THE INFLUENCE OF REINFORCEMENT HOMOGENEITY ON THE
DEFORMATION AND FRACTURE OF A DISCONTINUOUSLY REINFORCED
ALUMINUM MATRIX COMPOSITE

A Thesis in
Materials Science & Engineering

by

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ABSTRACT

Deformation processing (extrusion) has been used to homogenize reinforcement distribution in a discontinuously reinforced aluminum matrix composite (DRA 6092/SiC/25p). Reinforcement distribution after three conditions of varying extrusion reduction has been quantified using the homogenous length scale technique. Results indicate that deformation processing positively influences reinforcement homogeneity—increasing deformation asymptotically leads to an increasingly uniform reinforcement distribution. After heat treatment to peak age, the compressive flow behavior both along and transverse to the extrusion axis has been determined for each condition. Using chevron notch short rods, the fracture toughness behavior in several orientations has also been assessed. Variations in flow behavior with deformation processing are mainly rationalized in terms of matrix texture with the combined effect of particle alignment along the extrusion axis and reinforcement homogenization being relatively small. Regardless of orientation, toughness is shown to increase with reinforcement homogeneity; in a particular transverse orientation, chevron notch fracture toughness \( K_{IV} \) demonstrates a nearly two-fold increase (10.8 MPa√m to 19.3 MPa√m) as a result of reinforcement cluster breakdown. Qualitative fractography indicates a generally ductile fracture process. Quantitative fractography indicates a strong positive relationship between fracture surface average roughness \( R_s \)—a measure of out-of-plane crack deflection—and toughness \( K_{IV} \), while a fractal technique used to characterize the fracture surface indicates a decrease in fractal dimension with increasing toughness. Using a simple model, the trend of increasing average roughness is interpreted as an increase in the fracture process length scale, while the decrease in fractal dimension is interpreted as a subtle decrease in the mean angle of crack deflection—a result expected with reinforcement homogenization. The increase in toughness is rationalized as a combined effect of decreasing crack deflection (leading to slower strain accumulation during crack blunting) in addition to a nominal improvement in plastic strain that can be supported by the interparticle ligament after reinforcement homogenization. Such a change in local ductility is qualitatively validated with SEM fractography.
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>LIST OF FIGURES</td>
<td>v</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>xii</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENTS</td>
<td>xiii</td>
</tr>
<tr>
<td>1. INTRODUCTION &amp; BACKGROUND</td>
<td>1</td>
</tr>
<tr>
<td>1.1 INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>1.2 FRACTURE IN DRA &amp; CRACK GROWTH BEHAVIOR</td>
<td>3</td>
</tr>
<tr>
<td>1.3 DUCTILE FRACTURE MODELS FOR DRA</td>
<td>27</td>
</tr>
<tr>
<td>1.4 QUANTITATIVE FRACTOGRAPHY</td>
<td>44</td>
</tr>
<tr>
<td>1.5 RESEARCH OBJECTIVES</td>
<td>59</td>
</tr>
<tr>
<td>1.6 REFERENCES</td>
<td>60</td>
</tr>
<tr>
<td>2. EXPERIMENTAL PROCEDURE &amp; METHODS</td>
<td>66</td>
</tr>
<tr>
<td>2.1 MATERIALS &amp; MATERIAL PROCESSING</td>
<td>66</td>
</tr>
<tr>
<td>2.2 MATERIAL CHARACTERIZATION</td>
<td>72</td>
</tr>
<tr>
<td>2.3 DEFORMATION BEHAVIOR</td>
<td>75</td>
</tr>
<tr>
<td>2.4 FRACTURE TOUGHNESS TESTING</td>
<td>76</td>
</tr>
<tr>
<td>2.5 QUANTITATIVE FRACTOGRAPHY</td>
<td>86</td>
</tr>
<tr>
<td>2.6 REFERENCES</td>
<td>87</td>
</tr>
<tr>
<td>3. RESULTS &amp; ANALYSIS</td>
<td>89</td>
</tr>
<tr>
<td>3.1 MATERIAL CHARACTERIZATION</td>
<td>89</td>
</tr>
<tr>
<td>3.2 MECHANICAL BEHAVIOR</td>
<td>110</td>
</tr>
<tr>
<td>3.3 FRACTOGRAPHY</td>
<td>122</td>
</tr>
<tr>
<td>3.4 QUANTITATIVE FRACTOGRAPHY</td>
<td>133</td>
</tr>
<tr>
<td>3.5 REFERENCES</td>
<td>151</td>
</tr>
<tr>
<td>4. DISCUSSION</td>
<td>152</td>
</tr>
<tr>
<td>4.1 MATERIALS CHARACTERIZATION</td>
<td>152</td>
</tr>
<tr>
<td>4.2 DEFORMATION</td>
<td>154</td>
</tr>
<tr>
<td>4.3 FRACTURE</td>
<td>155</td>
</tr>
<tr>
<td>4.4 REFERENCES</td>
<td>167</td>
</tr>
<tr>
<td>5. CONCLUSIONS</td>
<td>168</td>
</tr>
<tr>
<td>APPENDIX A</td>
<td>170</td>
</tr>
<tr>
<td>APPENDIX B</td>
<td>176</td>
</tr>
<tr>
<td>APPENDIX C</td>
<td>180</td>
</tr>
<tr>
<td>APPENDIX D</td>
<td>186</td>
</tr>
</tbody>
</table>
LIST OF FIGURES

1.1 SPECIFIC PROPERTY CHART FOR CONVENTIONAL METALS AND METALLIC COMPOSITES.......................................................... 1

1.2 (A) DRA F-16 FUEL ACCESS PANELS, (B) DRA HONDA PRELUDE CYLINDER LINERS, (C) DRA F-16 VENTRAL FIN, AND (D) DRA FAN-EXIT GUIDE VANE FROM A PRATT & WHITNEY 4000 SERIES ENGINES.......................................................... 2

1.3 MODES OF FRACTURE IN DISCONTINUOUSLY REINFORCED ALUMINUM (DRA).............................................................. 5

1.4 IDEALIZED INFLUENCE OF REINFORCEMENT VOLUME FRACTION ON COMPOSITE FLOW BEHAVIOR................................. 6

1.5 EFFECT OF VOLUME FRACTION ON FRACTURE RESISTANCE OF NUMEROUS DRA SYSTEMS...................................................... 7

1.6 EFFECT OF HEAT TREATMENT ON TOUGHNESS AND YIELD STRENGTH FOR SEVERAL DRA’S............................................. 9

1.7 DEPICTIONS REPRESENTATIVE OF THE (A) PARTICLE CRACKING IN AN UA HEAT TREATMENT OF A DRA AND (B) INTERFACIAL DEBONDING IN AN OA HEAT TREATMENT OF THE SAME MATERIAL OBSERVED BY LEWANDOWSKI.................................. 10

1.8 (A) SCHEMATIC DEPICTION AND (B) NEAR INTERFACE TEM PHOTOGRAPH OF A PRECIPITATE FREE ZONE (PFZ) THAT IS 1-2 μm THICK IN DRA 6092/15/SIC_p............................................................ 11

1.9 RELATIONSHIP BETWEEN TOUGHNESS AND YIELD STRENGTH IN AL 2080/SIC/X_p AS A FUNCTION OF REINFORCEMENT VOLUME FRACTION AND PARTICLE SIZE...................................................... 14

1.10 RELATIONSHIP BETWEEN TOUGHNESS AND ULTIMATE STRENGTH IN AL 2080/SIC/X_p AS A FUNCTION OF REINFORCEMENT VOLUME FRACTION AND PARTICLE SIZES........ 14

1.11 (A)-(D) INFLUENCE OF REINFORCEMENT COMPOSITION AND MEAN PARTICLE DIAMETER ON CRACK AND VOID DAMAGE ACCUMULATION RATES, AS MEASURED BY THE DAMAGE PARAMETERS D_E AND D_p, RESPECTIVELY......................................................... 16

1.12 INFLUENCE OF ASPECT RATIO ON THE UNIAXIAL DEFORMATION RESPONSE OF A COMPOSITE CONTAINING 20 VOL. % ALIGNED PROLATE ELLIPSOIDS................................................................. 17

1.13 COMPARISON OF THE MORPHOLOGICAL DIFFERENCE BETWEEN TWO TYPES OF REINFORCEMENT USED IN A AL-6061/SIC/25_p COMPOSITE, (A) AN F-600 (GRIT) POWDER AND (B) THE HBD POWDER. .................................................................................. 19
1.14 MECHANICAL BEHAVIOR OF THE F-600 AND HBD REINFORCED AL-6061/SIC/25p COMPOSITES IN THE (A) AS EXTRUDED AND (B) HEAT TREATED (OA AND T6) CONDITIONS............................................. 19

1.15 INFLUENCE OF HEAT TREATMENT ON GLOBAL FRACTURE MORPHOLOGY IN (A) F-600 AND (B) HBD REINFORCED AL-6061/SIC/25p COMPOSITES................................................................. 20

1.16 INFLUENCE OF HEAT TREATMENT ON FRACTURE MORPHOLOGY AND LOCAL FRACTURE TOPOGRAPHY IN F-600 AND HBD REINFORCED AL-6061/SIC/25p COMPOSITES........................................... 21

1.17 A NOVEL CT GEOMETRY FOR DETERMINING MECHANISMS OF CRACK PROPAGATION IN DRA, AND (B) THE RESULTING DEPICTION OF CRACK PROPAGATION THROUGH CLUSTERED (HIGH $V_F$) PARTICLE REGIONS................................................................. 23

1.18 DIFFUSE CRACK GROWTH WITH UNBROKEN LIGAMENTS IN THE CRACK WAKE OF A 20 VOL. % SIC 7XXX ALUMINUM COMPOSITE DUE TO CRACK GROWTH BY INTERFACIAL DEBONDING........... 24

1.19 (A) “BEAN” DRAS AND (B) THE RESULTANT R-CURVE BEHAVIOR FOR SEVERAL DIFFERENT MATERIALS RESULTING FROM (C) DUCTILE LIGAMENTS BRIDGING THE WAKE OF THE CRACK…….. 26

1.20 DISTRIBUTION OF STRESS AND STRAIN AHEAD OF A CRACK-TIP………………………………………………………………………………………………… 28

1.21 CRACK EXTENSION IN THE RICE & JOHNSON MODEL BY VOID IMPINGEMENT WITH THE CRACK-TIP........................................................................ 29

1.22 SCHEMATIC REPRESENTING THE GEOMETRY OF CRACK DEFLECTION MODEL............................................................... 35

1.23 CELLULAR MODEL FOR CRACK EXTENSION IN THE MAJUMDAR & PANDEY MODEL............................................................... 39

1.24 COMPARISON OF EXPERIMENTAL DATA AND TOUGHNESS PREDICTIONS FROM THE MAJUMDAR & PANDEY MODEL FOR DUCTILE FRACTURE IN DRA............................................................... 44

1.25 ROUGHNESS MEASUREMENT TECHNIQUES: (A) AVERAGE ROUGHNESS, (B) LINEAL ROUGHNESS, AND (C) THE THOMPSON MICRO-ROUGHNESS........................................................................ 47

1.26 THE FRACTAL KOCH CURVE.................................................................................. 48

1.27 ITERATIONS OF THE KOCH KERNEL THAT LEAD TO THE FRACTAL KOCH CURVE............................................................... 48

1.28 FRACTAL DIMENSION OF THE KOCH CURVE............................................................... 49

1.29 THE RICHARDSON STRUCTURED WALK........................................................................ 50

1.30 EFFECT OF MEAN SEGMENT LENGTH ON FRACTAL DIMENSION........... 52

1.31 EFFECT OF SEGMENT LENGTH STANDARD DEVIATION ON FRACTAL DIMENSION........................................................................ 53
<table>
<thead>
<tr>
<th>Section</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.32</td>
<td>EFFECT OF MEAN ANGULAR SEGMENT DEFLECTION ON FRACTAL DIMENSION</td>
<td>53</td>
</tr>
<tr>
<td>1.33</td>
<td>EFFECT OF SUPER-IMPOSED MICRO-ROUGHNESS/MACRO-ROUGHNESS ON THE FRACTAL BEHAVIOR OF PROFILES</td>
<td>54</td>
</tr>
<tr>
<td>1.34</td>
<td>RESULTS OF FRACTAL ANALYSIS ON STEELS FAILING BY DIFFERENT MECHANISMS, INDICATING THE ABILITY OF FRACTAL ANALYSIS TO IDENTIFY FEATURE LENGTH SCALES</td>
<td>56</td>
</tr>
<tr>
<td>2.1</td>
<td>DEPICTION OF THE POWDER EXTRUSION PROCESS</td>
<td>66</td>
</tr>
<tr>
<td>2.2</td>
<td>EFFECT OF MATRIX/REINFORCEMENT PARTICLE SIZE RATIO ON REINFORCEMENT CLUSTERING IN DRA</td>
<td>67</td>
</tr>
<tr>
<td>2.3</td>
<td>(A) PROCESSING STEPS USED TO CREATE CONDITIONS OF VARYING DEGREES OF MICROSTRUCTURAL HOMOGENEITY</td>
<td>70</td>
</tr>
<tr>
<td>2.4</td>
<td>HARDENING CURVE FOR THE STUDIED COMPOSITE SYSTEM AL-6092/25.0/SICp</td>
<td>71</td>
</tr>
<tr>
<td>2.5</td>
<td>SCHEMATIC DEPICTING A REFERENCE PARTICLE, ITS NEAREST AND SECOND NEAREST NEIGHBORS</td>
<td>73</td>
</tr>
<tr>
<td>2.6</td>
<td>A TYPICAL NEAREST NEIGHBOR DISTRIBUTION, WHERE THE SEPARATION (R) IS NORMALIZED BY PARTICLE DIAMETER (D)</td>
<td>74</td>
</tr>
<tr>
<td>2.7</td>
<td>SCHEMATIC OF THE CHEVRON NOTCH SHORT ROD GEOMETRY USED TO DETERMINE FRACTURE TOUGHNESS</td>
<td>78</td>
</tr>
<tr>
<td>2.8</td>
<td>COMPLIANCE MEASUREMENTS IN A LOAD DISPLACEMENT TRACE USED TO DETERMINE CRACK LENGTH</td>
<td>79</td>
</tr>
<tr>
<td>2.9</td>
<td>FIT OF STRESS INTENSITY COEFFICIENT (Y) VS. RELATIVE CRACK LENGTH (α) FOR W/B = 1.45</td>
<td>80</td>
</tr>
<tr>
<td>2.10</td>
<td>FIT OF RELATIVE CRACK LENGTH (α) VS. DIMENSIONLESS COMPLIANCE (CEB) FOR W/B = 1.45</td>
<td>80</td>
</tr>
<tr>
<td>2.11</td>
<td>CLOSE VIEW OF THE CNSR TEST SETUP</td>
<td>82</td>
</tr>
<tr>
<td>2.12</td>
<td>VIDEO DISPLACEMENT SYSTEM STILL PHOTOS USED FOR THE DETERMINATION OF CRACK LENGTH IN THE 46LT CONDITION AT (A) INITIATION, (B) ARREST, AND (C) NEAR INSTABILITY</td>
<td>84</td>
</tr>
<tr>
<td>2.13</td>
<td>CRACK PROPAGATION OUT OF THE INTENDED PLANE OF FRACTURE DUE TO SPECIMEN MISALIGNMENT</td>
<td>85</td>
</tr>
<tr>
<td>2.14</td>
<td>CROSSING PATTERN FOR THE COLLECTION OF TOPOGRAPHIC MAPS FROM FRACTURE CHEVRON NOTCH SHORT ROD SPECIMENS, AS WELL AS A SAMPLE TOPOGRAPHIC MAP</td>
<td>87</td>
</tr>
<tr>
<td>3.1</td>
<td>EXAMPLE OF FLUCTUATION IN VOLUME FRACTION AS A FUNCTION OF LENGTH SCALE IN DRA 6092/25.0/SICp</td>
<td>90</td>
</tr>
<tr>
<td>3.2</td>
<td>EFFECT OF CLUSTER FACTOR (Fc) ON THE MICROSTRUCTURE ARRANGEMENT IN SYNTHETIC MICROSTRUCTURES</td>
<td>91</td>
</tr>
</tbody>
</table>
3.3  EFFECT OF CLUSTER FACTOR ($F_C$) ON THE HOMOGENOUS LENGTH SCALE ($L_H$) FOR SEVERAL VOLUME FRACTIONS................................. 91
3.4  STANDARD DEVIATION IN VOLUME FRACTION AS A FUNCTION OF LENGTH SCALE................................................................. 92
3.5  APPLICATION OF THE MSAAF TECHNIQUE TO A SYNTHETIC MICROSTRUCTURE................................................................. 93
3.6  APPLICATION OF THE MSAAF TECHNIQUE TO THE SYNTHETIC CLUSTERED MICROSTRUCTURES OF FIG. 3.2......................... 95
3.7  SCHEMATIC DEPICTION OF THE DIRECTIONAL-MSAAF TECHNIQUE FOR THE DETERMINATION OF ISOTROPIC AND ANISOTROPIC HOMOGENOUS LENGTH SCALES IN ANISOTROPIC MICROSTRUCTURES......................................................... 96
3.8  REPRESENTATIVE VOLUME ELEMENT (RVE) FOR THE “1:1” EXTRUSION (INITIAL 20” BILLET); THE ARROWS INDICATE THE DIRECTION OF CONSOLIDATION................................................................. 98
3.9  REPRESENTATIVE VOLUME ELEMENT (RVE) FOR THE 8:1 EXTRUSION; THE ARROW INDICATES EXTRUSION AXIS....................... 99
3.10 REPRESENTATIVE VOLUME ELEMENT (RVE) FOR THE 46:1 EXTRUSION; THE ARROW INDICATES THE EXTRUSION AXIS............. 100
3.11 REPRESENTATIVE LONGITUDINAL (TL) AND TRANSVERSE (TS) SECTIONS OF EACH EXTRUDED CONDITION (1:1, 8:1, AND 46:1)..... 101
3.12 SIZE OF THE REPRESENTATIVE VOLUME ELEMENT DEFINED BY CONSTITUENT/ORTHOGONAL $L_{H,0.10-N}$ AS A FUNCTION OF EXTRUSION RATIO.............................................................................. 102
3.13 RELATIONSHIP BETWEEN HOMOGENOUS LENGTH SCALE ($L_{H1}/L_{H2}$) AND MEDIAN NEAREST NEIGHBOR SEPARATION IN THE PLANE TRANSVERSE TO THE EXTRUSION AXIS.......................... 102
3.14 NORMALIZED NEAREST AND 2ND NEAREST NEIGHBOR DISTRIBUTIONS FOR LONGITUDINAL (TL) AND TRANSVERSE (ST) SECTIONS FROM EACH DEFORMATION CONDITION...................... 103
3.15 EFFECT OF DEFORMATION PROCESSING ON REINFORCEMENT ALIGNMENT ALONG THE LONGITUDINAL DIRECTION FOR ALL DEFORMATION CONDITIONS.................................................. 105
3.16 INFLUENCE OF DEFORMATION PROCESSING ON THE REINFORCEMENT SIZE DISTRIBUTION...................................................... 108
3.17 EFFECT OF DEFORMATION PROCESSING ON LONGITUDINAL (TL) NEAREST-NEIGHBOR INCLINATION FOR THE (A) “1:1”, (B) “8:1” AND (C) “46:1” CONDITIONS.............................................................. 109
3.18 (A) RAW POLE FIGURES, (B) BEST FIT ODFS, AND (C) INVERSE POLE FIGURES FOR THE “1:1” EXTRUSION..................................... 111
3.19 (A) RAW POLE FIGURES, (B) BEST FIT ODFS, AND (C) INVERSE POLE FIGURES FOR THE 8:1 EXTRUSION

3.20 (A) RAW POLE FIGURES, (B) BEST FIT ODFS, AND (C) INVERSE POLE FIGURES FOR THE 46:1 EXTRUSION

3.21 ORIENTATIONS OF MECHANICAL TESTS RELATIVE TO THE REPRESENTATIVE VOLUME ELEMENT OF EACH MATERIAL CONDITION

3.22 (A) LONGITUDINAL (L) AND TRANSVERSE (T) COMPRESSIVE FLOW BEHAVIOR FOR EACH MATERIAL CONDITION AS WELL AS (B) THE INFLUENCE OF EXTRUSION RATIO ON YIELD STRENGTH IN EACH ORIENTATION

3.23 INFLUENCE OF EXTRUSION RATIO ON YIELD STRENGTH IN LONGITUDINAL (L) AND TRANSVERSE (T) DIRECTIONS

3.24 LOAD-DISPLACEMENT/Crack LENGTH MEASUREMENTS FOR SPECIMEN 46LTA

3.25 “RESISTANCE CURVES” FOR REPLICA TES IN THE 46LT CONDITION

3.26 NOTCH-ROOT-CORRECTED CRACK GROWTH RESISTANCE CURVES ($K_I - \Delta A$) FOR ALL CONDITIONS

3.27 FRACTURE SURFACE NEAR ARREST IN THE 1TL CONDITION (SPECIMEN B), ARROW INDICATES DIRECTION OF CRACK PROPAGATION

3.28 FRACTURE SURFACE NEAR ARREST IN THE 8TL (SPECIMEN B) AND 46TL (SPECIMEN C) CONDITIONS

3.29 FRACTURE SURFACE NEAR ARREST IN THE 8LT (SPECIMEN C) AND 46LT (SPECIMEN B) CONDITIONS

3.30 FRACTURE SURFACE NEAR ARREST IN THE 8TS (SPECIMEN D) AND 46TS (SPECIMEN D) CONDITIONS

3.31 LOCAL INFLUENCE OF REINFORCEMENT HOMOGENIZATION ON SURFACE TOPOGRAPHY IN THE TL ORIENTATION

3.32 LOCAL INFLUENCE OF REINFORCEMENT HOMOGENIZATION ON SURFACE TOPOGRAPHY IN THE LT ORIENTATION

3.33 LOCAL INFLUENCE OF REINFORCEMENT HOMOGENIZATION ON SURFACE TOPOGRAPHY IN THE TS ORIENTATION

3.34 INCREASE IN PRIMARY CAVITY GROWTH OVER THE (A) 1:1 CONDITION AFTER (B) EXTRUSION AT 8:1

3.35 CLEAVAGE MARKINGS ON CRACKED PARTICLES

3.36 TOPOGRAPHY COMPARISON OF FRACTURE SURFACES FROM THE TL ORIENTATION (ALL LENGTHS IN $\mu$m), ARROW INDICATES DIRECTION OF CRACK PROPAGATION
3.37 TOPOGRAPHIC COMPARISON OF FRACTURE SURFACES FROM THE LT ORIENTATION (ALL LENGTHS IN μM)…………………………………………………………. 135
3.38 TOPOGRAPHIC COMPARISON OF FRACTURE SURFACES FROM THE TS ORIENTATION (ALL LENGTHS IN μM)…………………………………………………………. 136
3.39 FRACTURE SURFACE ROUGHNESS (Rq) VS. CHEVRON NOTCH TOUGHNESS (KIV) OVER ALL CONDITIONS…………………………………………………………. 138
3.40 FRACTURE SURFACE ROUGHNESS (Rq) VS. CHEVRON NOTCH TOUGHNESS (KIV) BY ORIENTATION…………………………………………………………. 138
3.41 THE (A) MINKOWSKI-BOULIGAND COVERING AND (B) THE HAUSDORFF “BOX-COUNTING” COVERINGS…………………………………………………………. 139
3.42 ACCURACY OF VARIOUS TECHNIQUES FOR CHARACTERIZING THE FRACTAL DIMENSION OF A PROFILE…………………………………………………………. 140
3.43 REPRESENTATIVE VARIATIONAL COVERING OF DUBUC ET. AL……….. 141
3.44 WEIERSTRASS-MANDELBROT COSINE SERIES FOR D = 1.5, γ = 1.2 AND γ = 5……………………………………………………………………………………………… 143
3.45 WEIERSTRASS-MANDELBROT SINE SERIES, γ = 2, FOR VARIOUS D .. 144
3.46 FIDELITY OF THE IMPLEMENTATION OF THE VARIATIONAL METHOD OF DUBUC AND TRICOT TO THE FRACTAL DIMENSION OF THE WEIERSTRASS-MANDELBROT SERIES…………………………………………………………. 145
3.47 MEAN FRACTAL DIMENSION OF FRACTURE SURFACES FROM EACH CONDITION IN THE TL ORIENTATION…………………………………………………………. 148
3.48 MEAN FRACTAL DIMENSION OF FRACTURE SURFACES FROM EACH CONDITION IN THE TS ORIENTATION…………………………………………………………. 148
3.49 MEAN FRACTAL DIMENSION OF FRACTURE SURFACES FROM EACH CONDITION IN THE LT ORIENTATION…………………………………………………………. 149
3.50 FRACTURE TOUGHNESS (KIV) VS. FRACTURE SURFACE FRACTAL DIMENSION (D) FOR THE TL ORIENTATION…………………………………………………………. 149
3.51 FRACTURE TOUGHNESS (KIV) VS. FRACTURE SURFACE FRACTAL DIMENSION (D) FOR THE TS ORIENTATION…………………………………………………………. 150
3.52 FRACTURE TOUGHNESS (KIV) VS. FRACTURE SURFACE FRACTAL DIMENSION (D) FOR THE LT ORIENTATION…………………………………………………………. 150
4.1 COMPARISON OF MEDIAN NEAREST NEIGHBOR SEPARATION WITH OTHER MODELS USED IN DUCTILE FRACTURE…………………………………………………………. 153
4.2 COMPARISON OF MEASURED LONGITUDINAL (L) AND TRANSVERSE (T) YIELD BEHAVIOR WITH TEXTURE PREDICTIONS (EQ. 4.1) AS A FUNCTION OF EXTRUSION RATIO…………………………………………………………. 155
4.3 RELATIONSHIP BETWEEN NEAREST NEIGHBOR SEPARATION AND FRACTURE RESISTANCE BY ORIENTATION…………………………………………………………. 155
4.4 SCHEMATIC DEPICTION OF THE INFLUENCE OF CRACK DEFLECTION ON ROUGHNESS PARAMETERS…………………………………………………………. 158
4.5 INFLUENCE OF CRACK DEFLECTION ANGLE AND FRACTURE PROCESS LENGTH SCALE ON FRACTURE ROUGHNESS ................................. 159
4.6 FRACTAL DIMENSION AS A FUNCTION OF CRACK DEFLECTION ANGLE, AFTER DAUSKARDT ET. AL................................................. 161
4.7 COMPARISON OF MEASURED TOUGHNESS ($K_{II}$) AND THAT PREDICTED BY THE MAJUMDAR & PANDEY MODEL.......................... 163
4.8 $\beta$ AS A FUNCTION OF MEAN CRACK DEFLECTION ANGLE <$\theta$> AND LIGAMENT PLASTIC STRAIN ($\varepsilon_p$) THAT CAN BE SUPPORTED PRIOR TO RUPTURE........................................................................ 165
4.9 PHENOMENOLOGICAL MODELS RELATING $\beta$ TO NEAREST NEIGHBOR SEPARATION ($L/D$) FOR ALL ORIENTATIONS...................... 166
## LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1</td>
<td>COMPOSITION OF ALUMINUM 6061 AND 6092, BY WT. %</td>
<td>69</td>
</tr>
<tr>
<td>3.1</td>
<td>PARTICLE DISTRIBUTION, SIZE, AND ASPECT RATIO (AR)</td>
<td>104</td>
</tr>
<tr>
<td></td>
<td>STATISTICS FOR LONGITUDINAL (TL) AND TRANSVERSE (ST)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SECTIONS OF DRA 6092/25.0/SICp.</td>
<td></td>
</tr>
<tr>
<td>3.2</td>
<td>PARAMETERS CHARACTERIZING MATERIAL COMPRESSIVE</td>
<td>117</td>
</tr>
<tr>
<td></td>
<td>DEFORMATION RESPONSE</td>
<td></td>
</tr>
<tr>
<td>3.3</td>
<td>FRACTURE TOUGHNESS ($K_{IV}$) DATA COMPILED BY</td>
<td>121</td>
</tr>
<tr>
<td></td>
<td>CONDITION</td>
<td></td>
</tr>
<tr>
<td>3.4</td>
<td>FRACTURE SURFACE AVERAGE ROUGHNESS ($R_{a}$) COMPILED BY</td>
<td>137</td>
</tr>
<tr>
<td></td>
<td>CONDITION</td>
<td></td>
</tr>
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<td>FRACTURE SURFACE FRACTAL DIMENSION ($D$) BY</td>
<td>147</td>
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<td>CONDITION</td>
<td></td>
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<tr>
<td>4.1</td>
<td>FRACTURE SURFACE FRACTAL DIMENSION (DF) AND MEAN</td>
<td>161</td>
</tr>
<tr>
<td></td>
<td>CRACK DEFLECTION ANGLE ($\theta$)</td>
<td></td>
</tr>
<tr>
<td>4.2</td>
<td>MEASURED FRACTURE TOUGHNESS OF EACH COMPOSITE BY</td>
<td>164</td>
</tr>
<tr>
<td></td>
<td>CONDITION, AS WELL AS THAT PREDICTED BY THE MODEL OF</td>
<td></td>
</tr>
<tr>
<td></td>
<td>MAJUMDAR &amp; PANDEY, AND THE PARAMETER $\beta$ NECESSARY FOR</td>
<td></td>
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<tr>
<td></td>
<td>AGREEMENT</td>
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ACKNOWLEDGEMENTS

