

Final Year Project
Thesis Title
Fabrication and Characterization of Xydar® G-930
(Glass-Reinforced molding grade)



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Dedication

“This work is dedicated

To our beloved parents and

Our kind teachers”

ACKNOWLEDGEMENTS

All commendation is to Allah Almighty who made us fit for performing work. All the information, accomplishments, encounters and tries are because of his Generosity. The productive achievement of this task is owed to all those who collaborated, coordinated, gave facilities and assets and aided in the most ideal manner they could.

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ABSTRACT

Polymer industry is one of the prestigious and rapidly developing industrial sector in Pakistan as well as in the world. Comparing to other polymers of its class, LCPs are well known for its outrageous behavior and it has many applications in biological, sensing, electronics, automotive industries. That's what our moto was to portray LC polymers.

We imported the pellets of Liquid Crystal Polymer (Xydar® G-930) from Malaysia for nanoindentation experimentation. Before whatever else, manufacturing of LCP sheet was practiced by utilizing thermal press machine (Heat sensitive machine utilized for the transformation of polymer pellets to sheet having clear smooth surface). LC polymers are a moderately new class of high temperature polymers that have pulled in a lot of intrigue, both scholastically and mechanically and it can supplant ceramics, metals, composites and other plastics as a result of its exceptional quality at outrageous temperatures and protection from practically all chemicals, weathering, radiation and burning.

The objective of most of nanoindentation tests is to extricate elastic modulus and hardness of the sample material from load-displacement estimations.

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NOMENCLATURE

LCP	Liquid Crystal Polymer
LC	Liquid Crystals
PVC	Polyvinyl Chloride
ABS	Acrylonitrile butadiene styrene
FTIR	Fourier-transform infrared spectroscopy
SEM	Scanning electron microscope
TGA	Thermogravimetric analysis
NMR	Nuclear magnetic resonance spectroscopy
OB	Oxygen Barrier
MBBA	N-(4-Methoxybenzylidene)-4-butylaniline
PMMA	Polymethyl methacrylate
PC	Polycarbonate
PS	Polystyrene
UHMWPE	Ultra-high-molecular-weight polyethylene
LDPE	Low-density polyethylene
PEEK	Polyether ether ketone
FES	Finite element Simulation
HDPE	High-density polyethylene
PET	Polyethylene terephthalate
EVOH	Ethylene vinyl alcohol
PVDC	Polyvinylidene chloride

Chapter 1

INTRODUCTION

1.1 OVERVIEW

LCPs are a form of substance that exists between a solid and a liquid composition. Unlike other polymers such as thermoplastics, which include polyesters and other aromatic liquid crystal polymers, LCPs have a characteristic crystalline structured structure [1]. This chapter addresses the study and development of LCPs. LCPs offer benefits over other polymers, ceramics, metals, and materials. Its many varieties and which type does our Xydar® G-930 belong. This chapter will also feature the issue description, the aims of our research project, and the applications of our research-based project.

1.2 LIQUID CRYSTAL

There are three distinct states of matter: hard, liquids, and gas. Change in these three states may be caused by changes in the molecules heat. The warmer, the more unpredictable the molecules are, and hence the less order they have. The transition from a solid to a liquid and then to a gas is triggered by increasing temperature [3]. It is possible that there are atoms that do not fall into those 3 types above. Solid-to-liquid transitions in several participating more than one transition, confirming the existence of intermediate phases [2].

In terms of mechanical, optical, and structural properties, the new phases fall somewhere in between a crystalline solid and an equal-sized isotropic liquids. Liquid crystalline phases are the name given to these phases [4] [5]. An established state of matter, the polycrystalline phase, lies in between crystallites and isotropic liquids. Reinitzer discovered that cholesteryl benzoate had two distinct melting points in 1888, and the subject of particles was born. Afterward, Reinitzer noticed that the solid had transformed into a hazy liquid after raising its temp. The mixture became a translucent liquid at a warmer pressure [6].

An intermediate phase between solid and liquid states of cholesteryl benzoate was discovered in this study. In the ideal body state, a long-range orientational order and maybe a partial location regularity characterize the liquid crystal structure (state). Molds (molecules) in liquid crystals tend to point in one direction, which is known as the director (n). Molecules in the solid phase, on the other hand, are well structured and have little temporal flexibility. In liquid condition, there is no natural order to the structures. The normal isotropic order of the LCPs state is between the conventional solid and liquid phases and this is the basis of the phrase mesogenic state, commonly used relative with liquid crystal state. It is depicted in Figure (1.1) how molecules are aligned in each of these stages.

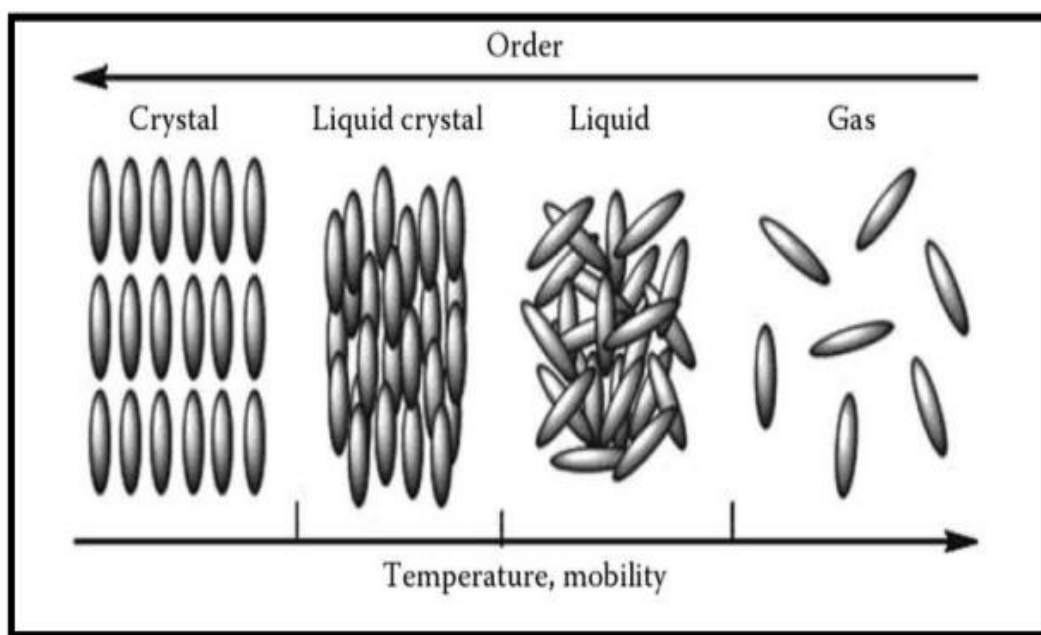


Figure: 1.1 Alignment of the molecules for solid, liquid crystal, liquid and gas phases.

"To quantify just how much order is present in a material, an order parameter (S) is defined. Traditionally, the order parameter is given as follows".

$$S = \left\langle \frac{3\cos^2\theta - 1}{2} \right\rangle$$

The angle recurring is referred to as a molecules. The total of the molecules in the sample is shown in the quotes. The order parameter is zero in an isotropic liquid because the typical angle factor is 0. The most of is one for a perfect crystal. As a result of kinetic molecular motion, the order parameter of a liquid crystal varies within 0.3 and 0.9, with the true number determined by heat.figure (1.2)

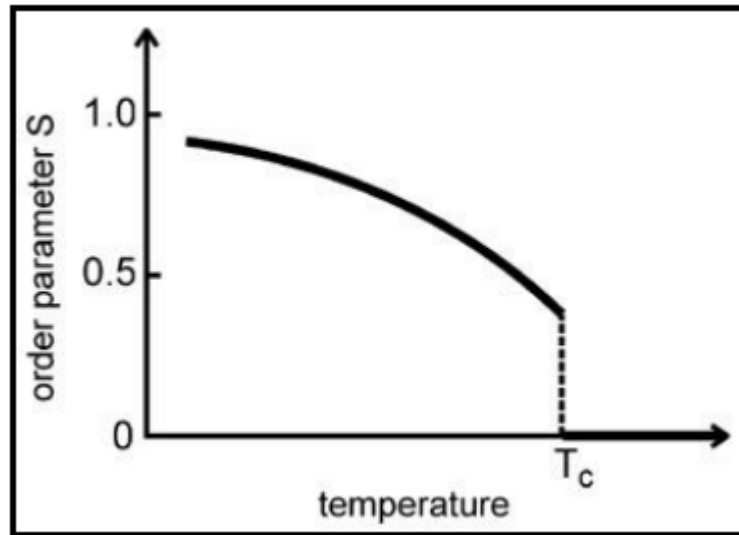


Figure: 1.2 Order parameter S vs temperature T_c [2]

Where, T_c is clearing temperature

Anisotropy is a result of the liquid crystal molecules tendency to point in the right path. The phrase refers to the fact that a qualities of a material change depending on which direction it is studied in. The optical properties of liquid crystals are due to their anisotropy [7]. For the most part, properties of liquid crystal materials may be compared. In addition to the rigidity of the midline and strong dipoles, rod-like molecular structures are among the other characteristics. Several different kinds of entities exhibit monoclinic characteristics. Certain chemical compounds, as well as micellar solutions of surfactants and polymers, and a wide range of biomolecules, are proven to be solid crystals [8].

1.3 CLASSIFICATION OF LIQUID CRYSTALS:

There are several ways to classify LCs, including low molar mass (monomeric and oligomeric) and high molar mass (polymeric) LCs; how the liquid crystalline phase was obtained, such as by adding solvent (lyotropic) or by varying the temperature (thermotropic); the nature of the constituent molecules (organic, inorganic, and organometallic); and the geometrical shape of the molecules (rod-loop) (nematic, smectic, columnar, helical, B phases, etc.). Figure shows the categorization of LCs (1.3).

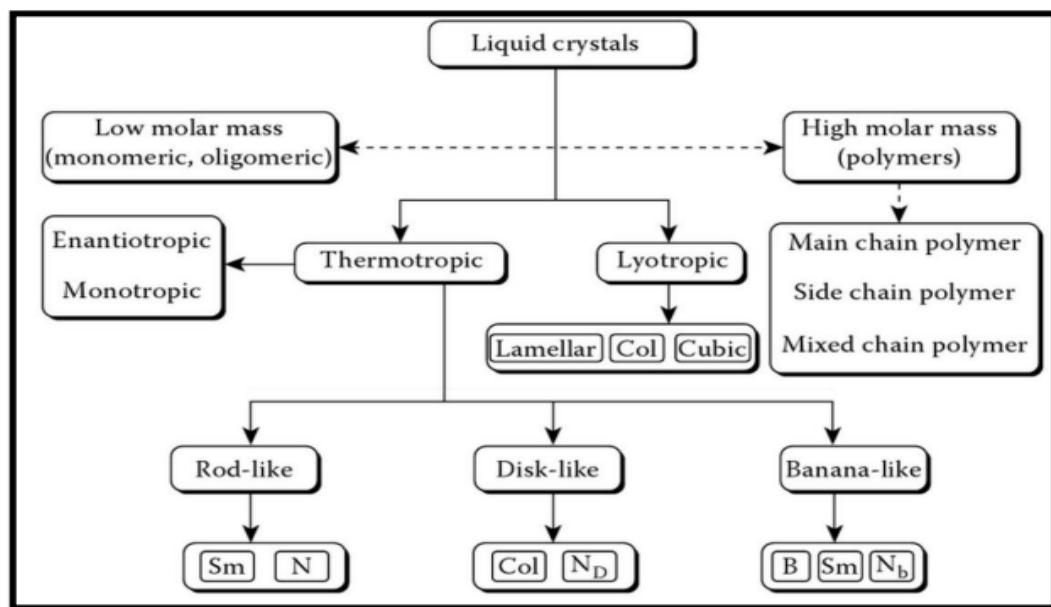


Figure: 1.3 Classification of LCs [9]

1.3.1 PHASES OF LC:

LCs generally exhibit two types of phases i.e Nematic and Smectic.

1.3.1.1 NEMATIC PHASES:

The nematic is one of the most common LC states. The Greek word nema, that means "knot," is the root of the name monoclinic. There is no positional order in a nematic phase of organic molecules, rather the personality to demonstrate long range orientational order with their long axes, generally parallel [10]. Consequently, the atoms move freely and their centres of mass are scattered as in a liquid, but their long-range directional order is maintained. In the majority of nematics, one axis is longer and preferred, while the other two are equally. Flexure nematics, which means that in addition to orienting along their long axis, some liquid crystals are also themes in nature, tend to be more common. [11].

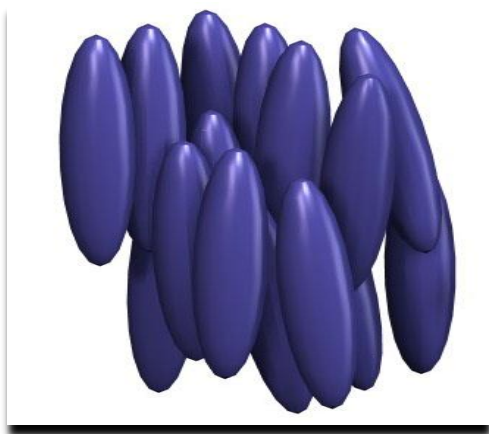


Figure: 1.4 Alignment in a nematic phase

1.3.1.2 SEMECTIC PHASES:

The smectic phases at low temperatures create distinctive layers that may glide over one another in a way akin to that of soap. The term "smectic" stems from the Latin word "smecticus", signifies cleansing, or having soap like characteristics. The smectics are therefore positionally organized in one direction. In the Smectic A phase, the molecules are orientated along the layer normal, whereas in the Smectic C phase they are tilted away from the layer normal. These phases are liquid-like inside the layers. There are numerous distinct smectic phases, all defined by varying kinds and degrees of positional and orientational order. [5][12]

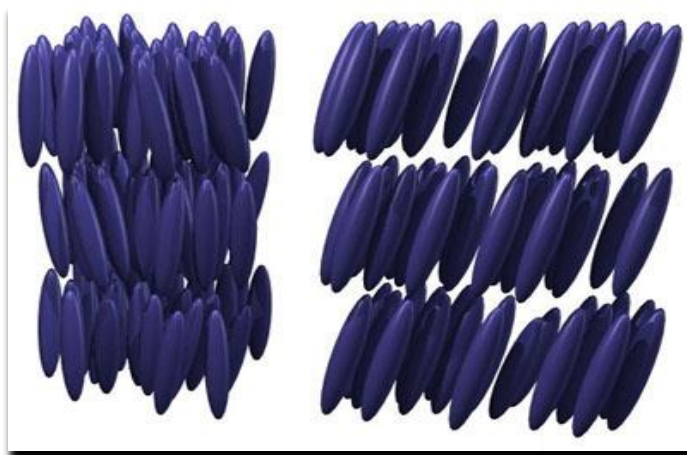


Figure: 1.5 Alignment in the smectic phases

The LCPs exhibiting significant attention nowadays notably in the domain of thermoplastics and synthetic liquid crystal polymers.

