Chapter – 2

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2.1 **Introduction:**

Structure and morphology of polyester yarns are extensively investigated and most of these works are related to the apparel grade polyester yarn where the intrinsic viscosity (IV) of the polymer is around 0.60. High tenacity polyester industrial yarns are produced from the polymer having intrinsic viscosity of 0.90 or higher. Polyester industrial yarns for specific applications were developed and patented by the world’s leading technical yarn manufacturers. Information relating to structure, morphology and properties of these polyester yarns are mostly available in patents only. No comprehensive research work is available in public domain which deals with the structure-property relationship of different types of polyester industrial yarns. Therefore, the present research work is essentially focused in this exclusive domain. This chapter deals with the literature review concerning the current project on “structure-property relationship of various types of polyester industrial yarns at different stages of its processing for specific application”.

The literature review on polyester fibre has been divided in the research areas, pertaining to

(a) Structure, morphology and properties of polyester yarns.
(b) Important downstream processes of the polyester industrial yarns and its influence on the structure and properties of the virgin yarns.
(c) Performance evaluation of the final products and its correlation with yarn / cord properties.

Even though the search for works either underway or already completed is focused chiefly on polyester yarn, it has been felt that it is worth to include a brief literature review on the other major industrial yarns which are also used for various technical textile applications. These fibres include nylon6, nylon66, aramid and ultra high molecular weight polyethylene.
2.2 Structure, Morphology and Properties of Nylon, Aramid and Ultra high molecular weight high density polyethylene yarns:

Nylon6 and nylon66 are the two most important and commercially successful yarns in the polyamide family. The stable crystal structure of both nylon6 and nylon66 is the α form and comprises stacks of sheets of planar hydrogen-bonded extended chain segments [1]. As spun nylon6 yarns contain a mixture of α and γ form of pseudohexagonal crystals. The perfection of crystals lattice and dimension of unit cell are significantly influenced by the processing conditions during spinning and drawing. With increasing draw temperature and draw ratio, γ form gets converted to α form [1-5]. Unlike nylon6, nylon66 does not have “directionality” of the molecules for formation of hydrogen bonds. Due to this, lattice disorders are less in nylon66 yarns [1].

Microstructural changes that occur during drawing and its relationship with the mechanical properties were investigated by researchers in the past [5-8]. The change in fibrillar structure in nylon6 yarns resulting from drawing and annealing were studied in detail by Murthy et al. [9]. The degree of orientation of the fibrils were found to be the same as that of crystallite orientation. With increase in draw ratio, diameter of the fibrils decreases while long period increases. Peterlin [10] showed that the extended interfibrillar tie molecules are the main factors for shrinkage of the yarns. Prevorsek et al. postulated that extended interfibrillar tie molecules in nylon / polyester yarns, may be considered as a separate phase which play an important role for its mechanical and thermal properties [11,12]. However, on this subject difference of opinion exists amongst researchers. The other group of scientists reasoned that mechanical properties of the yarn should be attributed primarily to the strength of the microfibrils [10]. Prevorsek et al. [13] determined the diffusion co-efficient of the amorphous phase of nylon6 yarns and attempted to correlate dye uptake with morphological characteristics of nylon fibres. Danford et al. [14] investigated the structure development of nylon66 yarn during melt spinning and compared the same to that of nylon6. Nylon66 was found to crystallize in the spin line to form α-triclinic structure. This behavior differs from that of nylon6 yarn (mixture of α and γ with pseudohexagonal structure). Jain et al.
studied the effect of thermal ageing on the changes in microstructure of nylon66 yarns and consequent effect on mechanical properties.

Aramid fibre was first introduced commercially by Du Pont in 1970s under the trade name of ‘Kevlar” (poly para-phenylene terephthalamide). It is known for having very high “strength to weight ratio” and used for reinforcement of high performance tyres, applications in composites, ballistics, ropes and protective textiles [16].

Para oriented aramid fibres belong to a class of material known as liquid crystalline polymers. These polymers are rigid and rod like and therefore, in solution under suitable conditions, they aggregate to form ordered domains in parallel arrays; the so called liquid crystalline form [17-19]. Aramid fibres are spun using the unique “Dry jet wet spinning” technology [16,20]. This facilitates an exceptional degree of alignment of long, straight polymer chains parallel to the fibre axis which is the key for obtaining the outstanding properties. The basic yarn morphology is the fibrillar structure like nylon or polyester. However, the degree of orientation and crystallinity are much higher [20-23] than nylon or polyester. The unique structure and morphology of Kevlar aramid fibre leads to very high tenacity and modulus, very low shrinkage and creep (therefore, excellent dimensional stability). However, it suffers from the limitations of inferior compression fatigue (compared to nylon / polyester) which is due to the compression buckling of the para-aramid molecules [16,21].

