close to the glass transition region took shape (Bergen, 1967).

As computer technology evolved, Finite Element Method (FEM) became the tool of choice to analyze the performance of polymer products (Pantelelis and Kanarachos, 1998; Veronda and Weingarten, 1975). The early creep models applied to FEA are not appropriate because they are not representative of the real time industrial applications (Sakaguchi and Kaiga, 1986). In addition, most of the earlier studies are related to the creep behavior under constant conditions of load and temperature leading to unrealistic behavior and less accuracy. Along with the increase in the use of polymer products, the number of characterization related studies increased (Sabuncuoglu et al., 2011; Dropik et al., 2002; Nunes et al., 2011). However, many of these studies focused on polypropylene and other materials, while only a limited work is done on selected materials. Most of the research works analyzed the creep behavior of polymers in an extremely short timespan where the primary creep phase is predominant; however, the long-term behavior is usually overlooked. Barbero and Ford (2004), applying the equivalent time and temperature method (ETT), analyzed the polymer aging problem.

Recently, with the development of rate-dependent creep modelling, researchers started to simulate and predict the relaxation due to creep of plastic materials based on experiment data conducted on specimen loaded in tension. Acceptable prediction of creep failure of PVC pipes under internal pressure is achieved (Laiarinandrasana et al., 2011). Other applications such as polymer composite has also been investigated, and the basic Norton creep model has been used in the research without considering the temperature effect (Pulngern, 2013). The literatures on HDPE and PVC pipping systems are primarily focused on pipes subjected to fatigue and other types of failure due internal pressure (Scavuzzo and Srivatsan, 2006; Mao et al., 2011; Hamouda et al., 2001). External forces such as bending are also considered because it is one of the major contributors to fracture failure. There is ample amount of research that has been carried out on the slow crack growth behavior of polyethylene (Lu and Brown, 1987; Lu et al., 1988; Lu and Brown, 1990). Wham et al. (2016) conducted experiments on a PVCO pipeline with bell-and-spigot joint subjected a simulated earthquake load. It shows that the PVCO pipeline has a good capacity to accommodate horizontal ground strain.

The data on relaxation of gasketed bolted flange joints is essentially focused on gasket creep (Bouzid and Chaaban, 1997). However, the contribution of the flange materials itself is significant in particular if it is made of HDPE or other polymer materials (Kanthabhabha Jeya and Bouzid, 2018). The rigidity of bolted joints greatly affects the level of relaxation (Bouzid and Nechache, 2010). This paper deals with the short-term bolt relaxation of an NPS

3 class 150 HDPE and PVC flanges subjected to a constant temperature. A finite element simulation that uses creep data from compressed circular ring samples is used to demonstrate the capacity of the proposed creep model to accurately predict bolt load relaxation.

7.3 Experimental Set-up

7.3.1 Universal Test Rig

The experimental test rig utilized for the compression creep tests of HDPE and PVC material samples is known as the Universal Test Rig (UTR), which is shown in Figure 7.1. The home built test rig consists of two platens that accommodate circular test samples with diameters ranging between 50 mm and 100 mm and a thickness of up to 10 mm. Additional information of this rig can be obtained from reference (Kanthabhabha Jeya and Bouzid, 2018).

7.3.2 HOBT fixture

The HOt Blowout Test (HOBT) fixture shown in Figure 7.2, was developed for the purpose of conducting the PTFE gasket test program (Derenne et al., 1999, Bouzid et al., 2001). The prime purpose of HOBT is to determine the maximum safe operating temperature of PTFE-based gaskets in order to avoid excessive extrusion that causes blow-out. Due to the capacity of the test rig to measure bolt load relaxation, it was found suitable for conducting polymer flange relaxation tests.

The NPS 3 Class 150 slip-on bolted flanged joint is chosen for this test because its relatively weak bolting and high failure rate experienced in the refinery and chemical plant piping systems. The upper metallic flange is replaced with either HDPE or PVC flange of same size and class depend on the type test to be performed. The objective of the test is to measure the bolt load relaxation of the bolted flange due to polymeric flange creep; therefore, no gasket or fluid media was used during the test. The two selected polymeric flanges exhibit significant creep as observed in the previous work (Kanthabhabha Jeya and Bouzid, 2018).

