IMF Newsletter

Industrial Materials for the Future

Winter 2001

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New mission plans for IMF

DOE's multiyear goals outline bold strategies for improved materials

By Charles Sorrell,
Department of Energy

The multiyear program plan outlines the mission, goals, and strategies of the Department of Energy (DOE), Office of Energy Efficiency and Renewable Energy, Office of Industrial Technologies (OIT) for conducting research and development activities in industrial materials.

OIT's Industries of the Future (IOF) process has shown that improved materials are a crosscutting need of many industries and one of the keys to cleaner and more energy-efficient and productive manufacturing.

The nine industries of the future on which OIT is focused are agriculture, aluminum, chemicals, forest products, glass, metal casting, mining, petroleum, and steel.

Supporting industries are forging, heat treating, welding, and carbon products.

The multiyear program plan outlines a new approach for integrating existing research and development projects into a unified Industrial Materials for the Future (IMF) program.

Deadline of 2004

The plan calls for integration of all of OIT's crosscutting efforts in advanced materials by 2004.

It addresses recent recommendations made by the National Research Council to sharpen the focus of materials research in

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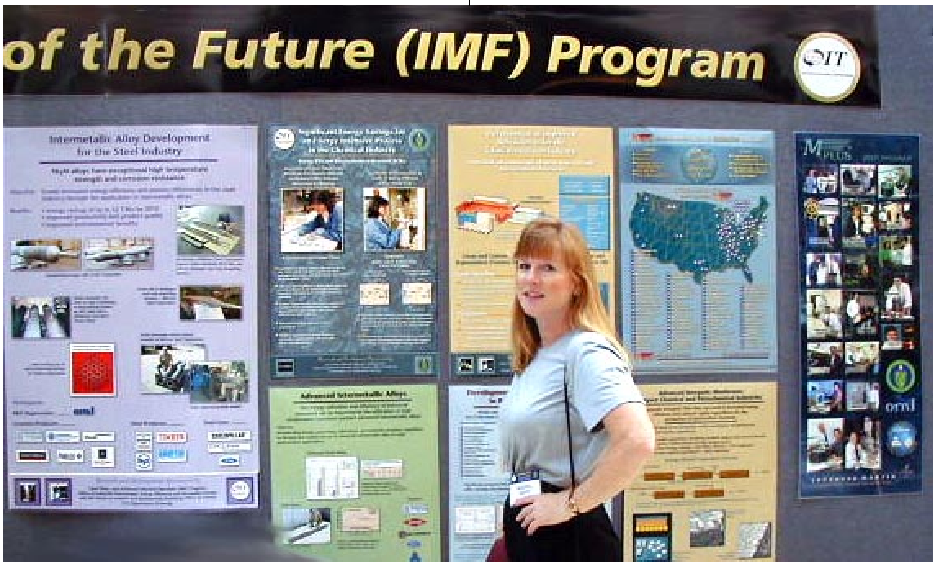

Merrill Smith, DOE Office of Distributed Resources, viewing the IMF display at the AIM/CFCC Conference earlier this year in Oak Ridge, Tennessee.

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OIT on industry-specific and crosscutting needs of the IOFs while maintaining efforts in core R&D areas.

OIT has $1.8 million in the fiscal year (FY) 2001 budget for cost-shared R&D.

All IOFs have certain characteristics, including:

- prepare a vision of the future,
- prepare one or more technology roadmaps,
- have a budget line in OIT,
- respond to competitive solicitations for cost-shared R&D in preparation of solicitations and in merit reviews, and
- have partnerships with national laboratories.

OIT currently has materials efforts in advanced industrial materials (AIM), continuous fiber ceramic composites (CFCC), and portions of advanced turbine systems, distributed generation, and combined heat and power.

Background

The first of these, AIM, is what remains of the energy conversion and utilization technologies program materials effort. In 1990 it became the materials program in Advanced Industrial Concepts. With the advent in 1994 of the IOF strategy, it became AIM, a national laboratory–based program with industrial partnerships.

Funded work has been in intermetallic alloys, membrane materials, uniform droplet metals processing, materials for kraft recovery boilers, refractories for glass, and metal/ceramic composites.

The budget has decreased over the past four years from $10.4 million to $6 million.

Six projects begun by AIM are now being funded by glass, chemicals, and steel.

The CFCC program, funded at $8 million for the past few years, has been a planned 10-year effort to solve critical technical problems for industrial implementation.

Scheduled to be completed in FY 2002, it has been conducted by industrial partners, with technical assistance from Oak Ridge National Laboratory.

Why the change?

Several factors have prompted changes in OIT’s Materials Development Program:

- Materials aspects of advanced turbines, distributed generation, and combined heat and power have been transferred to the Office of Power Technologies.
- The CFCC program is scheduled for completion in FY 2002.
- The AIM program has been noncompetitive.
- An integrated materials program is needed for political and other reasons.

The future

The IMF program calls for:

- Integration of OIT’s crosscutting materials efforts, beginning in FY 2000 and fully implemented by FY 2004.
- By FY 2004, all IMF projects will be selected by competitive selection.
- Solicitations will be for industrial applicants for industry-specific projects and national laboratory/university teams for base technology development.

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THE IMF NEWSLETTER

Industrial Materials for the Future

This newsletter is intended to facilitate communication among industrial, government, and university researchers involved in developing advanced materials.

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The plan's general IMF guidelines include the following:

- All projects will address materials needs identified in industrial technology roadmaps.
- Emphasis will not be on materials types but on industrial environments in which they operate.
- Priority will be given to technologies applicable to more than one industry.
- All proposals will be subject to technical merit reviews by industry and programmatic reviews by OIT.
- Funding will not be provided for one company to develop a product but to teams.

IMF's activity categories are to be

- Industry-specific R&D led by industrial partners including suppliers and users, as well as national laboratories and universities, as appropriate.
- Crosscutting R&D led by industrial partners to support OIT crosscutting programs. These currently include combustion, sensors, and controls.
- Core activities (technology base) led by national laboratories and universities to ensure continued development of new technologies.

The program will fund studies by independent organizations, workshops, program planning and analysis, program reviews, and advisory groups. It will also support the Materials Processing Laboratory User Center and other facilities to assist the IOFs with short-term materials problems.

Initial funding targets, which will be reviewed each year by OIT management, will be

- Industry-specific and crosscutting—50%
- Core activities—35%
- Directed activities—15%

Preparing the transition

Several things are being done to prepare for the transition to IMF:

- All existing projects are being assessed to determine time and funding required for successful completion.
- Competitive solicitation is being conducted with FY 2000 funds for $1.5 million, half for industry and half for national laboratories.
- The program plan is nearing completion and approval by OIT management.