The only “flexible chain fibre”, which has been commercialized as high performance fibre is the polythene fibre produced from ultra high molecular weight high density polyethylene (UHMWHDPE) [16,24,25]. “Spectra” and “Dyneema” are the two popular polyethylene fibres available commercially. It is distinguished by exceptionally high strength and modulus. Due to its lower density (around 0.97 g/cc) than most of the textile fibres, it has the edge with respect to “strength to weight ratio” than any other fibres. For example, it has tenacity of over 30 gpd, which is significantly higher than aramid fibre (~ 22 gpd) and over 3 times higher than high tenacity nylon and polyester fibres. It has modulus close to 2000 gpd, which is higher than aramid (~ 1500 gpd) and over 100 times higher than nylon / polyester fibre [16].
The key aspect of achieving these outstanding properties are:
- Ultrahigh molecular weight polymer (\(M_w > 10^6\)).
- Unique “gel spinning” technology followed by hot drawing to develop the appropriate yarn morphology with very little chain folding. This leads to create a fibre with very high crystallinity (\(~70\%\) by WAXS) and high overall orientation [26-28].

The basic yarn morphology is the fibrillar structure like nylon / polyester. Structure and morphology play an important role for achieving the exceptional yarn properties [16,29-33]. Inherently it is a highly crystalline and oriented fibre. Due to its very high strength, modulus and impact strength, this fibre is found to be particularly useful in applications like bullet proof clothing and high performance rope. However, it suffers from the limitations of having low compressive strength and susceptibility to high temperature application [16,34,35].

2.3 Structure, Morphology and Properties of Polyester yarns:

Structure and morphology of the final drawn polyester yarn is developed during drawing and heat setting process, which has been described in section 1.2.3.3. A two-phase model depicting crystalline and amorphous phase of polyester yarn is used widely by several authors, which has also been used in the present work [36-38].

The two-phase structural model of an oriented polyester yarn is described in Figure 2.1. According to this model the ordered (crystalline) regions alternate with less ordered (amorphous) domains. “Tie molecules” pass through several crystalline and amorphous regions, thus providing the coherence within the fibre. During spinning and drawing process, the crystalline regions and the amorphous regions are oriented along the fibre axis and thus the so called “fibrils” are formed.
The crystalline regions may be regarded as regular stiff block as shown in Figure 2.1. On the other hand the amorphous regions are random in terms of orientation and length of the amorphous domain (Figure 2.2). Therefore, the randomness causes a non-uniform distribution of stresses over the molecules in the amorphous regions on mechanical loading. In the two-phase model structure of the fibre, the lack of molecular coherence makes the amorphous regions the weak spots in fibres. Extended non crystalline molecules present between the fibrils also contribute to the mechanical properties.

![Two-phase model structure of oriented polyester yarn](image1)

**Figure 2.1:** Two-phase model structure of oriented polyester yarn [36,38]

![High level and low level of orientation of the tie molecules in the amorphous domain](image2)

**Figure 2.2:** High level and low level of orientation of the tie molecules in the amorphous domain [36]
It is important to note that like mechanical properties, thermal properties (especially dimensional stability at wide range of temperatures) are predominantly governed by the amorphous regions. Upon heating, the oriented molecules tend to coil (enhanced entropy), resulting shrinkage of the yarn.

Studies on influence of spinning process parameters on yarn morphology and its relationship with key properties of polyester yarn have been conducted by several researchers [36,38-44]. Huisman and Hauvel [38] have discussed on the effect of spinning speed and drawing temperature on structure and properties of polyester yarns. It has been inferred that drawn yarn produced with high spinning speed leads to an increase in chain folding. Higher chain folding in undrawn yarn [38] leads to more number of tie molecules present in the drawn yarn. Since, HMLS polyester yarns are produced at a much higher speed (discussed in section 1.2.3.3.) than that of regular polyester yarns, HMLS polyester yarn is likely to possess higher fraction of tie molecules.

In another work, Desai and Abraham [39] studied the orientation distribution in the neighborhood of a growing crystal. It has been postulated that during crystallization of polymers, preferential transformation of the chain segment occurs. This is dictated by their orientation with respect to the growing crystals. This is an important aspect considering the different set of drawing and heat setting conditions which are applied to make different types of polyester industrial yarns. Few other authors studied the influence of spinning process parameters on the structure and morphology of polyester industrial yarns and its relationship with various properties [40-44]. These articles provide useful information on the formation of the structure during spinning and thread line modification.

studies on structure-property relationship in heat set polyester fibres [48-51]. Influence of different heat setting conditions (relax and taut annealing) on structure and morphology and its relationship with various properties have been discussed in detail in these papers. All these basic research works have been conducted with a laboratory set up and on moderate tenacity polyester yarns. However, fundamentals described in these papers with respect to structure-property relationship are found to be useful in the current study too.