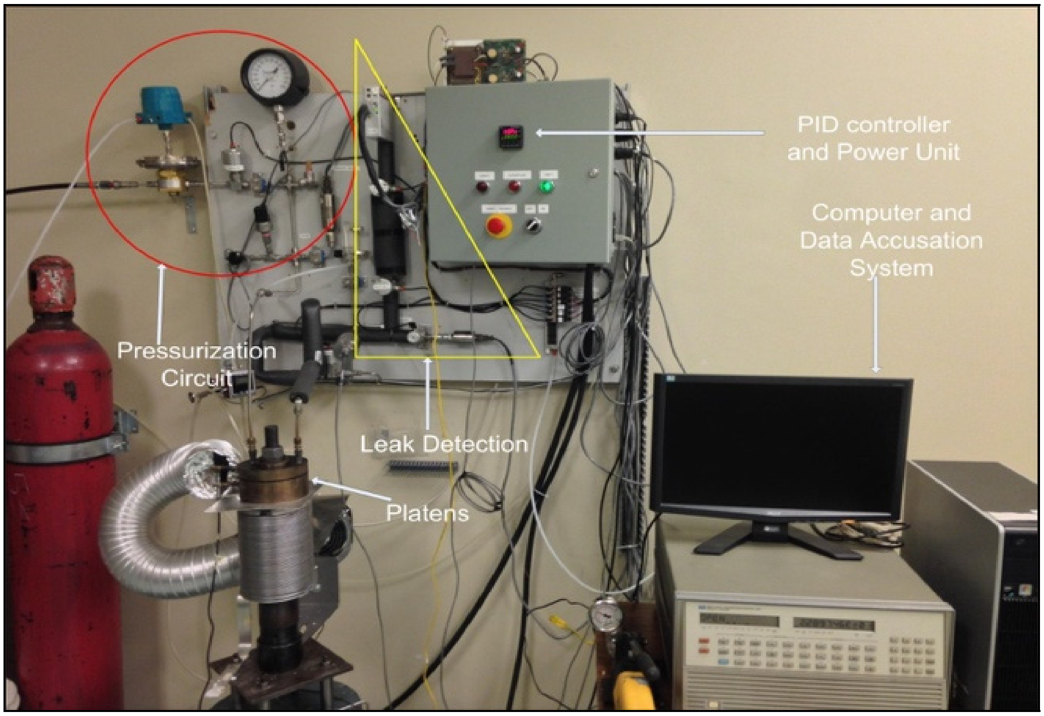

Figure 7. 1 Universal Gasket Rig

Figure 7. 2 HOBT fixture

7.3.3 Ring Specimen for Creep Analysis

Both HDPE and PVC ring samples utilized in the creep test are sliced from a 3-inch schedule 80 pipe of their respective materials. The Vertical Machining Center (VMC) is used for cutting ring samples with a nominal width of 12 mm as shown in see Figure 7.3. The maximum average width deviation of the ring specimens used in test is less than ± 0.1 mm.

Figure 7. 3 PVC ring sample

7.3.4 Bolted Flange Joint

In the market, there are two variants of readymade HDPE and PVC flanges available. They are PE4710 and PE3608 for HDPE material and solid and Van Stone type for PVC material. The difference between the two types of PVC flanges is that the solid flange is made of a single piece while the Van Stone flange is composed of two pieces. Likewise, the variation amongst the two type of HDPE flanges is mainly the material properties where PE4710 is superior than the PE3608. The 3-inch Schedule 80 solid slip-on flanges used in the experiment is manufactured for use in thermoplastic piping systems (Bouzid et al., 2001). Contrary to the PVC flange, HDPE flanges are generally stub flange type (Figure 7.4).

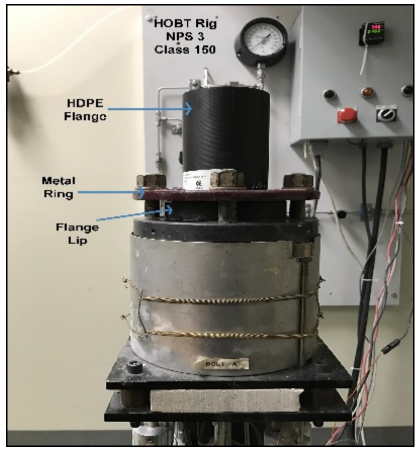

Figure 7. 4 HDPE stub flange

7.3.5 Procedure for creep test

The experimental test procedure commences with the application of heating to the target temperature; this step takes few hours for the temperature to stabilize and thereby getting rid of the deformation due to thermal expansion. Later, under steady state condition, the compressive load is applied to the polymer ring sample. The stress is applied manually on the sample by a hydraulic pump and a tensioner head. The tests last 4 to 5 days or until the secondary creep of the polymeric material is observed under the targeted test conditions. On a broader scale, a total of 24 short-term compressive creep tests were carried on the two selected polymer materials, among which 15 were conducted on HDPE material and nine on PVC material. The tests can be grouped under a constant temperature with different magnitude of compressive loads. Table 7.1 shows the details of the experiment test program.

Table 7. 1 Creep test program

High-Density Polyethylene					
No. of tests	Temperature (℃)	Compression load (MPa)			
4	23	7	10	14	21
4	40	7	10	14	17.5
4	50	7	10	14	21
3	60	7	10	14	
PolyVinyl Chloride					
No. of tests	Temperature (℃)	Compression load (MPa)			
3	25	10	25	30	
3	45	10	25	30	
3	60	10	25	30	

7.3.6 Bolted joint relaxation Test procedure

The flange relaxation test is conducted following two interchangeable procedures on the HOBT fixture. In the first test, the whole test fixture is heated up to the target temperature first. Then after the temperature is stabilized the initial bolt load is applied according to the instruction of polymer pipe system installation manual (Plastic Pipe Institute, 2013). In the second test, the bolts are initially tightened to the target load with the same crisscross tightening method as in the first experiment and then the whole assembly is heated up to the target temperature. The target temperature is only around 50°C, which is a representative of the hot weather environments. During the test, measurements of temperature, bolt load, flange or gasket displacement and time are displayed on the interactive LabVIEW interface every 10 seconds and all data are saved in a file at regular time intervals. The load applied on the two polymeric flanges is selected form their respective supplier manuals.

7.4 FINITE ELEMENT MODELING

7.4.1 Creep model

The creep properties in ANSYS are described under Rate-Dependent Plasticity, which additionally includes visco-plasticity material behavior. Rate-dependent plasticity defines the flow rule of materials, which depends on time, stress and temperature. Therefore the creep is defined as a material deforming under load over time which also be the function of neutron flux level and temperature (IPEX, 2016; Ansys Help, 2016). The von Mises stress is used for creep analysis, and the material is assumed to be isotropic.

