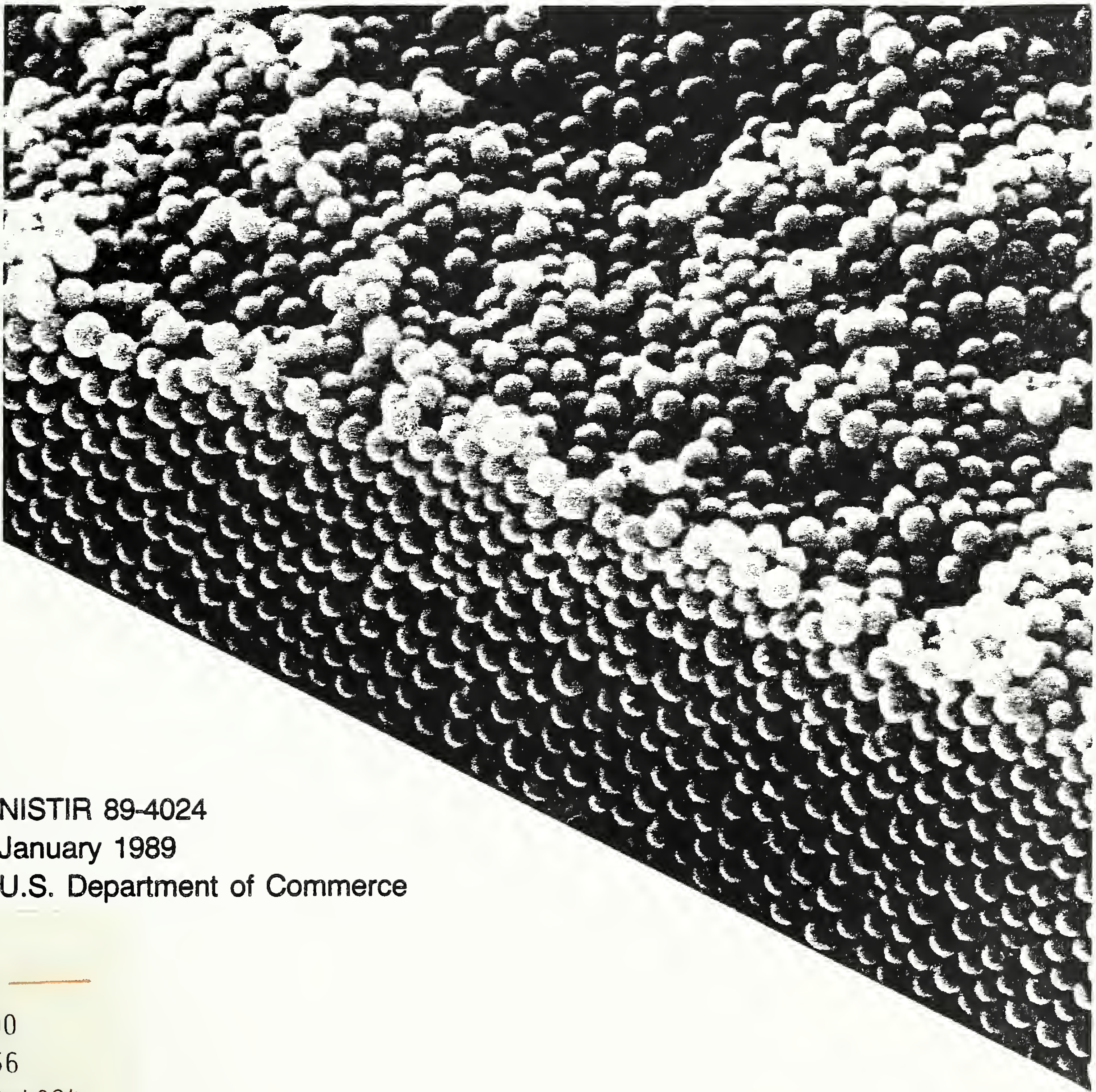
Intelligent Processing of Materials

Report of an Industrial Workshop Conducted by the National Institute of Standards and Technology



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Intelligent Processing of Materials

Report of an Industrial Workshop Conducted by
the National Institute of Standards and Technology

Gaithersburg, Maryland
August 31-September 1, 1988

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FOREWORD

Intelligent processing of materials has been established as a major new program area in the Institute for Materials Science and Engineering, National Institute of Standards and Technology (NIST, formerly the National Bureau of Standards). The goal of the program is to develop some of the generic scientific and technological bases for intelligent processing and, by means of selected pilot or demonstration projects, to encourage American industry to pursue and to adopt this powerful new approach to materials processing.

In developing this new program, NIST cooperated in organizing two national workshops in 1985-86 to help identify the principal industrial needs in this field of technology and to solicit guidance for program planning activities. The first of these two workshops [1] was concerned with the role of sensors in intelligent processing. The second workshop [2] addressed process models, artificial intelligence, and computer integration.

On August 30 and September 1, 1988, NIST convened a third workshop in this series. This one was comprised primarily of selected industrial specialists and was designed to define the specific materials processes upon which the NIST program should focus, and to discuss suitable approaches for accomplishing the work. This report documents the results of that workshop.

The workshop was held at the NIST facilities in Gaithersburg, Maryland. It opened with a plenary session on the morning of August 30, 1988, and closed with a second plenary session at noon the next day. Between the two plenary sessions the participants broke into three independent working groups concerned, respectively, with polymer processing, thermomechanical processing, and ceramics processing.

In the opening plenary session, Dr. Lyle Schwartz, Director of the Institute for Materials Science and Engineering (IMSE), welcomed the participants and highlighted the programs of the Institute. Dr. H. Thomas Yolken, Chief of IMSE's Office of Nondestructive Evaluation, then set forth the goals of the workshop. An ongoing intelligent processing program at NIST, which deals with the production of rapidly solidified metal powders, was then reviewed by

Dr. John R. Manning, leader of the Metallurgical Processing Group in IMSE's Metallurgy Division.

Following this intensive preparation, the three working groups adjourned to separate meeting rooms to pursue their respective assignments. A member of the NIST staff coordinated the activities and served as secretary of each working group; each group was chaired by an expert from the industrial sector. The following objectives were established for the working groups:

- to confirm the industrial importance of specific materials processes,
- to identify the principal technical problems that must be solved in order for the specified processes to be automated profitably, and
- to define the respective roles of industry and NIST in the development of intelligent automation technology for the processes.

Each of the working group chairmen presented the results of his group's deliberations at the closing plenary session. Also presented at the closing plenary session was a report on a separate workshop, dealing with hot isostatic pressing (HIP) of metals, which had been arranged by the Metallurgy Division. (A second workshop on HIP was convened several weeks later.)

The principal purpose of this report is to document the proceedings of the closing plenary session. By way of introduction, the body of this report opens with a description of the concept of intelligent processing and a review of its advantages. This is followed by the report by each of the three working groups and, then, a brief summary of the two HIP workshops.

The Workshop Organizing Committee is pleased to take this opportunity to thank the industrial participants in the workshop for their devoted efforts and valued advice. In particular, the contributions of the working group chairmen and those participants, who prepared and presented discussion papers, are sincerely appreciated.

References:

1. H. T. Yolken and R. Mehrabian, "A National Forum on the Future of Automated Materials Processing in U.S. Industry. Report of Workshop I. Role of Sensors," sponsored by: Industrial Research Institute and White House office of Science and Technology Policy, Committee on Materials, Working Group on Automation of Materials Processing, NBSIR 86-3341, 76 pp (May 1986).

