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Statistics and Materials Science

Report of a Workshop

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Preface

This report describes the scientific program and presents the findings of a workshop, *Statistics and Materials Science: Microstructure–Property–Performance Relations*, held at the National Institute of Standards and Technology (NIST) on July 26–28, 1993.

The workshop was sponsored jointly by the National Institute of Statistical Sciences (NISS) and NIST. It was organized by Edwin R. Fuller, Jr., of the Ceramics Division of NIST and Alan F. Karr, Associate Director of NISS. Stephen Freiman (Materials Science and Engineering Laboratory) and Robert Lundegard (Computational and Applied Mathematics Laboratory) of NIST were instrumental in articulating the goals of the workshop and securing funds from the Advanced Technology Program of NIST for preparation of this report. Raghu Kacker, of the Statistical Engineering Division at NIST, played a crucial role in early planning. Funds for participant support were derived in part from a grant by the National Science Foundation to NISS. The Conference Office at NIST handled local arrangements superbly.

Drs. Freiman, Fuller and Lundegard generously provided numerous comments and suggestions for improving the report, as did Jerome Sacks of NISS and Nozer Singpurwalla of the George Washington University.

This document may be read at various depths. The Executive Summary (§1) contains broad descriptions of the main issues; it can be read on its own, possibly with the assistance of Appendices C and D.

Section 2 is a description of the nature and problems of (some of) the data arising in materials science.

Section 3 presents in more detail the key materials science issues exposed at the Workshop, along with statistical strategies available or potentially available to address them; it is an expanded version of §1.6, and may be read almost independently of the latter.

Finally, §4, 5, 6 and 7 present technical details associated with quantification of microstructure, microstructure–property relations, materials performance and materials processing, excerpted from presentations at the Workshop. Section 8 treats the statistical theme of combining information, which is common to many of the materials science problems presented at the workshop.

Appendix A contains the names and addresses of Workshop participants, while Appendix B is the program. Appendices C and D are glossaries of materials science for statisticians and statistics for materials scientists, in an attempt to lessen the terminology impediment to cross-disciplinary research.

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1 Executive Summary

This report describes the scientific program and presents the findings of a workshop, *Statistics and Materials Science: Microstructure–Property–Performance Relations*, held at the National Institute of Standards and Technology (NIST) on July 26–28, 1993. More broadly, it addresses the nature and importance of cross-disciplinary research between materials and statistical scientists, as a key to progress in both fields.

To define the fields of materials science and statistics completely exceeds the scope of this report; however, it is important to identify key characteristics.

1.1 What is Materials Science?

In brief [77, 79],

> Materials science is the understanding of the nature, properties and use of materials and control of their basic building blocks: atoms, molecules, crystals, and noncrystalline arrays.

Seen in this light, materials are defined by chemical composition and structure at levels from the molecular to the macroscopic, both of which may vary in response to external influences, such as those exerted in processing or by the environment. Broadly, materials can be classified as

- Structural materials:
  - Metals
  - Ceramics
  - Composites (whose constituent phases are usually metals or ceramics);
- Polymers;
- Electronic materials;
- Biomaterials.

The structural materials are scientific neighbors, sharing an origin in mechanics, an emphasis on (poly-)crystalline structure and mechanical properties and an operative length scale of microns to millimeters. They are rather clearly differentiated from polymers, which lack crystal structure and whose theory is driven more by chemistry, electronic materials, whose behavior reflects quantum mechanical considerations at the nanometer scale, and biomaterials.

At a scientific level, the "objectives of materials science are the synthesis and manufacture of new materials, the modification of materials, and the understanding and prediction of materials properties and their evolution over time" [79]. Four themes are depicted in [77] as spanning materials science and engineering:

Materials microstructure, consisting of the size, shape and orientation of crystalline phases, and the location, orientation and connectivity of boundaries, defects, fibers and microcracks. Microstructure is accessible experimentally, can be manipulated in processing, and can be related to properties of materials.

Materials properties, such as mechanical properties (e.g., Young's modulus or fracture toughness), thermal properties (e.g., thermal conductivity) and electrical properties (e.g., electrical conductivity). Changes in properties over time, especially as a result of processing, use or environment, are important as well.

Component/materials performance in service. Often couched as reliability, performance relates to behavior, such as fatigue, failure and yield, that depends on extreme characteristics of microstructure, which are subject to severe variability. Environmental effects and degradation are also crucial, as is cost.

Materials processing, via which microstructure is manipulated, typically by application of stress, temperature and pressure, to produce materials and components with desired properties.

The relationships among these areas are equally significant.

1.2 What is Statistics?

The science of data,

Statistics is data-driven modeling and analysis.

In the best historical tradition of mathematics, statistics springs equally from applications in the sciences[1] and an internal imperative to understand and extend the theory.

A statistical analysis of a scientific problem poses three central questions:

- What is the problem?
- What are the data?
- How can the data be used to inform decisions regarding the problem?

With some oversimplification, many problems and techniques in statistics fall into three general categories:

- *Measurement*, which has a retrospective aspect of analyzing data collected previously, as well as a prospective aspect of designing informative means of data collection, such as experiments and surveys.

[1] This term is meant broadly, as a euphemism for the "real world."

- *Prediction* of unobserved or hypothesized phenomena. These range from not-yet- (but to-be-) observed behavior of physical systems to potential effects of policy decisions.
- *Modeling*, which links measurement to prediction, and analysis to data.

1.3 Background of the Workshop

The workshop was organized in response to growing belief that cross-disciplinary research between materials and statistical scientists is central to progress in materials science, and at the same time will stimulate development of new statistical strategies and theories having broad applicability. Previous workshops in November, 1992, organized by the National Institute of Statistical Sciences, and in June, 1993, organized by the Catholic University of America, addressed rather focused aspects of this issue, and a more broadly targeted workshop was seen as desirable.

The purposes of the workshop were to:

- Initiate productive communication between materials and statistical scientists;
- Formulate a cross-disciplinary research agenda leading to high-impact advances in materials science *and* statistics;
- Recommend actions by researchers, their institutions and funding agencies to address this agenda;
- Identify potential barriers to implementation of the research agenda, and means by which they may be overcome.

Two key classes of structural materials, ceramics and metals, were emphasized. This was deliberate, in order to limit the scope of the workshop, and reflects the similar origins and concerns of these classes and their importance to industry, as well as the perceived immediacy of statistics to central issues in them.[2]

The fifty-two workshop participants were materials and statistical scientists from industry, government laboratories and universities. Their names and addresses appear in Appendix A.

The workshop program, reproduced in Appendix B, comprised eleven lectures, one session of informal, ten-minute presentations, and a concluding panel discussion.

1.3.1 Materials and the Advanced Technology Program

Three industrial needs, all of which are targeted by NIST's Advanced Technology Program (ATP). motivated the workshop:

- Life-cycle engineering;

[2]This should not be construed to mean either that other classes of materials are unimportant or that statistics is irrelevant to them.

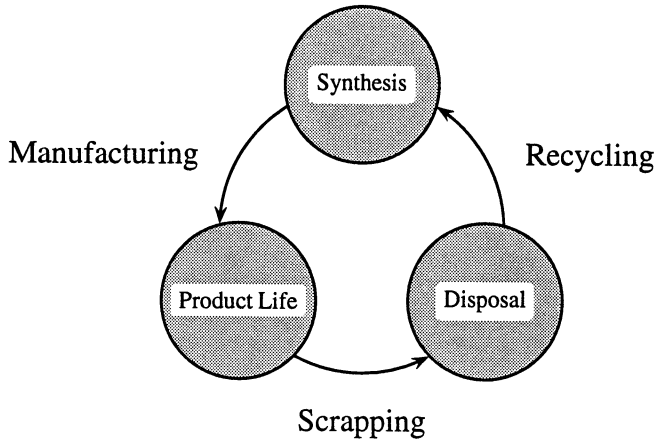


Figure 1: Life-Cycle Engineering

- Component and system performance;
- Industrial competitiveness.

Translated to the context of the ATP, the key needs for materials science are to

> **Design** components with desired **performance**,
> fabricated from materials with desired **properties**,
> and the **processes** to produce
> these components and materials
> via control of **microstructure**.

The ultimate goals are to

> Optimize materials properties and
> increase reliability of components and systems.

1.3.2 Life-Cycle Engineering

Life-cycle engineering, or *concurrent design*, posits that products be designed and evaluated in terms of their entire life cycles, as depicted in Figure 1.

At any point in its life, a product is characterized by its *product state*, consisting not only of physical variables, such as composition, microstructure, temperature, stress and shape,[3] but also economic variables, such as cost. In some settings, the appropriate state may consist not of simply the current values of such variables, but rather their entire histories.

Effective life-cycle engineering leads to reliable, affordable products, funds for needed research, a cleaner environment and jobs for scientists and engineers.

1.3.3 Component and System Performance

Component and system performance is the basis of product evaluations, whether prospective or retrospective. The need for credible prediction of performance and properties is overwhelmingly urgent. Indeed, of ten materials needs identified in [77] for eight industries,[4] four span all eight industries, and two of these focus on prediction:

- Prediction of service life;
- Prediction of physical properties.[5]

Performance, albeit often expressed in terms of physical components of product state, cannot be isolated from cost and other nonphysical factors. This is particularly true for "advanced materials," whose superior performance is often matched by high cost. In "high-tech" settings, performance may dominate, but in other settings, established, inexpensive materials may be preferred. Always, however, there remains the need to predict performance.

Increasingly, applications demand that materials perform in hostile environments, with respect, for example, to temperature, stress or corrosion. Such factors must be accounted in performance predictions.

1.3.4 Economic Competitiveness

Banalities notwithstanding, advances in materials science and engineering are widely acknowledged as crucial to U.S. economic competitiveness:

> New structural materials technologies will be a determining factor in the global competitiveness of U.S. manufacturing industries in the 1990s and beyond. Today, for example, materials account for as much as 30 to 50 percent of the costs of most manufactured products. New materials that can reduce overall production costs and *improve performance* [italics added] can provide a competitive edge in many products, including aircraft, automobiles, industrial machinery, and sporting goods. [81]

[3] All of which are fields, that is, functions of spatial location within the product as well as time.

[4] Aerospace, automotive, biomaterials, chemical, electronics, energy, metals and telecommunications

[5] The remaining "spanning" needs are near-net-shape forming and materials databases. The other needs are lightness/strength, high temperature resistance, corrosion resistance, rapid switching, efficient processing and material recycling.

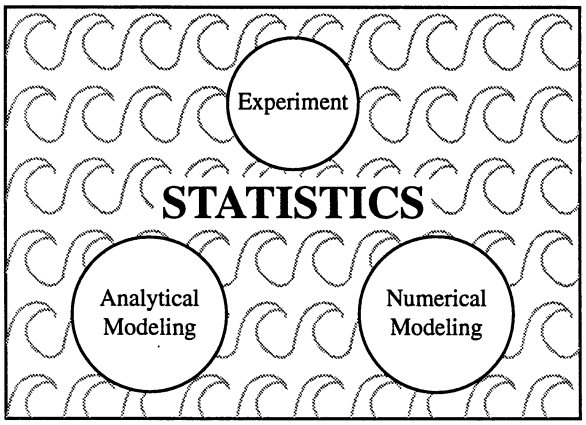


Figure 2: Materials Science in a "Sea" of Statistics (after S. Kurtz)

> An overriding theme for all the industries surveyed was the primary importance of *synthesis and processing* [italics added] of new materials and traditional materials, as well as fabrication of these materials into components and devices. Materials science and engineering, and processing in particular, plays a uniquely important role in these industries and in their ability to help maintain and improve the U.S. position in international competitiveness. [77]
>
> In recent years, a combination of factors has prompted recognition of the need for a stronger overall Federal commitment to materials R&D. A major concern is growing foreign competition, which already has had a significant impact on manufacturing and national competitiveness. [29]
>
> [I]t is clear that the scientific vigor, technological strength, and economic health of the nation all argue in favor of universities, government, industry, and professional societies stimulating and facilitating new collaborations between mathematical scientists and materials scientists. [79]

Thus, materials science and engineering are necessary (but not sufficient) in order that the U.S. improve its international competitiveness.

1.4 Findings and Recommendations

The workshop produced two principal findings:

> Crucial problems in materials science are inherently statistical, so that statistics is an enabling technology for progress in materials science.

and

> Fulfilling industrial needs and goals demands cross-disciplinary collaboration between materials and statistical scientists.

Materials structure is a complicated object, whose intrinsic variability can only be characterized statistically. Key experimental data are uncertain and incomplete. Relations among structure, properties, performance and processing, derived from a combination of experiment, analytical modeling and numerical modeling, require statistical characterizations. Indeed, modern materials science is embedded in a "sea" of statistics: see Figure 2.

It follows that neither materials science without statistics nor statistics without materials science can address the industrial needs outlined in §1.3.1.

The recommendations, then, are straightforward:

Statistical and materials scientists *need to collaborate*, for their mutual benefit. This will produce key progress in materials science, *and* will lead to important advances in statistics as well. There is no possibility that extant statistical strategies will suffice to resolve the materials science issues raised in this report.

Funding agencies should continue to *recognize the need* and *enhance their support* for cross-disciplinary, large-scale approaches to important issues in materials science.[6] High-impact projects will require funding at the scale of millions of dollars annually. Agencies[7] must realize that "cross-disciplinary collaborations require long-term commitments" [77].

Industrial and university managers, who select personnel and establish and implement reward structures, should *pay serious attention to cross-disciplinary research* generally, and to that between materials and statistical scientists in particular. Granting tenure or other rewards to a statistician for cross-disciplinary contributions to materials science, or *vice versa*, should be a goal, not a fear.

Institutions with extensive mandates and broad constituencies, NIST and NISS in particular, should set the standard for cross-disciplinary communication and collaboration. They are

[6] A conclusion echoed, in the broader context of all mathematical sciences, in [79].

[7] And researchers!

especially well positioned to perform and foster cross-disciplinary research. Moreover, communication, in multiple forms, including workshops, written reports and interaction with non-technical communities, is explicitly part of their mission.

The needs are urgent, but just one collaboration producing high-impact, high-visibility contributions to both materials science and statistics can serve as a powerful model. Others will then surely follow.

1.5 Materials Science Themes

We now move on to present a somewhat more technical summary of the workshop.

Several materials science themes emerged strongly and repeatedly. The importance of statistics and cross-disciplinary research to these themes is discussed in §1.6 and again in §3.

Implicit in each of these themes are *analysis and modeling*, which are identified[8] in [77] as "cut[ting] across all four of the elements of materials science and engineering." The importance of modeling is driven by increasing speed and capacity of computers, by availability of instruments able to make detailed quantitative measurements and by advances in theoretical understanding of materials properties, which allow construction of more accurate numerical simulations.

1.5.1 Use and Understanding of High-Dimensional Data

Microstructure is the epitome of complex, high-dimensional data. Experimentally accessible features must be characterized, and at the same time, crucial characteristics affecting materials properties identified and used to inform the measurement process. Although some needs for additional or improved data were identified — higher resolution and direct measurement of three-dimensional structure, in particular — use of available data seems to be a more pressing issue.

1.5.2 Heterogeneity

Nearly all real materials are heterogeneous at length scales finer than the microstructure. Many, for example, composites and some ceramics, are heterogeneous at larger length scales. Models are necessary that accommodate and represent heterogeneity. At the heart of this issue are the problem of dealing simultaneously with multiple length scales and an inevitable tension between representative properties and local variation.

Multiple, simultaneous length scales. Length scales relevant to microstructure (let alone components) range over at least four orders of magnitude (10^{-7} to 10^{-3} m), and must be handled simultaneously. Data regarding different length scales is of variable nature and quality.

[8] Together with instrumentation.

Representative elements and local variation. In order to provide a simplified description of a material or component, it is necessary to identify "representative" elements of it, but inevitably these suppress information regarding local variability, which may be crucial to macroscopic behavior.

1.5.3 Critical and Extreme Phenomena

Ceramic components, unlike metals, are *brittle*. Failure (fracture) is sudden and catastrophic, ordinarily without precursors. Fracture of ceramics results from propagation and coalescence of cracks [68], coupled with the inability of the material to relieve stress concentrations at flaws by means of plastic deformation. The process may be initiated by the "weakest link" in the component, an extreme characteristic subject to severe variation. As a consequence, ceramics exhibit size scaling: larger objects are weaker than smaller. In addition, object-to-object variability is higher than for materials in which component behavior reflects "average" rather than extreme material characteristics. Models are required that relate macroscopic failure to extreme microstructural characteristics. Mechanisms to strengthen ceramics (e.g., transformation toughening, the use of ceramic composites and introduction of microcracks during processing) are known, but predictive models of their effects are lacking.

1.5.4 Interfaces and Grain Boundaries

Important behaviors of brittle materials, such as fracture, are controlled by interfaces between grains and by boundaries between grains and other phases, including defects and voids. Stress concentrations associated with interface geometry and topology lead ultimately to fracture of the material. Description of interfaces and their relationship to key properties, despite recent progress, remains an important unmet need.

1.5.5 Time-Dependent Effects

Materials performance and processing are time-dependent. The product state — temperature, stress, composition, microstructure, shape and cost — varies in consequence of deliberate and inadvertent or uncontrollable actions. Performance depends on time-evolving fatigue, damage and environmental degradation (e.g., corrosion). Processing typically involves time-dependent application of heat, pressure and mechanical stress, and possibly chemical effects as well. The physical processes themselves are not understood completely, and available data are incomplete. To the extent that microstructure determines properties of interest in known ways, understanding *microstructural evolution* comprises one approach to these issues.

1.5.6 The Role and Evaluation of Complex Computer Models

Computer experiments have become an indispensable to the research process in materials science. Computer models discussed at the workshop portray phenomena as diverse as fracture of ceramics and hydration of cement. These models are complex, often with embedded finite element calculations. They may or may not be explicitly stochastic (Monte Carlo models). Integration of data from computer experiments with that from physical experiments is necessary. In addition, models themselves must be validated, and their parameters, which may have no innate physical meaning, must be "tuned" to available physical data.

1.5.7 Discretization of Data and Models

The ubiquity of computers, not only in modeling but also in data acquisition, can lead to approximations and errors. Much data regarding microstructure is collected as, or converted to, digital images. Finite element models entail similar discretization. The effects of discretization in data and models are not understood fully.

1.6 Materials Science Issues and Statistical Strategies

From the materials science themes in §1.5, we extract and summarize a set of materials science issues with three key characteristics:

- They were identified as important at the workshop: substantial progress on them will lead to improvements in performance, properties, and processing;
- Statistical strategies can be developed that will lead to progress on them;
- Development of such strategies will constitute progress in statistics as well.

Addressing these issues requires cross-disciplinary research between materials and statistical scientists.

This entire subsection is presented in expanded form in §3, where details concerning the statistical strategies are discussed.

1.6.1 Materials Performance

Issue: Performance prediction from diverse data. Component performance, in the form of failure probabilities on the order of 10^{-6}, must be predicted from data of varying nature, sample size and quality. Models must incorporate dependence of performance on materials, geometry, fabrication, load, testing, operating environment and time; they may involve heavy computational burdens. Uncertainty in predictions of performance must be characterized.

Statistical strategies: Design of physical and numerical experiments; combining information from physical and numerical experiments; simulation; statistical estimation of functional

relationships;[9] model validation; use of physical data to "tune" parameters of computer models;[10] statistically derived "fast prototype" simulations.[11]

Issue: Models of failure and fracture. Micromechanical models, analytical and numerical, of varying failure modes are required. Phenomena that must be accommodated include interfacial processes, heterogeneity and time dependence of materials properties in response to loads or environmental conditions.

Statistical strategies: Stochastic micromechanical models; physically-based statistical models for tails of performance distributions, model validation; tuning of computer models to physical data; statistically derived fast prototype simulations.

Issue: Testing. Effects of proof tests on components (currently assumed negligible) must be evaluated. Data from proof and verification tests must be combined informatively to predict performance. Nondestructive means are required to probe the state of components in service; resultant data must be incorporated effectively into predictions.

Statistical strategies: Statistical estimation of functional relationships; combining information; tuning of computer models to physical data; experimental design for nondestructive evaluation.

Issue: Environmental effects and degradation. Dependence of component performance on time histories of load and environment, which are observed neither accurately nor completely, must be represented. Feedback effects on materials properties may exist.

Statistical strategies: Dimension reduction;[12] statistical estimation of functional relationships; stochastic modeling of load and environment processes, as well as observations of them, and feedback on materials properties; model validation; tuning of computer models to physical data.

1.6.2 Materials Properties

Issue: Prediction of properties from microstructure. Microstructure–property relations are required to enable informed, efficient design and synthesis of materials with desired or optimal properties. Active microstructural variables must be identified. Physical data must be supplemented with data from numerical experiments.

[9] In this case, performance as a function of material, component geometry, fabrication, ...

[10] As opposed to direct measurement/estimation of model parameters; see §3.

[11] Simplified, fast running approximations of detailed simulations, derived by statistical means and used to facilitate more complete exploration of model behavior. See §3.

[12] Statistical identification of informative low-dimensional approximations of high-dimensional data, for purposes of application of other statistical strategies, as well as interpretation and visualization of data.

Statistical strategies: Estimation of functional relationships; dimension reduction; stochastic models of microstructure; design of physical and numerical experiments; combining data from physical and numerical experiments; tuning of computer models to physical data.

Issue: Environmental degradation. Microstructural mechanisms by which materials deform and degrade, and their effects on properties, must be modeled, together with partially observable environment processes. Microstructure and properties must be measured and inferred nondestructively.

Statistical strategies: Stochastic micromechanical models; statistical estimation of functional relationships; dimension reduction; model validation, statistically derived fast prototype simulations, combining information from physical and numerical experiments; design of experiments.

Issue: Validity of computer simulation models. Applicability and limitations of numerical simulations[13] (for example, finite element and spring network models) of microstructures and microstructural processes need to be characterized. Effects of discretization of data (e.g., digital images) and computational algorithms (e.g., finite element codes) require clarification. Representative elements of microstructure must be identified for various purposes.

Statistical strategies: Model validation; tuning of computer models to physical data; statistically derived fast prototype simulations.

Issue: Tools for microstructural design. Credible, "user-friendly" tools for microstructural design, in the form of software packages incorporating microstructure–property relations, must be developed.

