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Multi-Scale Approach to Investigate the Tensile and Fracture Behavior of Nano Composite Materials

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This report covers results addressing multi-scale measurements of deformation, strain, and failure behavior in particulate composites containing nano size particles. In addition, techniques are developed, based on multi-scale modeling approaches, to model particles interaction, damage initiation and evolution processes, and constitutive and crack growth behavior in particulate composites. The program's basic approach involves a blend of numerical and experimental studies. The results of these studies are evaluated and discussed.

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NOTICE

FOREWORD

This Final technical report, entitled “Multi-Scale Approach to Investigate the Tensile and Fracture Behavior of Nano Composite Materials,” presents the results of an in-house study performed under JON 23020378 by AFRL/PRSM, Edwards AFB CA. The Principal Investigator/ Project Manager for the Air Force Research Laboratory was Dr. Chi T. Liu.

This report has been reviewed and is approved for release and distribution in accordance with the distribution statement on the cover and on the SF Form 298.

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GLOSSARY

AL	aluminum
AN	ammonia nitrate
CASI	Computer Aided Speckle Interferometry
MPa	mega pascal
SIEM	Speckle Interferometry with Electron Microscopy
TPEG	Tetrahydrofuran-Polyethylene Glycol
VFP	volume fraction of particle
Vo	initial volume
ΔV	change of volume

1.0 INTRODUCTION

This program was concerned with the effects of nano-size particles on the tensile and fracture behavior of particulate composite materials. The program's basic approach involved a blend of experimental and analytical studies. In general, mechanisms and mechanics involved in the damage process and cohesive fracture were emphasized. Special issues that were addressed are: (1) To what extent and by what mechanism do the nano-size particles affect damage initiation and evolution processes, deformation process, and crack growth behavior? (2) What are the deformation and failure mechanisms on the meso and the macro levels? (3) What is the role of microstructure on damage initiation and evolution processes? and (4) How to model microstructure effects on damage process and crack growth behavior in a particulate composite.

2.0 OBJECTIVES

The objectives of the proposed research were to (1) obtain a fundamental understanding of the effects of nano-size aluminum particles on the constitutive and crack growth behavior of particulate composite materials; (2) investigate the effects of aluminum particle size on deformation mechanism and fracture strength under a constant strain rate condition; (3) determine the deformation and failure mechanisms on meso and macro scales; and (4) develop a numerical modeling technique to model damage process and crack growth behavior including microstructural effects.

In this study, there were three major tasks: (1) Task 1 – meso and macro scale strain measurements and damage analysis; (2) cumulative damage analysis; and (3) multi-scale modeling on damage initiation and evolution.

3.0 SUMMARY OF ACCOMPLISHMENTS

3.1 Task 1 – Meso and Macro Scale Strain Measurements and Damage Analysis

In the first phase of this program, the deformation and failure mechanisms in two matrix materials [Solithane 113 and Tetrahydrofuran-Polyethylene Glycol (TPEG)] were investigated (1-2). Uniaxial tensile specimens were made from the two materials. Tests were performed in a Hitachi scanning electron microscope (model S-2460N), which is equipped with a displacement controlled loading device with a crosshead that can travel continuously. The application of Speckle Interferometry with Electron Microscopy (SIEM) consisted of three procedures: the creation of micro/nano speckles on the specimen surface; recording and digitization of the speckle patterns before and after specimen deformation; and deformation analysis of the speckle patterns using an efficient program called CASI (Computer Aided Speckle Interferometry).

Experimental findings revealed that the strain distributions varied with the size of the area, A, in which the data were analyzed. The area, $A_{u,}$ in which the strain distributions were relatively uniform, or the material's microstructure had no significant effect on the strain distributions, varied with the materials investigated. The sizes of A_u for Solithane 113 and TEPG were 1.04 mm^2 and 0.1 mm^2, respectively.