2. H. M. Bloom and N. R. Kuchar, "A National Forum on the Future of Automated Materials Processing in U.S. Industry. Report of Workshop II. The Role of Process Models, Artificial Intelligence and Computer Integration," NBSIR 87-3544, 48 pp (April 1987).

WORKSHOP ORGANIZING COMMITTEE

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Mr. Joseph A. Carpenter, Jr.
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INTRODUCTION TO INTELLIGENT PROCESSING OF MATERIALS

Advanced materials are capable of providing outstanding or specialized properties, or combinations of properties, that cannot be obtained in conventional materials. These unique properties are the result of the sophisticated microstructure that is designed and built into the material. However, advanced materials generally require unusual processing operations in order to achieve their unique microstructures. Advanced materials also tend to be expensive because these operations are labor intensive and because of high rejection rates. In most cases, the raw materials account for only 5 to 10 percent of the total manufacturing cost for many advanced materials. Since the relationships between the processing parameters and the resulting material microstructures and properties are not fully understood or controlled, reproducibility of microstructure and resultant properties in these materials is often less than satisfactory. Rejection rates are much higher than desirable, and the unpredictable variability of properties prevents the designer from utilizing these materials to their full potential. For example, in the production of advanced ceramics, it is not uncommon to have 50 percent of the total manufacturing cost related to end-of-the-line inspection and rejected materials.

A promising direction toward overcoming these difficulties involves intelligent processing of materials. This approach controls the microstructure in contrast to conventional materials processing where process variables such as temperature and pressure are automatically controlled to preselected values. However, these process variables do not usually control the microstructures of advanced materials to a sufficient degree.

There are a number of significant benefits to be derived from the intelligent processing of materials. These benefits include a marked improvement in the overall quality of the product and a substantial reduction in subquality or rejected products. This automation concept is consistent with the broad-gauge, systematic approaches to planning and implementation now being undertaken in industry to improve quality. Since intelligent processing of materials involves building in quality rather than attempting to obtain it by inspection, there will also be a reduction in the expense devoted to post-manufacturing inspection and rejection. The flexibility to change manufacturing processes or material types quickly and economically is another potential benefit of intelligent processing. Finally, it is also reasonable to envision a shortening of the long lead-time needed to bring new materials from the development stage to mass production. Intelligent process control also holds promise for processing conventional materials, with similar benefits.

Intelligent Processing of Materials Facilities

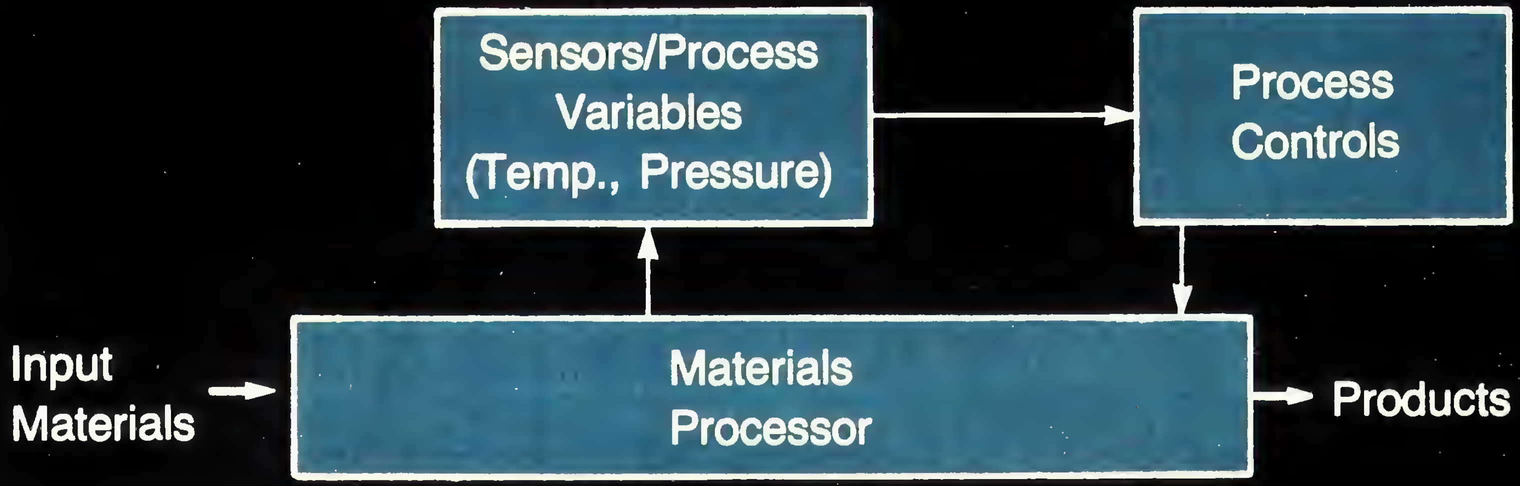
A conventional automated processing system is shown in Figure 1. Here automation involves utilizing sensors to monitor process variables such as temperature and pressure. The data from these sensors are compared automatically, and adjustments are made by the controllers to maintain preselected and preset values.

In the intelligent processing system shown in Figure 2, a new class of advanced Nondestructive Evaluation (NDE) sensors is utilized to characterize the evolution microstructure of the material in real time. Moreover, these data and data from the conventional process variable sensors are transmitted to a computerized decision-making system. This computer system transmits control signals based on the sensor data, a process model, and process data.

An intelligent processing of materials facility may be visualized as comprising four principal interconnected systems as indicated in Figure 2. One component of this intelligent processing system is the materials processor such as an apparatus to grow single crystals, a hot isostatic press to consolidate powders, a continuous metal caster, or a mold and autoclave for graphite reinforced polymer matrix composites. However, the processor may not necessarily be very different from other modern machinery used for conventional processing.

A second system, which is receiving increased attention, consists of advanced NDE sensors that can measure or monitor either directly or indirectly important microstructural characteristics of the material while it is being

A CONVENTIONAL AUTOMATED PROCESSING SYSTEM



Sensors/Process Variables (Temp., Pressure)