Characterization of structure and morphology of polyester fibre / yarn is an interesting area of research and many works have been published in this field [54-62]. A detailed literature review has been conducted in order to understand and choose the appropriate techniques for analysis of microstructure of polyester yarn, required for the current study. Crystallinity and crystal size are determined using wide angle x-ray scattering (WAXS) as per the method described by Dumbleton et al. [54] and Gupta et al. [55]. Important structural characteristics of polyester yarns which have been proposed to be studied in the present work to establish a co-relation between structure, morphology and properties include the following.

- Crystallinity by wide angle x-ray diffraction (WAXS)
- Crystallinity by density gradient principle
- Crystal size
- Crystallite orientation
- Birefringence
- Amorphous orientation

Herman’s crystallite orientation factor of the polyester yarn is determined from the Azimuthal scan of the three equatorial planes viz. (010), (110) and (100) according to the method described by Gupta and Kumar [56]. Values of “intrinsic birefringence of the crystallite phase (Δc)” and “intrinsic birefringence of the amorphous phase (Δa)” have been considered as reported by Gupta and Kumar [57]. Details of the equations and the methods for these characterizations have been described in chapter 3.

Heuvel et al. [63] have studied the molecular changes of polyester yarn during stretching. Mechanism of stress development on the crystals and particularly on the tie molecules in the amorphous phase has been described in
their work. The influence of structural parameters like tie molecules, amorphous orientation on stress-strain behavior of polyester yarns has been reported by several authors [64-67].

Kunugi et al. [68] studied the mechanical properties and super structure of high modulus and high strength PET fibres and had tried to investigate the influence of the amorphous phase on such properties of the polyester fibre. It has been understood that the orientation of the chains in the amorphous phase, the number and fraction of tie molecules play an important role on mechanical properties of the polyester yarn. The following empirical equation has been used to determine the amorphous modulus ($E_a$) of PET fibre.

$$\frac{1}{E} = \frac{V_c}{E_c} + \frac{1 - V_c}{E_a} \quad (2.i)$$

$E$ is the as-measured macro modulus. $E_c$ is the crystal modulus along the molecular chains and $V_c$ is the percent crystallinity (or volume fraction of crystallinity). $E_c$ is a constant, the reported value being 107.8 GPa. $E_a$ (amorphous modulus) of polyester fibre for a given crystallinity depends upon the morphology of the yarn, especially amorphous orientation and fraction of tie molecules. Fraction of tie molecules has been estimated as $\beta_E = E / E_c$. These inputs were found to be useful for interpretation of the results pertaining to structural parameters and properties of polyester yarns.

In the area of development of dimensionally stable polyester yarns, various structural parameters have been discussed in few patents [69-71]. Buyalos et al. [70] have patented the development of high strength polyester yarn for improved fatigue resistance. It has been emphasized that intrinsic viscosity (min 0.90) of the chips and the optimized spinning process conditions are the key aspects required to achieve dimensionally stable polyester yarn which has relatively low work loss and improved fatigue life.
2.4 **Influence of twist on properties and performance of cord:**

An outline of the downstream processes of polyester industrial yarns viz. twisting and weaving has been described in section 1.4. This section deals with twist and its influence on the properties and performance of the cord. “Helix angle” of twist has been described in Figure 2.3. If “T” is the length of one twist along the axis of the cord, then twist per unit length is 1/T, which is expressed at “Twist per meter (TPM)”. As twist per unit length increases, “helix angle” of the cord increases. When tensile force is applied along the axis of the yarn, the component of the force (Cosδ) along the yarn axis decreases with increase in helix angle. This leads to increase in elongation at break and decrease in initial modulus & strength of the cord, resulting lower “conversion efficiency [72-74].” “Conversion efficiency” of the twisting process is defined as follows.

\[
\text{Conversion Efficiency (\%)} = \frac{\text{Breaking strength of cord}}{\text{Breaking strength of yarn} \times \text{No. of plies}} \times 100
\]

Generally, conversion efficiency of tyre cord is around 90-92%. If the conversion efficiency is lower than this, it indicates higher loss of yarn strength during twisting / weaving process and it calls for checking the yarn path & guides to find out the causes for lower conversion efficiency.

![Helix angle of the twisted cord](image)

**Figure 2.3:** Helix angle of the twisted cord [73]

Twist plays a critical role for the product like tyre, which undergoes fatigue during service and therefore, influences the service life of the product. Durability
of the tyre is significantly influenced by the fatigue performance of the rubber-cord composite. Fritsch [75] has nicely presented the relationship of various properties (viz. tenacity, modulus and fatigue) with twist in a single graph as shown in chapter 1 (Figure 1.4).

