Chapter 6

Summary and Conclusions

6.1 Conclusions

A framework has been proposed in this study for assessing mode I and II delamination properties of interlayered polymer composites. The procedures developed in this framework provide guidelines for maximizing fracture toughness by controlling two parameters: interlayer thickness and interlayer toughening potential. In order to achieve this, it is necessary to assess the influence of the particles on delamination onset and growth.

The study commenced with examining toughening of Derakane 8084 vinyl-ester resin (VE) with ABS particles. It was concluded that this toughening approach was effective, making VE/ABS blends candidates for interlayer-toughening of the VE based composite materials. The study established non-linear mode I fracture toughness relationship with ABS content, where the toughness increased with increasing ABS content. The possible toughening mechanisms can be correlated to the mechanical influence of the second phase in the form of particles and to the chemical modification of the VE. Results from Raman spectroscopy showed chemical modification of the constituents, which might be a reason for the initial increase in toughness even with low ABS weight content blends (i.e. 3.5% and 7%). This chemical modification was in butadiene dissolution from the ABS particles into the VE. It was shown that the butadiene saturation corresponds to a change in the fracture toughness trend. This point was found to be around 7% ABS of addition. It was proved, by employing FEA modelling procedures, that below the butadiene saturation point the ABS particles do not influence fracture toughness and fracture behaviour of the material.

In addition, some aspects of standard delamination testing procedures for non-interlayered unidirectional composites were examined. Although some of these procedures are standardized there is still some ambiguity about their proper implementation. This part of the study established complementary methods for data analysis and the testing, and it was noted that the most influential factor on the toughness results is choice of the initial crack simulator. Fracture behaviour and toughness results under both loading modes for non-interlayered composites were assessed. This established toughness benchmarks for the following comparative study with interlayer toughened composites.

Mode I delamination testing and analysis of interlayer toughened composites was conducted in order to examine mode I fracture toughness as a function of two variables: interlayer thickness and ABS particle content. The materials examined in the first two chapters: VE/ABS blends and VE/glass fibre composite materials were combined in interlayer toughened configurations for the purpose of this study. It was shown that the interlayer thickness can influence fracture toughness through a confinement action upon the plastic zone around the crack tip. When the plastic zone is fully formed, the toughening potential of the interlayers, achieved through the ABS modifications, can be fully utilized. Hence, the mode I fracture toughness trend of the composites followed the toughness trend obtained for the bulk materials only when the interlayer thickness became sufficiently large to allow the full development of the plastic zone. The composite specimens failed within the interlayer, without any sign of an interfacial failure proving a good adhesion between the modified resins and the fibres. Also, cracks propagated through the ABS particles without debonding them from the surrounding resin. The ABS particles also initiated significant microcracking of the surrounding resin. This influenced the stability of the crack propagation which was improved with increasing the ABS content, followed by the subsequent increase in toughness. It was found that after the butadiene saturation point the change from unstable to stable fracture occurs, resulting in a steep increase in toughness. By employing a finite element analysis, a better insight of the role of the particles during the interlaminar fracture was established. The particles did not contribute to the interlayer yielding potential, but influenced fracture toughness through generation of microcracks. The major crack and microcracks propagated through the ABS particles and this resulted in triggering the rubber particle cavitation followed by shear yielding mechanisms.

The mode II fracture toughness trend remained independent of the interlayer thickness variation between 150 and 500μm. Also, a moderate improvement in the fracture toughness was recorded with increasing the ABS content. During the testing, all specimens failed in a stable fashion with the crack propagation between the first non-linear and the maximum load-displacement points, recorded during the test. This was defined as sub-critical crack growth. All interlayers failed under mode II loading interfacially, due to the hackle coalescence, which were generated after the material failure under the maximum tensile stress. It was found that the length of the sub-critical cracks was influenced by ABS concentration. Interlayers with ABS contents above the butadiene saturation point allowed 3mm longer sub-critical crack growth than those below that point. This resulted in significantly higher fracture toughness since all recorded load-displacement curves were similar regardless of the ABS content. A finite element modelling study was conducted to further explore the sub-critical crack growth. It was found that particles can induce localized matrix yielding and micro-cracking, at crack initiation and during the subsequent interfacial crack propagation. Also, as under mode I loading, ABS particles did not promote yielding of interlayer materials. It can be concluded that the secondary damage induced by the particles through their interaction with the main crack, could directly influence stability of the crack propagation. This possibly occurred by absorbing fracture energy through micro-cracking. Since the main crack did not propagate directly through the particles, their influence on fracture toughness was not as significant as it was under mode I loading.

