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# MATERIALS TESTING IN THE 21ST CENTURY

JAMES W. McCAULEY  
MATERIALS SCIENCE BRANCH

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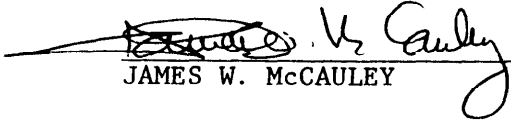
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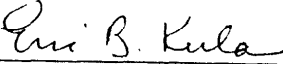
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## ABSTRACT

In the 21st century, society will require materials with the maximum achievable, performance and reliability at the lowest possible cost. High performance ceramic, polymeric and composite materials will have replaced traditional metals in many engineering applications. Exotic surface modification techniques will be in common use. In order to cope with these enormous changes, the advanced materials and testing communities must recognize the importance of materials characterization concepts for controlling and monitoring a material's full unique signature of chemical, structural, and defect characteristics, compared to traditional reliance on post manufacture flaw interrogation for property assurance. This will require the extension of traditional NDT into chemical and microstructural interrogation, transitioning sophisticated materials characterization techniques out of the research laboratory, establishing material unique signature and property knowledge bases and developing new concepts in sensor technology.

  
JAMES W. McCAULEY

APPROVED:

  
ERIC B. KULA  
Director  
Emerging Materials Division

## MATERIALS TESTING IN THE 21ST CENTURY

James W. McCauley  
U.S. Army Materials Technology Laboratory  
Materials Characterization Division  
Watertown, MA 02172

### ABSTRACT

In the 21st century, society will require materials with the maximum achievable performance and reliability at the lowest possible cost. High performance ceramic, polymeric and composite materials will have replaced traditional metals in many engineering applications. Exotic surface modification techniques will be in common use. In order to cope with these enormous changes the advanced materials and testing communities must recognize the importance of materials characterization concepts for controlling and monitoring a material's full unique signature of chemical, structural and defect characteristics, compared to traditional reliance on post manufacture flaw interrogation for property assurance. This will require the extension of traditional NDT into chemical and microstructural interrogation, transitioning sophisticated materials characterization techniques out of the research laboratory, establishing material unique signature and property knowledge bases and developing new concepts in sensor technology.

### INTRODUCTION

The successful and widespread commercialization of advanced ceramic materials, involves, among other factors, reducing the cost of raw materials and final product manufacturing, improving their reproducibility and reliability and minimizing their susceptibility to unpredictable catastrophic failure. Innovative materials testing/characterization concepts can make a significant impact on all of these problems. (1,2)

### ADVANCED MATERIALS

Current extremely high reject rates(3) ( $50 \pm 25\%$  of total manufacturing costs) of high performance ceramics and excessive problems with prototype high technology systems suggest that significant changes must take place given these current trends and future requirements. The emergence of advanced ceramic materials as viable candidates for metal-limited performance of many engineering systems has placed new demands on the technology surrounding these materials. Critical properties of emerging advanced ceramics in many cases depend on the characteristics of 30 to 1000 Å grain boundary phases. The interfacial region between fiber and matrix in composites has a

profound effect on properties. With the advent of new materials, joining techniques are relying more and more on advanced concepts over traditional welding and mechanical fastening. Optimum bonding in these cases depends critically on the nature (chemistry and structure) of the joining faces. The drive towards pushing the performance of materials toward their theoretical limits necessitates sophisticated characterization, since minute changes in chemistry and structure, besides defects, have profound property effects.

The emergence of advanced materials means that the 20th century engineering experience with traditional materials, like steel and aluminum, must be replaced in the 21st century by a functional appreciation of the unique characteristics of powders, fibers, advanced ceramics, composites, polymers, high performance metals and surface modified materials. Treating these materials as isotropic "black boxes" will result in enormous problems in the future.

Exacerbating the situation is the lack of uniform nomenclature in regards to material constituents and testing procedures. Over the years the metal's community have successfully evolved commonly accepted nomenclature for material compositions and processing treatment (e.g. Al 7075, T<sub>6</sub> condition). Presently the various advanced materials groups are still struggling with these issues and uniform nomenclature and standardization is many years off. Figure 1. is a schematic representation of a very generalized advanced materials technological progression from origination to incorporation into a system; the approximate 25 year time scale may even be over optimistic in many cases. This attempts to illustrate the required actions for the full maturing of a materials technology, including nomenclature and standardization. Vertically integrated industries that produce their own materials do not worry as much about the uniformity and standardization issues of nomenclature, testing procedures, and the like, however, the Department of Defense, like many other industries without in-house material production capabilities, require detailed specifications and verification procedures to assure the quality of purchased material. Further, even the vertically integrated industries, in some cases, must be able to demonstrate conformance of material components to required, standardized characteristics and properties.

In the past, over-design of engineering systems assured structural integrity by providing a "margin of error" in design accuracy or material quality. The structural integrity of a system, is defined as the assurance that all critical components will perform their functions (satisfactorily) in the environment in which they were intended to operate. Often times the design accuracy has been overemphasized over material correctness (quality), with the former being understood as relating to the following definitions:

- Uniformity: control of character variations in a single piece,
- Reproductibility: control of variations from piece to piece,
- Reliability: uniformity and reproducibility of a desired property value continuously,
- Quality: reliable conformance to requirements.

The extensive use of "forgiving" metals provided additional tolerance in engineering systems. However, the complex characteristics of advanced materials and the inherent brittle and surface sensitive nature of ceramics, together with reproducibility problems have reduced the margin for acceptable error to very small values. Hidden "defects" like subtle differences in porosity, phase composition, microstructure retained strain (residual stress) and sub-critical cracks can result in properties well below acceptable levels, even though traditional nondestructive interrogation revealed no gross inhomogenities, cracks or voids.