Thermoplastic polymers, which also comprises LCPs, which may be examine in everyday life use and are known by some other polymers as PVC, PTFE, PE, PP, PS, Kevlar or Xydar. LCPs preserve their value in crystalline nature in stable system. Partially ordered structure exhibits qualities of LCPs, such features are assortment of utilizations of LCPs. Today, LCPs demonstrated variety in many fields including laser beam deflectors in autos and aeronautical components of food containers. LCP thermoplastics especially may be organic molecules based on polyesters and can be aromatic molecules like polystyrene [1]. Mostly thermoplastic polymers occur as a main chain, side chain or mixed as stated above.

LCPs as a thermoplastic performed quite well in severe environment. They have strong thermal, chemical and electrical resistance. Unlike other polymers such as nylon, PVC and ABS, LCPs display a significant degree of anisotropy [13]. It indicates they have great stiffness, strength and thermal expansion. LCPs may be simply injection molded or they can transform pellets into sheets by employing thermal presser. They have high melt temperature and can handle high pressure. They are resistant to hydrolysis, acids and base, aromates, alcohols and hydrocarbons. They have excellent operating temperature. That's why, they discovered variety in its uses. Although LCPs have numerous benefits as well as downsides and we have to be mindful of to all of them. The anisotropic nature of the polymer may create weakness. They are pricey as well.

DuPont (scientist of United States) established the industrial production of high strength and high modulus fibers of the Kevlar (LCP) brand (annual output of over 10000 tons), while the Dutch business Akzo started steps to manufacture of the Tvaron fiber (LCP) with approximately the same output as Kevlar. Same investigations were undertaken at the Khimvolokno Research Institute (Mytishchi). Where a number of Soviet scientists under the leadership of some other qualified scientists, studied that the high strength fibers were obtained from the lyotropic solutions of aromatic copolyimides [14]. It appears that the growth in the manufacturing of this kind of high modulus fibers played the most part in the further developing of this kind of thermotropic polymers that contain mesogenic groups in main and side chains of macromolecules.

All above stated factors pushed the scientists to create new ultrahigh strength and high-modulus fibers self-reinforced or glass reinforced molded polymers such as Vectra and Xydar have great mechanical qualities. These are high performance compounds. These materials find a range of applications in bigger components for the aircraft, car industry and other civil and military sectors. Furthermore, portions of millions of components of radio, audio, and video facilities [15].

In late of 2007, Symitomo, a chemical business of Japan, reported a special increase of the production of LCPS for electronics, communications and telecommunications sectors with an annual output of 9200 tons and it is further expanding to create yearly output to 14 000 tons [16].

Now a days the deep study on liquid crystal polymers with the use of advanced specialized techniques including FTIR, tribometer, SEM, TGA, NMR spectroscopy, X-ray diffraction, tunneling microscopy and nano indenter is undergoing. Above said all the LCPs are created. Now there is only more study to increase the characteristics of these LCPs is going undertaken.

Xydar® G-930 an LCP is a glass reinforced injection molded polymer that is created specifically for the electric and electronic industry. Its tiny layer can compete thick layer of other polymers since it has super high-strength, this quality is special of xydar. It offers negligible warpage in molding and good weld line strength. It is offered in black or white pellets form which is converted into a sheet by employing pressing equipment. It has exceptional resistance against chemical, weathering and radiations. It is Glass Fiber, 30 percent Filler by Weight. It found its usage in bobbins, tubing, electrical and electronics devices and numerous industrial applications. Properties are shown in the table below

Table 1: Properties highlights of Xydar

Density (g/cm ³)	1.6	Drying temp. (°C)	149
Tensile modulus (MPa)	15900	Drying time (hr)	6 to 8
Elongation (%)	1.6	Processing temp. (°C)	320 to 360
Flexural modulus (MPa)	13400	Mold temp. (°C)	60 to 93
Tensile strength (MPa)	135		

1.4 NATURE OF LCP:

What Lehmann, Reinitzer, and others had identified another state of problem somewhere near to a solid and a liquid. The liquid had small segments which had all the marks of being crystalline in nature but were suspended in a liquid state. Likewise, not at all like ordinary unmixed substances which soften precisely at specified temperature are liquid crystals. The liquid crystal in associated era of state was consequently named a mesophase.

The individual atoms in the substance suited for framing a mesophase, or liquid crystals, are mesogens. These atoms are typically microscopic to ordinary size natural particles and arrange themselves in different degrees of affiliation. For instance, a mesophase structure may also can be observed in gelatin material [17].

Further developments in the analysis of liquid crystal polymers enhanced their unique feature. Two main sorts of liquid crystals were found: those that came to existence by heating and those that came about due to the dissociation in a solvent. Liquid crystals that might be created by solvation are termed as lyotropic; those of the kind that Reinitzer and Lehmann identified were because of heat and are named thermotropic liquid crystals [18].

In this manner, when examining mesophases, it maybe progressively fitting and for simplicity of understanding to regard the altering states or periods of the mesophase, as material advances from solid to liquid crystal and then to liquid. Both thermotropic and lyotropic mesophase types have unique relevance in industry and research.

Making up the mesophase involves the small atoms within it. While not all atoms are suited for framing mesophases, the potential for mesogenic particle types are practically endless.

Along these lines, mesogens often have some amount of non-consistency in their structure and network of molecules. This non-balanced nature shows that the atoms have some amount of directionality and that sure of their attributes or qualities vary as indicated by this directionality. This directionality attribute is termed anisotropy. For instance, the principal produced liquid crystal particle, is uneven and shows anisotropic features in its liquid crystal stage. On the other hand, atoms that are perfectly balanced have properties that do not depend on any directionality; they are termed isotropic [19]. These descriptions of isotropy and anisotropy may be extended out to higher sorts of problem to study solids, liquids, and gases. The amount of isotropic and anisotropic nature of the mesophase consequently regulates itself.

While liquid crystal polymer demonstrate unique behavior differing from solids and liquids, they also show group character. Basically, the array of liquid crystal have different arrangement of their mesophase atoms which contribute to their smaller scale crystalline structures. Aside from thermodynamics effects, the behavior of the liquid crystal polymer is along these lines controlled by the example of mesogen arrangement [20].

1.5 PROBLEM STATEMENT:

LCPs are capturing the market faster than the polymers like LDPE UHMWPE PEEK and others by the past few years due to their high strength and stiffness, it has ability to replace ceramics, metallic materials and other plastics because these polymers exhibit higher resistance to corrosion, chemicals, high temperature and weathering and also there is no nano indentation analysis done before on Xydar® G-930. So, we decided to choose LCP Xydar® G-930 for nano indentation analysis. In this analysis, formation and characterization of Xydar® G-930 (glass reinforced injection molding product) and its properties comparison with other polymers for various purposes is included.

1.6 OBJECTIVES:

The choice of research bases project is made with the following goals described below:

1. Formation of LCP sheet from Xydar® G-930 pellets by using thermal presser
2. Characterization of its mechanical properties using nano indenter
3. Compare its properties with other polymers that are not liquid crystal based
4. Selection of the best one for using in cable ropes and medical applications.
5. Replacement of other ceramics, metals and polymers with LCPs. As these are compatible with all other materials

1.7 APPLICATIONS OF LCPs:

There are several uses for liquid crystals in industry and medicine due to their electro-optical, magneto-optical, auto dimming, and crystal form capabilities. It is used in a wide range of fields such as LCD displays as well as health and aesthetic industries. Dials, clocks, and other small electronic devices are the most common applications of liquid crystal displays at the moment [21].

1. As it has high strength and its thin film is comparable with thick films of other polymers. It can replace the other polymers as xydar is versatile in nature.
2. Tubes mad from PVC, polyethylene, and polycarbonate can be replaced by LCP tubes.
3. It can be used in thin film chip circuit board.
4. Tubes made from this polymer used for cryogenic fluid, harmful chemicals and other applications.
5. It can be used as a rigid tubing for automotive fluid system
6. This LCP is used to build barriers for different gas barriers.
7. It founds its applications in blow molding like for bottles.
8. On combining with other polymers gives high strength (50% strength increment).
9. Used as a thin-films in the formation of circuit board.
10. It has high cost but more benefits as well

Chapter 2

LITERATURE REVIEW

2.1 OVERVIEW:

This chapter offers a literature review of theoretical and experimental knowledge, and a review of previous studies linked to our research. A quick review of nano indentation results from several research articles on various polymers is done in this chapter. Surface properties of materials, which are strongly dependent on mechanical properties of materials, have taken the attraction of researchers and experts in the field of mechanical and scientific sciences.

Nano indentation is simply a depth-detecting and instrumented indentation machine. Nano indentation is a popular instrument due to its unique qualities, such as its ability to record small loads and displacements with high accuracy, and it provides an analytical model to evaluate mechanical properties (hardness, modulus of elasticity, and so on) for materials. Hardness is defined as the measure of resistance to deformation when a load is applied by indentation or any other method. Certain materials have more hardness than others.

Modulus is the measure of resistance to elastic deformation when a load or tension is applied. It may be defined as the slope of the stress-strain curve. Some materials have a greater elastic modulus than others.

As we all know, micro indentation has been a highly recommended method in recent years for measuring mechanical properties, but it has also been found to have a significant influence on the experimental examination of material physics. During nano indentation, the phase change may also be detected [22].

A study of hardness, plasticity index, and modulus on four unique polymers, PMMA, PC, PS, and UHMWPE, was published in a scientific journal in January 1998. The impact of penetration depth and maximum load applied on modulus and hardness of these four polymeric materials is thoroughly discussed in this research. According to this study, the depth range is 10mm to 10m, with a maximum depth of 10m. The applied load is 300nN. This study also looks at the creeping phenomena [23]. This study shows that the penetration displacement, maximum force exerted on the hardness, modulus of elasticity, and plasticity index all have an effect.

The displacement Vs load curves are originally generated by maintaining the loading and unloading rates constant at 300N/s and the displacement range between 510 and 560 nm. The temperature was at 20 degrees Celsius, which is considered room temperature. Because UHMWPE is a crystalline polymer, the findings reveal that it is softer than any other amorphous polymers. As the penetration depth increases, so does the load and the distance covered by the tip, demonstrating the softer nature of UHMWPE because applying less weight covers greater distance within the polymer, demonstrating the softer nature of the polymer. PMMA and PS, on the other hand, are tougher in nature because when greater stress

is given, the needle covers less distance, indicating the harder nature of these two polymers.

If we look at the displacement and hardness data, we can see that in the instance of UHMWPE, the hardness of the polymer does not vary as the distance traversed by the needle changes. This demonstrates the softer nature of polymer. Other polymers, on the other hand, exhibit a substantial change in hardness as the displacement covered by the tip of the needle increases. In the case of PC, the hardness decreases at initially as the displacement increases, but after a while it stays constant. The same phenomenon was found with the other two polymers, which are harder and amorphous.

For displacement and modulus data, the modulus for UHMWPE is somewhat different and almost the same as the displacement applied increases. All other polymers initially drop and then virtually stay constant, demonstrating the tougher nature of polymers.

It is established that approaching the surface, the hardness and elastic modulus of the four polymeric solids increase. This is due to changes in the physical and chemical characteristics of the polymers during assembly. These differences in findings might be attributed to human error or defects in the indentation needle tip. In the case of PMMA, more precise findings are achieved, and no indentation effects seem to be seen.

The PS surface is the hardest, while the UHMWPE surface is the softest. PMMA and PS have similar modulus and hardness. The author was pleased with the findings and concluded that this is the most practical way for estimating the

mechanical characteristics of a polymeric surface. The findings revealed that the mechanical parameters estimated via the indentation process may be influenced by the testing approach and the indentation tip used. When high strain rates and imperfection of the indenter of a physical system are present, extreme caution is necessary. For reliable findings, there must be a process to eliminate the flaws and dispersed nature of polymers [23].

In 2013 micro indentation response on PEEK was done. Indentation is extremely simple and nondestructive technique to determine the mechanical properties of polymeric materials. In this study article load-displacement, elastic modulus or elasticity and hardness data is acquired as a response of nano indentation and presented as a function of contact depth of the tip of nano indenter. One of the primary results that can be gained from the study of this research is that nano indentation of the polymeric materials displays excellent surface hardening effect which is depending upon the contact circumstances like contact depth. As PEEK is the semi crystalline polymer in nature and has the features of bimodal nano indentation because of the occurrence of solid crystalline thin layer inside its non-solid amorphous phase.

The second main finding was the characteristics of bimodal is validated by the micro indentation response such load displacement, hardness and modulus data. Bimodal character is essentially having two peaks in its findings, and in the results it can be easily observed that PEEK has two peaks in its nano indentation response. It can also be seen that the polymers which have semi crystalline nature displays

distinct variations in their mechanical characteristics like hardness and modulus with the rise of displacement and penetration depth during micro indentation study. According to the author, nano indentation method is very easy and practical approach to discover the mechanical properties of any polymeric materials and it is also convenient way to determine the degree of crystallinity of any polymer which is semi crystalline in nature. This micro indentation technique has established its usefulness to give easy approaches to analyze morphological features of polymers and semi polymer surfaces [24].

In late 2013, the investigation of micro indentation response for softer polymer like silicon rubber was done. Silicon rubber is isotropic and generally occurs in hyper-elastic nature which shows minimal viscous behavior. Viscoelastic and adhesion effects were reported in micro indentation technology. The viscoelastic and adhesion effects may be determined by comparing the data acquired by numerical model with the force displacement data obtained by nano indentation. The comparison between force displacement data and numerical data includes finite parameters that are suitable to evaluate the adhesion effects.

Viscoelastic and adhesion model both can best describe the viscoelastic behavior of the silicon rubber in nano indentation. This investigation indicates that the silicon rubber exhibits surface adhesion. A varied testing method can be needed in nano indentation experiment for silicon rubber test. Therefore, it is vital to build inverse process which needs to be improved to find new factors for flexible circumstances in nano indentation technology.