In addition, several important conclusions must be underlined. Firstly, two important factors for improving fracture toughness under both loading modes are:

- Strong bonding between the interlayer and the rest of the composite
- Good bonding between the particles and the interlayer matrix

Strong interfacial bonding between the interlayer and the rest of the composite forces cracks to propagate through the interlayer, utilizing its full toughening capacity. Also, it was found that an optimum interlayer thickness can maximize fracture toughness under both loading modes. However, the optimum interlayer thickness under mode I is significantly larger than the one under mode II loading, due to a larger plastic zone sizes around the crack tip. Also, under mode II loading an interfacial failure, which is induced by the stress state and not by a weak interfacial bonding, always limits one half of the mode II plastic zone. Poor bonding between large particles (beyond 10μm in size) and interlayer matrix can result in the particle debonding during fracture.

This leads to the particles having a negligible influence on the fracture processes and can lead to poor toughness improvements. This phenomenon is in contrast with the fracture behaviour of sub-micron toughening particles (CTBN rubber for example), where the particle debonding triggers strong fracture energy absorbing mechanisms. An important conclusion from this study for fulfilling these two requirements is the necessity for the chemical interaction between the interlayer constituents.

Under both loading modes, two major fracture mechanisms were active: yielding and micro-cracking, and they clearly operated in synergy. Plastic deformation dissipated significant fracture energy within interlayers, while micro-cracking interacted with the main crack under the both loading modes. The latter, resulted in a stable crack propagation and overall higher fracture resistance.

6.2 Recommendations for future work

During this research project several issues remained beyond the scope of this work. These can be classified in three groups: manufacturing, mechanical testing and numerical modelling.

The interlayer toughening procedure was successfully applied to vinyl-ester based composites involving the hand lay-up manufacturing procedure. However, there is a need for research in the area of optimization of the current manufacturing process and its application to real-life structural parts. Also, modifications of the established manufacturing process are necessary for implementation of multiple interlayers in the composite.

Mechanical testing conducted in this study covered quasi-static delamination testing under mode I and mode II loading, and established some important milestones for characterization of this material. Tests that can obtain further insights in the material's behaviour are: mixed mode I/II quasi-static testing, impact tests, followed by the compression after impact test (CAI). In addition, further study may be required to study the influence of pre-cracking conditions for delamination testing of these materials. For example, the influence of pre-cracking on the sub-critical crack growth under mode II loading needs further investigation.

Finally, the FEA techniques used in this study simulated the processes of crack/particle interactions and subsequently captured stress/strain distributions that were undetectable by standard experimental procedures. In future work, FEA can be used to explain fracture behaviour of interlayered composites under mixed mode I/II and impact loading conditions.

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Appendix A

Generation Of Random Particle Distribution

The distribution of the ABS particles in interlayers was irregular and random. In order to develop representative FEA models of the interlayers with particles several approximations had to be made. Firstly, it was assumed that particles can be successfully represented by circles, random in size and randomly distributed within the given boundaries. A program in FORTRAN 77 was developed by using a random number generator (RNG) [115], to generate random particle distributions under this assumption. Each circle was defined by generating three numbers given as the output from the program: the radius and two coordinates of the center.