Tenacity of the virgin greige cord is decreased as twist increases. However, fatigue performance is enhanced with increase in twist upto certain optimum level. Jackson [76] has explained this phenomenon with “helix angle” and inferred that optimum twist level to achieve the best fatigue performance varies significantly with cord thickness. As cord denier / thickness increases, optimum twist (TPM) decreases. However, “helix angle” corresponding to the optimum twist level is found to be similar for different constructions of cords [76].

Many research works have been carried out in the area of “hybrid cord” where “twist” plays a vital role for the performance of the hybrid cord [77-82]. Applications of these hybrid cords are aimed for high performance tyres where fatigue performance is one of the most important characteristics. Different combinations of “hybrid cords” viz. Polyester / Nylon66, Aramid / Nylon66 have been explored by many researchers.

2.5 Dipping of polyester tyre cord fabric:

For applications in rubber-cord composites like tyre and conveyor belt, the desired level of adhesion between cord and rubber is essential to maintain its integrity during service. Rubber-cord adhesion is one of the most important factors which influences the service life of the composite products like tyre, conveyor belt etc. The classical way to achieve good rubber-cord adhesion is to impregnate the greige polyester fabric in a dip solution followed by drying and heat setting. For dipping of tyre cord fabric made out of HMLS polyester yarn (non adhesive activated), a double dip system is necessary. For adhesive activated type of polyester yarn single stage dip recipe is adequate. In the present study, only HT polyester yarn is adhesive activated type, whereas all others are non-adhesive activated type of polyester yarns. In section 1.4, a brief of the dipping process has been described. Formulation of the dip solution has been illustrated in section
3.3.4, which has been used for experimentation in the present work. This section of the literature review covers the research works in the following areas:

- Dipping system and mechanism of adhesion between polyester and rubber.
- Factors influencing rubber-cord adhesion.
- Effect of dipping process conditions on structure, properties and performance of the dipped cord.

2.5.1 *Dipping system and mechanism of adhesion*:

Adhesive system for rubber-cord composite has been studied by many researchers in the past [83-86]. Takeyama et al. [83] and Solomon [84] have studied the basic formulation of the dip solution and mechanism of adhesion elaborately. Resorcinol formaldehyde latex (RFL) based dip recipe is being used in the industry for over half a century. Lot of research has been done to develop an alternate adhesive system for rubber-cord composite which is superior and cost effective. However, till now, no commercially viable adhesive system could be developed which can replace RFL. It is important to note that the practical challenge lying with the manufacturers is to achieve the desired level of adhesion along with other target properties of dipped cords. Several factors in the upstream and downstream processes affect cord-rubber adhesion, which would be discussed in the subsequent sections.

Since polyester is non-polar in nature, it is more challenging to achieve the desired level of adhesion with non-polar rubber. Therefore, special chemicals like isocyanate and / or epoxy are used for dipping of polyester tyre cord fabrics. For dipping of tyre cord fabric made out of non adhesive activated type of polyester yarn, the dip unit should have the features of double dip bath system. The first dip bath contains special dip chemical (blocked isocyanate and epoxy) while the second dip bath contains regular RFL dip solution. After predip in the first dip bath, the fabric is dried (130-140°C) followed by heat setting at a higher temperature (around 230°C), to unblock the isocyanate. This is essential to facilitate bond formation between epoxy and isocyanate. In case of adhesive
activated type of polyester yarn, epoxy is added along with the spin finish during manufacturing of yarn. Therefore, a single dip bath system is adequate for dipping of fabric (for example belting fabric) made out of adhesive activated type of polyester yarn. Only isocyanate is required to add into the regular RFL dip solution [84].

Several interfaces are present in a rubber-cord composite [87] as described in Figure 2.4. Bonding mechanism between polyester and rubber has been postulated by the researchers [88-90]. A model study on reinforcement mechanism was carried out by Xue et al. [89]. It has been emphasized that both carboxyl and hydroxyl group of polyester can react with epoxy group present in first dip. Kurz [90] had depicted the chemical bond formation of both epoxy and isocyanate with polyester (Figure 2.5). A chemical bond is also formed between RFL and epoxy. The latex component present in the second dip, forms chemical bond with the rubber during curing (co-vulcanized) while making the rubber-cord composite.

![Figure 2.4: Interfaces between cord and dip](image1)

![Figure 2.5: Bonding mechanism of polyester cord with dip coat](image2)
The cross section of a polyester dipped cord, (Figure 2.6) indicates that the dip solution penetrates by only 2-3 filaments inside the cord which is adequate to provide the desired level of adhesion between cord and rubber. Dip pick-up of polyester tyre cord is around 2-3%. However, dipping in a pilot dipping unit leads to a bit higher dip pick up (~ 4%). The contribution of the first dip on total pick-up is only around 0.5%, and the rest is contributed by the second dip (RFL). A good “rubber coverage” is desired. A good coverage means that the interfacial bond strength (between RFL and rubber) is higher and the failure has happened within rubber. On the contrary, if the adhesion tested cord looks “pink”, it indicates the failure of the bond between RFL and rubber (Figure 2.7) which is not desired.

