Effects of Changing Environment and Loading Speed on Mechanical Behavior of FRP Composites

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ABSTRACT

Durability of fiber reinforced polymer composites (FRP) are controlled by the durability of their constituents: reinforcement fibers, resin matrices, and the status of interfaces. A great deal of research has been focused on attempting to assess the relationship between interfacial structure and properties of fiber-matrix composites. It is at the interfacial area where stress concentration develops because of differences in the thermal expansion coefficients between the reinforcement and the matrix phase. A significant mismatch in the environmentally induced degradation of matrix and fiber leads to the evolution of localized stress and strain fields in the FRP composite. The present investigation aims to study the effects of changing hygrothermal conditioning cycles (either by changing relative humidity and temperature is kept constant, or by changing temperature but relative humidity is maintained same) on moisture gain/loss kinetics and on interlaminar shear strength (ILSS) of varied weight fractions glass fiber reinforced epoxy and polyester matrices composites. The mechanical assessment is extended to evaluate the loading rate sensitivity of hygrothermally shocked glass/epoxy and glass/polyester laminates at 2mm/min and 50mm/min crosshead speeds. Observations on absorption/desorption kinetics are noticed to be dependent on nature of hygrothermal shock cycle and on weight fraction of fiber reinforcement. Results of mechanical performance are statistically significant at different stages of conditionings. Shear values are found to be greater at higher crosshead speed for all undertaken situations. Mechanical responses are observed to be dependent on matrix resin and type of hygrothermal shock cycle. Very little and limited literature is open to address the important interactions of polymer composites with this kind of realistic environmental situations.

KEY WORDS: glass fiber, epoxy, polyester, hygrothermal shock, absorption/desorption, Mechanical behavior, crosshead speed.

INTRODUCTION

The bond properties especially the bond strength during service period deteriorates depending on the environmental conditions [1]. Both short-term and long-term properties of a composite depend decisively on the microstructure and properties of the interface or interphase between the fiber and the matrix [2]. The recent analysis of FTIR images suggests that there is a variation in chemical structure of the matrix resin from the fiber to the polymer bulk due to different conversions arising from a gradient in the initial composition [3]. The position, form and intensity of spectral bands reveal useful information about the microstructure of polymer at a molecular level [4]. The rate of degradation of mechanical properties of a composite laminate could be higher than that of the individual constituents due to the synergy among the different degradation mechanisms [5]. The strength of composite materials depends not only on the substrate strength but also on the interface strength. The interface here does not have unique fracture energy unlike homogeneous materials [6]. The adhesive fibers elongate in a stepwise manner as folded domains are pulled open. The elongation events occur for forces of a few hundred piconewton. These are smaller than the forces of over a nanonewton, which are required to break the polymer backbone. When the force rises to a significant fraction of the force required to break a strong bond and threatens to break the backbone of the molecule, a domain unfolds. Thus, it could avoid the breaking of a strong bond in the backbone. These may represent the cumulative effect of multiple intra- and inter-chain bonds acting in concert [7].

All engineering plastics are affected by weather. Weather and radiation factors that contribute to degradation in plastics include temperature variations, moisture, sunlight, oxidation, microbiologic attack, and other environmental elements. Cyclic exposure is an important factor when considering the service environment of a composite material. Environmental conditions can promote brittle fracture in normally ductile plastics at levels of stress or strain well below those that could usually cause failure. Exposure to lowered temperature may cause a plastic to become brittle. Cracking and a propensity to fracture can occur. Exposure to elevated temperature can result in degradation of mechanical properties, cracking, chalking and flaking of polymers [8]. The first form of damage in laminated composite is usually matrix microcraks. These microcraks are transverse to the loading direction and are thus called transverse cracks. Matrix microcracks cause degradation in properties in composite laminates and also act as precursors to other forms of damage leading to laminate failure [9]. Delamination is a critical failure mode in composite structure. The interfacial separation caused by the delamination may lead to premature buckling of laminates, excessive intrusion of moisture and stiffness degradation. While in some cases a delamination may provide stress relief and actually enhance the performance of a composite component [10].

The structure of the resin matrix has potential to influence the uptake of water. It is well known that Fick's Law is frequently inadequate for describing moisture diffusion in polymers and polymer composites. Non-Fickian diffusion is likely to prevalent when a polymer composite consists of internal damage in the form of matrix cracks. Langmuir-type diffusion is reported to explain the penetration of

water in polyester and vinyl ester resins. The Langmuir model considers two phases of water, bound and unbound. Plasticization, swelling stress, hydrolysis, debonding and formation of cracks are the possible occurrence of environmental exposure and which should also influence the diffusion of water in the material [11, 12]. Moisture wicking along the fiber-matrix interface degrades the interfacial bond strength, resulting in loss of microstructural integrity [13].

A growing body of experimental evidence has given birth to the concept of interphase, which is a three-dimensional region existing between the bulk fiber and bulk polymer. The interphase of some finite thickness extending on both sides of the interface in both the fiber and matrix is different in structure and composition. Hydrothermal aging is possibly introducing chemical and structural changes in the resin matrix should influence the performance of a fiber-reinforced composite. The aging-induced new-phase at the interphase is more mobile than the matrix because it has its own glass transition at a lower temperature [14-16]. The shear strength of resin matrix is also known to suffer a loss on environmental aging. This could possibly change the locus of failure from the interface-interphase and produce cohesive failure in the matrix, particularly in composites having greater interfacial adhesion [17]. Composite structures must be designed to withstand the great diversity of environments, such as large variations in temperature and moisture. New causes of failure in composite materials are still being uncovered as service experience is gained. The rapid advancement of these materials has outstripped the understanding of appropriate failure analysis techniques [18]. Environmental exposure results in reduced interfacial stress transmissibility due to matrix

plasticization, chemical changes and mechanical degradation. Matrix plasticization reduces matrix modulus. Chemical degradation causes hydrolysis of bond. Mechanical degradation is the outcome of matrix swelling strain. An interfacial reaction may induce various morphological modifications to the interphase at the fiber/polymer interface [19, 20].