7.4.2 Bolted flange joint model

The entire bolted flange joint assembly was modeled in Ansys for both polymer materials. This includes the top HPDE and PVC flanges with the bottom metallic flange part of the HOBT test fixture. The model also includes for metallic bolts and supports. The model used in FEA is created based on the physical polymeric flanges, which has been discussed previously. The material properties of polymers are obtained from the data sheet of manufacturer and the experimental creep test results. Due to CPU time consumption and taking advantage of symmetry, the static ANSYS analysis (Figure 7.5) of the two polymer flanges are represented by 1/8 portion models of the actual bolted joint.

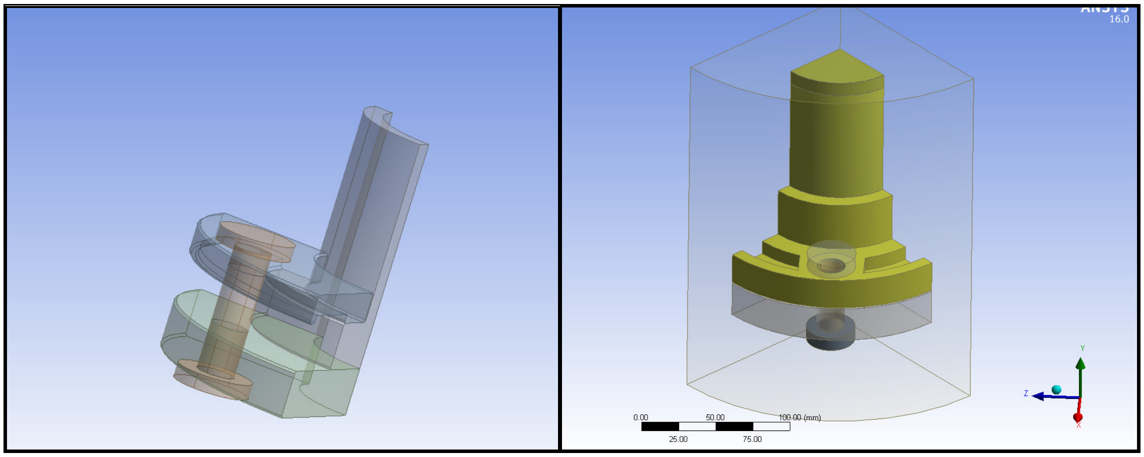

Figure 7. 5 1/8th static model of HDPE (left) and PVC (right)

7.5 Results and Discussion

7.5.1 Experimental Creep Analysis

The compressive creep behavior study of HDPE and PVC ring shaped specimens gave new insights into their material properties. The two polymers exhibit significant raise in creep strain with increase in applied compressive load and temperature. As expected out of the two influencing parameters, the magnitude of damage is highest with the highest combination of the two parameters.

Compared to PVC material, HDPE is severely vulnerable to compressive creep. The magnitude of creep strain increases with an increase in applied compressive load and applied temperature, where the highest combination of the two factors is most critical. All HDPE ring samples exhibit secondary creep phase under different test conditions. It is to be noted that the HDPE material reaches secondary creep within first 10 hours of creep test. The magnitude raise of creep strain is predominantly evident during the primary creep phase, where the growth of creep strain is almost proportional to the magnitude of applied compressive at higher temperatures. This is not so obvious with the creep strain curve at room temperature. The immense weakness of HDPE material to compressive creep is well-established by comparing the creep strain between HDPE and PVC at 10 MPa of load and at 60°C.