The transition timetable:

- FY 2000—89% of funding to be used for completion of existing projects, 11% for competitive solicitations.
- FY 2001—60% for existing projects, 25% for mortgages and new solicitations, 15% for directed activities.
- FY 2002—35% for existing projects, 50% for mortgages and new solicitations, 15% for directed activities.
- FY 2003—10% for existing projects, 75% for mortgages and new solicitations, and 15% for directed activities.
- FY 2004—85% for mortgages and new solicitations, and 15% for directed activities.

Guidance/Evaluation

Guidance and evaluation for the program will be accomplished by the following:

A subset of OIT's overall board will oversee the IMF program.

Assessment of current projects will feed into a more comprehensive study by an independent group to determine priorities, criteria for project selection, completion, and/or termination.

An independent group will identify and prioritize technology needs from industrial technology road maps.

The IMF program will be managed by OIT in the Office of Energy Efficiency and Renewable Energy. Other organizations will be involved in the implementation and coordination of the planned R&D activities.

Implementation

Program implementation will be handled primarily by the DOE Operations Offices. Industrial performers will probably play the lead roles in carrying out industry-specific materials and crosscutting R&D, with the national laboratories and universities playing supporting roles as members of the teams.

National laboratories and universities, in cooperation with industrial partners, will play the lead roles in carrying out core activities.

Coordination with other offices in DOE, other federal agencies, and the various IOF teams is a critical program management function.

Several organizations are involved in development, including the United States Advanced Ceramics Association, Metal Powder Industry Federation, ASM International, Materials Technology Institute of the Chemicals Process Industries, the American Iron and Steel Institute, Aluminum Association, and others.

The program will coordinate with these organizations through their ties to the IOFs.∆

Supporting technologies provide the infrastructure

Development and utilization emphasized

By Rick Lowden,
Oak Ridge National Laboratory

Effort has been made to build an infrastructure of supporting technologies for development and utilization of advanced materials.

As part of this effort, an infrastructure for characterization and evaluation is essential, as are generic or more fundamental tasks that support development.

The structure of a supporting technologies effort consists of four basic components:

- Design of and with the materials,
- Characterization,
- Test methods, and
- Performance-related phenomena.

Constant change needed

It is important to integrate tasks and users or makers and to understand that the effort must evolve, to constantly change with the needs of makers and users.

A variety of materials and applications were to be investigated in the program, including

- Chemical vapor infiltration,
- Sol-gel infiltration,
- Directed metal oxidation,
- C fiber/silicon nitride,
- Nitrite-bonded silicon carbide, and
- Polymer impregnation and pyrolysis.

The supporting technologies task consists of the more basic or generic supporting elements of composite design, materials characterization, test method development, and performance-related phenomena. This generic research will provide the scientific foundation for successful development of new materials.

Composite design

Within the area of composite design, work included both design of composites and design with composites. In this area, work was conducted by:

- Micromechanics—C.H. Hsueh, Oak Ridge National Laboratory (ORNL);
- Macromechanics (class 2)—John Kibler, Materials Science Corp.;
- Fiber architectures—Thomas Hahn, Pennsylvania State University;
- Failure modeling—Claudia Ostertag and Linda Braun, NIST; and
- Composite Thermal Properties (Scanning Thermal Conductivity Microscope)—Ralph Dinwiddie, ORNL.

Materials characterization

In the area of materials characterization, which has provided detailed information about constituents and interactions, work was conducted by

- Characterization (electron microscopy)—Karren More, ORNL;
- Interface studies (testing and fiber coatings)—Edgar Lara-Curzio, Karren More, Matt Ferber, and Rick Lowden, ORNL;

>

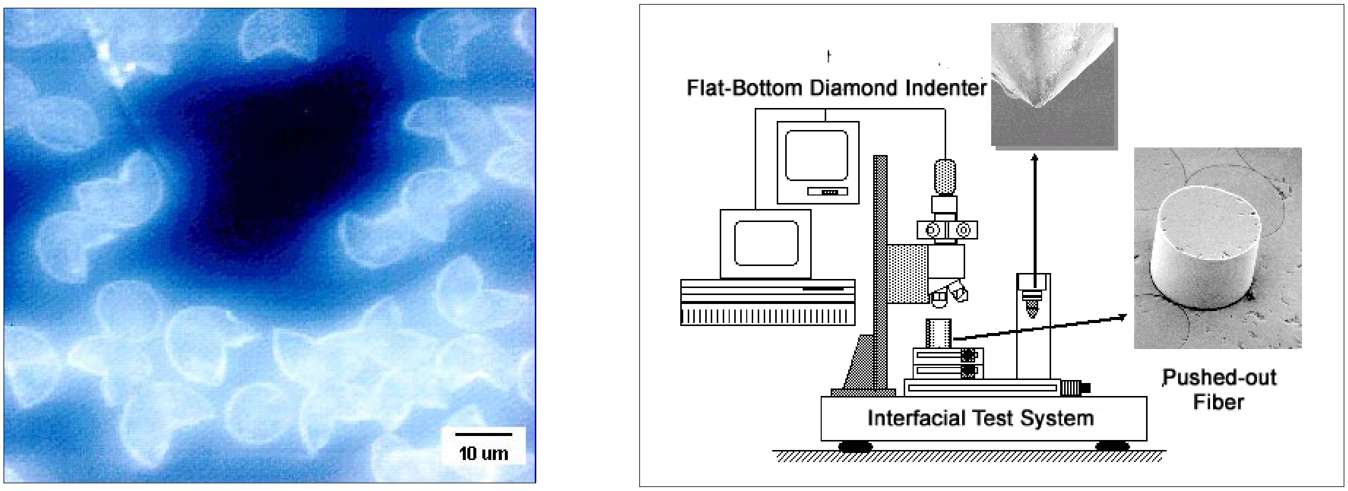
Sol-gel infiltration

Fiber/silicon nitride

Nitride-bonded silicon carbide

Polymer impregnation and pyrolysis

A variety of materials and applications are to be investigated in the IMF Supporting Technologies Program. Examples of several are shown here.



Specialized test methods and equipment have been developed to investigate in-situ properties of constituents. They include, at left, a thermal conductivity microprobe, and at right, an interfacial test system.