![Figure 2.6: Cross section of polyester dipped cord [83]](image1)

![Figure 2.7: Rubber coverage of adhesion tested polyester cord](image2)

Few research works have been published on the development of dip recipe for hybrid cord [78] and improvement of strength retention of the cord post cure [91-92].

2.5.2 Factors influencing rubber-cord adhesion:

Appropriate dip solution alone is not adequate to achieve the desired level of adhesion between polyester fabric and rubber. Time, Temperature and Tension (3 Ts) are the three key parameters of the dipping process, which need to be optimized to achieve the desired level of adhesion and other target properties of the dipped cord. A broad dipping process conditions have been provided in Table 1.3 (Chapter 1). Bhakuni et al. [93] has described the influence of dipping process conditions on adhesion and other properties of dipped cord.
Published research studies are available on various factors which adversely affect the rubber-cord adhesion viz. rubber compounding ingredients [94-98], atmospheric pollutants [99-102] etc. Iyengar [94] has emphasized that the adhesion retention (high temperature ageing test) of polyester tyre cord with rubber is adversely affected by the amines present in the rubber compounding ingredients (curatives) and the moisture content in the rubber stock. Non amine type of accelerators is recommended for polyester based skim compound. In another article, Wennekes et al. [98] has emphasized the influence of rubber curatives on adhesion of polyester tyre cords. Hartz [101] has highlighted that ozone alone and in combination with ultraviolet light has the most predominant adverse impact on rubber-cord adhesion. Mechanism of loss of adhesion has been explained as due to the attack of olefinic double bond of latex by ozone. This leads to depletion of the reactive sites required for co-vulcanization with rubber during curing of rubber-cord composite. Exposure of the dipped fabric in atmosphere should be avoided to retain its usable shelf life. Porter [99] has emphasized the adverse impact of metals and metallic salts present in water which is used for preparation of dip solution. Metals and its salts which have worst influence on adhesion are magnesium nitrate and cadmium chloride. Usage of DM water and routine quality checks of the water have been recommended [99].

2.5.3 Effect of dipping process conditions on structure, properties and performance of the dipped cord:

As mentioned in the preceding section in this chapter, that the fabric impregnated in dip solution is first dried at around 130-140°C, followed by heat setting at elevated temperature (230-240°C). Dipping process parameters (especially 3 Ts) need to be optimized to achieve the desired level of adhesion and other target properties of the dipped cord. Few research works have been published on planned experimentation [93,103,104] for establishing the relationship between dipping process parameters and dipped cord properties. Trask and Promies [103] have conducted a design of experiments and discussed the relationship between dipped cord properties (strength retention, part load
elongation and shrinkage) and important dipping process parameters (stretch, temperature and residence time in heating / normalizing zone). It has been highlighted that the heat setting temperature plays a major role in controlling stiffness of the cord which has an impact on the fatigue performance of the dipped cord. Bhakuni [93] has highlighted that excessive stretch in heat set zone and excessive relaxation in normalizing zone adversely affect the fatigue life of the cord. However, no literature could be found with regard to the influence of dipping process parameters on structure and morphology of dipped cord.

2.6 Degradation of polyester yarn / cord in rubber-cord composite:

For applications like tyre / conveyor belt, etc., retention of strength and other properties of the cord, post cure and during service are very important, because these are associated with the performance of the final products. There is degradation of polyester in rubber-cord composite. The mechanism of degradation of polyester is divided in two categories viz. (a) chemical degradation, which generally happens due to hydrolysis and sometimes catalyzed by aminolysis and (b) mechanical degradation, which is primarily due to fatigue. These are discussed in detail in the subsequent sections.

2.6.1 Chemical degradation of polyester yarn:

Chemical degradation of tyre cord in rubber has been studied by many researchers [96,105-110] in the past. Weighmann and Lamb [107] have emphasized the adverse influence of sulfonamide and thiuram type of accelerators on degradation of polyester due to aminolysis. Sawada et al. [108] have illustrated hydrolysis of polyester, catalyzed by amines. The detailed study indicates that prolonged heat exposure leads to breakage of ester bonds, resulting in decrease of strength of the polyester cord. Sawada has carried out lab experiments on polyester cord and rubber-cord composite by exposing it under thermal ageing at varied humidity. The major cause for degradation of polyester was found to be the
hydrolysis. This degradation becomes faster in presence of amines, present in the rubber compounding ingredients.