With age I gain a better appreciation for how many people have generously invested their time into me. Whenever I am disappointed, their selflessness is inspiring; whenever I feel apathetic, the thought of disappointing them provokes a renewed effort—their memory will live on through me and my willingness to return their favor to others. While the list of people I should specifically thank for enabling this work begins far before its onset, those most important to its completion include a colorful cast of characters that I am proud to know. Most importantly, I would like to thank my advisor, Don Koss for engineering the opportunity to do this work, his poignant insights, correcting several of my bad habits, and his general “human intelligence” from which I have learned a great deal. Along a similar vein, I would also like to thank Jonathan Spowart and Dan Miracle for their insight, support, patience, and friendship. My office mates who have made both my work and my life more enjoyable also deserve recognition: Bence Bartha, for being an eerily kindred spirit, and Michael Glavcic, for his friendship and certainly his assistance in texture measurement; I have learned a great deal about this subject from him, its effect on mechanical behavior, and even nickel and titanium processing—especially late in the evening when Lee comes into the office to rattle his cage. Thanks are also certainly due to Mike Scott for his assistance with mechanical testing and Oleg Senkov for his timely commentary and several very fruitful conversations. I would also like to recognize the management of the development section and metals branch in the Air Force Research Laboratory’s Materials & Manufacturing Directorate for their support, including Steve Russ for sneaking up on me, kicking the back of my chair, and shouting “write!”, as well as Rollie Dutton, who with a simple glance and a smile could make me feel guilty about not having completed my degree. Last, but most certainly not least, I would like to thank my father Ben for being a sounding board and “the man up in the booth.” I can only hope someday to be as a good a father as he has been.
INTRODUCTION & BACKGROUND

1.1.) INTRODUCTION

The primary interest in metal matrix composites (MMCs) stems from their ability to offer excellent structural efficiency, including high specific strength ($\sigma/\rho$) and especially high values of specific stiffness ($E/\rho$) that are unattainable in most conventional metallic systems, as can be seen in fig. 1.1. Although greater absolute specific properties can be achieved along the fiber axis in continuous fiber-reinforced composites, discontinuously (particle) reinforced MMCs can generate significant improvements in specific properties over conventional alloys with far less anisotropy in their mechanical behavior than continuous fiber composites. This isotropy particularly affects both elastic properties and fracture behavior—critical properties necessary for broadening the scope of application for these materials—and furthermore, permits material processing by conventional casting and powder metallurgy routes.

![Figure 1.1: Specific property chart for conventional metals and metallic composites [1].](image)

Although the elevated cost of MMCs does not justify their implementation for many consumer applications, discontinuously reinforced metal matrix composites have carved a
niche in a number of high-end commercial and military applications [1]; examples include: specific stiffness applications such as high performance drive shafts for automobiles, and exterior access panels (skin) on aircraft (fig. 1.2a). The excellent behavior of these discontinuously reinforced MMCs in wear and creep has also facilitated their implementation as cylinder liners for automobile engines (fig. 1.2b), while their similarly outstanding behavior in high cycle fatigue has seen them become a material of choice for certain (low temperature) air control surfaces on jet engines and military aircraft (fig 1.2c, 1.2d) [1].

Figure 1.2: (a) DRA F-16 Fuel Access Panels, (b) DRA Honda Prelude Cylinder Liners, (c) DRA F-16 Ventral Fin, and (d) DRA Fan-Exit guide vane for Pratt & Whitney 4XXX engines [1].
The material system for a number of these applications is some variant of a discontinuously reinforced aluminum (DRA) composite, with the result that this material is perhaps the most prolific metallic composite for structural applications—a tribute to its effective balance of mechanical properties. Unfortunately, as is the case with most advanced structural materials, the improvements in structural efficiency, creep, and fatigue of DRA’s over conventional aluminum alloys often come with a severe penalty in fracture resistance and ductility.

Since resistance to fracture constrains the broader application of DRA’s, it has been a subject of extensive study. Researchers have shown that DRA’s are sensitive to the most significant parameters that influence fracture in conventional aluminum alloys: yield strength, strain hardening, and second-phase (reinforcement) volume fraction [2]. However, the fracture resistance of DRA’s has also been related to a number of other variables, including, but not limited to the following: reinforcement distribution, composition, and shape, size/strength/Weibull Modulus, as well as matrix alloy and texture, heat treatment, residual stress, and matrix/reinforcement interfacial strength [3,4,5,6,7,8,9].

This thesis focuses on the effect of perhaps the most important un-quantified variable, the degree of reinforcement homogeneity, and in particular, attempts to understand the often observed debit in fracture toughness caused by reinforcement clustering via changes in the reinforcement separation, culminating with a modification to existing ductile fracture models to assist in its account. While the results of this study will be directly applicable to DRA’s, aspects of these results should be applicable to other metallic composites and heterogeneous materials such as the cemented carbides, and certain multi-phase high-temperature intermetallic alloys (ex: Mo-Si-B, Nb-Si).

1.2.) MECHANICAL BEHAVIOR OF DRA

Generally, DRA’s demonstrate low tensile ductility in which failure is controlled by crack initiation and some limited crack growth, reminiscent of, but not identical to the
ductile fracture of conventional high strength aluminum alloys [10]. In these composites, crack growth proceeds first by the nucleation of damage ahead of the crack tip—either cracked or debonded reinforcement—and is subsequently followed by the thinning of the ligament between this nucleated damage and the crack tip until ligament instability causes coalescence of this damage (and any ensconcing cavity growth) with the crack tip. This thinning and coalescence is the result of the imposition of small scale yielding under plane-strain conditions, in which the crack-tip stress field provides the mechanical constraint necessary to induce flow localization within the damage field on planes of high shear stress inclined relative to the crack plane. After a certain amount of crack extension, global mechanical constraints intercede and discourage continued propagation of an out-of-plane crack. Such a fracture process results in a rough, often tortuous fracture path consisting of a characteristic sawtooth, or “zig-zag” ductile fracture surface constructed of ligaments inclined ~45° relative to the fracture plane (fig. 1.3c)[11].

The strong microstructural heterogeneity of discontinuously reinforced MMCs however may lead to a variety of other interacting damage mechanisms which are ultimately determined by material composition and processing. This section therefore outlines specific studies of crack growth and damage propagation, as well as deformation response, as a function of important microstructure variables in order to set the stage for the models used to quantify crack initiation and growth.

*Reinforcement Volume Fraction*

From the onset of the study of the fracture of discontinuously reinforced MMCs, it was clear that volume fraction of reinforcement ($V_p$) has a significant influence on both deformation and fracture, with higher volume fractions leading to higher yields strengths and lower fracture resistance. The improvement in yield strength with volume fraction is a result of improved load shedding from the matrix to a greater volume fraction of higher stiffness and strength reinforcement, which for a given level of composite strain will disproportionately carry more load than the matrix up until damage initiation (usually concomitant with composite yield) [12]. This and many other aspects of mechanical
Figure 1.3: Modes of fracture in Discontinuously Reinforced Aluminum (DRA).
behavior of discontinuously reinforced metallic composites have been modeled by Bao, Hutchinson, and McMeeking [13]. Taking the simplest possible case of uniformly distributed rigid spheres in an elastic-perfectly-plastic matrix allowed for a straightforward finite element analysis of a square unit cell with periodic boundary conditions in which the volume fraction of the composite is the volume of the sphere relative to the volume of the unit cell (a cross section of which is depicted in the inset of fig. 1.4). The results of this analysis for uniaxial loading, depicted in fig. 1.4, show the general increase in equivalent flow stress of the unit cell $\sigma$ (normalized by the matrix yield strength, $\sigma_0$) with volume fraction as well as the accompanying expansion of the non-linear region between the onset of yielding and plastic flow. However, such studies based on representative volume elements do not address the effect (if any) of reinforcement clustering, as their microstructure consists of regular arrays of reinforcing particles.

![Figure 1.4: Idealized influence of reinforcement volume fraction on composite flow behavior [13].](image)

The increase in yield strength with volume fraction can lower toughness by encouraging flow localization, but—as will be discussed later—it is not the solitary source of the decrease in fracture resistance that accompanies increasing reinforcement volume.
fraction. The starkness of the trend of decreasing toughness with reinforcement volume fraction is captured quite well in a review article by Lewandowski in which data from several studies, where a measure of both toughness ($K_Q$, $K_{Ic}$) and volume fraction of reinforcement was available, was compiled and plotted, as seen in figure 1.5 [14]. Clearly visible from this plot is an upper bound on toughness as a function of reinforcement volume fraction that decreases steadily with increasing reinforcement loading.

![Figure 1.5: Effect of volume fraction on fracture resistance of numerous DRA systems [14.](image)](image)

When the mechanics of damage advance are identical among materials containing varying volume fractions of reinforcement, this *upper bound* on toughness tends to follow a $V_p^{-1/6}$ dependence—in close concord with the fracture toughness of high strength aluminum alloys [15]. Unfortunately, volume fraction is only accurate in describing the effect of reinforcement distribution on fracture resistance in homogenously reinforced materials—hence its utility in defining an upper bound on this quantity—however it can be grossly inaccurate at capturing the dependence of toughness in clustered (inhomogeneous) microstructures. Though other effects to be discussed certainly
contribute, the potential influence of reinforcement distribution is illustrated in fig. 1.5 by the absence of any clear lower bound on fracture toughness as a function of volume fraction. Furthermore, the reader will note that the greatest variation in fracture resistance occurs at low reinforcement loadings, where the greatest flexibility in reinforcement configuration exists.

**Matrix Microstructure**

As in conventional aluminum alloys, there is a strong effect of heat treatment on the mechanical behavior of discontinuously reinforced aluminum matrix composites. Typically, age hardening in DRA’s influences flow behavior in a manner similar to age-hardening in conventional aluminum alloys, and this in-turn influences crack growth through the effect of strain hardening and yield strength on crack tip plasticity, though the interaction of reinforcement particles with precipitation kinetics can produce both favorable and unfavorable consequences.

Like in conventional age-hardenable alloys, over age (OA) heat treatments can lead to a lower toughness than an underage treatment (UA) with the same yield strength—as can be seen in fig. 1.6 compiled by Lewandowski for a number of composite systems [14]—due to precipitate coarsening and concomitant embrittlement. However, there is also a strong potential for embrittlement of the matrix/reinforcement interface by way of preferential precipitation at the interface and the attendant formation of a precipitate free zone (PFZ) in an adjacent layer of the matrix. Crack growth in such a material proceeds entirely by interfacial debonding, dramatically reducing the fracture resistance of the composite system when compared to similar materials in which crack growth proceeds by particle cracking.
Figure 1.6: Effect of heat treatment on toughness and yield strength for several DRA’s [14].
For example, Lewandowski studied a 7000 series aluminum matrix composite reinforced by 15 and 20 vol. % SiC particles (~13 μm diameter) which was tempered to both under age (UA) and over age conditions, and compared the behavior of this composite to the “neat” (reinforcement free) matrix alloy [16]. Initiation toughness values in the UA and OA conditions of the neat alloy were nearly identical (~30 kJ/m²), while the same heat treatments produced values of 16.3 and 7.4 kJ/m² respectively for the 15 vol. % material, and 11.7 and 5.5 kJ/m² respectively for the 20 vol. % material. TEM observations of the material confirmed the interfacial precipitation of a Zn-rich phase and the formation of the PFZ, while SEM fractography depicted a clean interface, or a very thin “spider-web” of matrix material on reinforcement surfaces in the OA condition (characteristic of interfacial debonding), and cracked (or cleavage marked) particles in the UA condition (fig. 1.7a and b, respectively), regardless of reinforcement volume fraction.

![Figure 1.7: Depictions representative of the (a) particle cracking in an UA heat treatment of a DRA and (b) interfacial debonding in an OA heat treatment observed by Lewandowski.](image)

It should be noted that embrittlement does not necessarily occur during over-age treatments in all matrix alloys. In those alloys where precipitation is not preferential to the interface of the reinforcement—6000 series matrices, or 7000 series matrices subject to a truncated over-age treatment (compared to the “neat” matrix)—recovery of
toughness in the over-age condition comparable to that in the under age condition does occur since particle cracking tends to be the dominant mechanism of crack propagation [17]. In fact, it has been experimentally verified by Nagarajan and Dutta that the formation of a PFZ around a reinforcement particle and in the absence of an embrittling phase at the interface can be obtained through a 2-step heat treatment, resulting in an extremely beneficial increase in toughness in a slightly over aged condition [18]. In that study, a 6092 alloy reinforced with 17.5 vol. % SiC was solutionized, quenched, taken to peak age, quenched, reheated briefly at a higher temperature (below the solution temperature), and quenched a final time. This heat treatment dissolved the fine coherent Guinier-Preston Zones and precipitates near the reinforcement and rapidly allows for the absorption of vacancy clusters at the incoherent reinforcement interface—which would normally serve as sites for heterogeneous nucleation—producing a PFZ several microns in extent around the reinforcement without any interfacial precipitation, resulting in a toughness of 31 MPa√m, a value that exceeds the toughness of a nearly identical unreinforced alloy (6061) by 4 MPa. This behavior was presumably the result of an increased ductility in the interparticle ligament after damage nucleation (particle cracking) and prior to coalescence with the crack tip.

Figure 1.8: (a) Schematic depiction and (b) near interface TEM photograph of a Precipitate Free Zone (PFZ) that is 1-2 μm thick in DRA 6092/15/SiCp [18].
Perhaps the most novel illustration of the effect of matrix microstructure on mechanical behavior comes from studies of the pure aluminum matrix composites produced by the Mortensen group [19-24]. Recognizing that many variables contributing to mechanical behavior were present in discontinuously reinforced metallic composites and that separating the cross interaction of these variables was experimentally cumbersome, Mortensen and co-workers extensively studied “simplified” aluminum matrix composites in which materials were produced by gas-pressure-driven infiltration of molten pure aluminum into a cavity containing a high packing fraction of ceramic particles, resulting in materials free of any variations in matrix microstructure and—since it contained a high volume fraction of reinforcement particles (often in excess of 40 vol. %)—any significant variation in reinforcement distribution. These materials have excellent specific stiffness and toughness, though they are somewhat impractical due to their low yield strength.

The primary source of toughness in these materials is in fact attributable to their low yield strength. As a consequence of their low yield strength, the condition of small scale yielding is lost as is the concomitant elastic constraint in most common fracture specimen geometries, and for that matter, structural components. Consequently, there is no great driving force for flow localization, and fracture favored by high stress triaxiality does not necessarily occur in a tortuous “zig-zag” manner, and certainly not by deformation localization between damaged particles. In this regime, local mechanisms for failure are not as pronounced or important during fracture as generally the dissipation of energy plastically ahead of the crack (fig. 1.3d) overwhelms the local energy for crack advance in terms of contribution to the total work of fracture [22-24].

Reinforcement Composition, Size, and Strength

Reinforcement composition strongly impacts composite stiffness and damage accumulation behavior. Specifically with regard to the damage accumulation, which itself is strongly related to reinforcement size and strength, reinforcement composition and prior processing history can affect both the propensity for particle cracking and interfacial reactions with the matrix. Reinforcement size on its own tends to affect mechanical
behavior, with smaller particle sizes comparatively leading to much higher work hardening rates, as well as improved Weibull strength (resistance to stress induced cracking), which can indirectly improve the deformation response of a composite and its resistance to fracture. Nonetheless, the strong interaction of these parameters requires that they be discussed together.

The most common reinforcement compositions that tend to be chemically inert in an aluminum matrix are, SiC, B₄C, and Al₂O₃, though virtually all commercial structural DRA’s are reinforced with SiC, mainly because of availability and cost. Interestingly, the author is unaware of any study that incorporates partially stabilized zirconia (PSZ) as reinforcement in an discontinuously reinforced aluminum matrix composite, since its propensity for deformation induced transformation (and simultaneous volume change) should provide a significant compressive back-stress in the vicinity of a growing crack and retard crack growth in a manner similar to its application in certain technical ceramics [25].

The effect of reinforcement stiffness on composite stiffness is nearly universal regardless of particle processing history, leading to a monotonically positive relationship between reinforcement stiffness—as a function of composition—and composite stiffness through rule-of-mixtures type analyses [26]. The effect of reinforcement size on plastic deformation response is best illustrated by the work of Hunt who processed Al 2080 matrix composites reinforced with three different mean particle diameters—F-1000 (4 μm), F-600 (10 μm), and F-280 (36 μm)—at several volume loadings (10%, 15%, 20%, and 30 %) [27]. The effect of the variation of these parameters on the relationship between toughness and yield is depicted in fig. 1.9. Generally, the effect of volume fraction overwhelms any noticeable contribution of particle size to the inverse relationship between composite yield strength and toughness. However, the effect of particle size on ultimate strength is more dramatic, and is captured in fig 1.10 which shows that decreasing particle size can significantly increase the ultimate strength. When combined with the nominal effect of particle size on yield, the implication is that smaller particle sizes greatly enhance the work hardening rate, irrespective of volume fraction.
Figure 1.9: Relationship between toughness and yield strength in Al 2080/SiC/Xp as a function of reinforcement volume fraction and particle size [27].

Figure 1.10: Relationship between toughness and ultimate strength in Al 2080/SiC/Xp as a function of reinforcement volume fraction and particle sizes [27].
A secondary effect of particle size on composite toughness comes through the interaction of the particle-size/work-hardening-rate relationship on crack tip plasticity, which will be discussed in a later section. This is however, overshadowed by the effect of reinforcement size on Weibull modulus and the propensity for reinforcement cracking with deformation. Generally, smaller reinforcement particles tend to have a higher Weibull modulus—due to a decreased likelihood that they contain pre-existing flaws—and consequently, depending on the reinforcement composition, can require greater composite \textit{in situ} stresses to fracture [28]. Conversely, an increased propensity for particle fracture can severely debit the flow behavior after yield and greatly accelerate the fracture process. Theoretically, if the particle size was small enough—and the matrix/reinforcement interface was strong enough—it may be possible to induce a fracture mode reminiscent of ductile fracture in conventional alloys where crack growth would be dominated by inter-reinforcement void nucleation, coalescence, and growth.

Unfortunately, in the work by Hunt, the effect of particle size—and its interaction with particle strength—on toughness is overwhelmed by the effect of volume fraction. This is not the case in the work of Kouzeli, Weber, San Marchi, and Mortensen, who studied damage accumulation rates as a function of particle size and composition in pure aluminum matrix composites subject to uniaxial tension [21]. In this study, the evolution of two damage parameters ($D_E, D_p$) corresponding to the percentage decrease in elastic modulus and composite density with increasing strain were measured via changes in elastic compliance and the Archimedes method, respectively, in composites reinforced with varying sizes of either BN or Al₂O₃. Optical microscopy confirmed that decreases in stiffness in these materials were primarily a result of particle cracking, while decreases in density were a result of inter-reinforcement void nucleation and growth. As is illustrated in fig 1.11a-d—where materials are labeled with a prefix describing their mean particle size in microns and a postfix describing their composition (A-alumina, B-boride)—the stronger boride reinforced composites accumulated cracked particles much more slowly than the weaker alumina reinforced composites, while the opposite trend was observed in void growth. In all conditions, both types of damage were greatly accelerated by particle size.
Figure 1.11: (a)-(d) Influence of reinforcement composition and mean particle diameter on crack and void damage accumulation rates, as measured by the damage parameters $D_E$ and $D_\rho$ [21].

**Reinforcement Morphology**

Particle shape can strongly influence both the deformation and fracture of discontinuously reinforced metallic composites. Composite yield strength is improved with increasing reinforcement surface-area-to-volume ratio (increasing aspect ratio, and to a more limited effect, decreasing particle size) since this parameter strongly influences load transfer from the matrix to the higher stiffness reinforcement [29]. Simultaneously though there is a negative effect of this strengthening on work hardening rate and toughness due to increased flow localization between high aspect-ratio particles, especially chopped fibers [30].
In order to quantify the effect of particle morphology on composite deformation behavior, Bao, Hutchinson, and McMeeking studied—via finite element modeling of a particle in a unit cell with periodic boundary conditions—the influence of aligned prolate ellipsoidal particles in an elastic-perfectly-plastic matrix, as well as aligned cylinders and disks in a matrix conforming to a Ramberg-Osgood power hardening law given by

$$\varepsilon = \frac{\sigma}{E} + \alpha \left( \frac{\sigma}{E} \right)^N$$

(1.1)

where $\varepsilon$ is the applied strain, $\sigma$ is the matrix stress, $\alpha$ is a constant equal to 3/7, $E$ is the elastic modulus, and $N$ is the so-called stress exponent which is the inverse of the strain hardening exponent ($n$) [13]. Results of this analysis for a volume loading of 20% ellipsoidal reinforcement in an elastic-perfectly-plastic matrix (fig 1.12) indicate that higher reinforcement aspect ratios improve flow resistance ($\sigma_c/\sigma_o$) regardless of whether the load is applied parallel or perpendicular to the direction of particle alignment; flow curves for particles of aspect ratio 1, 5, and 10, parallel to the reinforcement major axis are designated by $a/b = 1, 0.2, \text{ and } 0.1$, and transverse by $a/b = 1, 5, \text{ and } 10$, respectively.

**Figure 1.12:** Influence of aspect ratio on the aligned and transverse uniaxial deformation response of a composite containing 20 vol. % aligned prolate ellipsoids of aspect ratio 1, 5, and 10 [13].
Similar results followed for disks and cylinders in a hardening matrix, though the influence of matrix strain hardening exponent was far more important in improving composite strain hardening than reinforcement aspect ratio. The near yield transition region that was discussed earlier—regarding the influence of the volume fraction of rigid spheres on flow behavior—also appears in the case of non-spherical particles in a work hardening matrix. In this case, not only does increasing volume fraction increase the width of this transition region, but so also does increasing reinforcement aspect ratio. Furthermore, for these non-spherical reinforcement geometries, the authors demonstrated that the strain hardening exponent for the composite \( N \), when outside of this transition region and in analogy to spherical particles, is identical to that of the matrix, though they are careful to point out that this may not be observed in some experiments due to the often low ductility of these composites. Ultimately, the authors found that a composite Ramberg-Osgood power hardening law, of the form

\[
\varepsilon_c = \frac{\sigma_c}{E_c} + \alpha \left( \frac{\sigma_o}{E_m} \right) \left( \frac{\sigma_c}{\sigma_N} \right)^N
\]

\hspace{1cm} (1.2)

fit their data very well, where \( N \) is the reciprocal of the strain hardening exponent \( (N = 1/n) \), \( \sigma_o \) is the yield strength of the matrix, \( \sigma_c, \varepsilon_c, E_c \) are the composite stress, total strain, and modulus respectively.

Unfortunately, this idealized flow behavior is only valid up until the initiation of damage, where the flow behavior becomes, with each increment of strain, more and more sensitive to those aspects of reinforcement morphology that contribute to deformation localization and limit ductility: damage initiation, growth, and coalescence. Realizing from previous experimental studies on reinforcement shape [31] that sharp, angular corners can lead to high local hydrostatic stresses in a composite even at relatively low loads—which would lead to relatively premature damage nucleation and growth with deformation—Spowart and Miracle studied the effect of reinforcement shape on the tensile ductility of two identically processed, powder metallurgy (P/M) composites containing 25 vol. % SiC in a 6061 matrix. The first composite contained an F-600 grade reinforcement (fig. 1.13a)
while the other contained a “high bulk density” (HBD) reinforcement (fig. 1.13b) obtained by mechanical comminution of the grit powder.

![Comparison of the morphological difference between two types of reinforcement used in a Al-6061/SiC/25_p composite, (a) an F-600 (grit) powder and (b) the HBD powder [31].](image)

By studying 3 different conditions of the two composites, including an “as-extruded” (AE) condition, as well as peak age (PA) and over age (OA) heat treatments, the authors observed a strong increase in tensile ductility with the HBD reinforcement (fig 1.14).

![Mechanical behavior of the F-600 and HBD reinforced Al-6061/SiC/25_p composites in the (a) as extruded and (b) heat treated (OA and T6) conditions [31].](image)
Though not under controlled crack growth conditions, this increase in ductility should primarily reflect a trend of increasing toughness, accompanying which was a large change in fracture surface morphology (fig. 1.15), as can be specifically seen in the macroscopically featureless fracture of the F-600 reinforced composite (AE condition) when compared to the tortuous region of stable damage growth (inside the ensconcing shear cone) in the HBD reinforced composite (also in the AE condition). Accompanying this change in morphology was a large increase in primary cavity growth stemming from cracked particles in the HBD material over the F-600 material. The primary damage mechanism in both materials (fig. 1.16) gradually transitioned from particle cracking to a near-interface microvoid coalescence with increasing duration of the heat treatment—and increasing precipitate coarsening—suggesting that the strength of the particles (resistance to fracture) had little to do with the improvement in fracture resistance. It was more likely rather that the decreased hydrostatic stress in the interparticle ligament of the HBD condition slowed the rate of void nucleation and growth with deformation which lead to higher critical strains prior to ligament collapse.

Figure 1.15: Influence of heat treatment on global fracture morphology in (a) F-600 and (b) HBD reinforced Al-6061/SiC/25_p composites [31].
Figure 1.16: Influence of heat treatment on fracture morphology and local fracture topography in F-600 and HBD reinforced Al-6061/SiC/25_p composites [31].
Using a tensile stage *in situ* in an SEM, Davidson observed damage propagation in a 2014 Alloy reinforced with 15 vol. % SiC heat treated to peak age, and in combination with surface deformation mapping, recognized that ductility was limited by regions of high particle density in which cracks and damage were nucleated at very low strains [32]. Even though this technique indicated that local plastic strains often exceeded 50%—comparable to those achieved in “neat” aluminum alloys—the author argued that the presence of tearing ridges rather than purely dimpled rupture on the fracture surface during crack propagation indicated that the strain to damage coalescence was not due to a change in the intrinsic ductility of the matrix in clustered regions, but constraints on plastic flow brought on by adjacent damage nucleation.