It is determined that the findings of the numerical model are significantly closer to the experimental force-displacement data in the unloading section rather than that of the loading and holding part of micro indentation response. There are several causes that might be the cause of this outcome including thermal drift, cracks in the surface of the polymer during the indentation process or thermal influences on the surface characteristics of material. The inaccurate estimations of the adhesion and viscoelastic effects in the unloading and loading or holding phases may be a solid cause for this outcome.

The author is satisfied with the reaction of nano indentation technology. But in case of discovering response of adhesion and viscoelastic effects for softer polymers like silicon rubber there are some adjustments necessary in inverse mode of micro indentation to obtain best possible and accurate findings. This is the finest and convenient model for mechanical properties without any alterations [25].

In 2011, a study was undertaken by J.A. Bencomo-Cisneros a, A. Tejada-Ochoato and some other researchers to describe the intrinsic behavior of single Kevlar-29 filaments under static loading, and nano indentation experiments, to evaluate their cross-sectional mechanical characteristics. The poly(p-phenylene terephthalamide) (PPTA) fibers investigated in this study were Kevlar-29, Kevlar is a commercial brand for aramid fibers and offer a unique combination of high strength, high modulus, toughness and thermal stability compared to many other organic fibers [26]. Kevlar fibers were designed for demanding industrial and advanced technology uses, including as ballistic protective armor, helicopter blades, pneumatic reinforcement, and sports goods.

The longitudinal behavior of single fibers had been investigated for a long time, including Kevlar fibers [27] [28], it was required to research the fiber reaction to mechanical stresses in other directions, such as the cross-section. The results of this study showed that the Young's modulus of Kevlar-29 is about half that of Kevlar-49 [29] [30]. Therefore it is not surprising that the former fiber, which was the object of this study, was found to be less ordered than the latter due to poor alignment of the molecular morphology induced during manufacture. The standard deviation displayed with the data suggests that there is substantial diversity in the mechanical characteristics of these fibers. Results reveal that the stress-strain curves of Kevlar-29 fibers remain essentially linear until failure as shown in following figure

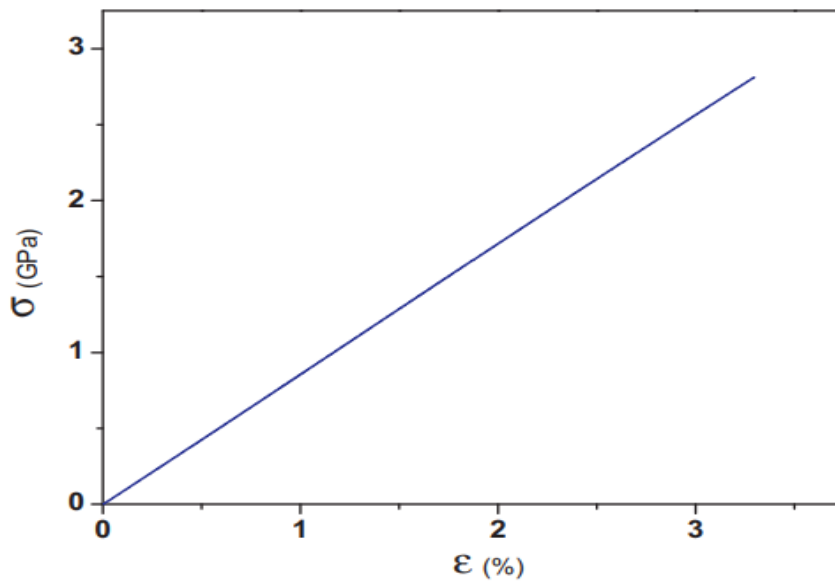


Fig: 2.1 Stress–strain curve of a Kevlar-29

The materials examined showed near flawless tension behavior. These had a good toughness, but there was a lot of variation in those characteristics. A severe cracking of the object could be seen in the fracture morphology under quasi-static loading conditions. When measured by impact testing, the elastic modulus of the fibers was four times lower in the transverse direction than when measured by tensile testing in the longitudinal direction. It was because of the great anisotropy of the fibers that were generated well during work process that the longitudinal and radial directions differed [31].

April 2019, the examination of influence of experimental circumstances on nano indentation response for LDPE was concluded by the professor Dr. Tanveer Iqbal and other faculty members. To study nano scale mechanical characteristics of any polymer may simply be determined by the technique called as nano indentation. To study nano mechanical characteristics using nano indentation as a function of experimental circumstances is highly condemning for building engineering components. In this research paper, different nano-indentation experiments were observed by considering different values of amplitude, frequency, strain rate, peak load and hold time their values are (1, 5, 10 nm), (11.2, 22.5, 45 Hz), (0.02, 0.05, 0.1, 0.2, 1s-1), (10, 30, 100 mN) (1, 3, 5, 10, 20, 50, 100 sec) to assess their impact on the mechanical properties like hardness and modulus of LDPE [32].

Extensive variants were explored for hardness esteems of LDPE at extremely low values of penetration depth. This precariousness is related to faults in the geometry of indenter tip, inaccuracies in the computation of surface or there is rough surface or impacts of indentation size [33].

At small piercing depth, a tougher and considerable altering behavior of nano indenter was found. This shift in behavior is owing to the changes in the physical properties of LDPE due to hostile environment before the micro indentation or in the time of the formation process of polymers.

The toughness of LDPE rises with as indentation displacement increases. This behavior reveals that there is the influence of indentation size. When there are metals present then there exists density, plasticity and location of plasticity. Indentation size effect cannot be recognized since the polymer have particular strain rate and elasticity [32].

In April 2020 there is another study on nano indentation response of UHMWPE done by Dr. Tanveer Iqbal. According to writer, to study nano scale mechanical characteristics of any polymer may simply be identified using the instrument called as nano indentation. Mechanical characteristics of a variety of materials may be computed notably for polymers this approach is optimal.

In this study effort micro indentation was done by choosing many parameters like frequency, amplitude, stress, strain rate, loading time, unloading time, contact depth and holding duration etc. and we detected the influence of all these factors to hardness and modulus.

For UHMWPE, the findings revealed that when there is peak load applied then load displacement curve go towards lesser indentation depth. There is substantial influence on strain rate and holding for hardness and modulus [34].

The creeping effect as a function of holding time is best described by this approach. Creeping is the phenomenon at which the polymer starts to deform when extra force is applied. For creeping effect there is approx. holding time of 10s.

There is tougher nature of UHMWPE when there is increased indentation amplitude. Significant rise in hardness and modulus when there is increase in strain rate. When increasing the holding time produce reduction in modulus and hardness for UHMWPE that may cause creeping effect takes place. i.e., there is no relationship between deformation and stress [34]. However, when there is prolonged holding time creeping impact might be minimal, that causes a reduced recovery of modulus of elasticity. There is high increased in the value of hardness and modulus when increase in contact depth. Indentation effect also feasible here.

The influence of creep behavior when there is peak load and holding time is applied. There should be various holding periods are offered like 1s, 10s, 20s, 30s. And two constant peak loads were employed 30 and 100mN and typical indentation mood was applied the resultant load displacement curve demonstrated that there is rise in the creep behavior as in the increase of holding duration. Same tendency was seen for the loading and unloading data [34].

Results disclosed that there is substantial influence on hardness as well as modulus of elasticity when the other factors like holding duration, amplitudes. Frequency, strain rate is varied. Load displacement curve diminishes as larger load is applied and indentation depth and holding period is increased.

Results demonstrated that there is substantial dependence of modulus and hardness on peak load and strain rate. It is discovered that the creeping effect may be decreased or eradicated by setting the holding duration 900 to 1100 seconds and two applied loads (30 to 100mN) [34].

Many indentation and other tests have been carried out to investigate the mechanical characteristics of the various materials. If we look back over the previous two centuries, we can observe how much has changed in this industry. Sensors and other devices that conduct indentations on a very tiny scale are being developed as part of this revolution. Nano indentation is a method that has gained prominence in the area of surface mechanical property measurement.

The most well-known characteristics calculated by indentation are modulus and hardness, while other mechanical properties such as hardening exponents, creep behaviour, and residual stresses also benefit from this approach. These measurements have a broad variety of applications in the physical and mechanical sciences, and several new researches on the topic have been published.

In 2010, a study of nanoindentation response was done by Huiying Zhang, Xiang Li, Wenjie Qian, and other researchers. They investigated the effect of nanocomposite coatings on 45 steel substrates in this study. They looked at the effects of pure epoxy (EP), the mixture of pure epoxy and molybdenum disulfide (EP/MoS₂), and the mixture of pure epoxy, molybdenum disulfide, and carbon nanofibre (EP/CNF-MoS₂) coatings on the elastic modulus, hardness and creep properties of materials.

According to the findings of this investigation, the modulus of elasticity of EP/CNF-MoS₂ coatings on steel increased by 37% compared to EP and 26% when compared to EP/MoS₂ [36]. They also tested the hardness of steel with pure EP, EP/MoS₂, and the mixture of EP/CNF-MoS₂ coatings. The hardness of the mixture of EP/CNF-MoS₂ coating on the steel surface increased 104.3% when compared to EP and 47.8% when compared to EP/MoS₂. In this research work, Nano-indentation was done by selecting various parameters like amplitude, stress, strain rate, loading time, unloading time, contact depth and holding time, etc.

Tribology experiments revealed that the composite material's coatings lower the friction coefficient and improve the coating material's wear resistance due to the CNF's high hardness [35]. The degree of the indentation increases with the duration of the holding stage, which was used to investigate the creep properties of coating material in that research.

The findings revealed that the creep displacement of pure epoxy (EP), the mixture of pure epoxy and molybdenum disulfide (EP/MoS₂) coatings is greater than the mixture of pure epoxy, molybdenum disulfide, and carbon nanofiber (EP/CNF-MoS₂) coating under various indentation loads (1mN, 2mN, 3mN, and 4mN). The creep displacement of the combination of pure epoxy, molybdenum disulfide, and carbon nanofiber (EP/CNF-MoS₂) coating decreased even at greater indentation loads such as 4mN. So, the coating of a combination of pure epoxy, molybdenum disulfide, and carbon nanofiber (EP/CNF-MoS₂) was found to be more efficient than other coatings [35].

In 1999, a study was conducted at The Australian National University by Alma Hodzic and other researchers. In this study, nanoindentation and scratch testing was done in order to analyze polymer-glass interfaces. The nanoindentation and scratch methods were used to explore the interfacial area in polymer-glass composite materials. To identify the characteristics of the material nanoindentation test was performed. In this test, indentations performed were up to 30nm in-depth to make the detection of properties of materials easy during the transition phase between the matrix and the fiber. Many indentations were produced on polymer and glass composite material in a line. To identify the different characteristics of the transition zone, 2-3 indentations were done. Nano scratch testing was also done to determine interface properties [36].

It was demonstrated that the nano scratch testing and the nanoindentation might be effectively applied in examining composite interphase. The phase transition area in composite has been the object of numerous research methods during the past twenty years. The findings displayed similar patterns; the observed width of the phase transition area varied due to the nature of the test. The nanoindentation had demonstrated to be a valid experimental approach in combination with the nano scratch test. The extent of the phase transition zone between the fiber and the matrix was identified by both procedures.

The nanoindentation test discovered material characteristics of distinct elements in composite, including the more important mechanical characteristics of the interphase region than the matrix in each material [36]. The significant variation

in the friction coefficient along the phase transition zone in the nano-scratch test verified these findings.

The mechanical characterization of the nanoindentation test indicated that the area in the composite impacted by the indent of a pyramidal tip indenter was considerably smaller in order than the width found in the experimental section. Therefore, the findings in the interphase zone could not be just impacted by the presence of the fiber since the experimentally measured interphase width surpasses the zone of the effect of fiber. So the scratch test seems a more sensitive approach for identifying interphases than the nanoindentation approach.

In 2017, a paper was published by Gheorghe Radu and other researchers. This study investigated the effect of processing temperature on the items made of HDPE, PMMA, and PC+ABS by nanoindentation. The effect of the processing temperature and pressure on the indentation modulus and surface hardness was studied using injection and the subsequent pressure.

The first section described the indentation modulus and hardness of high-density polyethylene (HDPE), polycarbonate, and acrylonitrile butadiene styrene mixture PC+ABS and polymethyl methacrylate (PMMA) changed depending on the operating temperatures by injection. At the same time, other factors that might influence the efficiency remained constant. The next phase of the research examined the variation in indentation modulus and hardness with increasing pressure via injection of high-density polyethylene, polymethyl methacrylate, and

PC+ABS mix. At the same time, other factors that might affect the operation remained constant.

The HDPE samples were collected at injection temperatures ranging from 180 to 220°C with a 10°C difference and rising pressures ranging from 800 to 1200 bar with a 100bar difference [37]. The PMMA samples were collected at injection temperatures ranging from 220 to 260°C with a 10°C difference and pressures ranging from 450 to 850 bar with a 100 bar difference. The PC+ABS specimens were made at injection temperatures ranging from 230 to 270°C with a 10°C difference and at subsequent pressures ranging from 500 to 900 bar with a 100 bar pressure difference. An injection machine was used to implant the samples. The indentation hardness and modulus were determined with the help of Nano Indenter G200 System equipment on model samples.

It was discovered that with the increase of operating temperature and followed by increasing pressure for the sample of high-density polyethylene, the indentation modulus and hardness were increased. It was found that by increasing the processing temperature from 220 to 250°C, by injection for PMMA, results in a marginal improvement in modulus and hardness, whereas the increase of pressure of PMMA, from 450bar to 850 bar, results in a slight reduction in indentation modulus and hardness [37].

In the scenario of PC+ABS, raising the process temperature from 230 to 250°C by injection leads to a slight improvement in indentation modulus and hardness. Raising the operating temperature from 250 to 270°C through injection led in a reduction in indentation modulus and hardness.

However, with increase of the subsequent pressure directly from 500 to 900 bar the indentation modulus and hardness were reduced [37].

This study gives an idea that we can use also perform mixing of xydar-G930 with other materials of high tensile strength such as PEEK, UHMWPE, PMMA etc for enhancing its strength and also with materials which can boost its surface smoothness.

In June 2018, detailed analysis of nano indentation response of fiber-matrix composite was done. Three-dimensional FE simulation was constructed to explore the nano indentation of fiber-matrix composite. It is simply a fiber reinforced composite and at three separate locations on fiber reinforced composite nano indentation was conducted.