Statistical strategies: Visualization and interpretation of complex data [122, 123].

1.6.3 Materials Structure

Issue: Characterization and quantification of microstructure. Three-dimensional microstructure must be inferred from two-dimensional data. The complex, hierarchical nature of microstructure must be reduced to a small number of context-dependent statistical descriptors (for example, to serve as inputs to microstructure–property relations). Differences among microstructures must be characterized, and important physical properties, such as anisotropy, detected. The experimental process should be informed by identified microstructural descriptors.

Statistical strategies: Stereology, especially for higher-order properties; "direct" averaging of grain shapes;[14] dimension reduction; statistical estimation of functional relationships;

[13] We use the term "simulation" broadly, to describe all models simulating physical reality, regardless of whether they are Monte Carlo models.

[14] Allowing definition of microstructural features as functionals of average grains, in addition to averages of functionals of grains.

design of physical and numerical experiments.

Issue: Models of microstructure. Improved models of microstructure are needed as inputs to models that simulate responses of materials to external factors. These models should reflect the hierarchical nature of microstructure and facilitate identification of appropriate "representative elements," yet reflect adequately local variations in structure. They must also represent interfacial structure and processes, which are crucial to performance.

Statistical strategies: Stochastic models of microstructure permitting dependence among grain shapes; design of numerical experiments; dimension reduction; tuning of computer models to physical data.

Issue: Fracture surfaces. Fracture surfaces should be used as data for materials microstructure as well as failure processes. Statistical descriptors of fracture surfaces must be identified. Models of fracture surfaces are necessary, possibly involving fractals, and must be related to micromechanical models of the processes that produce them.

Statistical strategies: Stereology; self-similar stochastic processes; dimension reduction; statistical estimation of functional relationships; statistically derived fast prototype simulations.

1.6.4 Materials Processing

Issue: Microstructural evolution. Models of microstructural evolution during processing are needed; together with microstructure–property relations, they lead to *process–property* relations enabling fabrication of materials with desired or optimal properties. Models of incompletely observable processing states are required, to be used in conjunction with models of microstructural evolution in order to create tools for microstructure-based process design.

Statistical strategies: Stochastic micromechanical models; statistical estimation of functional relationships; dimension reduction; model validation; combining information from physical and numerical experiments; tuning of computer models to physical data; statistically derived fast prototype simulations.

Issue: Constitutive equations. Parameters associated with constitutive equations describing materials processing must be estimated from sampled time trajectories of materials properties and applied stresses. Values of processing variables leading to desired material responses must be identified, in order to facilitate prediction for process design. Unobservable internal variables must be constructed.

Statistical strategies: Parametric estimation; statistical estimation of functional relationships; dimension reduction; model validation; combining information from physical and numerical experiments.

1.7 Research Priorities

The consensus of workshop participants, despite inter- and intra-community diversity, was that five research issues are the most pressing:

1. Developing techniques for *characterization and quantification of microstructure*;
2. Constructing design tools for *prediction of component performance*;
3. Developing tools for *microstructural design of materials* with prescribed properties;
4. Accommodating *time-dependent effects*, particularly environment and degradation;
5. Understanding and evaluating the *role of complex computer models* in research in materials science.

There was also consensus that significant progress on each one of these issues *requires and will result from cross-disciplinary collaboration between materials and statistical scientists.*

In several ways, microstructural characterization is the pivotal issue, since progress on other questions, especially microstructure–property relations and microstructural evolution, requires, as a prerequisite, informative, low-dimensional statistical characterizations of microstructure derived from experimentally accessible data.

1.8 Communication Between Statistical and Materials Scientists

Productive collaboration requires communication. Initiating communication between statistical and materials scientists was a principal goal of the workshop. Even though participants were selected on the basis of *a priori* willingness to try to communicate, the process was not easy nor was it brought to closure.

The "extreme cross-disciplinarity" of the workshop, as one attendee characterized it, was striking. The most apparent manifestation of this was each group's lack of familiarity with the "other field's jargon." To statisticians, such fundamental concepts as stress, strain and the finite element method, let alone more advanced notions, were unclear. Even the definitions of material and ceramic were uncertain. Conversely, materials scientists, even those with some appreciation of the key statistical concepts of variability and independence, were uninformed regarding more "exotic" statistical strategies, such as function estimation and Bayesian methods for combining information. The language barrier was greater than anticipated, and was overcome (albeit only partially) through determined efforts of the participants. Appendices C and D attempt to lower the language barrier for readers of this report.

Participants were virtually unanimous that future workshops are necessary and desirable, in order to continue and enhance the communication process.[15] Ultimately, of course, workshops will report on results of collaborations, rather than just exploring their desirability or urgency.

[15]To facilitate attendance by at least one community, these might be held in conjunction with society meetings.

1.9 Other Barriers

Compared to the "inability to communicate," other barriers to collaboration between statistical and materials scientists appear relatively minor, and are more excuses than true impediments. Many are generic barriers to cross-disciplinary research,[16] which history has demonstrated to be surmountable:

- *Individual barriers,* such as inertia, reluctance and (self-characterized) lack of knowledge of "the other field." Most of these are rationales, not true barriers. In particular, lack of knowledge can be overcome, initially by communication of the kind initiated by the workshop, and more fully in the course of cross-disciplinary collaboration. Statistical and materials scientists need not know everything about each others' fields in order to collaborate. On the contrary, one key tenet of team-based, cross-disciplinary research is that communication can overcome educational disparities.

- *Institutional barriers,* not only in regard to partnerships involving industry, government laboratories and universities, which cross-disciplinary research in materials science demands, but also within classes of institutions, as well as individual institutions. These include questions of intellectual property, publishability of research results, distribution of financial gains, and institutions' reward systems and "mores." There exist workable models for overcoming all of these.

- *Differing scientific styles,* including, for example, the role of postdoctoral fellows, the perceived scientific value of collaboration and the scale of projects. These are not an issue when there are true desire and need to collaborate.

- *Funding* is an obstacle currently because of pervasive shortages of funds rather than negative attitudes on the part of funding agencies toward cross-disciplinary research. On the contrary, agencies increasingly assign high priority to cross-disciplinary projects. Especially in the case of materials science, which is already acknowledged to be of national importance [29, 77], and which has cross-disciplinary roots of its own [77, 79], this appears not to be an issue.

- *Asymmetry,* in the sense that collaboration between materials scientists and statisticians may be seen as contributing only to materials science, *is* perhaps the most real risk, not only in reality, but also because its perceived existence is a deterrent. These fears are unfounded. Were all needs for statistics in materials science fulfillable by extant techniques, this would have happened already; the workshop produced compelling evidence that it has not. That hard problems in materials science will engender only trivial problems in statistics is simply not the case.

[16] For further discussion, see [50, 79, 106].

2 Data in Materials Science

Before proceeding, we describe some primary forms of data available in materials science, and their characteristics.

The various forms of data are not, of course, collected or applied in isolation from one another. They are linked in order, for example, to predict component performance from materials properties or properties from microstructure. Often, the links are manifested as computer models, which must be tested and evaluated.

2.1 Component and Materials Performance

Component performance, although in principle complex and multi-dimensional, is often reduced to dichotomous "reliability" terms: a component either performs as intended or it fails. Data are direct measurements (counts) of failures of components under controlled loads or in service, in which case loads may be unknown. Controlled-load tests may force failure (verification tests) or correspond to a nominal or design service load (proof tests). When components are costly, sample sizes are small (tens) for verification tests, and may be only in the hundreds for proof tests.

Data for components in service may go beyond their having survived; for example, nondestructive evaluation may yield information about current structure and properties. To the extent that predicted performance depends on history, such data are extremely important; however, relatively scant attention has been directed to their collection and analysis, or to incorporating them in predictive models.

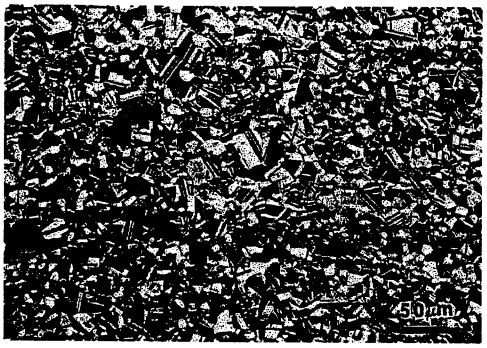
Problems with performance data include small sample sizes and incomplete load and environmental measurements. Complicated component geometry limits opportunities to combine data from different kinds of components.

2.2 Materials Properties

Ordinarily, materials properties are measured from laboratory specimens (with known, simple geometry) under controlled circumstances. Physical properties may be

- *Measured directly,* for example, fracture strength of ceramics.
- *Calculated from other measurements,* using established physical relationships or validated models. In some instances, this may be a matter of experimental convenience; in others (e.g., when direct measurement would destroy a specimen), "indirect" measurement is unavoidable.

Phenomenological properties are parameters of models of materials rather than attributes of materials themselves, and pose additional difficulties. By definition, they are not measurable directly, and must instead be inferred statistically. The extent to which they describe materials as opposed to models is sometimes problematic, as is their interpretation.



Phosphor Bronze

A Superconducting Oxide

Figure 3: Two Examples of Microstructure

Quality of properties data ranges from very high to fairly low, as a function of experimental technique and care, instrumentation and the nature of the property. Sample sizes, likewise, are highly varied. Other potential problems include measurement variability and experimental design, for example, sampling design and control for other variables.

2.3 Materials Structure

Physical data regarding microstructure is of two general classes:

- *Visual data* consist of micrographs, almost always in the form of digital images, that show a "picture" of the material. Experimental tools include not only older techniques such as light microscopy and ultraviolet spectroscopy but also more recently developed tools, for example, scanning tunneling microscopes and electron microscopes. Different magnifications, corresponding to different levels in the hierarchy of structure, are possible. Micrographs may comprise planar sections of the material (Figure 3) or may be *fractographs* depicting fracture surfaces (Figure 4). Chemical or other treatment of surfaces may be employed (Figure 5) in order to help reveal three-dimensional structure. Techniques from image analysis may be used to "enhance" digital data, for example, to aid in identifying grain boundaries.

- *Inferential data* constitute physical responses of specimens, from which microstructure is inferred by means of (verified) models or standards. Examples include bulk X-ray measurements, differential scanning calorimetry, electrical resistivity measurements and neutron scattering. Models used in this context are, in effect, microstructure–property relations (see §3.3), used to deduce structure from properties.

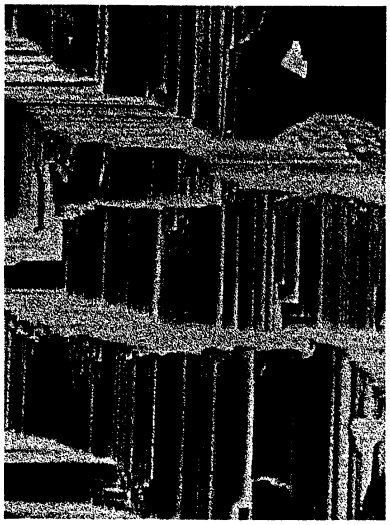


Figure 4: Scanning Electron Micrograph of a Fracture Surface in Alumina (Al_2O_3)

Figure 5: Data Revealing Three-Dimensional Structure of Silicon Nitride (Si_3N_4)

Microstructure data is generally plentiful, and occasionally overwhelming. The most evident shortcoming, discussed at some length in §3.4, is that *the data is two-dimensional, but microstructure is three-dimensional.* Also important are questions of adequacy of models used to analyze inferential data. Additional problems associated with microstructure data *per se* include noise and other distortions in visual data, which are particularly acute in regard to interfaces and insufficient resolution to detect features of interest. In addition, different imaging modalities (e.g., light and electron microscopy) yield data at disparate length scales.

2.4 Materials Processing

There is a striking dearth of "real" data pertaining to materials processing. Key variables are inaccessible experimentally, for example, because appropriate sensors do not exist or because the process is physically impossible to monitor (e.g., sintering of ceramics). In almost all instances, measurement would interfere with the process. Data collection may be seen as too costly, especially if meaningful uses of the data have not been demonstrated. The value of data concerning existing, possibly immutable, processes is problematic. Data collected for other purposes, such as process control, may be discarded once it is used, and be unavailable for other purposes.

The principal problem seems to be a lack of persuasive uses of data, compounded by general inattention to instrumentation in the U.S. [77] and a shortage of models to inform measurement design and data collection. The key issue is to define the entire process of measurement.

On scientific level, data regarding microstructural evolution are often merely time-dependent, time-sampled measurements of properties and microstructure. Comparable data on environmental and processing factors may be absent.

3 Materials Science Issues and Statistical Strategies

Here, we describe in more detail the crucial materials science issues raised at the workshop, together with statistical strategies applicable to them. This applicability ranges from rather apparent in some cases to speculative in others. Nevertheless, it is real in all, and stands ready to be realized by means of cross-disciplinary research between materials and statistical scientists.

The ordering of topics (materials performance, materials properties, materials (micro)structure, materials processing) represents a "top-down" approach to design: performance needs define properties needs, which in turn define desired microstructural characteristics, and, finally, processes must be devised that impart those characteristics.[17]

3.1 Statistical Strategies

Several "non-traditional" statistical strategies occur throughout this section. Rather than explain repeatedly, we summarize them here, and also provide references.[18]

3.1.1 Combining Information

This set of techniques, most "Bayesian" in nature, for informative fusion of data from disparate sources, is discussed in §8.

3.1.2 Design and Analysis of Numerical Experiments

A computer experiment is a number of runs of a complex computer model with various inputs. If the model is demanding computationally, only a few runs are possible. *Design* of numerical experiments deals with selection of inputs at which the model will be run, while *analysis* pertains to using the resultant outputs to meet various objectives, for example, to predict the response of the model at untried inputs or to optimize some functional of the response.

Three particular strategies discussed here — model validation, tuning computer models to physical data and statistically derived "fast prototype" simulations — address various aspects of numerical experiments. See [19, 82, 83, 84, 85, 103, 104].

3.1.3 Dimension Reduction

Statistical data is often characterized by complexity and high dimensionality: data sets may consist of many points, and individual observations may be complicated and high-dimensional. Techniques for data reduction are meant to alleviate such complexity, while minimizing the concomitant loss

[17] In terms of causality, the flow is reversed: processes define structure, which engenders properties, which — together with external effects — define performance.

[18] Definitions are given in Appendix D as well.

of information. Ordinarily, this occurs by forming lower-dimensional, perhaps data-dependent, functions of the original data.

Dimension reduction can serve to

Reduce sample size, by encapsulating key features of a large data set in a few summary statistics. For example, given data $X_1, \ldots, X_n$, the n-dimensional vector $(X_1, \ldots, X_n)$ might be summarized in terms of the sample mean $\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_i$ and the sample standard deviation $S = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(X_i - \bar{X})^2}$, reducing the effective dimension to 2.

Simplify data points, by taking some function of individual data points. Suppose that $X_1, \ldots, X_n$ are m-dimensional observations, where m is large. Dimension reduction is then effected by means of a function $K : \mathbb{R}^m \to \mathbb{R}^k$, where $k < m$, with X_i replaced by the lower-dimensional vector $X_i^* = K(X_i)$. In simple cases, K does not depend on the data,[19] but most in interesting cases, K depends on the entire data set. For example, in principal components analysis [96], K is a projection onto a subspace of $\mathbb{R}^m$ determined by all the data.

3.1.4 Estimation of Functional Relationships

Observed data often take the form of input-output pairs (x_i, y_i) for some physical system. The system may be conceived as a function F, which is at least partially unknown. The observations have, for example, the form

$$y_i = F(x_i) + e_i, \tag{3.1}$$

where the e_i are random errors. The inputs x_i may or may not be random.

Estimation of the functional relationship between inputs and outputs, then, requires estimation of the function F in (3.1), or of whatever aspects of it are not known.

Two main classes of function estimation problems[20] are

Parametric problems, in which F is of known qualitative form, but with (finitely many) unknown parameters. The simplest example is a *linear model*

$$y_i = (a + bx_i) + e_i,$$

where a and b are unknown constants.

Nonparametric problems, in which F is entirely unknown, or specified only to the extent that it belongs to some large (i.e., infinite-dimensional) class of functions.

For details, see [8, 32].

[19] For example, K might be a projection onto a k-dimensional subspace.

[20] Statistical *function estimation* is in fact somewhat broader, pertaining to any statistical model in which the unknown "parameter" is a function. *Density estimation* is one example of a function estimation problem that cannot readily be put in the form of (3.1).

3.1.5 Model Validation

Consider a computer model of the form (3.3), and suppose now that the *observed* physical data are modeled as

$$y_i = F^c(x_i, \mathbf{c}) + \sigma Z(x_i) + e_i, \tag{3.2}$$

where F^c represents the computer model, whose outputs are a function of inputs x and a vector $\mathbf{c}$ unknown parameters and e_i are errors.

In (3.2), Z is a stochastic process of known structure and σ is an unknown constant. The term σZ represents systematic departure of the computer model from "true theory:" if $\sigma = 0$ there is no departure, and so the model is "valid". Thus, the model validation problem is to test the statistical hypothesis $H_0 : \sigma = 0$ that there is no deviation of the observed data from the theory. Suitable assumptions allow determination of a likelihood function, which can be used to estimate all unknown parameters, including $\mathbf{c}$, and validation may be tested by means of a likelihood ratio test. See [14, 19, 103, 104] for details.

3.1.6 Tuning Computer Models to Physical Data

Computer simulation models of complex physical processes frequently involve parameters with no physical meaning, which therefore cannot be measured, even in principle.[21] Statistical techniques for estimating these kinds of parameters are said to "tune" the model.

If physical system is envisioned as an unknown function F satisfying

$$\text{Observed output} = F\big(\text{Observed input}\big) + \text{Error},$$

then the physical data are inputs $x_1, \ldots, x_n$ and corresponding outputs y_i given by (3.1), where e_i is the error associated with the ith observation. Both inputs and outputs may be multi-dimensional. The computer model has the general form

$$\text{Model output} = F^c\big(\text{Observed input, Model parameters}\big).$$

That is, for the same inputs $x_1, \ldots, x_n$, it produces outputs

$$y_i^c = F^c(x_i, \mathbf{c}), \tag{3.3}$$

that are also functions of the model parameters $\mathbf{c} = (c_1, \ldots, c_k)$.

Tuning the model involves selecting values for the c_i in such a manner that the model outputs have maximum fidelity to the observed outputs. One approach [14] is to view F^c as a realization of a stochastic process.

[21] Sometimes such parameters are termed *phenomenological.*

3.1.7 Statistically Derived "Fast Prototype" Simulations

Consider a (validated or tuned) computer model of the form (3.3):

$$y^c = F^c(x, \mathbf{c}^*).$$

Computational demands may prevent this model from being run on more than a few inputs $x_1, \ldots, x_n$, yet the outputs at other inputs may be of interest. A fast prototype simulation is a statistically derived, easier to evaluate, approximation $\hat{F}^c$, possibly viewing F^c as random, in the manner of [14], and using techniques for function estimation.

Fast prototypes can also be used to address model calibration issues, which arise when inputs are subject to variation but with unknown distribution. If there are physical output data available, then a fast prototype $\hat{F}^c$ can be used in lieu of F^c in order to match the parameters of the unknown distribution of the inputs to the observed data.

We now move on to discuss the relevance of these and other statistical strategies to key problems in materials performance, properties, structure and processing.

3.2 Materials Performance

Materials or component performance is concerned with predicting and improving how materials behave in service. Performance links inherent properties of materials with product design, engineering capabilities, cost and operating environment. Among the key issues are reliability, life prediction, life extension, life cycle cost, energy efficiency and safety for materials in service under varying, incompletely known operating conditions.

The fundamental issue is prediction, to which end there is particular need to understand failure modes and internal degradation. Computational modeling — of fabrication, deformation, degradation and failure — is a key tool.

3.2.1 Lifetime Prediction from Diverse Data

Materials performance is often expressed in reliability terms, with emphasis on estimation of very low failure probabilities (10^{-6}). Characterization of associated uncertainties, for use in deriving confidence, tolerance and prediction bounds, is crucial as well. The probability that a component fails in a given situation depends on properties of the material(s) from which it is made, its geometry, the techniques by which it was fabricated,[22] the load and consequent internal stresses to which is subjected, and other features of the operating environment, for example, temperature and exposure to corrosive chemicals. For some of these variables, their entire history (as opposed to only current values) is relevant.

[22] Sometimes, the major flaws in a component result from machining.

The data from which lifetimes are to be predicted vary in nature, number and quality. For ceramic components, data include measurements of properties of "laboratory" specimens and planned experiments, together with limited direct measurements of failures (or not) of the same or similar components in verification tests (in which failure is forced, regardless of the load required) and proof tests in production (in which loading approximates that anticipated in usage). Testing conditions comprise specimens and components of different sizes, varying loading conditions and multiple failure modes. Sample sizes are of the order 100 for specimens, 10–20 for verification tests, and 100 for proof tests. In addition, there may be data from nondestructive testing of components in service.

Properties data are linked to component performance by a model. Symbolically, the component failure probability is

$$P_c = F\Big(P, \sigma(\cdot, S, E), E\Big). \tag{3.4}$$

Here, P comprises materials properties,[23] E represents environmental variables, and $\sigma = \sigma(x, S, E)$ is the internal stresses within the component, which given by a function of location x, of environment and of external stresses and component geometry, denoted collectively by S. In (3.4), F is a function that models component reliability. Its form may or may not be known. The "external" variables P, E and S may be unknown or random, may vary over time, and in any event are observable only partially. The model (3.4) incorporates known or putative failure mechanisms, and may also entail scaling assumptions (e.g., those associated with Weibull moduli and inherent strength [56, 57]). There may be embedded numerical calculations of internal stress distributions (via finite element codes) and of environmental effects.

Thus, there is need for physically confirmed models, sound statistical tools, efficient algorithms and computing power. Among applicable statistical strategies are:

- Design for physical and numerical experiments;
- Methods for combining information;
- Bootstrap, Gibbs sampling and likelihood ratio techniques for characterizing uncertainties associated with reliability estimates;
- Nonparametric methods for estimation of functional relationships;
- Techniques for "tuning" complex computer models to physical data;
- Statistical approximation to provide fast prototype simulations.