Lewandowski, Liu, and Hunt used double-notched 4-point bend specimens to ascertain the effect of particle clustering on crack initiation in a 20 vol. % SiC reinforced 7000 series alloy [3]. After fracture of the primary notch, transverse sections of the unbroken notch—representative of the material immediately prior to fracture—indicated qualitatively that damage nucleated preferentially inside of particle clusters. In the over-age condition this damage consisted of interfacial debonding, while in the under-age condition this damage consisted primarily of particle cracking.

Since DRA’s with significant loadings of reinforcement often fail with little ductility, crack propagation can proceed quite rapidly. Therefore, a novel specimen configuration was employed in this same study to understand the growth of damage during crack propagation. This specimen geometry was essentially a standard CT specimen made of the studied DRA machined with a thick ductile backing of presumably the neat matrix Al (7000 series) alloy (fig. 1.17a). The ductile backing retarded crack growth to the point where the propagation of damage occurred at a rate that could be observed under stable crack growth conditions via a traveling microscope. Primarily observed during these controlled crack growth experiments is that when particle cracking occurs, damage occurs in particles *not* previously cracked (by deformation processing). Furthermore, by
comparing Voronoi tessellations of the region participating in fracture to randomly sampled regions of the microstructure, it was observed that crack propagation initiated and proceeded to grow through regions of high local volume fractions—clusters of reinforcement (fig. 1.17b), and in both heat treatments was very localized—limited to several (2-3) particle diameters away from the fracture surface—implying that crack growth resistance in these materials may be primarily controlled by the strain necessary to nucleate damage rather than the strain necessary to grow and coalesce this damage.

Figure 1.17: (a) A novel CT geometry for determining mechanisms of crack propagation in DRA, and (b) the resulting depiction of crack propagation through clustered (high $V_f$) particle regions [3].

Comparison of the UA and OA conditions in the same study elucidates a trend of much more diffuse damage (microcracking ahead of the tip/unbroken interparticle ligaments behind the crack tip) in the OA condition—when interfacial debonding is nucleated ahead of the tip—as well a strong propensity for crack bifurcation (fig. 1.18). Presumably this is
a result of the combination of the reinforcement inhomogeneity and the low strength of the interface in comparison to the Weibull strength of the reinforcement since no such micro-cracking/unbroken ligaments in the wake exist in the UA condition—when crack propagation proceeds by particle cracking.

Figure 1.18: Diffuse crack growth with unbroken ligaments in the crack wake of a 20 vol. % SiC 7XXX Aluminum composite due to crack growth by interfacial debonding [3].

When the reinforcement itself or the matrix/reinforcement interface is very weak, it is possible to imagine that the fracture process zone could delocalize (fig. 1.3b & 1.18), in which case it would be necessary to consider a non-classical mechanical analysis of the near crack-tip stress and strain fields since the damage could extend so far ahead of the position of maximum strain that the crack tip and the position of maximum strain are no longer coincident. In this scenario unbroken (bridging) ligaments in the wake of the crack effectively “shield” the crack-tip from the stress intensity it would otherwise experience. While unbroken ligament bridging has been observed in a monolithic discontinuously reinforced composite by Davidson [32], strong evidence—a decrease in toughness with an increase in unbroken ligament density—suggests that it is not as significant as bridging schemes for extrinsic toughening in other strongly heterogeneous systems such as the cemented carbides [33], ceramic matrix composites (CMCs) [34], concrete [35],
fiber-reinforced/laminate metallic composites [36], cancellous bone [37], and duplex intermetallic compounds [38].

The boundary between particle-by-particle ductile crack extension and this large-scale bridging though is certainly blurred by the above observations of Lewandowski and other authors who have shown that often there are many particles that fracture or decohere within the vicinity of the crack tip—particularly in high strength systems—though not all participate in the fracture process [3]. The only real criterion for distinguishing whether this micro-cracking and discontinuous crack behavior can contribute significantly to the fracture process is if there is a large increase in crack-growth resistance unequivocally attributable to this “discontinuous” crack growth. With regard to the just-discussed work of Lewandowski, it is unlikely that the diffuse damage in the OA condition contributed significantly to the fracture resistance since a separate study of this same material by the same author showed that the initiation toughness of the UA condition (11 kJ/m²) is approximately twice that of the OA condition (5.5 kJ/m²), while the tearing modulus—a dimensionless measure of crack growth resistance which should indicate the potency of this extrinsic toughening by bridging/microcracking—is likewise greater by one order of magnitude in the UA condition than the OA condition (1 compared to 0.1).

In contrast though, it is possible to capitalize on the concept of elastic shielding/bridging with reinforcement inhomogeneity via a form of ductile phase toughening, as has been obtained in a so-called “bean” DRA processed by Majumdar, Pandey, and Miracle in which large aluminum particles were incorporated during the consolidation of an otherwise “typical” (monolithic) DRA (fig. 1.19) [39]. These large aluminum particles bridge the wake of the crack and lead to appreciable resistance curve (R-curve) behavior, in stark contrast to the low-strength/plasticity related R-curve demonstrated in other composite systems [40, 24], though both are related to the loss of intensity in the tip singularity that occurs with their particular kind of crack growth.
It has been observed that particle clustering promotes the nucleation and propagation of damage in DRA’s and generally contributes to decreased fracture resistance. However, since reinforcement distribution has not been controlled in any quantifiable manner—through careful processing, for example—it is difficult from the available studies to ascertain the magnitude of any possible variation of reinforcement distribution on the mechanisms of damage nucleation, propagation, and the resulting fracture initiation toughness and crack growth resistance. The issue of reinforcement distribution is especially important for materials with low reinforcement volume fractions where significant variations in fracture toughness are often observed, but it tends to be muted at higher loadings where the fracture resistance is generally poor and the material is unsuitable for structural applications.
1.3.) SPECIFIC CRACK GROWTH MODELS FOR DRA

Several models have been used to describe the *intrinsic* fracture toughness of DRA and similar metallic-based ductile materials in the absence of *extrinsic* toughening mechanisms (bridging, crack deflection). The interrelation and development of those models most relevant to the problem at hand will be outlined in the order of their evolution after a brief synopsis of crack tip plasticity and the mechanics of damage growth. When appropriate, the relationship of these models to extrinsic toughening mechanisms will also be discussed.

*Crack-Tip Plasticity*

McMeeking used a finite element mesh to analyze the state of stress and strain ahead of a *stationary* crack without regard for any criteria for crack advance. The analysis performed describes these fields under large crack tip geometry changes (large strains)—in contrast to the small strain analysis of Hutchinson Rice & Rosengen—so that the state of stress and strain may be well characterized near the crack tip up until an arbitrary criteria for crack advance can be applied. While this analysis is excellent for understanding the conditions surrounding the crack tip during crack initiation, it is important to remember that—depending on the continuum behavior of a material—*growing* cracks tend to develop an elastic enclave in their wake which reduces the intensity of the singularity as the crack grows, making this analysis uncertain for understanding long crack growth/resistance curve behavior [41]. Regardless, the results of this analysis are captured succinctly in fig 1.20.

While the stress field ahead of the crack is marginally sensitive to the angular deviation from the crack plane (θ)—decreasing at higher angles—the variation in intensity of the strain field caused by the crack tip singularity (in an isotropic material) is noticeably more intense at higher angles from the plane of crack propagation than it is directly ahead of the tip. More importantly though, if the CTOD (δ) is the mode I crack tip opening displacement, the peak stress occurs roughly 2 CTOD ahead of the crack tip, while the
peak strain occurs very near the tip and decreases rapidly. In materials subject to small scale yielding, this asymmetry between the stress and strain fields guarantees that stress induced damage (cracked or debonded particles) will exist broadly ahead of the tip, though there may be an angular dependence on the intensity of the damage. The precise amount of deformation (strain) required to further nucleate, grow, and coalesce this or new damage with the crack tip—based on local material geometry and properties—is largely what controls the intrinsic toughness in many materials, and is the key subject of specific fracture models.

Figure 1.20: Distribution of stress and strain ahead of a crack tip [41].

*Rice & Johnson Model*

The original model for quantifying ductile fracture was that of Rice & Johnson [42] who recognized that the “process zone” of a material subject to both high triaxial stress and
high strain is confined to a small region roughly 2 CTOD directly ahead of the crack tip. In this model (fig. 1.21), crack advance is assumed to be controlled by the growth and coalescence (by impingement) of a void, nucleated at a second-phase particle, (within the process zone) with the blunting crack tip. Crack advance occurring, in a non-hardening material, when the process zone encompasses the ligament between the void and the crack tip or when the crack-tip opening displacement becomes greater than (approximately) half the average void-initiating particle separation ($\lambda$),

**Figure 1.21:** Crack extension in the Rice & Johnson model by void impingement with the crack-tip.

Considering both small scale yielding and full plastic yielding, Rice & Johnson proposed a criteria for local failure which can be stated in terms of the crack tip opening displacement at crack initiation ($\delta_{ic}$) as

$$\delta_{ic} \propto \lambda$$

(1.3)

Using the proportionality between crack tip opening displacement and the $J$ contour integral ($J$) for a stationary crack—a measure of strain energy density for elastic plastic
fracture mechanics analogous to the linear elastic stress intensity factor $K$—this expression can be restated as

$$J_k \propto \sigma_y \lambda$$  \hspace{1cm} (1.4)

where $\sigma_y$ is the yield strength of the material. As with most ductile fracture models, this model is a local idealization—toughness is single valued (irrespective of crack length) and assumed to be controlled by a short range process that is representative of many events occurring simultaneously along the crack front. The strength of this model resides in its emphasis on the spacing between void-nucleating particles and the incorporation of void coalescence into a mechanistic explanation of crack extension. Unfortunately, this model fails to quantify the relationship between all particles and the subset of void-nucleating particles, incorrectly captures the amount of energy spent up to coalescence, and predicts an inexplicable increase in toughness with alloy yield strength.

**Hahn & Rosenfield Model**

The Hahn & Rosenfield model for ductile fracture [15] is one of the first and most frequently used models to describe the fracture of discontinuously reinforced metallic composites [43]. Similar to the Rice & Johnson model, it is a model based on the failure of the ligament in the strain field between an advancing crack tip and a void-nucleating particle, even using the same criterion for crack advance (eq. 1.3). However, Hahn & Rosenfield proposed that the average interparticle spacing ($\lambda$) of a second phase in a material can be reasonably approximated by a regular distribution consisting of a hexagonal array of particles with volume fraction $V_f$ and diameter $D$, as given by

$$\lambda = D \left[ \frac{\pi}{6V_f} \right]^{-1/3} \frac{2}{\pi}$$  \hspace{1cm} (1.5)
Furthermore, Hahn & Rosenfield assume, using their own observations and those of other authors [15], that all particles in the vicinity of a crack-tip rupture or decohere, which for the purposes of describing crack propagation, makes the volume fraction of void-nucleating particles identical to the volume fraction of all particles \((V_f)\). With these observations it is possible to substitute the expression for interparticle separation (eq. 1.5) into the Rice & Johnson relationship (eq. 1.4), and use the transformation relating \(J\) and stress intensity \((K)\) at initiation to obtain a model for toughness [44],

\[
K_{ic} = \sqrt{E' J_{ic}} \approx \sqrt{E' \sigma_y \lambda} \approx \sqrt{2E' \sigma_y D \left( \frac{1}{V_f} \right)^{1/6}}
\]

where \(E\) is the modulus of the system, and \(E'\) is the stress-state-dependent reduced modulus which for plane-strain is equivalent to \(E/(1-\nu^2)\).

Though this model rationalizes some of the challenges presented in the Rice & Johnson framework with reasonable explanations, and captures an often observed dependence of toughness on \(V_f^{-1/6}\), its principal retractions are similar to that of the Rice & Johnson model. Specifically, this model predicts an incorrect positive correlation between toughness and yield strength and as a consequence, it inaccurately describes the dependence of composite toughness on heat treatment. Furthermore, as observed by Van Stone and Psioda [45], toughness typically does not increase with the size of a void nucleating particle; this is an unfortunate consequence of normalizing interparticle separation to particle diameter in the determination of ligament spacing from volume fraction, itself a result of describing particle arrangement via a hexagonal lattice. Most importantly though, this model ignores the effect of particle distribution by assuming that particle separation is only dependent on volume fraction—an approximation that is reasonable at high volume fractions, but very poor at lower loadings.
**Stress-Modified Critical Strain Models**

In order to overcome some of the inadequacies of the Rice & Johnson-type models, several authors [46] considered that for crack growth, the local equivalent plastic strain ($\varepsilon_p^*$) must exceed a critical value ($\varepsilon_p^*$) that depends strongly on the local stress state, and that it must be evaluated over a microstructurally significant length scale such as the mean void-nucleating particle spacing ($\lambda$). For example, Ritchie considered a near-tip strain distribution—very similar to that proposed by McMeeking—in terms of the distance ($x$) directly (at $0^\circ$ inclination) ahead of the crack, in the plane of the crack:

$$\varepsilon_p \propto \left( \frac{J}{\sigma_y r} \right)^{1/n} \sim B \left( \frac{\delta}{x} \right)$$  \hspace{1cm} (1.7)

where $r$ is the radial distance ahead of the crack, $n$ is the strain hardening exponent from a Holloman-type strain hardening law ($\sigma = \sigma_0 \varepsilon_p^n$, where $\sigma_0$ is a reference stress—a fit parameter), and $B$ is assumed a constant.

When, at a microstructural distance ($\lambda$) this equivalent plastic strain exceeds the critical value ($\varepsilon_p^*$), the crack tip opening displacement at crack initiation can be approximated by

$$\delta_{ic} \sim \varepsilon_p^* \lambda$$  \hspace{1cm} (1.8)

A quantity that can be related to the J-integral at criticality by once again using the proportionality of $J$ and $\delta$ for an elastic-plastic material,

$$J_{ic} \sim \sigma_y \varepsilon_p^* \lambda$$  \hspace{1cm} (1.9)

Then the relationship between $K$ and $J$ at crack initiation can be used to derive an expression for toughness as a function of yield strength, the critical plastic strain, and the
interparticle separation that incorporates a simple dependence on stress state through the reduced modulus \( E' = \frac{E}{(1-\nu^2)} \) referred to as a stress modified critical strain model:

\[
K_{lc} = \sqrt{E'J_{lc}} \approx \sqrt{E'\sigma_y \varepsilon_p^* \lambda}
\] (1.10)

The advantage of this type of model is that it incorporates a physically interpretable criterion for damage into a fracture model. This criteria could be measured from, say observed damage in a (notched) tensile test subject to an appropriate stress state [47], or even predicted by mechanical models of void growth and the strain to failure at coalescence [48]. Unfortunately, this type of model does not effectively decouple the critical strain for crack extension from the influence of second phase distribution. Furthermore, this model also unreasonably predicts increasing toughness with increasing yield strength.

*Garrett & Knott Model*

In an earlier study based on a simplified plastic zone [49], Hahn & Rosenfield observed that the extent of the plastic zone \( l \) for a number of ductile alloys subject to plane-strain seemed sensitive to alloy strain hardening exponent \( n \)—again, as determined from a Holloman power hardening law—observing a dependence of the form

\[
l(n) = Cn^2
\] (1.11)

where \( C \) is a constant. Using this observation, Garrett & Knott proposed a critical strain model for crack extension that contended that fracture was governed by the maximum strain ahead of the crack tip \( \varepsilon_{MAX} \) applied over the length of the plastic zone [50], resulting in an expression for a critical crack mouth opening displacement as a function of a critical strain \( \varepsilon_{MAX} = \varepsilon_c^* \),

\[
\delta_{lc} = 2\varepsilon_c^* l^*(n)
\] (1.12)
Again, using the proportionality of $J$ and $\delta$ for a stationary crack as well as the transformation relating $J$ and $K$ at criticality, a stress-modified critical strain model for fracture toughness under plane strain conditions can be obtained:

\[
K_{ic} = n_s \sqrt{\frac{2EC\sigma_y \varepsilon_c^*}{(1-\nu^2)}}
\]

This relationship captures the correct inverse dependence of toughness on yield strength since $n$ decreases rapidly with increasing yield strength, However, it relies on the critical strain for ligament failure to capture entirely the dependence on microstructural features, offering no explanation of its dependence on interparticle separation. Furthermore, the dependence of $K_{ic}$ on $n$ is based on a representation of the plastic zone which is not entirely developed like that of McMeeking or even the HRR (small strain) analysis of Shih [51] who showed that the strain distribution ahead of a stationary crack is comparatively insensitive to constitutive mechanical properties—particularly strain hardening exponent. An important concept developed by this model though is that ductile crack propagation need not necessarily occur, as assumed by other stress modified critical strain-type models, “straight on” (at an inclination of $0^\circ$ to the crack plane). Even though crack propagation along a $0^\circ$ inclination should be encouraged by the fact that the critical strain should be a minimum along this direction—since the hydrostatic stress, and consequently, the stress triaxiality are maximized on the $0^\circ$ plane (as can be seen in the McMeeking analysis)—it is quite possible that non-minimal critical strains can be achieved by the steeper strain gradients available at higher angles prior to the minimum critical strain being satisfied at a $0^\circ$ inclination. Though ultimately deficient in explanation, this model suggests that there may be an intrinsic rationale based on local out-of-plane crack deflection that explains the relationship between toughness, critical strain, and particle distribution.
Extrinsic Toughening by Crack Deflection

In heterogeneous materials it is possible for the microstructure to encourage substantial deflection of a crack out of the plane of fracture [52, 53]. In this case the material appears (through measurement) tougher than it may actually be intrinsically. This concept was originally explored, and quantified for linear-elastic materials by the work of Faber & Evans [54], who developed a simple explanation for the account of this *extrinsic* toughening by crack deflection. To understand their model, consider the crack and an interacting particle depicted in fig. 1.22. Here, the in-plane crack approaches the particle and is deflected by an angle $\theta$.

![Figure 1.22: Schematic representing the geometry of crack deflection model [54].](image)

The resulting stress intensity field is given by the local mode I and mode II stress intensity factors, $k_1$ and $k_2$, that, as a function of the *applied* stress intensity factors ($K_I$, $K_{II}$) are defined by the expressions

$$k_1 = K_I \cos^3(\theta/2) - 3K_{II} \sin(\theta/2)\cos^2(\theta/2) \quad \text{(1.14)}$$

$$k_2 = K_I \sin(\theta/2)\cos^2(\theta/2) + K_{II} \cos(\theta/2)(1 - 3\sin^2(\theta/2)) \quad \text{(1.15)}$$
From these relationships, it is then possible to compute the effective stress intensity ($K_{eff}$) that the material experiences from the relationship

$$K_{eff} = \sqrt{k_1^2 + k_2^2}$$ (1.16)

In the absence of mode II loading, this relationship reduces to

$$K_{eff} = K_I \cos^2(\theta / 2)$$ (1.17)

which indicates that the measured stress intensity ($K_I$) can be improved in a linear-elastic material by up to a factor of 2 through an increasing angle of microstructurally induced local crack deflection, though often toughness improvements are more modest (~x1.2-x1.4). Moreover, in their model, and has been demonstrated experimentally by other authors [55], Faber & Evans showed that an increasing volume fraction or clustering of a second phase, for many possible particle arrangements, increases the amount of extrinsic toughening by crack deflection since an increase in particle proximity necessarily increases the angle of deflection, though an even larger contribution is theoretically possible because of crack twist (deflection out of the plane of fracture, but orthogonal to direction of crack propagation).

*Extrinsic Toughening by Bridging and Shear-Ligament Toughening*

While the Faber & Evans model accurately captures the linear-elastic effect of out-of-plane crack deflection, in heterogeneous materials that demonstrate ductile fracture there can also be an extrinsic plastic contribution to toughness resulting from crack deflection. In the wake (behind the tip) of the crack—at larger length scales than where ductile fracture models are applicable—unbroken ligaments bridging the crack-wake shield the crack-tip from the full applied stress intensity. While generally this effect is termed *bridging*, when there is significant plasticity in the unbroken ligaments which themselves must be ruptured (ex: the Majumdar & Pandey “bean” DRA), this effect is termed *shear-*
**ligament toughening**, since the unbroken ligaments must be literally sheared apart to grow the crack.

Generally, bridging can be quantified by the degree of resistance curve behavior contributed to fracture in excess of the intrinsic toughness of the material by a relationship of the form

\[ K_{\text{Effective}} = K_{\text{Intrinsic}} + K_{\text{Bridging}} \quad (1.18) \]

In the case of linear-elastic bridging ligaments, the bridging contribution \( K_{\text{Bridging}} \) can be experimentally estimated from a comparison of the crack-tip mouth opening displacement \( \delta \) behind the crack-tip with the parabolic surface displacement expected from a linear-elastic un-bridged crack, which explicitly determines the load-carrying capacity of the unbroken ligaments—the bridging traction that shields the tip—and the corresponding contribution to toughness \( K_{\text{Bridging}} \) [34].

In the specific case of shear-ligament toughening, Chan has developed a detailed empirical model in which the effective toughness is given by the expression

\[ K_{\text{Effective}} = \left( K_{\text{Intrinsic}}^2 + \frac{v_l l E \gamma_l^*}{1-v^2} \left[ 1 + \left( \frac{L}{l} \right) \tan \phi \right] \right)^2 \quad (1.19) \]

where \( v_l \) and \( l \) are the ligament volume fraction and width respectively, \( v \) is the poisson ratio, \( E \) is the elastic modulus, \( \tau_l \) and \( \gamma_l^* \) are the material fracture stress and strain in shear, \( L \) is the projected crack length, and \( \phi \) is the angle of crack deflection causing ligament inclination [38]. This model has been effectively applied to a number of heterogeneous intermetallic materials, but in a manner similar to models for describing bridging by linear elastic ligaments, requires a measured density of ligaments participating in the bridging process rather than providing a microstructure-based origin for this quantity.
To date, the most accurate model for describing ductile fracture in DRA is that of Majumdar & Pandey. Their model is a stress-modified critical strain approach that incorporates a dependence of the critical strain on particle arrangement in a manner reminiscent of the approach of Hahn & Rosenfield. It also explains the dependence of toughness on yield strength and strain hardening in a manner consistent with the crack-tip analysis of McMeeking while simultaneously incorporating the effect of crack deflection on the *intrinsic* toughness of discontinuously reinforced MMCs [56].

Majumdar and Pandey start from the same common origin as all other models for ductile fracture, the proportionality between crack opening displacement, $\delta$ and $J$

$$\delta \propto J$$  \hspace{1cm} (1.20)

and use the small strain (HRR) finite element results of Shih to capture the dependence of this relationship on strain hardening exponent and yield strength via

$$\delta = \frac{d(n)J}{\sigma_y}$$  \hspace{1cm} (1.21)

where for a DRA-like material (with $\sigma_y/E = 0.004$, and $n \leq 0.1$), the dependence on strain hardening exponent is given by

$$d(n) = 0.78 - 2.73n + 3.065n^2$$  \hspace{1cm} (1.22)

though other expressions for this dependence are generally available [51]. Recognizing the normal relationship between $J_{lc}$ and $K_{lc}$ under plane strain, $K_{lc}$ can be expressed as

$$K_{lc} = \sqrt{\frac{EJ_{lc}}{1 - \nu^2}} = \sqrt{\frac{E\sigma_y\delta}{1 - \nu^2 d(n)}} = \sqrt{\frac{E\sigma_y\delta}{d(n)(1 - \nu^2)}}$$  \hspace{1cm} (1.23)
Similar to other critical strain approaches, the criteria for crack advance in this model is based on an extension of the crack from one broken particle to another, though in a unique manner when compared to other ductile fracture models. From the stress analysis of McMeeking [41], Majumdar & Pandey observe that local strains, for any angle $\theta$ relative to the crack plane, do not display the strong dependence on $n$ argued by Garret & Knott and Hahn & Rosenfield. Thus, there is no preference based on continuum properties of mechanical behavior ($n, E, \sigma_y$) for a critical length scale for describing the fracture process. Rather, they observe that even in low-strength heat treatments of the DRA system they studied, there is a strong propensity for intense slip localization between broken particles (similar to that observed in high strength steels and aluminum alloys), and that the position of damage ahead of a crack tip forces intense slip localization in a band between the crack-tip and near-tip damage at non-zero angles relative to the crack plane [56]. To accommodate for this, they formulate an expression for a critical strain criteria for interparticle ligament failure based on an arbitrary inclination rather than at $0^\circ$ relative to the crack plane, a stark difference from most other ductile fracture models (fig. 1.23).

**Figure 1.23:** Cellular model for crack extension in the Majumdar & Pandey model.
The determination of a critical strain at an arbitrary inclination requires a novel analytical approach. Majumdar & Pandey developed a method for determining this critical strain from the Cockcroft-Latham damage parameter, \( D \):

\[
D = \int \sigma_{\text{max}} d \varepsilon_{\text{plastic}}
\]

(1.24)

when it exceeds some critical value \( (D \geq D_{\text{critical}}) \). In this expression, \( \sigma_{\text{max}} \) is the maximum principal stress ahead of the tip, and \( \varepsilon_{\text{plastic}} \) is the associated equivalent plastic strain. This parameter has been successfully used to characterize failure in a number of materials during deformation processing [57], seemingly accurate in characterizing damage regardless of deformation path.

Noting that for the stress analysis of McMeeking that \( \sigma_{\theta\theta} \) is a good estimate of \( \sigma_{\text{max}} \), a good approximation of eq. 1.24 translated to the tip of the crack is

\[
D \cong \sigma_{\theta\theta} \varepsilon^*_{\text{p}}
\]

(1.25)

where \( \varepsilon^*_{\text{p}} \) is the critical plastic strain at which ligament instability occurs. Now, for a hardening composite material observing Ramberg-Osgood flow behavior, it can be shown that the critical value of the Cockcroft-Latham damage parameter as limited by the ductility in uniaxial tension for the matrix material, \( \varepsilon^f_m \) is equivalent to

\[
D_{\text{critical}} = \left( \frac{N}{N + 1} \right) \varepsilon^f_m \sigma_{\text{UTS}}
\]

(1.26)

where \( N \) is the inverse of the strain-hardening exponent \( (n) \). As a result, the relationship for crack extension is satisfied when
\[ D \geq D_{critical} \]

\[ \sigma_{\theta \theta} \epsilon^*_p \geq \left( \frac{N}{N + 1} \right) \epsilon'_m \sigma_{UTS} \] (1.27)

or, more importantly, solving for the critical strain for ligament collapse,

\[ \epsilon^*_p = \frac{1}{\sigma_{\theta \theta}} \left( \frac{N}{N + 1} \right) \epsilon'_m \sigma_{UTS} \] (1.28)

which, after several rough approximations and the observation that \( \sigma_{\theta \theta} \) is relatively constant (compared to the variation in strain)/insensitive to direction relative to the crack plane, Majumdar & Pandey reduce this to

\[ \epsilon^*_p = \left( \frac{\epsilon'_m}{0.002} \right)^{0.02} \left( \frac{\epsilon'_m}{(2.2 + 7n)(n + 1)} \right)^{a} \] (1.29)

This critical strain for ligament failure must be mechanically related to the mouth opening displacement, through some relationship of the form

\[ \delta = \beta \lambda \] (1.30)

where \( \lambda \) is the distance to the nearest cracked particle, and from a fit to the McMeeking data for a 45\(^{\circ} \) inclination between cracked particles, \( \beta \) is empirically determined to be

\[ \beta = \frac{0.3 + 100 \epsilon^*_p}{9.4 + 35 \epsilon^*_p} \] (1.31)

Majumdar and Pandey argue, based on experimental observations of intense shear banding in their composite systems, that 45\(^{\circ} \) relative to the crack plane is an appropriate
choice of misorientation for the determination of $\beta$; the inverse of this value representing the maximum normalized distance ($\delta/\lambda$) from the crack tip at which failure will occur—clearly though a broken particle must also be within this distance to facilitate fracture.