#### FUTURE PERSPECTIVE

The problems discussed in the preceding section are not insurmountable, but will require much planning and hard work to overcome. Furthermore, much of the effort will involve the less "glamorous" areas of materials science and engineering - characterization and property data collection, storage and analysis. Of course some very practical determinations must be made on how much effort to put into certain long range efforts. For example, the total cost of the material component (e.g. \$100-100,000/kg) would demand special attention. Life dependency on even the cheapest of material components (e.g. <\$1.00/kg) would also demand special attention. Further, sometimes the performance requirement alone (e.g. winning the Indianapolis 500) would demand special attention to key components. All of these factors must be totally analyzed in advance to evolve a practical material's quality program.

The writer's perception is that total quality can be achieved, even in the most complex of materials, if so desired. The path to this goal will be long and sometimes torturous, but it is achievable. Major institutional barriers will intercede along the way, primarily because of major changes to commonly accepted philosophical approaches to the problem and the cost and length of time associated with the total effort required.

It is now generally agreed that the quality of advanced materials must be improved if they are ever going to have large scale applications in many high performance applications. The route to this end is still somewhat controversial with some groups(4), for example, recommending the use of ideal starting materials (powders) while other groups are recommending in-process manufacturing/process controls.(5-8) These two alternatives are not mutually exclusive and a careful evaluation of the end use will, in many cases, help determine the optimum approach to material quality. For example, the use of advanced ceramics in electrical or primarily mechanical systems require different types of control on material characteristics.(9) In other cases, the availability of "ideal" powder may be impossible so an alternative approach may have to be used.

Methodologies also exist to use a variety of quantitative methods for improving the quality of manufactured goods.(10-12) The Union of Japanese Scientists and Engineers (JUSE), besides being a major quality control educational center, have even recognized the importance of Deming's concepts to quality control by establishing the world famous Deming Prize. Unavailability of methodology for the application of these quantitative methods on advanced materials is a major impediment for their more widespread use.

Traditionally, there has been a reliance on flaw detection after production and methods like proof testing and mathematical/statistical lifetime predictions prior to use. Use of the emerging, high performance materials and the desire to extract the maximum performance from them in engineering systems almost assuredly will make these methods unacceptable in many situations. One of the keys to the successful utilization of these quantitative manufacturing methods is the quantitative knowledge of key material characteristics and properties in all stages of production and usage. For some materials and material components, limited aspects of this concept is already possible. In the 21st century, the writer believes that full interrogation of material in production and in service will be possible and practical in a strict (or nearly strict) completely nondestructive mode. Contact sensor packages (transducers) and test/characterization equipment will be designed for permanent or temporary adherence to a material for the periodic evaluation of the materials characteristics and properties - even in fielded systems. Information, like date and place of production, original composition and structure, remaining service life, etc. will be coded into the material, or nearby in the system, by special interrogatable techniques similar to grocery store bar codes or specially designed micro-computer chips or sensor packages. These micro packages could also be designed to count cycles, temperature excursions, etc. in a particular engineering system and by coupling to a properly configured computer system will make maintenance a routine matter. Of course, it will also be possible to obtain much material information in a completely non-contact mode and choice of any method will involve all the issues mentioned previously.

#### REQUIREMENTS FOR FUTURE PERSPECTIVE

Basically, for efficient, high quality productivity and reliable utilization, methods will be needed in many situations to evaluate a material in all phases of the manufacturing process and while it is in service. In order to accomplish this, quantitative methods are needed to unambiguously define a material and its properties. There are also instrumental methods required to obtain this information from the material and to provide it to a human or specially configured computer system for decision making during production or while in service. The key aspects of this perspective consist of 1.) Materials Information Technology, 2.) Instrumental Requirements, and 3.) Management Support.

#### Materials Information Technology

Any engineering system requires certain material properties for performance. In an ideal world, any extrinsic property of a material is a function of its intrinsic characteristics. Therefore, by identifying the unique characteristics of the material the properties are also defined in an unambiguous way. The converse is not true. Identification of a property does not uniquely define a material. This is the fundamental ambiguity of advanced materials. In the real world the unique characteristics of any material not only consist of chemistry and physical structure of its constitutive components, but also include defects.

Materials characterization has been very succinctly defined by a National Academy of Science Materials Advisory Board Report.(13) "Characterization describes those features of the composition and structure (including defects)

of a material that are significant for a particular preparation, study of properties, or use, and suffice for reproduction of the material." The unique signature of any material therefore is a set of the necessary and sufficient characteristics of a material suitable for unambiguous, quantitative description of a material" or in equation form(14):

$$P = f(c,M,PD) \tag{1}$$

- c = chemistry, phases
- M = Microstructure (physical structure)
- PD = Processing Defects
- P = Property

Therefore, any property of a material can be unambiguously defined by its unique signature. Using this simple concept, a material's full life cycle can be schematically illustrated as shown in figure 2. A variety of concepts are illustrated in this figure. In order to insure certain performance requirements, performance specifications should be replaced with material specifications and design allowables obtained via a materials science and engineering approach: materials science being the relation between material characteristics and properties, while materials engineering is the relation of properties to performance criteria. Figures of merit have typically been used to relate a required performance to a set of properties. In theory, therefore, materials can be tailored to meet specific requirements. The right side of this chart defines the technological pushers, while the left side defines the pullers.

Materials characterization is critical in defining the unique signature of the processed material (c,M,PD) and also for assessing the service effects. Chemistry, microstructure, and processing defects may change as a result of the service environment, besides the formation of new defects (SD). The service related signature may, therefore, be defined as follows:

$$P' = f(\Delta c, \Delta M, \Delta PD, SD) \tag{2}$$

- $\Delta c$  = changed composition
- $\Delta M$  = changed microstructure
- $\Delta PD$  = changed processing defects
- SD = new service-related defects
- P' = changed property

Figure 3 illustrates an overall future concept for implementation of full cycle materials testing/characterization. The key area of focus involve the evaluation of techniques to fully characterize starting materials and material during processing. The techniques should be amendable to automation and computerization for feedback control. Powder characterization and green body characterization will be critical. For in-process characterization, the relation between measurable characteristics or properties and desired properties via contact or noncontact sensors is the key aspect for this part of manufacturing. Sensor technology must be advanced as well as quantitative relationships developed between measurable characteristics and desired properties.