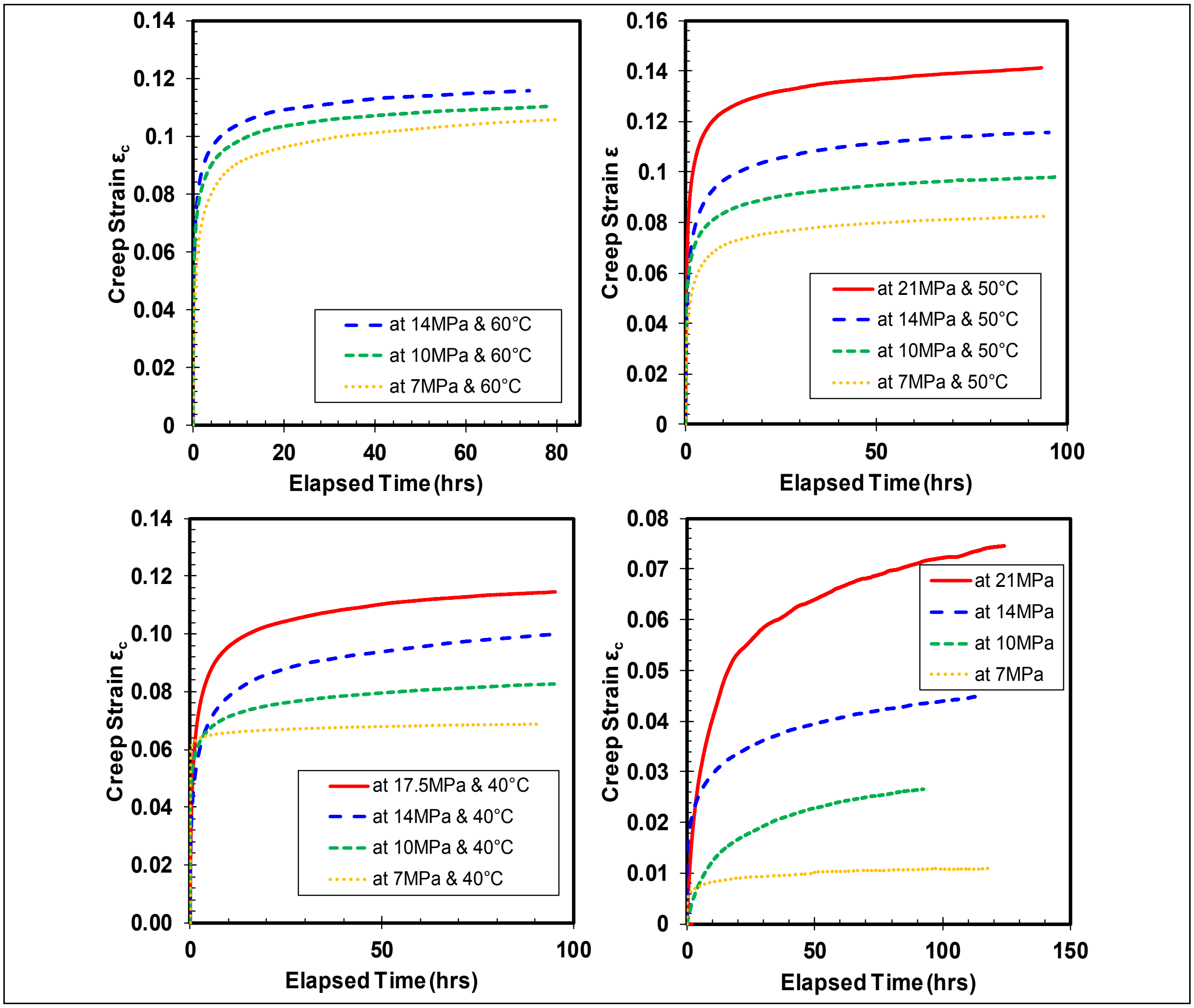

Figure 7. 6 Creep strain of HDPE under different loads at 60°C (top-left), at 50°C (top-right), at 40°C (bottom-left) and at 23°C (bottom-right)

With respect to PVC material, the maximum creep strain obtained under 30 MPa and 60°C (Figure 7.8 left) is 50 times higher than maximum creep strain at the same stress but at 25°C (Figure 7.8 bottom). Similarly, the creep curve between 60°C and 50°C (Figure 7.8 right) shows a 10 time rise in magnitude. It is important to that there is steep shift or tremendous growth in creep strain curve between 20 MPa and 30 MPa at higher temperatures. This clearly highlights the effect of temperature on the creep response while pointing out the susceptibility of the material under relatively high temperature. In addition, all the nine curves demonstrate only primary creep stage during 5 days of test.