Similar to the argument made by Hahn and Rosenfield, Majumdar & Pandey assume that all particles in the vicinity of the crack-tip are fractured or decohere at a *negligible strain*, making the set of void nucleating particles indistinguishable—as far as crack propagation is concerned—from the set of all particles; an argument they support with a Weibull-type analysis of particle damage near the fracture surface in tensile specimens. This being the case, Majumdar & Pandey argue that the distance between nearest particles can be defined by

$$\frac{\lambda}{D} = 0.74 \left( \frac{\pi}{6} \right)^{1/3} V_p^{-1/3}$$

(1.32)

where a cubic array ($\lambda/D = [\pi (6V_p)]^{1/3}$) has been initially assumed, but perturbed by a factor of 0.74 to account for the nearest neighbor spacing [58], rather than the particle spacing originally suggested by Hahn & Rosenfield. This results in a general expression for $\delta$

$$\delta = 0.74\beta D \left( \frac{\pi}{6} \right)^{1/3} V_p^{-1/3}$$

(1.33)

which can be substituted into eq. 1.21 to obtain

$$K_{lc} = 0.77 \sqrt{\frac{E\sigma_o \beta D}{d(n)(1-\nu^2)}} V_p^{-1/6}$$

(1.34)

In theory, the Majumdar & Pandey model separates fully the critical length scale for fracture and the critical strain for ligament failure, and seems to effectively predict an
upper bound on the general trend of fracture resistance with respect to particle distribution (through volume fraction) while incorporating other constitutive parameters ($\sigma$, and $n$) and the degree of crack deflection into an explanation of intrinsic toughening during ductile fracture.

Clearly though, this model ignores a significant amount of the complexity of the interaction of damage and the crack tip with regard to stress state, and material softening in the ligament due to secondary void growth, such as described by the Gurson, Tvergaard, Needleman (GTN) model [59]; all of these factors are bundled into a critical strain for ligament collapse along a particular direction, as well as its fundamental assumption that this process does not change with ligament size (and by corollary, the particle distribution). Moreover, as particle separation decreases below a certain separation, it is entirely possible that crack growth may not necessarily occur by linking of damage along a 45° direction, invalidating the single approximation used for $\beta$. Specifically, as has been demonstrated by the crack deflection work of Faber & Evans, it is geometrically necessary for non-uniform distributions to promote higher angles of crack deflection when compared with uniform distributions [54]. Furthermore, it is easy to imagine clustered particle distributions for which eq. 1.32 does not adequately describe the nearest neighbor separation.

In regard to these issues, the results of Majumdar & Pandey’s experimental work and modeling [60,56] clearly indicate a preference for non-planar ($\theta\sim 45^\circ$) propagation of a ductile crack, though the effect of such angular deviation on the length-scale of fracture is never explicitly considered in their model. This fact is perhaps most explicitly captured by their own results in fig. 1.24, where their model (eq. 1.34) is used in conjunction with microstructural data to predict the toughness of various heat treatments of a 7093/SiC/15_p DRA. Although the model for 45° propagation captures the trend of decreasing toughness with increasing yield strength quite nicely in comparison to the prototypical 0° propagation assumed in other ductile fracture models, in absolute terms, the model under-predicts toughness by ~20%. If one were to consider that particles inclined 45° relative to each other in a cubic array are separated by a distance $\sqrt{2}$ times greater than in the 0°-
plane, this increase in particle spacing would translate into a pre-factor of 0.92 in eq. 1.34, rather than 0.77, an increase that roughly accounts for gap between the experimental data and the models predictions.

![Graph](image)

**Figure 1.24:** Comparison of experimental data and toughness predictions from the Majumdar & Pandey model for ductile fracture in DRA [60].

1.4.) QUANTITATIVE FRACTOGRAPHY

Since very gross (qualitative) changes in fractography are not typically associated with variations in reinforcement or second phase distribution, it is necessary to use quantitative fractography techniques—various measures of fracture surface roughness—to characterize differences in fracture behavior. Roughness as a concept is generally used through a variety of models to mechanically quantify either intrinsic toughening via fracture surface formation energy or extrinsic toughening via crack tip deflection. In the prior case, roughness is an indicator that supports further analysis of the local surface topography to determine criteria for damage, while typically at larger length scales a variation in roughness supports a reevaluation of the mechanics of the crack itself. Fundamentally at issue is the fact that most basic measures of roughness are intrinsically
dependent on length scale and may not capture information on the fracture process of interest. This has led to the development of techniques for quantifying surfaces on multiple length scales—fractal techniques.

Unfortunately, there is no universal measure of roughness which can be used to quantify fracture behavior without regard for material, mechanism, or length scale. Rather, measures of roughness should be complimentary features of fracture which must be interpreted only when the fundamental mechanism of fracture is understood, as well as the mechanics of damage advance. While there are many means of quantifying surface roughness, we will introduce the most relevant to the purpose of our further discussion: average roughness ($R_A$), lineal roughness ($R_L$), and the so-called “surface” roughness ($R_s$), and for modeling purposes later, the micro-roughness ($W$) of Thompson & Ashby.

**Average Roughness**

Average roughness ($R_A$) is the most common measure of surface roughness; for a 2-dimensional profile it is a measure of the area between a profile and its mean height, normalized by the projected length of the profile (fig. 1.25),

$$R_A = \frac{1}{L} \int_0^L |R(x)| \, dx$$

(1.35)

where $R(x)$ is the profile amplitude, and $L$ is its projected length. A simple analog also exists for 3-dimensional surfaces which integrates the height of a surface over a selected area. In general, $R_A$ endures as the most common measure of roughness because of the ease with which it is measured; typically by dragging a mechanical stylus across the surface of interest, while coupled analog electronics rectified, integrated, and averaged that signal to compute $R_A$ [61].
Lineal Roughness

Lineal roughness \((R_L)\) otherwise known as the profile length ratio, is a measure of the total length of a profile \((L)\) normalized by its projected length \((L')\), as can be seen in fig. 1.25,

\[
R_L = \frac{L}{L'} \quad (1.36)
\]

Since lineal roughness (profile length) is very sensitive to resolution of measurement, it is difficult to quantify without an analysis over multiple length scales. An awareness of the significance of this behavior has over time come into vogue and is the very heart of fractal quantification of rough surfaces.

Surface Roughness

Surface roughness \((R_s)\) represents the actual area of a surface, normalized by its projected area; though difficult to measure directly, it can be approximated from the lineal roughness by the relationship

\[
R_s = \frac{4}{\pi} (R_L - 1) + 1 \quad (1.37)
\]

and has been successfully related to microvoid diameter and intergranular facet size in high strength steels and aluminum alloys [62]; though not directly—to the author’s knowledge—to strain criteria for damage. Since this measure is an analog of the lineal roughness, there is a similar built in sensitivity of this measure to length scale.
Several authors have recognized that during ductile fracture, the ratio of dimple height to diameter \( W = \frac{h}{D} \) can be used as a measure of the local critical strain for void coalescence \([63,64]\), as represented by the relationship

\[
\varepsilon^* = \ln(W) = \ln(h / D)
\]  

(1.38)

Though this relationship can be misleading since the onset of void coalescence typically occurs at much smaller strains than those described here—therefore over predicting critical plastic strain—though it is useful in the capacity of placing an upper bound on the
amount of influence that critical strain can have on measures of fracture surface roughness.

**Fractal Analysis**

Fractal analysis is a means of determining geometric self-similarity (or deviations from it) over multiple length scales. A common example of fractal behavior that the reader may identify with is the Koch curve in which a line segment is kinked to form a central equilateral triangle (fig. 1.26).

![Figure 1.26: The fractal Koch Curve.](image)

By applying the same transformation iteratively (kinking each constitutive line-segment in an identical manner) ad infinitum (fig. 1.28), a pattern develops that is self-similar—regardless of magnification (length scale)—whose length increases rapidly as the yardstick used to measure it (length scale) is decreased.

![Figure 1.27: Iterations of the Koch kernel that lead to the fractal Koch Curve.](image)

More quantitatively, a curve is said to be fractal if its length ($L$) obeys a “scaling law” of the form
\[ L = L' \eta^{(1-D)} \]  

where \( \eta \) is the length scale (yardstick) at which the profile length is measured, \( L' \) is again the projected length of the profile, and \( D \) is a constant quantity termed the fractal dimension, a parameter that is a length-scale independent measure of roughness representing the fractional dimension of the curve. For example, any plane curve should have a value of \( D \) between 1 and 2, with 1 representing a perfectly straight line, and 2 representing a tortuous curve that completely fills the area of the plot. Perhaps a more intuitive explanation of the fractal dimension of a profile (or surface) is to simply think of it as the exponential rate at which profile length (surface area) increases with increasing resolution in length scale. For a plane curve, the fractal dimension is most directly measured as the slope of a log-log plot of the length of the curve vs. length scale. Such a plot for the Koch curve is depicted in fig. 1.28.

![Graph showing the relationship between Log(Profile Length) and Log(Length Scale / Yardstick Length)](Attach:Graph.png)

**Figure 1.28:** Fractal Dimension of the Koch Curve.
There are a number of different ways to compute the length of a curve, with their own associated efficiencies and errors. The method used by Mandelbrot in his seminal work on quantifying the fractal behavior of fracture surfaces is to measure profile length as a function of length scale/yardstick via the Richardson structured walk [65]. This method takes a yardstick of a certain length ($\eta$) and then starting at the origin, successively finds the nearest intercept on the examined curve (fig. 1.29). When the end of the curve is reached, the number of line segments necessary to cover the curve is computed and multiplied by the segment length to determine the length of the profile. This process is repeated for each length scale of interest to compute a plot similar to (fig. 1.30) from which the fractal dimension can be determined.

Figure 1.29: The Richardson structured walk.

*Fractal Quantification of Fracture Surfaces*

At the core of fractal analysis of fracture surfaces is the concept that the roughness of a fracture surface that we perceive at a visual length scale is a function of some self-similar process whose kernel is at a much smaller length scale. Several studies, including the
original work by Mandelbrot have explored the fractal behavior of fracture surfaces
[66,67,68], most of which repeatedly confirm that fracture surfaces for a number of
different materials are indeed fractal, and then directly try to relate fractal dimension to
toughness in a first order fashion. Unfortunately, few studies attempt to connect these
measurements to some process occurring on a small length scale that may be controlling
fracture, such as the failure of an interparticle ligament by void coalescence in a
discontinuously reinforced metallic composite. Rather, most works take a different
approach, and try to quantify the effect of “fractal” crack tip deflection on the mechanics
of a linear elastic crack [69], in analogy to the model for crack deflection proposed by
Faber & Evans [54], ignoring entirely any effect of changes in intrinsic toughening that
could be manifested by changes in fractal behavior of the fracture surface.

Several very good works quantifying the fractal behavior of fracture surfaces do exist
though, including that of Dauskardt, Haubensak & Ritchie [70]. In this work, synthetic
fracture surface profiles were constructed by randomly sampling segment lengths from
Gaussian distributions (with mean length, $M$, and standard deviation $s$) and inclining each
successively sampled segment relative to the last with a constant angular deflection ($\theta$)
that alternated in direction (up or down, $\pm \theta$). Synthetic fracture surface profiles
constructed in such a manner could then be used to probe the effect of these parameters
($M$, $s$, $\theta$) on profile fractal behavior, specifically $D$. To do this, lineal roughness ($R_L$) of a
profile was measured as a function of the length scale $\eta$ via the Richardson structured
walk and fit to the relationship,

$$R_L = \frac{L}{L'} = \eta^{(1-D)}$$  \hspace{1cm} (1.40)

From the analysis of Richardson plots of these synthetic fracture surfaces (figs. 1.30-32,
note the graphical depiction of the segment length distribution depicted below the
Richardson plots), three key observations were made [70]:

1. Mean segment length has a strong positive effect on surface roughness (as $M$ increases, so does $R_L$), but has no effect on fractal dimension (fig. 1.30)—$D$ is insensitive to any variation in $M$ (while $\theta$ is held constant).

2. Segment length standard deviation ($s$) has a weak negative (inverse) effect on fractal dimension (fig. 1.31)—$D$ increases as $s$ decreases, weakly.

3. Mean angular deflection has a strong positive effect on fractal dimension and roughness (fig. 1.32)—$R_L$ and $D$ increase with increasing $\theta$.

Figure 1.30: Effect of mean segment length on fractal dimension [70].
Figure 1.31: Effect of segment length standard deviation on fractal dimension [70].

Figure 1.32: Effect of mean angular segment deflection on fractal dimension [70].
The principal logical consequence of these observations is that the fractal dimension of a fracture surface should, in many cases, be useful as a measure of the angular degree of crack deflection, while changes in the lineal roughness should—at a particular length scale, $\eta$—should correspond to variations in the feature size of the fracture process.

Moreover, on superimposing a micro-roughness—distributed on a relatively small length scale—onto a macro-roughness (fig. 1.33), in effect simulating the cooperation of two distinct features of a fracture process that operate on separate length scales, a disjunction in Richardson plots was observed. At low length scales the fractal dimension corresponds well with the parameters of the micro-roughness, while at larger length scales the fractal dimension resembles parameters of the macro distribution. This indicates that it should be possible to separate the effects of multiple fracture mechanisms if they act on multiple length scales.

**Figure 1.33:** Effect of super-imposed micro/macro-roughness on the fractal behavior of profiles [70].
Using the same methodology that was applied to synthetic fracture surface profiles, the authors subsequently studied fracture surfaces profiles from three different steels displaying three different modes of fracture (fig. 1.34): a mild steel (AISI 1008) fractured at -196 °C demonstrating transgranular cleavage (1), as well as a 31 wt. % Mn-steel failing by intergranular fracture at -196 °C (2) and by microvoid nucleation, growth and coalescence at room temperature (3). The results of the analysis of fracture surface profiles from these materials were compared to the size of microstructural features relevant to each fracture process.

During transgranular cleavage (fig. 1.34a) fracture surface profiles in the mild steel demonstrated a transition from a fractal behavior with a low dimension to a higher dimension behavior when length scales transitioned from those corresponding to river markings (2-15 μm) to the larger length scales representative of the material grain size (15-75 μm). Accompanying intergranular fracture in the Mn-steel was a similar transition in fractal behavior between three different length scale regimes corresponding to grain boundary slip steps (6-15 μm), grain boundary particle separation (0.8-4 μm), and grain boundary size (20-150 μm) presumably caused by the contributing micro-roughness of each of these features. Finally, associated with microvoid coalescence in the Mn-steel was a Richardson plot primarily dominated by fractal behavior over a regime of length scales corresponding to the ductile dimple size/inclusion spacing (6-50 μm) with a lower dimension behavior below this length scale correlated with slip steps and ripples in microvoid walls (0.8-4 μm).
Figure 1.34: (a)-(b) Results of fractal analysis on steels failing by different mechanisms, indicating the ability of fractal analysis to identify feature length scales [70].
Figure 1.34: (c)-(d) Results of fractal analysis on steels failing by different mechanisms, indicating the ability of fractal analysis to identify fracture feature length scales [70].
From these observations on real fracture surfaces, it was concluded that:

1. Generally there is a good correlation between the length scales of fractal behavior in Richardson plots, and length scales of features critical to the relevant fracture process.

2. A universal correlation of fracture surface fractal dimension and fracture toughness is unlikely since there is a clear dependence of fractal behavior on fracture mode.

3. Fractal analysis would be most useful when characterizing differences in fracture surface roughness that were a result of a specific kind of failure mode, such as fracture during microvoid coalescence, second-phase crack path deviation in ceramics, and during near threshold fatigue in metallic systems.

In a very comprehensive article [71], Milman, Stelmashenko, and Blumenfeld reviewed this work and that of many other authors, and observed that the fracture of many brittle systems (mainly ceramics and ceramic composites) demonstrates a strong positive correlation of fracture surface fractal dimension and fracture toughness. Given the observations of the strong positive relationship between fractal dimension and angular deflection observed by Dauskardt, Haubensak & Ritchie, as well as the strong influence that crack deflection can have on toughening brittle materials, this correlation is very reasonable.

Conclusions regarding the application of fractal techniques to ductile fracture surfaces are more uncertain, but generally coincide with the observations of Dauskardt et. al. The reviewers cited a number of studies that showed a positive correlation of fractal dimension and toughness, some an inverse relationship, or yet still others, no correlation whatsoever when comparing fracture surface fractal dimension and fracture toughness [71]. Explanations of the observations were varied, but mainly stemmed from improper technique used in quantifying fractal dimension, and the dissimilar modes of fracture in
the materials compared in each study. In general, it was concluded that results from any one study were not typically comparable to that of another, and consequently, any broad trend in fracture toughness and fractal behavior in ductile fracture remained an open problem, but was generally unlikely. The authors explicitly confirmed—by examining those studies which compared multiple fracture modes—that there existed a substantial dependence of fractal measurements on fracture mode, and in some cases of mixed fracture mode behavior, a noticeable “cross-over” behavior occurred in a Richardson-type plot at a particular length scale, indicating the dominance of one mechanism over another at particular length scales, similar to the super-imposed roughness results observed by Dauskardt et. al.

The consequences of these observations is that, from a fractal analysis of fracture surface profiles, it should be possible to identify—through the fractal dimension ($D$)—a relative propensity for crack deflection ($\theta$) for a given process operating over a given range of length scales ($\eta$), while from general measures of roughness ($R_L$), it should be possible to identify variations in fracture process length scale. Furthermore, if there is a variation in fractal behavior over several length scales, it should be possible to identify features contributing to the fracture process and their variation.

1.5.) RESEARCH OBJECTIVES

Ultimately, all of the above crack growth models describe only an approximate influence of reinforcement distribution on fracture toughness. Specifically, measures of mean particle separation based on volume fraction are only a good “upper bound” on particle separation, and consequently, fracture behavior. Since fracture is a “weakest link” process, it seems more pertinent that these models should also examine the worst case scenario—what is the shortest path for damage to link as a function of local variations in interparticle separation—and particularly how this microstructure distribution affects the partitioning of work of fracture between length scale, critical strain, and crack deflection. Considering this perspective would then enable the definition of a lower bound on
fracture behavior as a function of reinforcement distribution—arguably a far more useful, and potentially accurate measure of a particular microstructural configuration.

The principal argument of this thesis is that the critical length scale of fracture as well as the strain to failure of an interparticle ligament may be sensitive to the distribution of the second phase. In order to ascertain this, it should be possible, through processing, to create several material conditions that contain a variation in reinforcement distribution that reflects an improvement in the purported lower bound limiting the fracture process. Subsequently, quantitative fractography may be used to elucidate the influence of this change in reinforcement distribution on the length scale, degree of crack deflection, and the critical strain associated with the fracture process. This analysis will culminate with a generalization of the Majumdar & Pandey model—viewed as largely correct—to account for variations in reinforcement homogeneity/particle distribution.

1.6.) REFERENCES


II

EXPERIMENTAL PROCEDURE & METHODS

2.1) MATERIAL & MATERIAL PROCESSING

In order to study the effects of reinforcement homogeneity on the mechanical behavior of DRA, it was necessary to process the material in a manner that controlled this quantity while minimizing the influence of all other variables. Ultimately constrained by the facilities available, powder extrusion (P/E) was the primary processing route considered to accomplish this objective. During P/E, pre-alloyed matrix powder and reinforcement are blended, welded into a vacuum tight container (canned), the atmosphere in the can is volatilized (degassed), and the can is subsequently rammed through a die at an elevated temperature (fig. 2.1). In most conventional alloys systems, this process leads to full densification and a near-net-shape for prismatic/beam products [1].

![Figure 2.1: Depiction of the powder extrusion process, after [1].](image)

At the outset, it seemed the best way to control homogeneity and clustering in DRA was with a so-called particle-size-ratio (PSR) technique [2]. In material made in this manner,
varying particle sizes of matrix powder and reinforcement are blended to create interstitial sites in the matrix powder where reinforcement can agglomerate (cluster); the larger the ratio of matrix to reinforcement particle diameters, the greater the degree of clustering (fig. 2.2). With a large enough PSR, these particle clusters can even be retained after extrusion.

![Diagram showing reinforcement clustering in DRA](image)

**Figure 2.2**: Effect of matrix/reinforcement particle size ratio on reinforcement clustering in DRA.

An unfortunate complication of reinforcement homogenization via the PSR route—of which the author is unaware of any open addressing in the literature—is that clustering reinforcement in this manner frustrates densification by preventing penetration of particle
clusters by any matrix material, leaving a significant porosity that scales positively with many metrics of clustering, even after extensive extrusion. The effect of porosity is known to significantly influence the fracture of P/M systems [1] and thus, changes in the porosity level accompanying material processing would make it difficult to isolate the influence of reinforcement distribution. Initial work detailing this result is described in Appendix A. The need for another processing strategy to control reinforcement distribution resulted in consolidating material via vacuum hot press followed by deformation processing, which broke down the clusters of reinforcement initially present. Specifically, the shear induced by extrusion to varying reduction ratios homogenizes the reinforcement distribution [3].

Material

This study analyzes a 6092 aluminum alloy reinforced with 25 vol. % SiC particles initially consolidated by DWA composites (Chatsworth, CA)\(^1\). The reinforcement particles are nearly equiaxed with a maximum aspect ratio (over all conditions) of 2.4. The median equivalent circular diameter of the reinforcement is approximately 2.2 \(\mu\)m. The matrix alloy is an Al-Mg-Si system very similar to the more common 6061 alloy, but it is tailored for P/M applications. For comparison, the composition of these two alloys is delineated in table 2.1. In P/M aluminum alloys, refined oxides are often added or simply present as a result of processing. While these oxides improve creep strength and wear resistance, they also pin grain boundaries during heat treatment/processing, making the grain refiners present in 6061 unnecessary [4]; the ceramic reinforcement in the composite considered plays a similar role. The additional Cu in this alloy over 6061 is primarily added as a sintering aid [5].

\(^1\) As a disclaimer, it is important to note that while the material was obtained from DWA Composites Inc., any results described herein should not be considered representative of their commercial products.
Table 2.1: Composition of Aluminum 6061 and 6092, by wt. %.

<table>
<thead>
<tr>
<th>Species</th>
<th>6061</th>
<th>6092</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>0.40 - 0.8</td>
<td>0.40 - 0.8</td>
</tr>
<tr>
<td>Fe</td>
<td>0.7</td>
<td>0.30</td>
</tr>
<tr>
<td>Cu</td>
<td>0.15-0.4</td>
<td>0.7 - 1.0</td>
</tr>
<tr>
<td>Mn</td>
<td>0.15</td>
<td>0.15</td>
</tr>
<tr>
<td>Mg</td>
<td>0.8 - 1.2</td>
<td>0.8 - 1.2</td>
</tr>
<tr>
<td>Cr</td>
<td>0.04-.35</td>
<td>0.15</td>
</tr>
<tr>
<td>Zn</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>O</td>
<td>---</td>
<td>0.05 - 0.50</td>
</tr>
<tr>
<td>Ti</td>
<td>0.15</td>
<td>0.15</td>
</tr>
<tr>
<td>Others</td>
<td>0.05 Each</td>
<td>0.05 Each</td>
</tr>
<tr>
<td>Al</td>
<td>Balance</td>
<td>Balance</td>
</tr>
</tbody>
</table>

Deformation Processing

The initial material supplied by DWA was in the form of three 50.8 cm (20”) Ø billets, the result of uniaxial vacuum hot-pressing of blended reinforcement and pre-alloyed matrix powders. One of these initial billets was retained for study as its own condition, while from this common origin, deformation processing was used to derive two additional material conditions, as depicted in fig. 2.3. The second condition was formed by extruding one of the initial billets at a ratio of 8:1, leaving a 17.8 cm (7”) Ø extrusion. For the third condition, a significant degree of deformation was desired, but due to available extrusion die limitations, had be conducted in two steps. The initial billet was extruded first at a ratio of 7:1, quartered, machined down to a 7.8 cm (3”) Ø cylinder, and re-extruded down to a final diameter of 3.175 cm (1.25”), for a total reduction of 46:1.

All extrusions were drawn/rammed at a rate of 2.54 cm/min, while the die was held at a temperature of 400 ºC. Instrumentation on the extrusion press indicated that this die temperature corresponded to a nominal temperature (effective processing temperature) of 500 ºC in the material during extrusion. In further discussions each of these material conditions will be referred to by their total corresponding reduction ratios: “1:1”, “8:1”, and “46:1”.

Figure 2.3: (a) Processing steps used to create conditions of varying degrees of microstructural homogeneity.
**Heat Treatment**

After deformation processing and prior to mechanical test specimen machining, identical blanks were sectioned from the center of the deformation products and heat treated to peak age (T6). Generally, the schedule for this heat treatment included a solution treatment at 555 °C for 3 hrs, followed by an ice-water quench, a 12 hr aging at 163 °C, and finally an air cool. Rockwell hardness ($R_B$) was the primary technique for verifying the efficacy of the heat treatment. The mean value of 5 separate Rockwell-B hardness measurements made in a crossing pattern on a blank’s free surface was computed for each blank, with an average value of 81±2 indicating an acceptable heat treatment to peak age, as indicated by the hardening curve in fig. 2.4 [6].

![Figure 2.4: Hardening curve for the studied composite system Al-6092/25.0/SiC_p[6].](image)
2.2) MATERIAL CHARACTERIZATION

Microstructure Preparation

Optical metallography from orthogonal sections of scrap blank material was used to characterize the microstructure both parallel to and transverse to the axis of deformation processing. While sectioning (at peak age) with a high speed abrasive saw is uncomplicated, the primary difficulty in preparing metallographic sections from this material stems from its relative hardness and the resulting difficulty of grinding specimens flat. Experience showed that after mounting, the material was often more abrasive than even the most coarse of grit paper, and required first a pass on a surface, or high speed grinder, followed by several (~3) sheets of coarse (80-160 grit), changed every minute, at a ~150 rpm on an 8” wheel. Once sufficiently flat, relatively quick grinds on 240, 320, 600, and 800 grit sand paper would finish the grind. If a vibrational polish was available, an overnight treatment in a 1 μm diamond/Hyprez solution would yield a crisp final polish. Otherwise, the specimen would be polished successively on nylon at < 100 rpm on an 8” wheel with 25, 13, 6, 3, and 1 μm diamond suspensions at increasingly longer intervals.