The findings demonstrated that there exist a ratio of d/r for a particular displacement load curve. Above this ratio, the indentation response will be minimally impacted by the other component of nano indentation. We may argue that, given a certain ratio of d/r , the maximum displacement covered by the tip of nano should not be rise for the corresponding critical displacement to prevent the influence from the other component of nano indentation [39].

It shows that for a systematically divided fiber-reinforced composite, the mechanical response by nano indentation of the composite will move from matrix dominated to fiber-dominated response with the increase of the volume fraction of the fiber in fiber reinforced composite within the indentation zone. That shows that,

if greater fiber volume percentage is there then more fiber-dominated reaction, and similarly if less fiber volume is present then less fiber dominated response.

The findings demonstrated that, on the basis of the stress field, computation of the elastic modulus will be more efficient when the polynomial equations are provided for the fiber when the indentation takes place on the fiber reinforced composite.

However, the linear equation or polynomial equation will be more suited to compute the elastic modulus of the fiber-matrix when there is more stress field extends from the matrix, before the indenter tip directly strikes the fiber. Once the indentation tip come in touch with fiber surface then the linear equation will not be adequate for elastic modulus calculations owing to deformation zone within the fiber and matrix.

The findings demonstrated that when the fiber is horizontally connected to the surface of the matrix then the shape of indenter and its orientation have minor influence on the nanoindentation response. On the other hand, when the fiber is vertically installed in the surface of matrix, the tiny value of modulus may be determined by utilizing the Berkovich indenter (which relies upon the orientation of indenter) can be significantly different from its conical counterpart. The study demonstrates that when the ratio of fiber-to- indentation distance is modest over the diameter of fiber, then nanoindentation response will rely upon orientation of fiber and distance between indenter and fiber as well as geometry of indenter [39].

It has been found that, when the fiber is mounted vertically to the surface of matrix then the orientation of the indenter can damage the effective tip angle

relative to the fiber, and accordingly raise the effective stress field within the fiber and the matrix. Hence, when a non-axis-symmetric indenter was employed, then greater caution is required [38].

C.T. Wang and C.H. Chien conducted research on the Nano indentation response for nickel metal surfaces in three orientations in July 2010. MD simulations are utilized to determine anisotropic properties for a crystallographic nickel surface with three distinct surface orientations (100), (110) and (111) while executing the micro indentation procedure.

Many parameters may be seen during the nano indentation response for nickel with three orientations. Atomic stress distributions, pile-up patterns at high indentation depth or displacement, and material (nickel) characteristics at various crystallographic orientations are among the causes.

The findings show that the strain energy of the substrate's nickel surface is stored in the substrate through nucleation formation during loading. It is also dissipated from the substrate during aircraft unloading (111). The slip angle of the third orientation is responsible for the variance in the load-displacement curves (111). The pile up pattern of three oriented nickel substrate, one of the components, demonstrates that the crystalline nickel metal displays the pile up pattern from the nano indentation response [39].

Crystallographic nickel substrates display a variety of pile up patterns that extend out in various crystallographic orientations. The influence of surface features of FCC nickel material on nano indentation may be seen by slip angle of

(111) plane, which is their nickel metal orientation. This slip angle may have an effect on the hardness and modulus values acquired by nano indentation. Young's modulus is the cohesive energy for various crystallographic nickel surfaces that is also impacted by the slip angle of third orientation [39].

Researchers were satisfied with the reaction of nano indentation for the nickel surface having three orientations. There are various downsides like it impacts the hardness and modulus values by slip angle of third orientation.

Amin H. Almasri, George Z. Voyiadjis in the year of 2008-09 studied the nanoidentation of FCC metals. The nanoidentation of different metals can be compared with our respective material liquid crystal polymer. For several face-centered cubic metals with various purities, the findings of this experimental study indicated the effects of nano-indentation size. Silver, copper, aluminium, lead, and nickel were the materials of choice. Berkovich indenter with continuous stiffness measuring process used for nano-indentation testing, where hardness is continually measured with depth of indentation. The hardness of the material was shown to increase or decrease with increasing indentation in three unique locations. A basic power law model, which includes the effect of grain boundaries, is used to explain this behaviour [39].

From nanometers to millimetres, the hardness vs indentation depth of different metals is measured. While the nano indenter was used to measure indentations as small as 10 nm to a few micrometres, the macro indenter was used to measure millimeter-scale indents. As indentation depths increase, the upper and lower confidence levels were observed to converge to the experimental sites.

Because the indentations in the aluminium and lead samples were so deep (10 m), this was particularly apparent. Other samples with indentations larger than 10 m should exhibit the same convergence trend.

However, the 10 micrometre indentation could only be achieved with Aluminum and Lead, which are both rather soft. Purity does not necessarily decrease in the hardness-indentation curve, as the higher-purity Copper sample had a cleaner region compared to the lower purity sample [39].

Ti-6Al-4V alloy is widely employed in the aerospace, biomedical, and automotive industries because of its high strength to weight ratio and low density due to its unique features. Because of inadequate wear resistance and poor mechanical strength at high temperatures, its use in many technical industries is restricted. Further study has led to changes on Ti-6Al-4V (alpha and beta phase) alloy by reinforcing with hard particles such as zirconia and potentially titanium nitride to increase mechanical qualities at elevated temperatures.(TiN) [40].

In 2017, experimentation was done by Mosima Edith Maja, Oluwasegun Eso Falodun and other co-researchers on Ti-6Al-4V alloy at University of Johannesburg, South Africa. In this study mechanical properties of sintered Ti-6Al-4V reinforced with 1-4 vol% TiN was investigated by ultra-nanoindenter (UNHT) technique.

A Swiss ultra nanoindenter (UNHT) was used for nanoindentation testing. This inquiry made use of a Berkovich diamond indenter with a three-sided pyramid. The highest load applied was 3 mN and held for 10 s in order to determine the time-

dependent deformation properties. It was discovered that the microstructure's grain shape is controlled by the presence of reinforcements [40].

Furthermore, the results of nanoindentation showed that the hardness and modulus of elasticity of the alloy matrix are dependent on the presence of TiN. In comparison to alloy, sintered compact containing 4 percent TiN (by volume) exhibited the highest indentation hardness value of 7517 MPa and the highest modulus of elasticity of 156 GPa [40]. Additionally, the maximum indentation depth of sintered nanocomposite materials decreased while their hardness increased, suggesting that these materials may have much better wear resistance than alloy [40].

MMCs are supposed to offer reinforcement to provide strength and stiffness, while the matrix material provides ductility and strength to the composite structure. This is due to the fact that alloys cannot give both strength and stiffness to structures at the same time. Because of the tiny grain, phase, and strength of nano-sized materials, nanoscale reinforcement may improve mechanical properties [40].

The study revealed that reinforcing Ti–6Al–4V with TiN limits dislocation movement while enhancing mechanical characteristics. This inhibits the spread of cracks from one phase to the next. So, titanium nitride provides high strength to alloy which gives an idea about the reinforcement ability of Xydar-G930. On addition of measured amount of Xydar-G930 in different materials which have loose structures can provide reinforcement to their structures [40].

In 2012-2013, A. Molazemhosseini, H. Tourani, M.R. Naimi-Jamal, and A. Khavandi used nanoindentation to examine the micromechanical properties of PEEK-based hybrid composites reinforced with short carbon fibers (SCF) and nano-SiO₂ particles. The melt-mixing procedure at 400 degrees Celsius was used to create the composites. The neat polymer was granulated, the nanoparticles were 13nm in size, and the original length of the short carbon fibers was 6 mm [41].

Each specimen was subjected to more than 30 nanoindentation tests. AFM was used to assess the surface topography of the indentation and the effects of reinforcing particles on the standard nanoindentation load-displacement curves of composites were examined. The overall reinforcing effects of carbon fibers and nanoparticles were explored as well. The nanoindentation responses and micro frictional characteristics of the matrix and fiber phases of the composites vary substantially. The use of reinforcing compounds increased hardness and elastic modulus significantly [41].

The results of this study showed that the local nano mechanical characteristics of the composites are heavily influenced by the kind of existing phase that is indented. In reality, hysteresis will develop during loading and unloading cycles if the PEEK matrix or fibre matrix interphase has been indented. However, when carbon fibres are indented, elastic behaviour is observed. Elastic behaviour is shown by a linear trend in the load-displacement curve during both the loading and unloading phases [41]. Furthermore, nanoparticles cause the load-displacement curves of the indented point on the matrix region to shift to lesser indentation depths.

The incorporation of short carbon fibers into clean PEEK improves the material's decreased elastic modulus and hardness significantly (143 percent increase in reduced elastic modulus). Furthermore, adding up to 2% weight percentage of nano-SiO₂ particles to conventional composite (PC) leads in a second improvement in the decreased elastic modulus and hardness, such as 44% increase in the E-modulus [41].

It was in 2011 that Adams, Michael J; Gorman, David M; Johnson, and Simon A. performed nano indentation on PMMA. According to the results of the study, the load increases theoretically with the square of the total penetration depth when a flexural symmetric half-space is indented. The validity of this relationship between an organic polymer and a Berkovich indenter has been demonstrated experimentally, if that the tip fault and samples are properly considered. A solver for the ratio of impact height to final density was also shown to exist. Results from this study aid in developing ways for getting the rate-dependent mechanical characteristics of organic films or polymers with spatial physical behavior.

When filling a flexural monomer at a fixed load and taking into consideration the end fault, the quadratic loading relationship is accurate. For creases made at a constant velocity or loading rate, the bell curve factor decreases [42]. The contact depth-to-total penetration depth ratio, which remains constant under loading at a constant strain rate, is a valuable statistic for analyzing subsequent creep and creep recovery data.

In 2021, Dr. Tanveer Iqbal, Naveed Ramzan, Ujala Farooq, and Sairo Bano did a study on the combination of ABS and polycarbonate (PC). ABS and PC are now well structural nylon because of their widespread application in automobiles, 3D printers, and circuit boards. The combination of ABS and PC, on the other hand, improves toughness, processability, and thermal stability. Micro is being used to comprehend the slurry erosion characterisation of ABS/PC hybrids in the current work.

Sheets of ABS/PC polymer mixes (75:25, 50:50, and 25:75) were made by melting and pressing plastic melts together. The intermolecular interactions between the mixes' parts were examined using Ftir, a Factorization of the spectra. It was necessary to conduct a significant number of micro tests to understand the surface mechanical properties of ABS and PC blends. After loading and before discharging, creeping effects were seen [43].

Indentation, elasticity, indentation hardness, and creep values were calculated as a function of penetration displacement for the quasi-continuous stiffness mode (QCSM). The load-displacement curves indicated that as the ABS percentage climbed, the displacement increased, but the hardness and elastic modulus values declined [43].

The nano surface mechanical characteristics of clean ABS, PC, and ABS/PC blends were investigated experimentally in this work. Fifty percent ABS/fifty percent PC, 25 percent ABS/75 percent PC, 25 percent ABS/50-percent-percent-percent and PC were all created by melt-processing followed by a hot-pressing procedure.

Each sample's chemical structure was examined using the FTIR method. No plastics were decomposed during the mixing process, which was carried out by hand. A maximum load of 100 mN and a hold period of 20 s resulted in an 8.1-micron penetration depth for ABS and 7.2-micron density for PC. As the ABS content grew, so did the penetration depth [43].

Additionally, the modulus and the hardness of the indentation were observed. The hardness and elastic modulus of all samples dropped as the amount of ABS in them increased. Increases in indentation hardness and elastic modulus were noted as PC content increased. Conversely, a decrease in creep rate and creep was noted with a rise in PC content, but 50 percent ABS/50 percent PC trends were different from other tests. Prepare concerns, environmental influences, the inclusion of cavities and flaws, and inaccuracies in surface determination were assumed to be the source of these uncertainties.

Atomic force microscopy was used by I.Karapanagiotis, D.F.Evans, and W.W.Gerberic to examine the impact of nanoindentation-induced defects on polystyrene (PS) spin cast films on silicon (Si) substrates that were 50 and 120 nm thick (AFM). When bulk PS is heated over its glass transition temperature (T_g), indents form with residual penetration depths less than the film thickness. Curvature gradient-driven diffusion is used to describe the final levelling step. Soul values are very close to the equations [44].

Unentangled nanoindentation defects ($M_w=10,900$ g/mol) PS films were inspected using AFM when heated above T_g , according to the results. Initially, the indent was square, but as the circle grew, it changed shape to a circle.

Scaling with time (t), R_{tx} was determined to be as follows: for thick films, $x = 0.3$, and for nanofibers ($h = 50\text{nm}$), $x = 0.2$. Curvature gradient-driven diffusion was used to describe the levelling process. A good match was established between the computed flux ratios (D_s) and those of PS' bulk self-diffusivity. The D_s values for thin films ($h=50$ nm) were higher when the film was thicker ($h=120$ nm). The rim relaxation effect and the distance between the bottom of the indent and the substrate were cited as the causes of this. When it comes to thin films, these effects slow down the levelling process, compared to their effect on thick films [44].

LCPs give an alternative categorization of material which bestride between a solid and a liquid phase. From their initial admission in the research center to be acknowledgment that Crystalline Atoms furthermore exist in science, this type and unmistakable particle are now universal in an expanded usage of cutting-edge living. These polymers have important higher request structure notably visible in their liquid phase and are not the same as thermoplastics, for example, polyesters and other aromatic polymers.

These features provide LCPs a big range of notably advantageous material properties, for example, superior dielectrics, concoction reactivity (obstruction), and biocompatibility for clinical applications and goods.

Lehmann discovered the major gloomy liquid as the smooth strong softened just as Reinitzer had. He represented seeing different small crystalline arrangements with hilter kilter outskirts. Lehmann realized that this initial midway liquid appeared, by all indications, for crystalline character. Further investigating, adjusting thinking, Lehmann uncovered a discovery liquid crystalline substance. Lehmann's (and Reinitzer's) view given major consideration at that stage, particularly. Lehmann discoveries in late 90s. In reality, around mid of 20s numerous mixtures were came into existence to display liquid crystal polymer behaviour. Be that as it may, following this basic consideration excitement for the zone of technology before long waned [1].

Chapter 3

MATERIALS AND METHODOLOGY

3.1 OVERVIEW:

In this chapter, a detailed review of the materials and the procedure for carrying out the fabrication of Xydar® G-930 Liquid Crystal Polymer (LCP) sheet and the nano indentation test on the sheet. This chapter will also discuss the tools we used for fabrication of sheet and for its characterization.