- Fiber coatings—John Hellman, Pennsylvania State University;
- Oxide fiber coatings and matrices [metal-oxide chemical vapor infiltration (MOCVI)]—Sankar Sambasivan, Northwestern University (See page 8);
- Oxide coatings (inorganic precursor CVD)—Vinod Sarin, Boston University; and
- Environmental barrier coatings—Allen Haynes, ORNL.

Standardized test methods

In the area of standardized test methods, work was conducted by

- Thermomechanical testing (standardization)—Michael Jenkins, University of Washington, and Edgar Lara-Curzio, ORNL;
- Geometry-specific testing (develop techniques)—Edgar Lara-Curzio, ORNL; and
- Nondestructive characterization (computed tomography, infrared, and air-coupled ultrasound—Bill Ellingson, Argonne National Laboratory (see this issue of the IMF Newsletter, page 10).

Performance phenomena

In the area of performance-related phenomena as they address applications-related issues, work was conducted by

- Performance simulation (MRLife)—Ken Reifsnider and Mike Pastor, Virginia Polytechnic Institute;
- Environmental effects (trends and mechanisms)—Pete Tortorelli, ORNL;
- Mechanical reliability (performance envelopes)—H.T. Lin and Paul Becher, ORNL;
- Thermal shock (techniques and effects)—Raj Singh, University of Cincinnati; and
- Data Base—Edgar Lara-Curzio, ORNL, Michael Jenkins, University of Washington, Steve Gonczy, Gateway Materials.

Products

The products of supporting technologies are

- Computer models and codes,
- Specialized equipment and facilities,
- Advanced techniques and procedures,
- Data and information,
- Technical reports and publications,
- Expertise and experience, and
- Working relationships (teaming).

Preventing fragmentation

Since its beginning the direction of the supporting technologies effort was altered several times, but the original framework was maintained to prevent fragmentation, dilution of effort, and total chaos.

This framework consisted of

- Surveys of industrial needs and requirements,
- Steering committee,
- Working group meetings,
- Program management (DOE and ORNL),
- Informal communication.

Relationships complicated

One of the things learned during this effort has been that establishing relationships between materials manufacturers and national labs can be complicated.

This is because the goals and objectives for industry, national labs, and academia are different.

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It is also because not all materials and processes are at the same level of maturity.

Another complicating factor was that many materials continue to evolve. At times there are few "standard" compositions.

In addition, to maintain a competitive advantage, industry must keep secrets, which creates additional difficulties.

> *Another complicating factor was that many materials continue to evolve. At times there are few "standard" compositions*

Summary
To summarize our experience:

- Supporting technologies developed a network of facilities and personnel to support the manufacturing and utilization of advanced materials (CFCCs).
- The task has been well integrated, with significant cooperation between participants, and others, to make the use of advanced materials in industrial applications a reality.
- Supporting technologies evolved in response to the needs of industry. As the program progressed, efforts became less basic or generic and more "user" oriented.
- Supporting technologies have many "products" available. Techniques for the characterization and evaluation of advanced materials have been developed and optimized.
- Experience has yielded expertise.
- Teaming has been and continues to be an important part of the program.Δ

Membranes for gas separations

Energy efficiency and productivity boosts

*By David Devlin,
Los Alamos National Laboratory*

Separations are among the most energy-intensive and costly aspects of many industrial processes. They can account for 40 to 70% of both capital and operating costs and are critical to many industries such as chemical processing, petroleum refining, environmental/pollution abatement, agricultural, food processing, desalination, and materials processing.

> *Conventional processes such as distillation can require up to 50% more energy than is thermodynamically required*

Conventional processes such as distillation can require up to 50% more energy than is thermodynamically required and are thus not efficient. Membranes have the potential for greatly improving energy efficiency.

Within the advanced industrial materials (AIM) program at Los Alamos National Laboratory, we are pursuing two industrially important applications. The first of these is a high-temperature membrane aimed primarily at the removal of CO_2 from shifted syngas streams prior to use in gas turbines or high-temperature fuel cells (see Fig. 1).

Impact
This could have a significant impact on energy efficiency, waste reduction and productivity in power plants, the chemical processing industry and co-generation facilities. As a further benefit, should future regulations motivate or require carbon dioxide sequestration, a viable carbon dioxide separation technique will be essential.

The basis of the membrane system is the use of a reactive molten salt immobilized in a porous structure. The principle reaction of interest is:

$$M_xO(s,l) + CO_2(g) = M_xCO_3(l)$$

Where M represents metals from group I or II of the periodic table. The separation mechanism is based on the reversibility of this reaction and its dependence on the partial pressure of CO_2. In this system CO_2 is consumed on the upstream side to form the carbonate. It is then transported downstream through the molten salt membrane as carbonate ions, CO_3^{2-}, where the CO_2 partial pressure is sufficiently low to favor decomposition and CO_2 is released (Fig. 2).

Permeability of a membrane is the product of the diffusivity and the solubility of a given component. In these molten systems the very high reactive solubility combined with the fast liquid phase diffusivity promises to result in a membrane with good permeability. The selectivity of CO_2 over hydrogen results from relatively low physical solubility of hydrogen in the molten salt.

Candidate material
As a candidate material we are working with Li_2CO_3 and its low melting eutectics. Also significant is the fact that Li_2CO_3 and Li_2O

form a eutectic melt providing the O^{2-} in the melt for reaction with CO_2. Molten carbonates are used extensively for the matrix material in molten carbonate fuel cell systems. These materials are based on a tape cast porous lithium aluminate matrix containing the active carbonate salt. This approach has advantages in ease of fabrication, scalability, and a wet seal method for sealing of the membrane to the module.

Our second project is a joint effort with BP Amoco to develop a membrane for the separation of light gases from olefins. Ethylene and propylene are two of the largest commodity chemicals in the United States and are major building blocks for the petrochemicals industry. These olefins are separated currently by cryogenic distillation, which demands extremely low temperatures and high pressures.

Over 75 billion pounds of ethylene and propylene are distilled annually in the United States at an estimated energy requirement of 400 trillion BTUs. New separation processes are therefore needed to continually reduce energy consumption and remain competitive.

BP Amoco

BP Amoco has been a leader in incorporating new separation technology into its olefins facilities and has been aggressively pursuing noncryogenic alternatives to light gas separations.

The largest potential area for energy reduction is the cryogenic isolation of the product hydrocarbons from the reaction by-products, methane and hydrogen.

This separation requires temperatures as low as -150 F and pressures exceeding 450 psig. This project focuses on developing a membrane process to separate olefinic mixtures from light gas by-products at higher temperatures and lower pressures than are currently required in the demethanizer feed chilling system.