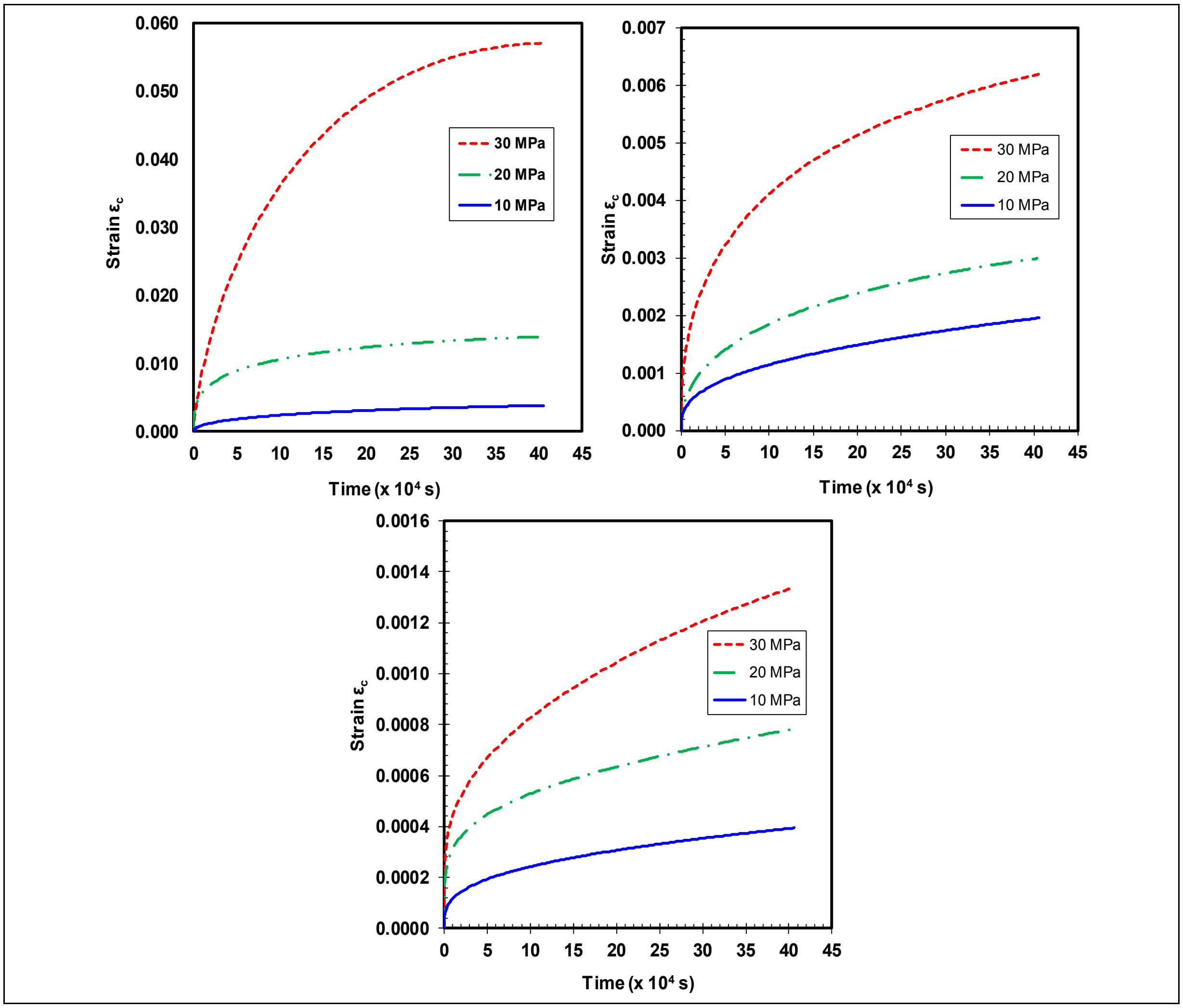

Figure 7. 7 Creep Strain of PVC under different loads at 60°C (left), at 45°C (right), at 25°C (bottom)

7.5.2 Creep Curve Fitting

The creep data obtained from polymer ring tests are analyzed and transformed to the mathematical creep model, which is then utilized in the FE analysis. The experimental creep strains are obtained by measuring the axial displacement of the ring samples over time at constant compressive load and temperature conditions. Later the constants of the most suitable creep model are determined from the experimental creep strain data using curve fitting. Among the various curve fitted models, the creep model that demonstrate highest accuracy (R^2) with physical creep test results is the Norton Bailey creep model (Equation 7.1) for both HDPE and PVC materials.

$$\varepsilon_{\mathrm{cr}} = A\sigma^{n}t^{m} \tag{7.1}$$

In Equation 7.1, ε_{cr} is the creep strain; A, n and m are constants that depends on temperature; t is the time and σ is the applied stress. The parameters A, n and m are replaced by functions f_1, f_2 and f_3 (Equation 7.2) such that:

$$\varepsilon_{\mathrm{cr}} = f_1(T)\sigma^{f_2(T)}t^{f_3(T)} \tag{7.2}$$

The corresponding coefficient of regression (R^2) values for the fitted curves at different temperature and loads conditions are close to 0.99, which is an acceptable range. All three functions (Table 7.2) of the Norton Bailey creep equation are of the form of second degree (Equation 7.3), in terms of temperature such that:

$$f\ (T) = aT^2 + bT + c \tag{7.3}$$

Table 7. 2 Creep Parameters

High-Density Polyethylene (HDPE)			
	a	b	c
f_1	4.21x10^{-06}	-3.79 x10^{-04}	8.41 x10^{-03}
f_2	-1.34 x10^{-03}	0.119	-2.07
f_3	-6.12 x10^{-06}	3.18 x10^{-05}	0.106
Polyvinyl Chloride (PVC)			
	a	b	c
f_1	-1.54 x10^{-03}	0.231	-1.89
f_2	2.72 x10^{-02}	-0.176	3.92
f_3	1.43 x10^{-05}	-4.54 x10^{-04}	0.334

Further to the above-mentioned creep model, modified time hardening creep model is utilized in ANSYS for both the materials as the FEA software does not provide for direct adaptation of Norton Bailey model. The modified time hardening creep model is simplified by combing the parameters related with temperature to the multipliable constant since the temperature is constant in each group. The modified time hardening is selected because it is the closest creep model to that of Norton Bailey.

7.5.3 Bolt Load Relaxation

As discussed earlier, the objective of this paper is to present a numerical model to predict the creep-relaxation of HDPE and PVC bolted flange joints. Three full-scale experimental test of NPS 3 Class 150 polymeric bolted flange joints were conducted on the bolted joint test rig. Two test on PVC bolted flange joints were performed to study the impact to two variations of the test procedure in which the application of heat before or after the bolt tightening. The test with the HDPE stub flange was carried to validate the numerical model adapted. Figures 7.8 and 7.9 show the decrease of bolt load of all four bolts over 5 days due to the creep-relaxation of the corresponding material flange. On average, the HDPE flange loses 35% of initial bolt load under ambient test conditions (Figure7.10) while the PVC flange loses 40% of primary bolt load at 50°C (Figure 7.11) operational temperature. As suspected, HDPE material is more vulnerable to creep than PVC, thereby losing considerably more joint tightness. In addition, it should be observed that the selected polymer flanges exhibit significant creep even without the use gasket. Hence, attention must be given to the design and maintenance protocol of these two polymer bolted flange joints, with an extra care for HDPE components. On comparing the numerical model (ANSYS Bolt Pretension) with experimental data, the difference observed between the two is less than 5%. This small difference is attributed to the uncertainties and drift of the measuring sensors and manual errors. Figures 7.11 and 7.12 shows that the order of heating has minimal effect on relaxation; moreover, the FE and experimental results correlates well under the test conditions. The curves superimpose each other indicating a good agreement between the two results with the FEA simulation slightly underestimates the relaxation. Creep data obtained with the ring samples on the UGR are under uniaxial constant load compression while the flange is a plate under a tri-axial stress state. There are parts of the flange that are under tension (Bouzid and Chabaan, 1997). The compressive load during the creep tests cannot be kept at absolute constant although the experimental test rig is equipped with an accumulator. A slow decrease of load in the range of 2 to 8 % is observed depending on the test conditions. This relaxation may have an influence on the creep constants, which in turn could underestimate the results.