The RNG has a uniform distribution of the generated numbers (i.e. all numbers between 0 and 1 have equal probability to be generated). By examining micrographs taken from the polished specimen edges, it was established that particle size ranges between approximately 1 and 200μm, having a nearly exponential size distribution. However, the particle for particle generation possessed pre-defined bpundaries. The minimum and maximum particle radii were chosen to be 5 and 75μm, respectively, while the minimum distance between particles was kept at 5μm. Finally, the smallest distance between the particles and the given area boundaries was also 5μm. These particle size and distribution boundaries were adopted due to the requirements of the element shape and size in the FEA models. Although the employed RNG did not produce an exponential distribution of numbers, the quality of generated particle distributions were found to be of sufficient accuracy for the purpose of this study.

Finally, the program terminates when the area of the generated particles is greater or equal to the pre-defined area size (denoted in percentages of the overall given area)

The program and its main variables are listed below:

iseed1-iseed3 – three positive integer numbers, called 'seeds', used by the RNG function **ran1** to generate random numbers between 0 and 1

R(I), X(I), Y(I) – radii, x and y center coordinates of the Ith circle

P1-P4 – limitations upon the generated circles: minimum distance and radius

```
PROGRAM PARTICLE GENERATOR
PARAMETER (M=99999)
INTEGER I,J,MP,SP,COUNT,iseed1,iseed2,iseed3,idum,idum2,idum3
REAL H,W,PC,X(M),Y(M),R(M),GX(M),GY(M),GR(M),A(M),AR
REAL P1,P2,P3,P4,P5
real*8,RND1,RND2,RND3
DATA DIST,RMIN,RMAX,PI /5,5,75,3.14159265359/
DATA GX(1),GY(1),GR(1),I,J,AR /0,0,0,0,1,0/
DATA MP,SP /0,0/
PRINT*, 'Input width and height of the area, W & H [microns] '
READ(*,*) W,H
PRINT*, 'Input average particle concentration, PC[%]'
READ(*,*) PC
PRINT*,'Input initial seeds for RNG, any 3 integers>0'
READ(*,*) iseed1,iseed2,iseed3
idum=-iseed1
idum2=-iseed2
idum3=-iseed3
PC=PC/100
```

```
      open(unit=1,file='gen-final-out.txt',
     +FORM='FORMATTED',status='unknown')
 10   I=I+1
 11   RND1=ran1(IDUM)
      RND2=RAN1(IDUM2)
      RND3=RAN1(IDUM3)
      R(I)=RMAX*RND1
      IF (R(I) .LT. RMIN) GOTO 11
      X(I)=W*RND2
      Y(I)=H*RND3
      IF ((X(I)+R(I)+DIST).GT. W .or.(X(I)-R(I)).LT. DIST .or.
     +    (Y(I)+R(I)+DIST) .GT. H .or. (Y(I)-R(I)) .LT. DIST) GOTO 11
      DO K=1,J
         P1=(GX(K)+R(I)+GR(K)+DIST)
         P2=(GX(K)-R(I)-GR(K)-DIST)
         P3=(GY(K)+R(I)+GR(K)+DIST)
         P4=(GY(K)-R(I)-GR(K)-DIST)
         IF ((X(I) .LT. P1) .AND. (Y(I) .LT. P3).AND.
     +       (X(I) .GT. P2) .AND. (Y(I) .GT. P4) .OR.
     +       (X(I) .GT. P2) .AND. (Y(I) .LT. P3) .AND.
     +       (X(I) .LT. P1) .AND. (Y(I) .GT. P4)) GOTO 11
      ENDDO
       IF (R(I).GE.30)THEN
         MP=MP+1
         ELSE
            CONTINUE
       ENDIF
      IF (R(I).GE.10.AND.R(I).LT.30)THEN
         SP=SP+1
           ELSE
            CONTINUE
      ENDIF
      J=J+1
      GX(J)=X(I)
      GY(J)=Y(I)
      GR(J)=R(I)
      A(J)=PI*(GR(J))**2
      AR=AR+A(J)
      COUNT=J-1
      P5=AR/(H*W)
      print*,COUNT,Gr(J),Gx(J),Gy(J),A(J),AR,(P5*100)
      IF (P5 .LT. PC) GOTO 10
      WRITE(1,20)COUNT,(P5*100),AR,mp,sp,(COUNT-MP-SP)
 20   FORMAT(/6X,'NUMBER OD PARTICLS=',I3/,
     +   /6X,'PARTICLE CONCENTRATION=',F4.1/,
     +  /6X,'AREA COVERED WITH PARTICLES=',F10.3/,
     +  /6x,'NUMBER OF LARGE PARTICLES,(30<R<75)=',I3/,
     +  /6X,'NUMBER OF MEDIUM PARTICLES,(10<R<30)=',I3/,
     +  /6X,'NUMBER OF SMALL PARTICLES,(3<R<10)=',I3/,
```

```
     +    /6X,'R:',10X,'X:',12X,'Y:',12X,'A(K)'/)
      DO K=2,J
          WRITE(1,30)GR(K),GX(K),GY(K),A(K)
      enddo
 30   FORMAT(3X,F8.3,5X,F8.3,5X,F8.3,5X,F10.3,5X)
      STOP
      END


      FUNCTION ran1(idum)
      INTEGER idum,IA,IM,IQ,IR,NTAB,NDIV
      REAL ran1,AM,EPS,RNMX
      PARAMETER (IA=16807,IM=2147483647,AM=1./IM,IQ=127773,IR=2836,
     +NTAB=32,NDIV=1+(IM-1)/NTAB,EPS=1.2e-7,RNMX=1.-EPS)
      INTEGER j,k,iv(NTAB),iy
      SAVE iv,iy
      DATA iv /NTAB*0/, iy /0/
      if (idum.le.0.or.iy.eq.0)then
      idum=max(-idum,1)
      do j=NTAB+8,1,-1
         k=idum/IQ
         idum=IA*(idum-k*IQ)-IR*k
         if (idum.lt.0) idum=idum+IM
         if (j.le.NTAB) iv(j)=idum
      enddo
      iy=iv(1)
      endif
      k=idum/IQ
      idum=IA*(idum-k*IQ)-IR*k
      if (idum.lt.0) idum=idum+IM
      j=1+iy/NDIV
      iy=iv(j)
      iv(j)=idum
      ran1=min(AM*iy,RNMX)
      return
      END
```

