ABSTRACT
Spatial arrangement of microstructural features (particles, voids, grains, etc) is an important facet of microstructural geometry that affects the fracture sensitive properties (ductility, strength, fatigue life, etc) of monolithic as well as composite materials. Nonetheless, the experimental techniques for quantitative characterization of the important descriptors of the spatial arrangement of microstructural features are not well developed. Such quantitative information is necessary to understand how parameters such as spatial clustering affect the fracture sensitive mechanical properties. The central objective of the research program was to develop general and flexible experimental techniques for quantitative characterization of spatial arrangement of features in material microstructures, and application of these techniques to quantify clustering and microstructural non-uniformities in the three-dimensional (3D) microstructures of discontinuously reinforced metal matrix composites. The research program involved reconstruction of 3D microstructures of discontinuously reinforced Al-alloy matrix composites from montage serial sections and detailed characterization of the 3D microstructural geometry. The research led to development of stereology and digital image analysis based technique for efficient estimation of two-point correlation functions in 3D microstructures. These correlation functions were then utilized for computer simulations of the “realistic” microstructures that are statistically similar to the corresponding real microstructures. The research program also led to the development of some fundamental theoretical results concerning the nearest neighbor distribution functions and higher order correlation functions.

The research results have been reported in 15 scientific papers in archival scientific journals and conference proceedings, and 3 more papers are in the review process. The research results have been also presented in 9 keynote/invited presentations and 6 contributed presentations in major national and international conferences. In addition, the PI has given 5 invited seminars in the universities and R&D laboratories. Significant contributions have also been made to the development of human resources: 2 postdoctoral fellows, 4 Ph.D. students (including a student jointly advised by Prof. Garmestani and the PI), and 2 undergraduate students (2002 summer) have been involved in this research program. The PI taught significant portions of two continuing education short courses on Stereology and Quantitative Fractography to material scientists and biologists. The PI has also written chapters on quantitative fractography and quantitative characterization of microstructural geometry in the recent editions of the ASM Handbooks to disseminate the knowledge of new methodologies to practicing metallographers. The new stereological techniques for estimation of correlation functions and the methodologies for computer simulations of microstructures have been incorporated in the graduate course “Quantitative Characterization of Microstructures” taught by the PI at Georgia Tech.

The research program has led to close and fruitful research collaborations with Dr. D. Miracle (AFRL), Dr. J. Spowart (AFRL), and Prof. H. Garmestani (Georgia Tech) that have resulted in numerous joint publications and presentations in conferences. In addition, unfunded collaborations have been initiated with Prof. Horstemeyer (Miss. State U.) for modeling damage evolution in the composites, and with Dr. Peter Mouton (NIH) for applications of the stereological techniques developed in this research program for estimation of two-point correlation functions and other microstructural attributes in the biological structures.
EXECUTIVE SUMMARY

A microstructure is a collection of volumes, internal-surfaces, lines, and points. Depending on its dimensionality, each microstructural feature (particle, pore, grain, fiber, etc.) has associated with it, size, shape, volume, surface area, curvature, etc., and location. The distribution of relative locations of microstructural features is manifested in the spatial patterns, correlations, clustering, spatial affinity, short and long range interactions, pair correlations and higher order correlations, microstructural gradients, segregations, etc. The theoretical models, numerical simulations, and qualitative experimental observations indicate that spatial microstructural distance distributions affect numerous mechanical and physical properties of materials. Nonetheless, the experimental techniques for quantitative characterization of the important descriptors of the spatial arrangement of microstructural features are not well developed. Quantitative characterization and modeling of spatial arrangement of features in three-dimensional (3D) microstructures of discontinuously reinforced metal matrix composites is particularly important because although these composites have excellent strength and stiffness, their ductility, fracture toughness, and fatigue characteristics are often not satisfactory due to nonuniform and clustered spatial distributions of the reinforcement phases. Numerous qualitative experimental observations [1,2] and computer simulations [3-8] have shown that non-uniform spatial arrangements of particles are responsible for the poor fracture related attributes of discontinuously reinforced metal matrix composites. Accordingly, the central theme of the research program was to techniques for quantitative characterization of spatial arrangement of features in material microstructures, and application of these techniques to quantify clustering and microstructural non-uniformities in the three-dimensional microstructures of discontinuously reinforced metal matrix composites. The specific research objectives were as follows.

1. To reconstruct large volumes of three-dimensional microstructures of a set of discontinuously reinforced Al-alloy matrix (DRA) composites to study spatial arrangement of reinforcement phase.

2. To develop a practical and efficient methodology for experimental quantitative characterization of the descriptors of spatial arrangement of microstructural features in opaque materials and to apply these methods to quantify three-dimensional spatial arrangement of particulate phases in discontinuously reinforced metal matrix composites.

3. To develop a methodology for computer simulations and modeling of microstructural geometry where the spatial arrangement of microstructural features may be non-uniform and/or anisotropic and to arrive at realistic computer simulated microstructures for non-uniform microstructures of MMCs based on the experimental data on metric properties and spatial arrangement of microstructural features.