Process Controls

Input Materials

Materials Processor

Products

Figure 1

AN INTELLIGENT PROCESSING SYSTEM

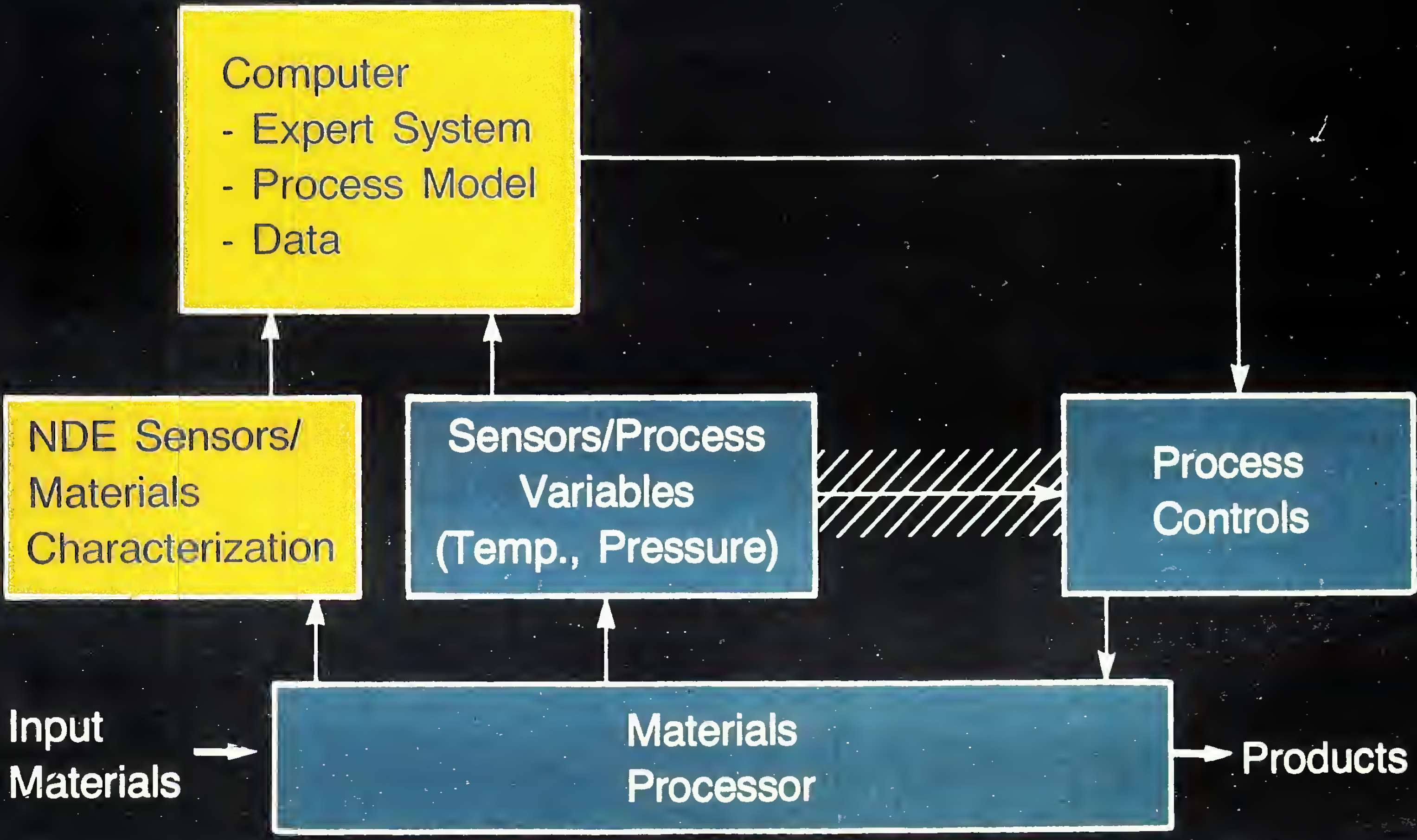


Figure 2

processed. These NDE sensors are based, for example, on ultrasonics, eddy currents, and optical fluorescence. The inspection of materials or products after processing or manufacturing is an essential feature of conventional industrial quality control. Inspection during processing is a new concept, and it is intended to help avoid the high costs of rejecting fully processed materials. Such inspection requires special sensors that can rapidly monitor meaningful materials characteristics reliably and nonintrusively, often in very hostile environments. Unfortunately, relatively few NDE sensors of this kind are readily available today. Conventional sensors for monitoring process parameters such as temperature and pressure are also a part of this system.

A third system of an intelligent processing of materials facility consists of conventional control devices such as valves, hydraulic cylinders, rheostats, pumps, and motors that regulate pressures, temperatures, velocities, and other processing parameters.

The final system closes the loop between the sensors and the process controls. In conventional control systems, sensing elements such as thermocouples or strain gages provide electrical signals that regulate temperatures or pressures via relays or switches with manually preset limits. If during production the material properties or the product characteristics begin to drift from the tolerable range, it is usually determined after the fact by destructive laboratory measurements. These measurements indicate to an operator the need for corrective action. The operator then manually readjusts the control limits in a way that is expected to return the process to the tolerable range. The efficacy of such adjustments is often less than adequate, depending on the skill and experience of the operator and the time delays before determining the need for corrective action and between the adjustments and their effects.

In intelligent processing of materials, the human operator in the fourth system is replaced by a computer with an applicable knowledge base. The most important element of the knowledge base is a model of the process, which is a mathematical representation of the interrelationships between the processing parameters, the material microstructure or characteristics, and the resulting material properties. Such models, which are rarely available, can be exceedingly complex and generally do not fully characterize the process. The theoretical and experimental knowledge base may be incomplete and, therefore, the development of a complete process model may not be possible. In this case, the formal knowledge base can be supplemented by (1) an expert system, i.e., rules of thumb that have been empirically devised by experienced operators, and (2) data (e.g., viscosity as a function of temperature) and records from past manufacturing runs that show how variations in the processing parameters have influenced materials properties. Replacing the human operator by this expert system can provide more precise and rapid control of the processing parameters and can lead to more uniform material of higher quality. A key feature of the expert system is that it can be enlarged and improved regularly, strengthening its capability for effective process control until little, if any, of the output of the facility needs to be rejected. At this point, productivity as well as quality will have been optimized.

With the present state-of-the-art, the enlargement of the expert system requires human intervention, at periodic intervals, to enter the data and other experience gained through the use of the facility. However, rapid developments that are being made in the new scientific discipline called artificial intelligence (AI) suggest that at some point in the future the processor will be capable of some measure of "learning". This means that the computer system will be programmed to enter into its knowledge base the data and results from successive processing operations as they take place. Until this can be accomplished, however, and perhaps thereafter as well, the most dramatic enlargements of the knowledge base may be expected to come not from accumulated processing data but from theoretical and experimental research that leads to more comprehensive and reliable process models.

REPORT OF THE WORKING GROUP ON POLYMER PROCESSING

Industrial Chairman
Joseph W. Miller, Jr.
Goodyear Tire and Rubber Co.

NIST Coordinator
Anthony J. Bur
Polymers Division

Intelligent processing is an ideal manufacturing concept that uses the full power of state-of-the-art measurement technology and real-time microprocessor control in conjunction with processing models to predict and control the materials properties and performance characteristics of a product. Bringing this concept into practice has become a goal of the polymer processing community because tighter controls on processing conditions are needed in order to produce polymer products which satisfy the demands of the marketplace. This demand for improved materials properties of existing polymers and for new high performance materials comes from many sectors of the marketplace but it is particularly strong in the automotive, aerospace, communications and computing, food packaging, industrial machinery, and defense industries. The use of polymers in hostile and demanding environments and the development of new polymer materials necessitate better understanding and improved control of the processing over a broad range of variables.

Polymer materials are made by combining one or more resins (plus fiber and filler for the composites and rubbers) with other additives in a process stream to produce products with specific chemical, morphological, mechanical, and electrical characteristics. Today, the large majority of polymer processing is controlled via on-line temperature and pressure measurements with set controls but without the measurement of other important processing parameters, without the benefit of feedback loops, and without an adequate model description of the process. Existing processing tools must be improved in order to meet the future challenges of developing intelligent processing technology and producing quality polymer products which are demanded by the end users of new high performance engineering plastics, polymer blends, elastomers, and advanced composites.