A.1 Random particle generator output

The typical output from the particle generator is given below. The program returns, in addition to radii and center coordinates of the particles, their surfaces, and their size clasification, based on the given definitions. All units are in micrometers, except areas which are in square micrometers.

NUMBER OD PARTICLS= 52

PARTICLE CONCENTRATION=27.0

AREA COVERED WITH PARTICLES= 67608.453

NUMBER OF LARGE PARTICLES,(30<R<75)= 9

NUMBER OF MEDIUM PARTICLES,(10<R<30)= 25

NUMBER OF SMALL PARTICLES,(3<R<10)= 18

R:	X:	Y:	A(K)
12.249	471.167	302.860	471.381
39.556	383.682	122.021	4915.554
8.704	164.382	146.503	238.009
40.674	288.355	106.491	5197.397
8.854	17.672	74.049	246.303
10.102	218.371	161.986	320.600
16.211	129.346	422.732	825.568
26.507	331.597	401.613	2207.349
36.788	46.640	269.375	4251.739
5.672	46.399	451.423	101.088
11.022	216.858	360.874	381.652
30.889	240.670	430.814	2997.419
7.094	122.999	189.143	158.084
36.990	390.952	226.187	4298.458
14.995	384.645	55.063	706.408
15.614	90.131	134.778	765.869
13.381	86.561	338.020	562.467
62.738	191.364	267.432	12365.339
16.916	322.611	223.723	898.924
19.076	384.190	310.871	1143.163
7.288	280.506	224.017	166.850
16.259	123.150	357.439	830.458
7.055	59.013	130.457	156.375
7.030	458.837	395.200	155.252
30.611	91.817	41.334	2943.854
30.544	174.521	96.745	2930.969
7.769	65.595	386.924	189.635
21.720	85.375	440.228	1482.033
6.257	455.953	330.365	122.986
12.118	408.434	373.092	461.303
18.652	310.009	307.127	1092.970
16.288	39.039	342.600	833.452
8.338	397.450	436.499	218.425
8.587	467.835	200.820	231.646