3.2 MATERIAL SELECTION:

3.2.1 Xydar® G-930:

Glass-reinforced injection moulding grade Xydar® G-930 Liquid Crystal Polymer (LCP) is ideal for externalising in the wire and cable industries. Moldability is great with this material. Low-temperature Xydar® G-930 can fill extremely thin walls over long flow lengths with little or no flash, even at mould temperatures below 200°F (93°C). It has a good weld line strength and causes low warpage in moulded objects. High hardness (even at elevated heat), good thermal stability, high deflection temperature, intrinsic flame resistance, and extraordinary rigidity to most toxins, ageing, and light are all features of this metal.

For nanoindentation experiments, we imported pellets of Liquid Crystal Polymer (Xydar® G-930) from Malaysia.

First of all, the LCP sheet was manufactured using a thermal press machine (Heat sensitive machine utilized for the transformation of polymer pellets to sheet

having clear smooth surface). LCPs are thermoplastics with properties intermediary between exceptionally ordered solid crystalline and amorphous disordered liquids across a specific temperature range. Average chain length, the kind,length of the flexible linker and atomic weight dispersion, all influence the crystallinity, adaptability, and strength of LCPs.

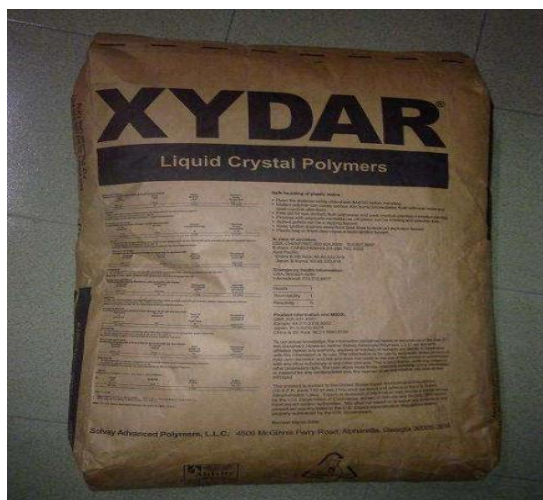


Fig: 3.1 Xydar® G-930

Copolymerization with different monomers, the addition of plasticizers and fillers, and mixing with other polymers such as polycarbonates, polyolefins, and polyesters may help to balance the properties even further. Plasticizers and fillers save costs, alter procedures, and even encourage handling. Graphite, for example, increases chemical and wear resistance, while carbon fibers and glass fibers improve quality and toughness, and carbon black improves electrical characteristics like electrostatic dispersion.

The effect of nanoindentation test parameters on the resultant nanomechanical response is crucial for the effective design of building engineering tools with sufficient surface characteristics.

3.2.2 Mold Release wax:

When it comes to mold release, composite fabricators turn to a special kind of high-gloss paste wax. A strong mold escape tool, it provides repeatability, luster and non-staining, hard releasing film, both of which are crucial to the maker. In order to see as many releases as possible each application, Mold Release Wax utilizes a proprietary combination of fats. Factory, new molds, and fiber glass usage all benefit greatly from this additive. This white non-silicone wax is aimed at creating a robust, long-lasting, large layer. Fungus publish wax has exceptional release properties. Mold release paste or wax, one of the first types of mold removal materials, has numerous benefits.



Fig: 3.2 Mold release wax

A mold release paste or wax, being one of the first forms of mold release agents, offers various advantages.

Advantages:

1. Low Price Point:

For a lot longer, the low cost of shellac mould release sprays and agents has been a major selling feature. Often are affordable, making as a viable option of businesses.

2. Quality Mold Surface:

Your product or prototype may have minor flaws, such as a soup to nuts on the edge. It is possible to smooth out the layer by using a wax or paste to fill in the flaws.

3.2.3 Thermal press machine:

A thermal press machine is equipment used in the manufacturing of desired sheets. This machine has a high temperature range of more than 400°C and can apply a pressure of 5000 psi pressure. This machine is available in a variety of types, which are chosen according on the work at hand. This machine is excellent for a variety of uses, such as permanent marking and printing on any object utilizing a customized mold.



Fig: 3.3 Thermal press machine

3.2.4 Nano-Indenter:

Nano-indentation is a depth-detecting strategy and a valuable and energizing method to research the surface mechanical properties of a vast range of materials, especially polymers. The impact of exploratory circumstances used during nano-indentation on the resulting nano-mechanical reaction is important for the productive structure of developing segments with suitable surface qualities. The objective of most of nano-indentation analysis is to separate flexible modulus and hardness of the under test material from load-relocation estimations [45].

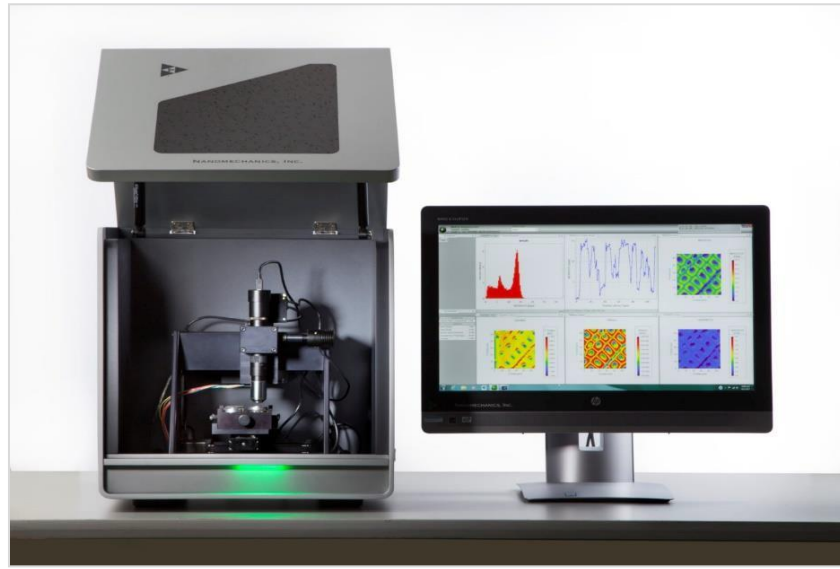


Fig: 3.4 Nano-indenter

3.3 METHODOLOGY

Basically, two types of procedures were applied to avoid/minimize mistakes and to enhance experimental abilities. Qualitative examination is a scientific approach of examination to compile non-numerical information. This form of inquiry "alludes to the implications, concepts, meanings, qualities, representations, and portrayal of objects" and not to their "counts or measurements". Quantitative strategies, on the other hand, emphasize target estimations and the arithmetic, mathematical, or numerical analysis of information gathered by surveys, polls, and tests, or by modifying pre-existing statistical data using computational processes.

1. First of all, the mold is cleaned and mold release wax is applied on the inner surface and also on the walls of the mold. This will help to remove the sheet from mold after the preparation. Another benefit of wax is it will help to seal up the pores in the inner surface or on the walls of the mold. Ensure the mold surface is properly

sealed (porous mold surfaces such as wood or plaster must be sealed prior to the application of any mold release agent) clean and free of contaminants.

2. We used a clean and lint-free cloth before applying the wax to the surface of mold in circular motions with a small amount of wax. Fill the whole mold base, giving particular attention to any tight corners. Allow the wax to settle, that would make the pores on the mold surface more noticeable, by allowing it to melt off a little.

This will take roughly 15 mins. Using a different, clean, lint-free cloth start to lightly whisk the wipe over the surface. This will start to slowly level out the swirl pattern of the wax.

3. We placed pellets in 4×4 sq. ft. and 1-inch die smoothly. The mass of pellets required for the fabrication of sheet of required volume is calculated. Then place the die in between the jaws of thermal presser. Next wait for it to attain the melting temperature of pellets. After that, we applied a pressure of 1600psi then we set the temperate at 360-400 degree Celsius. We placed the pellets for 15-20 minutes in the presser and then shut the supply off to cool the sheet. We let it cool for 1.5 hours. After that we get the sheet off from the die and cut into small pieces for testing its mechanical properties using nano-indenter and tribometer.

Nano indentation is a nondestructive experimental strategy which permits a few tests to be accomplished on a similar material without the necessity of optical imaging. Samples for nano-indentation conventional indentation experiments. Penetration displacement and indentation are the two essential limits which can be evaluated using nano-indenter. Hardness and Young's modulus can be assessed from numerical assessment of the acquired load-displacement data. Indenter load is applied on the specimen through the indenter tip for a specific time frame and the resultant indentation area is estimated as in normal indentation experimentation.

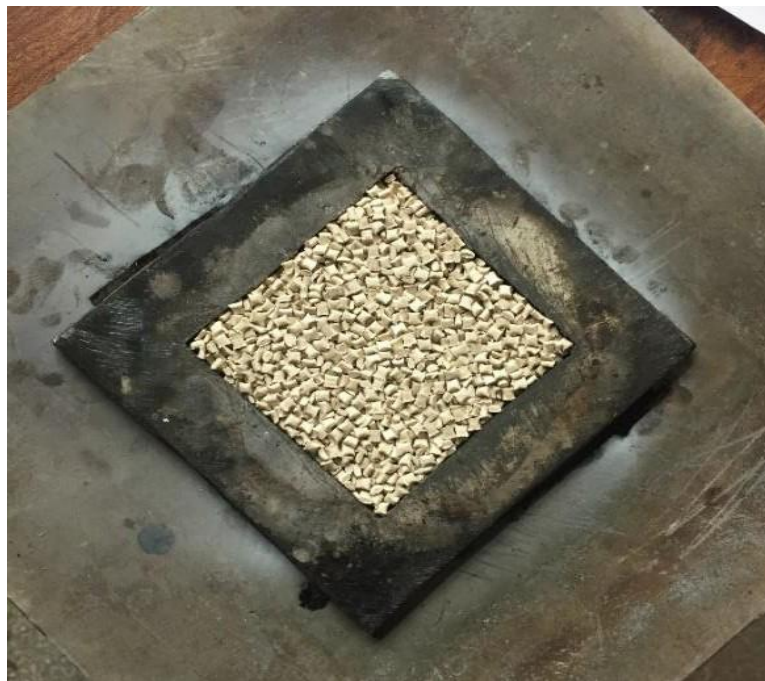


Fig: 3.5 Xydar® G-930 pellets in mold



Fig: 3.6 Sheet prepared and cooled



Fig: 3.7 Sheet cut into small pieces for nano-indentaton

3.4 BLOCK DIAGRAM REPRESENTATION:

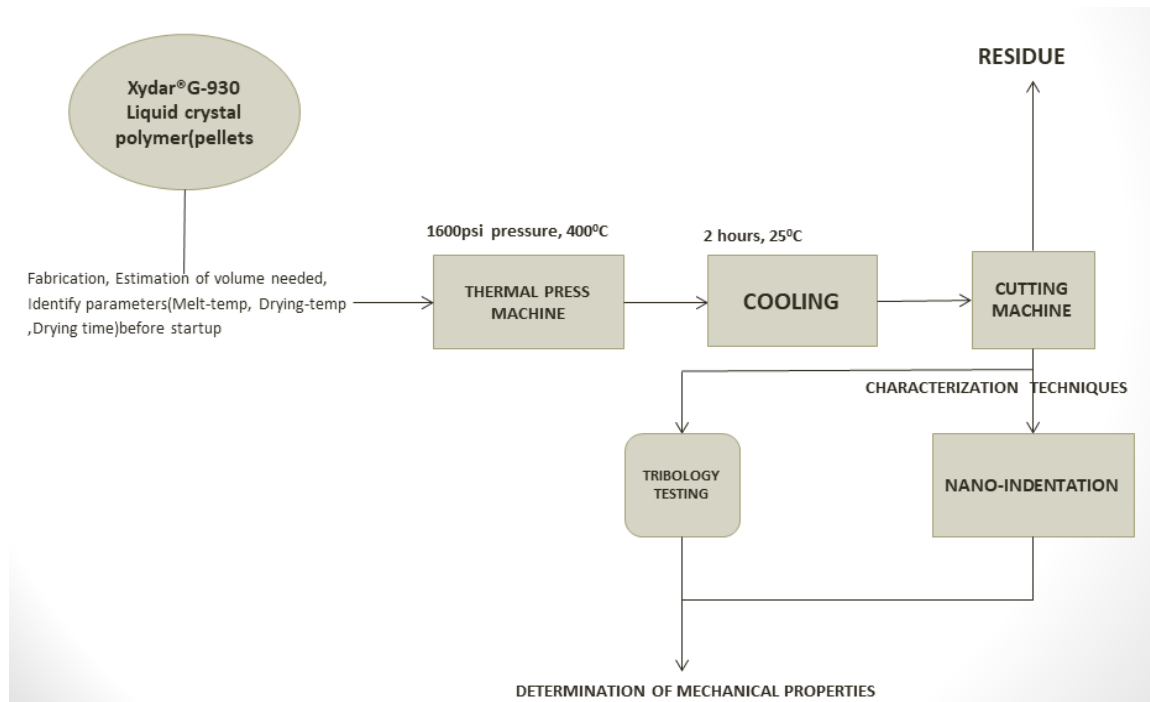


Fig: 3.8 Block Diagram representation of Final Year Research Project

Chapter 4

RESULTS AND DISCUSSIONS

4.1 OVERVIEW:

This chapter includes the Nano-indentation results of Xydar-G930 and the comparative study overview of Xydar-G930 with other compatible polymers such as LDPE, UHMWPE, PS, PC. It will demonstrate that how Xydar-G930 can compete with these polymers and can replace these in various applications. The results will demonstrate the different analysis performed during the nano indentation of Xydar-G930. This chapter also includes the social and economical impacts of the project.

4.2 NANOINDENTATION RESULTS OF XYDAR® G-930:

A contact compliance indentation mode is used by the nano indenter to analyze the surface mechanical characteristics of materials. As a result, the material hardness and elastic modulus may be computed without having to estimate the area of indentation. The loading circumstances have a large influence on nano-indentation measurements of a polymer.

4.2.1 Load displacement curve:

The load-displacement curve offers a "mechanical fingerprint" of a material response to contact deformation. Figure 4.1 below shows a load-displacement curve. The loading phase of the curve is caused by both plastic and elastic

disfigurement reactions of the contact, while the unloading segment is caused by elastic recovery of the contact.