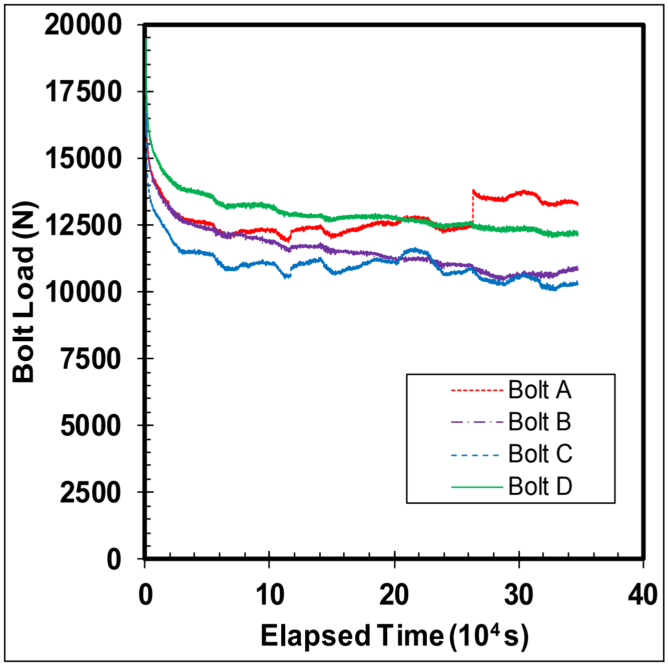

Figure 7. 8 Bolt load relaxation of HDPE flange

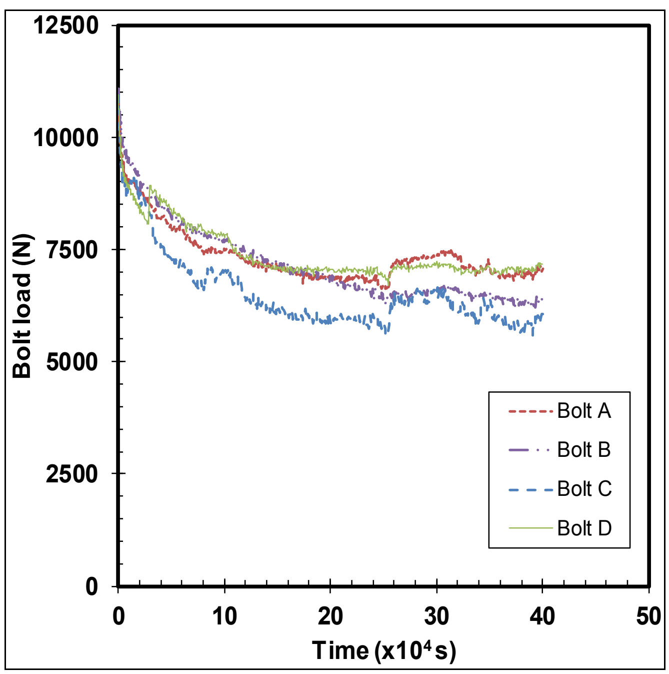

Figure 7. 9 Bolt load relaxation of PVC flange

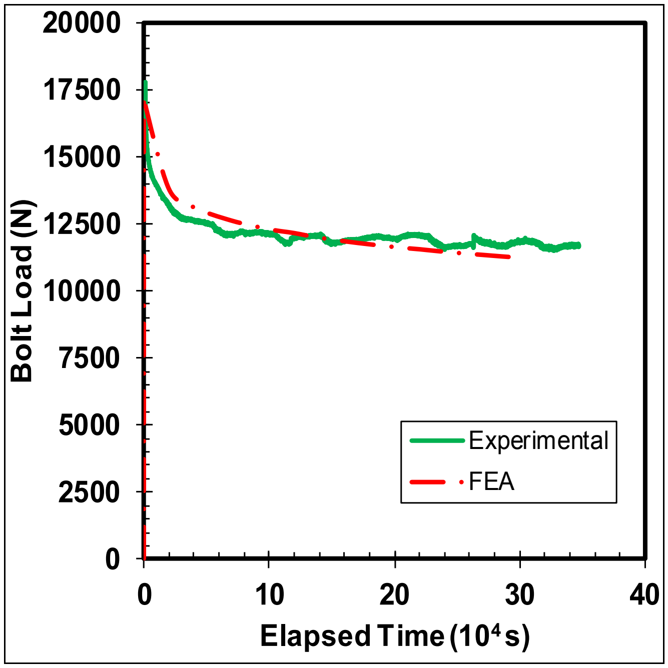

Figure 7. 10 Comparison of bolt load relaxation of HDPE

The load measurement fluctuations are another factor that may contribute to difference in the load relaxation value between FEA and the experimental results. The strain gages connected to the bolts are very much influenced by temperature fluctuation due to the ventilation system located near the test rig. The room temperature of the lab is controlled by a central air conditioning system, which is lowered during the night for energy savings.