The importance of addressing these processing problems is underscored by the economic magnitude of the U. S. polymer industry. In 1987, 57 billion pounds of plastics, fibers, and rubbers were produced. Based on U. S. Department of Commerce figures, the value of plastic products made from this raw material is approximately $80B. The fraction of the total that corresponds to high performance polymer products is approximately 25 percent--an estimate obtained by summing those markets which are impacted by high performance polymers.

The working group on intelligent processing of polymers consisted of twenty representatives from a cross section of the polymer industry: resin manufacturers, machinery manufacturers, processors, and end users. An attendance list is given in Appendix A. Considering the broad range of interests of the participants, the group did not focus on a particular processing activity, but rather sought to uncover that common ground which cut across the interests of the various segments of the industry. The objectives of the working group were:

- to identify important processing methods that would benefit from automation and improved technological understanding of the process;
- to isolate important generic problems that limit automation and productivity and impede the processing of high quality polymer materials;
- to identify technical and scientific barriers that must be overcome in order to solve processing problems;
- to identify concepts, measurement techniques, and other tools that can be applied to these problems; and
- to forecast the directions and emphasis of future polymer processing activities, define the respective roles of NIST and the industrial sector in these undertakings, and establish communication between the NIST laboratories and the U. S. processing community.

The meeting was organized to elicit information about those problems that have the broadest impact on the industry and to isolate the generic issues that can be attacked by ongoing collaborative NIST/industry programs. This information is not only important to many processing engineers and scientists and to directors of research and development programs, but also will assist NIST in defining materials processing programs that promise significant benefits to the industry.

Workshop Program

The workshop, which extended over one and a half days, was divided into several parts. The first morning session was a plenary session attended by participants from concurrently scheduled working groups on polymers, ceramics, and thermomechanical processing. After the plenary session, the polymer working group convened and followed an agenda, shown in Appendix B, consisting of presentations by NIST and industry representatives and a summary discussion session on the morning of the second day. A final plenary session was held during which the industry chairmen from the individual working groups summarized their workshop discussions.

In addition to presentations by ten speakers at the polymer working group, a questionnaire was distributed for the purpose of obtaining opinions from the participants on the current state of polymer processing, the important technical barriers that impede automation, and the current and future needs of the polymer processing industry.

Summary of Working Group Presentations NIST Polymer Processing Program

The objectives of the NIST polymer processing program are to develop new on-line measurement technology based on optical and fluorescence methods, to develop new models and improve existing models of polymer processes that utilize the new measurement technology, and to join with U. S. industrial partners in collaborative polymer processing programs that address the problems of this industry. A working hypothesis for the NIST program is that knowledge of processing conditions at the molecular and microscopic level are needed in order to predict and control the properties of the final product.

Fluorescence measuring equipment is being developed in order to monitor relevant processing parameters that, for the most part, are not being measured on today's processing equipment, such as, the quality of mix of product ingredients, non-Newtonian viscosity, molecular orientation, velocity, flow instabilities, and intersegmental penetration at the molecular level. Using fluorescence spectroscopy for these measurements requires the doping of a fluorescently active chromophore into the processing ingredients. Since the chromophore is added at dopant levels it does not affect the material properties of the final product.

Fluorescently active chromophores are chosen in accordance with their sensitivities to particular processing parameters. Information regarding the behavior and spectra of many chromophores can be obtained from existing data and literature, but the application of this information to the measurement and control of polymer processing parameters is a new concept that will require significant development. The ultimate application is to use fluorescence spectroscopy as an on-line nondestructive probe for real-time monitoring of processing parameters. Optical fibers can be used to transmit and receive optical signals to and from monitoring sites in the processing equipment. Optical energy that is sent to the monitoring site excites nearby chromophores which respond by radiating characteristic spectra. From an analysis of the spectra, processing conditions at the probe site can be determined.

This experimental program is in progress. The feasibility of the measurement technique has been demonstrated for two processing parameters: the quality of mixing of product ingredients, and viscosity under zero shear conditions. A future objective is to use the information obtained from these new fluorescence measurements to assist in the development of processing models that incorporate molecular and microscopic concepts and their relationship to the materials properties of the final product.

Active collaboration with industry on the NIST polymer processing program is invited and encouraged.

Industrial Perspectives

Nine presentations were made by industrial participants representing the interests of original equipment manufacturers, resin manufacturers, and processors. A number of common concerns and processing problems were expressed. These recurring topics included: measurement and control philosophy, on-line measurement and analysis tools, process models to include viscoelasticity and materials structure/property relationships, theoretical and fundamental understanding of processes, theoretical improvements in rheology, the complexity of new materials, inadequate materials database, and production and manufacturing problems.

Measurement and Control Strategy

Two basic concepts concerning measurement and control of polymer processing were presented:

- Control of a process parameter first requires its measurement ('You can't control what you can't measure');
- There is no point measuring and controlling a parameter that is not important.

These two items were revisited throughout the working group meeting because they capture the situation and the dilemma which polymer processors face today. New materials and new processing techniques demand ever expanding measurement capability and theoretical understanding. New measurement techniques beyond the usual pressure and temperature measurements are needed to monitor and understand the process, but precisely what those measurements should be is often an unanswered question because we do not fully understand the physics, rheology, and chemistry of the process. Well conceived process models, which establish a relationship between process parameters and desired materials properties of the final product, can illuminate parameters that are important. On the other hand, the application of new measurement technology can highlight an important process parameter and direct the observer to a new model concept or a method for improving an existing model. Thus, measurement technology and model development are best utilized in a complementary relationship.

Measurement and Analysis Tools

An extensive list of measurement and analysis requirements was accumulated during the working group discussions. The need for on-line, rapid observations was a common criterion. On-line instrumentation must be industrially hardened or "robust" so that it can survive the manufacturing environment. While all participants agreed on the value of utilizing closed loop feedback control, there was unanimous agreement that many preliminary goals must be achieved before feedback control is realized. Preliminary goals such as developing new measurement technologies and models, and improving our rheological understanding of new and complex materials are formidable challenges which, when overcome, will yield great opportunities for improved product quality and productivity.

The list of on-line measurement needs was divided into five categories: rheology, morphology, chemistry and chemical analysis, thermodynamics, and physical. In most cases the perceived needs were not supported by the rationale of a proven process model, but are viewed as important because of institutional experience or because of the existence of a working hypothesis. The stated need for new measurement capability varied in accordance with the process and the product specifications. However, many measurement requirements have common application to several processes. An expressed general need was for accurate measurements at high temperatures. In summary, workshop participants perceived current requirements for new on-line measurement technology to be:

- Rheology: viscosity, residual stress, velocity, and flow characteristics;
- Morphology: molecular orientation, fiber distribution and orientation, particle size distribution, quality of mix of ingredients, polymer microstructure, degree of flocculation;
- Chemistry and chemical analysis: infrared spectra, chemical reaction kinetics;
- Physical: density, electrical properties, spatial distribution of temperature, nucleation;
- Appearance: color, surface smoothness, and spatial dimensions.

Ideally, the set of measurement requirements for a given process will be determined by a fundamental understanding of the process via a tested model which emphasizes the important process parameters. In the absence of a working model, an empirical and/or statistical approach must be used to establish parameter importance.