A need probably exists for an assessment of mechanical performance of such potentially promising materials under the influence of changing environment and loading speed. A strong interface displays an exemplary strength and stiffness but is very brittle in nature with easy crack propagation through the interface. A weaker interface reduces the stress transmissibility and consequently decreased strength and stiffness. A crack here is more likely to deviate and grow at the weak interface. It results in debonding and/or fiber pull-out and contributes to improved fracture toughness [21]. Most polymers lose their ductile properties below their glass transition temperature. Cooperative chain motions involving main chain bond rotation become extremely restricted. Requirements for polymeric materials in cryogenic applications are severe and complicated. It has been proposed that local intermolecular rearrangements result in relaxations at low temperature [22]. Additional complexities involve absorption of water and subsequent influence of freeze-thaw cycling on the changes of materials behavior through microstructure modifications. The factors affecting the mechanical response of composites are fiber/matrix interfacial properties, volume ratios, load transfer mechanisms and fabrication techniques [23]. As the volume fraction of reinforcement fiber in

composites increases, more fiber-matrix interfacial area is created and the more energy may be dissipated by the interface [24].

The recent review article has reported the effect of varying loading rate on the tensile, compression, shear and flexural properties of FRP composites is yet to be conclusive and furthermore, a variety of contradictions is observed [25]. It suggests the need for immediate investigation in the pursuit of eliminating all disagreements that currently exist regarding the effect of loading rate on mechanical performance of polymeric composites. Composite material may contain randomly spaced microvoids, incipient damage sites and microcracks with statistically distributed sizes and directions. Therefore, the local strength in the material varies in a random fashion. The failure location as well as degree of damage induced in the material will also vary in an unpredictable mode. Therefore, the mechanical and fracture behavior can be strongly influenced by the loading rate, temperature and material microstructure [26]. The predominant failure mechanisms in a composite laminate are a very complex combination of energy absorption principle [27]. Despite several advantages over conventional materials, FRP composites are highly susceptible to heat and moisture when operating in harsh and changing environmental conditions. It is suggested that the composite sensitivity to strain rate is mostly driven by the resin behavior, which needs to be further studied for adequate modeling of polymer matrix composites [28]. The effects of strain rate on most polymers may be explained by the Eyring theory of viscosity, which assumes that the deformation of a polymer involves the motion of a chain molecule over potential energy barriers. Here, the yield stress varies linearly with the logarithm of

strain rate. The polymer matrix has less time to localize at higher loading rates [29]. The non-linear viscoplastic behavior of fiber reinforced composite materials was recently investigated for elastic-plastic response [30].

Humid aging is recognized as one of the main causes of long-term failure of organic matrix composite. There are several modes of humid aging: by plasticization of matrix; differential swelling; embrittlement of macromolecular skeleton by hydrolysis; osmotic cracking; hygrothermic shock; localized damage at the fiber/matrix interface [31]. Most of the earlier studies have dealt with the aging behavior at a particular temperature and relative humidity. Thermal and as well as hygrothermal shock waves are expected in many applications, rather these are usual service condition [32,33]. Thermal expansion coefficients of polymers are considerably greater, thus failure of the bond between fiber and resin may occur under extreme of temperature. The use of composites in safety-critical applications leads to uneasiness since the mechanical performance is not well explored in active and changing environmental situations [34].

The present experiment is carried out for varied weight fractions glass fiber reinforced with epoxy and polyester resin matrices. The essential objective of this study is to assess the moisture absorption and/or desorption kinetics and mechanical behavior of laminated composites under hygrothermal shock cycles. Hygrothermal shocks are given here by exposing specimens in quick succession in two different environments. Humidity is varied and temperature remains constant in one set of conditioning. Temperature is changed and humidity is kept fixed in another experiment. Effects of relative humidity (RH) and temperature during

hygrothermal conditioning are qualitatively assessed here in the present investigation. Loading rate sensitivity of hygrothermally shocked polymer composites are also reported at different crosshead speeds.

EXPERIMENTAL

An unmodified epoxy resin based on Bisphenol-A and an unsaturated polyester resin were used with woven roving E-glass fibers, treated with a silane-based sizing system to fabricate laminated composites. Three fiber weight percentages, 55,60, and 65% were targeted in the laminate fabrication process. They were cured at ambient temperature. The laminated plates were cut into short beam shear (SBS) specimens by diamond cutter. The ASTM standard (D2344-84) specimens were exposed to climatic chamber for diffusional and as well as for 3-point SBS bend test studies. The mechanical tests were carried out with shortest possible off-time after the conditioning. Multiple samples were tested at each point of the experiment and the average value of acceptable level was reported here. The breaking load of SBS test was used to calculate the ILSS value. The tests were performed at 2mm/min and 50mm/min crosshead speeds for all the hygrothermally shocked samples as well as for untreated specimens.

Changing relative humidity at constant temperature shock conditioning

Glass/epoxy and glass/polyester laminated SBS specimens were exposed to 60% RH and at 50˚ C temperature environment for 1 hour. Then they were immediately transferred to another climatic chamber with 95% RH and again at 50˚ C temperature for 1 hour. This was treated as one hygrothermal shock cycle at

constant temperature. The conditionings were continued upto 19 cycles for the present experiment.

Changing temperature at constant relative humidity shock conditioning

Here the specimens were first exposed to environmental chamber at 50˚ C temperature and 60% RH for 1 hour. They were then suddenly exposed to 70˚ C temperature and again with the same 60% RH condition in another climatic chamber for 1 hour. Here this was considered to be one hygrothermal shock cycle at constant relative humidity. The experiments were extended again for 19 cycles.