These strategies can also inform the experimental process, answering such questions as: What is the right data? What are the right experimental techniques?

Models are needed as well that incorporate heterogeneity, for predicting performance of materials, especially ceramics, whose microstructure is varied deliberately over different regions of a component. In the context of (3.4), the materials properties P become functions of x.

[23] Themselves a function of microstructure; see §3.3.

3.2.2 Models of Failure and Fracture

The links between materials properties (measured from laboratory specimens) and component performance are micromechanical models of the failure process, represented in (3.4) by the function F. Some of these are analytic, but most are computerized simulations, some of which are stochastic. Failure models are ordinarily phenomenological in nature,[24] and are based on parameters (e.g., Weibull modulus) associated with the models themselves rather than direct observation.

Statistical strategies can be used to address important needs for models that

- Depict additional failure modes for materials, such as ceramic components and *in situ*-toughened ceramics, to which "pure" weakest link scaling[25] is inapplicable;
- Incorporate heterogeneity of components, especially ceramics, in which microstructure is varied intentionally over different regions;
- Accommodate time dependence of materials parameters.

These strategies include semiparametric and Cox-type models of tails of performance distributions; alternative failure models [129, 135]; model validation; tuning of computer models to physical data and statistical approximation to provide fast prototype simulations.

3.2.3 Environmental Effects and Degradation

For ceramics and other brittle materials that fail catastrophically, modeling and assessing environmental effects and concomitant degradation become critical. Component reliabilities may depend not simply on current loads and environmental conditions, but on entire *time histories* of these processes. Incorporating histories converts (3.4) to

$$P_c = \tilde{F}\big(\mathcal{P}, \sigma(\cdot, \mathcal{S}, \mathcal{E}), \mathcal{E}\big), \tag{3.5}$$

where $\mathcal{P}$, $\mathcal{S}$ and $\mathcal{E}$ are the time histories of materials properties, external stresses and the environment, none of which is known completely, even *ex post facto*, and where $\tilde{F}$ is a more complicated version of the function F in (3.4).

Further complications ensue from feedback effects of loading and environment on materials properties.

The key issue remains *prediction of performance given available information*, including that from nondestructive evaluation of components in service. Such prediction requires statistical strategies such as stochastic modeling of environmental processes and their effects on materials properties (see also §3.5), function estimation, dimension reduction (the function $\tilde{F}$ in (3.5) is infinite-dimensional), validation and tuning of computer models and combining information. Observability of load and environment processes must be modeled as well. Statistical strategies are needed to inform experimental design and nondestructive evaluation.

[24] That is, they model observed phenomena of interest but are not derived from first principles.

[25] Which leads to Weibull distributions of strength.

3.3 Materials Properties

Properties are quantitative descriptors defining functional attributes of materials. They are envisioned as associated with a material itself, rather than specific objects fabricated from it. Broadly, properties include mechanical, electrical, magnetic, optical and thermal responses to external stimuli such as stresses, electric fields or thermal gradients. The workshop dealt mainly with mechanical properties.[26]

The crucial issue is to *design materials with desired properties*, as well as the processes by which they are produced. This can be done by relating properties to microstructure, and *designing the microstructure*.

3.3.1 Prediction of Properties from Microstructure

Current and foreseeable measurement, modeling and computing technology do not allow prediction of properties directly from atomic structure. An attractive alternative is to relate statistical features of microstructures to macroscopic properties of materials, by means of *microstructure–property relations*. This is feasible because, unlike atomic-level structure, microstructure, which is intermediate between the atomic and continuum levels, is accessible via experiment, and tools can be developed to describe and simulate it (§3.4).

Properties are a function of microstructure and environmental variables such as temperature. Symbolically,

$$\text{Properties} = F\big(\text{Microstructure}, \text{Environment}\big) \tag{3.6}$$

for some high-dimensional, unknown function F.[27] Knowledge of F would identify microstructures yielding a desired set of properties under specified environmental conditions, enabling microstructural design of materials.

The principal goal is to estimate F from available data, which consist of micrographs and other physical data, properties measured from laboratory specimens, simulated microstructures and computer simulations of materials properties associated with real or simulated microstructures. Statistically, this is a context marked by complicated, high-dimensional functions and small data sets of diverse types. Available strategies include dimension reduction to identify "active" microstructural variables associated with properties of interest, which also informs the processes of physical experimentation and microstructural quantification, tuning of computer models to physical data, nonparametric function estimation, and combining data from numerical and physical experiments.

[26] For ceramics, relevant mechanical properties include mechanical strength, fracture toughness, hardness, tribological (lubricating) characteristics, and resistance to erosion and corrosion.

[27] Possible time dependence of structure and environment is considered in §3.5.

3.3.2 Validity of Simulation Models

Physical data alone are often inadequate to test validity of proposed microstructure–properties relations, and numerical experiments are required as well.

Applicable tools include models of microstructure itself, such as pixellated grain growth models [33] and other Monte Carlo models [1, 114], which can serve as input to other numerical models that represent physical processes linking structure to properties of interest.

Several simulation models relating properties to microstructure were discussed at the workshop. They link global properties, such as effective conductivity and effective elastic moduli, to local behavior, such as current densities and stress distributions. Examples of materials and phenomena they represent include fiber-reinforced composites, cement pastes, interpenetrating phase composites, scaling in fracture, fracture of disordered microstructures, sintering, granular materials and polymer fracture.

Because they are implemented on computers, these models are discrete. The primary physical relationship of interest:

$$\text{Microstructure} \Longleftrightarrow \text{Properties}$$

is realized in the model in the discrete form

$$\text{Digital Image} \Longleftrightarrow \text{Finite Elements.} \tag{3.7}$$

Despite intense investigation, many numerical models are of unestablished (and publicly questioned) validity: it is simply not clear whether they represent the structures and phenomena of interest sufficiently well to be useful for prediction or design. Resolution of these questions is one of several important statistical issues:

Discretization, which is tied intimately to the question of representative elements. As noted in (3.7), numerical models discretize structure and its evolution. Comparison with experimental data is necessary to determine whether, in each case, the level of discretization is appropriate to the properties of interest.

Model validation and tuning. Discretization affects model validation. In turn, validation leads to questions of model tuning, since the models contain (phenomenological) parameters that must be estimated from physical and numerical experimental data.

Fast prototypes. With embedded, repeated finite element computations, many of these models, especially those also incorporating randomness in structure or evolution, are too complex to permit more than a few runs. Statistically derived fast prototypes aid in evaluation and use of such models.

3.3.3 Microstructural Design of Materials

Designing materials at the microstructural level, by means of statistically derived microstructure–property relations, holds promise of attaining desired properties systematically (rather than fortu-

itously) and, ultimately, of optimizing properties.

In conceptual terms, microstructural design involves the inverse of the function F in (3.6):

$$\text{Requisite Microstructure} = F^{-1}\big(\text{Specified Properties, Anticipated Environment}\big). \tag{3.8}$$

In order that (3.8) be useful, the microstructural characterization must be low-dimensional and be based on variables that can be manipulated during processing and fabrication.

Since even F itself must be estimated statistically, estimation of F^{-1} is even more difficult, and is complicated by the inaccessibility of F^{-1} as a function of environment. Moreover, the computational demands entailed by (3.8) are almost inevitably severe.

Beyond establishing microstructure–property relations themselves, it is necessary to develop *design tools* embodying them. Most of these tools will be software packages. In addition to questions of "user friendliness," statistical issues of visualization and interpretation of complex data (e.g., three-dimensional microstructure and multiple properties) must be addressed [122, 123].

3.4 Materials Structure

The most salient characteristics of materials structure are its *complexity* and its *hierarchical nature*. Structure is simultaneously interesting, important and experimentally accessible at levels from the atomic to the component (or object, or structure). Depending on the level, relevant physical theories range from quantum physics to thermodynamics to continuum mechanics.

For metals and ceramics, interest focuses on the *microstructure*, centered at the scale of 10^{-6} m. As depicted in Figure 3, the microstructure of a real material is a complicated arrangement of grains, surfaces, interfaces and defects, involving phase volume fractions, orientations, sizes, shapes and spatial distribution of phase domains and connectivity of phases. A metal, for example, typically has grains, grain boundaries, precipitates of second phases, dislocations and solute elements. For ceramics, defects in the form of porosity and microcracks (both deliberate and inadvertent) are present as well.

Materials structure is linked intimately with experimental data. Understanding and characterizing microstructure depends heavily on instruments for measuring and probing structure, and equally on statistical methods for analyzing, comprehending and visualizing the resultant data. By far the most salient aspect of that data is that *microstructure is three-dimensional, but virtually all of the data is* (at most) *two-dimensional*. In particular, the data depicts surfaces, whereas materials properties are driven by "interior" as well as surface structure.[28]

3.4.1 Characterization and Quantification

Techniques are needed in order to

- Identify low-dimensional, statistically-based characterizations of microstructure;

[28]Neither can be neglected. For example, surface and interior flaws induce different failure modes in ceramics.

- Determine whether two microstructures differ, and to characterize the nature of the difference;
- Determine whether important physical properties, e.g., anisotropy, are present in a given material or microstructure, and to what extent;
- Develop improved simulation models of microstructure. These may then be linked to other models that simulate response to external factors, such as stresses, in order to elucidate microstructure–property relations.

Some specific statistical issues are:

Inferring three-dimensional microstructure from two-dimensional data. Stereology [55, 98, 116, 117] provides procedures to infer some aspects of three-dimensional structure via analysis of lower-dimensional data, such as transects, slices and projections.[29]

Prior emphasis has been on mean behavior of functionals of shape, such as grain volume, number and surface area, total surface curvature, line length and curvature, and number of triple points. Recently, interest has turned to second-order and higher properties, which provide a more refined description of the material. Continued development of these techniques is necessary.

Other statistical strategies combine "conventional" stereology with stochastic geometry and morphology to yield "direct" averages of shapes [125], from which functionals can be computed. That is, rather than[30] the customary approach that microstructural features are averages of functionals of structure, features can be taken as characteristics of an "average" structure:

$$\text{Microstructural features} = \text{Functionals of averages of structure.}$$

Differences between averages of functionals and functionals of averages will also be informative.

Inference for topology (e.g., connectivity of phases in multiphase materials such as ceramic composites or cements [33]) cannot be based on stereology; new tools are required.

Dimension reduction. The complexity of microstructure entails need for low-dimensional characterizations. These will be statistical in nature. This is particularly necessary for derivation of microstructure–property relations (§3.3), because dimensionality of inputs to the function F in (3.6) must be tractably small.

Informed experimentation. To date, stereology has been driven by experimental accessibility ("What can be measured easily?") and mathematical tractability ("What can be estimated easily?") more than by need ("What should be estimated in order to derive microstructure–property relations?"). Statistical identification of such relations (§3.3) creates impetus for new

[29] See §4.1.

[30] Or, in addition to.

data needs, at the same time as progress in experimentation and instrumentation may yield means of fulfilling these needs.

Even in "conventional" stereology, sampling considerations can be crucial, Statistical properties of some stereological estimators derive from randomness in the sampling process rather than assumed randomness in the material structure (either within one specimen or from specimen to specimen). Populations of probes (lines, planes) must be defined and sampled carefully. See §4.1.

3.4.2 Models of Microstructure

Numerical experimentation for derivation of microstructure–property relations and development of tools for microstructural design require stochastic models of microstructure. Marked point processes [59] are prime candidates for bases of such models, which may then be linked to computer models that subject the material to stresses or other environmental influences. For example, crack growth and fracture [112, 136, 137] can be analyzed in this manner. Design of numerical experiments for the computer models is needed for selection of informative sets of input parameters. Fast statistical prototypes are needed when many runs of one of the linked models are required; models must be tuned (calibrated) to physical data; validation of the models is essential.

Microstructural modeling involves issues of particular importance:

Hierarchical aspects. Even at the scale of microstructure, the hierarchical structure of materials is evident, with length scales of interest spanning several orders of magnitude. Characterization and quantification are necessary over several length scales simultaneously. This raises issues of data relevance, compatibility and combination and of model linking.

Representative elements and local variation. To set of microstructural features, there correspond representative elements of a material, over which one can average in a given sample (e.g., a micrograph) in order to derive a statistical summary. At one level, average grain "size" may suffice, while at another, grain "sphericity" may be required as well. Additional phenomena, such as fracture of ceramics, are thought to be driven primarily by local variability, and no averaging may be possible. In every case, of course, there is trade-off between the simplification associated with representative elements and concomitant loss of information regarding variability.

Heterogeneity. At sufficiently small length scales, most materials other than single crystals are heterogeneous. Indeed, microstructural characterization is aimed at reducing heterogeneity to manageable proportions. Some materials, however, such as ceramic matrix or metal matrix[31]

[31] Such composites consist, for example, of single crystal ceramic whiskers, ceramic fibers or ceramic particulates embedded in a matrix of controlled microstructure. The "fibers" provide strength, stiffness and fracture toughness, and are bound together in proper orientation by the matrix. Composites of a ceramic fiber in a metal matrix have mechanical properties superior to those of the individual phases. Ceramic fibers are very strong at short lengths, but

composites, are deliberately heterogeneous, even at relatively large length scales. Models are necessary that represent relevant heterogeneity.

Micromechanical models of interfacial structure and processes. For metals and ceramics, properties of interest are driven by the structure of grain boundaries and other interfaces, or by their evolution during processing or in service. Spring network models [20, 21, 100] represent grain boundaries in a simplistic but nevertheless useful manner; however, details of geometry and energy considerations associated with crack formation and propagation are suppressed. Improved models of microstructure incorporating micromechanical properties of grain boundaries, crack bridging, and detailed treatment of energy are needed. These will be computer simulations, which require validation and tuning to experimental data. The computational burdens may be enormous, and statistically derived fast prototypes may be appropriate.

In the same way that most microstructural data is two-dimensional, so are most models depicting interfacial processes. Three-dimensional models, for example, of nonplanar crack growth, are needed; however, their development has been retarded by lack of three-dimensional data and extreme computational demands. Both of these issues can be addressed statistically, the former as described below and the latter via fast prototypes.

3.4.3 Fracture Surfaces

Inference from fracture surfaces (based on *fractographs*, such as Figure 4) presents additional challenges and opportunities. Data from fracture surfaces are of interest as another basis of inference for microstructure, and may also yield information about the nature and mechanisms of failure.[32] Fracture surfaces are very complex [74], rendering dimension reduction more necessary and difficult than for planar sections. At the same time, further summary parameters become available, such as surface roughness, an important feature of the fracture behavior of concrete [66].

Most stereological procedures assume the sampling mechanism to be independent of the material,[33] which is not the case for fracture surfaces. For inference about material-wide microstructural characteristics, this may cause problems. However, if fracture occurs largely along grain boundaries, then a fractograph may be very informative about grain shapes. For inference about fracture itself, of course, fracture surfaces are mandatory.

3.4.4 Other Data Issues

Several other issues are associated with microstructural data:

---

have severe size effect. Metal matrices, though less variable, may have lower stiffness and yield strength, and be susceptible to severe creep at high temperatures. The composite can have high strength, acceptable variability, high creep resistance and reasonable fracture toughness.

[32] Inference from fracture surfaces is relevant as well to component reliability (§3.2), where it is of interest to reconstruct the temporal development of fracture from the fragments of the failed component.

[33] See §4.1.

Three-dimensional data. As noted previously, most visual microstructural data is two-dimensional. This remains literally true even in cases where two-dimensional data contains more-or-less direct information about three-dimensional structure, as in Figure 5 or fractographs (Figure 4). Ultrasound tomography, used in nondestructive evaluation [102], yields "genuinely" three-dimensional data, albeit not at resolutions relevant to microstructure. It is important to devise methods for collection and analysis of three-dimensional data.

Inexactness in digital image data. Digital micrographs are noisy and discretized, so that essential features may be obscured or lost. Image analysis can be useful for obtaining enhanced visual data [28, 101, 102, 134], but has seen only limited application. Strategies are necessary to attenuate effects of noise and discretization, and to characterize their propagation through analyses and models.

Edge effects. Awareness of edge effects in visual data[34] is not new. Nevertheless, stereological and related procedures making full use of information at the edges of visual microstructure data need further development.

Combining multiscale data. Integrating visual data from two or more different modalities falls under the general rubric of image registration, which has been studied previously in the context of medical imaging. For microstructure, the problem is more complicated (and the potential usefulness enhanced) because different forms of data may represent different length scales.

3.4.5 Experimental Needs

The logical flow between experiment and instrumentation, on the one hand, and description and quantification of microstructure, on the other hand, is not unidirectional, even though historically descriptions of structure have been driven by available experimental data. Identification of key statistical descriptors of structure, via derivation of microstructure–property relations will drive data collection and development of instrumentation.[35] To some extent, this is happening already: various models have defined needs for

Three-dimensional measurements of structure, which will facilitate validation and tuning of models of microstructure, or detection of specific features of microstructures, such as anisotropy. Among these are measurements of connectivity and other topological properties that cannot inferred from two-dimensional data.

Data obtained nondestructively, which are of particular importance in regard to materials and components in service, in order to assess environmental effects and predict future properties.

Enhanced resolution, which is, of course, a perpetual need. In the future, it may be driven mainly by increased modeling sophistication and computational capabilities.

[34] Especially in other contexts, for example, spatial statistics.

[35] The latter is noted in [77] as neglected in the U.S.

Measurements of local variation, such as microstress, which at the scale of grain size are highly non-uniform, as well as residual stress and grain orientation.

Data involving interfaces. The crucial role of interfaces and grain boundaries has been noted in §3.4; however, relatively little "direct" data regarding interfaces and their characteristics is available. Grain boundaries appear as edges in digital microstructural data, which makes them particularly susceptible to noise and distortion introduced by image processing.

3.5 Materials Processing

Materials processing and synthesis are identified in [77] as weaknesses of materials science and engineering in the United States.

Materials processing is the control of structure to produce materials with desired properties and, ultimately, components with desired performance. It is an enabling activity. Processing is typically effected by means of macroscopic manipulations — for example, application of stress, pressure or temperature — that cause microstructural changes. Metal alloys, for example, can be created by melting or mechanical alloying or deposition techniques. Subsequent processing can include hot or cold rolling, forging, extrusion and thermal treatments. Other examples are grain growth and other aspects of origins of microstructure, coarsening, sintering of ceramic powders, recrystallization, directional grain growth, phase transformations during annealing, particle stacks and spinodal decomposition. Since processing entails control and manipulation of interfaces, better understanding of interfacial structure and processes is required.

In the same way that microstructural design (§3.3) uses microstructure–property relations to tailor materials properties, materials processing can be viewed as requiring analogous *process–property relations*, of the generic form (compare (3.6))

$$\text{Properties} = H\big(\text{Initial microstructure, Processing history, Environment}\big). \tag{3.9}$$

This formulation is, however, too general. Two more specific approaches — *microstructural evolution* and *constitutive models* — are feasible and useful.

3.5.1 Microstructural Evolution in Processing

Building on microstructure–property relations, one can predict time-varying behavior of properties by tracking microstructural evolution, that is, changes over time in microstructure.[36] Understanding and modeling this evolution enables prediction of materials properties.

In the context of materials processing, at an elementary level, one can build on (3.6). With t representing time, $P(t)$ properties at t, $M(t)$ microstructure at t, a simple model would take the

[36] We use the term generally, to connote development of microstructure during synthesis as well as changes to extant microstructure during processing or in service.

form

$$P(t) = F\big(M(t)\big), \tag{3.10}$$

where F is (effectively) the same function appearing in (3.6). This assumes no dependence of properties on processing history, which is plausible initially, if not ultimately. Otherwise a variant of the form

$$P(t) = F\big(M(t), \mathcal{S}(t)\big) \tag{3.11}$$

is necessary, where $\mathcal{S}(t)$ processing history (compare (3.5)) at t, representing, for example, stress, temperature and pressure.

If microstructure changes qualitatively, as in *in situ* toughening, then a "different" function F and a more complicated M may be necessary in (3.10).

If F has been estimated already, the sole issue is to model microstructural evolution, which might be done via a "evolution equation" (cf. (3.14))

$$M'(t) = G\big(M(t), \mathcal{S}(t)\big). \tag{3.12}$$

In some contexts, a "processing state" $S(t)$ might suffice in place of the history $\mathcal{S}(t)$.

Most models of microstructural evolution are computer simulations [1, 33, 35, 114, 136]. They might involve constitutive equations (discussed momentarily), cellular automata, Monte Carlo methods, time-dependent Ginsburg–Landau theories with random noise or tesselation-based models.

Materials science issues and statistical strategies germane to microstructural evolution include:

Models of microstructural evolution, corresponding to the function G in (3.12). These will be computer simulations based on stochastic micromechanical models of relevant interfacial processes. Their construction requires function estimation, dimension reduction, model validation, tuning of computer models to physical data, fusion of data from physical and numerical experiments and fast prototype simulations, the latter because of the complexity of the computer codes.

Models of processing states, that is, of the process $S(t)$ in (3.12). These may be constitutive models (discussed momentarily). In addition to issues of stochastic models, function estimation, dimension reduction, model validation and model tuning, the process $S(t)$ is only partially observable. The partial observability itself, as well as its consequences, must be modeled.

Feedback effects. Implicit in (3.10) and (3.12) is an assumption that properties do not feed back to modify the microstructure. There are cases in which this assumption fails: for example, deterioration of concrete depends on the interaction between permeability and microstructure [75, 119]. In other cases, an external stimulus causes microstructural or morphological evolution that alters the response of the material to the original stimulus [137].

Process design. Tools to design processes yielding desired or optimal properties are necessary in order that progress on microstructural evolution affect manufacturing practice. Ordinarily these will be realized as software packages. "User-friendliness," especially insofar as it pertains to display and interpretation of complex data [122, 123] is of particular concern.

Models that incorporate cost (see §1.3.1) are needed; essentially none has been developed to date.

3.5.2 Constitutive Equations

Constitutive equations relate time-varying stresses applied to a material to resultant properties. The ultimate engineering goal is to produce components with desirable characteristics.[37] In some ways, they may be construed as particular realizations of the evolution equation (3.12).

A set of constitutive equations consists of state and evolution equations. A *state equation*, of the generic form

$$P(t) = F\big(T(t), \varepsilon'(t), S(t), \theta\big), \tag{3.13}$$

expresses a vector P of materials properties as functions of controlled, external variables, such as temperature T and strain rate ε' (the time derivative of strain), a vector S of "internal variables," which represents microstructure, and a vector θ of unknown parameters characterizing the specific process.