14.182	184.946	26.519	631.901
7.281	169.896	189.630	166.550
12.985	300.505	181.507	529.740
18.166	91.566	188.105	1036.782
16.018	30.162	192.669	806.047
14.695	175.867	386.269	678.436
9.534	39.200	483.521	285.555
12.199	374.505	347.356	467.534
14.050	109.537	301.417	620.155
9.342	437.549	36.457	274.155
25.752	461.734	127.859	2083.350
7.449	358.339	440.763	174.325
12.937	249.316	167.008	525.762
16.016	188.691	457.938	805.883
6.801	484.467	328.504	145.319
31.299	443.291	443.366	3077.566
6.408	424.654	62.994	128.997
9.795	122.864	469.296	301.408

Appendix B

Franc 2D/L – FEA Crack Growth Algorithm

The FRacture ANalysis Code-2D with Layers (FRANC2D/L) is an FEA code that is a result of research efforts conducted at the Kansas State University (KSU), as a part of the NASA Airframe Structural Integrity Program (NASIP). NASIP has developed a range of engineering tools in the form of computer codes to be used by engineers and researchers in research institutions and industry. Originally developed at Cornell University by Wawrzynek and Ingraffea in 1987 (originally named FRANC), this software was designed to solve two-dimensional linear elastic fracture mechanics problems using the finite element approximation. The FRANC code was created in response to a need to model the true evolutionary behavior of crack propagation using finite elements. The main novelty implemented in the code was the use of an advanced topological database in order to save the relationships between all adjacent nodes, elements and element edges. This approach increases efficiency of the code during re-meshing operations during crack propagation analyzes.

The current FRANC2D/L version is a result of further development of the Cornell version of the software and is the result of two recent research projects at KSU. Sudhir Gondhalekar [74] extended the original two-dimensional code to model layered structures such as lap joints and adhesively bonded joints. The model was limited to in-plane loading and deformations only. Srinivas Krishnan [74] subsequently extended the code to include out-of-plane loading and deformations through linear, small strain bending. Finally, Mark James [74] conducted a further development of the software, as a part of his Ph.D. reserach project, by adding elastic-plastic tearing capability.

The following sections cover the basics of the Crack Growth Model implemented in the FRANC2D/L software. More information on this topic, together with verification studies and some additional information regarding implementation of elastic-plastic crack propagation models in FEA codes can be found in [74].

B.1 Crack Growth Methodology

In Figure B.1 the conceptual crack growth methodology is depicted. The process starts with the pre-processing stage, resulting in the initial representation of the analysis, R_i. This stage involves definition of the model's geometry, mesh, material properties and boundary conditions.

The analysis model generation step uses the topological database to prepare data for the analysis. This includes generation of node and element lists, together with minimization of the stiffness matrix band width. Finally, the data is structured and prepared for the solution procedure.

The solution procedure (i.e. solver) is responsible for implementation of the assigned boundary conditions and satisfying the equilibrium equations, by using nonlinear incremental-iterative schemes. As the solution progresses, fracture parameters are monitored at appropriate times during the analysis. Also, the solver triggers ap-