Eliminates energy needs

Additionally, this technology will eliminate the reboiling energy requirements for up to four distillation towers, which will result in substantial energy savings. The basis of this separation method is the well-known effect of capillary condensation.

The blocking of pores by the condensate provides a means of separating condensable from noncondensable gases. Heavier gases are preferentially adsorbed and condensed in the pores leaving the light gases behind (Fig. 3).

Transport of the condensed vapor through the membrane is primarily by capillary flow. Capillary pressures can result in high flow with very low actual

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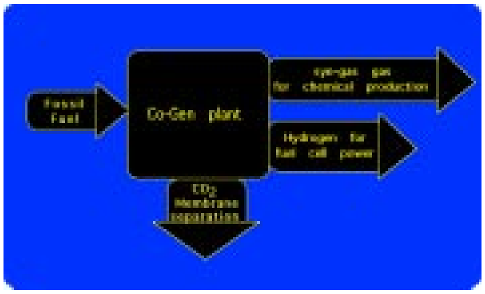


Fig. 1. Co-generation plant for prodution of hydrogen as fuel or syn-gas for chemical production.

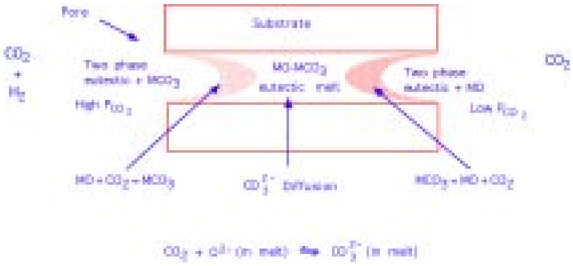


Fig. 2. Illustration of an immobilized molten carbonate salt functioning as carbon dioxide selective membrane.

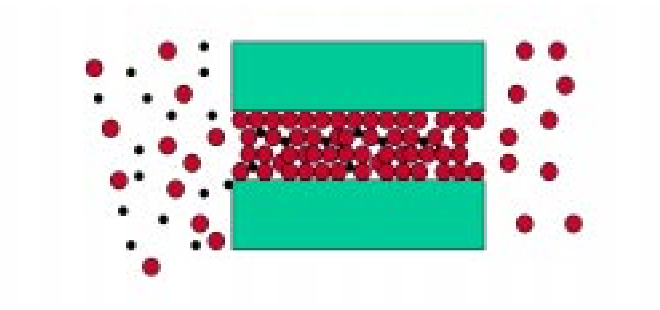


Fig. 3. An illustration c capillary condensatic membrane. Condens of the heavier hydroc blocks the lighters fro passing through the p

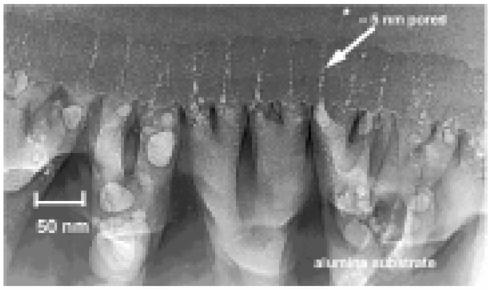


Fig. 4. Transmission electron micrograph of a magnetron sputtered carbon film with columnar grains and 5-nm pores as a result of "self-shadowing."

Fabric coating technologies

Process technologies for oxide CFCCs

By Sankar Sambasivan and Kimberly A. Steiner, Northwestern University

Under CFCC Task 2 funding, the Advanced Coating Technology Group (ACTG) of Northwestern University is primarily engaged in developing process technologies for the deposition of interface and protective fiber coatings for oxide CFCCs.

Based on the collective work of many researchers in the oxide CFCC community over the past 5 years, it is evident that the technical and scale-up challenges (interface coatings and matrix fabrication) for producing oxide CFCCs are quite different from the development of SiC-based CFCCs.

New interface concepts/materials have emerged for tailoring fiber-matrix properties. Specifically, the discovery of monazite ($LaPO_4$) and scheelite ($CaWO_4$) as "weakly-bonded" interface coatings has brought refreshing perspectives to interface design and engineering. Other interface compositions and concepts are also being developed.

Complex compositions

Most of the work reveals that complex compositions (with two or more cations) may be required to achieve the desired interface properties. Unlike the chemical vapor infiltration (CVI)-produced C/BN coatings for SiC-based CFCCs, solution-based dip-coating methods appear to be more suitable to ensure uniform "local" cationic stoichiometry.

While the solution-based methods offer low-cost advantages, obtaining coatings with a high degree of uniformity and good surface coverage without deleteriously affecting fiber strength is a significant challenge. Recent work by Hay and coworkersat the U.S. Air Force Research laboratory, however, demonstrated good strength retention characteristics for monazite coatings on Nextel™ grade oxide fibers.

From a technology perspective, availability of oxide-coated fabrics or preforms for composite fabrication will be of great benefit to the industry. Most of the current SiC-SiC composites are fabricated using C/BN coated preforms. It was indeed possible to infiltrate fabrics and woven preforms using CVI in the case of C or BN coatings, where gaseous species could easily diffuse into intratow spaces and condense uniformly on the substrate surface.

Key question

The key question is whether we can duplicate the success of C/BN to oxide coatings using a low-cost and relatively simple solution-based dipping process. The challenge is in efficiently wetting and infiltrating the tightly woven fiber tows.

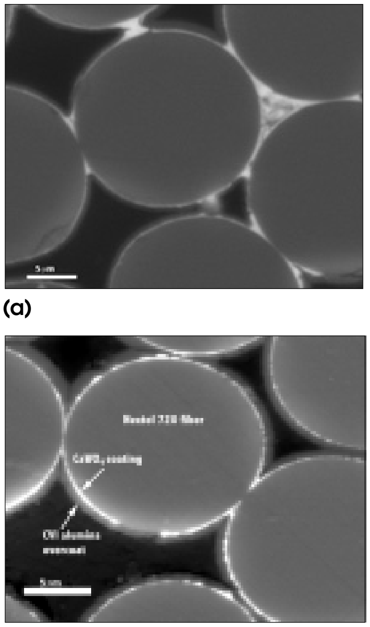
Hay's immiscible layer approach has worked quite well for coating fiber tows and, in principle, can be extended to coating fabrics and preforms. In brief, the technique involves the use of an immiscible layer placed on top of the precursor solution which assists in draining excess precursor after the fiber tow is passed through the solution such that fiber bridging is minimized.