The internal variables satisfy an *evolution equation*

$$S'(t) = G\big(T(t), \varepsilon'(t), S(t), \theta\big). \tag{3.14}$$

They serve as surrogates for real phenomena occurring during processing, may have no specific physical significance, and are not necessarily measurable, even in principle.

Constitutive equations are viewed as valid pointwise, at representative small elements of the microstructure. The qualitative forms of F and G are dictated by the laws of mechanics, but are not necessarily available as analytical expressions.[38] Instead, they may be embedded in computer models, such as finite element algorithms.

The observed data are experimentally measured time trajectories, sampled at discrete times, of P, T and ε'; in some cases, only final values are known. The observations are subject to noise and measurement error.

Specific issues include:

Estimation of θ, a parametric estimation problem, but not standard because of the unusual nature of the data and the absence of analytical expressions for F and G. Variability of estimators is of particular concern, as are model validation and tuning.

[37] For example, ultimate properties of a metal undergoing extrusion, forging or rolling are strain, deformation and micro-hardness.

[38] In this *parametric* formulation of the problem, F and G are of known form, but involve the unknown parameter θ. *Nonparametric* alternatives are discussed momentarily.

Prediction, in the direct sense of predicting properties associated with input values other than those treated experimentally, and also in the *process design* sense of determining values of the processing variables $T(t)$ and $\varepsilon'(t)$ leading to prescribed properties.

Identification of internal variables. In the example of [105], there are two internal variables. One represents the combined effect of all dynamically recoverable defects (which disappear if stress is removed or the material is heated), and the other, the combined effect of microstructural features that cannot be recovered during deformation, such as permanent hardening of the material. Statistical strategies can identify effective, low-dimensional internal variables. Recent advances in computational methods allow incorporation of experimental data.

Nonparametric models, which posit only that the functions F and G in (3.13) – (3.14) belong to some large, qualitatively defined class of functions, rather than treating them as known except for the value of θ. These models have the advantage of being more flexible and powerful, but may be ineffective when there is only limited data.

3.5.3 Environmental Degradation

The response of materials to deterioration[39] is similar conceptually to processing. Under load or environmental stress, the microstructure of a material changes, and so do its properties. Microstructural mechanisms by which materials in service deform, degrade and fracture, for example, contact damage, metal creep, fatigue and corrosion, are central processes to be understood and modeled.

By analogy with (3.6) and (3.10), a basic model takes the form

$$P(t) = F\big(M(t), \mathcal{E}(t)\big), \tag{3.15}$$

where $P(t)$ denotes materials properties at t, $M(t)$ is microstructure at t, and $\mathcal{E}(t)$ is the environmental *history* at t, including loads as well as "external" effects. Evolution of microstructure and the environment must be modeled as well: symbolically,

$$M'(t) = G\big(M(t), \mathcal{E}(t)\big) \tag{3.16}$$

$$\mathcal{E}'(t) = H\big(\mathcal{E}(t)\big). \tag{3.17}$$

Feedback effects, which would convert (3.16) to

$$M'(t) = G\big(M(t), \mathcal{E}(t), P(t)\big), \tag{3.18}$$

may be important, too.

Issues resemble those for microstructural evolution, but while some physical processes involved with processing and degradation are similar, the data and models are quite different.

[39] A crucial aspect of the nation's problem of deteriorating physical infrastructure [25].

Modeling of environmental processes, corresponding to the process $\mathcal{E}(t)$ and the function H in (3.16). Stochastic models of environmental conditions require dimension reduction to identify key variables, function estimation and model validation, all of which must account for the incomplete observation of the environment.

Measurement of properties and microstructure. For materials in service, ordinary measurements of microstructure and properties may not be possible. Components may be physically inaccessible, or carrying out the measurements might require removing them from service or destroying them, neither of which may be allowable. Instead, emphasis must be on data obtained via nondestructive evaluation, which may differ qualitatively from that discussed previously. Even "classical" questions, such as characterization of variability of measurements, require novel statistical strategies. Data from diverse sources must be combined. Issues of experimental design ("What should be measured?" "How should inputs be varied?") are relevant as well.

Modeling of degradation, as represented by the functions F in (3.15) and G in (3.16) involves function estimation, dimension reduction, model validation and tuning of computer models.

Several processing-related questions with significant statistical content were not addressed at the workshop:

- Materials synthesis, the chemical and physical means by which atoms and molecules are assembled to produce (new) materials, albeit similarly dependent on microstructural manipulation, operates on a smaller scale than processing, and often need not address of issues of efficiency, cost and control. Statistically derived process–property relations, supplemented by techniques of experimental design, would enable rapid synthesis of new materials with desired properties, lessening the level of "guesswork" involved.
- Novel, improved production techniques for extant materials and machinery for processing.
- Robust, nondestructive sensors and process control models and methods.

Dimension	Feature	Property	Classes
3	Grain	Volume Number Connectivity	α, β
2	Surface	Area Total Curvature Spherical Image	$\alpha\alpha, \alpha\beta, \beta\beta$
1	Line	Length Dihedral angle Curvature	$\alpha\alpha\alpha, \alpha\alpha\beta, \alpha\beta\beta, \beta\beta\beta$
0	Point	Number	$\alpha\alpha\alpha\alpha, \alpha\alpha\alpha\beta, \alpha\alpha\beta\beta, \alpha\beta\beta\beta, \beta\beta\beta\beta$

Table 1: Microstructural Features

4 Quantification and Inference for Microstructure

This section and the four that follow contain details of the presentations at the workshop. They are organized in the "scientific" order of microstructure, microstructure–property relations, materials performance and materials processing.[40]

4.1 Stereology

Professor DeHoff's talk, "Stereology and the Quantification of Microstructural Geometry," an overview of the subject, focused on microstructural features, stereological measurements, stereological relationships and sampling design strategies.

4.1.1 Microstructural Features

Consider a two-phase granular material with phases α and β. Key microstructural features may be categorized as in Table 1. The classes form a *co-dimension*: grains are only α or β, a grain boundary can between two α grains, two β grains or one of each, (and so has a 2-dimensional classification) a triple line, along which three grains meet, can be of four types (and has a three-dimensional classification), and quadruple points, of five kinds, have a four-dimensional classification. Interpretation of these as microstructural features entails an implicit "per unit volume" qualification. For the α phase, the grain volume feature is the *volume fraction* v_α, that is, the fraction of the volume of the material comprised of phase α.[41]

These features are illustrated in Figure 6. It is important to understand that since this figure is two-dimensional, only features of co-dimension 2 or lower can be shown, and each feature loses

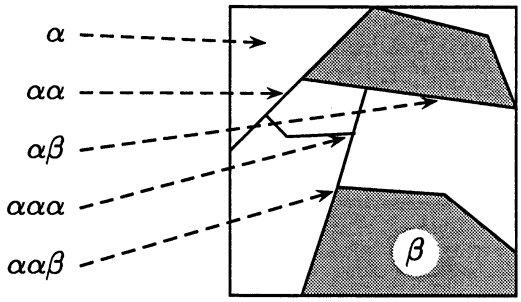


Figure 6: Classes of Microstructural Features

Probe	Example Counting Measurement
Point	Point counts of different phases
Line	Line intercept count of grain boundaries
Plane	Area point count Feature count Tangent count

Table 2: Stereological Probes and Counting Measurements

one dimension in the process of projection. Thus, in two-dimensional data, quadruple points are not observed at all, grain boundary surface becomes a line and a triple line becomes a point.

4.1.2 Stereological Data

Three-dimensional microstructure is (in general) not accessible directly; rather, it is sampled by means of *stereological probes*:

- *Volume probes*, parametrized by 3-dimensional position;
- *Plane probes*, parametrized by 2-dimensional orientation and 1-dimensional position;
- *Line probes*, parametrized by 2-dimensional orientation and 2-dimensional position;
- *Point probes*, parametrized by 3-dimensional position.

In reality, most microstructural data is two-dimensional, sampled with *point probes*, *line probes* and *area probes*. These probes provide various *stereological counting measurements*, some of which are given in Table 2. Data of any of these sorts ordinarily represent *repeated probes of the*

same microstructure.

Not all data is "complete." Edge effects, which arise because samples of microstructure are finite in extent, are a particular form of incompleteness, and have not, in general, been dealt with successfully.

4.1.3 Stereological Relationships

The link between stereological data and microstructural features is provided by stereological relationships, which are derived from assumptions regarding the nature of the material, the sample and the sampling process.

To consider one example, the volume fraction v_α can be estimated from point probe data. Let N be the number of point probes and N_α the number of those yielding phase α. Then, N_α/N is a plausible estimator of v_α, and, under certain hypotheses, is unbiased:

$$E[N_\alpha/N] = v_\alpha. \tag{4.1}$$

The hypotheses underlying (4.1) pertain mainly to the sampling mechanism. They are generically to the effect that "the population of (possible) probes has been sampled uniformly" and that the (planar section) data are "representative" of the three-dimensional microstructure. For probes selected randomly, this means that the expectation in (4.1) is solely with respect to the sampling mechanism.[42] The particular microstructure is taken as fixed rather than sampled from some class of microstructures, and v_α is regarded as simply an unknown physical constant.

If probes are selected systematically (to "span" the space of probes), but the microstructure is still taken as fixed, then it is unclear how to interpret the expectation in (4.1).

When individual microstructures are regarded as sampled from a class of microstructures (one possible definition of a "material"), then the volume fraction becomes a random variable V_α, whose mean v_α is a characteristic of a material rather than objects. Under uniformity assumptions regarding probes, (4.1) is valid conditionally on V_α:

$$E[N_\alpha/N|V_\alpha] = V_\alpha, \tag{4.2}$$

and if sampled microstructures are representative[43] of the material, in the sense that

$$E[V_\alpha] = v_\alpha,$$

then once more (4.1) holds.

[40] As opposed to the "engineering" order of performance, properties, structure and processing.

[41] To the extent that these features are taken as features of a material, rather than a particular sample, issues of representative elements and local variation (§3.4) are germane.

[42] The possibility that different parametrizations of the set of probes engender different manifestations of uniformity seems to be neglected.

[43] See the discussion of representative elements in §3.4.

4.2 Stochastic Geometry: Average Shapes

Professor Vitale, in his talk "Stochastic Geometry," described an approach to microstructural quantification in which the

$$\text{Features} = \text{Averages of functionals of structure} \tag{4.3}$$

paradigm inherent in stereology (embodied in (4.1), for example) is supplanted or supplemented by

$$\text{Features} = \text{Functionals of averages of structure.} \tag{4.4}$$

Some versions of (4.4) are based on a concept of *average shape*. To illustrate, one measure of size for a single grain G is the effective radius $r(G) = \text{vol}^{1/3}(G)$. To estimate $r(G)$ using the approach of (4.3), suppose that from grains $G_1, \ldots, G_n$ in three dimensions one samples not the "true" effective radii $r(G_1), \ldots r(G_n)$, but instead stereologically derived estimates $\hat{r}(G_i)$. Then, an estimated value for the effective radius is

$$\overline{r} = \frac{1}{n}\Big[\hat{r}(G_1) + \ldots + \hat{r}(G_n)\Big].$$

To employ (4.4) is this context, Minkowski addition [72] of sets can be used to form the *mean grain*

$$\frac{1}{n}[G_1 + \ldots + G_n] \stackrel{\text{def}}{=} \left\{\tfrac{1}{n}[x_1 + \ldots + x_n] : x_1 \in G_1, \ldots, x_n \in G_n\right\},$$

and its data-dependent version

$$\overline{G} = \frac{1}{n}\Big[\hat{G}_1 + \ldots + \hat{G}_n\Big],$$

where the $\hat{G}_i$ are stereological estimates of the grain shapes themselves. Then, the effective radius $r(\overline{G})$ can be computed. This provides a different summary parameter for the sample, since [126]

$$\overline{r} \leq r(\overline{G}). \tag{4.5}$$

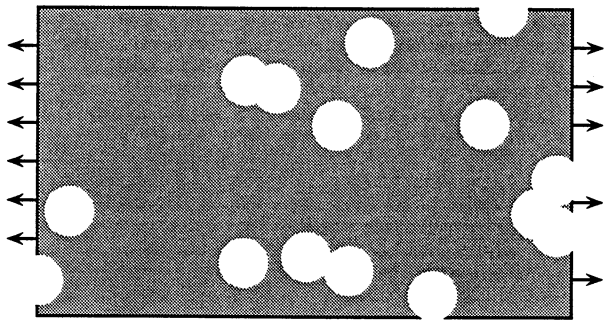
The "gap" $r(\overline{G}) - \overline{r}$ yields information about possible anisotropy in the material: it is larger when grains are oriented randomly than when the material is anisotropic.

Similarly, lower bound in the inequality

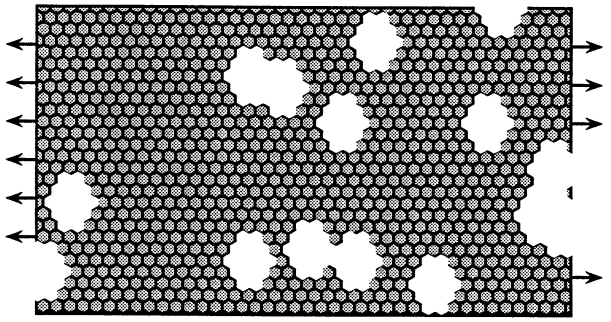
$$\overline{r} \leq \overline{\text{vol}}^{1/3} \stackrel{\text{def}}{=} \left(\frac{1}{n}\Big[\text{vol}(G_1) + \ldots + \text{vol}(G_n)\Big]\right)^{1/3}. \tag{4.6}$$

is identical to that in (4.6); comparing their upper bounds leads to the functional $\overline{\text{vol}} - \text{vol}(\overline{G})$, which may provide a novel way to quantify the overall sphericity of grains within the sample.

Many extant techniques of this kind require that individual grains be convex and that grains in the sample be (stochastically) independent, neither of which is true in real materials. Research is necessary in order to overcome these limitations.



A. Continuum Version



B. Discretized Version: Hexagonal Grid with Random Holes

Figure 7: Elastic Sheets with Random Holes

5 Microstructure–Property Relations

In [81], structure–property relations are identified as one of "the four most important R&D priorities." Models are a necessary (or even crucial) component of such relations.

Professor Day, in his talk on "Microscale Elastic Simulations for Random Materials and Composites," and Dr. Jagota, who spoke on "Property Simulations via Spring Networks and Finite Element Models," discussed two key classes of large, computerized simulation models that relate properties to structure:

- Finite element models (§5.1);
- Spring network models (§5.2).

Both classes of models simulate global properties from assumptions regarding local behavior.

Computer models are not the only avenue to structure–property relations. One widely pursued alternative, with roots in the field of mechanics, is analytically derived bounds on effective properties.[44] See [119].

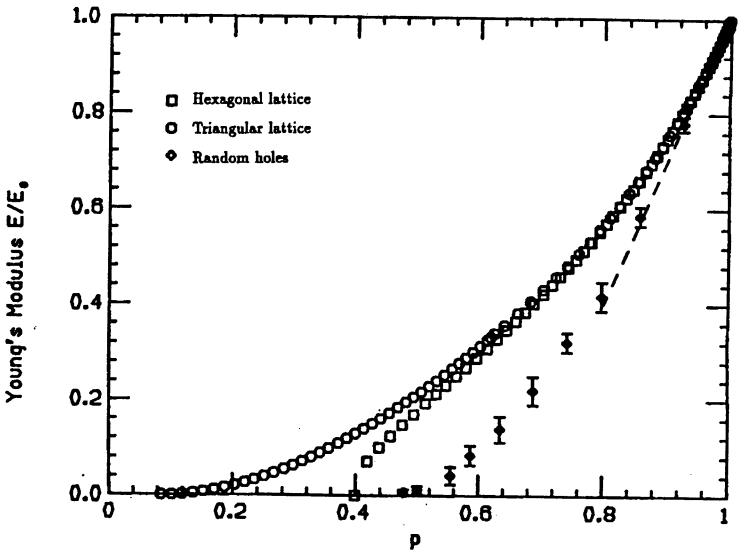


Figure 8: Young's Modulus for Elastic Sheets with Random Holes

5.1 Finite Element Models

The finite element models described by Professor Day are directed at computation of global properties of materials, for example, effective conductivity and effective elastic moduli, from simulations of their structure and its response to stress or other stimuli. Two particular models were presented, one for elastic moduli of sheets containing circular holes [24], and the other for interpenetrating two-phase composites.

These models are based on finite element computations. A continuum (for specificity, an elastic sheet subject to tensile stress) is discretized into a grid (for example, triangular, rectangular or hexagonal) of finite elements, with nodes at grid points. It is assumed that the strain ε at any interior point of an element can be calculated from the displacements u of the nodes. Then, the elastic energy of the system can be expressed as a quadratic function $E(u)$ of the displacements, and equilibrium corresponds to minimization of $E(u)$, which can be performed numerically. See [5].

5.1.1 Elastic Sheets with Random Holes

Consider an isotropic (two-dimensional) elastic sheet, perforated by randomly located holes (for simplicity, all of the same diameter), as in Part A of Figure 7, which is subjected to a tensile stress. The goal is to investigate the effective elastic modulus of the sheet as a function of the (expected) fraction of it occupied by the matrix.

The model reported in [24] discretizes using a 210 × 210 hexagonal lattice, (as in Part B of Figure 7), which is taken as the representative element in a doubly periodic system.

Results for this model are shown in Figure 8, which depicts the effective elastic modulus as a function of the area fraction p of the sheet occupied by the matrix.

5.1.2 Interpenetrating Two-Phase Composites

These models are used to investigate properties of two-phase composites in which each phase is topologically connected throughout the microstructure. Examples include porous ceramic filters, certain glasses and rocks [15] and bones.

A basic simulation model [15] functions as follows:

- A *random field* $I_0(x, y, z)$ is constructed by assigning a random number $I_0(x, y, z)$ in $[0, 1]$ to each point (x, y, z) in a 3-dimensional cubic lattice.
- The initial random field is smoothed, by convolution with a Gaussian kernel, to form another random field

$$I(x,y,z) = \frac{1}{(2\pi w^2)^{3/2}} \int\int\int I_0(x',y',z') e^{-[(x-x')^2+(y-y')^2+(z-z')^2]/2w^2}\, dx'dy'dz'.$$

  The parameter w controls the degree of smoothing.[45]
- The random field $I(x, y, z)$ is thresholded to produce the simulated two-phase material $M(x, y, z)$. There is a cutoff value $c_1 \in (0, 1)$, interpreted as the concentration of phase 1, such that $M(x, y, z)$ is assigned to phase 1 if $I(x, y, z) \leq c_1$ and to phase 2 otherwise.

Full interpenetration ("percolation") does not occur for all values of c_1, but does occur for $0.3 \leq c_1 \leq 0.7$ (approximately).

The phases have different properties, e.g., conductivity σ and Young's modulus E, and the goal is to calculate effective properties of the composite as a function of the concentration of phase 1.

The computations are done on lattice of size (on the order of) 100^3, using finite element codes, by minimizing an energy function, with the composite subjected to an electric field or stress, as appropriate. An advantage is that this permits calculation of local as well as global properties.

The results that follow pertain to composites in which phase 1 has low conductivity $\sigma_1 = 1$ and high Young's modulus $E_1 = 10$, while phase 2 has high conductivity $\sigma_2 = 10$ and low Young's modulus $E_2 = 1$.

The finite element approach allows computation, for example, of histograms of current magnitude, shown in Figure 9, with c_1 decreasing from .95 to .05. The principal peaks interchange places, yet there is not complete symmetry. When phase 1 dominates, the current magnitude is

[44] Some participants noted the relative absence of this approach among presentations at the workshop.

[45] In reality, of course, the integral is a sum.

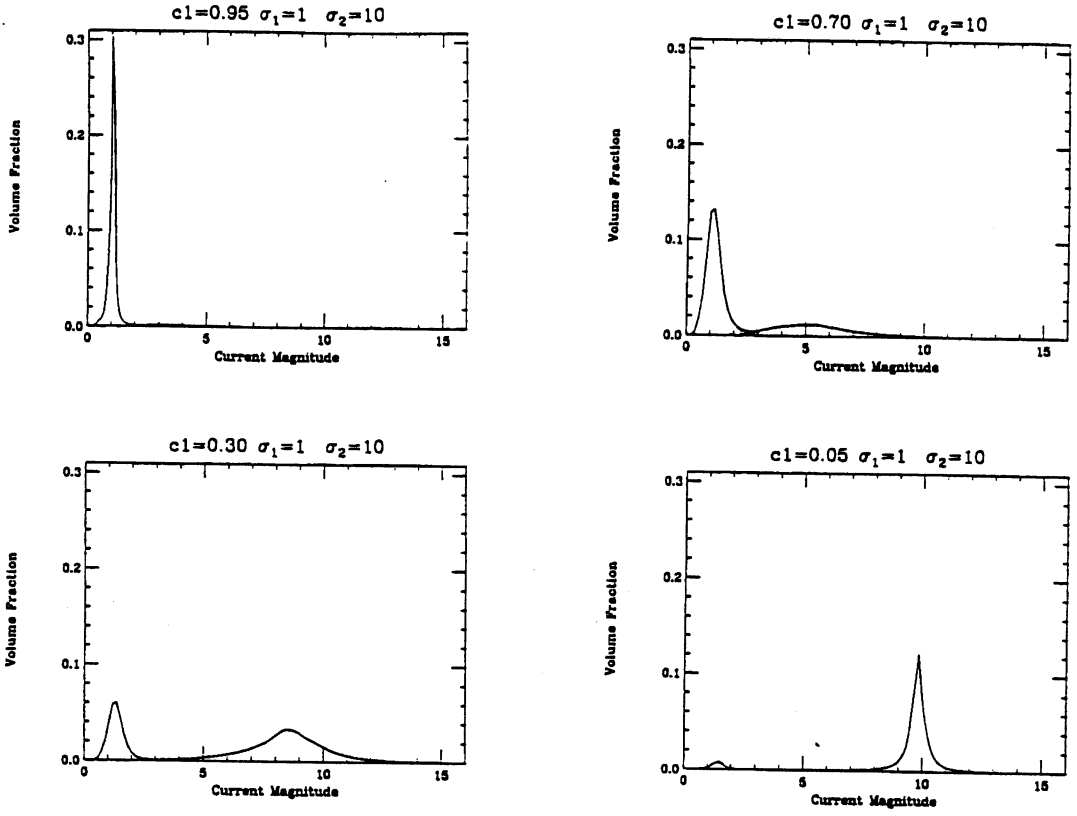


Figure 9: Current Distributions in Simulated Two-Phase Composities

peaked sharply at σ_1, but when phase 2 dominates, the peak at σ_2 is smaller and a visible peak at σ_1 remains. Strategies are not yet available to make effective use of the detailed information in these histograms.