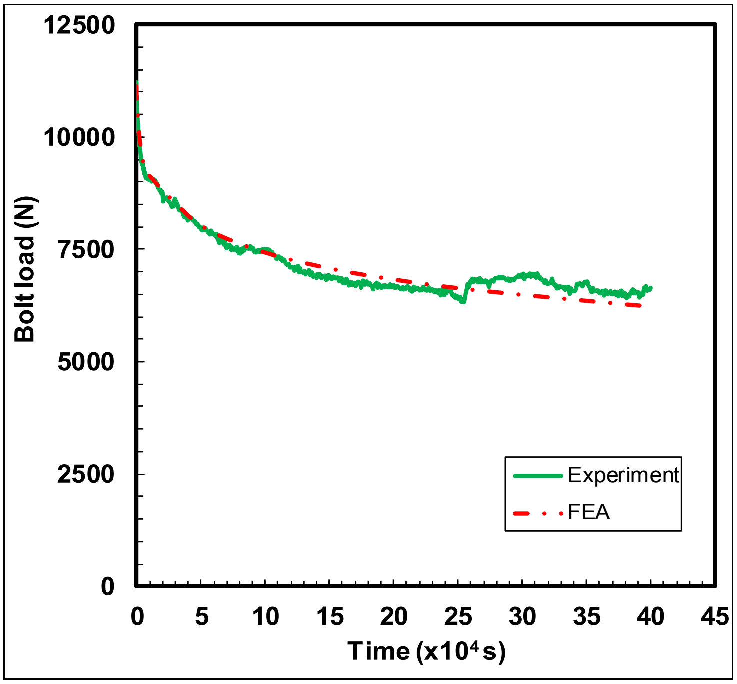

Figure 7. 11 Comparison of bolt load relaxation of PVC test 1 (heating first and then tightening)

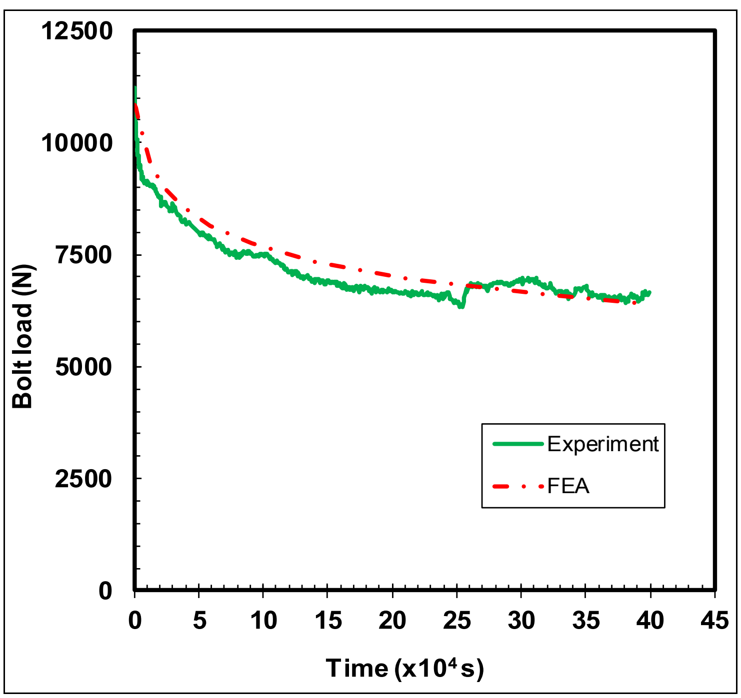

Figure 7. 12 Comparison of bolt load relaxation of PVC test 2 (tightening first and then heating)

7.6 Conclusion

The prime objective of this study is to evaluate the compressive creep behavior of HDPE and PVC materials and subsequently develop a creep model to predict polymeric flange relaxation over time. The fundamental creep tests are carried out on ring samples under compression using UTR fixture while the polymer flanges are tested on a NPS 3 Class 150 flange test bench to validate the adapted creep models. Unlike most loaded components of a piping system, polymeric flat face flanges are under compression rather than tension-compression. Based on the results, it is concluded that creep data obtained from compression tests on ring samples can be utilized to predict creep-relaxation behavior of selected polymeric products operated under compression load. Instead of using standard creep test samples, the rings directly cut from pipe material can be used for creep assessment. The time hardening model with interpolated parameters provides an accurate prediction of the bolt load relaxation over time of the two-selected polymer flanges.

CONCLUSION AND RECOMMENDATIONS

Since the early 1980's, the research on characterization of polymeric materials has been on a raise leading to a significant trend change in the selection of material for various applications. The monopoly of metallic materials has seen a shift in power with the raise of polymers, whose utilization ranges from simple children toys to load bearing aviation components (in terms of polymer/composites). Excellent corrosion resistance and protection against chemical attacks has made HDPE and PVC polymeric materials to be a natural replacement for rapidly ageing metallic materials in pressure vessel and piping applications. Hence, it is obvious that these materials has a lion's share among the polymer materials used in PVP domain. In addition to corrosion and chemical attack resistance, the selected two polymers are extremely lightweight and can be manufactured in rolls of 50 m pipes. This leads to easier installation and better maintenance of pipes, thereby reducing the operational cost. Combination of these factors paved the way for total dominance of HDPE and PVC polymeric materials in polymeric PVP domain.

In addition to the scientific criticism of low operational temperature and significant difference in material behavior under tension and compression, both HDPE and PVC PVP components lack historic data of operations. This drawback restricts in updating a dedicated design standard for polymeric PVP components. Moreover, the existing ASME and European polymeric PVP design standards are directly derived from the equivalents standards of their metallic counterparts. Hence, the questions raises on the acceptability of these standards in their existing form for different PVP applications. Here in the Static and Dynamic Sealing Laboratory, the research is focused on joints and sealing PVP components, hence this thesis is narrowed down to polymeric bolted flange joints and polymeric gaskets. The validation for selection of HDPE and PVC polymers is considerably detailed throughout this report. The characterization of two types of PTFE based and one of fiber based gaskets materials is also performed, as these gaskets are some of the commonly used gasket materials with HDPE and PVC bolted flange joints.