4. To develop and implement techniques to incorporate computer simulated microstructure models and digital images of non-uniform two- and three-dimensional microstructures in the models and simulation of mechanical response of materials.

The research results have been reported in 15 scientific papers in archival scientific journals and conference proceedings, and 3 more papers are in the review process. The research results have been also presented in 9 keynote/invited presentations and 6 contributed presentations in major national and international conferences. In addition, the PI has given 5 invited seminars in the universities and R&D laboratories. Significant contributions have also been made to the development of human resources: 2 postdoctoral fellows, 4 Ph.D. students (including a student jointly advised by Prof. Garmestani and the PI), and 2 undergraduate students (2002 summer) have been involved in this research program. The PI taught significant portions of two continuing education short courses on Stereology and Quantitative Fractography to material scientists and biologists. The PI has also written chapters on quantitative fractography and quantitative characterization of microstructural geometry in the recent editions of the ASM Handbooks to disseminate the knowledge of new methodologies to practicing metallographers. The new stereological techniques for estimation of correlation functions and the methodologies for computer simulations of microstructures have been incorporated in the graduate course “Quantitative Characterization of Microstructures” taught by the PI at Georgia Tech. The research program has led to close and fruitful research collaborations with Dr. D. Miracle (AFRL), Dr. J. Spowart (AFRL), and Prof. H. Garmestani (Georgia Tech) that have resulted in numerous joint publications and presentations in conferences. In addition, unfunded collaborations have been initiated with Prof. Horstemeyer (Miss. State
U.) for modeling damage evolution in the composites, and with Dr. Peter Mouton (NIH) for applications of the stereological techniques developed in this research program for estimation of two-point correlation functions and other microstructural attributes in the biological structures. Major research results are briefly summarized below; the details can be found in the publications that report these results.

1. Reconstruction of Three-Dimensional Microstructures from Montage Serial Sections

A precision polishing based set up put together by the PI and his students at Georgia Tech has been used for montage serial sectioning and reconstruction of three-dimensional (3D) microstructures of the DRA composites [9]. This set up is not as efficient as the Robo-Met developed at AFRL, but it is relatively inexpensive. An image analysis procedure is used to align the montage serial sections and reconstruct the 3D microstructure. These montage serial sections (~100 per specimen) generate a reasonably large volume of 3D microstructure (~1 mm³) at high resolution (~1 μm). As an example, Figure 1 shows segments of surface-rendered and volume-rendered 3D microstructure of 8.1 PSR composite reconstructed from the serial sections. Note that the volume segment shown is less than 10% of the actual 3D volume reconstructed.

![Figure 1](image1.png)

2. Stereological Estimation of 3D Two-Point Correlation Functions

For a two-phase microstructure containing particles (phase-1) and matrix (phase-2), the two-point correlation function $P_{11}(r, \theta, \phi)$ is the probability that both end points of a randomly located straight line of orientation $(\theta, \phi)$ and length $r$ are contained in the particles (phase-1), as illustrated by line AB in Figure 2a. The two-point correlation functions $P_{22}(r, \theta, \phi)$, $P_{12}(r, \theta, \phi)$, and $P_{21}(r, \theta, \phi)$ are defined in an analogous manner, but only one of the four two-point correlation functions is independent. The concept can be extended to three-point (triangle ABC in Figure 2b) and higher order correlation functions. A stereological method has been developed for efficient estimation of the direction dependent as well as orientation-averaged 3D two-point correlation functions from vertical 2D sections [10]. A digital image analysis-based procedure has also been developed that uses an in house C++ code for automatic measurements of two-point correlation from large-area high-resolution 2D binary image montages containing ~30,000 particles [10]. Using
this procedure, the two-point correlation functions can be precisely measured at distances ranging from 1 μm to 500 μm with a resolution of about 0.5 μm.

3. Computer Simulations of Microstructures Statistically Similar to Real Microstructures

An important inverse problem in the development of microstructure representation methodologies is that once a microstructure is quantified in detail, using such data, is it possible to simulate a microstructure that is statistically similar to the corresponding real microstructure in its geometric attributes including spatial arrangement (clustering, etc.) of the features? If this is possible, then the simulation parameters used for the microstructure simulation essentially represent all useful geometric information concerning the real microstructure. In this way, the “information” in the real microstructures can be separated from the “noise.” Although computer simulations of two-phase microstructures have been performed by numerous other investigators, almost all of such simulations (a) assume uniform random spatial distribution of the particulate phase, and (b) use simple model particle shapes such as spheres (circles in 2D) and ellipsoids (ellipses). Further, in most of the cases, the detailed geometric attributes of such simulated microstructures are not matched with those of the corresponding real microstructures to simulate the microstructures that are “realistic.” However, in the real microstructures, (i) the spatial arrangement of the constituent phases is often not uniform-random, and (ii) the particle (feature) morphologies are almost always very complex and cannot be realistically modeled using simple shapes such as spheres or ellipsoids. Recently, the PI and his students have made progress to resolve some of these issues. A computer simulation algorithm (and the code) has been developed to simulate the microstructures of the DRA composites of interest having exactly the same complex particle morphologies as in the corresponding real microstructures, any given volume fraction and size distribution of the SiC particles, and any given degree of microstructural spatial clustering and non-uniformities. A procedure is also being developed to match the two-point correlation function (and other attributes) of the simulated microstructure with the corresponding real microstructure.