Several processes were highlighted because of their demanding measurement requirements: RIM (reaction injection molding), RTM (resin transfer molding), and reactive extrusion of elastomers. RIM and RTM are processes used extensively by the automobile industry in production and development programs. In order that RIM and RTM yield quality parts, residual stresses must be minimized, part dimensions must be controlled to very tight tolerances, and the surface must satisfy class A specifications. Reactive extrusion will require on-line monitoring of chemistry and rheological properties and the control and monitoring of temperature distribution within the process stream.

Theoretical and Model Development

The general consensus of the workshop was that many new model developments and adjustments to existing models must be undertaken in order to meet the challenge of producing quality products for today's market. While many views were presented concerning the need for new models, it was also pointed out that optimum use of existing models may produce beneficial results. The more fundamental problems, however, concern an adequate description of new processes and the understanding of the rheology of new materials which are continuously introduced into the market. A major shortcoming of polymer processing models is that they concentrate on the process while treating the polymer as a simple viscous fluid. The result is that, to a first approximation, the process can be described, but the materials properties of the product are not described or predicted. Strong support for materials properties oriented models was expressed by the working group. The solution to today's processing problems depends on construction of models that include the following:

- Viscoelastic materials properties;
- Relationship between product microstructure and process parameters;

- Modern theory of viscoelastic flow in three dimensions;
- Mass, momentum, and energy transfer;
- Non-isothermal conditions;
- Chemical kinetics (for reactive processing);
- Phase morphology;
- Surface finish and spatial dimensions;
- Process instabilities.

The widest application of new model developments will ensue if they are generic rather than process specific.

Materials Database Requirements

Utilization of new model descriptions and the creation of empirical processing/product properties relationships will require an expanded materials database. In general, data at high temperatures are needed. The most frequently mentioned data base requirements were structure/property relationships and rheological properties.

Production Processing Problems

Several workshop participants pointed to practical manufacturing problems which pose impediments to the development and application of intelligent polymer processing. Problems such as product proliferation, raw material inconsistency, short production runs, and scale-up difficulties due to equipment differences present inconvenient obstacles which can thwart quality production runs. Intelligent processing schemes must be broad enough in scope to address these issues.

Responses To Questionnaire

A questionnaire was distributed to the participants for the purpose of inviting their comments to eight questions regarding the current and future state of polymer processing. Responses to the questionnaire, shown in Appendix C, are summarized as follows:

1. Processing Methods. In answer to this question a total of 17 processing operations were listed as the most important polymer processing methods

used today. In the order of the number of times mentioned the top five are:

- Injection molding
- Extrusion
- Thermoset composites
- Blow molding
- Mixing

2. <u>Automation</u>. A total of 16 processes were identified as methods that will benefit most from intelligent processing technology. The top six in the order of the number of times mentioned are:
   - Injection molding
   - Extrusion
   - Compression molding
   - Reaction injection molding
   - Thermoset composites
   - Mixing

3. <u>Technical Barriers</u>. The list of scientific and technical barriers to be overcome in order to develop intelligent processing technology was divided into three categories: measurements, process models, and fundamentals. The most mentioned concepts were:
   - New and improved on-line measurements:
     + Rheological properties
     + Polymer microstructure and morphology
     + Chemistry
     + High temperature measurements
     + Robust measurement sensors
   - Better process models:
     + Generic rather than equipment specific
     + Include kinetics/reaction descriptions
     + Develop methodology equivalent to chemical industry
     + Expert systems
   - Fundamentals:
     + Phenomenological understanding and quantification of what is happening in the process

4. Models and Concepts. Two needed improvements in present day models and concepts were mentioned most often:

   - Need to include viscoelasticity in models
   - Relate product microstructure to process parameters

   Also recommended were:

   - Add polymer blends to models
   - Add description of reaction to models (for reactive processing)
   - Add non-isothermal conditions
   - Consider non-melt portion of extruder
   - Incorporate surface waviness
   - Incorporate melt fracture

5. Measurement Technology. The total list of materials properties and processing parameters which need to be measured in real-time was given as:

   - Rheological parameters
   - Chemistry
   - Morphology
   - Quality of mix
   - Molecular orientation
   - Particle size distribution
   - Dimensions
   - Electrical properties
   - Nucleation
   - Color
   - Degree of flocculation
   - Density

6. Materials Properties Database. Respondents to this question recommended an expanded materials database to include:

   - Data at high temperatures
   - Structure/property relationships
   - Rheological properties
   - Specific heats
   - Diffusivities
   - Upper and lower critical solution temperatures for polymer blends

- Flory-Huggins parameters
- Composites characterization
- Biaxial viscoelastic properties at large deformations

7. Processing Trends. New or emerging processing methods in the next 5-10 years were predicted by the respondents. In the order of the number of times mentioned these were:
    - Resin transfer molding
    - Sheet molding compound to be replaced by injection molding
    - UV, microwave, electron beam curing
    - Batch processing to be replaced by continuous processing
    - Liquid molding of structural thermosetting composites
    - Pultrusion
    - Large parts via injection molding
    - Higher temperature processing
    - Thermoplastic sheet forming/stamping
    - Hollow gas molding
    - Co-injection molding
    - Net shape processing
    - Fusible core molding

8. New Materials. Workshop participants forecast that new classes of polymers will be processed in the next ten years. Among the nine materials mentioned, three received most attention:
    - Liquid crystal polymers
    - Interpenetrating networks
    - In-situ reinforced polymers

    Also mentioned:
    - Higher T_M thermoplastics
    - Intractable materials, e.g., ultra-high molecular weight polyethylene and polyimides
    - Highly filled thermoplastics
    - Continuous filament reinforcement
    - Recyclable materials
    - Organic/inorganic composites

SUMMARY

The subject matter of this working group was broad in scope. As such, the participants emphasized the many concerns of their production requirements. Although these specific interests included those of equipment manufacturer, resin supplier, processor, and end user, the working group participants were able to isolate common concerns and to recommend the directions of research and development that are needed to develop intelligent processing technology. Continuing themes and concepts that punctuated the discussions, presentations, and responses to the questionnaire underscored the necessity to improve measurement technology, process models, and fundamental understanding of the physics, rheology, and chemistry of polymer processes. Measurement technology must be robust and advanced so that rheology, material microstructure, and chemistry can be measured in real time. Existing models must be improved or new models constructed that include viscoelastic behavior, the relationship between product microstructure and process parameters, non-isothermal conditions, chemical reactions, and three dimensional descriptions. Advances in the fundamental understanding of polymer processing are needed in the areas of rheology of new materials, the thermodynamics and kinetics of the process, and the structure/property relationships of polymer materials.

APPENDIX A

ATTENDANCE LIST

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The following from the Polymers Division of NIST were in attendance:

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Donald L. Hunston

Steven C. Roth

Leslie E. Smith

APPENDIX B

AGENDA FOR WORKING GROUP ON INTELLIGENT PROCESSING OF POLYMERS

National Institute of Standards and Technology
Gaithersburg, MD 20899

Wednesday, August 31, 1988 (Rm A Administration Bldg.)