Kamiyama et al. [109] have reported the usage of a compound containing cyclic imino ether group to minimize / inhibit chemical degradation of polyester tyre cord in rubber. Hisaki et al. [92] used carboxylated latex based on conventional VP-Latex for improving both adhesion and strength retention post cure. The purpose of using carboxylated VP-Latex was to react with the amines of the rubber compounding chemicals and thus preventing polyester from degradation.

One of the aspects of the present work is to study the structure-property relationship of polyester yarn at cured cord stage. It has been planned to measure the carboxyl group number of the polyester cord (HMLS and HT), taken out from the rubber-cord composites. However, determination of these characteristics on rubberized cords is a cumbersome process. The first step is to separate out the polymer (polyester) from rubberized cord by dissolving it in a suitable solvent, followed by separation of the rubber particles by filtration and then precipitation of the dissolved polymer in a non-solvent [110]. No literature could be found on the method for determination of crystallinity of cured / fatigued cord. Presence of rubber (which is amorphous and thus likely to increase the amorphous phase contribution in x-ray diffraction scan) makes it difficult to measure crystallinity by wide angle x-ray scattering or by density method.

2.6.2 Mechanical degradation of polyester in rubber-cord composites:

Mechanical degradation here primarily refers to the degradation due to fatigue. Therefore, in the current context, it is relevant to study the mechanical degradation of HMLS and HT polyester tyre yarn / cord which are subjected to fatigue during service of a tyre or conveyor belt in particular. Many research works have been conducted on fatigue characteristics of reinforcing textiles which can be broadly divided into two categories viz. (a) works related to technique / methods for fatigue characterization and (b) mechanism of fatigue.
A laboratory test for fatigue characterizations of reinforcing textile is one of the very important evaluations since it is related to the field performance of the final products. However, no laboratory test method of fatigue characterization can truly simulate the complex scenario of the service conditions of a tyre or conveyor belt. Different test methods are followed by companies [111-113] across the world for their own in-house evaluation. Results of the fatigue test are expressed either in terms of the number of cycles till failure of the rubber-cord composite or strength retention of the reinforcing textile after fixed number of fatigue cycles. The test parameters used in a laboratory test are generally accelerated in nature and therefore, the results of the laboratory tests are mainly utilized for relative evaluation for developmental purpose.

Major laboratory test methods used for fatigue characterization of reinforcing textile are given below:

(a) Mallory tube fatigue tester [111,112]
(b) Scott flex fatigue tester (ASTM D430-73)
(c) Compression Tension (CT) disc flex fatigue tester (ASTM D6588M-11) [93,110,114,115]

Fatigue testers based on other principles have also been reported viz. “Shear Compression Elongation Fatigue” (SCEF) [113].

Mallory tube fatigue tester and compression-tension flex fatigue testers are the two most widely used test methods across the tyre industries, while scott flex fatigue tester is mostly used in the conveyor belt industry. In the present study, for HMLS polyester industrial yarn (which is primarily used for passenger radial tyres), compression-tension disc fatigue tester has been used. Scott flex fatigue tester has been used for fatigue characterization HT polyester industrial yarn, which is mostly used for fatigue testing of conveyor belt.

Mechanism of fatigue of reinforcing textile in rubber-cord composite has been studied by many researchers [116-119]. Cord-rubber interface failure during dynamic fatigue plays a crucial role on the fatigue life of the rubber-cord composites. Padovan [116] has investigated the cord-matrix load transfer problem in cord-rubber composite. Du et al. [117] have emphasized the impact of volume fraction of the reinforcement and the effect of cord angle in rubber-cord
composite. Directional effects on loss of cord strength in bias tyre have been reported by Forster et al. [113] which indicates that a more rapid loss of strength occurs in the leading portion of the cord (which enters the footprint region first). It has been also highlighted that in radial tyres, cords in the carcass experience mostly tension generated by the rotational shearing, regardless of the direction of rotation of the ply.

Wahl [118] has nicely presented the scenario of dynamic load, experienced by different components of the tyre during its service and has explained the mechanism of fatigue of rubber-cord composite which are discussed below.

Figure 2.8: Dynamic load experienced by different reinforcements during service of a passenger radial tyre [118]

Figure 2.9: Structure rearrangement of the cord during fatigue [118]
**Figure 2.8** illustrates that the carcass of a passenger radial tyre which consists of HMLS polyester yarn, is subjected mostly under tension (very little under compression) during service. **Figure 2.9** describes the structural rearrangement of the cord during fatigue of a rubber-cord composite. Twisted structure of the cord leads to axial compression and shear strain at the interface which are depicted in **Figure 2.10** and **Figure 2.11** respectively.