The effective conductivity σ_{eff} is simply the mean value of the current distributions in Figure 9, and is shown in Figure 10. Model outputs accord with calculations made using effective medium theory.

Similar results obtain for the effective Young's modulus E_{eff}.

5.2 Spring Network Models

Dr. Jagota addressed the use of spring network models [20, 21, 100] in representing properties of materials, especially fracture behavior of disordered microstructures. These models represent grain boundaries (or any element of the material) as elastic springs, capable of withstanding some

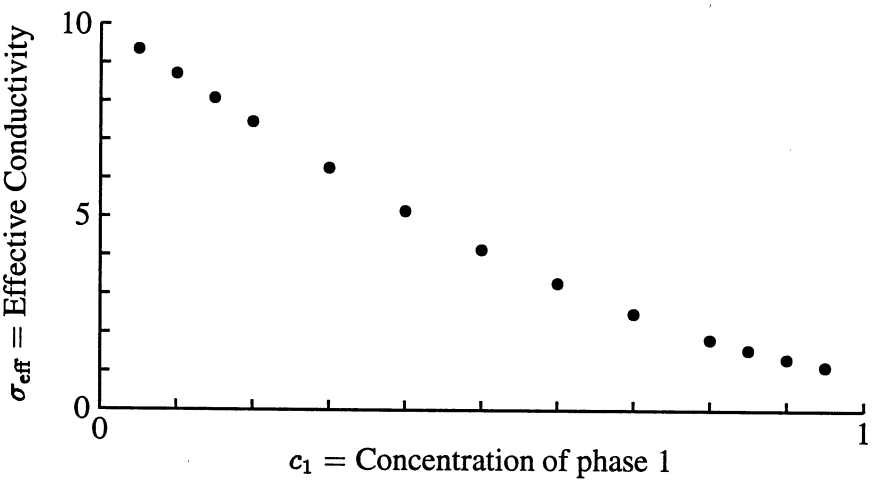


Figure 10: Average Current in Simulated Two-Phase Composities

combination of normal forces, transverse forces and moments. Springs may fracture at a critical force or a critical energy, or they may fail stochastically, based on energy, stress or strain. When a spring fractures, the resultant distribution of stresses on the remaining springs is calculated. Ultimately, the model material "fails."

These models capture many of the subtleties and issues associated with use of complex computer models in materials science.

5.2.1 Structure of Spring Network Models

Spring network models have the following general structure:

- *A digitized lattice*, such as the triangular lattice in Part A of Figure 11, along which grain boundaries lie.[46] This lattice is the basic unit of discretization for microstructure as modeled. As shown, not all lattice elements need be grain boundaries.
- *Grain boundaries*, modeled as (perfectly elastic) springs. Depending on the model, springs may resist
  - Central forces only, and be characterized by a single elastic constant k^n;
  - Both central and transverse forces (the Born model), and be characterized by constants k^n and k^t;
  - Moments, in addition to central and transverse forces;

[46] Both deterministic and random lattices have been employed.

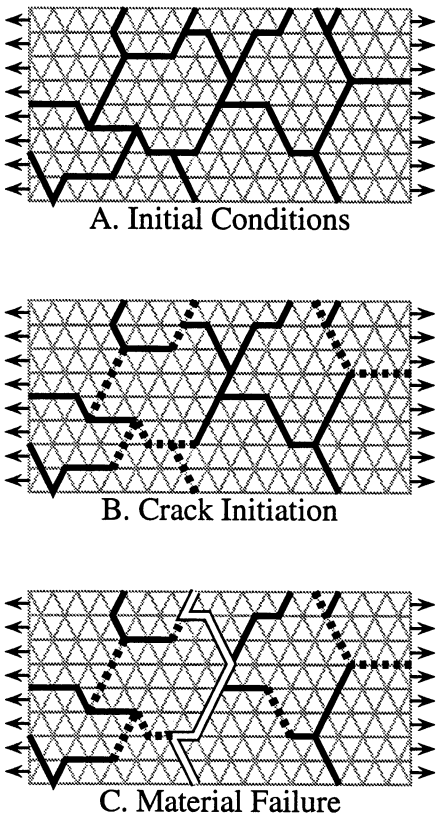


Figure 11: A Spring Network Model

– Bond bending, with adjacent intergranular springs connected by other springs.

Ordinarily, the characteristic constants vary randomly from spring to spring.

- An *external stress*, shown in Figure 11 as pure tension, but which might instead have a shear component as well.
- A *"fracture" criterion* for springs, which may be based on a critical force or a critical energy.
- *A numerical code,* which, each time a spring fractures, is used to calculate the resultant distribution of stresses on remaining springs.

As springs fracture, broken bonds become "cracks," as in Part B of Figure 11. If the stress is sufficiently high, then ultimately, the model material "fails," in the sense that it becomes disconnected, as shown Part C of Figure 11 and for a real material (alumina) in Figure 12.

In more complicated models, lattice elements not corresponding to grain boundaries may also be represented by springs (typically, stronger than those representing boundaries), which allows depiction of trans-granular fracture as well as inter-granular fracture.

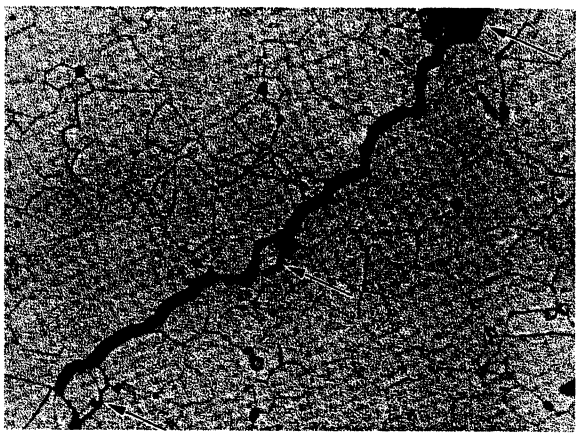


Figure 12: Microstructure of Fractured Alumina

Spring networks have been used to model a variety of phenomena, including

- General elasticity [49];
- Scaling in fracture [115];
- Fracture of disordered microstructures [20, 21];
- Sintering [53].

Most of these models are two-dimensional.

Limitations of spring network models include:

- *Computational restrictions*, which, with current capabilities, limit two-dimensional lattices to sizes on the order of 1000×1000, and effectively preclude meaningful 3-dimensional investigations.
- *Inability to depict important physical phenomena*, especially those, such as crack bridging (Figure 16), crack deflection, microcracking and crack pinning[47] that attenuate the brittle character of ceramics. All of these can be regarded as forms of heterogeneity.
- *Incomplete understanding of their relationship to continuum models.* In some cases, spring network models are meant as discrete approximations of continua.[48] Whether they are appropriate discretizations is not known.[49] Resolving this issue requires more refined knowledge of the inverse process of homogenization (passing from the discrete to the continuous) as well.

[47] These mechanisms inhibit formation of stress concentrations large enough to cause macroscopic failure [68].

[48] In other cases, they are simply exact models of discrete systems.

[49] Known difficulties exist even for the "canonical" problems of uniform strain and cracks in homogeneous, isotropic materials.

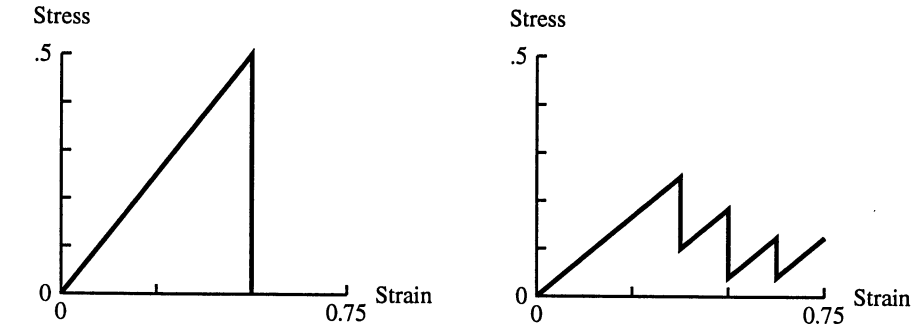


Figure 13: Behavior in a Spring Network Model as Residual Stress Varies

- *Lack of empirical validation.* Some relationships between parameters of spring network models and effective properties (for example, Young's modulus) of large networks are known, but effectively nothing has been done to validate or tune these models in the sense of §3.

5.2.2 Quasi-Brittle Behavior in Two-Phase Microstructures

One particular application is to modeling of two-phase microstructures in which the phases have different coefficients of thermal expansion. When such a microstructure is cooled, *residual stresses* are created because grains constrain each others' shapes. Roughly speaking, grains with the higher coefficient of expansion shrink more, and become subject to tensile stress, while grains with the lower coefficient become subject to compressive stress. Three parameters can be varied in the model:

- *The disorder of the microstructure*, that is, the degree of residual stress;[50]
- *The volume fraction of the phases*;
- *The scale of the microstructure*, that is, the number of grains.

Figure 13 depicts the effect of varying the disorder. Interestingly, as the disorder increases, the behavior of the microstructure changes from *brittle* to *quasi-brittle*. In the brittle case, the stress increases as the strain does until the material fractures, as in Part A of Figure 13. At this point, its stress-carrying capability vanishes. By contrast, in the quasi-brittle case in Part B of Figure 13. There, as strain increases, the material undergoes a series of "partial" failures that decrease the stress, but not to zero, and further strain is possible. In consequence, while the maximum stress is less, the ultimate strain increases.

Figure 14 shows similar effects of varying the volume fraction v of the phase under tension. When v is high, the material is brittle, but while v is low, it becomes quasi-brittle.

[50]Roughly, the mismatch between the coefficients of thermal expansion.

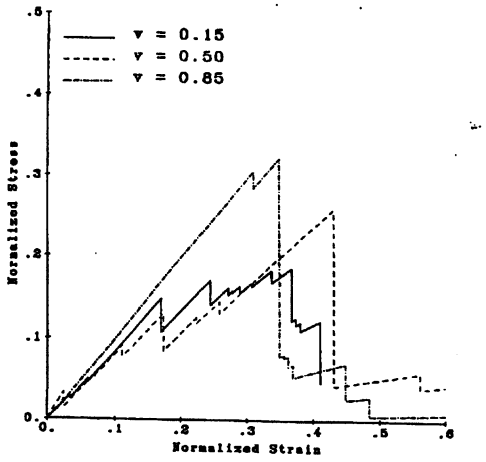


Figure 14: Behavior in a Spring Network Model as the Volume Fraction Varies

Effects of varying the scale of the microstructure remain to be elucidated.

5.3 Microstructure and Fracture Behavior

In his talk on "Microstructure and Fracture of In-situ Reinforced Silicon Nitride," Dr. Li treated the relationship between microstructure and fracture behavior for *in-situ* toughened Si_3N_4.

Silicon nitride exhibits "R-curve" behavior, as depicted in Figure 15: crack resistance increases with crack size.[51] Nevertheless, silicon nitride does not have high strength. Related qualitative properties are that

- Weibull modulus[52] of grains increases with grain size;
- Fracture toughness[53] increases with grain size.

Linearly elastic fracture mechanics is unable to account for this behavior.

Large grains are thought to be the predominant means of fracture origination in silicon nitride, because grain strength decreases with grain size. Crack growth is retarded by *bridging*, which is illustrated in Figure 16: grains lying across the path of the crack inhibit its growth, until under sufficient stress they either fail or debond (are pulled bodily away from neighboring grains). Bridging is relevant only in a *bridging zone* behind the crack tip.

[51] See Appendix C.
[52] See Appendix C.
[53] See Appendix C.

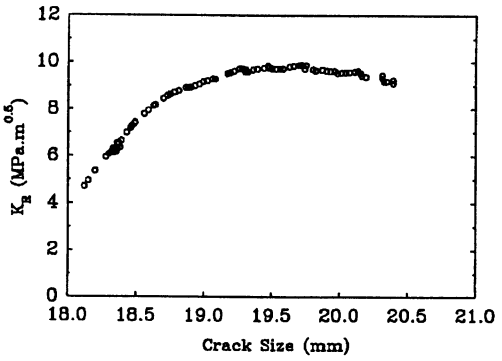


Figure 15: *R*-curve Behavior of *In Situ* Reinforced Si_3N_4

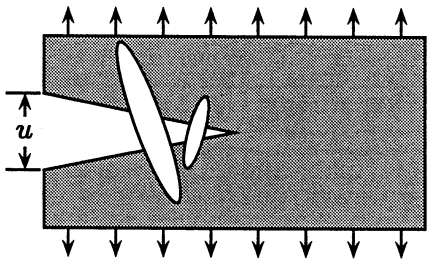


Figure 16: Crack Bridging in Si_3N_4

The talk serves as a case study of proceeding from data to a microstructure–property relation, in this case relating grain size — specifically, grain width — to two parameters describing fracture behavior:

- The *grain strength* σ_0, which is expected to decrease with grain size;
- The *debonding length* ℓ_{db}, at which the bridging zone collapses and fracture occurs, and which is expected to increase with grain size.

The data are 13 batches of Si_3N_4, of which Figure 17 is an example, each leading to approximately 50 samples, for which were recorded:

- Grain size distributions: length, width and (apparent) aspect ratio;
- Strengths, from which Weibull moduli were estimated;
- Flaw size distributions, from each of which is calculated an *equivalent flaw size* c_0.

The key grain size variable is taken to be d_{10}, the average width of the widest 10 percent of grains. This reflects the known importance of width of bridging grains in attenuating crack growth.

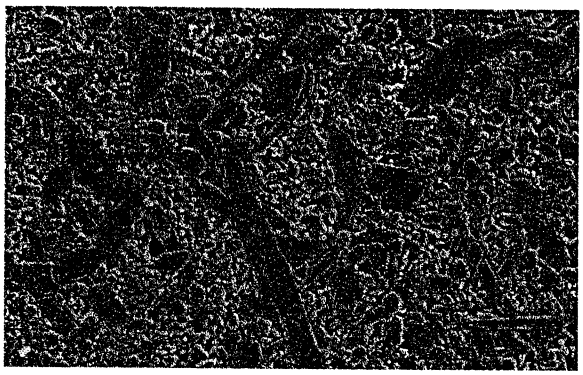


Figure 17: Micrograph of *In Situ* Reinforced Si_3N_4

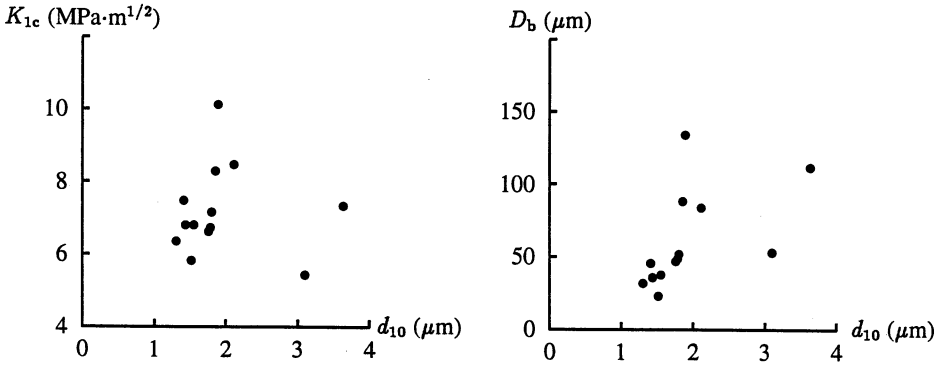


Figure 18: K_{1c} and D_b as Functions of Grain Size d_{10}

From the data, the fracture toughness K_{1c}, which peaks at an intermediate value, and the length D_b of the bridging zone are calculated by means of the stable crack growth model (see (C.1) in Appendix C)

$$\sigma_c = \frac{K_{1c}}{Y\sqrt{c_0 + D_b}}, \tag{5.1}$$

where σ_c is the critical stress, at which fracture occurs, and Y is a material constant. Values associated with the thirteen samples are shown in Figure 18.

Two crucial microstructural parameters are p_{max}, the maximum observed bridging stress, and u_{max}, the maximum crack opening, in which the influence of microstructure is embedded. These are related to K_{1c} and D_b by the *Dugdale model*: under uniform bridging stress,

$$K_{1c} = K_0 + \sqrt{\frac{4}{\pi}}\, p_{max}\sqrt{c_0 + D_b}\cos^{-1}\left(\frac{c_0}{c_0 + D_b}\right) \tag{5.2}$$

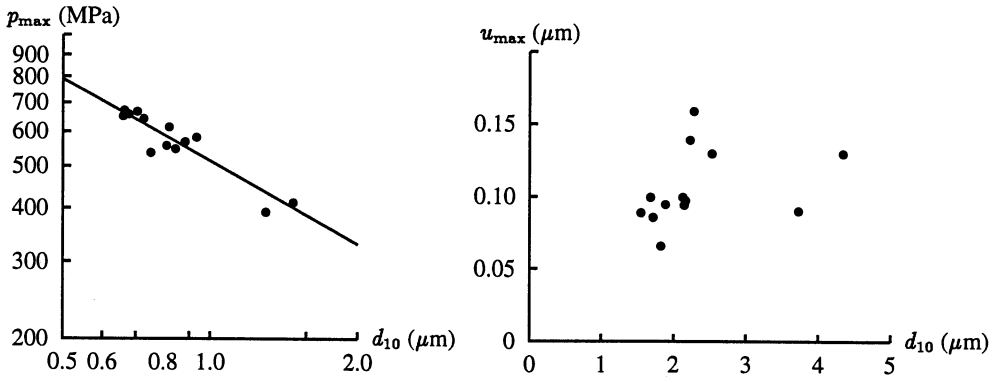


Figure 19: $p_{\max}$ and $u_{\max}$ as Functions of Grain Size d_{10}

$$u_{\max} = \sqrt{\frac{4}{\pi}} \frac{K_0\sqrt{(c_0 + D_b) - c_0^2}}{E'\sqrt{c_0 + D_b}} + \frac{4p_{\max}c_0}{\pi E'} \log\left(\frac{c_0 + D_b}{c_0}\right). \tag{5.3}$$

On the basis of the data, as shown in Figure 19, $p_{\max}$ decreases with grain size, while (although more ambiguously), $u_{\max}$ peaks at an intermediate grain size. This is consistent with expected qualitative behavior of σ_0 and ℓ_{db}.

Finally, from the micromechanical relations

$$p_{\max} = f\sigma_0 \tag{5.4}$$

$$u_{\max} = \frac{\ell_{db}\sigma_0}{2E_0}, \tag{5.5}$$

where f is the area fraction of bridging (which also is a function of the microstructure) and E_0 is the Young's modulus, it is possible to derive estimated values of σ_0 and ℓ_{db} as functions of grain size. From these, and the relation

$$p_{\max} \propto (d_{10}^2 \ell_{db})^{1/m'},$$

it is possible to recover the Weibull modulus m' of grains, which is of the order of 5, and is consistent with values determined by other means.

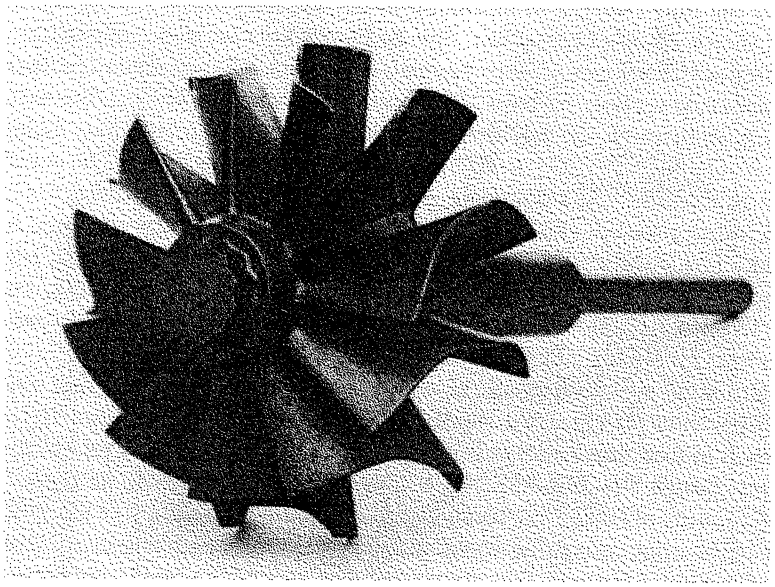


Figure 20: A Silicon Nitride Turbocharger Rotor

6 Materials Performance

6.1 Component Performance

Dr. Johnson, in a talk entitled "Probabilistic Aspects of Ceramic Failure," illuminated a number of issues associated with development of a comprehensive strength/life prediction methodology for ceramic components.

Ceramics are of interest for structural applications — including diesel and turbine engines (Figure 20), industrial dies and cutting tools, bearings and armor for military vehicles, filters and coatings — because of their high melting point, chemical stability, fracture strength, fracture toughness and lubricating (tribological) properties. See [9, 54, 61, 73, 121, 127] for details.

The fundamental relationship between strength and flaw size in ceramics and other brittle materials is the *Griffith criterion*:[54] the fracture strength σ_f (stress at fracture) is given by

$$\sigma_f = \frac{K_{1c}}{Y\sqrt{a}}, \tag{6.1}$$

where K_{1c} is the critical *stress intensity factor* (a property of the material), Y is a geometric factor representing flaw shape and position, and a is the flaw size, which is determined by processing history.

[54]For a derivation based on thermodynamic considerations, see [68].

Flaws in ceramic components arise from

- Materials fabrication;[55]
- Machining processes in component manufacture;[56]
- Damage during service, for example, as a result of impacts or corrosion.