11:00 - 11:10 AM: Welcome and Introduction, Anthony Bur (NIST)

11:10: Workshop Session begins, Joseph Miller (Goodyear), session chairman

11:10 - 11:35 AM:
Anthony Bur (NIST), "The NIST Polymer Processing Program";

11:35 AM - 12:05 PM:
James Tshudy (Armstrong), " 'You Can't Control What You Can't Measure' - Armstrong's Needs in Automatic Polymer Processing and Control";

12:05 - 12:35 PM:
Vic Thalacker (3M Co.), "Melt Processing of Polymer Materials - Directions and Needs";

12:35 - 1:00 PM:
Tony Dean (Ferro Corp.), "Needs for the Compounding Industry";

1:00 - 2:00 PM:
Lunch (Senior Lunch Club line and Employees lounge)

2:00 - 2:30 PM:
John Theberge (LNP Eng. Plastics), "Processing of Fiber Reinforced Plastics to Optimize Performance";

2:30 - 3:00 PM:
Alan Murray (Ford Motor Co.), "Fit and Finish of Thermoplastic RTM and RIM Body Panels";

3:00 - 3:25 PM:
Walter Schrenk (DOW Chemical), "Unresolved Problem Areas in Multilayer Co-extrusion";

3:25 - 3:50 PM:
Dean Reber (Cincinnati Milacron), "Polymer Processing from the Machinery Manufacturer's Point of View";

3:50 - 4:05 PM:
Coffee Break

4:05 - 4:20 PM:

Pawan Handa (Goodyear), "Reactive Elastomer Processing";

4:20 - 4:35 PM:

Bruce Whipple (Mobay), "Variation of Product Properties as a Function of Machine Parameters and Resin Variables";

4:35 - 5:15 PM:

Discussion

Thursday, Sept. 1, 1988

8:45 - 10:15 AM:

Discussion regarding final report of working group:

- Future emphasis and directions of processing R&D
- Measurement needs
- Manpower needs
- The roles of NIST and industry, etc.

10:15 - 10:30 AM:

Coffee Break

10:30 AM - 12:30 PM:

Convene in plenary session to present final oral report

Workshop adjourns at 12:30 PM

Optional

12:30 - 1:30 PM:

Lunch (NIST cafeteria)

1:30 - 2:30 PM:

Laboratory tour of polymer labs for those who wish.

APPENDIX C
QUESTIONNAIRE
WORKING GROUP ON INTELLIGENT PROCESSING OF POLYMERS

Please answer questions in accordance with your experience and your company's interests.

1. Processing: From your experience and your company's manufacturing activity, what are the most important polymer processing methods being used today?

2. Automation: What processes do you think will benefit most (in terms of improved process control, product quality, and productivity) by the development of intelligent processing technology?

3. Technical Barriers: Considering the process methods listed in question 2, what scientific and technical barriers must be overcome in order to develop intelligent automated processing for these methods?

4. Models/Concepts: Considering the process methods listed in question 1, what improvements in the present day model descriptions of these processes are needed? Do models describe the process adequately? Do models predict materials properties of the product?

5. Measurement Technology: In addition to accurate measurements of temperature and pressure, what materials properties and processing parameters would you want to measure in real-time during processing? What measurement parameters will be needed for the development of intelligent processing technology?

6. Materials Properties: Considering the polymers which your company processes, what improvements in the materials properties database are needed for improved processing control and productivity?

7. Processing Trends: In the next 5 or 10 years, do you foresee processing activity shifting to new or emerging methods? If so, what are these methods?

8. Materials: What new classes of polymer materials do you predict we will be processing in the next 10 years?

REPORT OF THE WORKING GROUP ON THERMOMECHANICAL PROCESSING

Industrial Chairman
John M. Chilton
Bethlehem Steel Corporation

NIST Coordinator
Yi-Wen Cheng
Fracture and Deformation Division

Thermomechanical processing (TMP) is the thermal treatment and plastic forming of metals using controlled deformation and temperature schedules to achieve the desired properties and dimensions in the finished product. TMP includes the primary metal working processes used by the metals producing industry such as rolling and extrusion, and secondary processes used by metal fabricators such as forging and forming. Currently, industrial process-control practices are based primarily on empirical models. However, industry is beginning to develop scientific-based models as a basis for improved design, automation, and control. Development and transfer of certain building-block technologies are needed to support industrial programs.

The working group on thermomechanical processing consisted of eight industrial representatives, one from academia, and three members of the NIST staff. See Appendix A. The working group's activities opened with the following two presentations which served as an excellent stimulus to the discussions which followed:

"Thermophysical Property Data and Sensor Technology for TMP," presented by Fred Schwerer, Alcoa Laboratories (coauthors: Owen Richmond and Larry Lalli).

"Product/Process Modeling for TMP," presented by Dhani Watanapongse, Inland Steel Company.

In addition, copies of the following paper were distributed to the attendees as a further resource:

"A Hot-Deformation Apparatus for Thermomechanical Processing Simulation," by Yi-Wen Cheng and Harry I. McHenry, NIST.

Sensor Development

Improved sensors and measurement systems are required for the measurement of both process parameters and product characteristics. These measurements must provide accurate and reliable data while performing in the hostile operating environments associated with primary metal production.

With regard to process parameters, real-time measurements are essential and direct measurements are preferred. The example discussed in detail was temperature measurement. At the present time, temperature is inferred from rolling mill loads sometimes as a substitute for direct measurement by temperature sensors. The direct measurement of temperature with a non-contacting device such as an infrared sensor is often unreliable because the measurement is affected by operating environments such as steam, vapor, etc., and the emissivity of the material which, in turn, is a function of surface condition. Newer techniques such as ultrasonic methods are still under development.

Most of the sensor discussion concerned measurement of material properties during processing. These include:

Mechanical properties
Grain size
Precipitation hardening
Transformation temperature
Degree of recrystallization
Crystallographic texture
Long-term durability

For many of these measurements both on-line and rapid off-line systems would be useful. As for the case of temperature measurement, it is important to consider the ultimate operating environment of the measurement system.

In Dr. Schwerer's presentation, a hierarchy of sensor requirements was outlined. He emphasized the need to base the measurement requirements on process models. The first-order sensor requirement is a laboratory-based system designed to improve understanding of the process. The second level of measurement is a rapid off-line system to provide measurements for statistical process control, the design of experiments, and process optimization. The ultimate sensor is an on-line measurement used for automatic control.

Sensor needs for material characterization include direct and indirect inference of microstructures, mechanical compliances, anisotropy, and strength. For these sensors, the general development requirements include a process/material model which correlates the measurement with the property, a quantitative understanding of the limitations of the correlation, and an assessment of gage capability and integrity. In addition, the sensor must be designed and calibrated for a specific application. This means that the specific operating parameter must be measured, the secondary variables must be restricted, and the sensor must work in the plant environment.

Process Models

A process model describes the conversion of incoming material of specific composition to a product with desired dimensions and properties. Models are used to facilitate process design, to assess measurement and sensor requirements, and to quantify control strategies. Currently, with few exceptions, models used in industry are qualitative and empirical. Research is needed to improve these models and to develop new ones based on physical principles. The overall model includes several components:

1. A thermal model to predict heating and cooling profiles;

2. A deformation model, including tool/workpiece interface, equipment responses, and constitutive characterization of the product at a given temperature, to predict product geometry such as gage, shape, crown and width;

3. A metallurgical model to predict the evolution of material properties and microstructure;

4. A failure criterion to indicate the process/material limits.

Significant technical challenges were discussed in each of these areas with the implication that an integrated process model would be a long-term goal. It was recognized that the development of some physical models needs long-term research, such as in the area of interface and constitutive modeling. In some cases, however, the basic physical principles are understood and simply need to be codified into practical models, such as the evolution of microstructure and properties in steel plate rolling. It was also recognized that current process models typically require lengthy numerical calculations; thus, the potential role of these physical models for intelligent process control is limited. Simplified models with fewer calculations have to be developed for intelligent process control.