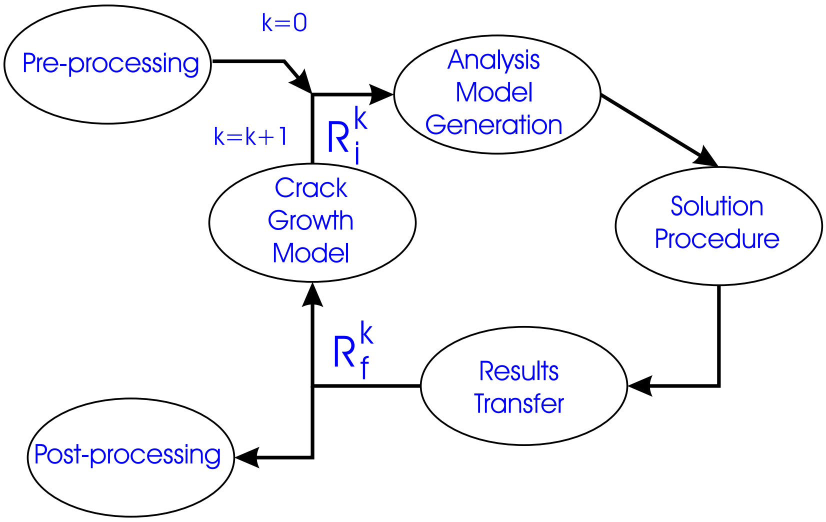

Figure B.1: Conceptual Crack Growth Methodology: how the crack growth model fits within the analysis architecture [74]

propriate actions when fracture criteria are satisfied (i.e. node separation process for generation of a new crack).

The output from the solution procedure phase is the results transfer phase. This process is used for transferring the equilibrium output from the solution procedure to the final representation appropriate for post-processing and the crack growth model.

B.2 Crack Growth Model

The Crack Growth Model is depicted in Figure B.2 in more detail. It receives the input from the results transfer phase in the form of R_f^k results vector (i.e. solver output), based on the current equilibrium state. The first step is application of the fracture and direction criteria to determine the crack growth direction. In the case of elastic-plastic fracture mechanics, the direction criterion is dependant upon the stress or strain state local to the crack tip. Naturally, this is obtained from the R_f^k.

The crack extension algorithms open the new crack using the calculated direction and user specified crack growth increment. In order to do this, this algorithm has to

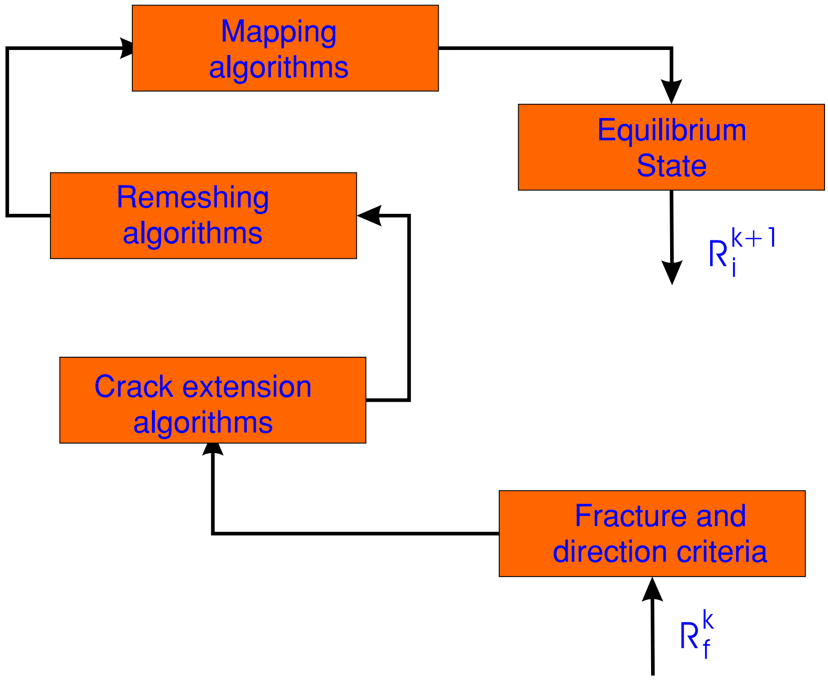

Figure B.2: Crack Growth Model: sequence of steps to grow a crack [74]

delete the current mesh in the vicinity of the crack tip and then extends the crack.

The re-meshing algorithms place a rosette of elements around the crack tip and then rebuild the mesh in the vicinity of the crack tip.

The mapping algorithms are responsible for transferring the plastic history and displacement solution from the old to the newly built mesh after the crack extension, through the re-meshing and crack extension algorithms. The plastic history includes nodal displacements, gauss point stresses, the stress work, radius of the yield surface, and flags that indicate yielding state.

The crack growth model then brings the FEA model to its equilibrium state. This results in a new initial representation model, R_i^k , at the new geometric configuration, which can enter a new cycle of the crack propagation analysis.