The metallographic samples were digitally imaged in the as-polished condition using a computer controlled Nikon microscope. The microscope stage was controlled by a Clemmex (Longueuil, QC) image analysis system, which enabled large fields of view (several mm in extent) at high resolution (200x) to be assembled by montage. The resulting fields of view could then be post-processed and probed with a battery of available quantitative characterization scripts provided with the Clemmex system, as well as routines developed by the author in order to determine two primary metrics of quantifying the particle distribution: (1) the nearest neighbor separation distribution—a method for determining local microstructural affects—and (2) the homogenous length scale technique, a method of quantifying the macroscopic (large length scale) concept of homogeneity.
Nearest Neighbor Separation

After image acquisition, images were processed by thresholding the gray level such that particles were black (gray level = 255) and the matrix was white (gray level = 0). A routine in the Clemmex software suite could then identify individual particles and record their centroid position \((x_i, y_i)\), diameter, and aspect ratio. From such a data set, it is possible to consider each particle (the reference particle depicted in fig. 2.5) and identify its nearest neighbor and second nearest neighbor by computing a matrix of separations between \textit{every} particle in the data set, and identifying the minimum and second minimum distances in this matrix for each particle. By calculating these parameters for all particles (as detailed in the script provided in Appendix B), it is then possible to observe how the interparticle separation is distributed in a microstructure. A typical interparticle separation distribution—which gives the probability of finding a particle at a given distance away from the reference particle—is depicted in fig. 2.6. As is common in nearly all fracture models, the nearest neighbor separation is normalized by the diameter of the reference particle \((R/D)\). As will be discussed later, it is anticipated that homogenization of the material will push the median of this distribution to progressively larger length scales while simultaneously curtailting the extent of the high-end tail.

\textbf{Figure 2.5:} Schematic depicting a reference particle, its nearest and second nearest neighbors.
Figure 2.6: A typical nearest neighbor distribution, where the separation \((R)\) is normalized by the particle diameter \((D)\).

Particle Alignment

With extrusion, it is anticipated that reinforcement particles will align themselves along the extrusion axis to minimize their resistance to deformation. As models have indicated, that there is an effect of reinforcement anisotropy/alignment on deformation response, it is important to characterize any such changes that occur with processing. Here, two separate issues exist, alignment of reinforcement by shape and particle centroid along the extrusion axis. Since the particles are not spherical, they may be modeled as prolate spheroids with major and minor axes; with deformation processing, it is anticipated that the particle major axis will align along the extrusion axis. Furthermore, particle centroids should become collinear with increasing extrusion aligning into a banded microstructure of stringers. To characterize such changes, data collected from particle fields was used to compute distributions of particle major-axis inclination and nearest-neighbor centroid-centroid inclination analogous to the nearest neighbor separations described earlier.
Texture Measurement

With deformation processing of a metal there is the potential of developing a preferred crystallographic orientation with an increasing degree of processing. As it develops, this texture will manifest itself as a transition from deformation response that is isotropic, similar to that of a random polycrystalline material, to a state of anisotropic deformation response representing an enhancement of certain single crystal components. Since this texture can contribute significantly to all aspects of flow behavior, it is necessary to measure its development so that it can be separated from any other factor contributing to the deformation response of the studied DRA. In order to determine the degree of texture in the aluminum matrix, X-Ray crystallography was performed on a Rigaku 2500 diffractometer using the Schultz reflection method with Cu-$K\alpha$ radiation, for each deformation condition. After planes were indexed by software on the diffractometer, pole figures for each condition were computed from these data via the preferred orientation package – Los Alamos (popLA) to determine the type and intensity of texture that develops.

2.3) DEFORMATION BEHAVIOR

Prior experience with tension testing of this material indicated a fundamentally brittle behavior (failure prior to yield) at peak age. Consequently, in order to understand any possible influence of reinforcement distribution on flow behavior, and most importantly, crack tip plasticity, compression tests were performed. This deformation mode is less sensitive to any distributed effect of particle damage (cracking or debonding) and consequently will demonstrate a nominally higher (~3%) stiffness and yield strength than in tension [7].

Two (replicate) compression specimens were machined via EDM from the heat treated blanks in the longitudinal (L) and transverse (R) orientations for each deformation product, with the exception of the initial forging (“1:1” extrusion), where only a longitudinal orientation was prepared due to material isotropy. Specimens were right
circular cylinders 0.635 cm in diameter and 0.952 cm tall (aspect ratio 1.5:1). The ends were trued with the assistance of a V-block and a polishing procedure similar to that used for the metallographic sections. Optical inspection of the polished ends indicated that the EDM recast layer contributed to less than 2% of the total cross-sectional area of the specimen, making it unlikely that it significantly influenced the measured flow behavior.

Specimens were tested at room temperature (25 °C) under a nominal strain rate on the order of 10^{-3}/s. Displacement (specimen length contraction) was measured via a video displacement system (Correlated Solutions Inc.). Prior to testing, the TZM anvils (load-frame fixtures) were lubricated with a spray coating of the lubricant boron nitride to minimize friction effects.

The resulting flow curves were then fit by a Ramberg-Osgood type strain hardening law [8] which follows the functional relationship

$$\varepsilon = \varepsilon_e + \varepsilon_p = \frac{\sigma}{E} + \left(\frac{\sigma}{\sigma_0}\right)^N$$

(2.1)

to determine relevant flow parameters. In this analysis the elastic modulus ($E$) is used to determine the 0.2 % offset yield ($\sigma_y$) and to compute the true plastic strain ($\varepsilon_p$) by subtracting the elastic strain response ($\varepsilon_e$) from the total true strain ($\varepsilon$). The offset and slope of a log-log plot of $\sigma$ vs.$\varepsilon_p$ are then used to compute the plastic flow coefficient ($\sigma_0$) and the strain hardening exponent ($n$), respectively. Unlike other hardening laws, the Ramberg-Osgood analysis described yielding as continuous process, introducing no discontinuity between elastic and plastic deformation, making it an excellent relationship for modeling purposes [8].

2.4) FRACTURE TOUGHNESS TESTING

Fracture toughness of each material condition was measured by a Chevron Notch Short Rod Specimen (CNSR) geometry, as depicted in fig. 2.7, and described in ASTM E1304.
Specimens conforming to E1304 are specifically designed to produce an overall measurement of fracture toughness, $K_{IV}$, which for a broad range of materials displaying toughness values less than 35 MPa√m, shows great congruency with more conventional tests for determining $K_{IC}$ [9]. When the specimen response is properly qualified, the fracture toughness can be computed directly from the expression

$$K_{IV} = \frac{P_{MAX} Y^*_m}{B \sqrt{w}}$$

(2.2)

where $P_{MAX}$ is the maximum applied load from a load-mouth-opening-displacement trace, $Y^*_m$ is the minimum in the crack-driving force coefficient, $Y(a)$—a dimensionless function representative of the stress state in a particular geometry as a function of crack length ($a$)—$B$ is the diameter of the specimen (12.70 mm), and $w$ is the length of the specimen from the load line to the back face (18.41 mm). For the configuration tested ($w/B = 1.45$), E1304 specifies that $Y^*_m$ is 29.21. These dimensions were chosen such that the longest dimension would be nominally less than the final dimensions of the most heavily deformed material condition, in this case the diameter of the 46:1 extrusion—31.75 mm.

The expression for toughness may be stated more generally as

$$K = \frac{PY(a)}{B \sqrt{w}}$$

(2.3)

a form that can be used to calculate the stress-intensity factor for any crack length ($K(a)$)—an R-curve) provided there is a suitable method for measuring crack length at a given load, and the functional form of the stress-intensity coefficient, $Y(a)$, is known. Using a proper calibration curve—an experimentally or numerically determined relationship between specimen compliance ($c$) and crack-length ($a$)—$a$ can be calculated via compliance as measured from loading-unloading events during a test, like those depicted in fig. 2.8, and subsequently used to generate R-curves by the method described.
Figure 2.7: Schematic of the Chevron Notch Short Rod Geometry used to determine fracture toughness.
While E1304 does not explicitly describe compliance and stress intensity factors as a function of crack length, such calibration data are available in the literature for the $w/B = 1.45$ geometry from both finite element analysis and calibration specimens [10,11,12,13]. For completeness, these calibrations are plotted in figs. 2.9 and 2.10 respectively; instead of absolute crack length ($a$, measured from the load line) and compliance ($C$, the inverse stiffness of each subsequent load drop measurement), these calibrations are described in terms of a normalized crack length $\alpha = a/w$, and dimensionless compliance $c = CEB$, where $E$ is the elastic modulus of the material. These data from the literature were fit with 4th degree polynomials ($R^2 \approx 0.999$) to obtain the closed form expressions for $\alpha(c)$ and $Y(\alpha)$:

$$\alpha(c) = -2.4595E-11 + 3.5080E-08c^3 - 2.0141E-05c^2 + 6.0144E-03c - 9.7031E-02 \quad (2.4)$$

$$Y(\alpha) = 1259.5\alpha^4 - 2668.1\alpha^3 + 2358.4\alpha^2 - 989.88\alpha + 183.51 \quad (2.5)$$
Figure 2.9: Fit of stress intensity coefficient ($Y$) vs. relative crack length ($\alpha$) for w/B = 1.45, after [12].

Figure 2.10: Fit of relative crack length ($\alpha$) vs. dimensionless compliance ($c_{EB}$) for w/B = 1.45, after [13].
Crack Arrest

The popularity of the CNSR geometry can be attributed to outstanding aspects of its design as related to crack growth. By looking at the stress intensity coefficient as a function of crack length (fig. 2.9), one observes that the crack driving force is initially negative in slope and approaches a minimum that causes crack arrest at a geometrically defined crack length ($\alpha = 0.55$ or, relative to the notch, $\Delta a = 4.1$ mm for the considered test geometry). The negative slope prior to arrest encourages metastable crack growth with an initially very large crack driving force, $K$; this eliminates the need for specimen pre-cracking when only a single-valued measure of toughness is desired. Furthermore, in the absence of any $R$-Curve behavior, peak load occurs coincident with the crack length at arrest (at $\Delta a = 4.1$ mm), greatly simplifying the determination of $K_{II}$ (eq. 2.3); in short, only the peak load is needed to compute the toughness of a material tested with this geometry in the absence of $R$-curve behavior.

Constraint

Another extremely beneficial aspect of using the CNSR geometry is that the “plunge” cut of the chevron notch from the outer specimen diameter behaves similar to side groves used on conventional compact tension or bend geometries; imposing a strong level of constraint, and reducing the minimum specimen size required to prevent any toughness/thickness interaction. An often cited [14] relationship for the minimum size requirements to maintain elastic constraint/small scale yielding is given by

$$a, B, (w - a) \geq 1.25 \left( \frac{K_{II}}{\sigma_y} \right)^2$$

(2.6)

Using anticipated values of toughness and yield strength with exceptional safety margin, the CNSR specimen dimensions were chosen such that there would be no difficulty in satisfying this relationship.
Methodology

All CNSR specimen testing was conducted in an MTS-0810 load frame with a 10 kN load cell, operated under displacement control, at a displacement rate of 0.003 mm/s. All specimens tested were knife-edge loaded 0.64 mm from the edge of the specimen flange (directly in line with the gauge seats depicted in fig. 2.7) via the grips depicted in fig. 2.11. The load cell was calibrated and certified prior to use, as was machine alignment. Grip alignment was checked between each test. Rather than monitoring crack mouth opening displacement ($\delta$) with a clip gauge, this parameter was monitored via a video displacement measurement system (Correlated Solutions, Inc.), which was synchronized with load cell data from the test frame via an integrated A/D card.

Figure 2.11: Close view of the CNSR test setup.
Crack Length Measurement

In order to validate the measurement of $K$ via the CNSR geometry, an accurate, redundant, measurement of crack length is necessary, especially since R-Curve behavior has been observed in materials similar to those in this study. Consequently, crack length was measured both by elastic compliance, and optically post facto from still photos taken via the video displacement system prior to each unloading. Compliance measurements were taken every 0.01 mm increment of mouth opening displacement, with a programmed load drop of 25% of the peak-achieved load, in order to avoid any misalignment on reloading, or a large effect of plasticity from the 95% unloading recommended in the ASTM E1304. Optical crack length photos were taken at the peak load prior to each compliance unload. In order to make the crack visible under incident lighting, it was necessary to polish off the EDM recast layer in the notch using index cards and successive grades of diamond paste. After polishing, each specimen was visually inspected to ensure that the position and curvature of the notch was within specifications of the ASTM standard. An ancillary benefit of this polish was to mitigate any effect of the recast layer on crack initiation and propagation. Sample images from which crack length measurements were taken are depicted in fig. 2.12.

Specimen Alignment

At each load drop/crack length measurement the video system was paused to measure the mouth opening displacement on both sides of the knife edge grips via a Vernier caliper. This validated the accuracy of the video system, and confirmed that specimen alignment was maintained during testing; an important necessity since at small loads, the CNSR configuration is inherently unstable, and can lead to erroneous results, even crack propagation out of the plane of the chevron may occur (fig. 2.13). Data from any specimen where this occurred was discarded.
Figure 2.12: Video displacement system still photos used for the determination of crack length in the 46LT condition at (a) initiation, (b) arrest, and (c) near instability; inverse color scale is used to more easily identify the crack tip; spacing between hash marks on the adhesive scale is 1mm.
Figure 2.13: Crack propagation out of the intended plane of fracture due to specimen misalignment.

Notch Root Correction

Since CNSR specimens were not fatigue pre-cracked, the measured toughness in the vicinity of the notch is significantly affected by its curvature/size. In this region, the size of the notch relative to a sharp crack acts to effectively reduce the effective stress intensity, causing erroneously high toughness values in this region. Using an elastic analysis [15], it is possible to obtain the relationship between the stress intensity at the notch ($K_I'$) and a sharp crack ($K_I$)

$$K_I = \frac{K_I'}{1 + \rho/2r}$$  \hspace{1cm} (2.7)

where $\rho$ is the radius of the notch and $r$ is the crack length measured from the center of curvature of the notch. Using this relationship, and the notch root radius of the CNSR configuration tested
(0.125 mm), $K_I$ and $K_{I'}$ become indistinguishable at (read: the notch exhibits an influence only up to) a crack length $\sim 2$ mm from the notch root ($\Delta a \approx 2$ mm). Such an analysis was used to determine the initiation toughness ($K_c$), in order to determine whether there was any significant R-Curve behavior.

2.5) QUANTITATIVE FRACTOGRAPHY

Fractography

Fracture surface features were observed via a Leica 460FEII SEM to ascertain the qualitative effect of reinforcement homogeneity on fracture surface features. After several initial tests it became readily apparent that it would be difficult to make a convincing argument regarding the effect of homogeneity on fracture behavior purely on qualitative fracture surface observations since differences in fracture surfaces between material conditions were evident but subtle.

Fracture Surface Roughness

After testing, visible variations in fracture surface roughness were observed, and quantitative fractography was pursued. The average roughness ($R_A$) of a fracture surface was quantified with a VEECO Wyko NT1100, an interferometric optical profiler. Five (5) adjacent 3D topographic maps were taken in the vicinity ($\pm 1$mm) of the crack arrest length ($\Delta a = 4.1$ mm) from each fractured CNSR specimen for which a valid toughness measurement ($K_{IV}$) was obtained. A typical topographic map, as well as the collection arrangement is depicted in fig. 2.14. These topographic maps measured 614 $\mu$m x 460 $\mu$m in extent. The lateral spatial resolution of the topographic maps was 0.498 $\mu$m, while the out-of-plane resolution was 9.49E-3 $\mu$m. The mean from the average roughness measured from each topographic map was then used to compute an average roughness for each specimen, and grouped subsequently to compute an average $R_A$ for each test condition.
Figure 2.14: Crossing pattern for the collection of topographic maps from fracture chevron notch short rod specimens, as well as a sample topographic map.

2.6. REFERENCES


III

RESULTS & ANALYSIS

3.1.) MATERIAL CHARACTERIZATION

Reinforcement Homogeneity

The homogenous length scale \((L_H)\) is a quantity developed to determine a representative length scale for a particular microstructure based on the variations in volume fraction of a second phase as a function of length scale \((\eta)\) [1]. To illustrate the behavior of variation in second phase volume fraction as a function of length scale, consider the different regions of the same microstructure depicted in fig. 3.1, from which it can be observed that as a microstructure is sampled at various length scales, there is a large variation in apparent volume fraction of second phase between sub-regions at small length scales \((\eta = 50 \mu m)\), while at larger length scales \((\eta = 200 \mu m)\) this variation becomes less significant. Qualitatively, the homogenous length scale \((L_H)\) for a particular microstructure is the (large) length scale at which the variation between each sub-region becomes imperceptible—\(L_H\) is that length scale at which a microstructure becomes statistically indistinguishable from a larger volume of material; \(L_H\) is the minimum length scale necessary to construct a volume element representative of a particular microstructure.

In a DRA-like microstructure, \(L_H\) is sensitive to local variations in reinforcement distribution, particularly nearest neighbor separation—a parameter clearly of consequence to ductile fracture. Spowart et. al. have demonstrated the utility of \(L_H\) in characterizing clustered microstructures via synthetic microstructures of constant second-phase volume fraction with varying degrees of clustering [1]. The degree of clustering was defined by a “cluster factor” \((f_c)\) equal to the fraction of particles included in clusters (fig 3.2); microstructures were “generated” by randomly placing a fraction \((1-f_c)\) of particles (disks) with no overlap, and then randomly placing the remaining fraction of particles \((f_c)\) in contact with, but also not overlapping, existing particles. As the cluster factor was increased—corresponding to a decreasing nearest neighbor separation—so did \(L_H\), regardless of volume fraction (fig 3.3); as the level of clustering increases, a larger volume element is required to characterize a microstructure (to an equivalent level of confidence).
Figure 3.1: Example of fluctuation in volume fraction as a function of length scale in DRA 6092/25.0/SiC$_p$. 
Figure 3.2: Effect of cluster factor ($f_c$) on the microstructure arrangement in synthetic microstructures [1].

Figure 3.3: Effect of cluster factor ($f_c$) on the homogenous length scale ($L_{H.0.04D}$) for several volume fractions of reinforcement [1].
The homogenous length scale of a real microstructure may be quantified by a technique suggested by Spowart et. al. [1] termed the multi-scale analysis of area fractions (MSAAF). In this method, a digitized microstructure is re-sampled at various resolutions (corresponding to various length scales, \( \eta_n \)) to determine the volume fraction of a particular sub-region. Take for example, fig. 3.4, in which an initial microstructure is subdivided into 3 lower resolutions/length scales: \( \eta_1 \), \( \eta_2 \), \( \eta_3 \). After subdivision, the effective volume fraction (\( V_f \)) is assigned to each sub-region, visually depicted as a gray level in fig 3.4. By using all points available at the chosen length scale, an average volume fraction and standard deviation (\( \sigma_{Vf} \)) in volume fraction may be determined as a function of length scale. Though the average volume fraction over all sub-regions for a particular length scale (\( \eta \)) will not change with length scale, the standard deviation in this quantity will. The evolution of (\( \sigma_{Vf} \)) normalized by volume fraction (\( V_f \)) as a function of dimensionless length scale (\( \eta/D \)—where \( D \) is the reinforcement diameter) for the microstructure of fig 3.4 is plotted in fig. 3.5.

**Figure 3.4:** Application of the MSAAF technique to a synthetic microstructure [1].
As has already been qualitatively described, after a certain length scale, the behavior of $\sigma_{Vf}/V_f$ clearly becomes self-similar (linear on the log-log scale). This self-similar (fractal) behavior of variation in volume fraction with length scale has been addressed by Spowart et al., who assumed that, if reinforcement particles are distributed discretely (according to Poisson statistics), the standard deviation in volume fraction of a particular sub-region of a material should be related to the number of particles in that region ($m$), according to the relationship

$$\sigma_{Vf} = \sqrt{m}$$  \hspace{1cm} (3.1)

and using the identity relating the number of (monosized) particles with mean diameter $D$, to their volume fraction in a given region of length $\eta$,

$$m\left(\frac{1}{4} \pi D^2 \right) = \eta^2 V_f$$  \hspace{1cm} (3.2)
it can be seen that the deviation in volume fraction is given by

$$\sigma_{Vf} = \frac{2\eta}{D^2} \sqrt{\frac{V_f}{\pi}}$$

(3.3)

or, if we normalize the deviation by the absolute volume fraction, and eliminate \(m\) with eq. 2, we obtain

$$\frac{\sigma_{Vf}}{V_f} = 1 \left( \frac{\eta}{D^2} \right) \frac{1}{2} \sqrt{\frac{\pi}{V_f}}$$

(3.4)

a dimensionless relationship that describes the variation in (normalized) volume fraction as a function of (dimensionless) length scale, which can be seen to quite accurately fit the results of the MSAAF analysis (fig. 3.5.) at larger normalized length scales. All this self-similar behavior is indicating is that as the size of a window used to view the microstructure is increased, the confidence that this window is a faithful representation of the microstructure is also increasing (microstructural variation is decreasing).

Experience with engineering materials has shown that the slope of this region of self-similarity is identical as long as compared microstructures contain nominally the same size and shape reinforcement. This concept is illustrated by MSAAF plots of the previously discussed synthetic (clustered) microstructures depicted in fig. 3.6. In this figure, it can be observed that all particle clustering (as measured by cluster fraction) does is shift the MSAAF plot further to the right. Therefore, it is possible to characterize a microstructure, and make comparisons of differences in homogeneity between microstructures, using a single value of length scale at a predetermined level of confidence \((\sigma_{Vf}/V_f)\). For this reason, the microstructural analysis of materials in this study will characterize homogeneity with the so-called 1% homogenous length scale, \(L_{H} \equiv L_{H-0.01}\)—the length scale at which the variation in microstructure becomes less than 1% (i.e., \(\sigma_{Vf}/V_f = 0.01\)), as indicated by the arrows in fig. 3.6.
When studying a volume of material that is anisotropic and inhomogeneous, it is useful to logically extend the concept of the homogenous length scale by measuring the directional variation in volume fraction as a function of length scale to compute a directional homogenous length scale, $L_{Hn}$, where $n$ is an integer index (valued from 1-3 for a 3-dimensional microstructure) denoting a particular (orthogonal) direction in the material [2]. Only a simple modification to the MSAAF technique is required to obtain values of $L_{Hn}$ from plane sections of a material. As is depicted in fig. 3.7, rather than sub-division of a microstructure into square elements, each line of pixels from a digital image of a microstructure—in the direction of interest—is concatenated into a linear array of pixels which is, for each length scale ($\eta$) of interest, subdivided into a corresponding number of strips. The variation in volume fraction of each of these strips is used to generate a directional-MSAAF plot from which a 10% homogenous length scale can be determined. A routine based on the MSAAF technique for the determination of both the homogenous length scale as well as the directional homogenous length scale from metallographic sections, is included in Appendix C.
Figure 3.7: Schematic depiction of the directional-MSAAF technique for the determination of isotropic and anisotropic homogenous length scales.
Homogeneity of Processed DRA

Deformation processing is highly effective in homogenizing reinforcement distribution, as can be qualitatively observed in figs. 3.8-10. These figure display 3-dimensional views of each material condition where each edge is labeled by the appropriate directional homogenous length scale ($L_{Hn}$). Fig. 3.11 depicts more detailed views of both the transverse (TS) and longitudinal (TL) sections for each degree of deformation. Qualitatively, it is apparent that the microstructure of the initially consolidated material is isotropic and clustered. After an 8:1 reduction in cross-section, the material demonstrates an anisotropic/semi-banded microstructure which is aligned parallel to the extrusion axis. This banded microstructure becomes thoroughly disrupted after a reduction of 46:1 although there is still a clear particle alignment along the extrusion axis in this condition.

Along the extrusion axis, there is an increase in the homogenous length scale ($L_{H3}$) with deformation, there is concomitantly a much greater decrease in the transverse homogenous length scales ($L_{H1}$ & $L_{H2}$). When these representative lengths of the microstructure are used to construct a representative volume element (RVE), such as those in figs. 3.8-10, it becomes apparent that the RVE is decreasing in volume ($V_{RVE}=L_{H1}xL_{H2}xL_{H3}$) with increasing deformation, as depicted in fig. 3.12. This result is fully anticipated since as the distribution of reinforcement becomes more uniform, it should take a smaller volume element to effectively characterize the material.

The decrease in the transverse homogenous length scale with deformation predominantly reflects an increase in nearest and second-nearest neighbor separation with extrusion (fig. 3.13), as indicated by the probability density and cumulative distributions of these quantities depicted in fig. 3.14 (nearest neighbors: solid curves, second nearest neighbors: dashed curves)—in this sense deformation processing seems to be very effective in homogenizing the reinforcement distribution.
Figure 3.8: Representative volume element (RVE) for the “1:1” extrusion (initial 20” Billet); arrows indicate the direction of consolidation.
Figure 3.9: Representative volume element (RVE) for the 8:1 extrusion; the arrow indicates extrusion axis.
Figure 3.10: Representative volume element (RVE) for the 46:1 Extrusion; the arrow indicates the extrusion axis.
Figure 3.11: Representative longitudinal (TL) and transverse (TS) sections of each extruded condition (1:1, 8:1, and 46:1).
Figure 3.12: Size of the representative volume element defined by constituent/orthogonal $L_{10,10,0}$ as a function of extrusion ratio.

Figure 3.13: Relationship between homogenous length scale ($L_{H1}/L_{H2}$) and median nearest neighbor separation in the plane transverse to the extrusion axis.
Figure 3.14: Normalized nearest and 2nd nearest neighbor distributions for longitudinal (TL) and transverse (ST) sections from each deformation condition.
From these distributions, it can be seen that the median normalized nearest neighbor separation 
\( \lambda_{0.50} = (R/D)_{0.50} \), centroid-centroid separation/reference particle diameter) is gradually pushed to larger and larger values with increasing deformation. Though the mean would be a valid statistic to quantify this distribution, the distribution is not normal, and therefore, the mean is not as sensitive as the median to variations in the microstructure occurring at shorter length scales—those that are most relevant to fracture. As predicted, changes in statistical homogeneity on very large length scales (400-900 \( \mu m \)) are the leveraged results of seemingly minor changes in particle separation and orientation occurring on much shorter length scales (< 10 \( \mu m \)). The statistics quantifying these distributions are tabulated in Table 3.1.

**Table 3.1:** Particle distribution, size, and aspect ratio (AR) statistics for longitudinal (TL) and transverse (ST) sections of DRA 6092/25.0/SiC<sub>p</sub>.

<table>
<thead>
<tr>
<th>Condition</th>
<th>((R_{1NN}/D)_{0.50})</th>
<th>((R_{2NN}/D)_{0.50})</th>
<th>(D_{0.50}) ((\mu m))</th>
<th>(AR_{0.50})</th>
</tr>
</thead>
<tbody>
<tr>
<td>1TL</td>
<td>1.58</td>
<td>2.56</td>
<td>2.39</td>
<td>2.21</td>
</tr>
<tr>
<td>8TL</td>
<td>1.71</td>
<td>2.67</td>
<td>2.34</td>
<td>2.26</td>
</tr>
<tr>
<td>46TL</td>
<td>1.80</td>
<td>2.72</td>
<td>2.28</td>
<td>2.40</td>
</tr>
<tr>
<td>1TS</td>
<td>1.51</td>
<td>2.28</td>
<td>2.39</td>
<td>2.21</td>
</tr>
<tr>
<td>8TS</td>
<td>1.92</td>
<td>2.72</td>
<td>2.30</td>
<td>2.23</td>
</tr>
<tr>
<td>46TS</td>
<td>2.10</td>
<td>3.10</td>
<td>2.28</td>
<td>2.15</td>
</tr>
</tbody>
</table>

**Homogeneity & Particle Alignment**

With deformation, it was anticipated that the non-spherical reinforcement particles should align along the extrusion axis. The orientation of each particles major axis and the associated statistics of their distribution were collected from TL sections of each deformation condition, and are reported in fig. 3.15; the frequency of occurrence for a particular angle of major axis orientation is reported in the “upper” plots, while a polar plot of the same probability distribution data is depicted in the “lower” plots. As can be seen from these analogs to orientation distribution functions, there is a clear propensity for increasing particle alignment along the extrusion axis (0°) with increasing deformation. It is unlikely that it is a coincidence that the homogenous length scale along the extrusion axis is a factor of ~2 larger than the transverse direction in both the 8:1 and 46:1 extrusions, since this is approximately equal to the reinforcement aspect ratio.
Figure 3.15: Effect of deformation processing on reinforcement alignment along the longitudinal direction form all deformation conditions.
Consequently it is reasonable to assume that the increase in the homogenous length scale along the extrusion axis with deformation reflects more the increase propensity of alignment of the reinforcement’s major axis along the extrusion axis, rather than any change in the normalized nearest neighbor separation, a quantity—in this plane (TL) of the microstructure—which is only modestly increased with deformation (fig. 3.14). The debit in homogenous length scale along the extrusion axis between the 8:1 and 46:1 conditions is most likely a result of the breakdown of the banded microstructure and the loss of the elongated clusters which are the origin of these reinforcement bands.