RESULTS AND DISCUSSION

Figure 1 shows the percentage of moisture gain of varied weight fractions glass fiber reinforced epoxy composites with number of changing humidity cycle. The percentage of moisture gain of different weight fractions glass fiber reinforced polyester is plotted against the changing humidity cycle in Figure 2. The moisture uptake kinetics is observed here to be dependent on weight fraction of constituent phases. It is further noticed that the saturation level of moisture gain is attained after certain shock cycles and no further observable gain is reflected for almost both the resin systems. The initial stages of absorption fit into Fickian model, i.e. proportionality between mass gain and the square root of exposure time. The deviations in latter stages may be attributed by hygrotheral shock-assisted microcracks and/or microdamage. These possibly might accelerate the diffusion process. The structure of resin has the potential to influence the uptake of moisture

for that FRP composite. Langmuir-type diffusion is also proposed to explain the penetration of water in polyester and vinyl ester resins [11]. Absorbed moisture induces plasticization, hydrolysis, debonding at the interface and formation of cracks, which should influence the diffusion of moisture in different stages of FRP composites. Moisture can weaken the fiber/matrix interface not only through chemical attack and reaction, but also through mechanochemical effects such as osmotic pressure. Moisture diffuses and separates out in voids of resin. It dissolves soluble materials of polymer. The resin acts as a semipermeable membrane and osmotic pressure develops inside the void because water continues to diffuse there. The pressure may contribute in the formation of blister or debonded areas [35].

Figures 3(a) to 3(c) show the variations of shear strength of glass/epoxy composites with the number of humidity shock cycles for successively 55, 60. and 65% glass fiber reinforcement composites. The ILSS values are reported here for 2mm/min and 50mm/min crosshead speeds at each point of conditioning cycle. In order for a polymer composite to have properties that are advantageous, the fiber/matrix interface must have good adhesion. The mechanism of attack at the interface is mostly governed by the chemistry, structure, morphology and modes of failure at the interface [11].

The variations in ILSS values are drawn here successively against the humidity shock cycles for 55, 60, and 65% glass fiber reinforced polyester laminated composites at 2mm/min and 50mm/min crosshead speeds in Figures 4(a) to 4(c). It

is reported that moisture may influence flexural and transverse tensile properties and matrix cracking is common occurrence but not so invariably. The role of moisture in reducing cracking presumably because of plasticizing the resin and reducing internal stresses [36].

Figure 5 shows the percentage moisture loss with the number of hygrothermal shock cycles at constant relative humidity for 0.55, 0.60, and 0.65 weight fractions glass reinforced epoxy composites. Similarly, the moisture desorption with the same type of cycles is shown in Figure 6 for 0.55, 0.60, and 0.65 weight fractions glass fiber/polyester laminated composites. The nature of desorption is widely different in two systems. Polymer composites which absorbs moistures in humid condition at 50˚ C temperature and then exposure to 70˚ C temperature leads to drive off. Here the high temperature may act as activator for desorption phenomena. The very fast drying and generation and regeneration of residual stresses may quite often induce matrix as well as interfacial crackings. These microcracks in turn provide fast desorption paths for the absorbed moisture.

The variations of shear values of varied weight fractions glass/epoxy system with the hygrothermal shock cycles (at fixed relative humidity) at 2mm/min and 50mm/min crosshead speeds are shown in Figures 7(a) to 7(c). Cyclic moisture absorption and desorption plays a significant role in influencing the mechanical behavior of polymeric composites [12]. Moisture diffuses in and out through the polymer matrix and is likely to concentrate at the fiber/polymer interface. This may

influence the performance of a composite. There are quite often chemical, mechanical and thermomechanical changes at the interface and in bulk polymer matrix, which may introduce spatial non-uniformity of thermomechanical properties at the interface [37].

Figures 8(a) to 8(c) show the changes of ILSS values with the thermal shock cycles at a constant humidity level for different volume fractions glass fiber reinforced polyester composites at 2mm/min and 50mm/min loading speeds. Cyclic exposure is an important parameter when considering the service environment. The temperature in Remo, NV, has a daily mean variation of 20° C temeparture. The very low residual interfacial strain developed by the polyester resin suggests that this material should perform well in changing environment [38].

The possible synergy between exposures to moisture and cyclic stresses (thermal as well as swelling stresses), that may be responsible for the limited understanding of the present area of investigation. The mechanism of failure by environmental aging generally includes interfacial adhesive failure and cohesive failure in the matrix. High speed (high strain rate) deformation favors nonductile failure while low speed (low strain rate) deformation induces more ductile failure [8]. FRP composite structures are expected to experience a range of hygrothermal environmental conditions during their service life. Absorbed moisture causes plasticization of polymer matrix, may alter stress state and degrade the fiber/matrix interface. Thus critical understanding of moisture absorption and desorption behavior is need to be

explored for predicting long-term material and structural performance. Very recent investigation has been carried out on the diffusion behavior of woven composites using micromechanics finite element models [39]. It reports that woven composites show faster diffusion than that of a unidirectional laminate with the same overall volume fraction. The nature of interfacial contact is decisively controlled by the presence of residual stresses. Most fibers have smaller thermal expansivity than the polymer matrix, the resultant stresses are compressive in the fiber and tensile in the matrix. This arises because the matrix contracts onto the fiber and compresses it. Some of the stresses in polymer matrix composites which are generated by differential contraction, are relaxed by viscoelastic flow or creep in the matrix [40]. The interactions between fiber and matrix resin are complex but important phenomena. Cure shrinkage of unsaturated polyester resin can induce additional residual stress. Moisture absorption and desorption can also lead to significant residual stresses. This could modify the local stress threshold required for interfacial debonding [2].Drying and remoisturizing seem to affect only the compressive strength but SBS strength appears to be unaffected by it in graphite/epoxy composites [41].

CONCLUSIONS

The present study may possibly reveal the following conclusions;

Moisture is absorbed in changing humidity cycle with a constant temperature environment. Nature of absorption is found to be dependent on the type of matrix resin as well as weight fractions of constituents.

Desorption phenomena is noticed in changing temperature cycle with a fixed relative humidity condition. Desorption is much faster with the conditioning cycles for epoxy system in compared to polyester resin matrix composites. Although higher moisture loss is observed in polyester system.

Variations shear values under humid shock cycles show small deviations with reference to the as-cast specimens. Small increments and decrements may possibly be attributed to the changing state of residual stresses. Sometimes, it imparts strain-free state in the composite due to nullification.

Effect of changing temperature and humid conditions has a little impact in degrading the ILSS values for both epoxy and polyester systems. Thermal shock effects are not so phenomenal here. This probable reason could be the presence of moisture during thermal cycling.

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FIGURE CAPTIONS

Figure 1. Percentage of moisture gain against the number of changing humidity cycle for 55% (●), 60% (▲), and 65% (■) weight percentage of reinforcement glass/epoxy composites.