Inevitably, flaw sizes are non-uniform, with two important consequences:

- High component-to-component variability in fracture strengths;
- Dependence of average strength on specimen size, with larger specimens weaker than smaller.

The latter is a consequence of "weakest link" scaling assumptions: a component fails if the stress at any single flaw is large enough.

6.1.1 Weibull Failure Models

A simple model for brittle fracture under uniaxial stress assumes

- A spatially homogeneous Poisson process of flaw locations $X_1, \ldots, X_N$ in the material;
- Independent, identically distributed flaw sizes $A_1, \ldots, A_N$, independent of the location process, whose distribution function F reflects the material and the processing;
- A stress distribution $\sigma(x, S, E)$, where x is location, S denotes external stresses and component geometry, and E represents environmental variables (for example, temperature);
- The Griffith failure mechanism of (6.1): the ith flaw causes component failure if $\sigma(X_i, S, E) > K/\sqrt{A_i}$.

If $1 - H(a) = (a_0/a)^{m/2}$ for all $a > a_0$, and if $\sigma(x, S, E) < K/\sqrt{a_0}$ for all x, then after rescaling [57, 59], the probability that the component fails is

$$P_c = 1 - \exp\left[-\int_V \left(\frac{\sigma(x, S, E)}{\sigma_0}\right)^m dx\right]. \tag{6.2}$$

This is a form used widely in applications, in which material properties are encapsulated in two phenomenological parameters: the *Weibull modulus* m and *inherent strength* σ_0.

[55] Structural ceramics are produced from extremely pure, extremely fine powders, which are densified at high temperatures, via several processes, two of the most important of which are *sintering* and *hot isostatic pressing*. Sintered components can be formed at low temperatures, into *green bodies*, which may be machined before densification Hot forming processes, such as hot isostatic pressing, combine can forming and sintering into one operation.

[56] Machining of ceramics, because of their hardness, is particularly difficult, and is a significant source of flaws.

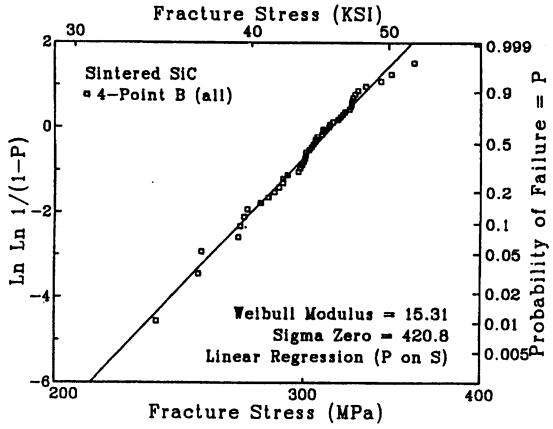


Figure 21: Failure Data for Sintered SiC

Size (mm)	Load Geometry[59]	n	$\bar{X}$	s	$\hat{m}$	$\hat{\sigma}_0$
20 × 2 × 1.5	3-point	18	388.11	33.36	14.57	430.99
20 × 2 × 1.5	4-point	17	312.85	34.83	9.43	459.24
40 × 4 × 3	3-point	18	350.96	31.12	12.20	449.93
40 × 4 × 3	4-point	48	303.31	24.17	14.28	430.28
80 × 8 × 6	3-point	18	325.65	21.69	16.39	418.83
80 × 8 × 6	4-point	18	283.78	22.35	14.48	441.08

Table 3: Data and Estimators for Sintered SiC Specimens

6.1.2 Estimation of Component Reliability

Data regarding laboratory specimens comes from 3- and 4-point bend tests; an example is illustrated in Figure 21.[57] The estimated values there — $\hat{m} = 15.31$ and $\hat{\sigma}_0 = 420.8$ — were obtained via maximum likelihood estimation.[58]

Different data sets arise from variations in the kind of test, specimen size and loading geometry. In the most complicated situation, multiple specimen sizes and geometries are tested, but component size and geometry differs from that of specimens. In such cases, (6.2) is assumed valid for both specimens and components. Estimators $\hat{m}$ and $\hat{\sigma}_0$ are constructed from specimen data, and then the estimated component reliability is

$$\widehat{P_c} = 1 - \exp\left[-\int_V \left(\frac{\sigma(x, S, E)}{\hat{\sigma}_0}\right)^{\hat{m}} dx\right]. \tag{6.3}$$

This is a statistical estimator, via dependence on $\hat{m}$ and $\hat{\sigma}_0$, and, ordinarily, also involves numerical approximation, in computation of σ as a function of x and calculation of the integral.

The estimators $\hat{m}$ and $\hat{\sigma}_0$ in (6.3) may be computed from individual tests (different sizes and geometries) or pooled data. Table 3 shows the former. There, n is the sample size, $\bar{X}$ and s are the sample mean and standard deviation of specimen strengths and the estimated values are obtained via maximum likelihood.

Alternatively, the data may be pooled, which leads to $\hat{m} = 14.22$ and $\hat{\sigma}_0 = 433.1$.

Confidence and tolerance bounds are required in order to employ such estimators, and may be produced by several methods, such as linear regression, maximum likelihood, likelihood ratios and parametric and nonparametric bootstraps [57]. Some of these are very intensive computationally.

6.1.3 Life Prediction Methodology

Future design needs make a comprehensive life prediction methodology highly desirable. Such a methodology two main components:

Modeling needs. Models are required that accommodate

- Reversible strength changes (mainly associated with temperature);
- Irreversible strength changes, resulting, for example, from high temperature slow crack growth, stress corrosion, cyclic fatigue and creep;
- Creation of new flaws, for example, by oxidation, corrosion, impact or proof testing;
- Anisotropic flaw populations;
- Non-planar crack growth;
- Multi-axial stress states and failure criteria.

Statistical needs. These include

- Efficient estimators from unpooled and pooled data;
- Confidence, tolerance and prediction bounds;
- Bias corrections for estimators and bounds;
- Tests for goodness of fit and homogeneity;
- Efficient experimental design;

[57] This *probability plot* contains the pairs $(X_{(i)}, [i - 1/2]/n)$, where n is the sample size and the $X_{(i)}$ are the order statistics of the failure stresses X_j.

[58] See Appendix D.

[59] In a 3-point bend test, the specimen is supported (from below) at the ends and stress is from above applied at its midpoint. In a 4-point bend test, the stress is applied at two points, located one-quarter of the length of the specimen from either end.

- Integration with data from proof tests and nondestructive evaluation of components in service.

All of these are accompanied by needs for computational power, efficient algorithms and "user-friendly" design tools.

6.2 Failure Models for Fibrous Materials

In his talk "Probabilistic Models for Microstructural Failure: the Bundle of Fibres Model and its Extensions," Professor Smith presented a series of stochastic models for failure of parallel fiber composite materials, which differ in regard to the stipulated physical structure of the material and the mechanism via which loads from failed fibers are re-distributed among surviving fibers.

6.2.1 Parallel Fibers

A simple model [22, 87] illustrates the key features of the analysis. Consider a bundle of n fibers in parallel subjected to a tensile load. Assume that

- Individual fiber strengths X_i are independent, identically distributed random variables with distribution function F;
- If some fibers have failed, the load is shared *equally* among those remaining.

Since the bundle will support n times the strength of the weakest fiber, *or* $n-1$ times the strength of the second weakest fiber, ..., *or* the strength of the strongest fiber, its strength expressed in terms of load per initial fiber is

$$S_n = \max_{1 \le k \le n} \frac{(n-k+1)X_{(k)}}{n}, \tag{6.4}$$

where $X_{(1)} \le X_{(2)} \le \ldots \le X_{(n)}$ are the order statistics of $X_1, \ldots, X_n$.[60] An alternative expression, with F_n the empirical distribution function[61] of the fiber strengths, is

$$S_n = \max_{0 < x < \infty} x[1 - F_n(x)]. \tag{6.5}$$

Exact (recursive) expressions for the distributions of the S_n are available, but in cases of physical interest, n is so large that appeal may be made to asymptotics. With x^* the value of x maximizing $x[1-F(x)]$, $m = x^*[1-F(x^*)]$ and $s = x^*\sqrt{F(x^*)[1-F(x^*)]}$, then for each y,

$$\lim_{n \to \infty} P\left\{\sqrt{n}[S_n - m]/s \le y\right\} = \Phi(y), \tag{6.6}$$

[60] That is, the values of $X_1, \ldots, X_n$ arranged in increasing order.

[61] The (discrete) distribution function with mass $1/n$ at $X_1, \ldots, X_n$.

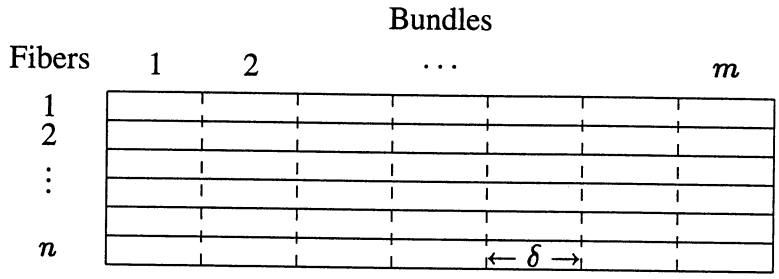


Figure 22: The Chain of Bundles Model

where

$$\Phi(y) \stackrel{\text{def}}{=} \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{y} e^{-z^2/2}\, dz$$

denotes the standard normal distribution function [16].

6.2.2 The Chain of Bundles Model

This model [39, 40] is a more complicated variant. Consider an object, as depicted in Figure 22, composed of m bundles, each composed of n parallel fibers, in series. Each bundle is δ long, where δ represents an *ineffective length*, on the order of microns, beyond which bundles do not interact. There is *local* load-sharing, described momentarily, within bundles, but *no* load-sharing among bundles, so that the chain fails when its weakest link does. Thus, with $F_{n,\ell}$ the distribution function of the strength of one n-fiber bundle of length ℓ,

$$1 - F_{n,m\delta}(x) = [1 - F_{n,\delta}(x)]^m. \tag{6.7}$$

Local load-sharing within a bundle means that when a fiber or group of fibers fails, the load is redistributed preferentially to nearby unfailed fibers. This may assume a variety of forms, depending on geometry and *stress concentration factors*: if r adjacent fibers fail, their load is distributed over q_r neighbors with stress concentration factors K_r.[62]

Exact analysis of this model is very demanding computationally. Regarding asymptotics, let $F_n^{(k)}(x)$ be the probability of failure, under load x, of k contiguous elements somewhere in a system of n elements. Suppose that a system of n elements contains $c(n,k)$ possible configurations of k elements, and that for each such configuration, the failure distribution satisfies

$$F^{(k)}(x) \sim \delta b_k \left(\frac{x}{x_1}\right)^{k\eta}, \qquad x \downarrow 0.$$

[62]For the simplest case, of linearly arrayed fibers, $K_r = 1 + r/2$.

k	1	2	3	4	5	6	7	8
Scale parameter	0.82	2.01	2.57	2.82	2.92	2.96	2.96	2.96
Shape parameter	5.6	11.2	16.8	22.4	28.0	33.6	39.2	44.1

Table 4: Experimental Data for a Chain of Bundles Model

where b_k is a constant representing the local mechanics of the system. Then,

$$1 - F_n^{(k)}(x) \sim \exp\left[-\delta b_k c(n,k)\left(\frac{x}{x_1}\right)^{k\eta}\right]. \tag{6.8}$$

This implies a *Weibull distribution* for k-failures.

The relationship of this model to experimental data for bundles of carbon fibers is treated in [129]. Estimated scale and shape parameters in (6.8) for various values of k are given in Table 4. The "critical" value of k, at which the estimated scale parameter stabilizes, is approximately 6.

In terms of dependence on length ℓ, one can simplify (6.8) to

$$F_\ell(x) = 1 - \exp\left[-\ell(x/x_0)^\beta\right].$$

An alternative

$$F_\ell(x) = 1 - \exp\left[-\ell^\alpha(x/x_0)^\beta\right],$$

where $0 < \alpha < 1$, depicts component failure by modes other than the "weakest link."

6.3 Phenomenological Models

Professor Krajcinovic' talk "Failure of Fiber Bundles" treated similar issues, with the goals of evaluating micro- and macro-responses (to stress) of brittle elastic solids weakened by a large number of microcracks, and of determining effective properties and the onset of the critical state (i.e., failure). Specific goals are to select an appropriate damage parameter; to determine the range of validity of local theories; to relate the damage model to fracture mechanics; and to study the brittle to quasi-brittle transition.[63] The underlying premise is that damage is a random process dependent on local stress concentrations and weaknesses in the material.

Three general classes of models are:

- Micromechanical models, which are accurate and unambiguous at the expense of computational demands and need for large data bases;
- Phenomenological models, which are efficient computationally, but ambiguous and require selection of materials parameters;

[63] See also §5.2.

Parameter	Notation
Specimen size	L
Size of representative volume element	L_{r}
Distance between adjacent defects	L_{c}
Decay length for local stress fluctuations	L_{d}
Size of cluster defect	ξ
Intrinsic length of microstructural disorder[64]	ℓ

Table 5: Length Scales Relevant to Specimen Failure

- Models from statistical physics, which accommodate defect geometry and critical phenomena, but are burdensome computationally.

The approach below falls within the second of these.

6.3.1 The Role of Geometry

An insightful formalism for dealing with multiple length scales is to introduce a family of *length parameters*, given in Table 5. These lengths may be used, for example, to identify various special cases and situations of interest, such as

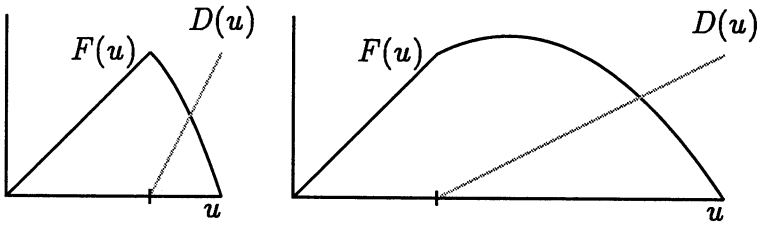
- *Macro-homogeneous solids*, for which $L \gg \ell \gg \xi$ and $L_{\mathrm{c}} > L_{\mathrm{d}}$;
- The case of *defect interaction*: $L_{\mathrm{c}} < L_{\mathrm{d}}$;
- *Non-existence of representative volume elements*: $\xi \to \ell$;
- *Cell models*, for which $\ell \cong \xi$;
- *Failure*, where $\ell \cong L$.

6.3.2 A Parallel Bar Model

A version of the parallel fiber model in §6.2.1 illustrates how a seemingly simple model can exhibit interesting, subtle behavior. The model is a system of N parallel bars, whose random strengths have density function p on the interval $[f_{\mathrm{min}}, f_{\mathrm{max}}]$.

Suppose that the system is subjected to tension, and that if some bars have failed, the load is sharing equally among those surviving. Then, in equilibrium the expected force needed to sustain

[64]The desired resolution of finite element models is ℓ, which for polycrystalline ceramics is on the order of the grain size.



A. Brittle (Ordered) B. Quasi-Brittle (Disordered)

Figure 23: Brittle and Quasi-Brittle Damage Behavior

a displacement u (relative to the no force condition) is

$$F(u) = ku \times N \int_{ku}^{f_{\max}} p(f)\, df, \tag{6.9}$$

where k is the stiffness of the material. The latter term in (6.9) is simply the expected number of surviving bars. The distribution function of rupture strengths is one possible *damage function*, representing the expected fraction of bars ruptured as a function of elongation.

For example, if the strengths are uniformly distributed on $[0, f_{\max}]$, then

$$F(u) = ku[1 - ku/f_{\max}]$$

and

$$D(u) = ku/f_{\max}.$$

Other alternatives, such as Weibull distributions, are possible, as are other measures of damage.

6.3.3 The Brittle to Quasi-Brittle Transition

A key parameter is the *bandwidth*

$$\Delta f = f_{\max} - f_{\min}. \tag{6.10}$$

In the limiting case that $\Delta f =$, all bars break at the same strength $f_{\min}$.

More generally, suppose that bar strengths are uniformly distributed on $[f_{\min}, f_{\min} + \Delta f]$. If $f_{\min} > \Delta$, then, as in Figure 23, the response is brittle: the damage at maximum force is zero. On the other hand, if $f_{\min} < \Delta f$, the response is *quasi-brittle*, in the sense that the partially damaged system can withstand additional force.

Scale	Displacement At Maximum Force	Force At Maximum Force	Force / Displacement At Maximum Force
4	1.09	0.54	0.495
8	1.83	0.92	0.503
16	3.08	1.54	0.500
24	4.17	2.09	0.501

Table 6: The Secant Modulus in Scaling

6.3.4 Lattice Models

Failure in real materials results from the interaction of stress concentrations and irregularities, such as weak links and hard spots, in the microstructure of the material. Lattice models with randomly distributed rupture strengths, for example, the spring network models of §5.2, are one avenue to study such phenomena.

Because these simulations entail finite element computations, mesh size must be of the order of the stress fluctuation decay length L_d in Table 5. In consequence, only small meshes can be simulated, so it is of interest to seek parameters that are scale independent.

6.3.5 Scaling Effects

The goal of studies of scaling effects is to determine extrinsic properties of large structures from (some combination of) simulations of small lattices and tests of lab specimens.

As an example, for central force lattices, with L denoting the specimen size, F denoting force and Λ denoting displacement, there are constants β and γ and a function Ψ, such that

$$\langle F \rangle = L^{\beta} \Psi(\Lambda / L^{\gamma}). \tag{6.11}$$

Simulations lead to the data in Table 6, which identifies the *secant modulus*, that is, the ratio of force to displacement at maximum force, as a size-independent damage parameter.

7 Materials Processing

As expressed by Dr. Richmond, materials processing extends from synthesis through recycling, and should be viewed in terms of *product design based on evolving product states.* See §1.

7.1 Microstructural Evolution

Dr. Garboczi, in a talk entitled "Simulation of Microstructural Development: Geometry and Topology," treated simulation models of microstructural evolution of cement-based materials. A basic goal is to develop models that, combined with microstructure–property relations (§5), allow calculation of time-varying materials properties, such as elastic moduli, fluid permeability, electrical and thermal conductivity, and thermal expansivity during hydration of cement (to form concrete).

The overall approach employs a paradigm discussed in §3.5:

- Microstructure at t is a digital image $M(t)$;[65]
- Properties at t are
$$P(t) = F\big(M(t)\big),$$
where F is a function, in this case a finite element-based computer code, that relates properties to microstructure.

The models below are computerized approximations to evolution equations (compare (3.12))

$$M'(t) = G\big(M(t)\big), \tag{7.1}$$

in which G, which is likewise available only as a computer algorithm, involves randomness but not additional state variables.

7.1.1 Models for Hydration of Cement

Concrete is a complex composite material produced from rocks, sand particles and cement via the chemical process of hydration [75], one of whose effects is to form calcium silicate that "fills in" initial pores in the material. During the course of hydration, the microstructure of the concrete develops; the process requires months to become substantially complete. Multiple length scales are relevant, from centimeters (rocks) through millimeters (sand particles) to microns (reacted and unreacted cement particles) to nanometers (pores). The models presented were at the scale of 25 microns.

The hydration models are computerized Monte Carlo simulations, with the microstructure a digital image, in which each pixel may correspond to undissolved cement paste, dissolved species, pores, or reacted concrete. See Figure 24 for an example. The models have the following structure:

[65] A $1024 \times 1024 \times 256$ color image is a function from $\{0, \ldots, 1023\} \times \{0, \ldots, 1023\}$ to $\{0, \ldots, 255\}$; the colors are a means of display without intrinsic significance.

Figure 24: Digital Model of Hydration of Cement

- The initial point is a suspension of cement particles in water, in which particle shapes may either be simulated (by another model) or obtained from experimental data;
- The hydration process itself is simulated iteratively, by representing
  - Dissolution of cement particles in water, modeled with a single probability of dissolution;
  - Diffusion of dissolved cement particles, modeled as a random walk in pores of the microstructure;
  - Reaction of dissolved particles to form calcium silicate, thereby generating the microstructure of the concrete. This is the "hardening" that fills pores with lower density (than the paste itself) particles, and occurs by "agglomeration" onto existing structure, modeled in conjunction with diffusion and the current state of the microstructure, or spontaneous crystallization.[66]

(Additional effects, for example, formation of microcracks that affect damage behavior, are not represented in current models.)

Related models [33] include

- Multiphase models, which depict more faithfully the heterogeneity of real cements. These entail more complicated hydration rules as well.
- Models of single interfaces (between sand or rock and cement paste), whose properties are crucial to performance of concrete in service [34]. See Figure 25.
- Models to test the effect of different constituents of the cement paste, for example, micron-sized particles of amorphous silica that fill pores more effectively.

[66] Adjustments are made to maintain the correct volume stoichiometry, based on initial composition.

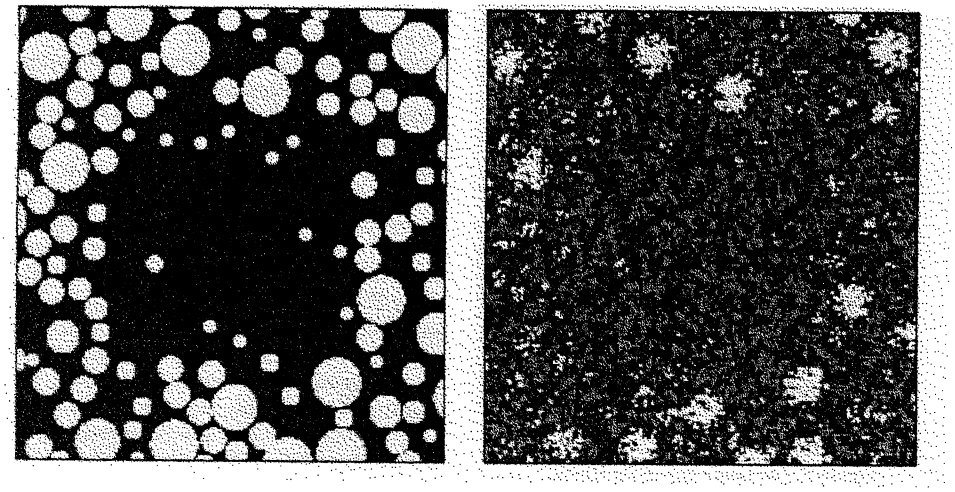


Figure 25: Simulation of the Interfacial Zone in Cement

7.1.2 Models of Connectivity

Yet another class of three-dimensional lattice models [34] (see also §6.3) can be used to address connectivity of phases in multiphase materials. This is especially important in light of the inaccessibility of connectivity data by means of stereological measurements.