Furthermore, the decrease in transverse homogenous length scale with extrusion should be primarily a result of increasing nearest neighbor separation rather than any effect of particle alignment since there are no apparent changes in alignment when similar measurements are made in the ST plane, an assumption supported by the stability of the median particle aspect ratio over all deformation conditions in the same orientation. Although at first glance this behavior was not intuitive since a decrease in particle aspect ratio transverse to the extrusion axis was expected, it is consistent with the fact that the reinforcement is more flake-like than acicular.

**Particle Size**

A primary concern of using extrusion to control reinforcement homogeneity is that with a large degree of deformation imposed, there could be a propensity for altering the size distribution of the reinforcement through particle fracture (fragmentation). Accordingly, fields of view with dimensions comparable to the homogenous length scale for each microstructure were used to determine particle size distributions for longitudinal and transverse sections from each forging. Statistics regarding particle size and aspect ratio are reported alongside nearest neighbor distribution data in Table 3.1. Generally, it is found that particle size (as measured by equivalent circular diameter, \(D\)) is distributed very similarly regardless of deformation condition (fig. 3.16). These data confirm that there is indeed little particle fragmentation with deformation, and that the median particle size (equivalent circular diameter) over all conditions can be approximated as 2.3 \(\mu m\).
Nearest Neighbor Inclination

Also of interest was whether, as a result of extrusion, there was any change in the angular location (centroid-to-centroid) of nearest neighbors. During the determination of nearest neighbor distribution for the longitudinal (TL) sections of each distribution, this angle (relative to the extrusion axis) was computed, results of this measurement are reported in fig. 3.17; similar to the measurements of particle alignment, the “upper” plots depict the frequency of occurrence as a function of angle from the extrusion axis while the “lower” plots depict a polar plot of these same data. As can be seen, concomitant with particle alignment along the extrusion axis, the polar plots indicate a slight increase in 4-fold symmetry along and transverse to the extrusion axis, indicating that deformation processing, and the associated reinforcement homogenization does preferentially alter the angular position of nearest neighbors; similar results were obtained for the TS planes. This suggests that there may be a fundamental microstructural reason to expect a change in damage localization and crack deflection, in addition to any effect of the increase in the transverse nearest neighbor distribution with extrusion.

Crystallographic Texture

From diffractometer measurements performed on transverse (TS) oriented plane sections from each deformation condition, crystallographic planes were indexed, and pole figures were computed for the (111), (200), (220), (311), and (220) planes; these are presented in figs. 3.18-3.20 (a). Corresponding best-fit orientation distribution functions (ODFs)—a list of Euler angles representing particular crystallographic planes and weights representing the intensity of alignment of those planes with the reference frame attached to the extrusion axis—are also plotted (b), along with corresponding inverse pole-figures (c), and a reference standard projection for an FCC system (after [3]). In all figures, the so-labeled “1” and “2” axes are those transverse to the extrusion direction, while the “3” direction is parallel to the extrusion axis.
Figure 3.16: Influence of deformation processing on the reinforcement size distribution.
Figure 3.17: Effect of deformation processing on longitudinal (TL) nearest-neighbor inclination for the (a) “1:1”, (b) “8:1” and (c) “46:1” conditions.
In the 1:1 condition the matrix texture is axisymmetric but nearly random. With further processing, there is an increasing propensity for alignment of the \{111\} and \{100\} planes along the extrusion axis as can be seen from the increase in grain alignment probability from 1.77x random to 2.56x random with extrusion at 8:1, and to 4.81x random with extrusion at 46:1. *In short, a substantial and increasing fiber texture with \{111\} and \{100\} components aligned along the extrusion axis is developed in the matrix with increasing extrusion*, consistent with other experiments on the extrusion of Al-Mg-Si systems [4]. This texture is likely a result of recrystallization rather than cold work since light microscopy of etched microstructures indicated that grains remained equi-axed with processing and the variation in yield strength with continued extrusion is relatively small. No strong texture was observed in the SiC reinforcement even though diffraction data showed clearly that the reinforcement was hexagonal—possessing a “hard” axis—strongly suggesting that reinforcement alignment occurred as a result of particle aspect ratio rather than crystallographic orientation. This fact, combined with the lack of a preferred particle orientation or nearest neighbor inclination justifies the investigation of flow behavior in the 1:1 extrusion with a single compression specimen orientation, and the fracture behavior of the same with a single CNSR orientation.

### 3.2.) MECHANICAL BEHAVIOR

The orientation of compression specimens and CNSR fracture specimens from each forging are described in fig. 3.21. Generally, two orientations of compression specimen were taken from each forging, one oriented along the extrusion axis (L), and one transverse (T), with the exception of the initial billet, that was, after microstructure characterization, assumed to be isotropic; in this case flow behavior in only the longitudinal orientation was assessed. Three orientations of CNSR specimens were also taken from each forging, oriented in the TL, LT, and TS planes (fig 3.21). In analogy to the compression specimens, because of the perceived isotropy in the undeformed billet, only one orientation of CNSR was taken from that forging, that along the TL orientation. For the sake of ensuing arguments, the behavior of this orientation (1TL) is assumed to be identical to what would be measured in the 1LT and ITS orientation.
Figure 3.18: (a) Raw pole figures, (b) best fit ODFs, and (c) inverse pole figures for the “1:1” extrusion.
Figure 3.19: (a) Raw pole figures, (b) best fit ODFs, and (c) inverse pole figures for the 8:1 extrusion.
Figure 3.20: (a) Raw pole figures, (b) best fit ODFs, and (c) inverse pole figures for the 46:1 extrusion.
Figure 3.21: Orientations of mechanical tests relative to the representative volume element of each material condition.
Deformation Response

Representative compressive flow curves for each test orientation are compiled in fig. 3.22a, while parameters describing deformation response—yield strength ($\sigma_y$), elastic modulus ($E$), and strain-hardening exponent ($n$) are reported in table 3.2. Generally, deformation processing and the resulting microstructure have only a modest effect on yield strength. Using the 1L flow behavior as a common origin for longitudinal (L) and transverse (T) orientations, it is observed that extrusion at 8:1 causes a debit in the transverse yield, while improving the longitudinal strength; further extrusion to 46:1 increases strength in both orientations simultaneously (fig. 3.22b). The effects of deformation processing seem to have no significant (measurable) impact on elastic modulus. An overall trend of decreasing strain hardening exponent in both the L and T oriented specimens attends increasing extrusion.

To separate the effect of texture on yield from those of reinforcement alignment and homogenization, each orientation and degree of deformation requires a more detailed analysis of the collected texture data. To estimate the effect of a textured ensemble of grains on yield strength, the Taylor factor ($M_i$)—a measure of plane strength relative to the critical resolve shear stress ($\sigma_x/\tau_{CRSS}$)—is computed for each plane in the ODF [5]. From the associated weights representing the intensity of plane alignment with the reference frame ($w_i$), it is then possible to compute an ensemble Taylor factor using the weighted average of all the Taylor factors via

$$M = \frac{\sum_i w_i M_i}{\sum_i w_i} \quad (3.1)$$

This ensemble Taylor factor for each deformation condition (in uniaxial compression) is reported for both the longitudinal (L) and transverse (T) orientations in Table 3.2, and plotted as a function of extrusion ratio in fig. 3.23.
Figure 3.22: (a) Longitudinal (L) and transverse (T) compressive flow behavior for each material condition as well as (b) the influence of extrusion ratio on yield strength in each orientation; 1:1 transverse yield is assumed equal to longitudinal yield from material isotropy.
Table 3.2: Parameters characterizing material compressive deformation response.

<table>
<thead>
<tr>
<th>Condition</th>
<th>$E$ (GPa)</th>
<th>$\sigma_y$ (MPa)</th>
<th>$n$</th>
<th>$M$</th>
</tr>
</thead>
<tbody>
<tr>
<td>46L</td>
<td>145</td>
<td>483</td>
<td>0.084</td>
<td>3.24</td>
</tr>
<tr>
<td>8L</td>
<td>148</td>
<td>476</td>
<td>0.085</td>
<td>3.23</td>
</tr>
<tr>
<td>1L</td>
<td>148</td>
<td>445</td>
<td>0.098</td>
<td>3.06 (L)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>3.09 (T)</td>
</tr>
<tr>
<td>8T</td>
<td>143</td>
<td>439</td>
<td>0.090</td>
<td>3.03</td>
</tr>
<tr>
<td>46T</td>
<td>148</td>
<td>459</td>
<td>0.086</td>
<td>3.10</td>
</tr>
</tbody>
</table>

Figure 3.23: Influence of extrusion ratio on Taylor factor in longitudinal (L) and transverse (T) directions.
Fracture Toughness

From load vs. mouth opening displacement ($P-\delta$) traces for chevron notch specimens from each condition, load and compliance were measured for each unloading, and compared with the measured optical crack length relative to the notch ($\Delta a$) to compile overlaid plots of $P-\delta$ and $\Delta a-\delta$ for each specimen, such as the one depicted in fig. 3.24. An important observation that is common to every such plot is that there exists a consistent discrepancy between compliance predictions of crack length and optical measurements at short crack lengths, and that these two measurements ultimately converge at increasing $\delta$ / larger crack lengths ($\Delta a$). This discrepancy in crack length predictions is an effect of the relatively blunt notch root having a lower effective stress intensity than a sharp crack, such as what one would expect from a pre-cracked specimen—the type of crack for which available compliance calibrations are designed.

This behavior emphasizes the necessity of using optical crack length measurements to locate the crack tip, and compute $K-\Delta a$ from the collected data via the appropriate weight function $Y(\Delta a)$, such as is done for all replicates in the 46LT condition in fig. 3.25. Notice that the effect of the notch is visible for up to ($\Delta a$) 2 mm away from the notch root, well before the (arrest) crack length at which $K_{iv}$ is computed ($\Delta a = 4.12$ mm). In order to account for the effect of the notch and ascertain if there is any resistance curve behavior, it is necessary to apply a notch root correction in the vicinity of the root ($\Delta a < 2$ mm). For simplicity, we identify the load in the $P-\delta$ plot at the onset of non-linearity to determine $K$ at crack initiation ($K_c$), and then apply the Paris notch root correction to estimate the correct initiation toughness. Plotting the average value of this corrected $K_c$ with average values from $K_{iv}$ (at arrest) and $K$ at instability, an approximation of the resistance curve behavior for each condition can be obtained, this is depicted in fig. 3.26. Computed average values for $K_{iv}$ are reported in table 3.3. Generally, it can be seen that deformation processing—and presumably the associated reinforcement homogeneity—greatly enhances the fracture resistance of all orientations, though as expected from material anisotropy, each orientation improves at a different rate with degree of deformation.
Figure 3.24: Load-displacement/crack length measurements for a 46LT specimen (A).

Figure 3.25: “Resistance Curves” for replicates in the 46LT condition.
Figure 3.26: Notch-root-corrected crack growth resistance curves ($K_I - \Delta a$) for all conditions.
Table 3.3: Fracture toughness ($K_{IC}$) data compiled by condition.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Specimen ID</th>
<th>$K_{IC}$ MPA√m</th>
<th>$\langle K_{IC} \rangle$</th>
<th>$\sigma_{IK}$</th>
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<td>19.4</td>
</tr>
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</table>
From the lack of any apparent resistance curve behavior—over all conditions—it can be inferred that the fracture process zone in this material is highly localized since large-scale discontinuous crack growth, micro-cracking, unconstrained crack tip plasticity, or any other such delocalization of the fracture process zone is the origin of such resistance curve behavior. Consequently, the absence of any resistance curve behavior implies that a single-value toughness \( (K_{IV}) \) can be used to characterize this highly localized fracture process—a process that is most likely occurring on a length scale on the order of the interparticle ligament.

3.3.) FRACTOGRAPHY

Qualitative Observations

Macro images of fracture surfaces are depicted for each fracture orientation in figs. 3.27-30. The fracture surface from the 1TL condition is depicted in fig, 3.27, and the effects of increasing deformation (and reinforcement homogenization) are subsequently depicted for the TL, LT, and TS orientations respectively in figs. 3.28-30. Qualitatively, an increase in fracture surface roughness (undulation out of the plane of fracture) is visible when comparing successive degrees of deformation for any particular orientation.

On closer inspection, at smaller length scales (figs. 3.31-33), it seems that after extrusion at 8:1 there is a modest increase in the extent of primary cavity growth surrounding individual cracked particles (best illustrated in fig. 3.34). It should be further noted that cavities are equi-axed in the LT orientation, while in the TS and TL orientations cavities are elongated along the extrusion axis—transverse and parallel to the direction of crack propagation in the TS and TL orientations, respectively. This anisotropy in cavity shape directly reflects the alignment of reinforcement along the extrusion axis.

The initial reaction to this increase in cavity growth with deformation processing is that, during fracture, it is a manifestation of an increase in plastic strain necessary to rupture the interparticle ligament, while the variation in cavity anisotropy explains the variation in
toughness between orientations. However, as was observed by Davidson [6], the fracture surface topography may appear qualitatively different—on small length scales—simply because of a relaxation of the constraint imposed by nearby damage, implying that improvements in toughness may be simply a result of improved fracture process length scale (interparticle separation) rather than an increase in critical plastic strain with reinforcement homogenization. The absence of any change in the morphology of the fractured interparticle ligament between extrusion at 8:1 and extrusion to 46:1—particularly the absence of any characteristically different microvoid growth in the tearing ridges between particles—further obscures whether the increases in fracture surface roughness and toughness with reinforcement homogenization are due to an increase in fracture process length scale, an increase in the plastic strain associated with cavity growth (at rupture), or some combination thereof.

It is qualitatively unclear whether an “organic” extension of the (short length scale) cavity growth or the increase in interparticle separation is the origin of the apparent increase in (large length scale) surface roughness that occurs over all orientations with deformation processing. Any relationship between these two length scales and the relationship between all prior observations emphasizes the need for more quantitative metrics for evaluating fracture surface features.
**Figure 3.27:** Fracture surface near arrest in the 1TL condition (specimen B), arrow indicates direction of crack propagation.
Figure 3.28: Fracture surface near arrest in the 8TL (specimen B) and 46TL (specimen C) conditions.
Figure 3.29: Fracture surface near arrest in the 8LT (specimen C) and 46LT (specimen B) conditions.
Figure 3.30 Fracture surface near arrest in the 8TS (specimen D) and 46TS (specimen D) conditions.
Figure 3.31: Local influence of reinforcement homogenization on surface topography in the TL orientation.
Figure 3.32: Local influence of reinforcement homogenization on surface topography in the LT orientation.
Figure 3.33: Local influence of reinforcement homogenization on surface topography in the TS orientation.
Figure 3.34: Increase in primary cavity growth over the (a) 1:1 condition after (b) extrusion at 8:1.
Particle Cracking/Interfacial Debonding

Unlike many alloy systems, the reinforcement in the studied composite system is large enough to observe cleavage markings on the second phase directly from the fracture surface, or the residual matrix left on a particle during interfacial debonding. An inspection of specimens from all conditions indicates an overwhelming preference for particle cracking (ex: fig. 3.35), rather than interfacial debonding in those particles participating in fracture.

Figure 3.35: Cleavage markings on cracked particles.
3.4.) QUANTITATIVE FRACTOGRAPHY

Average Roughness Results

In order to confirm the qualitatively observed increase in fracture surface roughness that accompanied the increase in fracture toughness, interferometric optical profilometery was used to digitize fracture surfaces for subsequent quantification. Representative contour maps for fracture surfaces from each orientation are depicted in figs. 3.36-38. Again these images depict a visually clear trend of increasing out-of-plane crack deflection with increasing deformation processing/reinforcement homogenization, and fracture toughness for each orientation. Note that progressively larger regions (with increasing deformation) are colored to the extreme ends of the height spectrum, which is the same in all topographic figures.

The average roughness ($R_a$) was determined from profiles for each condition and is reported in table 3.4. Comparing the average roughness measurements with the measured toughness it is apparent that an increasing average surface roughness corresponds to increased toughness; this is depicted in fig. 3.39, and for clarity of the effect of extrusion on each orientation, in fig. 3.40. Though this trend confirms the qualitative observations, this observation by itself does not enhance understanding of the fracture process since the root cause of this trend of increasing magnitude of out-of-plane undulation with increasing toughness can theoretically have its genesis in either an increasing critical strain with reinforcement homogenization, an increase in the length scale of the fracture process (a decrease in the effective volume fraction of cavity nucleating particles through increased nearest neighbor separation or a decreased probability for cracking), an increase in extrinsic crack deflection, or as some intermediate combination thereof. Consequently, a further geometric (fractal) analysis of this topographic data seemed a logical means for determine what length scales were important to the fracture process, and ultimately elucidate which of the described scenarios is most likely.
Figure 3.36: Topography comparison of fracture surfaces from the TL orientation (all lengths in μm), arrow indicates direction of crack propagation.
Figure 3.37: Topographic comparison of fracture surfaces from the LT orientation (all lengths in μm).
Figure 3.38: Topographic comparison of fracture surfaces from the TS orientation (all lengths in μm).
Table 3.4: Fracture surface average roughness ($R_a$) compiled by condition.

<table>
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<tr>
<th>Condition</th>
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<th>$&lt;R_a&gt;$</th>
<th>$\sigma_{R_a}$</th>
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Figure 3.39: Fracture Surface Roughness ($R_a$) vs. Chevron Notch Toughness ($K_{IV}$) over all Conditions.

Figure 3.40: Fracture Surface Roughness ($R_a$) vs. Chevron Notch Toughness ($K_{IV}$) by Orientation.
Fractal Analysis

Many techniques for measuring fractal dimension are variations on the same theme, using a “covering” to measure the length of a profile as a function of the size of the covering unit (sized in some accord with the length scale $\eta$). For example, the “Minkowski-Bouligand” method covers a fractal curve with disks of various diameters (whose diameter corresponds to a particular value of $\eta$) and measures the integrated area of this covering as a function of $\eta$ (fig. 3.41a) [7]; the more common Hausdorff (“box-counting”) method similarly covers a curve with a number of square boxes of varying edge-lengths, $\eta$ (fig. 3.41b) [8]. For each of these techniques, and in analogy to the Richardson structured walk described earlier, the slope of a log-log plot of covering area vs. yardstick (disk-diameter/box-length), gives the fractal dimension of a considered profile. Yet another method, the power spectrum method, determines the fractal dimension of a profile purely from the slope of a Fourier transform (spectrum) of that profile.

![Figure 3.41](image)

**Figure 3.41:** The (a) Minkowski-Bouligand covering and (b) the Hausdorff “box-counting” coverings, after [7].

Unfortunately, these methods used to determine profile fractal dimension are only marginally accurate since the shape of the covering element (or in the case of the power spectrum method, the windowing function) can significantly skew results of the covering area measurement at higher or lower length scales. This results in an “error” in the fractal dimension measurement that can be very non-linear, depending on the actual fractal dimension of the profile of interest.
This results in each technique having a band of fractal dimension for which the measurement method is most precise. This trend was observed by Dubuc & Tricot, who compare the fractal dimension measured by each of the aforementioned techniques to the actual fractal dimension of reference functions (with known fractal dimension). The results of their comparison—the significant errors of each described technique (Box Counting: BC, Minkowski-Bouligand: MB, and power spectrum: PS)—is captured, through comparison to an ideal measurement (solid line) in fig. 3.42 [9]. Generally, it is observed that techniques for measuring fractal dimension tend to under-predict the actual fractal dimension of a curve.

![Figure 3.42: Accuracy of various techniques for characterizing the fractal dimension of a profile [9].](image)

In order to determine the fractal dimension of profiles with a greater degree of fidelity, Dubuc et al. developed a more sophisticated “variational” covering scheme which overcomes many of the inadequacies associated with the shape of the covering object. This method covers a profile with a “variational” unit (fig. 3.43), an element whose yardstick length ($\varepsilon$, analogous to $\eta$) is determined by its length in the $x$-direction, while its height in the $y$-direction (and by corollary, the area of the unit) is determined by the difference between an upper and lower bound on the profile ($u_\varepsilon(x)$ and $b_\varepsilon(x)$, respectively), which are constructed as a function of the local maximum
and minimum within the neighborhood (the set of points within a distance, \textit{equal} to the length-scale/yardstick, $\varepsilon$, of interest), of the point of interest $x$, or in other words, the covering area $V$ is equal to

$$
V_{\varepsilon}[f(x)] = \int_0^L (u_{\varepsilon}(x) - b_{\varepsilon}(x)) \, dx = \int_0^L \left( \max_{x - \varepsilon \leq \alpha \leq x + \varepsilon} [f(\alpha)] - \min_{x - \varepsilon \leq \alpha \leq x + \varepsilon} [f(\alpha)] \right) \, d\alpha
$$

over the length $L$, of the profile $f(x)$. This covering becomes variational through a parameter $\Delta$ which is the amount of distance (in $x$) this element/window is allowed to slide in order to find the maximum area for that particular yardstick.

This method of Dubuc & Tricot has been implemented by the author and is available as part of a larger algorithm for determining the fractal dimension of profiles from fracture surface data in Appendix D. Explicitly, this larger algorithm verifies and interpolates data as necessary from the collected topographic maps, extracts 20 evenly spaced profiles from the surface in the direction of fracture, and computes the variational covering of each profile as a function of length scale. Covering area is averaged over all 20 profiles for each length scale. The fractal dimension is taken to be the slope of a least squares fit to a plot of this average covering area vs. length scale.

![Figure 3.43: Representative variational covering of Dubuc et. al [9]](image-url)
Method Validation

In order to determine the efficacy of a particular method for determining the fractal dimension of a fracture surface profile, the technique in question is applied to a reference function (or functions) for which the fractal dimension is known. A common reference function is the Weierstrass-Mandelbrot function

\[ W(x) = \sum_{n=-\infty}^{\infty} \frac{1 - e^{i\gamma x}}{y^{(2 - D)n}} e^{i\theta_n} \tag{3.6} \]

which when separated into real and imaginary components, with constant phase (\(\phi_n = 0\) and \(\pi\), respectively), reduce to the series

\[
\begin{align*}
\text{Re} \{W(x)\}_{(\phi=0)} &= \sum_{n=-\infty}^{\infty} \frac{1 - \cos \gamma^n t}{y^{(2 - D)n}}, \\
\text{Im} \{W(x)\}_{(\phi=\pi)} &= \sum_{n=-\infty}^{\infty} \frac{(-1)^n \sin \gamma^n t}{y^{(2 - D)n}}
\end{align*}
\tag{3.7}
\]

These functions are thoroughly discussed in [10] and are capable of generating a wide range of fractal (self-similar) behavior through the variation of 2 parameters: \(\gamma\) and \(D\), where \(\gamma\) is a “shape” parameter determining the nature of the pattern that repeats at all length scales (fig. 3.44), and \(D\), the fractal dimension (fig. 3.45). The utility of each series is obvious, since they represent functions where a known fractal dimension may be “dialed-in”, and can then be measured by any desired technique to determine the accuracy of the technique.

To validate that the author’s implementation of the variational method is functional and correctly measures the fractal dimension of arbitrary profiles, a segment of the Weierstrass-Mandelbrot curve was tested for various fractal dimensions (\(D = 1.01..1.99\), fig 3.45); the fidelity of the algorithm is depicted in fig. 3.46, and is found to agree reasonably with the results of the implementation of the Dubuc & Tricot, which is noticeably more accurate than other techniques available.
Figure 3.44: Weierstrass-Mandelbrot cosine series for $D = 1.5$, $\gamma = 1.2$ (top), $\gamma = 5$ (bottom) [10].
Figure 3.45: Weierstrass-Mandelbrot Sine Series, $\gamma = 2$, for various $D$ (inset).
Fractal Analysis Results

From the qualitative fractography observation that variations in roughness at large length scale may have their origin on shorter length scales, the modified method of Dubuc & Tricot was used on gathered topographic data to determine the influence of reinforcement homogenization on the fractal behavior of fracture surfaces. Results of the analysis for each fracture condition are reported in table 3.5. Richardson-type plots of the relationship between covering area and length scale are depicted by specimen orientation in figs. 3.47-49 where one line representing mean data for each degree of deformation is depicted in each chart along with an accompanying slope of the line of best fit—representing the fractal dimension ($D$). As anticipated, and has been reported by many other authors, these charts indicate that DRA fracture surfaces are indeed fractal, demonstrating self-similarity (linear behavior on these charts) from length scales spanning $10 < \eta < 480 \ \mu m$. Fractal behavior was limited at large length scales by the extent of the topographic data while below $10 \ \mu m$, there was a uniform deviation from fractal behavior (linearity) in all
conditions. Lateral data resolution (0.49 μm) is a possible origin of this short-range deviation, though there exists a possibility that this deviation could be related to a legitimate length scale—though indeterminate—at which self-similar behavior begins, that is, a measure of the critical length scale for the fracture process (~ 10 μm).

It can be observed in figs. 3.47-3.49 that nearly all of the presented Richardson-type plots are coincident at shorter length scales, while deviations in measured covering area seem to develop continuously with length scale. In contrast to the work of Ritchie et al [11] in which multiple mechanisms were observed to act on multiple length scales, no discontinuity that would correspond to a change in dominant mechanism is observed at any length scale. This uninterrupted self-similarity over all measured length scales suggests that the changes in the fracture surface topography at large length scales—in the regime where $R_d$ was measured—are the result of changes in a much more local fracture process (and its topography). This is congruent with the previously observed absence of resistance curve behavior and the association of such behavior with a very localized fracture process. Coupled with qualitative fractography observations of increased primary cavity growth with increasing reinforcement homogeneity, suggest that a local, critical-strain-type model should be an effective descriptor of the fracture process. What remains therefore, is to ascertain what parameter(s) of the fracture process—crack deflection angle, fracture process length scale, and the critical failure strain of the interparticle ligament—are influenced by reinforcement homogenization.

As depicted in figs. 3.50-52 for each orientation, a trend of decreasing fractal dimension with increasing toughness is observed, in concord with the observations of other authors examining ductile fracture surfaces [12]. One possible interpretation of this trend—from the observations of Ritchie et al. that fractal dimension is intrinsically related to crack-tip deflection—is that with increasing reinforcement homogenization, there is a decreasing propensity for crack-tip deflection. This would be in agreement with the crack-tip deflection analysis of Faber & Evans that predicts a decrease in deflection with increased second-phase homogeneity [13], but would not explain the increase in toughness.
Table 3.5: Fracture surface fractal dimension ($D$) by condition.

<table>
<thead>
<tr>
<th>Condition</th>
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<th>$D$</th>
<th>$&lt;D&gt;$</th>
<th>$\sigma_D$</th>
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</table>
Figure 3.47: Mean fractal dimension of fracture surfaces from each condition in the TL orientation.