Figure 4 is a schematic representation of a typical powder processing situation. Areas where characterization is important are indicated in several different stages, including after the powder is in the processing container. The next figure (5) illustrates how the simple act of pouring a typical metal powder segregates the powder by size and distribution in the final container.(15)

Obviously, the determination of the full unique signature of a material may be cost or technically prohibitive. However, the concept provides a sound quantitative framework for the derivation of data bases suitable for computer interrogation in an artificial intelligence/expert system mode if desired. Two routes are possible: either direct measurements of unique signature or measurement of other characteristics (like density, sonic velocity, etc.) which are a direct function of the unique signature and can be carried out nondestructively.

The establishment of data bases suitable for these purposes involves generating techniques for the collection and storage of data in computer interrogatable form. Collection of information for the data bases must begin in the research laboratory. At the U.S. Army Material's Technology Laboratory a start on this problem has been initiated with a computer program (MIMS - Materials Characterization Information Management System(16) to help decide what information to measure, and to then store the information for administrative or technical use. Figure 6 is an illustration of the form used to develop the MIMS system.(17) At MTL we are also collaborating with the Department of Energy and Oak Ridge National Laboratory in Annex II of the International Energy Agency agreement between the United States, Sweden and the Federal Republic of Germany(18) to identify better analytical standards and standard procedures for characterizing advanced ceramic materials. Part of the task involves determining methodology for the data base storage of the unique signature of ceramic powders. Figure 7 is an illustration of a preliminary format for the storage of particle size distribution data.(19) Figure 8 is an example of a data set for Zr metal powder, including the critical use property of burn time. From these data a simple relationship was determined

$$\text{Burn Time (sec)} = 1.35 (\text{FSSS}) - 0.44 \quad (3)$$

to predict burn time by a simple Fisher sub-sieve size measurement.(20) Hence, for this case, the determination of the Fisher sub-sieve size (permeametry number) may be all that is required, in most cases, to assure the quality of the Zr metal powder for certain applications.

The proper collection and storage of materials information in computer interneegotiable data bases is the subject of much debate, discussion and programs(21-28). Since the processing of commercially available advanced materials is often proprietary, processing data bases will probably not be available publicly in usable form for the foreseeable future. This is exactly why the unique signature concept is so important in identifying final products and their resultant properties. If this is done properly, the processing conditions are not required. Figure 9 summarizes the data bases that should be assembled; vertically integrated companies can easily collect all of these data bases for their own use, whereas others must rely on the final product and service data bases for their decision making.

## Instrumental Requirements

There are many talks during this meeting dealing with state-of-the-art characterization and testing procedures, so these do not have to be reviewed here. The important point to understand is that the successful application of the preceding full life cycle materials quality concept will require some new directions in equipment development, design and utilization.(29,30)

Decreasing the size of equipment, minimizing power requirements and the interfacing of powerful, but small, computer systems is resulting, and will keep resulting, in major advances in material testing and characterization. The impact of the new high temperature superconductors will also be very important. The ability to process many electromagnetic signals simultaneously with two-dimensional detectors is also having a profound effect on all of the technology. The coupling of computers to these new systems allows for data collection and analysis in extremely short periods of time. Further, in these systems, extremely complex mathematical smoothing and modeling algorithms can also be used to extract information only dreamed of a few short years ago. For example, much information on material characteristics and properties can be obtained using X-ray diffraction and ultrasonic interrogation. The use of X-ray diffraction for measuring residual stress in material has progressed(31) from taking several hours in 1925 to where it will soon be possible to carry out these same measurements without any moving parts with a two-dimensional position sensitive detector in less than a minute(32). Further, similar methods may also be used as a fatigue damage indicator(33). Other applications of X-ray diffraction include fast evaluation of fiber and polymer microstructure(34,35). Figure 10 is an illustration of this technique on KEVLAR and the speed realizable by using a position sensitive detector. The technique is also adaptable to "SiC" fibers as is illustrated in figure 11. A quantitative measure of crystallite size in SiC fibers can be determined.(36)

Another very important technique is ultrasonic characterization of materials.(37) With the creative use of this technique direct correlations to mechanical properties can be obtained.(38) Figure 12 illustrates its use on large plates of SiC. By precise measurement of velocity, direct measure of subtle differences in density are easily determined.(39-41)

Eventually, the total theory and experimental data package for X-ray diffraction and ultrasonic techniques discussed above can be built into the hardware of small computer devices that will automatically provide microstructure, fatigue or remaining life - time data, as only a few examples. However, work must be kept going in all of the similar areas on materials projected for future use so that the technical principles and experimental data are well documented and amenable for incorporation into computer software and, eventually, hardware.

In some cases, the measurement technique requires that a special contact be made with the tested material. In other cases, a vacuum, or at least a partial vacuum, must be formed. Innovations, therefore, must occur in joining technology so that minimal disturbance of the material occurs, but also provides the necessary coupling condition for proper measurement.

More and more equipment that was normally confined to the research laboratory will be made available for use in the factory and the field and work should be emphasized to enable this to happen. For example, with portable suction cup-type vacuum systems, particle and photon beam surface and bulk analysis could be carried out anywhere. The computing power that can be coupled to equipment suggests that other probing techniques, like microwave or millimeter waves, could also be useful for material interrogation.

#### SUMMARY

In the 21st century miniaturized testing and characterization equipment will be available for factory and field use. For the optimum utilization of this equipment much work is needed in the following areas:

- materials information technology,
- contact and non-contact sensor packages,
- nondestructive attachment and vacuum forming procedures,
- further research on data available from all types of probing techniques.

Finally, management personnel and decision makers must fully support these evolving total quality concepts for long periods of time in order for them to succeed.

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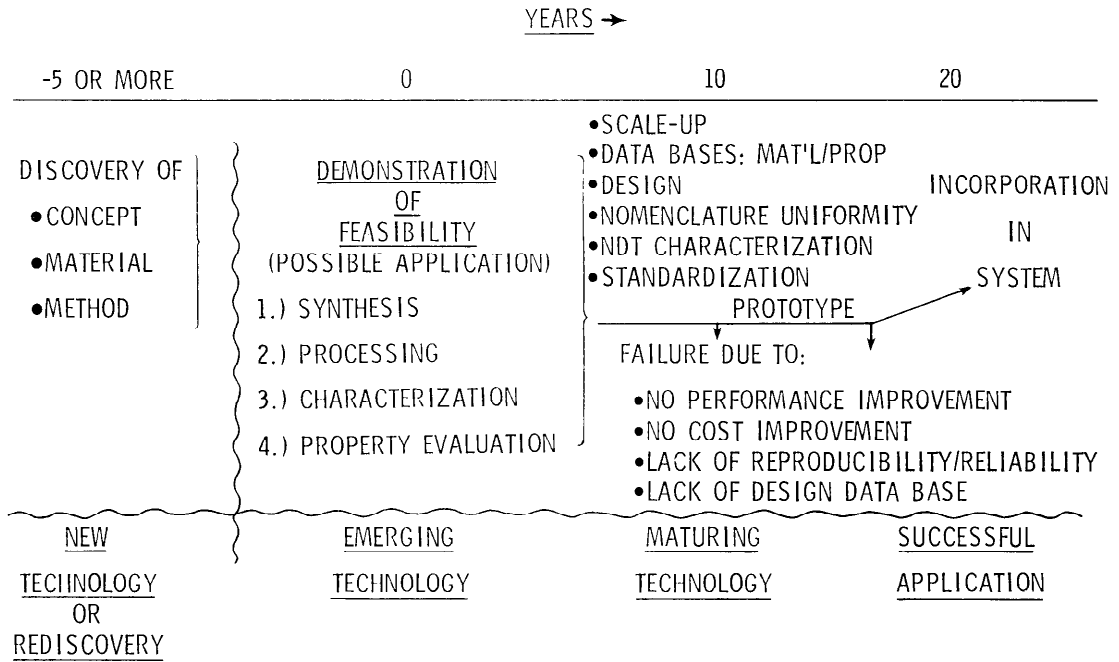


Figure 1. Materials Technology Evolution

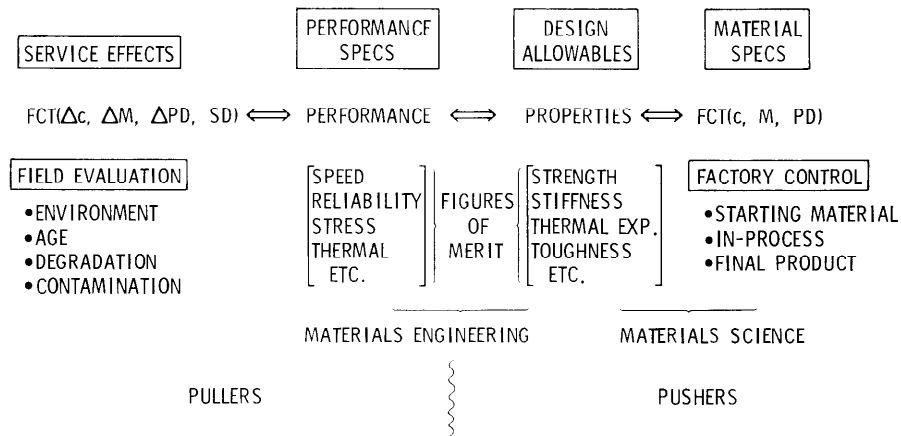


Figure 2. Full Life Cycle Materials Characterization

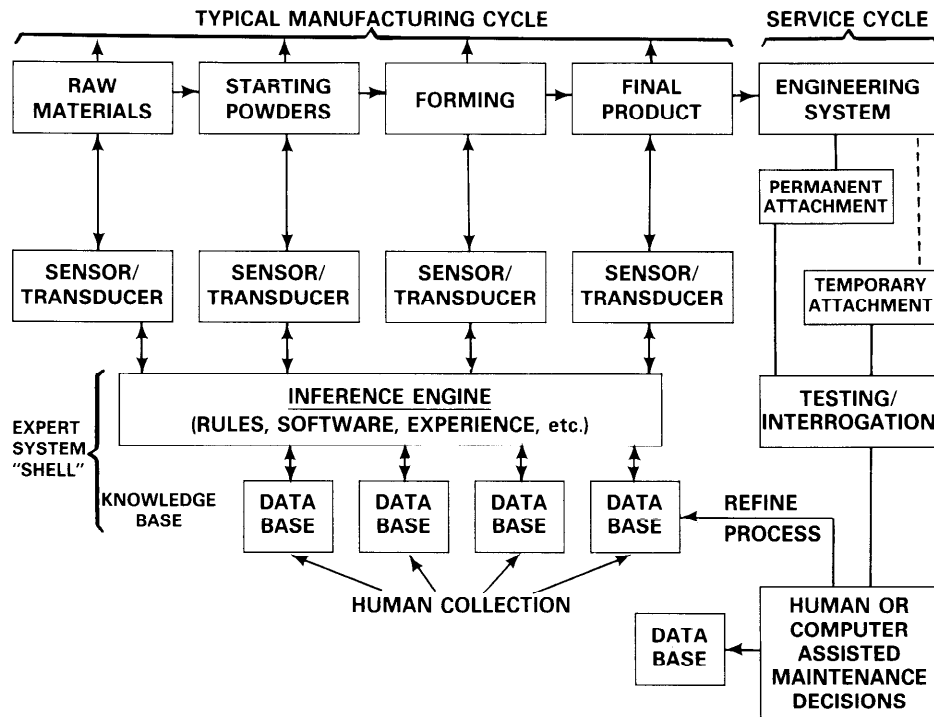


Figure 3. Implementation of Full Cycle Materials Testing/Characterization

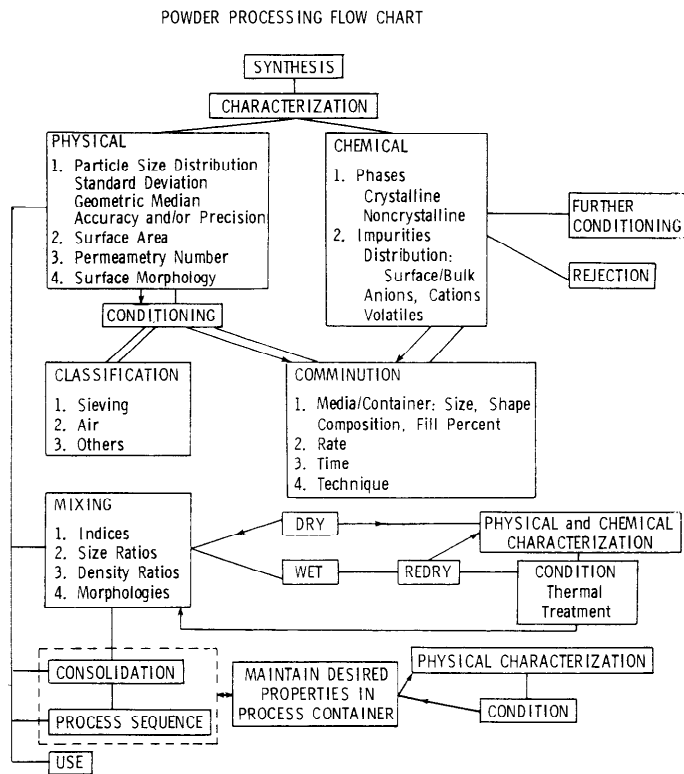


Figure 4. Aspects of Characterization in Processing Ceramic Powders

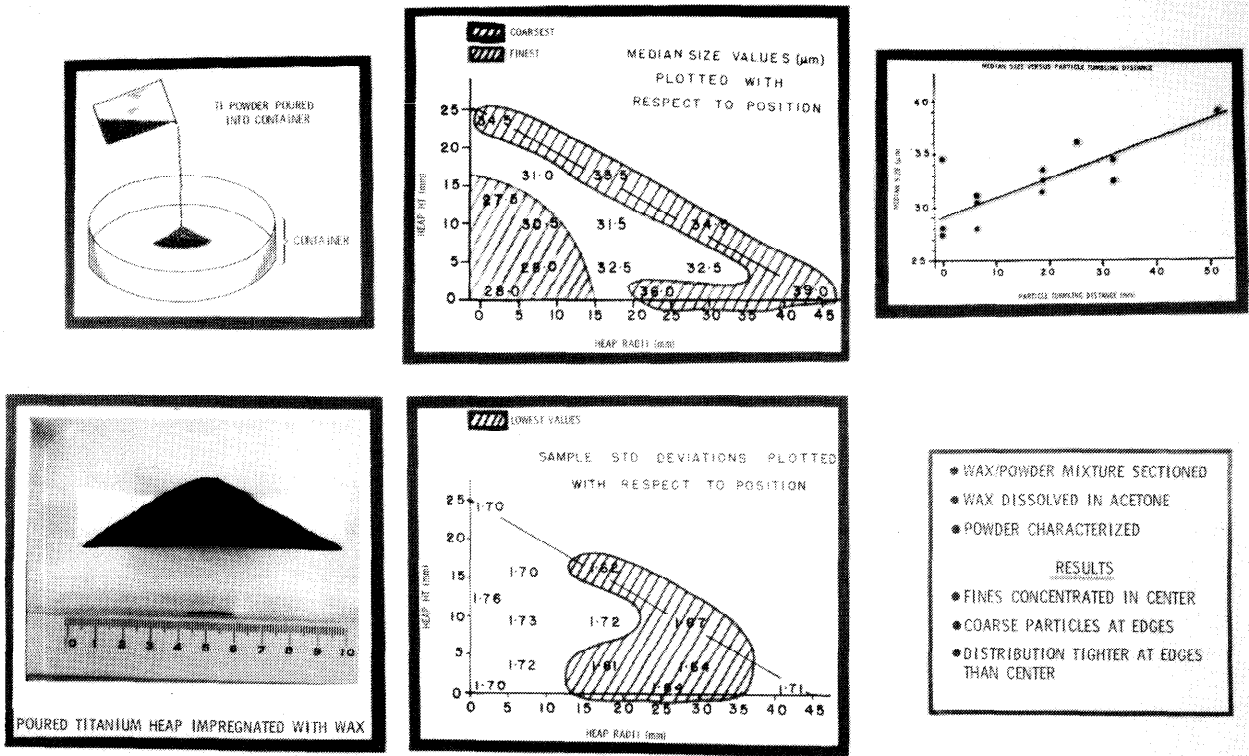


Figure 5. Powder Segregation in Processing Container


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Customer Job ID:		MIMS No:	Cost Code:	Non-MTL Org:		
MCD POC:		Phone:	Char. Cat:	Disposition of Samples:		
Priority: (Customer ___; MCD ___)    Max hours ___    Hours used ___    Date Required ___    Date Completed ___						
DESCRIPTION OF MATERIAL: Classification (U,C,S, or TS):    Title:						
No. of samples:		Army System Involved:				
Type (ORG/OMC/CER/CMC/MET/MMC/OTH):		Specific Type:				
History: MELT ___ SINT ___ TH/MECH ___ MACH ___ OTHER ___		Safety: RADIOACT ___ CAUS ___ EXPLO ___ OTHER ___				
Comp: MAJOR ___ MINOR ___ TRACE ___		COMMON NAME				
CHARACTERIZATION REQUIRED: [Make additional COMMENTS in the space below keyed to a (1)]	Bulk Chemistry:	QUAL ___	QUANT ___	S-QUANT ___	PHASES ___	CONTAMINANTS ___
	Spatial Chemistry:	SURFACE ___	GRAIN BOUND ___	FIB/MAT INT ___	OTHER ___	
[Make additional COMMENTS in the space below keyed to a (1)]	Structure:	DENSITY ___	ATOMIC ___	MICRO ___	MACRO ___	GRAIN/PORE SIZE ___
	Defects:	%MATRIX ___	%FIBER/FILLER ___	VOIDS/PORES ___	OTHER ___	
	Particulate:	RESIDUAL STRESS ___	RADIOGRAPHY ___	ATOMIC ___	OTHER ___	
BULK CHEMISTRY: ICAP ___ NAA ___ O/N ___ AA ___    LIST ELEMENTS FOR WHICH ANALYSIS WET ___ C/S ___ NMR ___ XRF ___ EL AN ___    IS DESIRED: Primary (up to 5): E-SPEC [ARC ___ SPARK ___] OTHER ___    Impurity (up to 4 + UN=unknown): COMMENTS (including compounds for which analysis is desired): Key to (2) in space below						
PHASE ANALYSIS: XRD ___ FTIR ___ NMR ___ JCPDS ___ OTHER ___    COMMENTS [Key to (3) below]						
SPATIAL CHEMISTRY: Auger ___ XPS ___ SIMS ___ STEM ___ ASEM ___ RBS ___ NRA ___ OTHER ___    COMMENTS (4)						
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SPECIAL INSTRUCTIONS AND PERTINENT INFORMATION						
QC/QA: BLANKS ___ DUPLICATES ___ ANALYZE UNKNOWN ___ DETECTION LIMITS ___ NBS STD REF MATL ___ OTHER ___						
Interpretation: DATA CHARTS ONLY ___ SUMMARY ANALYSIS ___ EXTENSIVE ANALYSIS ___						
Comments keyed to numbers in ( ):						
<div style="border: 1px dashed black; padding: 5px;">             (Use an additional page, or the back of this one, for more samples or additional comments.)           </div>						
MCD.	Customer's					
Sample No:	Sample No:	Comments				

Figure 6. Materials Characterization Information Management System

Powder	Lab	I.D.		Modes	d <sub>g</sub> (1)	σ <sub>g</sub> (1)	% [d <sub>g</sub> (1)]	d <sub>g</sub> (n)	σ <sub>g</sub> (n)	% [d <sub>g</sub> (n)]	Method	Normalized Data*				Standard
		IEA	Lab													

\*Normalization: Values normalized using reference material or Hatch-Choate equations.

Figure 7. Preliminary Format for Storage of Particle Size Distribution Data

I.D. Sample	Physical						Chemical				Property sec/10" (Burn Time)
	Sedigraph d <sub>g</sub> (50%)(μm)	σ <sub>g</sub> (84,16)	σ <sub>g</sub> (84)- σ <sub>g</sub> (16)	Sw (BET) (m <sup>2</sup> /g)	FSSS APS (μm)	Sw(BET)/ Sw(d <sub>g</sub> )	% Free Zr	% Mg	Oxygen Content (w%)		
									Leco	Neut. Act.	
J5078A-1	3.15	1.75	0.23	1.23	4.3	4.22	-	-	4.32	2.646	6.1
J5068A-3	1.88	1.64	0.15	1.74	2.4	3.56	89	-	4.43	3.503	3.3
J5068A-4	1.92	1.69	0.17	1.76	2.5	3.68	88.8	-	4.72	3.563	3.1
J5116A-1	1.89	1.65	0.15	1.67	2.9	3.44	88.9	-	4.82	3.575	3.2
J5068A-1	1.87	1.71	0.27	1.67	1.9	3.4	86.1	-	4.86	--?	2.7
J5042A-1	1.8	1.6	0.2	1.67	2.35	3.27	87.7	-	4.95	3.933	2.3
J5042A-3	1.85	1.57	0.13	1.64	2.2	3.3	87.4	-	4.95	3.89	2.2
J5043A-4	1.75	1.65	0.17	1.72	2.12	3.27	87.1	-	5	3.880	2.3
J5116A-2	1.89	1.73	0.15	1.75	3.8	3.6	88.7	-	5	3.773	2.4
J5043A-1	1.85	1.67	0.2	1.52	2.15	3.06	88.5	-	5.01	3.723	2
J5043A-2	1.88	1.61	0.22	1.61	2.2	3.29	87.1	-	5.04	3.912	2.2
J5042A-4	1.75	1.67	0.29	1.68	2.2	3.2	87.7	-	5.08	4.072	2.1
J5065A-1	1.8	1.65	0.32	1.70	2.25	3.33	87.4	-	5.15	3.969	2.4
J5051A-1	1.85	1.69	0.29	1.66	2.25	3.34	87.9	-	5.26	3.855	2.7
J5043A-3	1.8	1.54	0.4	1.88	2.1	3.68	87.7	-	5.41	4.065	2.4

SORTED ACCORDING TO ASCENDING LECO OXYGEN CONTENT

Figure 8. Powder Characterization Data Set for Zr Metal

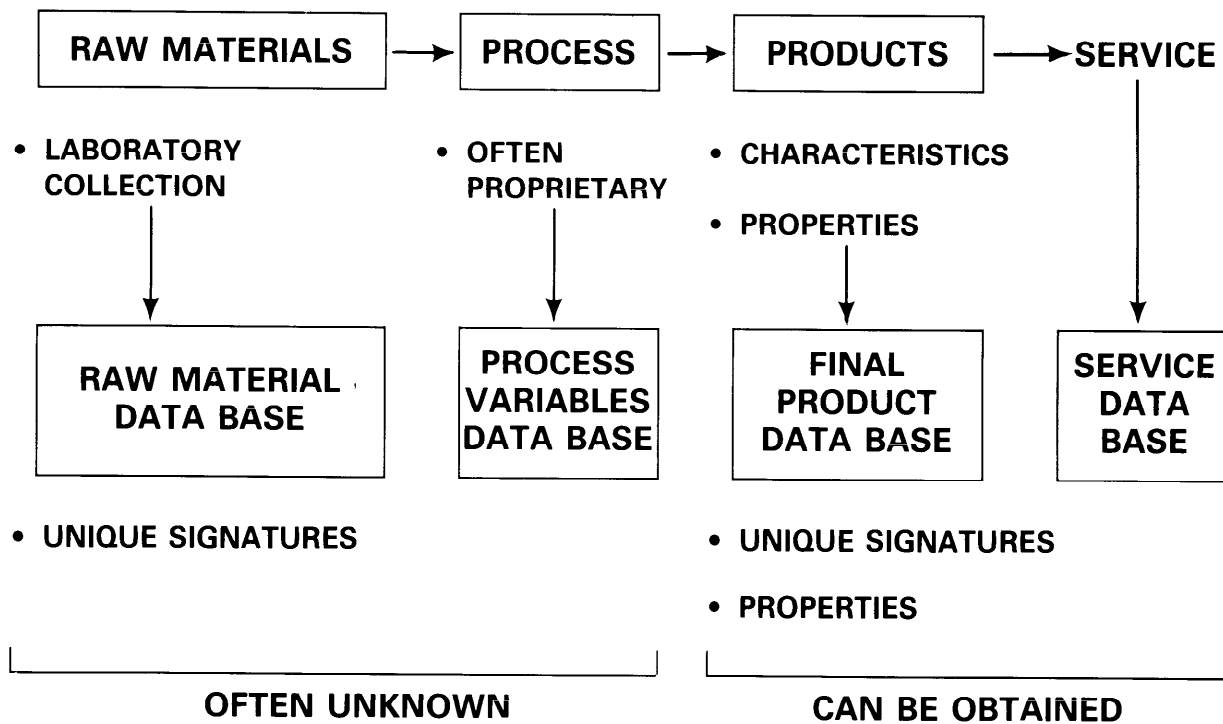


Figure 9. Material Data Bases

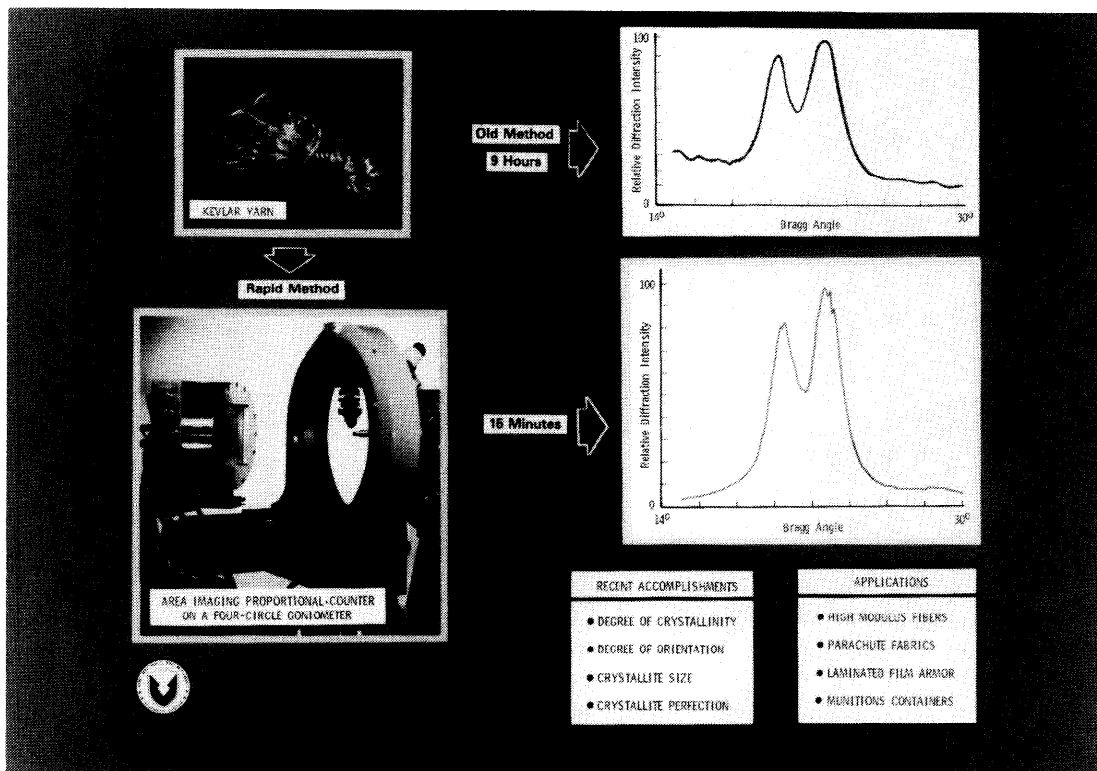


Figure 10. Rapid X-ray Characterization Technique

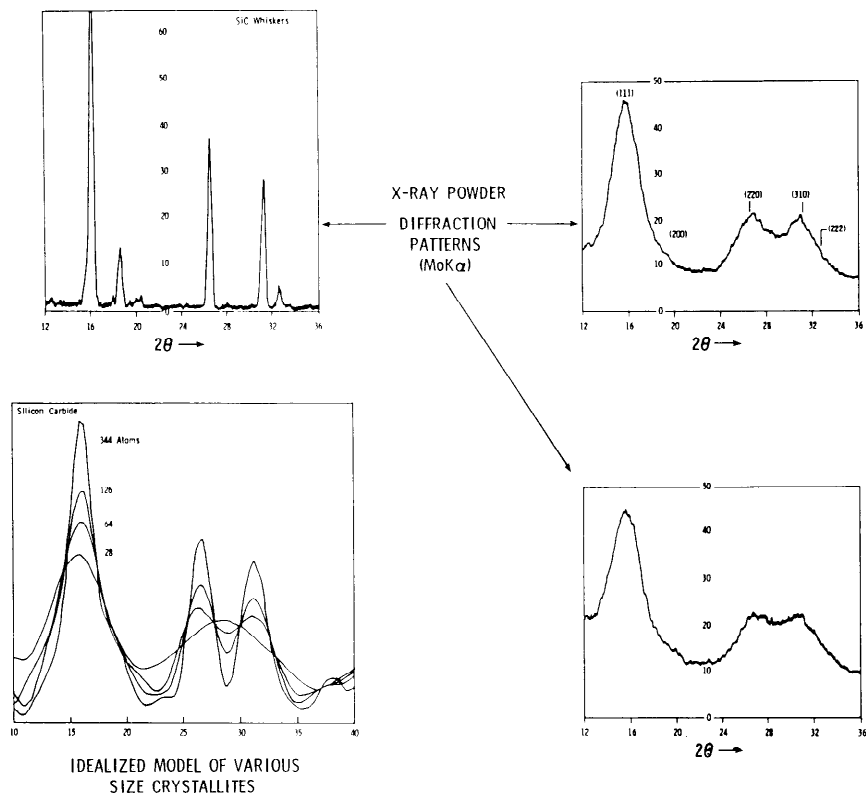


Figure 11. Structure of SiC Fibers

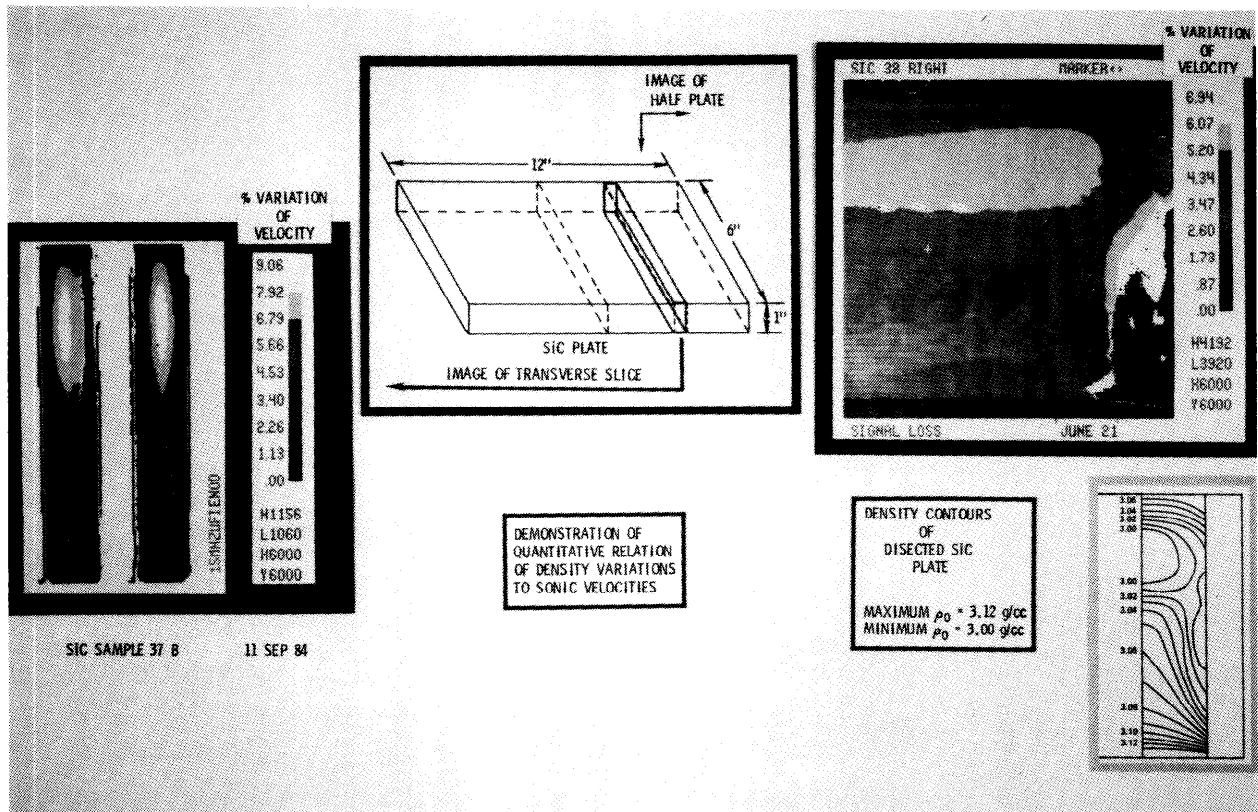


Figure 12. Ultrasonic Image Characterization of SiC Plates

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James W. McCauley

Technical Report MTL TR 88-32, October 1988, 20 pp -  
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In the 21st century, society will require materials with the maximum achievable performance and reliability at the lowest possible cost. High performance ceramic, polymeric and composite materials will have replaced traditional metals in many engineering applications. Exotic surface modification techniques will be in common use. In order to cope with these enormous changes, the advanced materials and testing communities must recognize the importance of materials characterization concepts for controlling and monitoring a material's full unique signature of chemical, structural, and defect characteristics, compared to traditional reliance on post manufacture flaw interrogation for property assurance. This will require the extension of traditional NDT into chemical and microstructural interrogation, transitioning sophisticated materials characterization techniques out of the research laboratory, establishing material unique signature and property knowledge bases and developing new concepts in sensor technology.

U.S. Army Materials Technology Laboratory,  
Watertown, Massachusetts 02172-0001  
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James W. McCauley

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