In the case of hydration of cement, whether the reacted concrete phase is connected, which closes off the pore space, producing a stronger material, is precisely such an issue. The lattice models have been applied to this question, with numerical results shown in Figure 26. Here the key parameters are the initial water to cement ratio W/C and the fraction of matrix that is connected. The more water present initially, the more hydration (in terms of time) is necessary to achieve connectivity; if $W/C \geq .6$, connectivity is not attained.

7.2 Constitutive Models for Manufacturing Processes

In his talk "Some Statistical Issues in Materials Science," Dr. Richmond described the role of constitutive equations in modeling evolving product states during product manufacturing and service.

Constitutive equations are sets of nonlinear differential and algebraic equations describing the material response to all possible thermomechanical histories (trajectories of strain and temperature). Responses include both structure and properties. A set of constitutive equations consists of

- *State equations*, which express materials properties as functions of controllable external variables, such as strain rate and temperature, and microstructural variables;

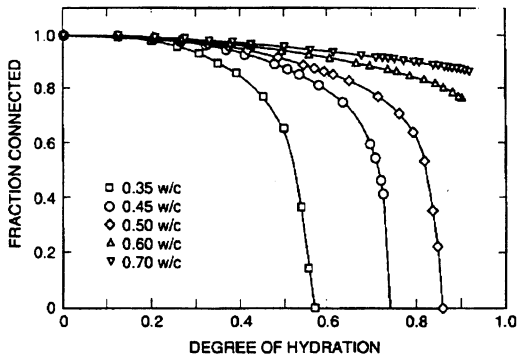


Figure 26: Capillary Porosity Percolation for Cement

- *Evolution equations* for the microstructural variables.

Constitutive equations are *local*, applying to a representative small element of the material, raising the "representativeness" theme discussed in §1.5 and 3.4. Unlike conservation equations, however, constitutive equations are typically ordinary rather than partial differential equations.

For example, as discussed in §3.5, a set of constitutive equations arising in hot rolling of polycrystalline metals, such as aluminum, consists of the state equation

$$\sigma(t) = f\Big(\varepsilon'(t), T(t), s_1(t), s_2(t)\Big) \tag{7.2}$$

and the evolution equations

$$\begin{aligned} s_1'(t) &= g_1\Big(\varepsilon'(t), T(t), s_1(t), s_2(t)\Big) \\ s_2'(t) &= g_2\Big(\varepsilon'(t), T(t), s_1(t), s_2(t)\Big) \end{aligned}$$

where σ is the stress with the material, ε' is the strain rate, T is temperature and s_1 is porosity and s_2 is hardness [105]. The functions f, g_1 and g_2 must be determined, an issue discussed below.[67]

Constitutive equations — in the example, the functions f, g_1 and g_2 — are generated in several ways:

- *Empirically*, from observation of actual trajectories, such as mechanical testing data, usually corresponding to strain rates and temperatures that are constant over time but may be varied from experiment to experiment. The data are curves (possibly time-sampled), to which differential equations must be fit.

[67]More generally, there are actual or surrogate microstructural variables $s_1, s_2, \ldots$, representing, for example, dislocations, precipitates, grains, pores, microcracks and corrosion damage.

- *From unit cell periodic array models,* by means of micromechanical computations, which can be combined with empirical data. Here again, the "representative element" issue must be confronted.
- *From nonperiodic array models*, which are necessary for phenomena, such as fracture and failure, that are driven by extreme values, clustering and other percolation-like effects.

Porosity in ductile materials under simple tension, an illustrative example, is represented by the state equation

$$\left(\frac{\sigma}{s}\right)^2 + 2f^m \cosh\left(\frac{\sigma}{2s}\right) - (1 + f^{2m}) = 0, \tag{7.3}$$

together with the evolution equations

$$\begin{aligned} \frac{ds}{d\varepsilon} &= \frac{n}{1-f}\sigma\left(\frac{\kappa}{s}\right)^{1/n} \\ \frac{df}{d\varepsilon} &= \frac{3mf^m(1-f)\sinh(m\sigma/2s)}{2\sigma/s + mf^m\sinh(m\sigma/2s)}. \end{aligned}$$

Here, tensile stress σ remains the response variable, while the microstructural variables are the volume fraction f and the matrix hardness s. The parameter m is a material constant representing strain hardening. The development of (7.3), which in its original form omitted the power m, was effected by combining measurements with micromechanical computations on a unit cell model.

8 Combining Information

Professor Berliner's presentation "Combining Information" addressed an issue that permeates science: means by which "information from a variety of sources in natural and social sciences is combined to produce more informative summaries and better decisions than those possible based only on each individual information source" [78].

Information, in this context, is of two primary forms:

Data, usually in the form of measurements or observations;

Judgement, in the form of "expert opinions," which can be embodied in subtle ways, such as selection of physical or probability models and structure of variables.

Combining information, then, is a form of learning for purposes of decision making and prediction. The major issue is *how* to combine information.

The discussion that follows pertains to combining information from "similar" experiments in order to reach stronger or more refined conclusions than are justified by the experiments individually. There is great unmet need to combine information from dissimilar experiments.

8.1 Approaches to Combining Information

In broad terms, approaches to combining information may be categorized as statistical or not. Within these categories, some key methodologies are

Statistical: Meta-analysis; random effects models, empirical Bayes procedures, hierarchical Bayes procedures and models of space and time dependence.

Non-Statistical: Fuzzy sets, expert systems, artificial intelligence, neural networks, pattern recognition, adaptive learning and genetic algorithms [36, 63, 76].

To illustrate some statistical approaches, consider K different experiments intended to measure the same physical constant c, which produce (unbiased) statistical estimators $\hat{c}_i$ with (known or estimated) variances V_i.[68] One means of combining these to obtain a single estimator $\hat{c}_{\text{comb}}$ is to form the weighted sum

$$\hat{c}_{\text{comb}} = \frac{\sum_{i=1}^{K} \hat{c}_i / V_i}{\sum_{i=1}^{K} 1/V_i}. \tag{8.1}$$

Relatively more precise estimators (those with smaller variances) contribute more to the weighted sum.

[68] These might reflect measurement devices and sample sizes, for example.

8.2 The Bayesian View

In fact, the very notion of what constitutes a "constant" may be questioned, and one may conceive of two classes of constants:

- "Traditional constants," such as the speed of light [47];
- Observables in complex physical settings, which are ordinarily functionals of lower level states, such as the thermal conductivity of copper [71].

For traditional constants, the key measurement problem is *bias*, or *systematic error*. For the ith experiment in the setting of §8.1, this occurs if $E[\hat{c}_i] \neq c$.

For complex observables, on the other hand, the key issue is *variability*. In the context of the example in §8.1, the experiments may be regarded as having their own values $c_1, \ldots, c_K$, where these values are unknown, but somehow similar.[69] Then, as an alternative to individual estimators $\hat{c}_i$, it is possible, by combining information, to "borrow strength" across experiments to improve individual estimators. This may be effected, for example, via estimators

$$\hat{c}_i^* = \alpha_i \hat{c}_i + (1 - \alpha_i)\hat{c}_{\text{comb}}, \tag{8.2}$$

where $0 \leq \alpha_i \leq 1$ and $\hat{c}_{\text{comb}}$ is a combined estimator of the average of the c_i, possibly although not necessarily that in (8.1).[70] Borrowing strength improves the overall mean squared error of the estimators, and sometimes all of the individual mean squared errors.

More generally, the Bayesian view of combining information is to *conceive properties as random, and to calculate their (conditional) distributions given the observed data.*

Mathematically, this view exploits the property that a joint distribution of random variables can be written as a product of conditional distributions. In the case of three random variables, for example,

$$\begin{aligned} P\{X_1 = x_1, X_2 = x_2, X_3 = x_3\} \;=\; & P\{X_1 = x_1 | X_2 = x_2, X_3 = x_3\} \\ & \times P\{X_2 = x_2 | X_3 = x_3\} \\ & \times P\{X_3 = x_3\}. \end{aligned} \tag{8.3}$$

Often, the terms on the right-hand side of (8.3), especially under certain simplifying assumptions, are easier to understand or more informative than the left-hand side, or provide improved modeling capability.[71]

Controversy regarding "Bayesian statistics" has nothing to do with (8.3), but rather with what is regarded as random and what values are assigned to the components on the right-hand side of (8.3).

[69] The mathematical concept of exchangeability makes "similar" precise.

[70] *Empirical Bayes* procedures provide means of calculating the α_i from the data. See also §8.3.

[71] Among other useful consequences of (8.3) is *Bayes' theorem*; see Appendix D.

8.3 Example: Hierarchical Linear Models

The example on §8.1 may be used to illustrate, by means of a *hierarchical Bayes linear model* for the experimental situation there. One such model stipulates that

- Underlying all K experiments is a physical constant c_0.
- There is a random perturbation C^* of c_0 common to all the experiments.[72]
- Associated (only) with experiment i is a *random effect* ("experimental effect") Z_i^*. Thus the "true" underlying value for this experiment is

  $$Z_i = c_0 + C^* + Z_i^*.$$

  Note that the Z_i are then correlated, because the definition of each involves the common effect C.
- Hierarchically, then, $\hat{c}_i$ is the sum of Z_i and measurement error associated with experiment i:

  $$\hat{c}_i = Z_i + E_i = c_0 + C^* + Z_i^* + E_i. \tag{8.4}$$

The model is put with the conditional distribution context of (8.3) by a series of assumptions, for example,

- The common perturbation C^* is normally distributed with mean 0 and variance τ^2.
- *Conditional on* C^*, the random vector $Z = (Z_1, \ldots, Z_K)$ is normally distributed with mean vector $(c_0 + C^*, \ldots, c_0 + C^*)$ and covariance matrix D_2.
- *Conditional on* C^* *and* Z, the random vector $(\hat{c}_1, \ldots, \hat{c}_K)$ has a joint normal distribution with mean vector Z and diagonal covariance matrix $D_1 = \text{diag}(V_1, \ldots, V_K)$, where $V_1, \ldots, V_K$ are constants.

An alternative interpretation is that the Z_i^* are independent of each other and of C^*, and normally distributed with mean 0, while the E_i are normally distributed, and independent of each other, of the Z_i^* and of C^*.

For example, if $\tau^2 = 0$, that is, $C^* = 0$, then (8.4) implies that $(\hat{c}_1, \ldots, \hat{c}_K)$ is normally distributed with mean $(c_0, \ldots, c_0)$ and covariance matrix $D_1 + D_2$, which is the model

$$\hat{c}_i = c_0 + Z_i^* + E_i, \tag{8.5}$$

where Z_i^* represents systematic error associated with experiment i and E_i is measurement error.[73]

In this same setting, if $D_2 = \text{diag}(\sigma_2, \ldots, \sigma_2)$ (the experimental perturbations have the same variance σ^2), then in (8.2), one can take $\hat{c}_{\text{comb}} = (1/K)\sum_{i=1}^{K} \hat{c}_i$, and the weights become $\alpha_i = \sigma^2/(V_i + \sigma^2)$. The more precise experiment i, in the sense that the error variance V_i is small, the more weight given to $\hat{c}_i$.

[72] For example, all might use the same time standard, whose value differs randomly from the "true" time, but which is the same for all experiments.

[73] Note that even though the mathematical roles of Z_i^* and E_i are identical, their interpretations differ.

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B Workshop Program

Monday, July 26, 1993

9:30 AM Introductory Remarks
* R. Lundegard (NIST)
* A. Karr (NISS)

Plenary Lectures
* O. Richmond (Alcoa): Some Statistical Issues in Materials Science
* C.-W. Li (Allied-Signal): Microstructure and Fracture of In-situ Reinforced Silicon Nitride

1:30 PM *Quantification and Inference for Microstructure*
* R. DeHoff (University of Florida): Stereology and the Quantification of Microstructural Geometry
* E. Garboczi (NIST): Simulation of Microstructural Development: Geometry and Topology
* R. Vitale (University of Connecticut): Stochastic Geometry

7:30 PM Informal Session

Tuesday, July 27, 1993

9:00 AM *Materials Performance*
* C. Johnson (General Electric): Probabilistic Aspects of Ceramic Failure
* R. Smith (University of North Carolina at Chapel Hill): Probabilistic Models for Microstructural Failure: the Bundle of Fibres Model and its Extensions
* D. Krajcinovic (Arizona State University): Failure of Fiber Bundles

1:30 PM *Structure-Property Relations*
* A. R. Day (Marquette University): Microscale Elastic Simulations for Random Materials and Composites
* M. Berliner (Ohio State University): Combining Information
* A. Jagota (Du Pont): Property Simulations via Spring Networks and Finite Element Models

Wednesday, July 28, 1993

9:00 AM Panel Discussion: Key Research Issues and Opportunities
* Moderator: A. Karr (NISS)
* Panelists: E. Fuller (NIST), S. Kurtz (Pennsylvania State University), W. Tucker (General Electric)

Material	Young's Modulus (GPa)	Density (g/cc)	Strength		Weibull Modulus	Max Use Temp (° C)
			30° C (MPa)	600° C (MPa)		
Hot pressed Si_3N_4	290	3.3	830	805	7	1400
Sintered Si_3N_4	290	3.3	800	725	13	1400
Reaction bonded Si_3N_4	200	2.7	295	295	10	1400
Hot pressed SiC	430	3.3	550	520	10	1500
Sintered SiC	390	3.2	490	490	9	1500
Reaction bonded SiC	413	3.1	390	390	10	1500
ZrO_2	205	5.9	1020	580	14	950
Steel	200	7.1	1500	140		600
Aluminum	70	2.7	370	0		350

Table 7: Properties of Selected Ceramics and Metals

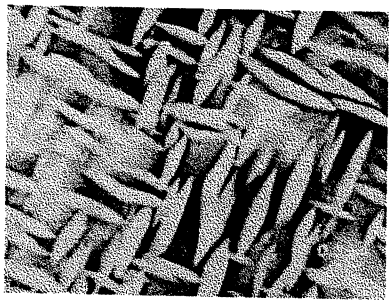


Figure 27: Microstructure of Partially Stabilized Zirconia (ZrO_2)

C Glossary of Materials Science for Statisticians

Terms appearing in sans serif type are defined elsewhere in the glossary.

Brittleness. Absence of plastic deformation of a material prior to fracture.[74] A characteristic property of ceramics.

Ceramic. An inorganic, non-metallic, man-made solid, primarily with ionic (but also covalent) bonding. Examples include alumina (Al_2O_3), zirconia (ZrO_2), silicon carbide (SiC) and silicon nitride (Si_3N_4). Ceramics are characterized by high melting point, strength, chemical inertness, light weight, low thermal conductivity and low coefficient of friction. Values of selected mechanical properties for some common ceramics are shown in Table 7.

The main limiting characteristic of ceramics is brittleness, which engenders size effects — larger components are weaker than smaller — and part-to-part variability.

Ceramic components are produced by forming *green bodies*, ordinarily comprised of powders of the ceramic or its constituents, together possibly with binders and other compounds, such as sintering aids. The green bodies are then *densified*, by heating, perhaps in the presence of constituents of the final product, and possibly under pressure. The principal means of densification are *reaction bonding*, sintering and *hot isostatic pressing* [113, 121].

Properties of ceramics, such as partially stabilized zirconia (Figure 27), depend on phase transformations during processing or induced by stresses [9].

Structural applications of ceramics include turbine and diesel engines, industrial dies and cutting tools,[75] bearings and armor for military vehicles [9, 54, 61, 73, 121, 127].

Composites. Materials comprised of fibers of one phase held together by a matrix of a second phase, and having properties superior to either phase alone. The fibers provide strength, stiffness and fracture toughness, while the matrix binds fibers together in proper orientation. The most common types are ceramic matrix composites [69], metal matrix composites [81] and polymer matrix composites [81].

Constitutive equation. Equation(s) describing the time evolution of materials properties as functions of structural variables and processing regime, including stress and temperature.

Crack resistance. Rate of change of surface energy associated with a crack, with respect to surface area of the crack, measured in Joules per square meter. Intrinsic crack resistance, the integral of cohesive stress (with respect to separation distance) is based on assumptions of linear elasticity. In general, crack resistance depends on crack size (Figure 29), and explanations require nonlinear models [68].

Elastic moduli. Constants of proportionality in linear relationships between stress and strain in a material, usually expressed in units of gigaPascals (1 GPa = 10^9 N/m^2).

For most materials, the stress required to produce small values of strain is proportional to the strain, as expressed by *Hooke's law*.[76] For the three strains in Figure 30,

$$\begin{aligned} \sigma &= E\varepsilon \\ p &= -K\delta \\ \tau &= G\gamma; \end{aligned}$$

E is the *Young's modulus*, K is the *bulk modulus* and G is the *shear modulus*.

[74] Connotations of fragility are inapplicable; in some settings, "inductile" has been proposed as an alternative to "brittle."

[75] The same hardness that makes ceramics desirable as cutting tools makes machining of ceramic components especially difficult.

[76] At larger strains, the relationship becomes nonlinear, and ultimately even non-monotonic.

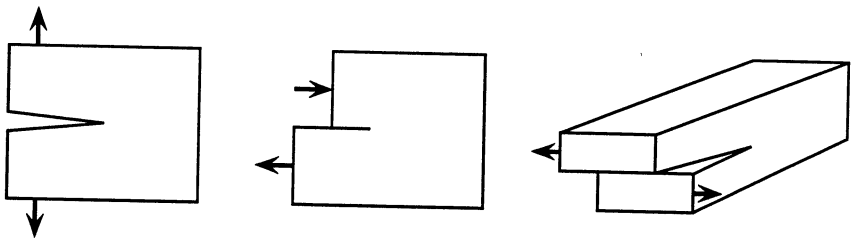


Figure 28: Fracture Modes

Elasticity. Recoverable deformation (strain) of a material as the result of applied stress; recovery occurs when the stress is removed. Nearly all materials exhibit elastic behavior under sufficiently small stresses.

Finite element method. Family of numerical techniques for solution of differential and partial differential equations, especially those with non-regular boundary conditions or other discontinuities that render analytical methods inapplicable. The finite element method is particularly useful in a variety of "engineering" contexts, for example, computation of stress distributions and in structural analysis.

In an application of the finite element method, the region of interest is discretized as the union of geometrically simpler regions (*finite elements*), which are connected at *nodal points*. Simple mathematical functions, such as polynomials, are used to represent the solution (for example, displacement or stress) within each region, and these must be fit together to produce a solution for the entire region. In the process, qualitative mathematical simplification occurs: for example, differential equations become systems of linear algebraic equations. See [5].

Fracture. Failure of a material by progressive stretching and eventual rupture of interatomic bonds across a crack plane, as the result of applied stress. Most models of fracture in brittle materials are derived by viewing cracks as thermodynamic systems in equilibrium [68]: crack extension is just on the verge of occurring.

The three main fracture modes — opening, shear and tearing — are illustrated in Figure 28. In brittle materials, such as ceramics, opening is the principal operative mode.

Key associated materials properties include crack resistance and fracture toughness.

Fracture toughness. Critical value of the stress intensity associated with a crack, as in (C.2): when it is exceeded, unstable fracture occurs. It is a property of the material.

Griffith criterion. A failure criterion for brittle materials based on flaw size and fracture toughness: a flaw of size a causes failure if the local stress σ satisfies

$$\sigma > \frac{K_{1c}}{Y\sqrt{a}}, \tag{C.1}$$

where K_{1c} is the fracture toughness and Y is a constant reflecting specimen and load geometry. Note the analogy to (C.2). The criterion is derived from thermodynamic considerations, under assumptions of elasticity [68].

Hardness. Resistance of a material to penetration, comprising an indication of deformation behavior. Hardness, measured in units of N/mm^2, is determined by forcing a diamond shaped point into a specimen under constant load and measuring the size of the resultant impression [107].

Material. Substance defined by its chemical composition and structure, especially crystal structure, at the molecular and macroscopic levels.[77] Both composition and structure may vary over time and within the material, sometimes in response to external conditions.

Primary classes of materials are

- Structural materials: Ceramics, composites, metals;
- Polymers;
- Electronic materials; semiconductors;
- Magnetic and photonic materials;
- Superconducting materials;
- Biomaterials.

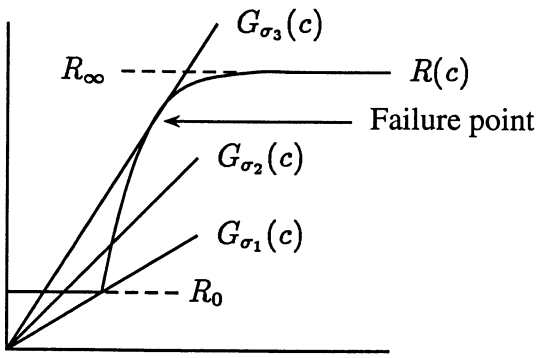
These cannot be delineated unambiguously.

Metal. An electropositive chemical element, possessing mobile electrons. In bulk, metals are characterized by crystalline structure, ductility, electrical and thermal conductivity, (relatively) high melting point, denseness, opacity, luster and plasticity.

Microstructure. Structure of a material at the (approximate) scale of $10^{-8} - 10^{-4}$ m, that is, between the atomic lattice and continuum levels. Microstructure is characterized by the size, shape and orientation of crystalline phases, and the location, orientation and connectivity of boundaries, defects, fibers and microcracks.

Phenomenological property. A materials property associated with a particular model rather than direct measurement, for example, the Weibull modulus. Phenomenological properties ordinarily can only be derived statistically.

[77] Composition alone does not suffice: alumina and sapphire have the same chemical composition but different crystal structure, as do diamond and graphite.



R = crack resistance; G_σ = global energy release rate

Figure 29: R-Curve Behavior

Plasticity. Permanent deformation of a material or object as the result of applied stress. Plasticity may be desirable or crucial for manufacturing, but undesirable in components in service. Brittle materials, such as ceramics, are effectively unable to undergo plastic deformation.