The data was gathered at a constant strain rate, and the highest displacement recorded was 7.8 μm . The end outcome demonstrates the tougher nature of the polymer as long as the indentation load is applied

To determine material hardness, indentation pressures ranging from $1\mu\text{N}$ to 100 mN may be used. The highest displacement change was seen at the start, and it then climbs virtually linearly to the maximum load.

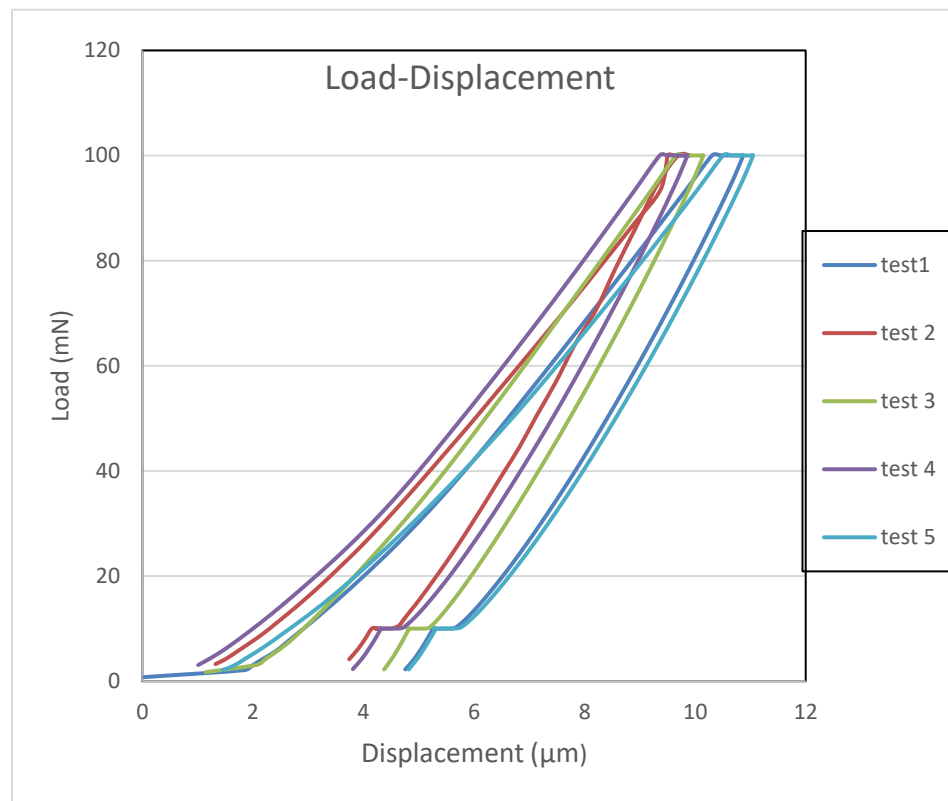


Fig: 4.1 Load-displacement curves

Fig. 2 provides a clear representation for a better comprehension of these usual curves. There is a steady rise in displacement from point A to B (Loading) as the indentation load increases. Creep is noticed in the load-displacement data from B to C as a rise in displacement during a hold time at a maximum load.

This creep tendency is material dependent and generally reduces to extremely low levels after few seconds, around 10 seconds. In materials science, creep is the propensity of a solid material to gradually or permanently deform under the influence of constant mechanical stresses. It may occur as a result of long-term exposure to high levels of stress that are yet below the material yield strength.

When we analyze the Unloading section (from C to D), the indenter load disappears and the displacement diminishes, nearing zero. From D to E (Recovery), the polymer returns to its natural form, relinquishing its identity.

Material stiffness may alternatively be computed as:

$$\text{STIFFNESS} = L/D$$

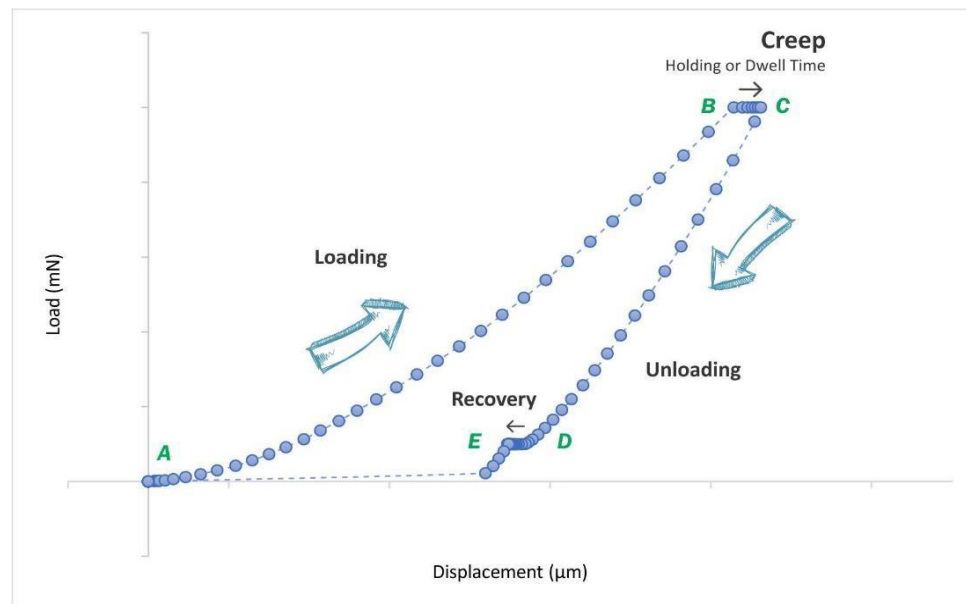


Fig: 4.2 loading and unloading Load-displacement curve

4.2.2 Elastic Modulus:

The stress-to-strain ratio below the proportional limit is defined as elastic modulus. It is a measure of the rigidity or stiffness of a substance. For determining elastic deflections, the modulus is an important design parameter. The connection between elastic modulus and indentation contact depth is seen in Fig 4.3. As contact depth rises, E-Modulus decreases abruptly at first. The modulus decrease rate eventually slows and becomes constant until maximum load is reached.

A toughened material has a high Young's modulus and only slightly changes form under elastic stresses. Elastic modulus falls as indentation contact depth rises, as illustrated in the figure below. This is due to a decrease in the hardness of the substance. Resistance to elastic deformation reduces as the indenter tip enters the specimen.

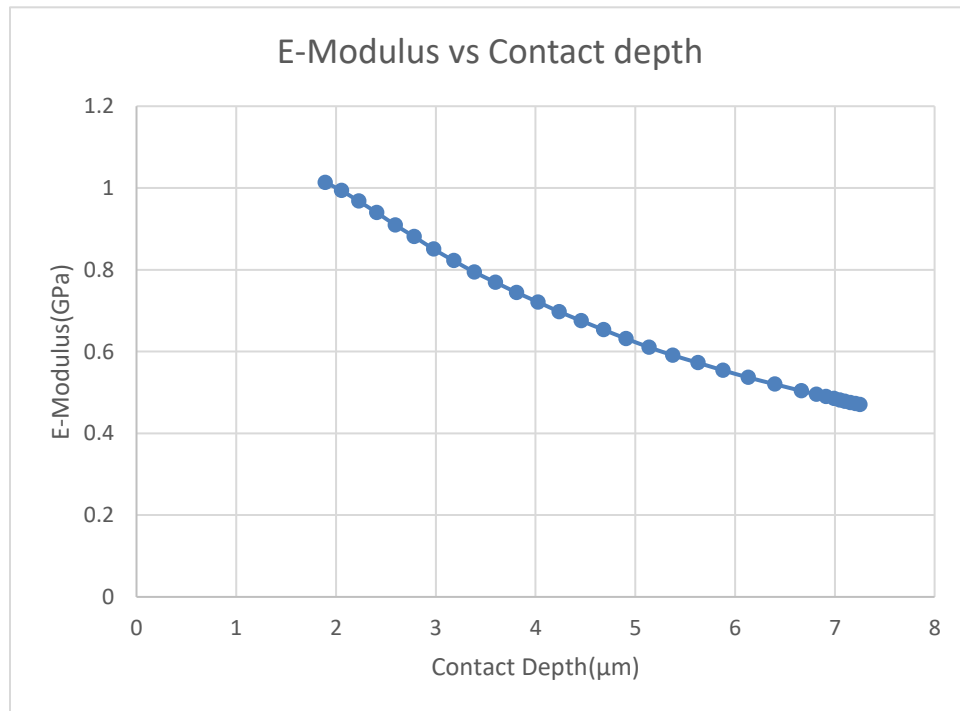


Fig: 4.3 Elastic modulus Vs contact depth

Displacement as a function of modulus

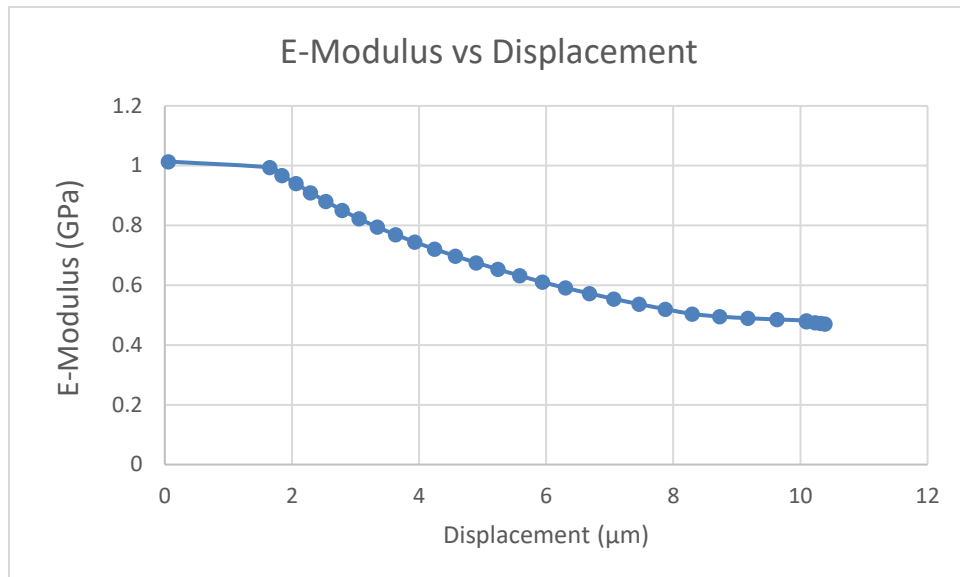


Fig: 4.4 Elastic modulus Vs displacement

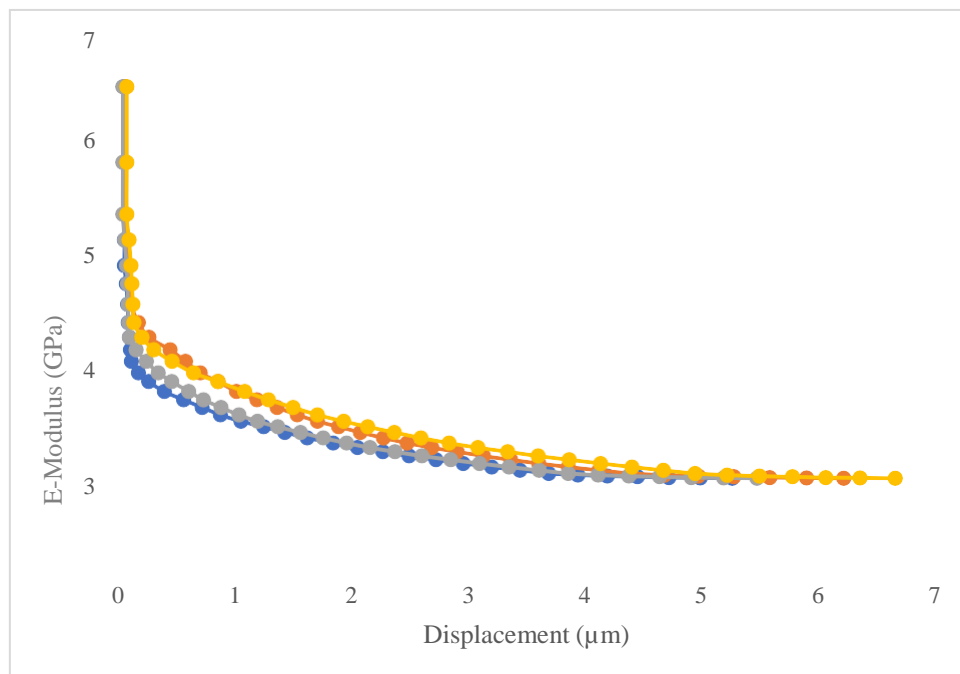


Fig: 4.5 Elastic modulus Vs displacement of different samples

4.2.3 Hardness:

The ability of a substance to resist plastic deformation, often via indentation, is characterized as its hardness. Amazing vacillations were seen for hardness esteems at low displacement estimates. A harsher and more changeable behavior was seen at very low penetration depths. This might be related to significant changes in the physical properties of the material caused by harsh conditions before or during the polymer production process.