ACTG's CFCC effort is focused on developing alcohol-based precursor solutions for both scheelite and monazite, and it has optimized the immiscible layer method (originally developed by Randy Hay) to obtain fairly uniform coatings on woven Nextel™ fabrics (Fig. 1).

First time

To our knowledge, this is the first time direct application of oxide interface coatings on woven

Images of interface coatings used in production of new oxide CFCCs



(a)

(b)

Fig. 1. Interface coatings on woven Nextel 720 fabrics, covered by a MOCVI alumina coating: (a) monazite (b) scheelite.

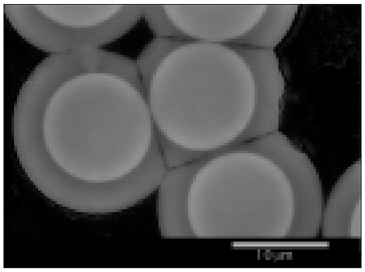


Fig. 2. MOCVI of alumina on woven Nextel™ 720 fabric.

fabrics has been demonstrated using a solution-based process.

In addition, ACTG has also developed a suitable metal-organic chemical vapor infiltration (MOCVI) process for deposition of alumina (Fig. 2) and other alumina-based compositions (Fig. 3) as protective coatings directly on monazite- or scheelite-coated woven fabrics. Along with a relatively high deposition rate (up to over a micron per minute), excellent infiltration efficiency is achieved by this process, with coating thickness ranging from 0.5-1 µm across the thickness of the fabric.

A 6-layer preform of Nextel 610 fabric was uniformly coated with this process (Fig. 4). The alumina coating also serves as protective aid during metallographic preparation such that the true distribution of interface coating can be observed.

Aggressive chemistries

There is a need to protect fibers and interface coatings comprising complex chemistries during matrix processing. To develop dense oxide matrices using solution-based methods, aggressive "chemistries" may have to be used while preserving low cost of fabrication. Thus, a matrix coating to protect the interface coating and fiber may be a critical intermediate step in the fabrication of CFCCs.

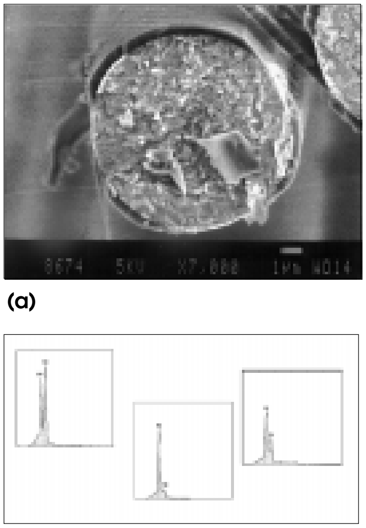
The protective matrix coating also offers a suitable nonwetting interface with the fiber coating as shown in Fig. 5 where debonding appears to occur at both fiber-coating and coating-matrix interfaces. The as-deposited alumina coatings produced by the MOCVI process is amorphous in nature and is converted to α-alumina upon annealing to 1200°C. More recently, ACTG is developing doped-alumina coatings that allow conversion to alpha alumina at lower temperatures.

Recent efforts

Recent ACTG efforts include fabrication of alumina-based oxide minicomposites by MOCVI. Figure 6 shows a cross-sectional SEM image of minicomposite (with scheelite coated fibers) fabricated by MOCVI. Testing of minicomposites (by Dr. Edgar Lara-Curzio, ORNL) revealed extensive pull-out of the fibers with debonding occurring primarily at fiber-coating interfaces.

While further improvement in fabrication of minicomposites is essential to use reliably this to screen interface coatings, preliminary results look promising.Δ

More images of interface coatings



(a)

(b)

Fig. 3. (a) Aluminosilicate coating on a Fiber, and (b) EDS profiles from 3 alumino-silicate coatings demonstrating the compositional range of the process.

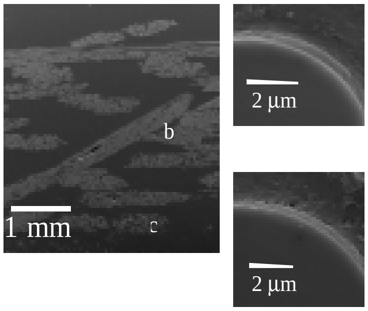


Fig. 4. SEM of alumina coated 6 layer Nextel 610 preform. (a) The preform (b) fiber in the center of the preform (c) fiber on the outer edge of the preform.

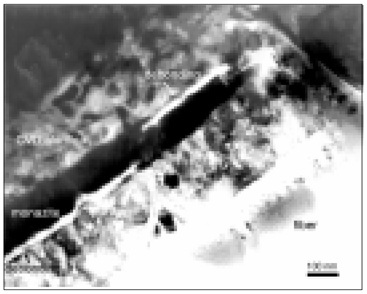
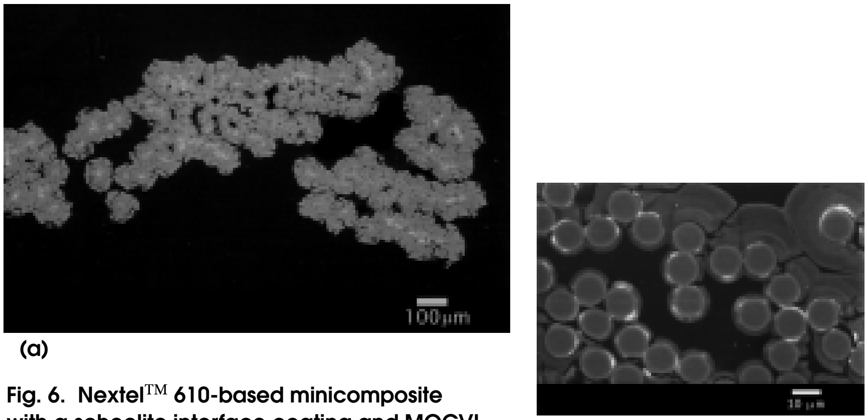


Fig. 5. TEM micrograph of a monazite and alumina-coated Nextel™ 720 fabric after annealing to 1200°C, 1 hour.



(a)

Fig. 6. Nextel™ 610-based minicomposite with a scheelite interface coating and MOCVI alumina matrix.

(b)

NDC work at Argonne lab

New techniques using filmless nondestructive X-ray imaging method

By Bill Ellingson,
Argonne National Laboratory

X-ray imaging is a common, highly regarded diagnostic method for medical as well as industrial applications.

Usually the X-ray images we are familiar with are those which use film. However, X-ray film must be processed in a series of chemical baths. This chemical process is time consuming, and disposal of processing chemicals becomes an environmental concern.