Process models are more process/product specific than sensors. The details of process/property models are more or less proprietary. Thus, it was recommended that NIST concentrate on the development of fundamental understanding and of generic (process and product independent) models of constitutive and tool/workpiece interface behavior instead of specific and global process/product models. The development of process models will proceed from simple qualitative cause-and-effect reasoning to parametric, statistical modeling with the ultimate goal of physically based modeling.

Material Property Data

Material property databases are needed to fine tune and verify the process models. Standard reference data are needed on thermophysical properties (such as conductivity, elastic moduli, thermal expansion, and specific heat) at elevated temperatures and thermomechanical properties (such as yield strength and flow characteristics) as a function of strain rate and temperature. These data need to be quantitatively related to the microstructural state of the material (i.e., solutes, precipitate size and distribution, grain size and texture, etc.). In many cases, improved measurement methods are needed to develop these databases. In order to provide a sufficiently comprehensive database, physical models will be needed to permit extrapolation and interpolation of the data.

System Integration

The integration of sensing, process modeling, and computer control was discussed at length. A multidisciplinary approach must be coupled with detailed knowledge of the products and processes involved. Concern was expressed regarding the ability of a nonindustrial research laboratory to develop a generic approach which could be used for intelligent control of TMP. The peculiarities of specific pieces of equipment and the wide range of product variations make the integration process a difficult one involving the proprietary knowledge and expertise of the company involved. In addition, concerns were expressed over the feasibility of intelligent control of certain complicated high-speed processes, such as hot strip rolling. In these cases, it may be necessary to take an incremental approach to intelligent control of subprocesses with particular attention being given to minimizing interaction time required between sensors, process models, and control systems. However, increases in computer capabilities and speed will increase the range of future applications.

National Facility for TMP Research and Development

Generation of basic property databases and constitutive and interface models to support the development of intelligent control of TMP requires experiments using versatile multipurpose equipment that can simulate the temperature and deformation histories of representative processes such as rolling, forging, and extrusion. The equipment must be fully instrumented to measure and control the experimental variables. This type of equipment is not generally available to American industry and is too expensive for many individual companies. Thus, the participants recommended that a national facility be established for TMP research. This facility would include the multipurpose equipment and the associated instrumentation to verify the research results. It would also provide a test bed for some phases of the sensor development projects. The participants expect that industry would make use of the facility and would separately provide the opportunity for full-scale trials on industrial production facilities.

SUMMARY

Workshop participants agreed that improvement in design, automation, and control of TMP will benefit the industry in reduced cost, improved quality and uniformity of existing products, reduced lead time for development of new products, and the potential for grade consolidation. Currently, industrial TMP process-control practices are based primarily on empirical models. Improvement in automation and control can be achieved by developing the following building-block technologies:

- sensors and measurement systems for process parameters and for product characteristics:
- improved global process models which include models for evolution of material properties and practical descriptions of tool/workpiece interface conditions;
- reference data on thermophysical and thermomechanical properties.

Each of these building-block technologies is of immediate importance to industry, and their integration into intelligent control systems is a suitable goal for the long term.

They recommended that NIST play the following roles to help industry in achieving improved design, automation, and control of TMP:

1. To conduct research directed towards developing the building-block technologies that industry can use to improve their TMP practices.

2. To facilitate the development and transfer of TMP technology from all sectors of the research community to the metals-producing industry.

3. To establish and operate a national facility for TMP research and development.

4. To develop sensors and measurement systems, process models, and reference data for direct use by industry which would be applicable to a broad class of TMP problems.

Participants represented companies with a wide range of in-house capabilities. In addition, the participants expressed differences in the extent that their companies consider TMP process models and supporting technology to be proprietary and to provide competitive advantage. All agreed with Items 1 and 2 and with Item 3 to the extent to which it supports building-block technologies and on the basic work on constitutive models and on the tool/workpiece interface in ideal systems. Item 4 and equipment-specific work on Item 3 were not supported by all participants as appropriate roles for NIST.

All participants expressed support for and a willingness to collaborate in NIST programs on TMP. Industry will assist program planning and review, and will provide technical support, expertise, and specialized equipment.

APPENDIX A

ATTENDANCE LIST

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REPORT OF THE WORKING GROUP ON CERAMIC PROCESSING

Industrial Chairman
Thomas J. Whalen
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NIST Coordinator
Joseph A. Carpenter, Jr.
Ceramics Division

Representatives of nine industrial firms and seven NIST staff members participated in the discussions of the Ceramics Working Group; an attendance list is attached. Five of the industrial firms (GTE, Dow, Norton, Carborundum, and Coors) are primarily suppliers of ceramic materials and components; three (Chrysler, Deere, and Ford) are mainly users; and one (Garrett) is both a supplier and user. Based on these discussions, the program outlined below is suggested for a NIST initiative on intelligent processing of ceramics.

Details of Discussions

The objective of the proposed program is to demonstrate the potential technical and economic viabilities of the concept of intelligent processing of advanced ceramics to the point that U.S. industry would feel that the technical and economic risks had been reduced enough to consider further development work aimed at the eventual adoption of the concept in industrial ceramic processing. The U.S. advanced ceramics industry's annual sales, currently on the order of $4-5 billion, have been variously estimated to have the potential to increase to the $100 billion range in the 1990-2010 era provided ways can be found to cost-effectively produce reliable components. Assuming a knowledge base exists unequivocably relating the structure of a component to its in-service reliability, intelligent processing will primarily increase the cost effectiveness by reducing reject rates through in-stream changes so that out-of-specification material can still be brought to the desired structure, by eliminating the time an energy spent on further processing of irretrievably out-of-specification material, and by timely detection of drift-out of process conditions. In the latter case, intelligent processing will enhance statistical process control now being used by some parts of the U.S. ceramics industry.

Work will concentrate on intelligent processing for densifying a few representative ceramics as opposed to processing a single ceramic from starting materials to end product. The industrial participants felt that the latter approach would be too extensive for the level of funding likely to be available and that meaningful results, sufficiently convincing for industry to consider adopting the technology, could be obtained by the first approach. Powder production and characterization and injection molding were considered, but the industrial participants strongly made the point that, despite many years of research in academia, government and industry, unacceptable variabilities in the properties of densified bodies remain. SiC (silicon carbide), Si_3N_4 (silicon nitride), and Al_2O_3 (alumina) were suggested at the workshop as the representative ceramics, as they are used or being considered for use in functional as well as structural applications, making wider the potential impacts of studies of their intelligent processing.

The studies will concentrate on novel diagnostic techniques and mechanistically-based process models for densification at high temperatures both with and without applied pressure; the computer and control technologies required to complete an intelligent processing system will be of secondary importance. It was strongly suggested that the NIST program emphasize diagnostics for structures and models at the microstructural level and not just at the phenomenological level currently being emphasized today by industry and programs such as the Department of Energy's Ceramics Technology for Advanced Heat Engine Program. Despite the emphasis on the microstructural level, priority will be given to developing information that will lead to practical process diagnostic and control techniques that could be used in an industrial setting; specimens studied will be as large as possible in order to more closely resemble pieces that would be processed in industry.