**Figure 2.10:** Axial compression mechanism of rubber-cord composite under fatigue [118]

**Figure 2.11:** Shear strain mechanism of cord-rubber composite under fatigue [118]

Axial compression mechanism explains the important role of the interface. Initiation of the adhesion failure at the cord-rubber interface leads to open structure and finally fatigue failure. Shear strain mechanism (Figure 2.11) emphasized that the high shear strain between edge of separating ply and surrounding matrix leads to crack initiating points during compression cycle of the composite.
Scanning Electron Microscopic (SEM) examination of the broken ends of the failed cord indicates that “bias breaks” of the filaments are the most frequent mode of fatigue failure \cite{64,69,73} as shown in Figure 2.12.

![SEM photo](image1)

Tensile break (Tensile test)  
Bias break (due to fatigue)  

**Figure 2.12:** SEM photographs of the filament breaks: normal tensile break and bias break (due to fatigue) \cite{64}

In case of conveyor belt, a multiple ply belt when passed through end pulleys, the top ply experiences primarily tension fatigue and the bottom ply experiences higher extent of compression fatigue. Assaad \cite{120} has studied on fatigue characteristic of rubber-cord composite made out of multiple ply of fabrics and has emphasized the differences in fatigue performance between top and the bottom ply. All these inputs are found to be useful for deciding the test parameters for fatigue characterizations of cords made out of HMLS and HT polyester yarns.

In the recent years too, research works have been published in the area of fatigue performance of reinforcing textiles \cite{80,82,110,121,122}. Cho et al. \cite{121} has reported the influence of fine structure on fatigue resistance of polyester tyre yarn. Fatigue performance of the polyester yarns having smaller long period has been reported superior. Naskar et al. \cite{110} has highlighted the drop in intrinsic viscosity of the cord after fatigue. Sawada et al. \cite{108} has inferred that the degradation of polyester due to fatigue in a rubber-cord composite is attributed to amine catalyzed hydrolysis apart from mechanical rupture of the filament due to fatigue. This leads to increase in carboxyl group number and decrease in intrinsic viscosity of polyester.
2.7 **Relationship between properties of polyester industrial yarns and performance characteristics of the final products:**

Each type of polyester industrial yarn is tailor made to meet the performance requirements of the final products. Three major product segments viz. (a) passenger radial tyre (b) conveyor belt and (c) coated fabric have been discussed in this section. Different types of polyester industrial yarns taken in the current study are used mostly in these product segments. It is worth to understand the relationship between properties of various types of polyester industrial yarns and the performance characteristics of the final products.

It has been mentioned previously that HMLS polyester yarn is primarily used as carcass (body ply) in passenger radial tyre. Cross section of a passenger radial tyre showing major components of the tyre has been described in section 1.4.1.2. Suitability of various reinforcing textiles for passenger radial tyres was reviewed by many authors in the past [75,123-128]. In the recent years, developments in tyre technology are focused towards improvement in rolling loss, heat buildup and ride & handle characteristics of tyre [125,126]. Dimensional stability at high speed is the most critical requirement of the passenger radial tyres which is influenced primarily by the modulus and shrinkage characteristics of the polyester tyre cord used in the carcass. Modulus of the carcass cord at different extent of strains is very important considering tyre manufacturing as well as tyre performance. Mukhopadhyay [129] depicted the ideal stress-strain curve of the reinforcing textile to satisfy the requirements of the tyre manufacturing process as well as the performance during service (Figure 2.13). During manufacturing process of passenger radial tyre, the carcass cord should provide reasonable strain. However, during service, the reinforcing cord should provide higher modulus to achieve the desired service requirements viz. rolling resistance, durability, comfort and handling.
Stress-strain curves of major reinforcing materials [84] have been depicted in Figure 2.14. It may be noted that no single reinforcing material can match the ideal stress-strain curve as described above. Development of the tailor made HMLS polyester industrial yarn is towards meeting this requirement. Studies on the stress-strain curves of the different types of polyester industrial yarns and its linkages with its structural characteristics assume paramount importance in the present work which has been discussed elaborately in Chapter 4.

Figure 2.13: Ideal stress-strain curve of reinforcing textile (carcass) for a high speed passenger radial tyre [129]

Figure 2.14: Stress-strain curves of the major reinforcing materials [84]
As mentioned above, few desired aspects of the service performance of the tyres are lower rolling loss, lower heat build-up, durability, ride and comfort. Each of these performance characteristics of the tyre has relationship with the properties of the reinforcing textile. “Rolling loss” is the mechanical energy converted into heat by a tyre moving for a unit distance on the roadway [130]. Rolling loss of tyre is attributed to dynamic properties of the rubber-cord composites and hence, dynamic property (tanδ) becomes an important characteristic of the HMLS polyester yarn which is used in passenger radial tyre. It is to be noted that tanδ also influences on wet traction (especially important for tyre grip in cold countries - below 0°C). Lower tanδ is desired considering lower heat build up [130-133], however, too low tanδ adversely affects wet grip [134-136].