Figure 3.48: Mean fractal dimension of fracture surfaces from each condition in the TS orientation.
Figure 3.49: Mean fractal dimension of fracture surfaces from each condition in the LT orientation.

\[ K_{IIr} = -95.377D + 145.31 \]
\[ R^2 = 0.8953 \]

Figure 3.50: Fracture toughness \((K_{IIr})\) vs. fracture surface fractal dimension \((D)\) for the TL orientation.
Figure 3.51: Fracture toughness ($K_{IV}$) vs. fracture surface fractal dimension ($D$) for the TS orientation.

Figure 3.52: Fracture toughness ($K_{IV}$) vs. fracture surface fractal dimension ($D$) for the LT orientation.
3.5) REFERENCES


IV

DISCUSSION & MODELING

4.1) MATERIAL CHARACTERIZATION

From interparticle separation and homogenous length scale data, the effect of extrusion on particle arrangement is clear; in the plane transverse to the extrusion axis (TS), nearest neighbor separation is pushed to continuously greater separation, while in the plane parallel to the extrusion axis (TL), there is a more subtle increase in the same quantity (fig. 3.14). Ultimately, this increased particle separation acts to decrease the homogenous length scale in all orthogonal directions with increasing deformation, leading to a substantial reduction in the representative volume element (RVE) necessary to characterize the microstructure—large length-scale changes in homogeneity are the manifestation of changes in small length-scale particle rearrangement. Striking about this process is the variability that can occur on a short length scale for a microstructure with a fixed second phase volume fraction. Consider fig 4.1 which shows the evolution of median nearest neighbor separation as a function of imposed deformation as well as the interparticle separation predicted by volume fraction in the ductile fracture models of Hahn & Rosenfield, and Majmudar & Pandey [1,2]. As can be seen, these models would predict, respectively, a significant overestimate or underestimate of the actual interparticle separation (assuming $V_f = 25\%$). Though in this case it would appear that the separation predicted by Hahn & Rosenfield’s hexagonal array of particles is a reasonable “upper-bound” estimate on nearest neighbor separation, and the Majumdar & Pandey appears to be a good “lower-bound” estimate, this is purely coincidental for the volume fraction of the composite studied. The factor of 0.74 suggested by Majumdar & Pandey to reduce the interparticle separation of the Hahn & Rosenfield model becomes more inaccurate at both higher and lower volume fractions of reinforcement since more configurations of reinforcement are accessible at dilute concentrations, while significantly fewer configurations are accessible at higher loadings. This variance possible in local microstructure arrangement demonstrates how incorrect it is to determine interparticle separation—and fracture process length scale—as simply a function of volume fraction since the associated errors can be substantial; interparticle separation should be measured directly rather than determined via volume fraction.
The anisotropy that develops in the RVE necessary to describe the studied microstructures (fig. 3.8-3.12) is more than likely a result of the increased propensity for particle alignment that occurs with increasing deformation (fig. 3.15). Although the RVE is clearly anisotropic in both extruded conditions, the shrinking of the RVE along the extrusion axis between the 8:1 and 46:1 conditions indicates that the major share of RVE anisotropy—and particle alignment—comes after extrusion at 8:1 and only modestly increases with further deformation; at this point, the contribution to RVE shape from reinforcement alignment is overwhelmed by the increasing reinforcement homogenization/nearest neighbor separation. In effect, reinforcement alignment occurs with a relatively gentle degree of deformation processing, while reinforcement homogenization continues substantially with further deformation. Again, large length-scale changes in RVE anisotropy are the leverage result of changes in particle alignment and separation occurring on very local (small) length scales.
Since the particle size distributions are relatively unchanged with deformation, there is no particle fragmentation, and this parameter cannot be affecting homogeneity. Moreover, from the statistics of nearest neighbor orientation (fig 3.17), it can be seen that there is only a weak preferential direction (angular dependence) for a particles nearest neighbor—regardless of mechanical test specimen orientation or degree of deformation—suggesting that this parameter is not influential in determining the size or shape of the RVE (and the degree of homogeneity) in this material.

Not captured by the variations in homogenous length scale with deformation is the development of a combined intensifying \{111\} and \{100\} fiber texture parallel to the extrusion axis in the matrix as well as the absence of any texture in the reinforcement. It is anticipated that the anisotropy introduced into the material by this matrix texture combined with the tendency for particle alignment with increasing deformation processing will contribute to variations in mechanical tests by orientation while the overarching trend of reinforcement homogenization contributes to any trends that develop over all testing orientations.

### 4.2) DEFORMATION RESPONSE

In order to identify any contribution of reinforcement homogeneity on deformation response, it is necessary to isolate the individual effects of reinforcement alignment and texture. It is possible to determine an estimate of the effect crystallographic texture on yield strength as a function of extrusion ratio and compression specimen orientation by use of the taylor factors and yield strength data reported in Table 3.2; to do this, the yield strength of the 1:1 L condition (\(\sigma_0\)) is normalized by the taylor factor of that orientation to determine the critical resolve shear stress (\(\tau_{CRSS}\)) of the material. This can then be used to determine the yield strength of any orientation by multiplication of \(\tau_{CRSS}\) by the Taylor factor for that orientation (\(m\)),

\[
\sigma_y = m \tau_{CRSS}
\]  

(4.1)

A plot comparing the evolution of yield strength using this technique with the actual yield data as a function of extrusion ratio is depicted in fig. 4.2. As can be seen, texture accounts for the
majority of the modest variation in yield strength of the composite with deformation processing. Generally, extrusion increases the texture induced strength in both orientations, although there is a slight debit in the transverse yield initially at 8:1 before a gradual increase at 46:1.

![Graph showing yield strength vs extrusion ratio](image)

**Figure 4.2:** Comparison of measured longitudinal (L) and transverse (T) yield behavior with texture predictions (Eq. 4.1) as a function of extrusion ratio.

Though an analysis could be performed to separate the effect of texture from the overall yield of the material and determine the combined effect of reinforcement alignment and homogeneity on yield, such an analysis would give contributions of these effects less than the error of measurement. Clearly, in this material, the effect of matrix texture dominates the deformation response, and the effects of reinforcement alignment and homogeneity on yield behavior are diminutive.

### 4.3) FRACTURE BEHAVIOR

The clear trend of increasing reinforcement homogeneity and fracture resistance is depicted in fig 4.3, which shows the increase in chevron notch fracture toughness ($K_h$) with nearest neighbor...
separation; regardless of orientation, there is an overarching trend of increasing toughness with degree of deformation/level of homogenization that overwhelms any difference between toughness by orientation, although there are subtle variations between orientations.

![Graph](image)

**Figure 4.3:** Relationship between nearest neighbor separation and fracture resistance by orientation.

Since the chevron notch fracture resistance of the conditions studied is insensitive to crack length (there is no resistance curve behavior in figs. 3.26), there are no discontinuities present in the Richardson plots for the determination of fractal dimension (figs. 3.26)—differences that Duaskardt *et. al.* associated with the dominance of different features at different length scale [3]—and qualitatively the fracture surface is locally ductile (figs. 3.47-49), it seems reasonable to assume that fracture is itself a very localized process, and that it can be described by a Majumdar & Pandey-like stress-modified critical strain-type model of the form

\[
K_{IC} = \frac{E\sigma_y\delta}{\sqrt{d(n)(1-\nu^2)}} = \frac{E\sigma_y\beta l}{\sqrt{d(n)(1-\nu^2)}}
\]  

(4.3)
where $E$ is the elastic modulus, $\nu$ is the Poisson ratio, $\sigma_y$ is the yield strength, $d(n)$ is the strain hardening component derived from the analysis of Shih, and $\beta$ is the constant of proportionality relating the fracture process length scale, $l$, to the crack mouth opening displacement, $\delta$. $\beta$ is a complex factor strongly dependent on crack deflection angle ($\theta$), the plastic strain sustainable by the material, $\varepsilon_c$, and $l$ [2].

Since the net effect of reinforcement homogeneity on elastic modulus and strain-hardening exponent are imperceptible, and the observed variations in yield strength are only modest, the effect of these deformation parameters on fracture toughness are probably most effectively captured “as-is” in this generalized model; a reasonable assumption since the Majumdar & Pandey model accurately describes the fracture of many heavily processed composites [2]. However, what remains unanswered is a quantitative understanding of how, in the framework of this model, to partition the beneficial effect of reinforcement homogenization on toughness between possible competing contributions due to variations in crack-tip deflection angle ($\theta$), fracture process length scale ($l$), and the critical strain accumulated over that length scale prior to ligament collapse ($\varepsilon_c$). The answer to this question must be found by an appropriate interpretation of the quantitative fractography results.

**Quantitative Fractography**

The two quantitative trends in fracture surface behavior that emerge coincident with reinforcement homogenization are an increase in average roughness ($R_a$) with increasing toughness (figs 3.39-40) and, over all conditions, a decrease in fracture surface fractal dimension (figs 3.50-52). These two trends can be reconciled with parameters critical to the fracture process when the combined effects of length scale and crack deflection are considered on $R_a$ and $D$. Interpreting the trend of decreasing $D$ with increasing $K_{IV}$ along the lines of the work of Dauskardt et. al.—which showed that fractal dimension was most sensitive to the mean angular deflection—would lead one to conclude that mean angular crack deflection is decreasing with reinforcement homogeneity; a result entirely expected from the work of Faber & Evans, who
predicted that an increasingly homogenous distribution of crack-deflecting particles would lead to a decrease in the mean angle of crack deflection, $\theta$ [4].

Another physical analog for interpreting the fractal dimension of a fracture surface is to use it as a measure of surface area at a particular length scale. From the standard relationship describing the length of a profile as a function of its fractal dimension (and measuring unit) it is possible to determine the lineal roughness parameter $R_L$. Recall, that this parameter is simply the ratio of the total line length normalized by the projected length ($L'$) over which the total profile length is measured, and is given by

$$R_L = \frac{L}{L'} = R_o \eta^{(1-D)}$$

where $\eta$ is the measuring length scale and $R_o$ is a constant of proportionality [3]. From this representation, as fractal dimension increases, the length of the profile, relative to the projected length must increase. Therefore, in the context of the observed trends, at question is how a decreasing profile length ($R_L$) can occur in conjunction with increasing $R_A$. To illustrate this consider the simplified crack path depicted in fig. 4.4, where $R_L = 4d/L'$.

![Figure 4.4: Schematic depiction of the influence of crack deflection on roughness parameters.](image)

If we assume that this simple pattern of deflection repeats *ad infinitum*, the average roughness for this simplified surface segment is given by
\[
R_A = \frac{\text{Area}}{\text{Length}} = 4 \left( \frac{1}{2} A \left( \frac{1}{4} \right) \right) = \frac{1}{2} \frac{A}{d} = \frac{1}{2} d \sin \theta
\]  
(4.5)

while the lineal roughness is given by the relationship

\[
R_L = \frac{\text{Arc Length}}{\text{Length}} = \frac{4d}{L} = \frac{d}{L} \cos \theta
\]  
(4.6)

Notice that \(R_A\) can increase with increasing crack deflection \((\theta)\), however, this will naturally incur a concomitant increase in surface length \((R_L)\), and by analogy, fracture surface area. Observe though that it is possible for average roughness to increase if the feature separation \((d)\) increases while the fracture surface length can still simultaneously decrease as long as the improvement in feature separation overwhelms any increase in the angle of crack deflection. Such a scenario is depicted in fig. 4.5, where \((R_L)_1 = 4d_1/L_1 > (R_L)_2 = 4d_2/L_2\).

\[\text{Figure 4.5: Influence of crack deflection angle and fracture process length scale on fracture roughness.}\]

Consequently, the increase in fracture surface average roughness with increasing toughness is interpreted as a manifestation of the increase in fracture process length scale. Simultaneously, the decrease in fracture surface fractal dimension with increasing toughness is a result of a decrease in mean angle of crack deflection as reinforcement homogeneity is increased.
Fracture Process Length Scale

The fracture process length scale can change in two distinct manners. If fracture progresses simply from nearest neighbor to nearest neighbor, then an increase in nearest neighbor separation clearly must increase the fracture process length scale. Alternatively, if the distribution of particles via homogenization decreases the propensity for particle cracking in the vicinity of the crack tip, this too will increase the fracture process length scale (by decreasing the effective volume fraction of particles participating in fracture). In high strength composites, Majumdar & Pandey have observed a strong propensity for the former over the later, and support these observations with a Weibull-type analysis that demonstrated the probability for particle fracture as a function of near-tip stress normalized by the fracture strength of the reinforcement particle. At the strengths achieved in a 7XXX series composite reinforced with SiC, particle fracture was virtually guaranteed for a large region ahead of the crack-tip [2]. Consequently, it seems reasonable that the increase in the fracture process length scale should track at least identically with the increase in nearest neighbor separation. Therefore, \( l \) in the model of eq. 4.3 can, in this case, be equated with the nearest neighbor separation in the plane of fracture—as determined experimentally via direct measurement—to provide an estimate for the effect of reinforcement homogenization on fracture process length scale.

Crack Deflection Angle

It is possible to determine the angle of crack deflection using the results of Duaskardt et. al. for synthetic fracture surfaces (depicted in fig 4.6) [3]. From the model reported in fig. 4.6, the mean deflection angle is computed for each condition from the appropriate fractal dimension reported in table 3.5 and subsequently reported in table 4.1. From this analysis, it can be seen that there is a perceptible decrease in the mean deflection angle with deformation processing and reinforcement homogenization, regardless of fracture orientation. Although this variation in mean deflection seems small, the gradient in plastic strain ahead of a blunting crack is very severe near the angles reported, and can contribute significantly to the accumulation of plastic strain ahead of the crack-tip.
Figure 4.6: Fractal dimension as a function of crack deflection angle, after Dauskardt et. al [3].

Table 4.1: Fracture surface fractal dimension ($D_F$) and mean crack deflection angle.

<table>
<thead>
<tr>
<th>Fracture Condition</th>
<th>Fractal Dimension, $D_F$</th>
<th>Mean Deflection Angle, $&lt;\theta&gt;$</th>
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<td>1TL</td>
<td>1.412</td>
<td>55.9°</td>
</tr>
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<td>8TL</td>
<td>1.366</td>
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<td>1TL</td>
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<td>55.9°</td>
</tr>
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<td>1.344</td>
<td>49.5°</td>
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Crack Deflection & Plastic Strain

In the Faber & Evans model for crack deflection in a brittle material [4], the intrinsic Mode I fracture toughness of a material is modified by a factor that is a function of the angle of deflection out of plane ($\theta$) to determine an extrinsic (measured) toughness, $K_{ext}$, given by

$$K_{ext} = \frac{K_{int}}{\cos^2(\theta/2)} \quad (4.7)$$

The Faber & Evans model therefore predicts that the extrinsic toughness will increase monotonically with an increase in the angle of out-of-plane deflection, opposite the present results. In contrast, the large displacement analysis of McMeeking [5] shows that the degree of crack deflection is intrinsically related to the rate that plastic strain is accumulated as a function of mouth opening displacement ($\delta$), with greater plastic strains being accumulated at higher angles for the same $\delta$ (fig. 1.20). Therefore, when particle fracture ahead of a blunting crack results in crack-tip deflection angle—due to a more inhomogenous particle arrangement—plastic strain is accumulated faster than it would be at a lower deflection angle, provided the crack retains its Mode I character. The strain required to rupture the ductile ligament between the crack and the fractured particle is subsequently decreased, along with the mouth opening displacement (and load) necessary to advance the crack, ultimately resulting in a reduced toughness.

According to the large displacement analysis of McMeeking, a decrease in deflection angle leads to an increase in the toughness that a material can support and vice versa. If indeed crack deflection is directly correlated with fractal dimension, this would explain the often observed trend of decreasing fractal dimension with increasing toughness for ductile fracture as opposed to the opposite trend observed for brittle fracture [6]. Again, as has been shown separately by Faber & Evans, the origin of this decreased deflection with increasing reinforcement homogeneity is geometrically necessary [4], and can be seen in the subtle increase of four-fold symmetry that occurs in the angular nearest neighbor distribution with increasing deformation processing of the material in this study (fig. 3.17)—with homogenization there does seem to be a theoretical and material-based rationale for a decrease in the angle of deflection for linking cracked particles.
Depicted in figure 4.7 is a comparison of the measured fracture toughness with the predictions of the generalized model of Majumdar & Pandey (eq. 4.3) in which fracture process length scale is computed from nearest neighbor measurements (not volume fraction). This figure illustrates that the model of Majumdar & Pandey is very accurate for homogenous composites, but is increasingly inaccurate for more inhomogeneous composites (those with comparatively small nearest neighbor separations). In their analysis, Majumdar & Pandey assumed that crack deflection occurred exclusively along a 45° inclination; this, combined with an assumption to describe the rupture strain of the interparticle ligament, fixed the parameter $\beta$ in the generalized model of eq. 4.3 [2]. However, as has been measured, there is a strong argument for crack deflection at angles greater than 45° that decreases with reinforcement homogenization, suggesting that these assumptions should be relaxed. By comparing the measured fracture toughness of all conditions with the generalized model (eq. 4.3), it is possible to solve for $\beta$, since all other parameters are known; the results of such an analysis are tabulated in table 4.2.

![Figure 4.7: Comparison of measured toughness ($K_{i\nu}$) and that predicted by the Majumdar & Pandey model.](image-url)
Table 4.2: Measured fracture toughness of each composite by condition, as well as that predicted by the model of Majumdar & Pandey, and the parameter $\beta$ necessary for agreement.

<table>
<thead>
<tr>
<th>Fracture Condition</th>
<th>Measured Fracture Toughness, $K_{Fv}$ (MPa√m)</th>
<th>Toughness, Majumdar &amp; Pandey Model, $K_{IC}$ (MPa√m)</th>
<th>$\beta$ ($\mu$m/$\mu$m)</th>
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</tbody>
</table>

As can be seen in table 4.2, if the contraints of the Majumdar & Pandey model are relaxed, $\beta$ must strongly decrease with reinforcement homogenization. Recall that $\beta$ is a measure of how much a crack-tip can blunt relative to a characteristic dimension of the microstructure (the interparticle separation). By interpolating the results of McMeeking, as is done in fig 4.8, it is possible to gain a greater appreciation for how sensitive this parameter ($\beta$) is to crack deflection angle ($\theta$) and the plastic strain ($\varepsilon_p$) a material can support ($\beta$ for various levels of plastic strain is depicted by semi-solid lines in fig. 4.8). As can be seen, the amount of blunting ($\beta$) necessary to achieve a certain plastic strain strongly depends on the angle of deflection, with less stretch necessary to achieve a given plastic strain at higher angles. Super-imposed on these interpolated $\beta$-contours are the data collected in this study: measured values of $\beta$ as a function of the measured crack deflection angle. The first trend that can be observed from the depicted data is that with reinforcement homogenization, there is a sensible rationale for how crack deflection can affect the amount of crack blunting and subsequently improve material toughness—regardless of orientation. Moreover, reinforcement homogenization also appears to support slightly elevated levels of plastic strain in the interparticle ligament prior to particle rupture.
The absence of any strong variation in fracture behavior between fracture orientations is reasonable given that reinforcement homogenization (an increase in the nearest neighbor separation), though less pronounced along the extrusion axis, is generally uniform. Most of the variation between orientations seems reasonable given the alignment of the reinforcement; this variation would probably be eliminated with equaxed reinforcement. The apparent increase in the plastic strain (going from ~ 4% to ~ 6%) that can be supported in the interparticle ligament with homogenization is congruent with qualitative observations of improved cavity growth near cracked particles (fig 3.34), though the origin of this behavior is not clear. A plausible explanation could be a reduced constraint (and stress triaxiality) near cracked particles by improved particle separation that slows microvoid growth and coalescence, and ultimately delays ligament instability to greater plastic strains. Understanding the contribution of these factors, as well as the general nature cavity of growth from a large fractured particle near a blunting crack-tip, should be the subject of further modeling efforts.

**Figure 4.8:** $\beta$ as a function of mean crack deflection angle $\langle \theta \rangle$ and ligament plastic strain ($\varepsilon_p$) that can be supported prior to rupture for a Mode I crack, interpolated from the analysis of McMeeking [5]
The simplest way to capture the effect of deflection and ligament critical strain on the toughness of a composite with a particular level of reinforcement homogeneity is to use the generalized model of eq. 4.3, but to recognize that there is an effect of microstructural configuration on the parameter $\beta$. This can be captured phenomenologically by comparing $\beta$ with the dimensionless nearest neighbor separation $l/D$ as is done for all fracture orientations studied in fig 4.9; an arbitrary fit of these data is also reported in the fig. 4.9. As can be seen, both transverse orientations generally obey the same fit, and would probably represent what would be expected for a composite reinforced with an equi-axed reinforcement at a 25 vol. % loading, while along the longitudinal orientation (TL), $\beta$ increases at a faster rate with reinforcement homogenization. This anisotropy is most likely a combined result of matrix texture and reinforcement alignment, and should be much more severe with increasing reinforcement aspect ratio, particularly expected would be a reduction in the maximum $\beta$ that could be achieved with homogenization. Again, these variations should be the subject of future work.

![Figure 4.9: Phenomenological models relating $\beta$ to nearest neighbor separation ($l/D$) for all orientations.](image-url)
4.4.) REFERENCES


In this study, deformation processing (extrusion) was used to homogenize reinforcement distribution in a discontinuously reinforced aluminum matrix composite (DRA 6092/SiC/25\%). Reinforcement distribution after three degrees of deformation processing was quantified with the homogenous length scale technique while matrix texture was characterized via X-ray diffraction. Deformation and fracture behavior were characterized in peak-aged material with compression and chevron notch short rod specimens, respectively. Fracture surface average roughness and fractal dimension were quantified. Results of this investigation indicate the following:

1.) Deformation processing is very effective in homogenizing reinforcement distribution in the present discontinuously reinforced aluminum matrix composite system containing 25 vol. % SiC particulate. This homogeneity, as reflected by a decreasing representative volume element with increasing deformation processing is primarily the result of local microstructural rearrangement, particularly, an increase in nearest neighbor separation.

2.) Deformation processing significantly affects particle alignment and the directional homogenous length scale; this must be taken into account before using the homogenous length scale directly as a measure of interparticle separation. A further degree of anisotropy is also induced by extrusion in the form of a strong fiber texture in the matrix with \{111\} and \{100\} components aligning along the extrusion axis. This matrix texture accounts for most of the modest variation in yield strength of the material between extrusions, indicating that the effects of homogenization and reinforcement alignment on yield are negligible. Elastic and strain-hardening behavior are unaffected by these material variations induced by extrusion.

3.) Fracture toughness demonstrates virtually no resistance curve behavior in the peak age condition of this material regardless of the degree of reinforcement homogeneity. This fact permits fracture to be entirely characterized by a single value, $K_{IC}$ and strongly suggests that fracture can be modeled effectively by a highly localized process, specifically, a stress modified critical strain-type model.
4.) Improvements in fracture toughness result from reinforcement homogenization ($K_{IV}$ increases with decreasing size of the representative volume element), regardless of fracture specimen orientation, though the effect is less pronounced in longitudinally oriented specimens. As an upper bound on behavior, fracture toughness seems to improve by nearly a factor of 2 with reinforcement homogenization for crack growth in transverse orientations.

5.) An analysis based solely on the reinforcement volume fraction ($V_f$) cannot account entirely for nearest neighbor separation, or for the effect of deformation induced particle redistribution. Nearest neighbor measurements must be performed and used as inputs into fracture models.

6.) A positive relationship exists between fracture toughness ($K_{IV}$) and average fracture surface roughness ($R_a$) over all conditions. This is a manifestation of an increasing fracture process length scale that scales well with the increase in nearest neighbor separation (as a result of extrusion induced reinforcement homogeneity).

7.) Fracture surfaces are fractal down to a length scale of < 10 $\mu$m, indicating that fracture in all orientation is controlled by a geometrically self-similar, short length-scale process. For each fracture orientation, an inverse correlation exists between fracture toughness and the fractal dimension of specimen fracture surfaces. This behavior is interpreted as a decrease in the degree of crack deflection with increasing reinforcement homogeneity.

8.) Assuming that Mode I character has been maintained during fracture, the decrease in crack deflection contributes strongly to the amount of blunting ($\beta$) that can occur prior to crack advance; this accounts for a large share of the variation in toughness with increased homogeneity while the remainder is attributable to a modest increase in plastic strain that can be accommodated by the interparticle ligament. These effects can be captured by a generalized stress-modified critical strain model in conjunction with a phenomenological model for the parameter $\beta$ in which $\beta$ is strongly sensitive to the degree of reinforcement homogeneity, as measured by the median nearest neighbor separation normalized by the particle diameter, $l/D$. 
APPENDIX A

REINFORCEMENT HOMOGENIZATION IN DRA VIA THE PARTICLE SIZE RATIO TECHNIQUE

In this study, three conditions of “as-extruded” DRA (6061/15.0/SiCp) were examined, subject to identical processing with the exception that each condition was consolidated with a different particle size ratio (PSR) between the initial matrix and reinforcement powders (prior to powder extrusion): 8:1, 3:1, and 2:1. From these three extrusions 17 tensile coupons were tested; 6 each from the 8:1 and 3:1 conditions, and 5 from the 2:1 condition. The resulting flow data are illustrated in fig. A.1. It can be seen that plastic strain data tend to fall into distinct bands (depicted by color) based on the condition of the tested material. Generally, flow stress increases with decreasing matrix/reinforcement PSR. It was found that a Ramberg-Osgood model described the plastic strain data very well, and the results of the fit of the collected data to such a model are reported in table A.1, including the average values of yield strength ($\sigma$), elastic modulus ($E$), strain hardening exponent ($n$), and elongation to failure ($\varepsilon_f$). In general, yield strength, ductility, and elastic modulus in the as-extruded condition are all sensitive to the particle size ratio used during composite consolidation, all quantities seeming to improve positively with increasing reinforcement homogeneity (decreasing PSR), although the sensitivity of strain hardening exponent to PSR is statistically indeterminable.

Table A.1: Influence of initial particle size ratio (PSR) on parameters of mechanical behavior.

<table>
<thead>
<tr>
<th>Condition PSR</th>
<th>Modulus, $&lt;E&gt;$ (GPa)</th>
<th>Yield Strength, $&lt;\sigma&gt;$ (MPa)</th>
<th>Hardening Exponent, $&lt;n&gt;$</th>
<th>Ductility, $&lt;\varepsilon_f&gt;$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2:1</td>
<td>127.6 ± 4.5</td>
<td>151.9 ± 1.0</td>
<td>0.15 ± 0.1</td>
<td>9.2</td>
</tr>
<tr>
<td>3:1</td>
<td>121.5 ± 1.5</td>
<td>142.2 ± 2.4</td>
<td>0.13 ± 0.1</td>
<td>7.9</td>
</tr>
<tr>
<td>8:1</td>
<td>88.0 ± 5.8</td>
<td>124.4 ± 7.4</td>
<td>0.13 ± 0.1</td>
<td>7.5</td>
</tr>
</tbody>
</table>
In an effort to determine the effect of reinforcement distribution on strain criteria for damage, metallographic sections transverse to the fracture plane (along the strain gradient) were prepared for each condition. It was observed that during metallographic polishing of each material condition, large sections of clustered SiC seemed easily removed (as depicted in figs. A.2-4), while during further examination in the SEM—even great distances away from the fracture surface (outside of any strain gradient), material in particle clusters appeared undensified (fig. A.5). This suggests that there may be an effect of particle clustering via this processing route on overall density.