As part of the nondestructive characterization (NDC) development work at Argonne National Laboratory (ANL), research has included study of filmless X-ray imaging (referred to as digital radiography).

Filmless X-ray imaging

Filmless digital radiography X-ray imaging uses solid state digital X-ray detectors coupled to computers with various image display software packages. Solid state digital radiographic image methods provide direct viewable images and allow quick sharing of image data through internet transmittal.

Development of solid state digital radiographic X-ray imaging detectors is an on-going research effort. There are basically two configurations for using solid state X-ray imaging detector systems. These are shown in Fig. 1 and Fig. 2. Figure 1 shows what is referred to as a "one-dimensional" solid state X-ray detector array shown in an X-ray computed tomographic (XCT) imaging system.

Such an X-ray imaging system is often referred to as a "CAT" scanner ,and ANL has applied this X-ray imaging modality to several CFCC components.

Two dimensions

In the case of a one-dimensional detector, the X-ray detector only detects X-rays transmitted in one plane—the horizontal plane.

However, by translating the object up and down in front of the detector or, conversely, moving the X-ray source and the detector up and down in synchronization, one can generate a projection 2-dimensional (2-D) digital radiographic image similar to that which might be seen on a film.

However, the one-dimensional detector can also be used in an X-ray imaging system in the CAT scan mode of image acquisition. In a CAT scan imaging mode, one can generate cross-sectional images of an object and "see" interior details just as if the object had been destructively sectioned.

This is a very important aspect in the use of solid state X-ray detectors, that is, they can be used to obtain digital radiographic projection images similar to film type images or, if coupled with the proper hardware and software, they can be used as a detector in CAT scan image acquisitions.

Recent work on solid state X-ray imaging detectors has focused on 2-D or area detectors. These new area X-ray detectors are being developed from amorphous silicon (a-Si) or amorphous selenium (a-Se).

Area detectors

The advantages of using area detectors for X-ray imaging is obvious. In order to generate a projection or 2-D image, just like a film image, one does not have to translate the object or the X-ray source and detector.

Figure 2 shows a schematic of such an X-ray imaging set.

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Fig. 1. Schematic Diagram of a one-dimensional solid state X-ray imaging detector in a typical X-ray imaging system. Note that both digital radiographs and CAT scan images could be obtained in this system.

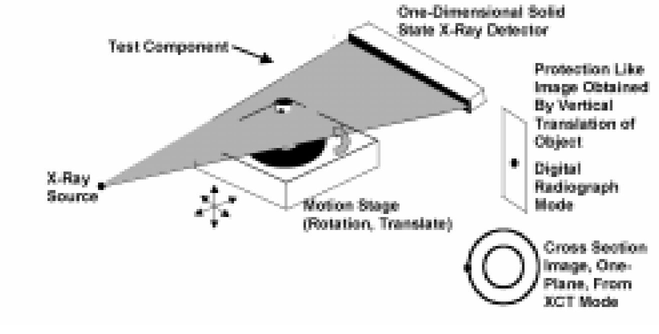
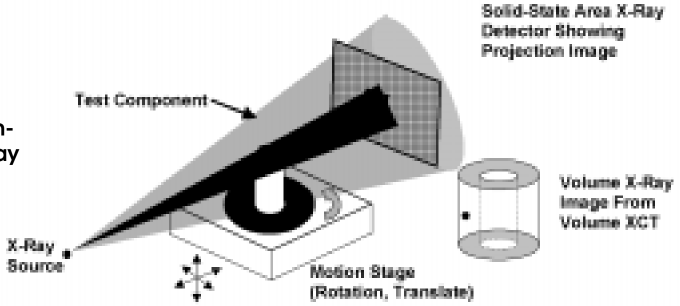


Fig. 2. Schematic diagram of a two-dimensional solid state X-ray imaging detector in a conventional projection X-ray imaging set up.



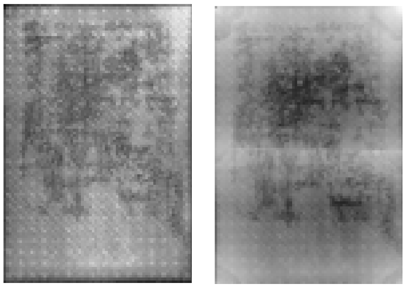


Fig. 3. Comparison of through-transmission X-ray images of melt-infiltrated SiC/SiC 8-ply layup, 3-mm-thick CFCC plate. At left is a film radiograph. At right is a digital radiograph.

Fig. 4. Photograph of 30-in. diam/13-in diam MI SiC/SiC combustor liner pair used in an advanced turbine.

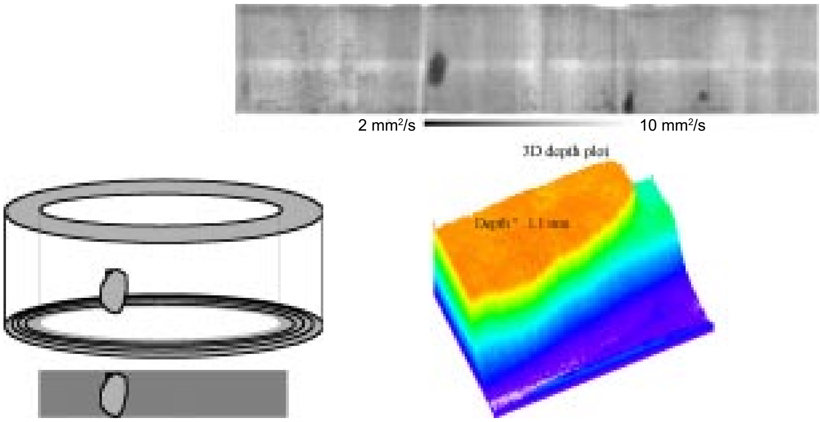


Fig. 5. Schematic diagram and predicted depth below surface of delamination of CFCC liner using thermal imaging methods.

Figure 3 shows a comparison between projection, through-transmission X-ray images taken of a 6- by 9-in. (15- by 22.5-cm) melt infiltrated (MI) SiC/SiC 8-ply flat plate. The plate was 0.125 in. (3 mm) thick, and the images were obtained using conventional film and a solid state a-Si 8- by 8-in. area detector.

The a-Si detector used in this case had 512 × 512 elements. Each detector element was 400 × 400 µm in size. One can assess the relative image quality between the images by comparing size of detected features and presence or absence of features.

Differences

There are several differences between a-Si and a-Se detectors. First, a-Se detectors are direct converters of X-ray photons. That is they do not require an intermediate converter.