Figure 2. Percentage of moisture gain against the number of changing humidity cycle for 55% (●), 60% (▲), and 65% (■) weight percentage of reinforcement glass/polyester composites.

Figure 3(a). Variation of ILSS with number of humidity shock (at constant temperature) cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds for 0.55 weight fraction glass fiber reinforced epoxy laminated composites.

Figure 3(b). Variation of ILSS with number of humidity shock (at constant temperature) cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds for 0.60 weight fraction glass fiber reinforced epoxy laminated composites.

Figure 3(c) Variation of ILSS with number of humidity shock (at constant temperature) cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds for 0.65 weight fraction glass fiber reinforced epoxy laminated composites.

Figure 4(a). Variation of ILSS with number of humidity shock (at constant temperature) cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds for 0.55 weight fraction glass fiber reinforced polyester laminated composites.

Figure 4(b).   Variation of ILSS with number of humidity shock (at constant temperature) cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds for 0.60 weight fraction glass fiber reinforced polyester laminated composites.

Figure 4(c).   Variation of ILSS with number of humidity shock (at constant temperature) cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds for 0.65 weight fraction glass fiber reinforced polyester laminated composites.

Figure 5.   Percentage moisture loss versus number of thermal cycle at constant relative humidity for 55% (●), 60% (▲), and 65% (■) weight percentage of reinforcement glass/epoxy composites.

Figure 6.   Percentage moisture loss versus number of thermal cycle at constant relative humidity for 55% (●), 60% (▲), and 65% (■) weight percentage of reinforcement glass/polyester composites.

Figure 7(a).   Variation of shear strength with number of hygrothermal shock (at constant RH) cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds for 0.55 weight fraction glass fiber reinforced epoxy laminated composites.

Figure 7(b).   Variation of shear strength with number of hygrothermal shock (at constant RH) cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds for 0.60 weight fraction glass fiber reinforced epoxy laminated composites.

Figure 7(c)   Variation of shear shtrength with number of hygrothermal shock (at

constant RH) cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds

for 0.65 weight fraction glass fiber reinforced epoxy laminated

composites.

Figure 8(a).   Variation of ILSS with number of hygrothermal shock (at constant RH)

cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds for 0.55

weight fraction glass fiber reinforced polyester laminated composites.

Figure 8(b).   Variation of ILSS with number of hygrothermal shock (at constant RH)

cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds

for 0.60 weight fraction glass fiber reinforced polyester laminated

composites.

Figure 8(c).   Variation of ILSS with number of hygrothermal shock (at constant RH)

cycle at 2mm/min (▲) and 50mm/min (♦) crosshead speeds for 0.65

weight fraction glass fiber reinforced polyester laminated composites.

Figure 1

Figure 2

24
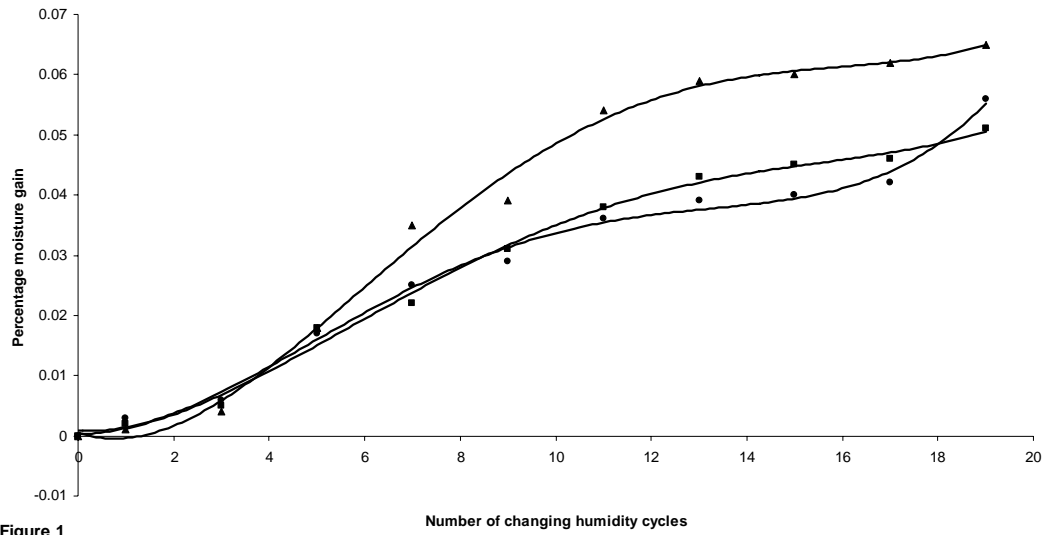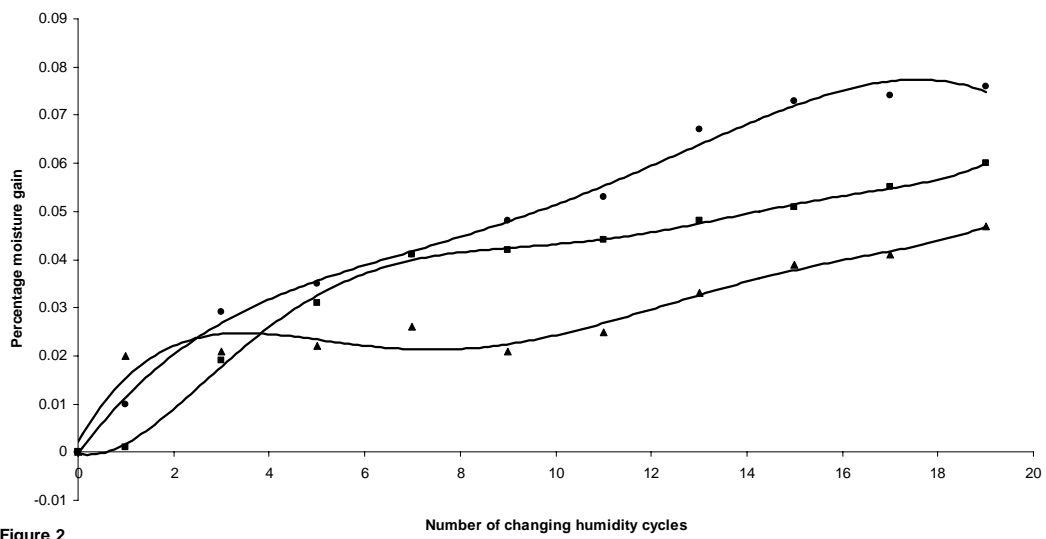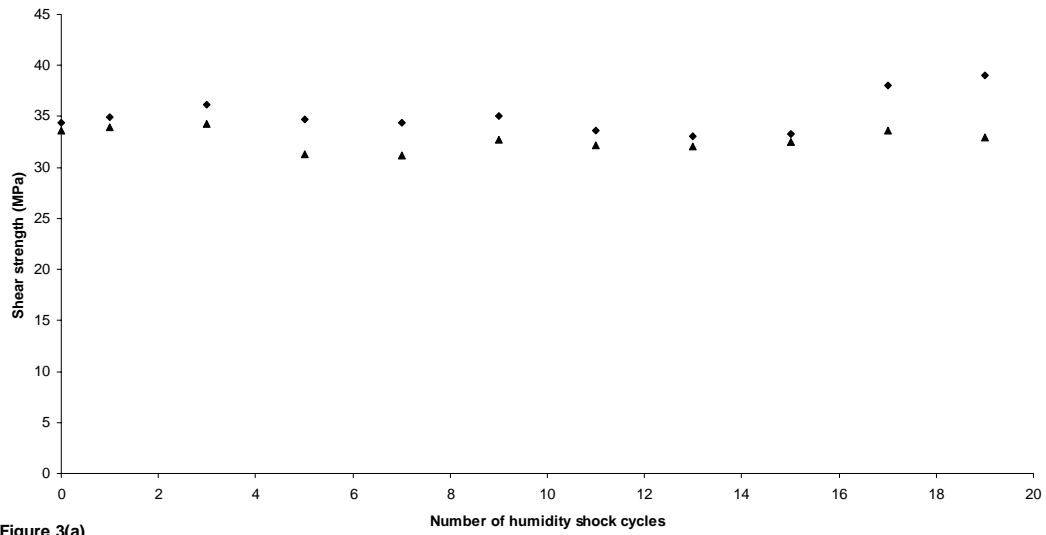
Figure 3(a)