Important microstructural parameters as well as gross features of the bodies will be measured by techniques based upon various phenomena. Examples of microstructural parameters that might be measured include size and shape of grains, pores and phases, and compositional and strain inhomogeneities. Gross features such as size, shape, and surface condition might also be measured. Phenomena suggested for study as the bases of the measurement techniques included ultrasonic velocimetry and attenuation; electrical and electronic properties such as capacitance, ac spectra, and microwave interference; eddy currents; thermal waves; x-ray tomography, topography, and diffraction; nuclear magnetic resonance; small angle neutron scattering; and fluorescent dyes for powder preparation.

It was proposed that the work be performed at NIST with inputs from industry on the program and, where appropriate, active participation. Universities will be invited to provide expertise, students, and linkages to other programs. There was a consensus achieved at the workshop that industry would directly implement the results if the program is reasonably successful. It was felt that an industry-government-academia consortium will have the best synergism and prospects for success.

SUMMARY

Workshop participants expressed support for NIST launching a program in intelligent processing of advanced ceramics for structural and functional applications. The U.S. ceramic industry and the nation will benefit substantially from such a program by reduced cost and improved quality and reliability of ceramics. Improvement in automation and control can be achieved by advancement in the following technologies:

- sensors and measurement systems for powders, compacts, and product properties;
- process models and understanding; and
- reference data on thermophysical and thermochemical properties of various systems.

It was also recognized that different products' properties would require different sensors and property measurement systems; properties important to one product may not be important to another. The industrial participants recommended that NIST concentrate its efforts in one or two unit processes such as pressureless sintering or hot isostatic pressing (HIPing). From an economic point of view, if a breakthrough can be achieved in pressureless sintering, the impact would be the greatest. There is sufficient interest among the individual participants in HIPing for high-performance materials.

It was agreed that industry would support, participate, and help guide the program at NIST.

APPENDIX A
ATTENDANCE LIST

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REPORT OF WORKSHOPS ON THE HOT ISOSTATIC PRESSING OF METAL ALLOYS

Robert J. Schaefer
Metallurgy Division
National Institute of Standards and Technology

The Metallurgy Division is currently carrying out a project in which the concepts of intelligent processing of materials (IPM) are being applied to hot isostatic pressing (HIP). This project, jointly supported by DARPA and NIST, is specifically directed toward HIP of titanium aluminides and titanium aluminide matrix composites. These are materials that have low densities and high strength at elevated temperatures, making them outstanding candidates for advanced aerospace applications. They are, however, difficult to fabricate and HIP of powder materials or composite tapes represents one of the more promising methods for producing them in near-net shape. The project is being carried out in collaboration with the BDM Corporation, which is developing the control software.

As part of this project two workshops have been held in which the project was discussed with members of the HIP community. Attendees included manufacturers of HIP equipment, operators of commercial HIP facilities, academic researchers studying HIP, and titanium aluminide specialists. The first workshop was devoted to a presentation of the project plan and IPM concepts, and a discussion of industry practices and problems. The program and the attendance list for this workshop are attached as Appendices A and B.

The participants in the workshop generally felt that the information provided by real-time density or microstructure sensors, as developed by this project, would be extremely useful. The modification of a temperature-pressure cycle on the basis of such information during a production run would, however, require a change in outlook toward process specifications. The current practice is generally to establish a temperature-pressure cycle during a development stage and then to carry out production runs using conditions duplicating this cycle within specified limits. A successful IPM approach must, therefore, demonstrate that a superior product can be made by a variable process cycle.

In the second workshop, held September 22, 1988, progress toward the development of components of an intelligent controller for the HIP was presented. Eddy current sensing of the diameter of cylindrical samples has been shown to be highly successful. Eddy current sensors for other geometries are also being considered but have not yet been demonstrated. Experiments with in-situ ultrasonic microstructure sensors have been started. The process model for powder densification is based on the work of Professor M. F. Ashby (Cambridge University) who is a collaborator on the project. The workshop members were shown how the Ashby model is being modified to apply more directly to real HIP cycles, and how it has been tested by comparison with densification data obtained for high-purity copper. The special problems encountered in applying such a process model to titanium aluminides or composite materials were also discussed. Finally, development of the control system software by BDM was presented, and a HIP run of a TiAl sample with eddy current density sensing was demonstrated in the laboratory.

During discussions at the end of the second workshop there was general agreement that the availability of a good density sensor could greatly shorten the development time required to optimize the process cycle for new alloys.

Consolidation of metal powders currently constitutes a relatively small fraction of the commercial HIP business, with larger volumes of work being concerned with ceramics or healing defects in castings or used parts. Intelligent processing concepts are likely to find their most fruitful applications in the manufacture of more advanced materials such as metal-matrix composites, where the controller system may need to be able to compensate for variations in the starting materials.

APPENDIX A

Hot Isostatic Pressing Workshop
December 18, 1987
BDM Corporate Office, Arlington, VA

This workshop was organized to introduce members of the industrial HIP community to a new program in which the concept of intelligent processing of materials (IPM) will be applied to HIP of TiAl. The program would be sponsored by DARPA and NIST and carried out by NIST and BDM.

Dr. Phillip Parrish gave an introductory overview of the DARPA program in IPM, a major objective of which is to reduce the time lag between R&D and production for advanced materials.

NIST participants then described how sensors installed within a HIP chamber can be used to monitor the densification process and compare the measurements with a process model. A sensor was described that is capable of operating in the HIP environment and that can measure the diameter of cylindrical samples. The densification model to be used in the program is that of Professor M. Ashby of Cambridge University, who will collaborate with the program under a subcontract.

BDM participants described the application of intelligent processing to HIP, in which an intelligent controller network would direct modifications of the HIP cycle to correct for deviations from the planned densification rate.

A series of questions relating to prevailing practices and attitudes within the HIP community were then discussed.

APPENDIX B

Attendance List

From the HIP community

Dave Peltier
ASEA Autoclave Systems

Dan Watkins
Carnegie-Mellon University

Mike Conway
Conaway, Inc.

Bill Eisen
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Charles Pierce
Howmet

Peter Price
IMT

Stephen Kampe
Martin Marietta

Shankara Sastry
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Arnold Bowles
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From DARPA

Phillip Parrish

National Institute of Standards and Technology

Roger Clough
Richard Fields
Ward Johnson
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From BDM

Roger Geesey
Brian Kushner
Rick Preston
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GAITHERSBURG, MD 20899

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Office of Nondestructive Evaluation
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10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

Intelligent processing of materials has been established as a major new program area in the Institute for Materials Science and Engineering, National Institute of Standards and Technology (NIST, formerly the National Bureau of Standards). The goal of the program is to develop some of the generic scientific and technological bases for intelligent processing and, by means of selected pilot or demonstration projects, to encourage American industry to pursue and to adopt this powerful new approach to materials processing.

In developing this new program, NIST cooperated in organizing two national workshops in 1985-86 to help identify the principal industrial needs in this field of technology and to solicit guidance for program planning activities. The first of these two workshops was concerned with the role of sensors in intelligent processing. The second workshop addressed process models, artificial intelligence, and computer integration.

On August 30 and September 1, 1988, NIST convened a third workshop in this series. This one was comprised primarily of selected industrial specialists and was designed to define the specific materials processes upon which the NIST program should focus, and to discuss suitable approaches for accomplishing the work. This report documents the results of that workshop.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

Intelligent processing of materials, sensors; NDE, process models

13. AVAILABILITY

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