As a summary, this Ph.D. thesis is structured based on the methodology developed at the nascent stages of this research and it can facilitate the readers to comprehend the evolution of this research from introduction of the problem statement to the state of art research findings. The milestones of this research can be visualized in terms of research finding published or submitted in scientific journals, with the most important of them is the establishment of problem statement. As the title suggests, the thesis is predominantly deals with the creep and thermal ratcheting characterization of soft materials. The significant characteristic traits of selected soft materials are abridged in the following paragraphs.

An assessment of short-term compressive creep and thermal ratcheting behavior of ePTFE and vPTFE shows substantial thinning and deformation. The cumulative damage due to thermal ratcheting of ePTFE material gets saturated after 12^{th} thermal cycle, meaning only minute or fractional changes in thickness of the material under compression with further thermal ratcheting. However, vPTFE material exhibits continuous and significant cumulative damage even after 20 thermal cycles. The results indicate that ePTFE material is better resistant to thermal ratcheting than vPTFE material. In terms of CTE, both applied load and thermal ratcheting controls this material property. The probable reason cited for this cause is the densification of material under load and thermal cycling.

The second paper brought insights to the behavior of HDPE to compressive creep and thermal ratcheting phenomenon. It was found that as the applied magnitude of applied compressive stress increases the magnitude of creep strain increases. The material demonstrates a 7 and 28 % increase in the creep strain under change in magnitude (from lowest to highest) of temperature and applied load, respectively. HDPE shows significant vulnerability to thermal ratcheting in term of thinning and the magnitude of cumulative damage is influenced by the applied load, ratcheting temperature, pre-exposure creep time and number of thermal cycles. The impact of pre-exposure is visible in terms of hardening of material thereby decreasing thermal ratcheting damage. This phenomenon is consistent with other characterization tests too.

The consequence of thermal ratcheting on the compressive creep behavior of three type of gasket materials is presented in chapter 5. The two Teflon based materials, ePTFE and vPTFE, exhibits tremendous vulnerability to compressive creep as a consequence of thermal ratcheting. The magnitude of damage is higher for vPTFE than ePTFE; however, CNA demonstrates insignificant change in compressive creep behavior with the impact of thermal ratcheting. The creep modulus behavior of all three gaskets are similar to their corresponding creep strain response but in the inverse direction. It has to be highlighted that all three tested gasket material exhibits an increase of compressive creep strain with a decrease of material operational temperature.

The paper 4 elaborates the impact of thermal ratcheting on the creep response of HDPE material. The behavior of HDPE is similar to the PTFE gaskets, where the creep strain increases by 17% due to thermal ratcheting. The creep strain and modulus response of HDPE clearly indicates that thermal ratcheting amplifies the damage caused by compressive creep. In addition, the impact of lowering of material temperature on the intensification of creep damage is evident for HDPE. A relatively long-term creep test was performed on HDPE sample to analyze the effect of thermal ratcheting on compressive creep. The results point to a decrease in the magnitude of cumulative damage with increase in pre-exposure creep. However, the consequence of ratcheting damage is not nullified and there is still augmentation of cumulative damage at the end of 30 thermal cycles after 45 days of creep pre-exposure. This means that the damage due to thermal ratcheting did not saturate even after severe tested conditions. The results show a 2.4% increase in creep strain due to thermal ratcheting after 45 days of pre-exposure creep.

The chapter 7 deals with the short-term experimental and numerically creep-relaxation behavior of HDPE and PVC bolted flange joints. The compressive creep behavior of HDPE and PVC respects Norton-Bailey creep law. Extensive amount of creep tests were carried out under different temperature and compressive load sets. Subsequent comparison with a numerical simulation, using ANSYS, provided less than 5% error in estimating the bolt load loss due to creep-relaxation of the materials.

Overall, the thesis points out that blindfolded utilization of design standards based on existing metallic design standards is not sufficient, especially with respect to the selected materials as they demonstrate important difference under basic mechanical load and temperature related properties. Even though, the impact of thermal ratcheting decreases with an increase in pre-exposure creep, it is not completely negligible and moreover, thermal ratcheting causes severe damage during the initial period of operation.

The future works of this research can be directed towards the thermal ratcheting behavior of soft materials under traction or multi-axial loading to cover PVP applications other than bolted flange joints. Since the difference in material properties of the selected soft materials under traction and compression is well noted (Zhang and Moore, 1997; Bezergui and Payne, 1985), it is important to experimentally study the impact of thermal ratcheting under traction and multi-axial loadings. These results will lead to better understating of behavior of soft materials for long-term applications.

Performing full-scale creep-thermal ratcheting tests on polymeric bolted flange joints is a must to update the bolted flange design standard. For this, alteration of the current HOBT test bench is needed to replicate a proper thermal ratcheting test. Subsequently, developing a numerical model to predict the thermal ratcheting response of polymeric bolted flanges would be an added value.

Finally, a study on cryogenic ratcheting of polymer materials can be performed. This type of tests are important because many polymer materials have low operational cryogenic temperatures and there is extremely limited information on the characterization of polymer materials under cryogenic temperature. Much similar to thermal ratcheting, the research findings on the cryogenic ratcheting of polymers or soft materials is extremely rare or close to none. As the application of polymer PVP components are growing in colder regions of the world, it is necessary to look into the behavior of these materials and their mechanical properties in cryogenic temperatures.

The mentioned future work recommendations may not be limited to only the selected soft materials, large sections of polymer-composites are suspected to be susceptible to thermal and cryogenic ratcheting. The polymer-composite applications vary from building (concrete-HDPE composite) to future aviation parts (glass fiber-HDPE composite), where the materials are subjected to variety of temperature and mechanical loadings. Hence, thermal and cryogenic ratcheting characterization is an essential.

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