Figure 3(b)

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Fgure 3(c)

Figure 4(a)

26
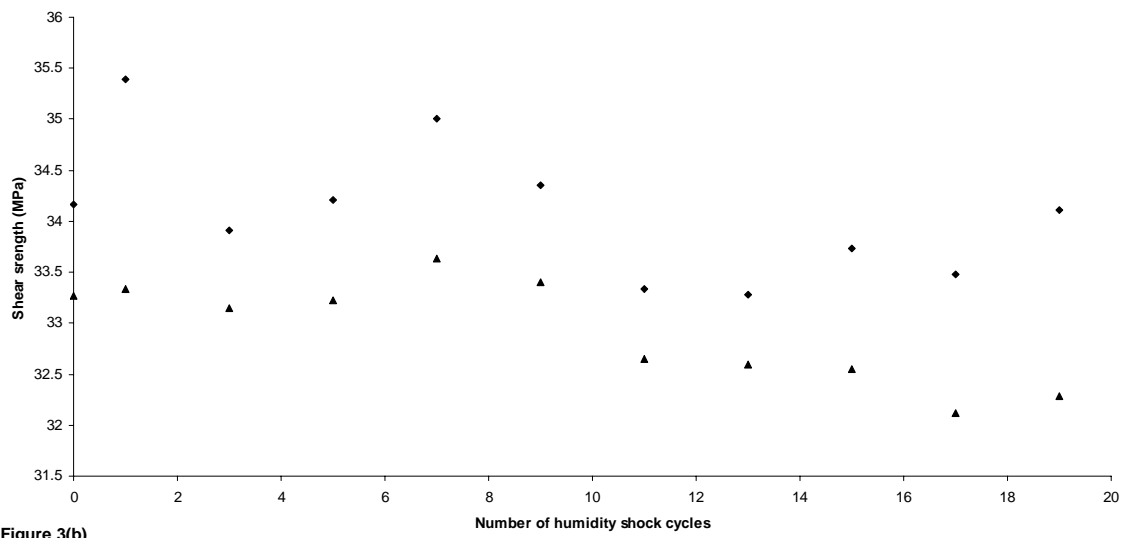