Amorphous silicon detectors are indirect systems and require the use of an X-ray to light the converter screen in front of the detector. Second is the speed at which images can be acquired.

A-Si area detectors allow faster read out times. Usually the read out time for a-Si is < 2 s per frame, whereas for a-Se it is 30–60 s per frame.

The size of the individual detector elements (pixels) in either the a-Se or the a-Si can range in size from 135 to 400 µm depending upon which specific panel one selects.

The overall sizes of panels available range from 4 by 4 in. (10 by 10 cm) up to 17 by 17 in (42.5 by 42.5 cm). The former has 100 µm elements (a 1000 by 1000 array) and the latter has 400-µm size elements or a 1250 by 1250 array.

Applications

ANL has successfully developed and employed digital X-ray imaging technology to several CFCC components and test specimens for DOE's Industries of the Future (IOF) and the advanced turbine programs.

It is important to realize that when using X-ray imaging in the simplest, lowest-cost mode, that is, in the through-transmission digital radiographic imaging mode, there is little sensitivity to delaminations if those delaminations are in a plane perpendicular to the path of the X-rays.

However, if the digital X-ray imaging mode is the CAT-scan mode, then there is essentially no better method to detect or determine internal structurevariations.

By coupling various aspects of digital X-ray imaging technology with other NDC methods, such as thermal imaging (*CFCC News*, No. 10, 1998, pp. 1–5) or acoustic methods (*CFCC News*, No. 11, 1999, pp. 10–13) very powerful arguments can be made about the size and through-thickness locations of density variations, delaminations, voids, size of impact damage zones or other defects or features of interest.

Predicting component life

Coupling of such data to life time prediciton methods could present designers with the ability to predict component life. The following applications sections

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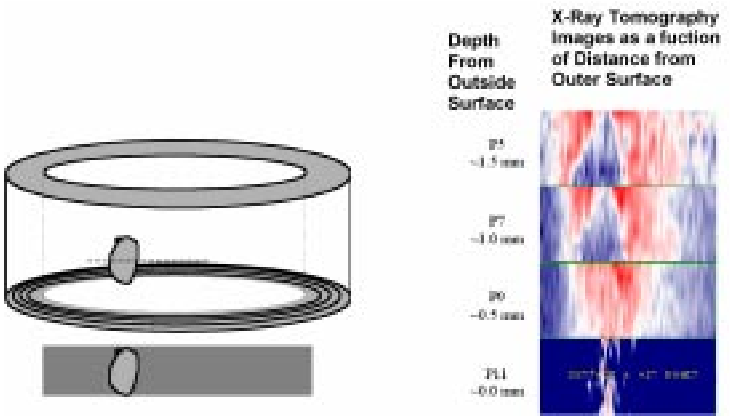

Fig. 6. Schematic diagram and resulting digital X-ray image data demonstrating the ability to determine the depth below the surface of delamination shown in Fig. 5.

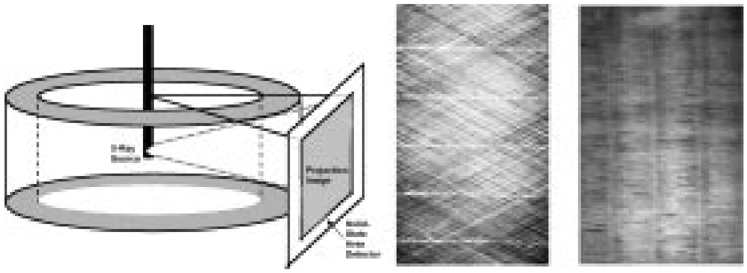

Fig. 7. Through-transmission single wall X-ray images of 2 different fiber architecture CFCC cylinders. The rectangular image on the left is oxide/oxide with filament winding. The rectangular image on the right is SiC/SiC with 3-D weave.

Recently, large diameter CFCC liners for industrial gas turbines have been developed (see Fig. 4).

During one recent set of NDC tests, it was discovered that a likely delamination had occurred somewhere inside the 3– to 4-mm-thick wall.

Knowledge of the location of the delamination within the wall thickness became critical as it would effect thermal stresses.

Uncertainty in results

ANL has been developing one-sided thermal imaging along with necessary software to predict the depth below the surface of delaminations. However, because this is a developing technique, there is currently an uncertainty in the results.

Figure 5 shows the thermal image and the predicted depth below the surface of 1.1 mm for the delamination detected on one 13-in. liner.

By using digital X-ray imaging, this time using the CAT scan mode with the images stacked and "peeled" (see Fig. 6), one can determine the depth below the surface where the delamination starts and stops.

Figure 6 shows a schematic diagram and resulting data which verifies that the depth below the surface of the delamination was about 1.25 mm. Thus the correlation is very good between the thermal data and the X-ray XCT data.

Detecting fiber orientation

In CFCC materials, it is also important to know fiber layout and general fiber orientation.

Using through-transmission X-ray imaging with the X-ray source (e.g., a rod anode) on the inside of a cylinder, and with digital detectors on the outside, one can easily determine differences in fiber architecture as shown in Fig. 7.

Figure 7 shows single-wall X-ray images of two significantly different fiber architecture 8-in.-diam cylinders.

Figure 7a is one single-wall X-ray image showing the filament winding pattern in an oxide/oxide 3-mm-thick wall of an 8-in.-diam cylinder, and Fig. 7b is one single-wall X-ray image showing the fiber architecture of a 3-D weave pattern in an 8-in.-diam, 3-mm-thick wall SiC/SiC cylinder.

By using digital detectors, and a rotation stage for the CFCC component, one can obtain image data similar to these but in near real time during object rotation.

Density uniformity

In many CFCC materials, it is useful to know the general as-infiltrated density uniformity. While thermal imaging methods may provide this information indirectly, through-transmission X-ray imaging provides this directly.

This can be shown by looking at Fig. 3. The lack of uniform gray tone across the images is the result of nonuniformity of density.

Exactly how nonuniform the density is can be estimated through use of a calibration method such as a simple step wedge. Inclusion of a step wedge or a set of samples with known

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density differences allows the image to be calibrated for gray tone versus known density.

Conclusions
Digital, filmless, X-ray imaging technology using solid state X-ray detectors provides many advantages over conventional film radiography for application to CFCC components.