“Heat build up” during running conditions not only influences rolling loss but also adversely affects fatigue characteristic and hence durability. One of the major reasons of service failure of tyre is the separation of the cord from the rubber matrix due to failure of the dynamic adhesion which is attributed to fatigue and heat build-up. Hysteresis characteristic (measured as work loss) of the rubber-cord composite is attributed to heat build-up. Tanδ is related to dynamic test, which is performed at high speed (10 to 25 Hz) whereas hysteresis (work loss) is related to slow speed test, generally performed at a speed of 5 to 10 cycles / min. Therefore, tanδ characteristic is more important and relevant for HMLS polyester yarn, whereas hysteresis characteristic assumes more relevance for HT polyester yarn which is used in conveyor belt (which is subjected to cyclic loading at a lower frequency).

Many reports [137-144] have been published on rubber-cord composites and on properties of polyester yarn / cord which have linkages with the tyre performances. Lamb et al. [137] have estimated the temperature rise at the cord-rubber interface at different parts of the tyre viz. shoulder, crown and side wall regions. The study on dynamic properties was conducted in laboratory on cord-rubber composites. Dynamic properties were measured at 20 Hz (equivalent to a speed of around 80 mph) for passenger tyres. Maximum temperature of around 100°C at the shoulder region has been reported. Kwon et al. [138] has also studied
the viscoelastic properties of tyre cords and rise in tyre temperature. Rolling resistance has been determined in this study which also illustrated the influence of viscoelastic properties on rolling resistance and fatigue endurance. Lim et al. [139] have studied on tensile properties of various tyre cords in laboratory simulating tyre curing and post cure inflation (PCI) conditions. It has been inferred that post cure inflation had an effect on increasing modulus of carcass cords in tyre and it must be carried out below glass transition temperature of the carcass cord. Papero et al. [141] have studied on mechanism of flat spotting of tyres and its relationship with cord properties. Power [144] has conducted laboratory evaluation of hysteresis characteristic of tyre cord at different “extent of stress relief” by simulating the tyre service conditions. In this work, it has been shown that work loss increases exponentially with increase in “extent of stress relief”. Inputs from this study have been taken for conducting hysteresis characterization of HMLS polyester tyre cord.

There are not many research papers available on HT, LS / SLS polyester yarn with special reference to conveyor belt and coated fabric applications. Farboodmanesh et al. [145] have studied on the effect of fabric construction on its mechanical behaviour which has some relevance on coated fabric made out of polyester yarn. Lachmann [146] has studied on hysteresis characteristic of polyester cord considering the service conditions of conveyor belt where the peak load for the hysteresis test was as high as 40% of the breaking load of the cord.

A relationship matrix between key product performances and the properties of the different types of polyester industrial yarns are summarized in Table 2.1.
As mentioned previously, three key product segments viz. passenger radial tyre, conveyor belt and coated fabric have been considered for making this relationship matrix. One the endeavours of the present work is to establish a linkage between these properties of the polyester yarns and its structural characteristics.

Table 2.1: Relationship Matrix: Key product performance vs properties of polyester industrial yarns

<table>
<thead>
<tr>
<th>Key product segments</th>
<th>Key product performance</th>
<th>Properties of polyester industrial yarns</th>
</tr>
</thead>
<tbody>
<tr>
<td>Passenger radial tyre</td>
<td>Dimensional stability</td>
<td>Shrinkage and modulus</td>
</tr>
<tr>
<td></td>
<td>Lower heat generation</td>
<td>Hysteresis and loss tangent</td>
</tr>
<tr>
<td></td>
<td>Lower rolling resistance</td>
<td>Hysteresis and loss tangent</td>
</tr>
<tr>
<td></td>
<td>Endurance</td>
<td>Fatigue</td>
</tr>
<tr>
<td>Conveyor belt</td>
<td>Full belt tensile strength (FBTS)</td>
<td>Tensile strength and heat resistance</td>
</tr>
<tr>
<td></td>
<td>Troughability</td>
<td>Modulus, elongation and glass transition temperature</td>
</tr>
<tr>
<td></td>
<td>Lower growth</td>
<td>Creep and hysteresis</td>
</tr>
<tr>
<td></td>
<td>Endurance</td>
<td>Tensile strength and fatigue</td>
</tr>
<tr>
<td>Coated fabrics</td>
<td>Dimensional stability</td>
<td>Shrinkage, modulus and creep</td>
</tr>
<tr>
<td></td>
<td>Tear strength</td>
<td>Tensile strength, modulus</td>
</tr>
<tr>
<td></td>
<td>Durability</td>
<td>Heat resistance, weather resistance</td>
</tr>
</tbody>
</table>
2.8 References:


