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Development of a Material Property Database on Selected Ceramic Matrix Composite Materials

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Introduction:

Ceramic Matrix Composites, with fiber/whisker/particulate reinforcement, possess the attractive properties of ceramics such as high melting temperature, high strength and stiffness at high temperature, low density, excellent environmental resistance, combined with improved toughness and mechanical reliability. These unique properties have made these composites an enabling technology for thermo-mechanically demanding applications in high temperature, high stress and aggressive environments such as turbine engines, combustors, nozzles, heat exchangers, boilers, fusion reactor first wall and blanket and so on. On a broader scale, CMC's are anticipated to be applicable in aircraft propulsion, space propulsion, power and structures[1] in addition to ground based applications. However, it is also true that for any serious commitment of the material toward any of the intended critical thermo-mechanical applications to materialize, vigorous research has to be conducted for a thorough understanding of the mechanical and thermal behavior of CMC's. The high technology of CMC'S is far from being mature.

In view of this growing need for CMC data, researchers all over the world have found themselves drawn into the characterization of CMC's such as C/SiC, SiC/SiC, SiC/Al_2O_3, SiC/Glass, SiC/C, SiC/Blackglas. A significant amount of data has been generated by the industries, national laboratories and educational institutions in the United States of America. NASA/Marshall Space Flight Center intends to collect the "pedigreed" CMC data and store those in a CMC database within MAPTIS (Materials and Processes Technical Information System).

The task of compilation of the CMC database is a monumental one and requires efforts in various directions. The project started in the form of a summer faculty fellowship in 1994 and has spilled into the months that followed and into the summer faculty fellowship of 1995 and has the prospect of continuing into the future for a healthy growth, which of course depends to a large extent on how fast CMC data are generated. The 10-week long summer fellowship has concentrated, basically, on establishing the procedure for a smooth transfer of data into a CMC database on MAPTIS which is a vital part of the following broader picture of the project.

CMC Database in MAPTIS

- **Data Acquisition**
  - Phone Contact
  - Lab Visits
  - Data From Literature
- **Data Analysis**
  - Literature Search For Models For Data Validation
    - Reading Reports To Check For Adequacy Of Information On the Material, Test Techniques and Material Properties
- **System Development**
  - Hardware & Software Compatibility & Procurement
- **Data Transfer**
  - Screen and Input File Generation In MAPTIS

Data Acquisition:

Several major industries, national laboratories and educational institutions of America have been involved in the generation of CMC data. Some of those are:

NASA Lewis / Rocketdyne / GE / Brockmeyer and Schnittgrund[2] :
Ceramic Composites for Earth-to-Orbit Engine Turbines
DuPont Lanxide / Wright Patterson AFB[3] :
C/SiC Turbine Rotor for an Expendable Missile Engine
WPAFB / Williams International / DuPont / Dow-Corning-Kaiser / General Atomics[4] :
Ceramic Composites such as HPZ/SiC, C/Si_3N_4, SiC/Al_2O_3(with SiC_p) for combustor components in the WR24-7 engine.
Southern Research Institute/WPAFB[5] :
Ambient and Elevated Temperature behavior of C/SiC composites

In general, CMC's are being researched vigorously for applications such as Gas Turbine Engines, Hot Aerostructure, Space Based Systems, Thermal Protection Systems, Antenna Windows, Rocket Propulsion

Contacts :

Throughout the fellowship and contract, contact has been established and maintained with the following: DuPont Lanxide, Williams International, Southern Research Institute, McDonnell Douglas, Wright Patterson AFB, Dow-Corning / Kaiser, BF Goodrich, Pratt & Whitney, NASA Lewis, Oakridge Laboratory, Voight Corporation, Boeing, Northrop / Grumman, CINDAS / Purdue, General Electric, Lockheed / Martin, University of Michigan.

This effort has led to the acquisition and promise of a variety of CMC data for storage into the CMC database here at NASA/Marshall Space Flight Center. To mention a few:

1. Ceramic Composite Turbine Component Evaluation : Williams International Report
Contains information on flat panel and combustor component (combustor cover and primary plate) testing of four cmc's, namely, DuPont Lanxide fabricated HPZ/SiC (CVI) and Nicalon/Al_2O_3(SiC_p reinforced) via DIMOX, General Atomics fabricated C/Si_3N_4 via a forced flow thermal gradient CVI method and Dow-Corning/Kaiser fabricated Nicalon/SiNC via Polymer Infiltration and Pyrolysis (PIP). The data presented in the report are comprised of mechanical property tests conducted at the University of Michigan, thermal conductivity tests at Purdue University and thermal expansion tests at Harrop Industries and of as received as well as engine tested data. The report also carries information on the use of Non Destructive Techniques (NDE) such as Thermal Wave Imaging as well as stress analysis data.

2. Turbine Rotor Development Program : DuPont Lanxide Composites, Inc. Report
Presents data on a silicon carbide matrix reinforced with carbon fibers (C/SiC) generated with the objective of developing and demonstrating the technology necessary to produce a CMC turbine rotor for a missile engine. A variety of physical and mechanical data show the effect of optimum fiber structure, reinforcement unit cell size, porosity and fiber volume, oxidation protection. Information on NDE techniques as tools for the evaluation of infiltration of rotor shaped test articles (C/SiC rotor disks), identification of optimum steps between CVI cycles to machine thick CVI parts etc. have been also incorporated. Blade and disk subelements were developed for rig evaluations to address critical areas of concern for structural requirements unique to the blade root and rotor bore regions of a turbine rotor.

As is the case with any good database, it is absolutely essential to look for "pedigreed" data. With this in mind, a specific request sheet has been made available to the data suppliers. The format of the files required by MAPTIS is likely to help refine this request sheet. The question of pedigree also calls for effort in two additional directions:
a. Evaluate the standards maintained and care taken in the experimental procedures used to generate the data. A thorough study of the reports, asking questions, and, especially, visiting labs has been found to be of considerable help.
b. Study the current literature and extract information on how CMC's ought to behave and look for the possibility of applying any model to the data. The literature, has at times, led to useful data also.

The Data :

The data have been supplied by the data sources in the form of both graphs and tables. The reports also provide information on the specimen geometry, objectives, experimental procedures etc. which is very helpful in the assessment of the quality of the data. The biggest concern relates to the procedure for a smooth transfer of the data into the database without losing any of the original flavor.

The CMC Database in MAPTIS:

The data will, after proper formatting, be stored into MAPTIS as a CMC database. MAPTIS can read in only ASCII datafiles and cannot store scanned images. Since the incoming data is usually in the form of graphs, tables, information text, a large part of the research has involved figuring out how best to transfer data most effectively.

Information Represented By The Data:

The database will provide information regarding Data Source, Type of the CMC, Coating (both external surfaces and fiber-matrix interfaces), and Test data accompanied by information such as test technique, loading condition, temperature, environment etc. Tests are likely to be as follows:

Physical Properties : Density, Porosity

Mechanical Properties : Tension, Compression, Fracture Toughness, Fatigue, Creep/Stress Rupture, Flexure, Torsion, Impact

Thermal Properties : Specific Heat, Diffusivity, Conductivity, Thermal Expansion, Emissivity

NDE : X-Ray, Ultrasonic, Acoustic, Computed Tomography, Pulsed Thermal Wave Imaging etc. providing information on density distribution, flaw detection, ultrasonic velocity in the material etc.

Ideally, search would be possible within the database with all of these as search criteria. An overview file and a help file containing information on how to search with any of these will be available. The format of the data will be the relevant set of numbers accompanied by the information relevant to the test.

System Development :

Software and Hardware required for data input :

MAPTIS, in its present form, cannot read in graphs or images. So the following actions had to be taken:

The Digitizer :

MAPTIS requires graphs and images to be turned into ASCII files. A time consuming search of the market led to the purchase of the SUMMASKETCH II PROFESSIONAL digitizer and the supporting software called "ROCKWORKS" which contains a digitizing software. This setup converts graphs into x-y files (also x-y-z files if necessary) which is then edited using the DOS EDITOR.

The Scanner:

An additional hardware and software need has arisen in connection with the transfer of the tables supplied by the data sources. It would be impractical to type the tables into a DOS text file manually, A scanner driven by an OCR (Optical Character Reader) software has been anticipated to be the solution. During the 1995 fellowship, information has been collected on this and the purchase will be made outside of the fellowship (under the contract). The HP 3C scanner supported by the CALERA WORDSCAN OCR software has been considered to be a good configuration so far.

Visits For Data Acquisition :

During the fellowship, a trip was taken to Oakridge National Laboratory (ORNL) in Tennessee. The visit comprised of :

1. Individual discussions with the CMC research personnel

2. Tour of the Mechanical and Thermal Properties test facilities and the lab in general.
3. Collection of ORNL reports, published and unpublished data generated using ORNL fabricated CMC's as well as other recent publications of data and concept related interest.

Southern Research Institute at Birmingham, Alabama was visited during the 1994 summer fellowship and the visit was similar in format. Other intended visits are to : DuPont, Williams International, University of Michigan, NASA/Lewis, Wright-Patterson AFB.

Literature : Samples :

The following publications are likely to provide valuable information toward the selection of data for the database.

1. High Temperature Behavior of Ceramic Composites ; edited by S. V. Nair and K. Jakus; Butterworth-Heinneman, 1995.
2. Ceramics and Ceramic Matrix Composites : Flight Vehicle Materials, Structures and Dynamics, Vol. 3, edited by S. R. Levine, A. K. Noor, S. L. Venneri, 1992.

Critical Issues In Testing CMC's[6]:

Specimen Design:

Test specimens may be : Straight sided or Dog bone, cylindrical or flat. DuPont used a blade subelement (a tensile coupon) with a gage section of elliptical shape, to simulate the leading edge of the rotor blade.

Finite Element Methods can be used in the design of specific test specimens to ensure that stresses are as a result of the manufacturing or fabrication process and cannot be corrected by machining or grinding due to other testing constraints.

At Oakridge Lab, they have acquired capacitance extensometers; however, the measurements are made only on one side of the specimen.

Control Variables : Temperature, Heating Rate, Environment, Load and Loading Rate.

Effort will be made to tag the data with this kind of information as far as possible.

Research Data : The literature search has made available data on some of the less studied properties and incorporating those into the database may be beneficial to facilitating interlaboratory comparison for the discerning of the testing reproducibility and limitations, material behavior and its variability for Ceramic Composites.

Example : Research Data on Interface Behavior :

The stress-strain behavior and damage tolerance of Ceramic Composites are significantly influenced by the strength of the fiber-matrix interface. This makes the characterization of the interfaces and their properties (co-efficient of friction, residual clamping stress, residual axial stress and debond stress) in continuous fiber reinforced ceramics an essential part of CMC characterization.

The forces between the fibers and the matrix in a CMC can be modified by introducing interphases (such as Nicalon fibers coated with carbon and SiC using polypropelene and a mixture of methyltrichlorosilane and H_2) that protect the fiber during processing. Fiber pushout tests with variation in coating thickness yields information on changes in fiber bonding and sliding characteristics [8]. The summary of the results of the tests reported in ref 8 shows the following :

1. The interfacial shear stress for a SiC(Nicalon)/Graphite/SiC CFCC decreases when the thickness of the fiber-matrix interphase layer increases from 0.03e-06m to 1.2e-06m.
2. The co-efficient of friction decreases with increasing coating thickness.

Similarly, the CMC database could contain information on environmental effects on crack growth in CMC's as crack velocity versus applied stress intensity measured in different environments[9].

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9. C. H. Henager, Jr., R. H. Jones : "Environmental Effects on Elevated Temperature Subcritical Crack Growth of SiC/SiC Composites", presented at the meeting on "Critical Issues in the Development of High Temperature Structural Materials", March 7 - March 12, 1993, Kailua-Kona, Hawaii.