These experimental observations revealed that there exists a length scale below which the material's microstructure has a significant effect on the strain distributions. In other words, a representative area, which is defined as an area in which the material's microstructure has no significant effect on the strain distribution, exists for the materials investigated in this study. It is interesting to point out that for the two materials investigated, the normal strain distributions measured in a small area of 0.002 mm^2 at 1500X were highly nonuniform, and both tensile and compressive strain fields existed. In addition, for Solithane 113, positive transverse strains were developed at certain locations in the materials. The triaxial tensile strain fields were the potential failure locations in the materials.

In addition to determining the strain fields, the damage mechanisms near the crack tip were also investigated. Although the deformation mechanisms, large displacement and ligament formation near the crack tip of the three materials were similar, the damage mechanisms near the crack tip depended on the material. For Solithane 113, a highly damaged region was developed at the crack tip. Inside the damage region, through-thickness voids were formed. The crack growth mechanism involved void formation ahead of the crack tip and the coalescence of the main crack tip with the void. For TPEG, no through-thickness voids were formed near the crack tip. Instead, microcracks were formed in the ligaments. The growth of the microcrack resulted in the fracture of the ligament.

In the second phase of this study, the deformation and failure mechanisms in two composite materials (TPEG with 10% by weight of 6 micron aluminum particles, denoted as Composite 1, and TPEG with 10% by weight of 0.2 micron aluminum particles, denoted as Composite 2) were investigated under a constant strain rate condition (3-4). In addition, crack propagation tests as well as cyclic loading and constant strain tests were conducted on specimens made of the two composite materials.

Experimental findings revealed that the normal strain distributions measured in a small area of 0.002 mm^2 at 1500X were highly nonuniform, and both tensile and compressive strain fields existed. In addition, positive transverse strains were developed at certain locations in the materials. The representative areas for the two composite materials were 0.74 mm^2.

The results of crack growth analysis showed that the crack grew faster in Composite 2 than in Composite 1. Experimental data showed that the critical load for the onset of crack growth for Composite 2 was higher than that for Composite 1. The higher crack growth rate observed in Composite 2 was mainly due to the increase in the relaxation modulus and the available elastic energy for crack propagation.

In the third phase of this study, the deformation and failure mechanisms in two composite materials were investigated (TPEG with 20% by weight of 6 micron aluminum particles and 10% by weight of 40 micron AN particles, and TPEG with 20% by weight of 0.2 micron aluminum particles and 10% by weight of 40 micron AN particles). Experimental findings revealed that the deformation mechanisms, large displacement and ligament formation, and the failure mechanisms, voids formation and elongation around the particles of the two materials were similar.

3.2 Task 2 – Cumulative Damage Analyses

Throughout the loading history, the progressive development and interaction of various damage modes changed the state of the material, or the mechanical response of the material. In general, when the particulate composite material is tested under a constant strain rate condition, the initial linear portion of the stress-strain curve is associated with a stretching on undamaged material, with the filler particles bonded to the binder. As the external load is continuously increased, at a certain critical stress level, dewetting occurs. When the density of the dewetted particles reaches a critical value, the rigidity of the material is thereby reduced, and usually this critical dewetting state coincides with the transition from linear response to nonlinear behavior. As the specimen is continuously stretched, the number of dewetted particles is increased, and the formed voids start to grow and coalesce. This damage process is related, primarily, to the nonlinear response of the material, and it can be characterized by bulk volume change during stretching. The bulk volume change during straining is usually known as the strain dilatation, which is partially caused by the nucleation of new voids, and partially caused by the growth of the existing voids. The extent of the volume dilatation depends on the nature of the binder/particle system, the testing temperature, and the strain rate. Therefore, to effectively use the material in structural applications, one needs to understand the damage initiation and evolution processes, the effects of damage and crack development on the material's response, and the remaining strength and life of the structures.

In this task, a linear cumulative damage theory was used to derive a time-dependent damage parameter, $D(t)=\left[\int_0^t \sigma^{\beta}\, dt\right]^{1/\beta}$. Experimental findings revealed that, for a given time, the damage parameter is highly dependent on the strain rate. However, the critical damage parameter for breaking the specimen is insensitive to the strain rate.

In addition to developing a phenomenological cumulative damage model, Lockheed-Martin Research Laboratory's High Resolution Digital X-Ray Systems were used to monitor the microstructure change, damage process, and volume dilatation under an incremental strain condition.

The microstructure change and the crack formation in a particulate composite, containing 78% by weight of hard particles embedded in a rubbery matrix and subjected to an incremental strain loading condition, were investigated using real-time x-ray techniques. During the test, Lockheed-Martin Research Laboratory's High-Resolution Digital X-Ray System was used to investigate the characteristics of the damage initiation and evolution processes. An Instron table model tensile testing machine, which was placed between the x-ray radiation source and the x-ray camera, was used to strain the specimen under an incremental strain condition. The recorded x-ray data were processed to create a visual indication of the energy absorbed in the material.

X-ray data revealed that the microstructure changes as a function of the applied strain. It is interesting and important to point out that, at a critical applied strain level, a crack is formed in the weak region of the specimen, and it doesn't propagate. As the applied strain level is increased, the number of non-propagating cracks increases. Finally, two non-propagating cracks coalesce, resulting in a long crack which propagates and leads to the fracture of the specimen. Experimental findings revealed that the degree of inhomogeneity of the material's microstructure and the number of non-propagating crack increases as the applied strain is increased. Also, the strain distribution is highly non-uniform when the applied strain is high.

In order to obtain a fundamental understanding of the mechanism associated with the non-propagating crack, numerical modeling analyses were conducted to determine the effect of inhomogeneity of microstructure on the stress field at the crack tip. In the modeling analysis, the inhomogeneous specimen consisted of an edge crack in a soft region in the specimen. The soft region was modeled by reducing the volume fraction of particle, and the corresponding Young's modulus was calculated by the use of Mari-Tanaka method. The results of modeling analyses showed that the normal stress at the crack tip decreases as the volume fraction of particle is reduced. If the stress is below a critical stress for crack growth, the crack will not propagate.

In addition to investigating microstructure evolution, we also analyzed the x-ray data in 2mm x 2mm areas where a crack was formed. The results of the analyses revealed that the statistical density function is a function of the applied strain. On the first approximation, when the applied strain level is below a critical applied strain level, the statistical density function can be assumed to be a normal statistical density function. When the applied strain level is above the critical applied strain level, the statistical density function starts skewing to the right

Besides investigating micro-structural change, damage initiation and evolution processes, and crack growth behavior, a technique was developed, based on x-ray data, to predict volume dilatation, $\Delta V/Vo$, as a function of applied strain. The volume dilatation is a physical damage parameter and is used to develop a nonlinear constitutive model, which is incorporated in a computer code and used to predict the non-linear constitutive and crack growth behavior in solid propellants with good accuracy. Experimental findings revealed that a good correlation exists between the predicted and the measured dilatations. This experimental finding gives us confidence in using the x-ray technique to predict volume dilatation and the critical x-ray intensity for the onset of crack growth.

The damage field near the crack tip in edge-cracked sheet specimens subjected to cyclic loads was also investigated using Lockheed Martin Advanced Technology Center's High-Energy X-Ray Systems. The specimen was subjected to an incremental cyclic-strain loading condition, which had a triangular shape and seven strain cycles. The minimum strain level for each cycle was 0%; and the maximum strain level for each cycle was 1.75%, 4.55%, 9.5%, 9.5%, 10%, 15%, and 19%. After the last strain cycle, the specimen was pulled to fracture at a constant strain rate of 0.05 cm/cm/min. that was used during the cyclic-strain test.

Experimental findings revealed that, when the specimen is strained, the high intensity of stress near the crack tip will damage the material. As the material is damaged, the x-ray image shows a light area near the crack tip. Initially, the size of the light area or the size of the damage zone increases with increasing applied strain level. This phenomenon is expected, because the local stresses near the crack tip increase with increasing applied strain level. However, the damage zone size will not continuously increase with increasing applied strain level. Real-time x-ray data reveal that the damage zone size starts decreasing when a certain applied strain level is reached. This applied strain level is close to the strain level corresponding to the maximum applied stress. Since the applied stress will continuously decrease beyond the maximum applied stress, the local stress near the crack tip will also decrease. Since damage zone size is directly proportional to the local stress near the crack tip, the decrease in applied stress results in a reduction in local stress near the crack tip, which, in turn, results in a reduction in damage zone size.

Experimental findings revealed that under the constant strain condition, the crack propagates, and the damage zone size and the intensity of damage increases. It is known that for a viscoelastic material the stress in the material will relax under the constant strain condition. The growth of the crack and the increase in the damage zone size near the crack tip is probably due to the material's viscoelastic nature. In other words, a time scale or phase shift exists between the applied stress and the local stress in the material, especially near the crack tip. Under this condition, even the global stress starts relaxing but the local stress near the crack tip is still high enough to propagate the crack. It is interesting to point out that, after the specimen is unloaded to 0% strain from a higher applied strain during the cyclic loading test, the damage zones still exist. Due to the viscoelastic nature of the material, the microvoid size will decrease with increasing time. Consequently, the damage zone size and the damage intensity will decrease as the length of time is increased.

3.3 Task 3– Multi-Scale Modeling on Damage Initiation and Evolution

In this task, progressive damage in the specimen was simulated using a multi-scale technique. In this approach, the macro-level (i.e., composite specimen level) and the micro-level (i.e., the particle and matrix material level) were interconnected in analysis in a staggered way. First of all, the effective composite material properties were computed from the particle and matrix material properties, and the macro-analysis (i.e., composite structural analysis) was performed to calculate composite stresses and strains using the finite element method. The stresses and strains at the composite structural level were decomposed into the stresses and strains at the base material level; i.e., particles and matrices. Then, damage initiation and propagation were described for the particle and matrix materials. In the present study, only matrix damage was considered, because the particles were very strong compared to the matrix. Therefore, matrix cracking was the main damage parameter. The damage discussed from now on will be matrix damage. When the degraded matrix material properties were computed from a damage theory, effective composite material properties were computed from the particle and degraded matrix material properties. This iteration process of computation continued as long as the damage continued to grow or until the applied load reached the designated level.

The results of numerical modeling analysis revealed that as sporadic damage grew, damage saturation was reached at certain applied strain levels. The saturation of damage implied that a crack was formed. However, the developed cracks did not propagate beyond a certain length. In other words, there were non-propagating cracks developed in the specimen. When the applied strain was increased, the non-propagating cracks coalesced to form a longer crack, which propagated and caused specimen fracture. These computer-simulated damage initiation and evolution processes, as well as crack growth behavior, agreed well with experimental observation.

Because matrix failure was the main damage in the composite, the matrix stress and the damage plots showed some correlations, especially at the highly accumulated damage locations. The major damage locations were also well illustrated in the macro-strain plot. However, the macro-stress plot did not indicate any clear damage location.

In simulating the constitutive behavior of the inhomogeneous material, the specimen of size 24 mm by 24 mm was meshed into sub-domains of the size of the characteristic area, which was determined in the x-ray analysis, and it had a size of 2 mm by 2 mm. In each sub-domain, the material was divided into 16 elements, with each element having a different volume fraction of particles (VFP). However, the average VFP in the sub-domains was the same, which was equal to 0.65 with a standard deviation of 0.1. The variations in VFP were determined using normally distributed random number generation. Once normally distributed random numbers were generated for each sub-domain, their values were scaled properly to maintain the average volume fraction of particle over each sub-domain.

Numerical modeling analyses were conducted to simulate the stress-strain curves for a specimen with uniformly distributed VFP and for specimens with different characteristic areas in which the VFP of each element was different. The results showed that the stiffness was almost the same among different specimens regardless of the size of the characteristic area. Even the uniform specimen, which has no variation of particle density from element to element, had almost the same stiffness as the non-uniform specimens. However, the failure strengths were lower for the specimens with non-uniform VFP when compared with the uniform VFP case. On the other hand, different characteristic sizes of the sub-domains had the same failure strength.

The next study was focused on the simulation of crack growth behavior of a centrally-cracked specimen. A global analysis was conducted first to determine the deformation in the specimen. The deformation around the crack tip, determined from the global analysis, was used for the local analysis, which had a very fine mesh around the crack tip. The global analysis model had 144 elements that had the same VFP for every element, while the local analysis model had six elements, each of which was sub-divided into 256 elements. The VFP of the 256 elements varied from element to element, with the average VFP the same as that used in the global analysis. In addition, for a comparison purpose, a uniformly distributed VFP case including the local elements was conducted.

In the analysis, the size of the saturated damage zone was considered as the incremental crack length. For the cases of random distribution of particles, the analysis was repeated five times with normal random generation for statistical analysis. The results of the analysis revealed that, by comparing with the uniformly distributed VFP case, the randomly distributed VFP case resulted in crack growth under a smaller applied strain and a longer crack length for a given applied load.

In addition to conducting multi-scale analysis to predict the constitutive and crack growth behavior, we also developed a technique, based on a combination of the boundary element method and the homogenization method, to investigate the effect of particle size and arrangement on the material property of particulate composites. The results of the modeling analyses revealed that, for perfect bonding between the particle and the matrix, particle arrangement (regular and random) and particle size have no effect on the predicted effective Young's modulus and Poisson's ratio, as long as the volume fraction of particle is the same. For both Composites 1 and 2 as defined earlier, the predicted Young's modulus and Poisson's ratio were 0.51 MPa and 0.4999, respectively. However, experimental results show that the Young's modulus for Composite 1 and Composite 2 were 0.53 MPa and 0.66 MPa, respectively. It is seen that the predicted Young's modulus for Composite 1 compared well with the measured value. However, the predicted and the measured values for Composite 2 differed by 20%.

In this study, two methods – boundary element method and element overly method – were used to investigate the stress distribution in a unit cell with different numbers of particles.

In the first study, linear elastic analysis, based on the boundary element method, was used to determine (1) the stress distribution among the randomly distributed particles with different size distributions; and (2) the effective material properties of the material. The results of modeling analysis revealed that as long as the particle volume fraction is the same and there is no debonding at the particle-matrix interface, the effective material properties, Young's modulus and Poisson's ratio, are independent of the distribution of the particle size. For example, for a 10% volume fraction of particle, the Young's modulus and the Poisson's ratio are 0.95 MPa and 0.499 for two different particle size distributions (200 and 0.2 microns) and (200, 50, 0.2 microns).

It is known that the efficiency of the boundary element method diminishes when the material undergoes nonlinear deformation. In order to analyze particulate composites with the matrix material deformed nonlinearly, a method, based on a combination of the element overlay method and the homogenization method, was developed, which is suited for the mesoscopic analysis of particulate composites. In the method, each particle and its vicinity is modeled by a finite element mesh, which is called a local model or mesoscopic model. A unit cell or a structure made of the composite is discretized by a coarse finite element mesh, which is called a global model or macroscopic model. In the element overlay method, the local models are superposed on the global model, and the material properties are specified such that the global model has the material properties of matrix material only, and the materials other than matrix material are specified in the local model. By comparing with the ordinary finite element method, the element overlay method is much simpler to build and modify when a numerical model consists of a large number of particles. In order to evaluate the accuracy of the element overlay method, stress distributions in a plate containing one or four circular holes under a uniform tension were determined, based on the element overlay method and the ordinary finite element method. The results of the analysis showed that a good correlation exists between the stresses determined by the two methods.

The element overlay method was used to determine the two-dimensional stress distribution in a unit cell containing either 250 randomly distributed holes or 250 randomly distributed particles, and the three-dimensional stress distribution in a unit cell containing either 35 randomly distributed voids or 35 randomly distributed spherical particles. The results of the analyses indicated that, for the void model, high stresses were developed on either side of the void; whereas, for the particle model, high stresses were developed at the top and the bottom of the particle. In addition, the randomly distributed particles induced randomly distributed high stress locations in the material.

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