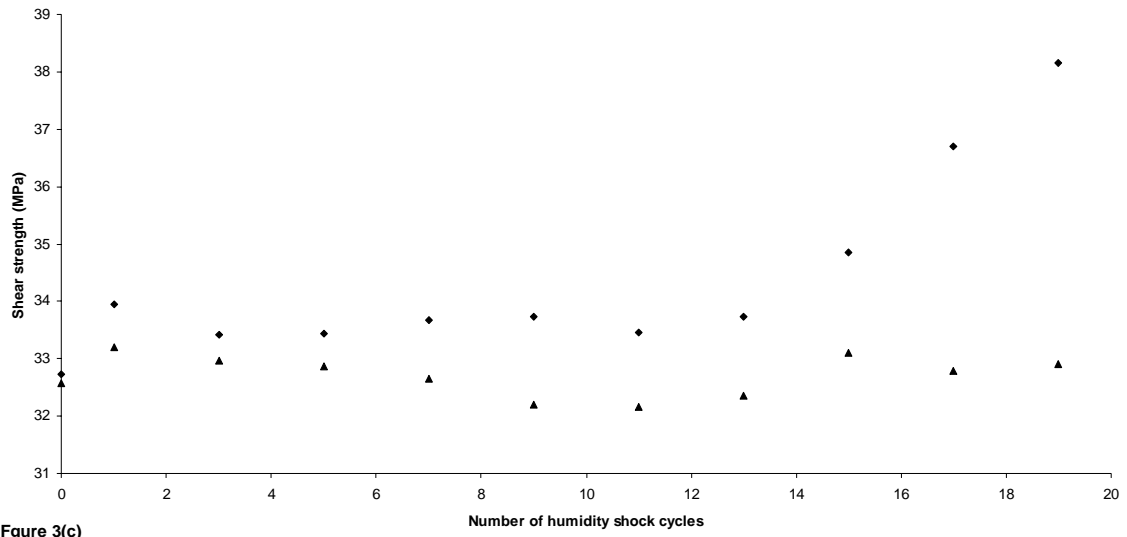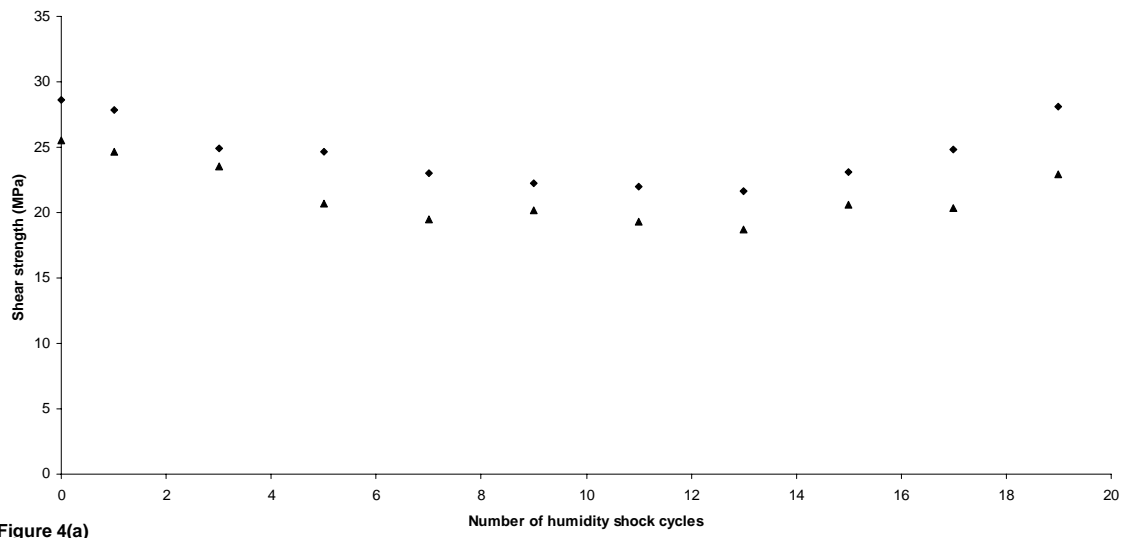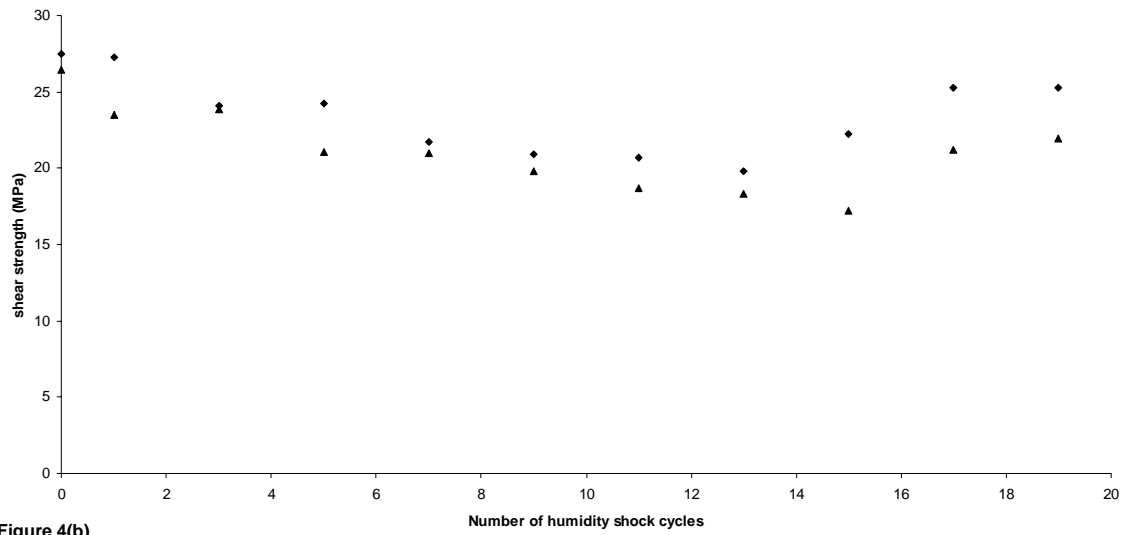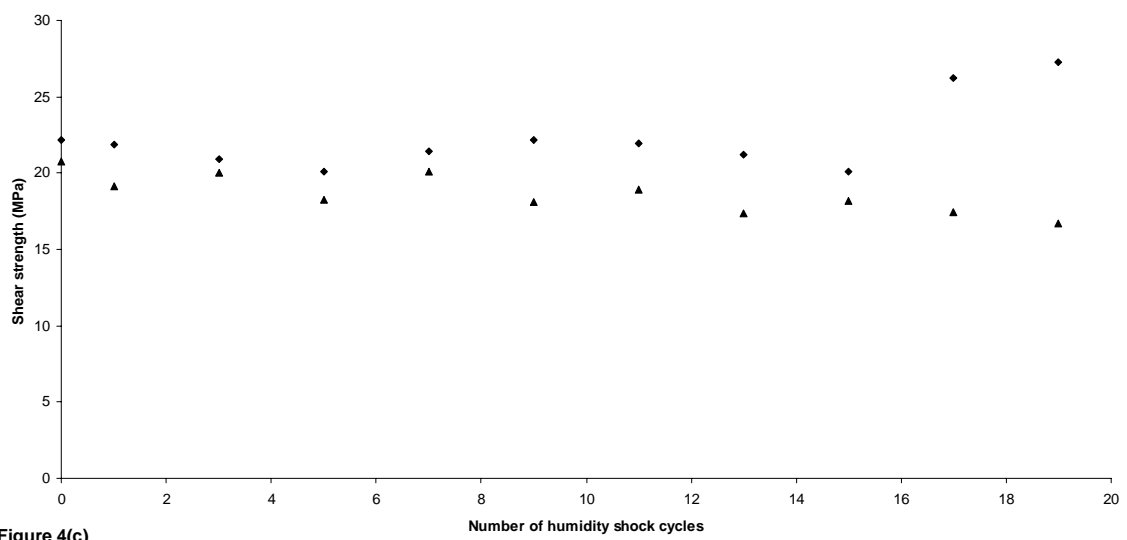
Figure 4(b)