Poisson ratio. The ratio of the transverse contracting strain to the elongation strain in a material subjected to a tensile stress.

Polymer. Material composed of long, chain-like molecules comprised of repeated simple molecules (monomers) with covalent inter-molecular bonds, held together by van der Waals bonds. Polymers are characterized by amorphous structure, low density, low thermal and electrical conductivity and deformability.

R-curve behavior. Increasing crack resistance as a function of crack size, a phenomenon exhibited by some but not all materials. A typical R-curve is illustrated in Figure 29. Explanations typically invoke some form of crack shielding, whereby the tip of the crack is shielded from stresses [68].

Figure 29 also illustrates a "tangency" condition for failure. The linear curve there represents the global mechanical energy release rate, for three values $\sigma_1 < \sigma_2 < \sigma_3$ of the applied stress. Equilibrium requires that $R(c) = G_\sigma(c)$; if $G'_\sigma(c) < R'(c)$, as for σ_1 and σ_2, the crack grows stably, but when $R'(c) \geq G_A,(c)$, which occurs for σ_3, the growth becomes unstable and the material fails. In this case, σ_3 is the strength of the material.

Slow crack growth. Chemically assisted fracture of a brittle material, especially during service. See [68].

Sintering. One process by which ceramics are densified, converting a loosely bonded powder into a dense body by means of heat. After moisture and organic impurities are "burned off,"

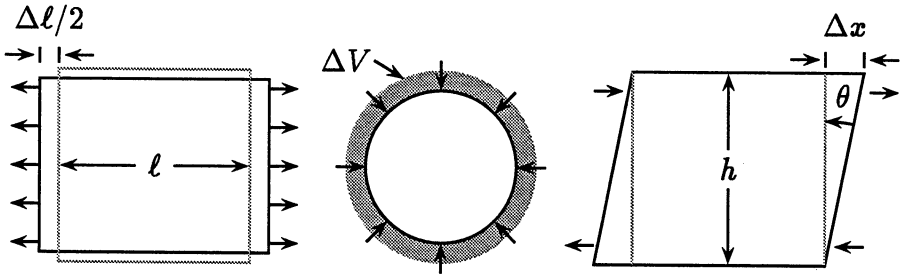


Figure 30: Stresses and Strains

further heating causes diffusion on an atomic scale that fills voids among particles. "Sintering aids" are ordinarily needed in order to achieve full density.

Strain. Relative deformation (therefore, dimensionless) of a specimen or material as a consequence of stress. Figure 30 illustrates three principal strains, which result from tension, hydrostatic pressure and shear:

- *Tensile strain* $\varepsilon = \Delta\ell/\ell$, where ℓ is the original length. *Longitudinal* strain is (lengthening) in the direction of the tensile stress; *lateral* strain is (shortening) in the perpendicular direction.
- *Volumetric strain*[78] $\delta = \Delta V/V$, where V is the original volume;
- *Shear stain* $\gamma = \tan\theta$, where θ is the angle by which the material deforms.

Like stress, strain is also a tensor.

Stress. Force per unit area acting on a solid object, measured in units of megaPascals (1 MPa $= 10^6$ N/m^2). The three principal stress effects — *tension*, *hydrostatic pressure* and *shear* — are illustrated in Figure 30. Tensile stress is ordinarily denoted by σ, hydrostatic pressure by p and shear stress by τ.

In reality, stress in two dimensions is a tensor

$$\begin{bmatrix} \sigma_x & \tau_{xy} \\ \tau_{yx} & \sigma_y \end{bmatrix}.$$

The shear stresses τ are indexed by direction and point of application of forces: τ_{xy} is the shear stress associated with force applied in direction x to the "top" face (y-face) of the object, while the normal stresses σ_x are essentially tensile stresses.

[78] Also termed *dilatation*.

Stress intensity factor. Under assumptions of elasticity, the constant K in approximations

$$\sigma(x) \sim \frac{K}{\sqrt{2\pi r}} f(\theta), \tag{C.2}$$

for stress $\sigma(x)$ at a point x in the vicinity of a crack tip. In (C.2), (r, θ) is the cylindrical coordinates of x relative to the crack tip and f is a function.

Units of the stress intensity factor are MPa $m^{1/2}$; its value depends on the crack size and shape, the loading stress and specimen geometry [68, 112].

Strength. Stress at which a material fails; taken to be a material property.[79]

Weibull modulus. A phenomenological property of brittle materials associated with strength. The applied stress causing failure is taken to have a (two-parameter) Weibull distribution:

$$F(\sigma) = 1 - e^{-(\sigma/\sigma_0)^m}. \tag{C.3}$$

The parameter m in (C.3) is the Weibull modulus; σ_0 is the *inherent strength.* Estimated values of m for real materials range from 5 to 30.

[79] Rather than a measure of component performance.

D Glossary of Statistics for Materials Scientists

As in Appendix C, terms in sans serif type are defined elsewhere in this glossary. General references are [60] on probability and [4] on statistics. The encyclopedia [64] is also exceedingly useful.

The field of statistics should be construed broadly, as *data-driven modeling and analysis.*

Asymptotics. Properties of statistical procedures as the amount of data becomes infinite. The main virtues of asymptotic theory are that it is simple and broadly applicable, whereas exact small sample theory is difficult and situation-dependent.[80]

Bayes theorem. Computational formula for conditional probabilities: if $A_1, A_2, \ldots$ are a partition of the sample space of a random experiment,[81] then for each event B with $P(B) > 0$ and each j, $P(A_j|B) = P(B|A_j)P(A_j)/\sum_{i=1}^{\infty} P(B|A_i)P(A_i)$.

Bias. For a statistical estimator $\hat{\alpha}$ of a parameter α, the quantity $E[\hat{\alpha}] - \alpha$, the average difference between the estimator and the quantity it estimates.[82]

Conditional distribution. Given (for example) random variables X and Y, conditional probabilities $P\{X = x|Y = y\}$.

Conditional probability. Relative likelihood that an event A occurs given that another event B is known to have occurred, expressed mathematically by $P(A|B) = P(A \cap B)/P(B)$.

Confidence interval. Interval[83] whose endpoints are statistics, and which contains the unknown parameter of a statistical model with prescribed (high) probability.[84] Typically, the confidence interval is centered at a statistical estimator. The width, construed as a measure of the variability of the estimator, is calculated from knowledge about the distribution of the estimator.[85]

Correlation. *Linear* relationship between random variables X and Y, expressed in terms of the dimensionless *correlation coefficient* $\rho_{XY} = \big(E[XY] - E[X]E[Y]\big)/\sigma_X\sigma_Y$, where σ_X and σ_Y are the standard deviations of X and Y.[86]

[80] For example, the data $X_1, \ldots, X_n$ correspond to a *sample size* of n, and asymptotics concern behavior of various statistical procedures as $n \to \infty$. For estimators, the key asymptotic properties are *consistency*, that is, convergence of $\hat{\alpha}_n$ to α, and *asymptotic error distributions*, which, scaled suitably, are often normal distributions.

[81] They are disjoint and their union is the entire sample space.

[82] $\hat{\alpha}$ is *unbiased* if its bias is 0.

[83] For higher-dimensional parameters, a more complicated set.

[84] The latter statement holds with respect to independent replications of the random experiment.

[85] Resampling techniques provide one method of approximating such distributional information when it is unavailable analytically.

[86] An alternative is the *covariance* $E[XY] - E[X]E[Y]$.

Density function. For a random variable X, a function f_X satisfying

$$P\{X \leq t\} = \int_{-\infty}^{t} f_X(u)\,du, \qquad t \in \mathbb{R}. \tag{D.1}$$

Dimension reduction. Simplifying high-dimensional data by forming lower-dimensional functions of them. Linear regression and principal components analysis are examples.[87] Often in applications there is attempt to identify "active" components or combinations of components of the data X, so that dimension reduction carries a connotation of sensitivity analysis.

Distribution function. For a random variable X, the function $F_X(t) = P\{X \leq t\}$.

Empirical distribution function. For data $X_1, \ldots, X_n$, the (distribution function-valued) statistic $F_n(t) = \frac{1}{n}\sum_{i=1}^{n} \mathbf{1}(X_i \leq t)$.

Event. A set of outcomes of a random experiment.

Expectation. Weighted average $E[X]$ of the values assumed by a random variable X, where the weight of each value is the probability of the event on which it is assumed.[88] Also known as the mean of X.

Experimental design. Statistical techniques for planning and analysis of physical or numerical experiments in order to decrease cost (e.g., number of trials) or increase informativity of the resultant data. An experiment comprises a set of *trials* intended to evaluate how several parameters influence one or more *responses*; parameters may be controllable *factors* or uncontrollable *noise*. The design specifies the *level* of each factor in each trial, with the goal of ascertaining both *main effects* (of varying individual factors) and *interactions* between or among factors.

A *full factorial design* tests all possible combinations of the factor levels; it provides the best information for estimation of main effects and interactions, but is time-consuming and expensive. *Fractional factorial designs* examine cleverly chosen subsets of factor levels, and are represented by matrices in which columns correspond to factors and rows list the factor levels for each trial.[89] Fractional factorial designs allow fewer trials, and hence, more factors and levels, as well as more repetition within trials,[90] but cannot resolve some factor effects, especially higher-order interactions. See [6, 7, 30, 31].

[87] More generally, for k-dimensional data X, dimension reduction produces m-dimensional data, where $m < k$, $X' = H(X)$, where H is a function from k-dimensional space to m-dimensional space.

[88] If X takes values in the countable set C, $E[X] = \sum_{t \in C} tP\{X = t\}$, while if X has density f_X, then $E[X] = \int_{-\infty}^{\infty} u f_X(u)\,du$.

[89] The *Taguchi method*, for example, is based on *pairwise orthogonal arrays* of the minimal size necessary to resolve main effects.

[90] Which reduces variability.

Function estimation. Statistical models and associated techniques in which the unknown parameter α is a function. A simple case is linear regression, in which the unknown function is linear. At the other extreme, in (nonparametric) *density estimation*, the unknown is the density function of independent, identically distributed data. See [8, 32, 99].

Independence. Absence of probabilistic interaction among random variables or events. Independence is assumed commonly in statistical models, not always explicitly.[91]

Integration of physical and numerical experiments. Statistical techniques specifically directed at combining data that comes in part from physical experiments and in part from (not necessarily compatible) numerical experiments.

Least squares estimation. Statistical estimation method in which statistical estimator $\hat{\alpha}$ is chosen to minimize a quadratic function of the parameter α and the data. Linear regression is the leading example.

Likelihood ratio test. Procedures for tests of hypotheses in which the test statistic is

$$T = \frac{\ell(\alpha_1; X_1, \ldots, X_n)}{\ell(\alpha_0; X_1, \ldots, X_n)},$$

where ℓ is the likelihood function of (D.2).[92] Large values of T are evidence in favor of the alternative hypothesis, so the null hypothesis is rejected if T exceeds a critical level.

Linear regression. Statistical model in which the data are random variables $X(t_1), \ldots, X(t_n)$ satisfying

$$E[X(t)] = a + bt,$$

with a and b the unknown parameters of the model. Statistical estimators $\hat{a}$ and $\hat{b}$ are usually computed by least squares estimation, to minimize $\sum_{i=1}^{n} \left(X_i - [a + bt_i]\right)^2$.[93]

Maximum likelihood estimation. Statistical estimation technique in which $\hat{\alpha}$ is that value of the parameter maximizing the probability of the actually observed values of the data. The *maximum likelihood estimator* is computed by maximizing the *likelihood function*

$$\ell(\alpha; x_1, \ldots, x_n) = P_\alpha\{X_1 = x_1, \ldots, X_n = x_n\}. \tag{D.2}$$

Mean. The expectation $E[X]$ of a random variable X, interpreted as the average value of X.

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[91] Mathematically, random variables $X_1, \ldots, X_n$ are independent if $P\{X_n \le t_1, \ldots, X_n \le t_n\} = P\{X_1 \le t_1\} \cdots P\{X_n \le t_n\}$ for all $t_1, \ldots, t_n$. Events $A_1, \ldots, A_n$ are independent if $P(\bigcap_{i \in I} A_i) = \prod_{i \in I} P(A_i)$ for every subset I of $\{1, \ldots, n\}$.

[92] The interpretation is that T is the relative likelihood of observed data under α_1, compared to that under α_0.

[93] Explicit expressions are available: $\hat{a} = \left[(\sum t_i^2)(\sum X_i) - (\sum t_i)(\sum t_i X_i)\right] / \left[n \sum t_i^2 - (\sum t_i)^2\right]$ and $\hat{b} = \left[n \sum t_i X_i - (\sum t_i)(\sum X_i)\right] / \left[n \sum t_i^2 - (\sum t_i)^2\right]$.

Mean squared error. For a statistical estimator $\hat{\alpha}$ of a parameter α, the quantity $E\left[(\hat{\alpha}-\alpha)^2\right]$, the average squared deviation of $\hat{\alpha}$ from α.

Method of moments. Statistical estimation technique used in cases in which the parameters of the model are expressible as a function of (population) moments of the data. The estimators are then the same function of corresponding sample moments. [94]

Moments. Expectations of powers of a random variable. The most important are the mean, variance and standard deviation.

Normal distribution. The distribution function Φ with density function $\phi(x) = (1/\sqrt{2\pi})e^{-x^2/2}$, $x \in \mathbb{R}$.

Numerical experiment. An experiment in which the observed data are generated by a numerical computer model rather than a "physical" system.

Order statistics. Given data $X_1, \ldots, X_n$, the statistics $X_{(1)} \leq X_{(2)} \leq \ldots \leq X_{(n)}$, the values of $X_1, \ldots, X_n$ arranged in increasing order.

Prediction. Estimation of the value of an unobservable random variable — one *not* a function of the data — by means of one that *is* a function of the data. Akin to statistical estimation, but what is predicted is random and unobservable rather than deterministic and unknown.[95] For data $X_1, \ldots, X_n$ and unobservable Z, the predictor is a random variable $\hat{Z} = k(X_1, \ldots, X_n)$ chosen to minimize the mean squared error $E\left[(Z - k(X_1, \ldots, X_n))^2\right]$.

Principal components analysis. Dimension reduction, effected by means of a least squares fit of a low-dimensional space to the data. Linear regression, which fits a line (a one-dimensional space) is the most widely used special case.[96]

Probability. A function P defined for events, which measures their likelihoods of occurring,[97] such that $P(A) \geq 0$ for all A; $P(\Omega) = 1$; and $P(A \cup B) = P(A) + P(B)$ whenever $A \cap B = \emptyset$.[98]

Random experiment. An experiment, not necessarily physical, whose outcome cannot be foretold in advance. The "randomness" may arise from several sources, including observation mechanisms (e.g., measurement error), sampling and other forms of incomplete observation and factors relevant to the experiment but ignored in a statistical model of it.

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[94] For example, if $\alpha = E[X_i]$ is the population mean, the sample mean $\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_i$ is the method of moments estimator.

[95] In Bayesian settings, however, the distinction blurs; see §8.

[96] The sample mean is the best zero-dimensional fit to the data.

[97] Various interpretations are possible. The two most common are that $P(A)$ is the long-run frequency of occurrence of A in repeated, independent trials, and that $P(A)$ is a subjective assessment of the likelihood of A.

[98] This property is known as *additivity* of P.

Random variable. A (real-valued) function $X(\omega)$ of the outcome ω of a random experiment.

Random variables are *identically distributed* if they have the same distribution function.

Resampling. Class of simulation techniques used to estimate distributional characteristics of statistical estimators not accessible by means of theory or direct estimation. The basis is to simulate additional pseudo-data from the empirical distribution function of the original data, and to estimate the distributional characteristics (used, e.g., to produce confidence intervals or standard errors) from these. The *bootstrap* [27, 37] is the most widely known of these techniques.

Sample mean. Given data $X_1, \ldots, X_n$, the statistic $\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_i$.

Sample variance. Given data $X_1, \ldots, X_n$, the statistic $S^2 = \frac{1}{n-1}\sum_{i=1}^{n}(X_i - \bar{X})^2$.

Sample standard deviation. Given data $X_1, \ldots, X_n$, the statistic $S = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(X_i - \bar{X})^2}$.

Simulation. Class of computer models and techniques for studying systems involving randomness, but for which analytical solution is not feasible.

Standard error. Statistical estimator of the standard deviation of another statistical estimator. For example, given data $X_1, \ldots, X_n$, "the" standard error of the sample $\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_i$ is often taken to be $S/\sqrt{n}$, where S is the sample standard deviation.

Standard deviation. For a random variable X, the quantity $\sigma_X = \sqrt{\sigma_X^2}$, whose interpretation is similar to that of the variance σ_X^2, but is in the same units as X.

Statistic. In a random experiment, any random variable that is a function of the data. Given data $X_1, \ldots, X_n$, common statistics are the sample mean, sample variance, sample standard deviation and order statistics and empirical distribution function.

Statistical estimation. Techniques for estimating from data the parameter value α actually governing a random experiment, applied within the context of a particular statistical model for the experiment.

Three general principles of estimation are maximum likelihood, the method of moments and least squares. In some settings, two or all three of these methods lead to the same estimators. *Ad hoc* methods are widely employed as well.

Statistical estimator. Statistic meant to estimate the value of the unknown parameter α of a statistical model. Given data $X_1, \ldots, X_n$, estimators, as statistics, are functions of the data, and are usually denoted by carets: $\hat{\alpha} = h(X_1, \ldots, X_n)$ for a function h specified by the estimation procedure. Two key characteristics of an estimator $\hat{\alpha}$ are its bias and mean squared error. Zero bias and low mean squared error are desirable properties, but ordinarily one is enhanced at the expense of the other.

Statistical model. A mathematical representation of a random experiment, consisting of

- A *sample space* Ω, the set of all possible outcomes of the experiment. Outcomes may range from simple ("yes" or "no") to complicated (micrographs in materials science).
- A collection of potential governing mechanisms for the experiment, represented mathematically as an indexed family $\{P_\alpha : \alpha \in I\}$ of probabilities on Ω. One of these probabilities, corresponding to a specific value α, actually does govern the experiment.
- *Data* comprised in simple cases of random variables $X_1, \ldots, X_n$,[99] and interpreted as measurements or observations. The form of the data may be dictated by the "physics" of a particular application or be under control of the scientist/statistician, who may decide which measurements to take.

Within this setting, the goal of statistical inference is to construct and evaluate meaningful statements concerning the mechanism actually governing the experiment, based on the data.[100]

Stochastic process. A family $\{X_t : t \in T\}$ of random variables, where T is any index set, typically representing time or space.

Systematic error. Error resulting from an inappropriate statistical model, and so caused by inconsistency between assumptions underlying the model and the random experiment being modeled. Examples are unwarranted assumptions of independence or inapplicable distributions. Systematic error[101] potentially invalidates all inferences drawn using a particular model.

Tests of hypotheses. Statistical procedures for ascertaining the support of the data for statements about the unknown parameter of a random experiment. *Simple* hypotheses specify one value for the parameter; *compound* hypotheses specify multiple values.

Hypothesis tests are expressed in terms of a *null hypothesis*[102] H_0, for example, that $\alpha = \alpha_0$, and *alternative hypothesis* H_1, say that $\alpha = \alpha_1 \neq \alpha_0$. A test based on data $X_1, \ldots, X_n$ is specified by a *test statistic* $T = h(X_1, \ldots, X_n)$ and a *critical region* C, such that H_0 is rejected if $T \in C$. Often, the critical region is an interval.

Being based on uncertain data, hypothesis tests are not flawless. Two kinds of errors are possible. A *Type I* error constitutes a "false rejection" of a true null hypothesis, while a *Type II* error is acceptance of the null hypothesis when the alternative is true. Ideally, the probabilities of both errors should be small, but decreasing one almost always increases the other. The

[99] In other instances, the data may be a sequence $X_1, X_2, \ldots$ or a stochastic process.

[100] Customary usage designates as *parametric* models in which the index set I is finite-dimensional and all others as *nonparametric*.

[101] Also known as *model mis-specification*.

[102] Typically, a "straw man," stating that what is suspected not to be true (for example, that a coin is fair) actually is true.

Neyman–Pearson paradigm minimizes the probability of type II error subject to a prescribed upper bound on the type I error.[103] The *power* of the test is the probability of rejecting the null hypothesis when the alternative holds.

Test results can also be expressed as *p-values*. For a ("one-sided") test in which the rejection region is an interval $[c, \infty)$ (that is, H_0 is rejected if $T \geq c$, where c must be determined), the p-value (sometimes, *observed significance*) is the smallest value of p_0 for which the null hypothesis would be rejected on the basis of the observed data.[104] The smaller the p-value, the more the data support the null hypothesis.

Tuning computer models to physical data. Statistical procedures for using available physical data to select values of parameters ("tuning constants") in numerical simulation models. These parameters are ordinarily phenomenological, that is, cannot be measured physically, even in principle. Tuning differs from statistical estimation: here model outputs are only obtainable numerically, and are not necessarily comparable to the physical data. Error may be present in all measurements. See [14] for one formulation.

Variance. For a random variable X, the quantity $\sigma_X^2 = E\left[(X - E[X])^2\right] = E[X^2] - E[X]^2$, which is the average (squared) deviation of X from its mean $E[X]$.

Weibull distribution. Distribution function given by (C.3), developed in [130] for study of failure of materials.

As m increases, the mean stabilizes at σ_0 and the variance converges to zero,[105] so that materials with higher Weibull moduli exhibit less variability.

[103] That is, the optimal critical region C^* solves the optimization problem of minimizing $P_{\alpha_1}\{T \notin C\}$ subject to a constraint of the form $P_{\alpha_0}\{T \in C\} \leq p_0$, where p_0 is the bound on type I error.

[104] That is, the p-value is the statistic $q(T)$, where $q(t) = P_{\alpha_0}\{T \geq t\}$, which can be calculated (from the data) without specification of an alternative hypothesis.

[105] More precisely, the mean is $(\sigma_0/m)\Gamma(1/m)$ and the variance is $\sigma_0^2\left[(2/m)\Gamma(2/m) - [(1/m)\Gamma(1/m)]^2\right]$, where $\Gamma(x) = \int_0^\infty t^{x-1}e^{-t}\,dt$ is Euler's gamma function.

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