For example:

1. The image data generated can be immediately transferred over the internet to those in need of the data.
2. The data are easily archived with little, if any, data deterioration.
3. Digital X-ray images can be used with new powerful digital image processing methods to extract and quantify data.
4. New solid state area detectors such as a-Si and a-Se provide large area images similar to film but without the need for potentially environmentally hazardous chemicals.
5. Digital X-ray imaging has been demonstrated to provide information for CFCC components relative to fiber architecture, delamination size and location, and general overall as-infiltrated density.
6. Digital radiography can be cost effective but does have certain limitations. As long as these limitations are recognized, then as an NDC method, digital radiography is a very viable NDC method.
7. When digital detectors are used in a X-ray imaging system in a CAT scan mode, cross-sectional image data provided are very quantifiable and provide through-wall data not obtainable by any other current nondestructive technology.∆

American Ceramic Society names Krishan Luthra Fellow


Krishan Luthra

Krishan L. Luthra, manager of the Thermal Structural Ceramics Program, has been named a Fellow of the American Ceramic Society (ACerS).

The prestigious designation—only 7 percent of the society's membership are Fellows —recognizes an esteemed leader in the ceramics field.

Fellows are elected by the ACerS Board of Trustees. The decision is based on their outstanding contributions to the ceramic arts or sciences, broad and productive scholarship in ceramic science and technology, conspicuous achievement in the industry of ceramics, or outstanding service to the Society.

Krishan joined General Electric's (GE's) Corporate R&D in 1976 as a materials scientist. In 1989 he began managing activities in ceramic composites, thermal barrier coatings, high- temperature thermochemistry, and oxidation corrosion.

Prior to joining GE he earned his bachelor's degree in metallurgical engineering from Malaviya Regional Engineering College, India; his master's degree in metallurgical engineering from the Indian Institute of Technology, India; and his Ph.D. in metallurgy and materials science from the University of Pennsylvania.

He has authored more than 70 publications and received 15 patents.∆

Membranes for gas separations

—Continued from page 7

pressure drop across the membrane. The Kelvin effect, which is the reduction of vapor pressure in a small pore, leads to reduced pressures or elevated temperatures over which the separation can be accomplished.

Columnar grains
Thin film deposition by evaporation and sputter techniques can result in microstructures consisting of columnar grains separated by porous regions.

The development of porous structures is dependent on a number of deposition parameters, but the porous structures are generally observed for conditions where the surface mobility of depositing atoms is limited.

The effect is a consequence of a process known as "self shadowing." We are fabricating and testing sputter deposited carbon films with mesoporous structures for this application (Fig. 4).∆

News notes

Industrial Materials for the Future and related notices

NEW TEXTRON TASK LEADER

Raymond Suplinskas has been named task leader of the Textron Immersion Tube Project. He replaces Gary DiBona, who has taken a position with another firm. He can be reached at rsuplins@systems.textron.com

LIN NAMED SECRETARY OF ACS

H. T. Lin has been elected secretary of the Engineering Ceramics Division of the American Ceramic Society for 2000–2001. Lin has for 11 years been a senior research staff member in the Structural Ceramics Group, Metals and Ceramics Division, at Oak Ridge National Laboratory. He works in the area of high-temperature mechanical properties and creep behavior of structural ceramics, ceramic composites, and intermetallics.

LARA-CURZIO AUTHORS CHAPTER FOR ENCYCLOPEDIA

Edgar Lara-Curzio this year authored a chapter for Elsevier's 2000 Comprehensive Composite Materials Encyclopedia. "Properties of CVI-SiC Matrix Composites" reviews experimental results and models of the physical, thermal, and mechanical properties of fiber-reinforced CVI-SiC matrix composites. Experimental results are included for elastic constants, tensile and shear strength in various material planes, interfacial properties, fracture resistance, performance-related behavior, and thermal and physical properties.

OIT MATERIALS PROGRAMS BECOME IMF

(See more details about this in the article on page one of this newsletter.) The Office of Industrial Technologies is integrating two veteran programs— Continuous Fiber Ceramic Composites (CFCC) and Advanced Industrial Materials (AIM)—into its new Industrial Materials of the Future (IMF) program. The new program will operate in the customer-driven manner typical of OIT's other activities. IMF will conduct a nationwide effort to research, design, develop, engineer and test new and improved materials, as well as discover more profitable uses of existing materials for Industries of the Future.

The new IMF program will fund industry-specific activities, crosscutting activities, and core activities. IMF will also support studies by independent organizations, workshops, review meetings, program planning, analysis and evaluations. As for funding, the IMF initial target is 50% for industry-specific and crosscutting technologies, 35% for core activities, and 15% for directed activities. Projects currently funded by CFCC and AIM programs will experience a transitional period through the year 2004.

In fiscal year 2000, a comprehensive assessment of all existing materials-related projects in OIT will determine time and funding required to conclude them. This will be followed by additional studies by an independent organization to identify and prioritize materials' needs from the technology roadmaps in order to determine future directions and priorities for the IMF program. (From *OIT Times*)

Calendar

AMERICAN CERAMIC SOCIETY 103rd ANNUAL MEETING
April 22–25, 2001
Indianapolis, Indiana
Indiana Convention Center & RCA Dome
This year's symposia and division programming will cover basic science and processing of ceramics and glasses, whitewares and materials, engineering and electronic ceramics, refractories and cements, glass and optical materials, and art ceramics. Of the 16 symposia, 12 will focus on device manufacturing and information technology; energy manipulation and environmental issues; and chemical, mechanical, and biomedical applications.
contact: http://www.ceramics.org/meetings/am2001/

23RD ANNUAL INDUSTRIAL ENERGY TECHNOLOGY CONFERENCE
MAY 1–4, 2001 — Houston, Texas
JW Marriott Hotel
The conference provides answers to energy and related environmental concerns affecting industrial facilities. this year the conference includes an energy managers' workshop, steam systems evaluation workshop, and a heat exchanger design workshop.
contact: Lana Tolleson, program coordinator; phone: (979) 847-8950;
fax (979) 862-8687; email: ltolles@esl.tamu.edu.

INTERNATIONAL CONFERENCE ON COMPOSITES ENGINEERING
August 5–11, 2001
Tenerife, Spain
Sheraton Mencey Hotel
The 8th ICCE, which will be held on one of Europe's most beautiful islands, will focus on (1) bridging the gap between materials science, mechanics, and processing of composites; (2) bridging gaps in interdisciplinary research between aerospace technology, biomaterials, chemistry, electronics, fluid mechanics, infrastructures, powder metallurgy, sensors/actuators; and (3) leveraging of research resources and encouraging joint research.
contact: http://www.uno.edu/~engr/composites.html

IMF Newsletter
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Oak Ridge, TN 37830