Figure 4(c)
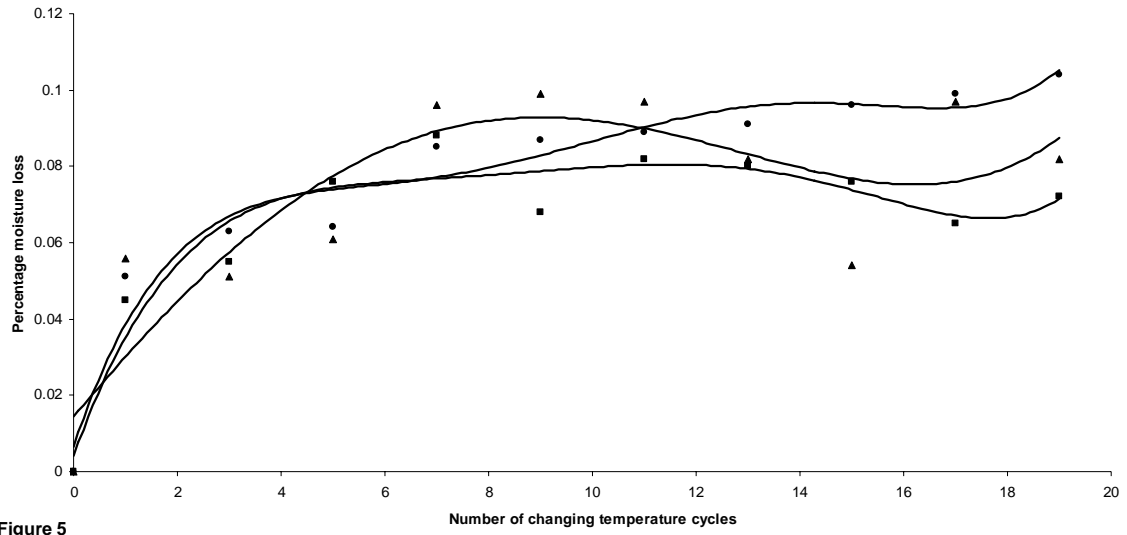
Figure 5
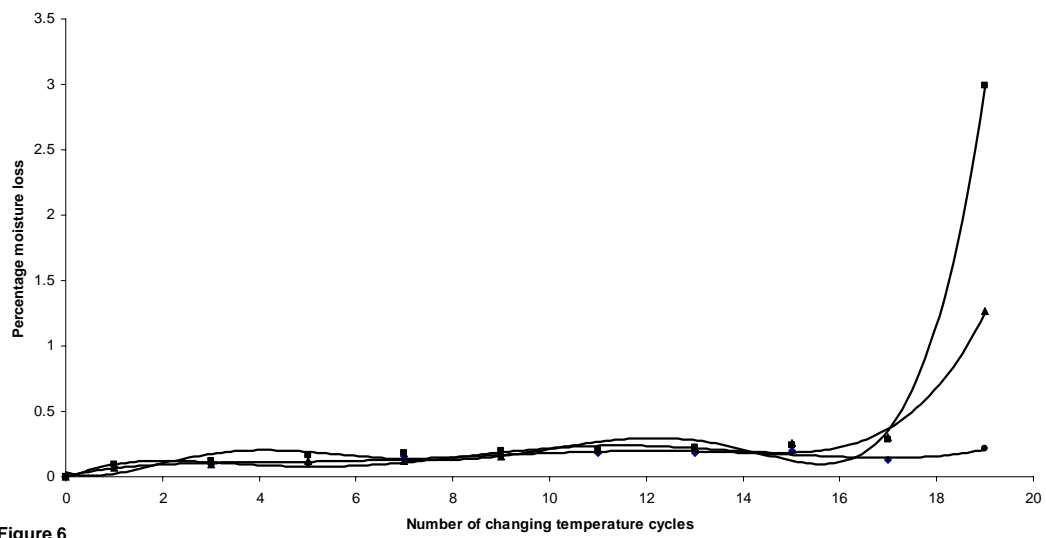
Figure 6
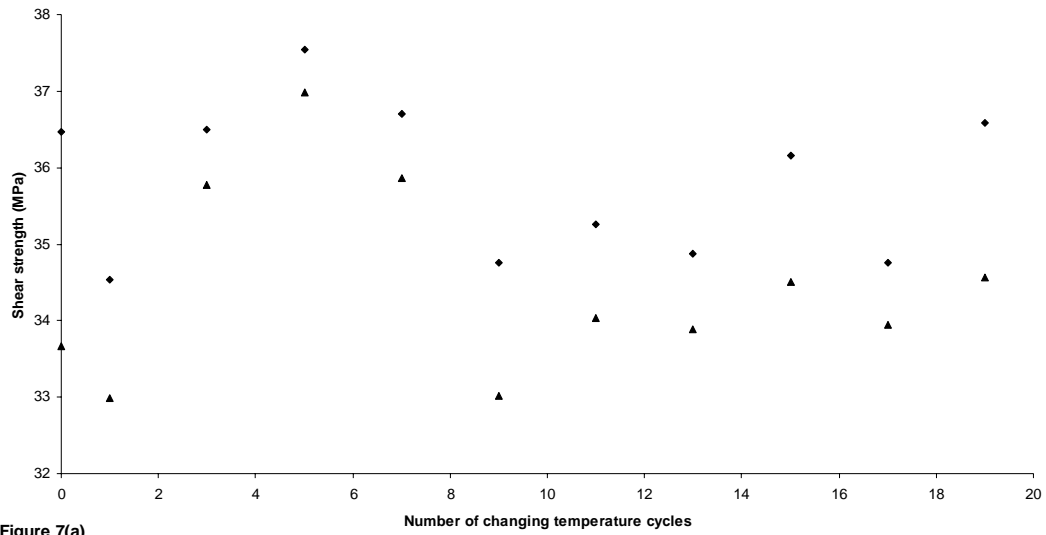
Figure 7(a)