4. Theoretical Results on Geometrical Constrains on N-Point Correlation Functions

The PI has derived a series of stochastic geometry based equations that relate the limiting behavior of two-, three-, and four-point correlation functions at small distances. These constrains are in addition to the well known limits of these functions as r approaches zero and infinity, which are related to the volume fractions of the constituent phases. For a two-phase microstructure, a three-point correlation function $P_{112}(r, \alpha_1, \alpha_2, \beta, \theta, \varphi)$ is the probability that a randomly located triangle, in a plane of orientation $(\theta, \varphi)$, having base length $r$ and angles between the base and the other two sides $\alpha_1$ and $\alpha_2$, and angle $\beta$ between the base and a reference direction in the plane of the triangle is such that the two corners of the triangle that are associated with the base are in the phase-1 (particles) and the third corner in the phase-2 (matrix). The PI has shown that as $r, \alpha_1$, and $\alpha_2$ all approach zero, the ratio of three-point correlation $P_{112}(r, \alpha_1, \alpha_2, \beta, \theta, \varphi)$ and the area of the triangle $\Delta$ approaches the following limit.

$$\lim_{r, \alpha_1, \alpha_2 \rightarrow 0} \left[ \frac{P_{112}(r, \alpha_1, \alpha_2, \beta, \theta, \varphi)}{\Delta} \right] = \frac{[T_A]_{abs}}{6} \quad \text{...(1)}$$
In equation (1), $[T]\text{abs}$ is the absolute (not net) number of tangents formed by a sweeping straight test line of orientation $\beta$ in the plane $(\theta, \varphi)$ with the boundaries between phase-1 and phase-2 per unit area of the sweep. The limiting value of the ratio of the probability $<P_{1,2}(r, \alpha_1, \alpha_2)>$ averaged over the orientation angles $\beta, \theta,$ and $\varphi$, and the area of the triangle $\Delta$ is given by the following equation.

$$\lim_{r, \alpha_1, \alpha_2 \to 0} [<P_{1,2}(r, \alpha_1, \alpha_2)> / \Delta] = \frac{Q_v}{(6\pi)}$$ ..........................(2)

In equation (2), $Q_v$ is the integral absolute mean curvature of the interfaces between the two phases. If the microstructure consists of randomly oriented convex particles of phase-1, then $Q_v$ is equal to the integral mean curvature $M_v$ of the interfaces between the two phases. An analogous limit exists for the four-point correlation function that is related to the integral of the absolute Gaussian curvature of the interfaces, and for a set of randomly oriented convex particles, it is directly related to the total number of particles per unit volume. The corresponding limit for the ratio of two-point correlation function $P_{1,2}(r, \theta, \varphi)$, and $r$, as $r$ approaches zero, is related to the total projected area of the interfaces per unit volume.

5. Computer Simulations of $N^{th}$ Order Nearest Neighbor Distributions in Microstructures

To model the mean nearest neighbor distances between particles/inclusions/voids in uniform random microstructures, extensive computer simulations have been performed to understand the first, second, and higher order nearest neighbor distribution functions of the centers of mono- and poly-size particle populations in one-, two-, and three-dimensional microstructures. It is shown that the volume fraction dependence of the mean value of $n^{th}$ nearest neighbor distance ($n = 1$ to $6$), $<H_n>$, in random 3D microstructures containing mono-size spheres can be represented as follows [11].

$$<H_n> / <d_n> = 1 + B_n f^2 \left( \frac{f}{N_v} \right)^{1/3}$$ ..........................(3)

In the above equations, $f$ and $N_v$ are the particle volume fraction and number density respectively, and $<d_n>$ is the mean value of the same order nearest neighbor distance in a uniform random distribution of points (zero size) having the same number density $N_v$, which is known from Poisson point process statistics. A similar equation has been derived for 2D microstructures that is applicable to uniaxial fiber composite [12]. These results are useful for incorporating 2D/3D nearest neighbor distances (which are extremely difficult to measure experimentally) in the theoretical models and simulations for the effects of inter-particle distances on the properties of materials containing inclusions/precipitates/dispersoids.

REFERENCES

PERSONNEL SUPPORTED

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<tr>
<th>Name</th>
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PEER REVIEWED ARCHIVAL PUBLICATIONS FROM AFOSR SUPPORTED RESEARCH

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Conference Proceedings Publications From AFOSR Supported Research

Invited/Keynote Presentations in Major Conferences Based on AFOSR Funded Research


Invited Presentations in Universities, National Labs, and Industries Based on AFOSR Supported Research


13. A.M. Gokhale: “Modeling and Representation of Microstructural Geometry: Implications for Materials Design”, Department of Materials Science and Engineering, University of Science and Technology, Beijing, China, April 29, 2004


Contributed Presentations in Conferences Based on AFOSR Funded Research


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