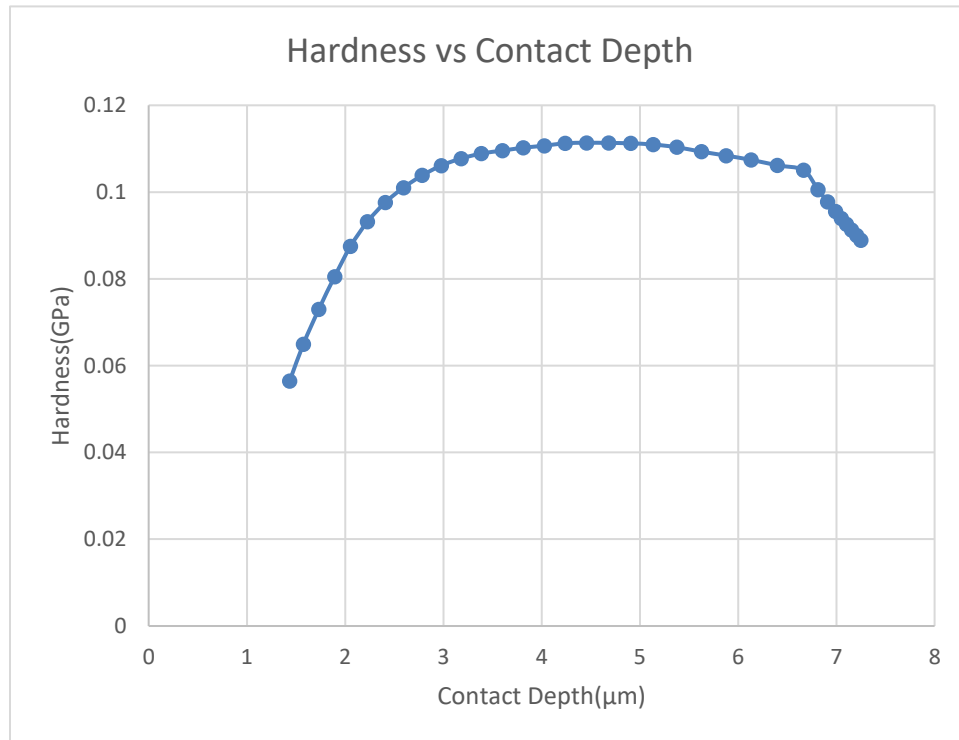


Fig: 4.6 Hardness VS contact depth

Hardness as a function of displacement:

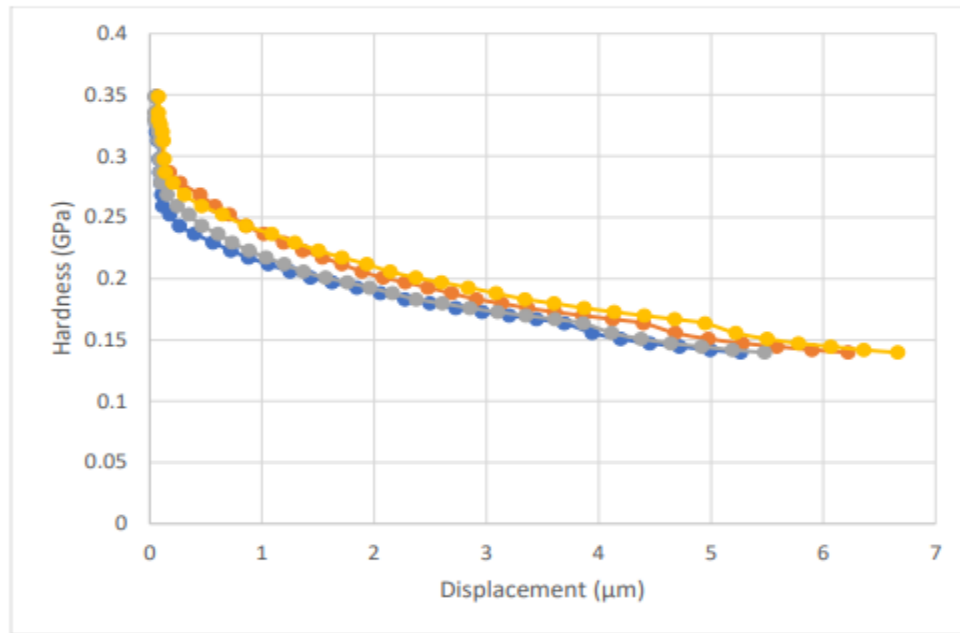


Fig: 4.7 Hardness as a function of displacement

4.2.4 Creep Analysis & Yield strain

Creep rate is the rate of deformation of material. In creep analysis we analyze the rate at which the material deforms. If the load is continuous then the material will go to permanent deformation. By creep analysis we can get expected deformation of our material and then take suitable measures to avoid the failures.

Yield strain is the ratio of the hardness of the material to the elasticity of material. In yield strain Vs contact depth relation the curve goes up because the hardness of material increases with contact depth but the elasticity decreases. So the yield strain will increase.

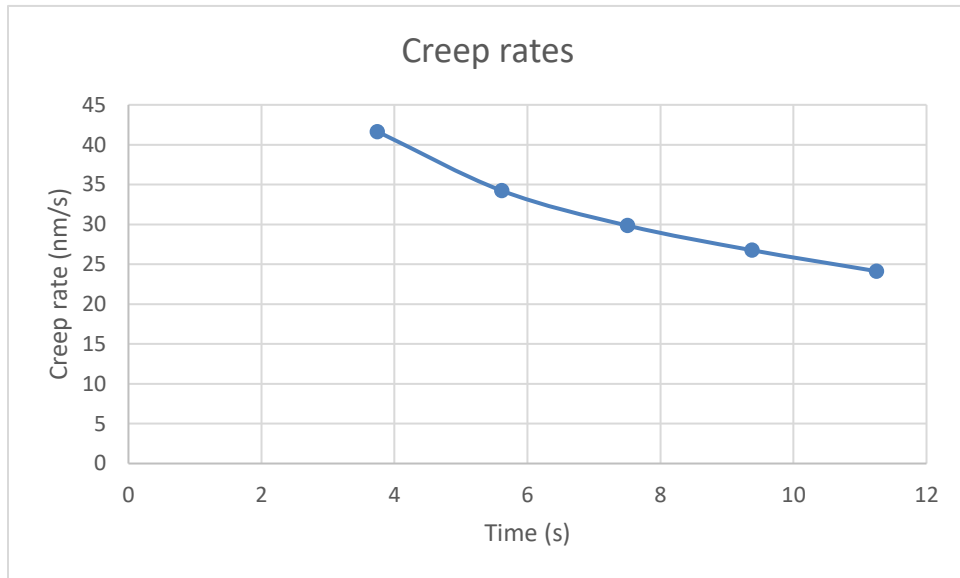


Fig 4.8 creep rates Vs time

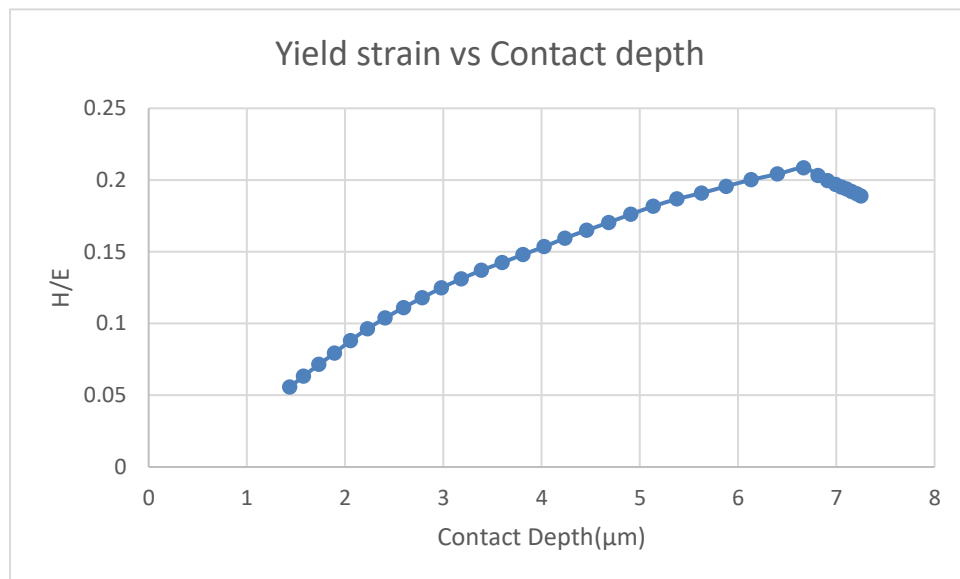


Fig: 4.9 yield strain Vs contact depth

4.3 ANALYSIS OF RESULTS:

Liquid crystal polymers (LCPs) are a relatively new group of high temperature polymers that have attracted academic and corporate attention. In comparison to ultra-high-quality conventional polymers, the temperature dependability of LCPs is a distinguishing feature that makes them suitable in a wide range of applications.

Best possible outcomes:

Indentation load as a function of displacement for different tests:

As the load on the polymer sample increases, the needle penetrates the polymer and travels some distance. As the load rises, the distance covered increases linearly. It signifies that there is a linear connection between load and displacement, demonstrating the hardness of polymer.

Elastic modulus as a function of contact depth and displacement

The elastic modulus of polymer decreases not constantly as the depth and displacement covered by the needle of nanoindentation increases, but it becomes constant after covering some displacement.

Hardness as a function of contact depth and displacement

Indentation depth or area is inversely related to hardness (inverse relation).

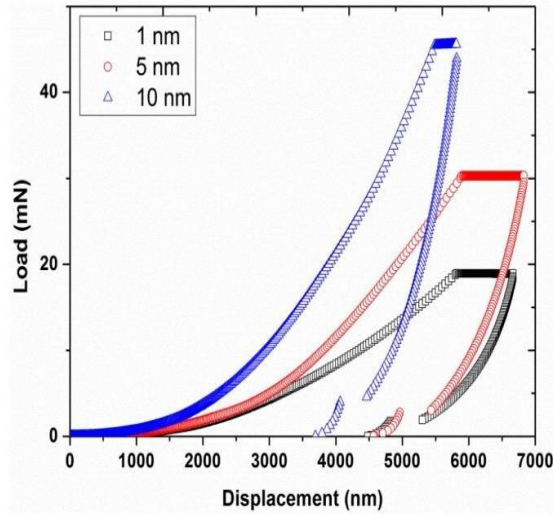
4.4 NANO-INDENTATION RESPONSE OF COMPATIBLE POLYMERS

4.4.1 Nano-Indentation Response of UHMWPE

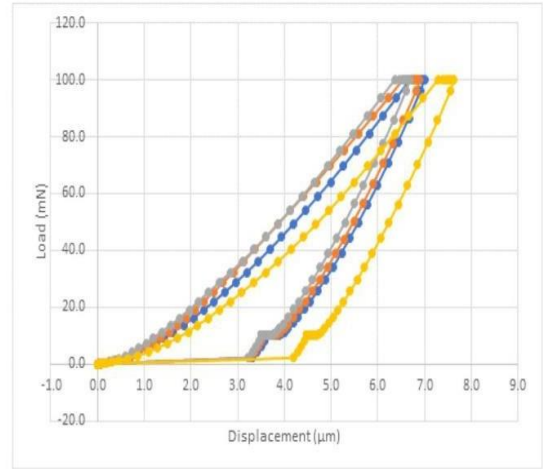
UHMWPE (ultrahigh molecular weight polyethylene) is a polymer with a repeating unit, $[C_2H_4]_n$, where n means the degree of polymerization of the unit. Unlike most other polymers utilized in the biomedical industry, Xydar® G-930 offers better toughness, abrasion resistance, and a lower coefficient of friction.

Most of nanoindentation tests are designed to derive elastic modulus and hardness information from load-displacement calculations. The transducer exerts a force during the technique, and the displacement is continually measured to generate a typical force vs. displacement curve.

The resultant force vs. displacement curve represents 'mechanical fingerprint,' from which quantifiable nanoscale material attributes may be calculated. The customer receives quantitative data on the mechanical characteristics of the sample after examining the measured force vs. displacement curve (particularly the unloading phase). Other data can be acquired, including as fracture toughness, stiffness, delamination force, and film thickness. This section compares the nanoindentation findings of Xydar® G-930 to the results of UHMWPE.

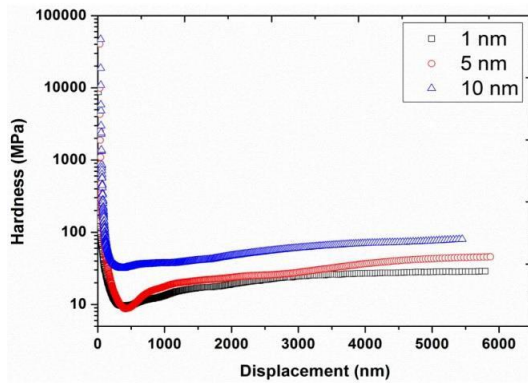


(a)

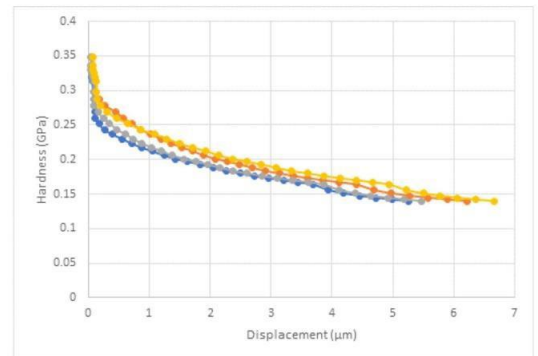


(b)

Fig 4.10 a) Indentation load as a function of displacement for the three different amplitudes (1, 5 and 10 nm) at a frequency of 45 Hz. (b) Indentation Load as a function of displacement for a specific values of amplitude, harmonic frequency and strain rate.



(c)



(d)

Fig 4.11 (c) Hardness as a function of displacement for the three different amplitudes (1, 5, and 10 nm) at a frequency of 45 Hz. (d) Hardness as a function of displacement, keeping all other parameters constant.

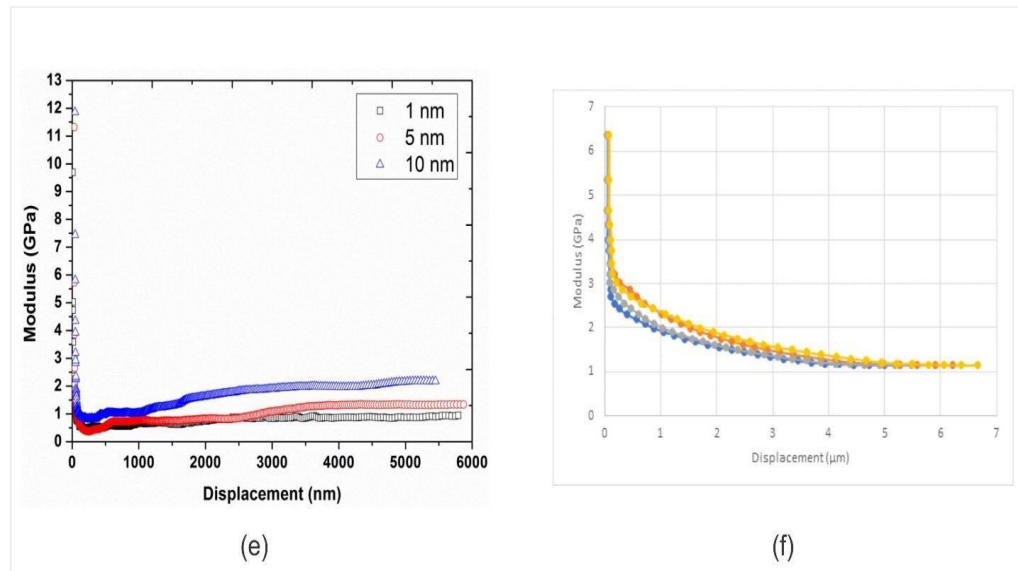


Fig 4.12 (e) Modulus as a function of displacement for the three different amplitudes (1, 5, and 10 nm) at a frequency of 45 Hz. (f) Modulus as a function of displacement, keeping all other parameters constant.