Figure 7(b)

29
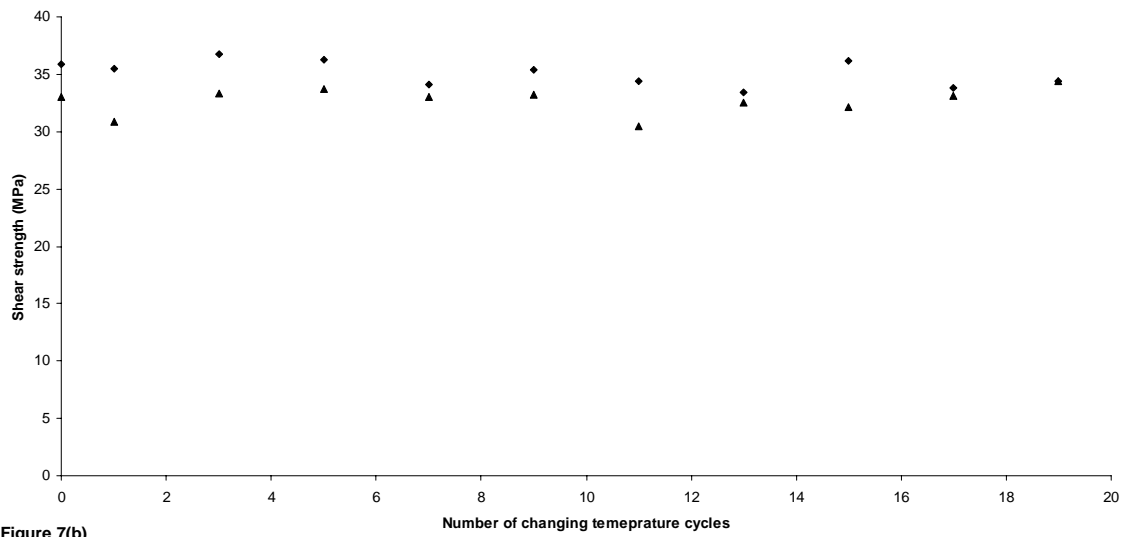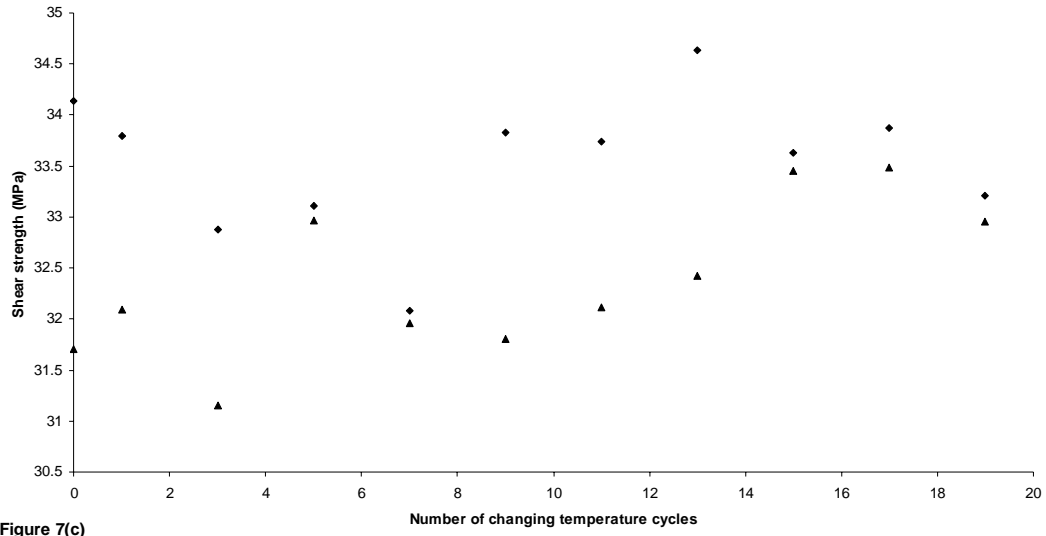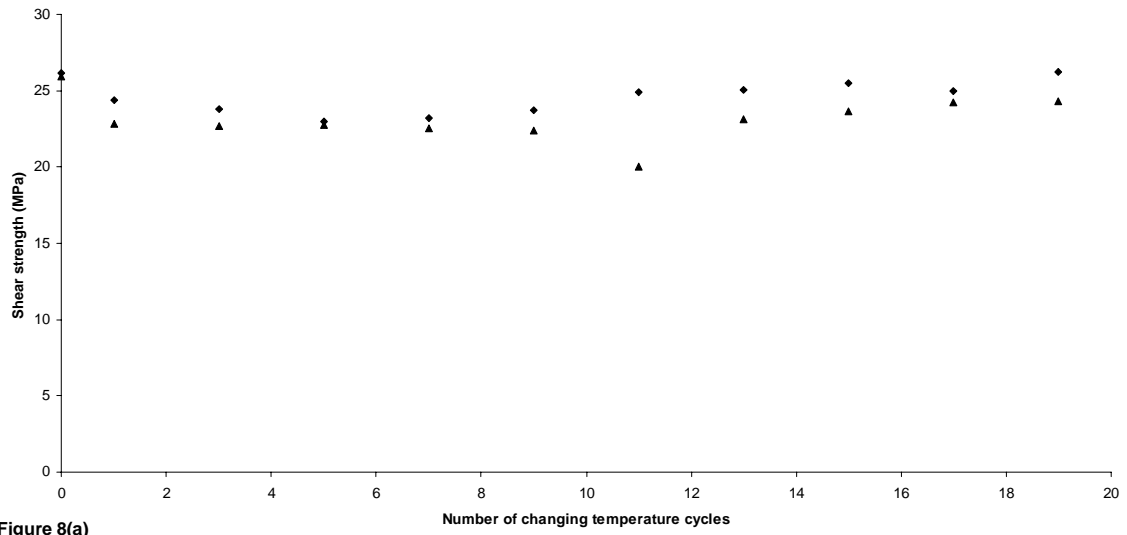
Figure 7(c)

Figure 8(a)

Figure 8(b)

Figure 8(c)

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