Figure A.1: Plastic strain response for various PSR conditions of an Al 6061/15.0/SiCp.
Figure A.2: Microstructure of a 2:1 PSR DRA (Al-6061/25% SiC$_p$) (125x)—low porosity.

Figure A.3: Microstructure of a 2:1 PSR DRA (Al-6061/25% SiC$_p$) (125x)—intermediate porosity.

Figure A.4: Microstructure of a 8:1 PSR DRA (Al-6061/25% SiC$_p$) (125x)—high porosity.
Figure A.5: Undensified particle cluster away from the fracture surface.

Since the grip section of each condition experienced little or no strain, these regions provide an excellent site from which it is possible to determine the initial density of the composite prior to deformation. Using the Archimedes method, the density of each condition was determined and compared to a theoretical “full” density of 2.90 g/cm³, as determined by the rule of mixtures, these results are reported in table A.2; the error of these measurements is less than ±1%.

Table A.2: Material density and ductility by PSR condition.

<table>
<thead>
<tr>
<th>Condition (PSR)</th>
<th>Density, ρ (g/cm³)</th>
<th>Density, ρ (% max.)</th>
<th>Porosity, ξ (%)</th>
<th>Ductility, &lt;εf&gt; (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2:1</td>
<td>2.87</td>
<td>99.0</td>
<td>1.0</td>
<td>9.2</td>
</tr>
<tr>
<td>3:1</td>
<td>2.79</td>
<td>96.0</td>
<td>4.0</td>
<td>7.9</td>
</tr>
<tr>
<td>8:1</td>
<td>2.73</td>
<td>94.3</td>
<td>5.7</td>
<td>7.5</td>
</tr>
</tbody>
</table>
Clearly, there is a systematic increase in density with decreasing PSR. Though the maximum variation in density between conditions is only on the order of 4.7%, this would explain well the variation in mechanical properties observed since virtually all mechanical properties are very sensitive to the overall level of porosity, particularly as a material approaches full density ($\rho > 92\%$). Take for example, ductility; in P/M systems, for which German has suggested the following model [German]:

$$Z = \frac{(1 - \xi)^{3/2}}{(1 + C\xi^2)^{1/2}}$$

(1)

where $Z$ is the relative ductility (ductility of a P/M component normalized to that of a wrought material), $\xi$ is the porosity ($= 1 - \rho$), and $C$ is an empirical constant. In P/M steels, $C$ can vary from 100 to $10^5$. This model for several values of $C$ is depicted in fig. 6.

Figure A.6: A model describing the effect of porosity on relative tensile elongation [German].

For comparison, a fit of porosity and elongation data in this study is presented for the composites studied in fig. 7.
Unfortunately it seems that this PSR processing route introduces porosity that scales with the degree of clustering, an undesirable affect since it will presumably mask any impact of particle distribution on mechanical behavior.
This is a Mathematica 5.2 script for determining particle distribution statistics from data files obtained via Clemmex microstructural imaging software. The input data file consists of an array of information for each particle (row). Particle Size, Aspect Ratio, and x centroid, y centroid, and major axis orientation are consecutively delineated in each column.

<<Graphics'Graphics'
<<Statistics'ContinuousDistributions'

Clear[pt, DM, dislist, dislisto, Mindis1, Angle, Minang1, Mindeltax, Mindeltay, Psize, PAngle, Assratio, MindisoverPsize];
pt = Import["D:\temp\Clemmex Data\1CL-coord-N.txt", "TSV"];

index = Dimensions[pt][[1]]
deltax = Table[0, {i, 1, 1}, {j, 1, index}];
deltay = Table[0, {i, 1, 1}, {j, 1, index}];
DM = {};
PAngle = {};
Mindis1 = {};
Minang1 = {};
Mindeltax = {};
Mindeltay = {};
Psize = {};
Assratio = {};
MindisoverPsize = {};

Clear[deltax, deltay];
For[i = 1, i <= index, i++, (deltax = pt[[All, 1]] - pt[[i, 1]];
deltay = pt[[All, 2]] - pt[[i, 2]];
dislist = Sort[Sqrt[(deltax)^2 + (deltay)^2]];
dislisto = Ordering[Sqrt[(deltax)^2 + (deltay)^2]];
Mindis1 = Append[Mindis1, dislist[[2]]];
Angle = ArcTan[deltay[[dislisto[[2]]]], deltay[[dislisto[[2]]]]]*180/3.1415927;
If[deltax[[dislisto[[2]]]] == 0, (Angle = 89.9; deltay[[dislisto[[2]]]] = 0.1)];
If[deltay[[dislisto[[2]]]] == 0, (Angle = 0.1; deltay[[dislisto[[2]]]] = 0.1)];
Minang1 = Append[Minang1, Angle];
Mindeltax = Append[Mindeltax, deltay[[dislisto[[2]]]]];
Mindeltay = Append[Mindeltay, deltay[[dislisto[[2]]]]];
Psize = Append[Psize, pt[[dislisto[[2]], 3]]];
PAngle = Append[PAngle, pt[[dislisto[[2]], 5]]];
Assratio = Append[Assratio, pt[[i, 4]]];
MindisoverPsize = Append[MindisoverPsize, Mindis1[[i]]/Psize[[i]]];
)];

PAngle2 = PAngle;
Minang2 = Minang1;
For[i = 1, i <= index, i++, (If[PAngle2[[i]] > 90, PAngle2[[i]] = (180 - PAngle2[[i]])];

APPENDIX B

SCRIPT FOR DETERMINING REINFORCEMENT DISTRIBUTION STATISTICS FROM RAW DATA

This is a Mathematica 5.2 script for determining particle distribution statistics from data files obtained via Clemmex microstructural imaging software. The input data file consists of an array of information for each particle (row). Particle Size, Aspect Ratio, and x centroid, y centroid, and major axis orientation are consecutively delineated in each column.
If[Minang2[[i]]<0, Minang2[[i]]=(-1*Minang2[[i]])];
If[Minang2[[i]]>90, Minang2[[i]]=(180-Minang2[[i]])];

Clear[loverdx,loverdy];
minora=2*(Psize/2)*Sqrt[1/Assratio];
majorb=2*(Psize/2)*Sqrt[Assratio];
loverdx=2*Abs[Mindeltax]/majorb;
loverdy=2*Abs[Mindeltay]/minora;

(* Compute Distributions *)

Off[FindRoot::lstol];
dist = GammaDistribution[alpha, beta];

RandomSampleWR[lis_List,num_]:=Module[{len},
   len = Length[lis];
   newlis = Table[Part[lis, Random[Integer, {1, len}]], {num}]
]

data=RandomSampleWR[Psize,2500];
Print["Psize"];llhf = Tr[Log[PDF[dist,#]]& /@ data];
est = Solve[{ Mean[dist]==Mean[data], Variance[dist]==VarianceMLE[data]}, {alpha,beta}][[1]];soln2=FindRoot[ Evaluate[{D[llhf,alpha]==0, D[llhf,beta]==0}], {alpha,{.975alpha,1.025alpha}/.est},{beta,{.975beta,1.025beta}/.est}];Psizesoln2=soln2

data=RandomSampleWR[Assratio,2500];
Print["Assratio"];llhf = Tr[Log[PDF[dist,#]]& /@ data];
est = Solve[{ Mean[dist]==Mean[data], Variance[dist]==VarianceMLE[data]}, {alpha,beta}][[1]];soln2=FindRoot[ Evaluate[{D[llhf,alpha]==0, D[llhf,beta]==0}], {alpha,{.975alpha,1.025alpha}/.est},{beta,{.975beta,1.025beta}/.est}];Assratiosoln2=soln2

data=RandomSampleWR[MindisoverPsize,2500];
Print["MindisoverPsize"];llhf = Tr[Log[PDF[dist,#]]& /@ data];
est = Solve[{ Mean[dist]==Mean[data], Variance[dist]==VarianceMLE[data]}, {alpha,beta}][[1]];Mindis1soln2=FindRoot[ Evaluate[{D[llhf,alpha]==0, D[llhf,beta]==0}], {alpha,{.975alpha,1.025alpha}/.est},{beta,{.975beta,1.025beta}/.est}];

(* Plot Distributions *)

DisplayTogether[Histogram[Psize, HistogramCategories->Table[1*i,{i,0,10}], Ticks->IntervalBoundaries,HistogramScale->1],
Plot[PDF[GammaDistribution[Psizesoln2[[1]][[2]][[1]],Psizesoln2[[2]][[2]][[1]]],x], {x,0,10},PlotRange->{0,1},AxesOrigin->{0,0}];

Print["Particle Size (Gamma ) ", " ",Mean[GammaDistribution[Psizesoln2[[1]][[2]][[1]],Psizesoln2[[2]][[2]][[1]]]],"," ",StandardDeviation[GammaDistribution[Psizesoln2[[1]][[2]][[1]],Psizesoln2[[2]][[2]][[1]]]]]
DisplayTogether[Histogram[Assratio, HistogramCategories->Table[1*i,{i,0,10}], Ticks->IntervalBoundaries,HistogramScale->1],Plot[PDF[GammaDistribution[Assratiosoln2[[1]][[2]][[1]],Assratiosoln2[[2]][[2]][[1]],x], {x,0,10}],PlotRange->0,1,AxesOrigin->{0,0}; 
Print["Aspect Ratio (Gamma) ", " " 
","Mean[GammaDistribution[Assratiosoln2[[1]][[2]][[1]],Assratiosoln2[[2]][[2]][[1]]]] ","StandardDeviation[GammaDistribution[Assratiosoln2[[1]][[2]][[1]],Assratiosoln2[[2]][[2]][[1]]]] 
DisplayTogether[Histogram[MindisoverPsize, HistogramCategories->Table[1*i,{i,0,10}], Ticks->IntervalBoundaries,HistogramScale->1],Plot[PDF[GammaDistribution[Mindis1soln2[[1]][[2]][[1]],Mindis1soln2[[2]][[2]][[1]],x], {x,0,10}],PlotRange->0,1,AxesOrigin->{0,0}; 
Print["Distance (Gamma) ", " " 
","Mean[GammaDistribution[Mindis1soln2[[1]][[2]][[1]],Mindis1soln2[[2]][[2]][[1]]]] ","StandardDeviation[GammaDistribution[Mindis1soln2[[1]][[2]][[1]],Mindis1soln2[[2]][[2]][[1]]]] 
DisplayTogether[Histogram[PAngle2, HistogramCategories->Table[10*i,{i,0,9}], Ticks->IntervalBoundaries,HistogramScale->10],PlotRange->0,1,AxesOrigin->{0,0}; 
DisplayTogether[Histogram[Minang2, HistogramCategories->Table[10*i,{i,0,9}], Ticks->IntervalBoundaries,HistogramScale->10],PlotRange->0,1,AxesOrigin->{0,0}; PsAssNNSOriNNA=Transpose[{Psize, Assratio,MindisoverPsize,PAngle2,Minang2}]; Export["D:\temp\Clemmex Data\1CL-PsAssNNSOriNNA.csv", PsAssNNSOriNNA];
SAMPLE OUTPUT

The primary output of this script are histograms and gamma distribution fit data for particle size, particle aspect ratio, nearest neighbor separation (centroid-to-centroid spacing/particle size), particle orientation, and a distribution of nearest neighbor disclination.
APPENDIX C

SCRIPT FOR DETERMINATION OF $L_{H,N}$ FROM A FIELD OF PARTICLES

This is a script that implements (in *Mathematica* 5.2) the MSAAF (multi-scale analysis of area fractions) technique and its anisotropic counterpart on a digitized optical image of a DRA microstructure in order to generate plots of volume fraction variation as a function of length scale. From these plots, the homogenous length scale and anisotropic homogenous length scale can be obtained—in pixels.

(* Load relevant libraries and initialize pertinent variables *)

<<Graphics'
<<Statistics`DiscreteDistributions'
<<LinearAlgebra`MatrixManipulation'
Clear[infile, Micropic, Micropic1, Micropic2, ResStats];

(* Input the path of the starting image into the statement in the expression for 'infile'. Note that the picture must be square and of a resolution which is an integral power of 2. *)

infile:= Import["D:\My Documents\Thesis Work\Homogenous Length Scale Pics 2\CL-26-4-gs-t.tif"]

(* Use this commented command below to determine the structure of the data file, and locate the matrix representation of the image (gray levels) in the data structure, currently, this is setup to strip data from an 8-bit TIFF file. Subsequently store the matrix representation in Micropic. *)

(* Shallow[InputForm[infilea]]; *)

Micropic=infile[[1,1]];  
Micropic1=Micropic;  
Micropic2=Transpose[Micropic];

ImagePower=1/Log[Dimensions[Micropic][[1]],2];
ResStats = Table[0,{i,1,ImagePower},{j,1,9}];
For[i=1,i≤ImagePower, i++, (  
plot1=ListDensityPlot[Micropic/255,Mesh→False,DisplayFunction→Identity];  
plot2=ListDensityPlot[Micropic1/255,Mesh→False,DisplayFunction→Identity];  
plot3=ListDensityPlot[Transpose[Micropic2/255],Mesh→False,DisplayFunction→Identity];  
Show[GraphicsArray[{plot1,plot2,plot3}], ImageSize→{1024,256},DisplayFunction→$DisplayFunction];
  (* Gather Statistics *)
  data=Flatten[Micropic];
data1=Flatten[Micropic1];
data2=Flatten[Micropic2];
  ResStats[[i,1]]=Dimensions[Micropic][[1]];  
  ResStats[[i,2]]=N[StandardDeviation[data]];  
  ResStats[[i,3]]=N[1-Mean[data]];  
)
ResStats[[i,4]] = N[2^ImagePower/Dimensions[Micropic][[1]]];
ResStats[[i,5]] = (ResStats[[i,2]]/ResStats[[i,3]]);
ResStats[[i,6]] = N[StandardDeviation[data1]];
ResStats[[i,7]] = (ResStats[[i,6]]/ResStats[[i,3]]);
ResStats[[i,8]] = N[StandardDeviation[data2]];
ResStats[[i,9]] = (ResStats[[i,8]]/ResStats[[i,3]]);

(* "Reduce" the image and save it in the upper quadrant/half of Micropic *)

For[m = 1, m ≤ ResStats[[i,1]]/2, m++,
    For[n = 1, n ≤ ResStats[[i,1]]/2, n++,
        Micropic[[m,n]] = (Micropic[[2*m-1,2*n-1]] + Micropic[[2*m,2*n-1]] +
                             Micropic[[2*m-1,2*n]] + Micropic[[2*m,2*n]])/4;
    ];
]

For[m = 1, m ≤ ResStats[[1,1]], m++,
    For[n = 1, n ≤ ResStats[[i,1]]/2, n++,
        Micropic1[[m,n]] = (Micropic1[[m,2*n-1]] + Micropic1[[m,2*n]])/2;
        Micropic2[[m,n]] = (Micropic2[[m,2*n-1]] + Micropic2[[m,2*n]])/2;
    ];
]

(* Pare off the extraneous quadrants from Micropic *)

Micropic = SubMatrix[Micropic, {1, 1}, {ResStats[[i,1]]/2, ResStats[[i,1]]/2}];
Micropic1 = SubMatrix[Micropic1, {1, 1}, {ResStats[[1,1]], ResStats[[i,1]]/2}];
Micropic2 = SubMatrix[Micropic2, {1, 1}, {ResStats[[1,1]], ResStats[[i,1]]/2}];

)

ResStats//MatrixForm
plot4 = LogLogListPlot[ResStats[[All, {4, 5}]], PlotStyle -> PointSize[0.0175], DisplayFunction -> Identity];
plot5 = LogLogListPlot[ResStats[[All, {4, 7}]], PlotStyle -> PointSize[0.0175], DisplayFunction -> Identity];
plot6 = LogLogListPlot[ResStats[[All, {4, 9}]], PlotStyle -> PointSize[0.0175], DisplayFunction -> Identity];
Show[plot4, DisplayFunction -> $DisplayFunction, ImageSize -> {512, 256}];
Show[plot5, plot6, DisplayFunction -> $DisplayFunction, ImageSize -> {512, 256}];

SAMPLE OUTPUT

The output of this routine is a series of images that subdivide the volume fraction of the original image at increasingly large length scales to determine the isotropic and anisotropic homogenous length scales. Subsequently, a table consisting of rows corresponding to each of the previous rows of images and columns of several important statistics. Each column, respectively, contains “image/subimage size (pixels)”, “standard deviation in volume fraction of all isotropic quilting units”, “volume fraction of dark phase”, “quilt edge length/strip length”, “standard deviation in volume fraction for each strip in the horizontal direction”, “previous column normalized by volume fraction”, “standard deviation in volume fraction for each strip in the vertical direction”, “previous column normalized by volume fraction”. This is then followed by an isotropic MSAAF plot, then and anisotropic MSAAF plot.
APPENDIX D

SCRIPT FOR THE FRACTAL ANALYSIS OF FRACTURE SURFACE TOPOGRAPHY DATA

This is a Mathematica 5.2 script that extracts fracture surface profiles from fracture surface topographic data sets, interpolates missing data, and conducts the variational covering method of Dubuc & Tricot on each extracted profile to measure the fractal dimension their respective fractal dimension.

(* Fractal Dimension *)

(* Load Data *)

(* This block imports the region of interest obtained from a data file created by the Veeco software into the variable infilea; this data is in the form of an array of height values. A second data file—infilex—is imported which contains a 1-dimensional array of the fixed lateral coordinates of the points in infilea. *)

<<Graphics`MultipleListPlot`

Clear[infilea, infilex];
infilea := OpenRead"F:\Roughness Small Regions\1CLA-Interpolate.txt";
infilex := OpenRead"F:\Roughness Small Regions\profilexvals.txt";
ind = newarray/10;
xs = Import[infilex,"List"];

(* Data Preprocessing *)

(* This block initializes several variables *)

numprofiles = 20;
width = Part[Dimensions[ind], 1];
proindex = 1;
lenx = Part[Dimensions[xs], 1];
For[l = 1, l < lenx, l = l + 1, (xs = Insert[xs, (Part[xs, 2*l - 1] + Part[xs, 2*l])/2, 2*l])];
profilearray = {xs};

For[k = 1, k <= numprofiles, k++, ( (* This block extracts a profile from the dataset and ensures that endpoints are not missing/null datapoints *)

profile = Part[ind[[{proindex}]], 1];
len = Part[Dimensions[profile], 1];
While[! (NumberQ[Part[profile, 1]] && NumberQ[Part[profile, len]]), ( proindex++;
profile = Part[ind[[{proindex}]], 1];
len = Part[Dimensions[profile], 1];
)];

Print[proindex];

(* Internal datapoints of a profile are interpolated if "Bad" *)

For[i = 1, i <= len, i++, (
If[NumberQ[Part[profile, i]] == False, (a = i - 1;
    For[b = i, NumberQ[Part[profile, b]] == False, b++;
    For[j = i, j < b, j++, (holder = (Part[profile, b] - Part[profile, a])*(j - a)/(b - a) + Part[profile, a];
       profile = ReplacePart[profile, holder, j];
     );]
  );
)
(* Doubles the number of points via interpolation, stores the profile in an array of extracted profiles *)
For[l = 1, l < lenx, l = l + 1, (profile = Insert[profile, (Part[profile, 2*l - 1] + Part[profile, 2*l])/2, 2*l])];
ListPlot[profile];
profilearray = Append[profilearray, profile];
proindex = proindex + IntegerPart[width/(numprofiles)];
)
(* Algorithm will not function correctly if values of k progress too rapidly, k_n < 2 k_{n-1} *)
k = {1, 1, 1, 2, 4, 6, 8, 12, 16, 20, 24, 28, 32, 40, 48, 56, 64, 80, 96, 112, 128, 160, 192, 224, 256, 320, 384, 448, 512, 640};
imax = Part[Dimensions[k], 2]; (* # yardstick lengths *)
nmax = Part[Dimensions[xs], 1]; (* # points in profile *)
kcoeff = profilearray[[1, 2]] - profilearray[[1, 1]]; (* pixel length *)
R = 640; (* Variational Parameter, R *)
lldata = SparseArray[{}, {imax, 1 + numprofiles}]; (* array for log - log data *)
For [p = 1, p <= numprofiles, p++,
    Clear[u, v, uinit, vinit, utemp, vtemp, uold, uold];
    (* Read data/profile into the last, unused, column of u and v, in the domain of nmax + 1 to 2*nmax *)
    Timea = SessionTime[];
    (* Extract profile from profilearray *)
    uinit = Part[profilearray[[1 + p]], 1];
    vinit = Part[profilearray[[1 + p]], 1];
    ListPlot[uinit];
    (* Zero - pad profile *)
    uinit = PadLeft[PadRight[uinit, 2*nmax], 3*nmax];
    vinit = PadLeft[PadRight[vinit, 2*nmax], 3*nmax];
    (* Mirror data about nmax + 1 and 2*nmax so that endpoints do not cause errors in fractal measurement *)
    For [n = 1, n <= nmax, (n++),
        uinit = ReplacePart[uinit, Part[uinit, nmax + n], nmax - n + 1];
        vinit = ReplacePart[vinit, Part[vinit, nmax + n], nmax - n + 1];
        uinit = ReplacePart[uinit, Part[uinit, 2*nmax + 1 - n], 2*nmax + n];
        vinit = ReplacePart[vinit, Part[vinit, 2*nmax + 1 - n], 2*nmax + n];
    );
);
Timeb = SessionTime[];

(* Main Routine *)

(* Compute u1 and v1, the upper and lower bound for the smallest length scale *)

utemp = uinit;
vtemp = vinit;

δ = k[[1, 1]];
For[n = (nmax + 1) - k[[1, imax]], n ≤ (nmax + 1 + R) + k[[1, imax]], n++, (  
  If[Part[uinit, n - δ] > Part[uinit, n + δ], utemp = ReplacePart[utemp, Part[uinit, n - δ], n],  
    utemp = ReplacePart[utemp, Part[uinit, n + δ], n]];  
  If[Part[vinit, n - δ] < Part[vinit, n + δ], vtemp = ReplacePart[vtemp, Part[vinit, n - δ], n],  
    vtemp = ReplacePart[vtemp, Part[vinit, n + δ], n]];  
)];

u = {utemp};
v = {vtemp};

(* Calculate ui and vi, the upper and lower bounds are computed at all lengths k, using data from the previous k *)

For[i = 2, i ≤ imax, i++, (  
  uold = utemp;
  vold = vtemp;
  Timec[i] = SessionTime[];
  δ = k[[1, i]] - k[[1, i - 1]];
  For[n = (nmax + 1) - k[[1, imax]], n ≤ (nmax + 1 + R) + k[[1, imax]], n++, (    
    If[Part[uold, n - δ] > Part[uold, n + δ], utemp = ReplacePart[uold, Part[uold, n - δ], n],    
      utemp = ReplacePart[uold, Part[uold, n + δ], n]];    
    If[Part[vold, n - δ] < Part[vold, n + δ], vtemp = ReplacePart[vold, Part[vold, n - δ], n],    
      vtemp = ReplacePart[vold, Part[vold, n + δ], n]];    
  )];
  u = Append[u, utemp];
  v = Append[v, vtemp];
  Print[p, ", < >", k[[1, i]]];
  Timed[i] = SessionTime[];
)];

(* Compute ui - vi, the difference between the lower and upper bound, then compute log-log data for all k*)

For[i = 4, i ≤ imax, i++, (  
  lldata[i, 1] = Log[10, (k[[1, i]]*kcoeff)];  
  lldata[i, p + 1] = 0;  
  For[n = nmax + 1, n ≤ (nmax + 1 + R), n++, (    
  )];  
)
lldata[[i, p + 1]] = lldata[[i, p + 1]] + (u[[i, n]] - v[[i, n]]);

lldata[[i, p + 1]] = Log[10, lldata[[i, p + 1]]*(1/(k[[1, i]]*kcoeff)^2)];

plotdata = Part[Part[Reap[For[i = 4, i ≤ imax, (i++), Sow[ Part[Part[Reap[Sow[lldata[[i, 1]]];Sow[lldata[[i, 2]]]], 2], 1]]]], 2], 1];

outfile = OpenWrite["F:\Roughness Small Regions\1CLA-R640.txt"]; Export[outfile, lldata, "CSV"]; Close[outfile];

(* This short block can be used to observe the bounds on various profiles storred in u and v *)

Clear[upper, lower];
upper = {}; lower = {}; testplot = {};
For[o = 1, o ≤ 1 + R, o++, (upper = Append[upper, u[[20, nmax + o]]]; lower = Append[lower, v[[20, nmax + o]]]; testplot = Append[testplot, u[[1, nmax + o]]]; )]

MultipleListPlot[upper, lower, testplot, PlotJoined -> {True, True, True}, SymbolShape -> {PlotSymbol[Triangle], PlotSymbol[Triangle], None}]

(* This block creates a convenient matrix of log-log data averaged over all profiles with error bars that is then plotted and exported *)

Clear[derivativearray];
derivativearray = SparseArray[{}, {imax, 7}];
For[p = 4, p ≤ imax, p++, (derivativearray[[p, 1]] = lldata[[p, 1]]; llfrac = {}; llfdim = {}; For[g = 1, g ≤ numprofiles, g++, (llfrac = Append[llfrac, 10^lldata[[p, 1 + g]]]; llfdim = Append[llfdim, (lldata[[p, 1 + g]] - lldata[[p - 1, 1 + g]])/(derivativearray[[p, 1]] - derivativearray[[p - 1, 1]])]; derivativearray[[p, 2]] = Log[10, Mean[llfrac]]; derivativearray[[p, 4]] = Abs[(1/(10^derivativearray[[p, 2]]*Log[10]))*StandardDeviation[llfrac]]; derivativearray[[p, 6]] = Mean[llfdim]; derivativearray[[p, 7]] = StandardDeviation[llfdim]; )];
lplot1 = {}; lplot2 = {}; lplot3 = {};
For[p = 5, p ≤ imax, p++, (derivativearray[[p, 3]] = (derivativearray[[p, 2]] - derivativearray[[p - 1, 2]])/(derivativearray[[p, 1]] - derivativearray[[p - 1, 1]]); derivativearray[[p, 5]] = (1/(derivativearray[[p, 1]] - derivativearray[[p - 1, 1]])*((Abs[1 - derivativearray[[p - 1, 2]]] - derivativearray[[p, 4]]) + (Abs[derivativearray[[p, 2]] - 1]*derivativearray[[p - 1, 4]])); lplot1 = Append[lplot1, {derivativearray[[p, 1]], derivativearray[[p, 2]], ErrorBar[derivativearray[[p, 4]]]}];

189
The principle output of this code is a series of images of the profiles extracted from the surface topography data, and a plot of the resulting average measurements over all profiles, as well as a data file containing this plot data.
Garth Barrett Wilks was born in Pittsburgh, Pennsylvania on February 13th, 1980 to his parents Denise and Benjamin Wilks. After the passing of Denise, Garth was raised by his father, an engineer, in Pennsylvania, Virginia, North Carolina, and Ohio. Garth attended the Pennsylvania State University where he took separate B.S. degrees with honors in Physics and Engineering Science in August of 2000 and an M. S. in Engineering Mechanics in August of 2003. At that time he joined the research group of Professor Donald Koss and began his dissertation work in collaboration with the Metals Development group of the Air Force Research Laboratory’s Materials & Manufacturing Directorate at Wright-Patterson Air Force Base in Ohio. Garth completed the requirements for the degree of Doctor of Philosophy in November of 2007, and has accepted a post-doctoral position with AFRL.