4.4.2 Nanoindentation Response of LDPE

LDPE gives great execution when contrasted with different polymers, glass, or metal. For most part, the uncommon properties of LDPE are simple handling, better electrical, compound, scraped area and effect opposition, and higher strength. LDPE acceptable chemical resistance and great electrical properties, anyway it has a high thermal expansion and will in general failure under mechanical and thermal stress.

Unlike LCPs, LDPE has extremely low quality, bad heat resistance and high coefficient of thermal expansion. The information on creep conduct of LDPE is essential to forestall creep distortion and crack in light of the fact that LDPE is referred to show bigger creep trend when contrasted with other polymers like HDPE because of lower crystallinity.

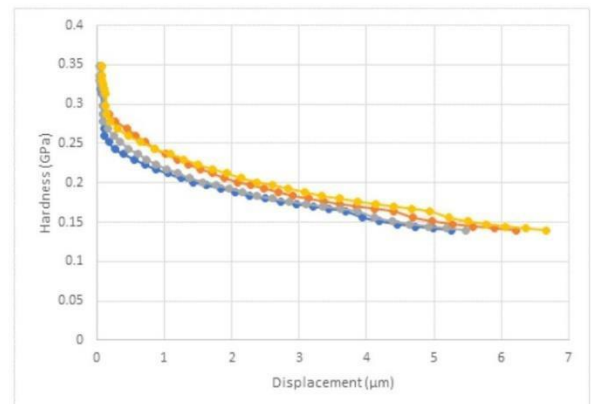
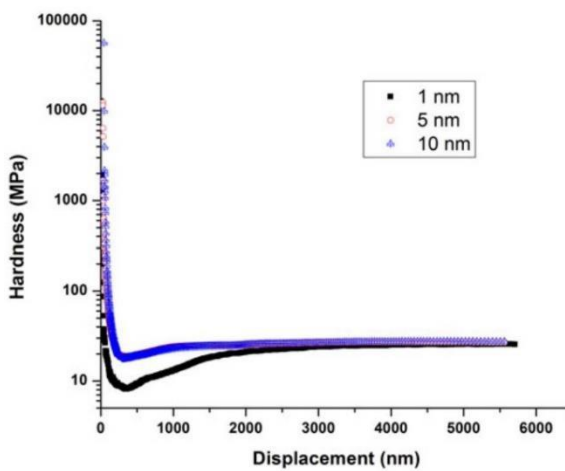
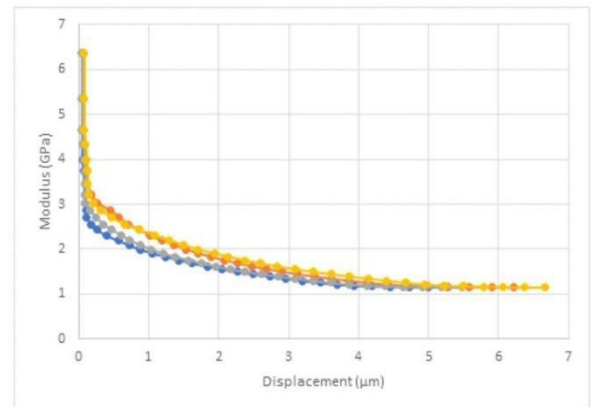
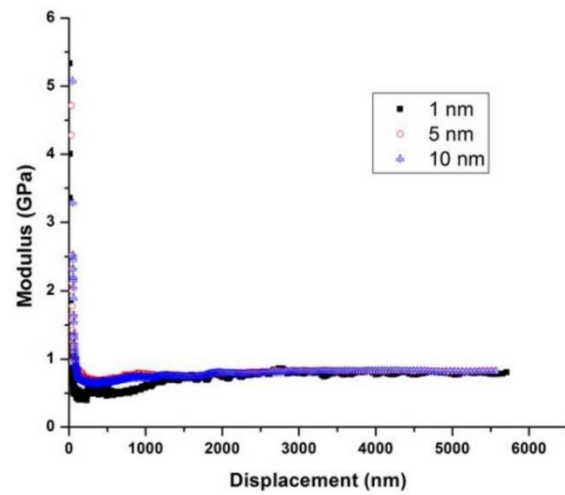
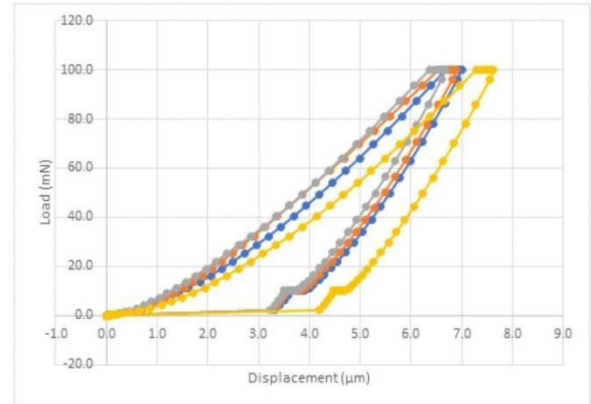
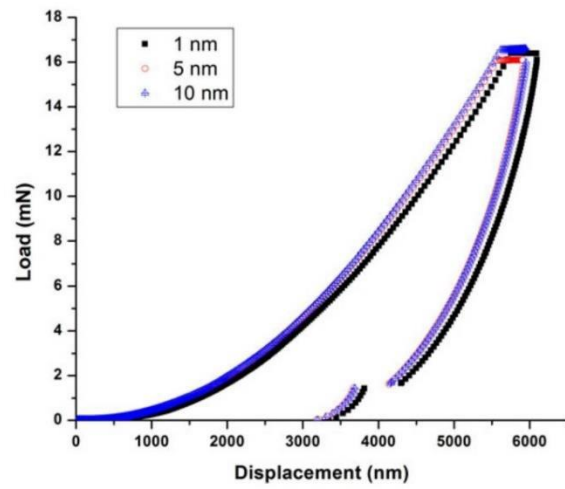


Fig: 4.13 Comparative overview of nanoindentation results

Table 2: Comparative analysis of Xydar®G-930 with other compatible polymers

	Load VS displacement	Displacement VS hardness	Displacement VS modulus	Maximum Load	Displacement range	Contact depth range
Xydar®G-930	Linear relation	First decrease and then constant	First decrease and then constant	100mN	7μm	5.3μm
LDPE	Linear relation	At start decreases then increase	At start decreases then increase	80mN	6800nm	10000nm
UHMWPE	Application of Little load corresponds to huge displacement	Hardness almost remains same	At start slightly decrease and then remains constant	Less than 300mN	560nm	10nm-10μm
PC	Slightly linear	Decreases at start and then constant	Decrease at start and then remains constant	Less than 300mN	530nm	10nm-10μm
PS	Linear relation	Show fluctuations	Sharp decrease at start then remains constant	Less than 300mN	510nm	10nm-10μm
PMMA	Linear relation	At start decrease and then constant	Sharp decrease at start then remains constant	Less than 300mN	510nm	10nm-10μm

- In case of low density polyethylene (LDPE) we see that the nanoindentation resultant curves obtained at different amplitude keeping all other parameters constant. While if we consider Xydar® G-930, resultant curves obtained for different tests keeping all the parameters constant.

Since the nanoindentation for LDPE is performed at various amplitudes. That's why they show different behavior for hardness and modulus

- In case of UHMWPE single test is performed when all the parameters are constant. It can be seen from the resultant curves that this polymer is softer in nature as compared to Xydar® G-930.
- For PC there is also single test performed when all parameters are same. It shows the same behavior as Xydar® G-930.
- For PS single test performed when all parameters same. It can be seen that for hardness and modulus there are some fluctuations in the resultant curves.
- In case of PMMA there is also single test performed. It shows similar behavior as Xydar® G-930.
- The main difference of the results is that the displacement range for Xydar® G-930 and LDPE started from zero while for UHMWPE, PC, PMMA and PS the displacement range started from 10nm. That's why different behavior is observed.

4.5 SOCIOECONOMIC CONSIDERATIONS

4.5.1 Social impacts

Present day headway in innovation has improved great personal satisfaction in the general public. LC polymers have assumed a huge job in this rising headway. They are obvious in the cutting edge complex clinical, transport, sensor, and agrarian gear now in the market which are comprised of metals and modified polymers.

Replacement of human parts with polymer, for example prosthetic limbs, knee, implant, and the new era of drug delivery. LCPs are presently changed by scientist and these alterations modify their physical and synthetic properties for different cultural needs, for example, sensors. These sensors are used in ecological observing for poisonous gases, contamination in stream and portable water. The sensors are likewise utilized in process plants for quality control. Xydar® G-930 has the accompanying social impacts insight movies, cylinders and blow molded items. In medical applications, a distinctive endoscopic surgical instrument with a polymeric tube (Liquid Crystal Polymer) joining the handle actuator to the surgical gadget.

Some more interesting uses of Xydar® G-930 incorporate lightweight pipe that is inalienably fire resistant, tubing for cryogenic liquids, hazardous chemicals, other mechanical channel applications, strong tubing for automotive fluid framework, basic tubing for outdoor supplies and recreational gear.

LCP covers are used for small chip bundling and are expected to replace polyimide film and thin fiberglass-epoxy for chip size bundling. For flexible printed circuit boards, as well as high frequency digital and microwave circuit boards, LCP should compete really well polyimide and fluoropolymers [46].

4.5.2 Economic impacts

Cost Estimation of Polymeric sheet:

Raw material (Xydar® G-930) cost

Thickness of the sheet = 10mm

Cost of 1kg bag = 3830PKR

Cost of 1gram raw material = 3.83PKR

When volume is 16.875cm^3 - mass required to fabricate sheet is 27grams.

When volume is 50cm^3 - mass required to fabricate sheet is 80grams.

Table: 3 Cost estimation of sheet

Raw material	Volume (cm ³)	Cost (PKR)
Xydar® G-930	16.875	103.5
	50	306.5

4.5.3 Cost Benefit Analysis

All types of LCPs used in thin multilayer construction compete well with other materials like ethylene vinyl alcohol (EVOH), high density polyethylene, low density polyethylene (LDPE), polyvinyl chloride (PVC), polyethylene terephthalate (PET) and other materials. But if we have a look on the cost the price of LCP is higher than the other materials but Xydar® G-930 is relatively less costly as compared to other LCPs.

We can use the LCP with other materials combined because a thin layer of LCP has great positive effects on its applications. Special extrusion dies have been built to coextrude and orient LCP with other thermoplastics [46].

1. At relative humidity 85 percent, the oxygen barrier of LCP is 6-8 times greater than that of EVOH, although LCP is three to four times more expensive. The application of the LCP results in a net material cost savings of 33-50 percent. The key to ensuring the savings is to make the LCP as thin as 6 micrometres.

2. A three-layer structure was coextruded, yielding PET-tie layer-LCP structures with a total thickness of 25 to 50 micrometres and an LCP content of 10 to 30%. These coatings offer outstanding water vapour barrier characteristics. LCP layer can be coextruded with other thermoplastics to produce high-performance, low-cost films.
3. LCPs may be manufactured into thin liners for compressed and liquefied natural gas tanks by biaxial film manufacturing, substituting thick and high-density polyethylene (HDPE) liners or pricey aluminium liners. An LCP liner only 0.051mm thick provides ten times the barrier of an HDPE liner 9 -13mm thick while weighing one-tenth the weight and saving thirty percent on material costs.
4. Due to issues with melting, recycling, and burning of PVDC, users of high barrier films are actively seeking for alternatives to PVDC as a barrier layer. LCP laminates and extruded films are being studied since research shows that they may be burnt as readily as PET, and because of their strong barrier qualities, the LCP layer will be 4 to 5 times thinner than PVDC for equal barrier performance.

LC polymers are significantly more expensive than normal thermoplastics, yet they offer excellent mechanical, electrical, and chemical resistance. They are used in a variety of sectors for extremely demanding applications. Bulletproof vests, tennis strings, hockey sticks (as a composite), snowboards, tyre reinforcement, jet engine enclosures, brake and gearbox friction components, and gaskets are just a few examples.

Chapter 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

In comparison with other polymers as discussed earlier Xydar® G-930 exhibits:

- Much stiffer behavior
- Displayed shorter line in creep deformation region
- Less deformation observed as compared to other polymers (higher Modulus of Elasticity)

Because of its outstanding characteristics, Xydar® G-930 may replace other materials such as metals, ceramics, and composites. A thorough cost-benefit analysis reveals that we can employ Liquid Crystal Polymers (particularly Xydar® G-930) in the manufacturing/process industries as well as the biomedical sector.

According to the results, Xydar® G-930 has a high hardness. Even as the penetration depth rises, the hardness does not decrease. This demonstrates that the material has a hard nature since it exhibits hardness to its depth.

This leads to the conclusion that it is capable of replacing high-strength polymers, which are less cost-effective and have a shorter lifespan under extreme temperature and pressure circumstances.

Furthermore, because of its chemical resistance, it may be employed in toxic chemicals storage, toxic chemical handling, and the fabrication of protective gear.

Although Liquid Crystal Polymer is costly, the end effects are unparalleled. The primary goal of our FYP was to characterize Xydar® G-930 in order to evaluate its nanomechanical characteristics under severe temperature and pressure conditions.

5.2 RECOMMENDATIONS

After extensive investigation, the following recommendations may be the best possible:

1. We recommend using Xydar® G-930 tubes instead of alternative high strength polymeric tubes for handling/processing hazardous substances such as cryogenic fluids or corrosive compounds.
2. Rigid and structural tubes should be manufactured of LC polymer, such as Xydar® G-930, because these tubes perform well in the automotive sector.
3. We strongly advise installing oxygen barriers comprised of LC polymers since the OB of LCP is 6-8 times that of EVOH. Although money is a major consideration, the end results are amazing.
4. Because LC polymer may be coextruded with polymers such as PET, PVC, and EVOH, LCP is extremely cost effective.
5. LCP may fill unusually thin walls over extended stream lengths with little or no flash, even at mold temperatures below 200 °F.

6. A 10-15% concentration of LC polymers in the blow molding process, mixing, and alloy creation improves the quality of finished products.
7. We recommend to use Xydar® G-930 in protective gears to prevent from toxic chemicals as well as for the storage of toxic chemicals.

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